8-10 November 1989

Proceedings
AUSTRALIAN INSTITUTE OF NUCLEAR SCIENCE AND ENGINEERING

6TH AUSTRALIAN CONFERENCE ON NUCLEAR TECHNIQUES OF ANALYSIS

LUCAS HEIGHTS N.S.W.

Wednesday 8th November 1989

Sessions commence 10.30 a.m.
Conference Luncheon 12.40 p.m.
Sessions conclude 6.30 p.m.
Invitation Dinner 6.30 p.m.

Thursday 9th November 1989

Sessions commence 9.00 a.m.
Sessions conclude 7.00 p.m.
Conference Dinner 7.00 p.m.

Friday 10th November 1989

Sessions commence 9.00 a.m.
Sessions conclude 3.00 p.m.

Conference President

Prof. J.S. Williams
Australian National University

Conference Committee

Dr. J.R. Bird
Dr. J.W. Boldeman
Dr. D.D. Cohen
Dr. R.G. Elliman
Mr. J.J. Fardy
Dr. G.J.F. Legge
Dr. B.V. King
Dr. S.H. Sie
Dr. R.B. Gammon

ANSTO
ANSTO
ANSTO
R.M.I.T.
CSIRO
University of Melbourne
University of Newcastle
CSIRO
AINSE

Conference Secretary

Ms J. Watson
AINSE
### SUMMARY

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WEDNESDAY, 8th NOVEMBER 1989 - LUCAS HEIGHTS

TIME

10.30 Opening Remarks - Conference President
Prof. J.S. Williams (A.N.U.)

SESSION I  FACILITIES AND INSTRUMENTATION I
Chairman: Prof. J.S. Williams (A.N.U.)

10.40 ANSTO, R & D and the Industrial Interface
- A Case Study. J.S. Charlton (Tracerco Australasia)

11.10 The ANSTO Tandem Accelerator. J.W. Boldeman (ANSTO)

11.40 Microphase Separation in Polymers and Paraffin Crystals.
M. Epperson (Argonne National Laboratory), R. Snyder
(University of California), J.W. White (A.N.U.)

12.10 Radiometric Gauges in Industry and Geophysics.
W. Durante (ANSTO)

12.40 CONFERENCE LUNCH - STEVENS HALL

SESSION II  INDUSTRIAL APPLICATIONS
Chairman: Dr. G.J. Clark (IBM Australia)

1.40 Applications of Nuclear Techniques in Industry.
J.R. Bird (ANSTO)

2.10 Industrial Opportunities and Application of Ion
Beams for Materials Analysis. C.A. Evans,
P.C. Kenroy (Charles Evans)

2.40 AFTERNOON TEA

SESSION III  TOUR OF ANSTO

5.00 SESSION III  POSTER SESSION A - AMENITIES CENTRE

Low Energy Ion Beam Mixing During SIMS Depth
Profiling. M.A. Sobhan, B.V. King, R.J. MacDonald
(Univ. of Newcastle)

Quantitative SIMS Depth Profiling of Ion Implanted
Semiconductors. P.J.K. Paterson (R.M.I.T.),
M. Petracic (A.N.U.)

Angle Resolved Secondary Ion Mass Spectroscopic
Studies of Ni3Al. S. Johnson, B.V. King,
R.J. MacDonald (Univ. of Newcastle)

Ion Assisted Deposition of Diamond Films
Characterisation of Apparatus. M.J. Paterson,
D.K. Sood (R.M.I.T.)

Structure Analysis of O on Ni(001) and Ni3Al(001) Surfaces
by Low Energy Ion Beams. Y.G. Shen, D.J. O'Connor,
R.J. MacDonald (Univ. of Newcastle)
Empirical Formulae for Proton Energy Straggling in Elemental Materials. Q. Yang (Shandong Univ.), D.J. O'Connor (Univ. of Newcastle), Z. Wang (Beijing Univ.)

Ion Beam Analysis on the Lucas Heights Tandem. D.D. Cohen, G. Bailey (ANSTO)


Selenium in the Blood and Food of Australians. G.D. McOrist, J.J. Fardy (ANSTO)

Ion Beam Induced Resistivity Changes in Glassy Carbon. D. McCulloch, S. Prawer (R.M.I.T.)

Detection of Trace Copper Levels in Polymers by RBS. M.J. Kenny, J. Hodgkin (CSIRO)

Ion Beam Induced Damage and Element Loss in Biological Tissue. B.J. Kirby, G.J.F. Legge (Univ. of Melbourne)


Universal Ion Implantation Manipulator. R.A. Clissold, D. Stevenson (CSIRO)

Ion Beam Induced Phase Changes in GaAs. S.T. Johnson, R.G. Elliman (R.M.I.T.), J.S. Williams (A.N.U.)


Ion-Beam-Induced Epitaxial Crystallization of GeSi_{1-x}/Si Heterostructures. R.G. Elliman, M.C. Ridgway (R.M.I.T.), J.S. Williams (A.N.U.)

Theory of the Momentum Distribution of Swift Ions and its Application to Surface Scattering. M. Kato (Univ. of Newcastle)
THURSDAY, 9TH NOVEMBER, 1989 - LUCAS HEIGHTS

SESSION V  MATERIALS SCIENCE APPLICATIONS I
Chairman: Dr. G.J. Legge (Univ. of Melbourne)

9.00 Nuclear Microscopy of Technological Materials at Oxford.  
D.N. Jamieson (Univ. of Oxford)

9.30 Diffraction Analyses of Zirconia - Toughened Ceramics.  
C.J. Howard, E.H. Kisi (ANSTO)

9.50 Simultaneous Profiling of $^{14}$N and $^{15}$N.  
M.J. Kenny, L.S. Wielunski (CSIRO)

10.10 Ion Beam Modification of the Surface Wear Characteristics of Diamond.  
G.C. Anderson, S. Prawer (R.M.I.T.)

10.30 Towards Practical Uses of Ion Bombardment in Ceramics.  
D.K. Sood (R.M.I.T.)

10.50 MORNING TEA

SESSION VI  MATERIALS SCIENCE APPLICATIONS II
Chairman: Dr. B.V. King (Univ. of Newcastle)

11.10 Pitfalls in the Use of Ion Beams for Quantitative Materials Analysis.  
J.A. Davies (McMaster Univ, Canada)

11.40 Ion Beam Analysis of Thin Film Photovoltaics.  
G.C. Morris (Univ. of Queensland)

12.00 Ion Beam Damage Phenomena in GaAs.  
R.A. Brown (Univ. of Melbourne), J.S. Williams (A.N.U.)

12.20 The Effect of Some Impurities on the Annealing Behaviour of Ion-Implanted Silicon.  

12.40 Ion-Beam Induced Epitaxial Crystallization of Metal Silicides.  
M.C. Ridgway, R.G. Elliman (R.M.I.T.), J.S. Williams (A.N.U.)

1.00 LUNCH
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SESSION VII  FACILITIES AND INSTRUMENTATION II
Chairman: Dr. J.A. Davies (McMaster Univ.)

2.00  Very High Resolution Powder Diffractometry. T.M. Sabine (Univ. of Technology, Sydney)

2.20  Thermal Neutron Scattering as a Probe of Atomic Structure - A Brief Overview. R.L. Davies (AINSE)


3.00  Microanalytical Problems in Industry and the SPMP. G.J.F. Legge, A. Saint (Univ. of Melbourne)

3.20  STIM Tomography. G. Bench, M. Cholewa, K.A. Nugent, A. Saint, G.J.F. Legge (Univ. of Melbourne)

3.40  AFTERNOON TEA

SESSION VIII  MATERIALS AND SURFACES
Chairman: Dr. S. Sie (CSIRO)


4.20  Ion Scattering Analysis of CaF$_2$/Si(111). B.V. King (Univ. of Newcastle), M. Kato, M. Katayama, M. Aono (Inst. of Physical and Chemical Research, Japan)

4.40  Application of Out-of-Plane MEIS to Surface Structure Analysis. C. Xu, D.J. O'Connor (Univ. of Newcastle)

5.00  Reionization in Low Energy He-Pb Scattering. A.D.F. Kahn, D.J. O'Connor (Univ. of Newcastle)

5.20  Polarity Determination of Epitaxial Structures of CdTe on GaAs by Channelling Technique. L.S. Wielunski (CSIRO)
THURSDAY 9TH NOVEMBER 1989 - LUCAS HEIGHTS

POSTER SESSION B  -  AMENITIES CENTRE

High Zinc Concentration in Spider Fangs.  
G. Bench, G.J.F. Legge (Univ. of Melbourne),  
H.W. LeFevre (Univ. of Oregon)

Substrate Orientation Dependence During Annealing  
of Amorphous Layers Produced by Stoichiometric Ion  

SIMS Analysis of Semiconductor Structures.  
M. Petravic, M.C. Ridgway, P.J.K. Paterson (R.M.I.T.),  
J.S. Williams (A.N.U.)

A 50 KV Target Bias System.  M.J. Kenny,  
L.S. Wielunski (CSIRO)

The ANSTO/ANU $^{36}$Cl Accelerator Mass Spectrometry  
Measurement Program.  R.F. Davie, L.K. Fifield, J.R. Bird  
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(A.N.U.), G.B. Allison, G.R. Walker, I.D. Jolly,  
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Ion Beam Induced Amorphization and Dynamic Annealing  
Processes in Silicon.  R.D. Goldberg, R.G. Elliman,  
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K.T. Short (A.T. & T. Bell Labs., U.S.A.)

Zinc Ion Implantation into <001> Sapphire.  

A Data Compression Algorithm for Nuclear Spectra.  
J.F. Mika (Varian Techtron), L.J. Martin (A.R.L.),  
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An Investigation into the Minimisation of Parasitic  
Aberrations in the Fringe Field Region of a Magnetic  
Quadrupole Lens.  G.R. Moloney, G.J.F. Legge (Univ. of  
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Lead Implantation into Sapphire.  F. Placidi,  
D.K. Sood, A.P. Pogany (R.M.I.T.)

A Study of High Dose Nitrogen Implantation in the  
Surgical Implant Alloy, Ti-6Al-4V.  V.C. Nath,  
D.K. Sood (R.M.I.T.)

Implant Induced Electrical Isolation of Gallium  
Arsenide.  D. Sengupta, J.M. Zemanski  
(R.M.I.T.), J.S. Williams (A.N.U.)

Fluorine in Australian Coals: A Nuclear Physics Technique.  
E. Tiller (D.D.I.A.E. retired)
A Comparative Study by SIMS and RBS-C of Formation of β-SiC in Silicon by Carbon Ion Implantation.

Physical Characterisation of Mercury Cadmium Telluride (HgCdTe) Films Grown by MOCVD.

Study of the Near Surface Damage in Silicon Created by P$_2^+$ and P$^+$ Implantation.
Z.W. Fang, R.G. Elliman (R.M.I.T.), C.L. Lin (Shanghai Inst. of Metallurgy)

Trace Element Distribution in Native Gold.
S.H. Sie, D.R. Cousens, W.L. Griffin, C.G. Ryan (CSIRO),
S.J.M. Garrett, R.R. Large (Univ. of Tasmania)

GENUREAC - A User Friendly General Purpose Nuclear Reaction Code for IBA.
D.D. Cohen, E.K. Rose (ANSTO)

Design and Fabrication of a Semiconductor Particle Detector.
L. Robinson, A.L. Cox, G.K. Reeves (R.M.I.T.)

CONFERENCE DINNER - ANSTO CANTEEN
FRIDAY, 10TH NOVEMBER 1989 - LUCAS HEIGHTS

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SESSION X  ACCELERATOR MASS SPECTROMETRY - ARCHAEOLOGY OR ART
Chairman: Dr. J.R. Bird (ANSTO)


9.30 PIXE and Art. E. Clayton (ANSTO), S. Laidler (Art Gallery of N.S.W.)


10.30 MORNING TEA

SESSION XI  GEOLOGICAL APPLICATIONS
Chairman: Mr. J.J. Fardy (ANSTO)


11.30 The Accuracy of Standardless PIXE Microanalysis in Geoscience Applications. C.G. Ryan, W.L. Griffin, S.H. Sie, D.R. Cousens (CSIRO)

11.40 Low Level U and Th Determination in Australian Dune Systems. J.T. Hutton, J.R. Prescott (Univ of Adelaide)


12.30 Thorium-Radium Dating of Floodplain Sediments. A. Johnston, A.S. Murray, R.J. Wasson (Office of the Supervising Scientist, Jabiru)

12.50 LUNCH
TIME

SESSION XII

BIOMEDICAL APPLICATIONS

Chairman: Dr. D.D. Cohen (ANSTO)


2.00 Beam Induced Damage in Organic Targets. M. Cholewa, G.J.F. Legge (Univ. of Melbourne)

2.20 Distribution of Cu, Fe and Zn in Brindled and Normal Mouse Kidney Tissue. B.J. Kirby (CSIRO), G.J.F. Legge (Univ. of Melbourne), H. McArdle, D.M. Danks (Royal Children's Hospital, Victoria)

2.40 Closing Remarks - Conference Chairman

Prof. J.S. Williams (A.N.U.)
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Universal Ion Implantation Manipulator.  
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Ion Beam Induced Phase Changes in GaAs.  
S.T. JOHNSON, R.G. ELLIMAN, J.S. WILLIAMS  

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Theory of the Momentum Distribution of Swift Ions and its  
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Diffraction Analyses of Zirconia - Toughened Ceramics.  
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M.J. KENNY, L.S. WIELUNSKI  

Ion Beam Modification of the Surface Wear Characteristics  
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G.C. ANDERSON, S. PRAWER  

Towards Practical Uses of Ion Bombardment in Ceramics.  
D.K. SOOD  

Pitfalls in the Use of Ion Beams for Quantitative  
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The Effect of Some Impurities on the Anealing  
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Ion-Beam Induced Epitaxial Crystallization of Metal Silicides. M.C. RIDGWAY, R.G. ELLIMAN, J.S. WILLIAMS

Very High Resolution Powder Diffractometry. T.M. SABINE

Thermal Neutron Scattering as a Probe of Atomic Structure - A Brief Overview. R.L. DAVIES

New Ion Beam Facilities at ANU. J.S. WILLIAMS, R.G. ELLIMAN, P.J.K. PATERSON

Microanalytical Problems in Industry and the SPMP. G.J.F. LEGGE, A. SAINT

STIM Tomography. G. BENCH, M. CHOLEWA, K.A. NUGENT, A. SAINT, G.J.F. LEGGE


Ion Scattering Analysis of CaF$_2$/Si(111). B.V. KING, M. KATO, M. KATAYAMA, M. AONO

Application of Out-of-Plane MEIS to Surface Structure Analysis. C. XU, D.J. O'CONNOR

Reionization in Low Energy He-Pb Scattering. A.D.F. KAHN, D.J. O'CONNOR

Polarity Determination of Epitaxial Structures of CdTe on GaAs by Channelling Technique. L.S. WIELUNSKY

High Zinc Concentration in Spider Fangs. G. BENCH, H.W. LEFEVRE, G.J.F. LEGGE


A 50 KV Target Bias System. M.J. KENNY, L.W. WIELUNSKY


Ion Beam Induced Amorphization and Dynamic Annealing Processes in Silicon. R.D. GOLDBERG, J.S. WILLIAMS, K.T. SHORT, R.G. ELLIMAN, M.C. RIDGWAY
Zinc Ion Implantation into <0001> Sapphire.  
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An Investigation into the Minimisation of Parasitic Aberrations in the Fringe Field Region of a Magnetic Quadrupole Lens.  G.R. MOLONEY, G.J.F. LEGGE

Lead Implantation into Sapphire, F. PLACIDI, D.K. SOOD, A.P. POGANY

A Study of High Dose Nitrogen Implantation in the Surgical Implant Alloy Ti-6Al-4V. V.C. NATH, D.K. SOOD

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ANSTO, R & D AND THE INDUSTRIAL INTERFACE
- A CASE STUDY

by

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Abstract

The ANSTO Strategic Plan [1] highlights the increasing importance of ANSTO’s role in ensuring that the full benefits of nuclear technology, and associated spin-offs, are made available to Australian industry. The relationship between ANSTO and industry is seen as symbiotic. Industry benefits through improved process efficiency, better quality control and new or improved products. ANSTO is rewarded with the revenue it receives for its services but, equally importantly, it is exposed directly to problems important to industry and the community and this ensures the continuing relevance of the applied research programmes.

As part of the move towards greater involvement with industry, ANSTO has entered into an agreement with ICI Australia to form a Partnership company, Tracerco Australasia. This paper examines the role and activities of Tracerco Australasia and uses them to exemplify some of the ways in which the fruits of ANSTO research can be brought to industry.

The parentage of Tracerco Australasia is apt. Both Partners have extensive experience in the application of radioisotope technology to study industry's problems. ANSTO’s experience ranges across a wide cross-section of industry and the environment while ICI worldwide has a vast experience of applications in the chemical, oil and gas industries. This international dimension is important. Tracerco Australasia shares in a world-wide pool of experience; it is exposed to problems drawn from an international network, with enhanced possibilities for ANSTO R & D. It also provides one possible route for the commercialization of ANSTO developments overseas.

The formation of Tracerco Australasia as a Partnership with a major industrial company is itself an expression of one of the ways in which ANSTO interacts with industry. An examination of Tracerco's operations provides a deeper insight into the nature of the interaction.

Tracerco is concerned with the application of nuclear techniques to solve problems in industry and the community. One important category of application is the use of radioactive tracer methods to study the fluid dynamics or mass transport properties of flowing media.

The principles of such methods are well known [2]. Radioactive tracer in an appropriate chemical and physical form is injected into the system of interest, thereby labelling a portion of the flowing material with radioactivity. The labelled material can then be detected as it moves through pipes, vessels or conduits, by strategically positioned radiation detectors. Table 1 lists some of the investigations and measurements which can be made by applying this technology. The measurements are performed on on-line plant with essentially zero disruption of process operations. The
ability to diagnose faults in this way leads to the minimization of plant shut-downs with large savings, both in terms of better-targeted maintenance and avoidance of lost production. Also, in providing essential input data to plant optimization programmes, radioactive tracer methods assist in the improvement both of the output and the quality of product, with attendant improvements in profitability.

TABLE 1

Investigations Using Radioactive Tracers

<table>
<thead>
<tr>
<th>Flowrate Measurements of Liquids and Gases:</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a) On-line calibration of flow-meters</td>
</tr>
<tr>
<td>(b) Measurement of flow distribution in a network</td>
</tr>
<tr>
<td>(c) Effluent flowrate measurement</td>
</tr>
<tr>
<td>(d) Mass balance computations</td>
</tr>
<tr>
<td>(e) Efficiencies of pumps and turbines</td>
</tr>
<tr>
<td>(f) Steam quality measurements</td>
</tr>
<tr>
<td>(g) Environmental flowrates</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>Leakage Testing:</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a) Internal leaks in process vessels</td>
</tr>
<tr>
<td>(b) Underground leaks</td>
</tr>
<tr>
<td>(c) &quot;Carry-over&quot;. Entrainment of liquid in gas streams</td>
</tr>
<tr>
<td>(d) Chanelling in hydrocarbon reservoirs</td>
</tr>
</tbody>
</table>

<table>
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<tr>
<th>Residence Time Studies:</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a) To compare actual plant performance with design predictions</td>
</tr>
<tr>
<td>(b) To identify malfunctions in flow patterns; bypassing; stagnant or &quot;dead&quot; zones</td>
</tr>
</tbody>
</table>

Dispersion Studies
Sediment Transport Studies
Measurement of mixing and blending efficiencies

As an indication of the potential for the use of radiotracers in industry, it is worth noting that Tracerco's UK associate undertakes in excess of seven hundred investigations of this type each year [3].

Though each investigation is unique in some respect, these are not research projects in the usual sense of the word. Rather, they are services, requiring a service organization to undertake them effectively. Tracerco's role is to ensure that the customer is provided with the service he requires, when he requires it, at consistently high quality and in a cost-effective manner. Should a problem be encountered which requires significant development work - say a requirement for the accurate characterization of multi-phase flow patterns, then with the customer's permission, Tracerco would involve its parents in the search for an appropriate solution.
In this way Tracerco performs two functions: it provides a mechanism for ensuring that developed technology is made available to industry in an effective and timely manner and also acts as a channel through which problems encountered at the industrial interface can be fed back to the appropriate research group.

A further Tracerco activity is the provision of nucleonic instruments for analysis, measurement and control on industrial plant. Such instruments incorporate a radioactive source and utilize one (or more) of the interactions of radiation with matter as the means of measurement [4]. Typically, radiation from the source is directed at the vessel or medium of interest. By detecting and analysing the transmitted or scattered radiation it is possible to draw conclusions about the contents of the vessel or the physical properties of the medium.

Instruments in Tracerco's range are listed in Table 2.

**TABLE 2**

_Installed Nucleonic Instruments for Industrial Applications_

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Basis for Measurement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Level gauges (alarm and proportional systems)</td>
<td>Gamma-ray absorption</td>
</tr>
<tr>
<td>Density gauges</td>
<td>Gamma-ray absorption</td>
</tr>
<tr>
<td>Belt-weighers (for continuous measurement of product mass on moving belt systems)</td>
<td>Gamma-ray absorption</td>
</tr>
<tr>
<td>Interface Alarms</td>
<td>Neutron moderation</td>
</tr>
<tr>
<td>Boron Analysers</td>
<td>Neutron absorption</td>
</tr>
<tr>
<td>H/C ratio analyser</td>
<td>Beta-particle scattering</td>
</tr>
<tr>
<td>Density profiler</td>
<td>Gamma-ray absorption</td>
</tr>
<tr>
<td>Effluent flowmeter</td>
<td>Radioisotope dilution</td>
</tr>
<tr>
<td>Potash deposit locator</td>
<td>Natural gamma-ray detection</td>
</tr>
<tr>
<td>Installed corrosion monitor</td>
<td>Neutron activation of plant components</td>
</tr>
<tr>
<td>Thickness gauges</td>
<td>Gamma-ray absorption and Gamma-ray Scattering</td>
</tr>
</tbody>
</table>
The advantages of nucleonic instruments stem from the fact that, in
general, the instruments are completely non-invasive. For this reason they
are ideal for measurements on high-pressure, high-temperature, acidic,
toxic or otherwise "difficult" process materials. Additionally, they can
be installed, calibrated and maintained while the plant is on-line. Substantial economic benefits are realized through improved process control
and shut-down avoidance.

It is worth noting that Tracerco is not a conventional instrument
company. The instruments it provides are sold very much as "solutions to
problems" rather than "black boxes" and each is tailored to the customer's
requirements - again, emphasising Tracerco's role as a service company.
Should the customer's requirements extend beyond what can be achieved by
instruments in the current range, then ANSTO (or ICI, as appropriate) may
be involved in developing a solution to his problem. Like the radiotracer
activity, Tracerco's nucleonic instrument business satisfies the perceived
need for a proactive relationship with industry; it is a vehicle for
introducing developments in nuclear instrumentation into industry and for
transmitting back the stimuli for further research and development.

Tracerco Australasia is a new company and the relationship between it
and ANSTO is still developing. Even at this stage, however, there are
clear indications that the model is sound and that Tracerco and other
ventures like it will play useful parts in forging closer links between
ANSTO and industry. In this way, we will ensure that the "problems in
search of solutions" and the "solutions in search of problems" need not
seek too long, or too far.

References

[1] Australian Nuclear Science and Technology Organisation,


Pergamon Press, Oxford (1971)
THE ANSTO TANDEM ACCELERATOR

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ANSTO has purchased a large Tandem accelerator laboratory from Rutgers University in the State of New Jersey in the USA. This famous university established in 1766 was a pioneer in many aspects of nuclear physics. However, in recent years the university has been unable to attract the funding necessary for the accelerator laboratory so the decision was made to close the facility and shift the research work to neighbouring universities at Yale and Princeton. ANSTO has been seeking to install a Tandem accelerator for many years and the availability of this entire laboratory provides a major opportunity for applied nuclear science and technology in Australia.

The intention is to install a world class applied nuclear science facility which will have the capability of satisfying existing strong Australian needs for Tandem accelerator facilities but which will also have sufficient flexibility to respond to new developments in the application of charged particle beams.

The proposed research and development program for the Lucas Heights Tandem has the following five major elements:-

a) The establishment of a national radiocarbon dating service for applications in quaternary research, oceanography, glaciology and genetic engineering;

b) The development of an ultra sensitive accelerator mass spectrometry service for applications in the fields of groundwater hydrology, salinity, erosion and sedimentation studies and nuclide migration;

c) The application of ion beam analysis techniques for industrial, environmental and biological studies;

d) The provision of high intensity heavy and light ion beams for materials science applications and industrial uses; and

e) The provision of intense neutron fluxes for safeguards development, industrial applications and radiobiological studies.

The total cost will be approximately $2.3M. A special allocation to ANSTO of $0.5M towards the cost of this facility was received from the Federal Government as part of the new science package.

Progress for the installation of this facility will be discussed.
MICROPHASE SEPARATION IN POLYMERS AND PARAFFIN CRYSTALS
by
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Abstract

INTRODUCTION - This paper is an example of research using the small angle neutron scattering technique.

The question of polymer compatibility and microphase separation is currently of practical and fundamental interest. In the practical domain two general problems are of current interest. Firstly, the extent to which polymer alloys can be made by blending commercial polymers is a potentially important way to design materials with intermediate properties. Secondly, environmental constraints now raise the desirability of polymer recycling and this poses the question of the degree to which small components of second polymer may affect properties and hence eventual uses of the recycled material. At a fundamental level the problem of microphase separation is intrinsic to the properties of block and graft copolymers and the extent to which such chemical methods give access to new materials by design. For example, also, we have found recently that the amphiphilic properties of some soluble electrically conducting polymers leads to micelle and other forms of microphase separation in the solutions and in the solid phases and that such separation is important for controlling the electrical and mechanical properties of these materials.

A most subtle example of microphase separation and the usefulness of neutron small angle scattering for exploring it concerns the separation of deuterated and protonated species of the same polymer. This work illustrates the generality of the phenomenon and the extent to which theories such as the Florey Huggins model are applicable. Here we give the first results in another very simple system involving phase unmixing of simple paraffin hydrocarbons, in particular mixtures of C30H62:C36D74 at temperatures well below the freezing point of either component.

PARAFFIN HYDROCARBON SOLUTION

There is a long standing controversy on the structure of binary paraffin solid solutions and phase diagrams for the C30H62: C35H74 system show an extremely flat lower boundary in the $\delta\alpha+\delta\beta$ phase region. It appears that the question of whether there are additional demixing effects in this region cannot be settled by calorimetry alone. Indeed Dorset has suggested that for the $n$C30H62/$n$C36H74 system the phase diagram is complex. Using the fact that great contrast in the scattering of neutrons can be produced in such systems by taking one of the the components as fully deuterated and the other hydrogenous we have investigated the $n$C30H62/$n$C36D74 system at a C30/C36 composition ratio of 1:4. Our particular objectives were to see whether there was any detectable superstructure in quickly cooled and slowly cooled melts of this composition. A search was made for any superstructure which annealed in at room temperature over periods of months and which could be associated with the recently discovered low temperature heat capacity anomalies in this mixture.

Neutron small angle scattering measurements were made from samples contained in fused quartz cells, 1mm thick, the components having been melted together in the cell and
allowed to mix freely in the liquid state for many hours before cooling. In the first series of experiments a sample which had been so prepared and allowed to anneal for six months was measured at room temperature, 46°C and 55°C all temperatures well below the melting point of the mixture. In a second series of experiments the sample was melted and homogenized above the melting point and quickly cooled to 27°C by immersion in cold water. The neutron scattering pattern was recorded as a function of time for annealing at this temperature. In the third experiment the liquified sample was allowed to cool slowly (over a period of two hours) to 27°C the scattering pattern being measured periodically during this period.

All measurements were made on the Small Angle Diffractometer (SAD) at the Intense Pulsed Neutron Source, Argonne National Laboratory. This instrument uses a large part of the wavelength spectrum from the pulsed source and is thereby able to record small angle scattering in the range of momentum transfers 0.006 < Q/Å^-1 < 0.3. The momentum transfer

\[
Q = \frac{4\pi}{\lambda} \sin \theta
\]

where \( \theta \) is the half scattering angle in the experiment. All wavelengths between 2Å and 13Å were used in the measurements. The samples proved to be very strong scatterers and typically, a good pattern could be recorded in about half an hour. Since the sample thickness is known precisely all measurements were corrected to an absolute scale of scattering intensity using standard samples for instrument calibration.

RESULTS
Figure 1 shows the small angle scattering from the as received C_{30}H_{62}·C_{36}D_{74}:1:4 mixture which had been allowed to equilibrate at room temperature for approximately six months (Curve A). This should be compared with measurements at low and high temperatures of the pure C_{36}D_{74} crystal (Curve B). It is evident that there is much small angle scattering from the mixed crystal which is not apparent in the pure C_{36}D_{74} sample. The peak in that sample at \log Q=-2.02 (Q=-132\,\text{Å}^{-1}; d=47\,\text{Å}) corresponds to the 002 reflection for the C_{36} compound. Annealing at 46°C, even for one hour, causes a loss of scattering intensity in the range 0.015<Q/\text{Å}^{-1}<0.132. Annealing for a total of 150 minutes at 46°C produced further small decreases of this signal as shown in the figure. We conclude that some form of superstructure is being annealed out in this process.

The effect of continuing the annealing at 53°C is shown in Figure 2, Curve C. The small angle scattering below Q=0.027\,\text{Å}^{-1} is now essentially identical to that from the pure C_{36}D_{74} material, there remaining only a broad background of scattering between this Q and Q=0.30\,\text{Å}^{-1} where statistical uncertainty makes measurements no longer possible. It is quite conceivable that this background is the natural background due to the extra incoherent scattering from the proton containing component of the mixture. Figures 1 and 2 show that a microphase component is present in the longtime annealed system and that this can be progressively annealed out at temperatures between 47°C and 53°C - temperatures which are approximately 20°C and 10°C below the melting point of the pure C_{36}D_{74} and C_{30}H_{62} components respectively.
In an attempt to observe directly this annealing-in phenomenon the sample at 53°C was quickly chilled to 28°C and the small angle scattering pattern recorded each hour over a twelve hour period. Figure 3 shows the ingrowth of small angle scattering at 27.5°C. The scattering at low angles is almost identical for all of these runs and at high angles it is the same within the statistical uncertainty. The chief development has been the growth of two peaks in the mid-Q region. Since the scattering is so close at high and low angles one may emphasize the ingrowth of these peaks by dividing the measured scattering pattern by that obtained after the first sixty minutes of annealing at 27.5°C. These divided spectra are shown in Figure 4. A progressive increase in the scattering at Bragg d values of the order of 179Å and at 65Å occurs and there may be other minor variations in the intensity at higher momentum transfers.

CONCLUSION

These data show the remarkably speedy ingrowth of structure at 27.5°C - some 50°C below the melting point of the major component of the mixture. We suppose that this structure is attributable to a superlattice of the short and long chain components, the maximum value corresponding to that which will be obtained from a 1:4 mixture of C_{30}:C_{36} in the approximation of Vegard’s law being appropriate and each of the lattice parameters for the separate components being double that normally found for the C axis of the isobestic crystals. Other possibilities for this crystal could involve a Vernier effect of the packing of the short and long molecules but the present data do not allow these two to be resolved at present.

REFERENCES

RADIOMETRIC GAUGES IN INDUSTRY AND GEOPHYSICS

by

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ABSTRACT

A variety of nuclear gauges (that is, gauges in which the operational mode depends on the interaction of nuclear radiation with matter) exist today. Broadly speaking, such gauges can be divided into two groups: those using charged particles (alpha and beta particles), and those using non-charged 'particles' (neutrons and gamma rays). Since charged particles interact strongly with matter through ionisation, their range is very limited, and consequently they can supply information on the properties of only thin layers of matter. Thickness gauges using charged particles have been developed for the manufacturing industry. On the other hand, neutrons and gamma rays have much longer ranges, and in principle, can yield information on the bulk properties of matter. It is therefore not surprising that neutron and gamma gauges are being used increasingly in agronomy, hydrology, civil engineering and industry.

A typical example is the neutron backscatter gauge used to determine the moisture content of soils and other bulk media. Such a gauge consists, in essence, of a fast neutron source (typically $^{241}$Am-Be) in close proximity to a thermal neutron detector (such as a BF$_3$ or He$^3$ detector). The source neutrons are thermalised by collisions with surrounding nuclei. Because hydrogen nuclei are the most effective at slowing neutrons down, the thermal neutron density in the vicinity of the source/detector is dependent on the surrounding hydrogen concentration. It is therefore possible, in principle, to relate the thermal neutron detection-rate to the moisture content in the surrounding medium. However, in practice, it is often difficult to interpret the gauge output as the thermal neutron density is also dependent on the composition and bulk density of the surrounding matrix material. Thus independent measurements of the compositional effects (in particular, the neutron absorption cross-section) and the matrix density are frequently necessary.

Gamma backscattering gauges are commonly used for in situ density determination.

The main fields of application of neutron and gamma gauges are in studies of soil water storage and movement and of irrigation practice, and in the determination of the density and moisture content of compacted foundations in the construction of dams, roads, airfields and buildings. Nuclear gauges are also used in determining the moisture content of blast furnace coke, foundry sand and sand for ready-mix concrete. Nuclear interaction techniques are also well established in oil well logging. These and other applications will be discussed.
APPLICATIONS OF NUCLEAR TECHNIQUES IN INDUSTRY

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1. INTRODUCTION

Atoms, nuclei and fundamental particles are remote concepts to most people - including many scientists. However, in recent years, it has become possible to "see" individual atoms, to construct materials atom-by-atom and to analyse materials by atom counting. As a consequence, techniques previously used only in research laboratories, such as those requiring access to reactor beams, ion beams and radiation sources and detectors, are now being applied to solve problems in many fields from microelectronics to archaeology and from heavy industry to medical diagnosis. The direct production of high technology materials by transmutation, implantation, ion assisted thin film deposition and other techniques has also become possible through the advances in knowledge and the special analytical capabilities of nuclear and atomic techniques.

2. FACILITIES

Nuclear techniques generally require one of three sources of radiation: radioisotopes, particle accelerators or reactors. Radioisotopes can be transported for use at any field or laboratory location. They are readily available in Australia and have been applied to a wide range of industrial problems involving materials analysis and tracer studies. High energy electron and ion accelerators require relatively large laboratory facilities. At least six research laboratories in Australia have such facilities and a lot of pioneering work on methods of analysis has been done with these. However, we have yet to see the installation of dedicated facilities in specific industries such as is happening overseas. The same is true in the case of reactors used to provide neutron irradiations. Australia has only two reactors and one accelerator used for neutron beam production, all at Lucas Heights, whereas in many countries a wider range of organisations have their own facilities. A list of Australian accelerator and reactor facilities is included in this proceedings.

3. APPLICATIONS

Many new analytical techniques based on X-ray, laser or low energy electron beams are now available but this has not halted the expansion in the use of nuclear techniques and closely related atomic techniques. The following description summarises the scope of such applications and additional information is contained in other papers in this proceedings.

a. Radioisotopes

Radioisotope-based analytical techniques involve many unique types of analysis including bore-hole logging, moisture probes and special-purpose instruments for in-situ assay of minerals and process streams or for density and thickness determinations. Instruments giving excellent performance in many applications in primary and secondary industries have been developed in Australia and marketed world-wide. A recent development in the detection of naturally-occurring radioisotopes at levels down to 1 part in $10^{19}$ of the stable element uses high energy mass spectrometers. The high energy makes it possible to identify the mass and
atomic number of each ion passing through the accelerator and thus to determine $^{10}\text{Be}$, $^{14}\text{C}$, $^{36}\text{Cl}$, etc. This technology has also been established in Australia and is being used in studies of water resources and other topics.

b. Ion Beams

Ion beam analysis can be separated into two regimes which involve the use of high energy (>100 keV) ions or low energy (< 10 keV) ions. Intermediate energies are occasionally used. At high energy, ion irradiation leads to ion scattering, elastic atom recoil, X-ray emission nuclear reactions and activation. Each of these processes can be exploited for rapid non-destructive multi-element or isotope analysis. Because of the limited penetration of the ions (10 - 100 μm), the results apply to surface and near surface atomic layers and it is often possible to obtain information on the depth dependence of atomic concentrations within these layers. It is also possible to locate specific types of atoms and defects within a crystal lattice.

At low energies, ion-atom interactions are most important leading to ion scattering, the sputter removal of surface atoms and photon emission. Commercial instruments are available to exploit these processes for analysis of the topmost atomic layer and the study of surface structure. Using ion-induced signals or other probes such as electron or X-ray beams, a depth profile can be obtained by changes with time as the incident ion beam sputters away successive layers. Both low and high energy ion microprobes have been developed with a spatial resolution of 1 μm or better. By rastering such a beam across the surface of a sample, any of the analysis techniques mentioned above can be used to obtain spatial distributions of specific elements or isotopes. Some of this development work has been done in Australia and low or high energy microprobes are in use in a number of research laboratories.

Industrial applications of ion beam analysis can be grouped into three areas. First, major, minor, trace and ultra-trace element analysis provides a valuable extension of other more widely available techniques. For example, the determination of light elements such as H, C, N and O is particularly useful for monitoring the properties of metals, glasses and other industrial products. Fluorine can also be readily determined down to ppm levels in minerals such as coal, in wastes and in the industrial environment. Multi-element analysis of aerosols has been another major application of ion beam analysis.

The second, and most dramatic application of ion beam analysis has been in the development and production of high technology topics such as semiconductors, thin film devices, wear and corrosion reduction and other fields. Materials produced by techniques such as ion implantation, ion-assisted film deposition, sputtering, etc. can all be studied in detail by ion beam analysis to gain an understanding of the surface physics involved and to obtain detailed information on atomic distributions. Thirdly, the low energy and high energy ion microprobes provide new capabilities compared to those of the electron microprobe, for example for the study of light element distributions, surface degradation and contamination, mineral phases, and so on. Australia has the facilities and know-how in all these areas of application to support the further growth of frontier industries.

c. Neutron Beams

Neutron beams can penetrate many cms of most materials and they provide unique capabilities in composition and structure analysis for truly bulk material which even electron and X-ray beams can't provide. Neutron activation analysis is well known, especially for the
high sensitivity (ppb) for many elements such as the rare earths. It has been widely used for
many years and continues to be a preferred technique for many studies. For example, uranium
assay with a sensitivity of 1 ppm or better requires only one minute measuring time and has
been extensively used in uranium exploration in Australia. The observation of microscopic
fission tracks in some mineral phases or in photographic film placed against a sample provides
a very sensitive method for mapping the occurrence of uranium which is present in almost any
kind of sample. A reactor neutron beam also provides unique capabilities for radiographing the
H distribution in metals, soils, etc. - even to the ability to record real-time circulation of oil in
engines. High energy neutron beams (from radioisotope sources, neutron generators or large
ion accelerators) also have special applications for activation analysis of oxygen and other
difficult nuclides, for the assay of elements such as hydrogen, nitrogen, etc.

Neutron diffraction is also well-established as a complementary technique to other methods
for determining structure and atomic motion in crystals and powders. Neutrons are scattered
strongly by hydrogen and are also affected by magnetic fields within a crystal and so they
provide important information on structure and atomic motion which cannot be obtained from
other scattering or diffraction techniques. The change in scattering power when deuterium
replaces the hydrogen provides a method for locating H atom positions in crystals, powders
and even in biological materials by comparing diffraction patterns from hydrated and
deuterated samples. This method is of great value in unravelling the structure of many
industrially important materials which contain hydrogen, changes in structure introduced by
phase changes and even the quantitative determination of the phase composition of materials
such as partially stabilised zirconia, synroc, etc. New topics such as warm superconductors,
catalysts, novel battery designs and a host of other potential industrial products require
structure information for completion of development projects. In recent years, small angle
scattering of neutrons (particularly at very low energies) has been used to provide valuable
information on inhomogeneities such as porosity, phase separations, etc.

4. CONCLUSION

Nuclear techniques constitute a wide range of capabilities which are being used in all
stages of industrial operation from the exploration for raw materials, operation of process
plants and development of new products to the monitoring of pollution, occupational health
and long term environmental change. Australia has the capabilities and know-how in most
aspects of such applications but there is a need for updating of facilities to introduce new
techniques.
INDUSTRIAL OPPORTUNITIES AND APPLICATIONS OF
ION BEAMS FOR MATERIALS ANALYSIS

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Abstract

Ion beams in many forms are currently used in industrial applications for materials analysis and characterization. The increasing sophistication of these ion beam based analytical techniques has significantly expanded the range of applications to industrial materials. This paper will briefly discuss industrial applications of classical secondary ion mass spectrometry (SIMS) and Rutherford backscattering spectrometry (RBS) as well as introduce new applications made possible by recent developments in the related techniques of time-of-flight mass spectrometry (TOF-SIMS) and hydrogen forward scattering spectrometry (HFS).

Industrial applications of SIMS analysis will be demonstrated for the semiconductor, metal, polymer and biomedical fields. Applications will include depth profiling, bulk analysis and three dimensional imaging.

RBS is used to characterize semiconductor and non-semiconductor thin films, catalysts, and powders, evaluate surface treatments or aging processes of polymers or glasses, determine heavy metal contamination on most surfaces and evaluate the quality of surfaces of crystalline materials. A variety of these applications will be presented as well as a brief discussion of quantitative methods used in automated data reduction.

Time-of-flight secondary ion mass spectrometry (TOF-SIMS) is an evolving analytical technique typically used to analyze the top several monolayers of nearly any type of material. TOF-SIMS employs a rapidly pulsed ion beam for bombardment of the sample surface, followed by time-of-flight analysis of the emitted secondary ions. TOF-SIMS has numerous applications in surface analysis including: biology, polymers, pharmaceutical, catalysts and semiconductors. Applications presented will include conventional mass analysis and the imaging capabilities of newer TOF-SIMS instrumentation.

Hydrogen forward scattering analysis is performed using the same instrumentation as for RBS with a different geometrical arrangement of the detector relative to the sample and the high energy ion beam. The instrumentation and methods used during routine hydrogen forward scattering analysis will be explained. Applications presented will include quantitative measurement of hydrogen in a variety of materials including polymers, geological materials, semiconductors and catalysts.
LOW ENERGY ION BEAM MIXING DURING SIMS DEPTH PROFILING

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ABSTRACT
Ion beam induced mixing has been investigated during 4KeV Ne⁺ sputter depth profiling of Ni/Ag multi-layered sample using the technique of secondary ion mass spectrometry(SIMS). The quantity of mixing, (Dt/ΦFt), was determined by modelling the sputtering and mixing processes by a diffusion approximation to the transport equation. The mixing value was found to be 125 Å/eV. This value is 1.5 order of magnitude higher than that of the similar high energy mixing experiment in Ag.

INTRODUCTION
Ion beam induced mixing is one of the noble techniques for material modification. It has opened a new dimension in the field of science and technology because of producing altered surface layers with improved properties. The semiconductor and materials processing industries have been revolutionised by the energetic ion beam since last two decades[1]. The basic research in ion mixing is confined to the medium (100KeV) and high energy regime using the well known technique of Rutherford Backscattering Spectrometry(RBS) [2]. The layer by layer removal of a sample surface by ion beam sputter etching has the ability to produce high depth resolution. On the other hand, atomic mixing takes place in the deeper interfaces due to the damage created, deep inside the solid, by the projectile ion upon bombardment. This mixing is one of the factors affecting the depth resolution of sputter profiles and hence must be taken into account while performing any depth profiling experiment. In this paper we report the mixing efficiency, (Dt/ΦFt), of Ni with Ag during 4KeV Ne⁺ sputter depth profiling studied by SIMS.

EXPERIMENTAL
The thin film multilayers were grown by ion beam sputter deposition onto polished silicon substrate. The sample geometry was shown in fig.1. Typically, 1KeV, 10mA Ar⁺ beam from a Kaufmann source was used for sputtering target. The base and deposition pressures were 8X10⁻⁷ and 5X10⁻⁵ mbars respectively. The deposition rate (0.3-0.5Å/s) was checked by a quartz crystal monitor. The total film thickness and uniformity were measured by a stylus profilometer. The measurement agreed to within ±9%. The sample was then transferred to a combined SIMS/LEIS analysing system where 4KeV Ne⁺ beam was used for analysis and sputter etching. The sputtered secondary ions were mass analysed through a quadrupole mass filter mounted 90° onto the sample surface. The beam size was 250μm, rastered over 6 beam diameters and the signal from the central 6% of the crater was accepted to avoid the crater edge effects. The current density was measured by a Faraday cup. The density was checked before and after each depth profiling run.

RESULT and DISCUSSION
The depth profile of Ni/Ag is shown in figure 1. After sputtering to depths much greater than the original impurity marker position the decrease in impurity concentration, c(x,t), at the eroded surface, x=0, is exponential with sputter time t and erosion rate U:

$$c(0,t) \propto \exp\left(-Ut/\lambda\right)$$

(1)

where λ is the decay length (i.e. the depth over which the Ni signal falls to 1/e of its peak value) and is calculated from the slopes of the trailing edges at low impurity concentrations, typically below 20%, where sputter erosion rates should remain constant. The exponential
decay arises from the solution of a diffusion approximation [3] to the general transport equation:

\[
\frac{\delta c(x,t)}{\delta t} = D \frac{\delta^2 c(x,t)}{\delta x^2} + U \frac{\delta c(x,t)}{\delta x}
\] (2)

together with the boundary condition

\[D(0) \frac{\delta c(x,t)}{\delta x} = U(r-1) c(x,t)\] (3)

where \(r\) is the ratio of the component sputter yields \(Y_{Ni}^c\) and \(Y_{Ag}^c\) [4] and \(D\) is the diffusion constant given by

\[D = \frac{0.42J/6NEd}{(dE/dx)_n} <d^2>\] (4)

where \(N\) is the density of the matrix atom, \(J\) is the flux of the projectile ion, \((dE/dx)_n\) is the deposited damage energy due to nuclear processes, \(E_d\) and \(<d^2>\) are the displacement energy and the mean square displacement distance of the impurity atom respectively. The mixing is calculated from a fit of the solution of eqn.(1) to the experimental profiles.

From eqns.(2) and (3) it is evident that the value of \(\lambda\) depends on preferential sputtering as well as \((Dt/\phi Fd)\). The computer programme TC (TRIM Cascade) [5] was used to determine the amount of preferential sputtering of Ni from alloy of the form \(Ni_{0.2}Ag_{0.8}\). The value of surface binding energy used for Ni is \(\gamma_{NiAg}\) and is given elsewhere [6]. The value of \(\lambda\) is presented in table-I. The dependence of \((Dt/\phi Fd)\) on \(\lambda\) and \(r\) is shown in figure 2.

From table-I, it is seen that the value of mixing efficiency of the present work is higher by a factor of 1.5 order of magnitude than that obtained by similar high energy mixing experiment [7]. The difference could be due to several number of reasons and they are: (1) may be because of different ion energy between the two sets of experiment, (ii) may be because of different temperatures or (iii) may be because of the surface effects like sputtering and segregation which can be neglected in the high energy mixing experiment. However, there is much uncertainty in estimating the values of \(r\) from both theory and experiment. For a small change of \(r\), significant change in \((Dt/\phi Fd)\) can occur. Therefore, the sensitivity of mixing efficiency to uncertain preferential sputtering ratio cannot be defyed.

CONCLUSION

Mixing efficiency of Ni in Ag has been reported during room temperature sputter depth profiling of multilayer thin film using 4KeV \(Ne^+\). The result has been compared with similar high energy mixing result.

<table>
<thead>
<tr>
<th>Table-I</th>
<th>Values of the parameters used in the calculation of ((Dt/\phi Fd)).</th>
</tr>
</thead>
<tbody>
<tr>
<td>Element</td>
<td>Matrix</td>
</tr>
<tr>
<td>Ni</td>
<td>Ag</td>
</tr>
<tr>
<td>Ni</td>
<td>Cu</td>
</tr>
<tr>
<td>Ni</td>
<td>Ag</td>
</tr>
</tbody>
</table>

* Value used for \(F_d\) for 4KeV \(Ne^+\) at the depth of the impurity layer in Ag was 57.24eV/\(\AA\), as calculated from the TRIM.
Figure 1. SIMS sputter depth profiles of Ni and Ag from the Ni/Ag multilayer shown.

Figure 2. The dependence of mixing efficiency, (Dt/ΦFu), on λ and r.

REFERENCES
Introduction

The combination of high sensitivity with good depth and lateral resolution makes SIMS an analytical technique well suited for application in materials research in general and in ion implanted materials in particular. The required accuracy and precision of analysis often necessitates the use of sophisticated quantification procedures. The purpose of this paper is to review the currently existing SIMS quantification methods, to indicate which of these methods can best be applied to ion implanted materials and to discuss some of the factors influencing the accuracy of the analytical results.

Quantitative Analysis

The aim of quantitative analysis is to determine the average value of the concentration of a given element A, \( C(A) \) across the probed volume. For samples inhomogeneous laterally or in depth this concentration is usually different to the concentration averaged over the whole sample. Discrepancies between SIMS data and other bulk analytical techniques are often due to this problem.

Methods for Quantitative SIMS Analysis

SIMS quantification relates the measured secondary ion intensity \( I(A) \) of element A to the concentration of element A in the sample. This may be expressed as
\[
I(A) = I_p f_p f_i C(A) \tag{1}
\]
where \( I_p \) is the primary ion current, \( f_p \) is a secondary ion emission parameter, \( f_i \) is a fudge factor and \( f_i \) is an instrumental factor. From equation (1) three different methods of quantification can be envisaged.

(a) First principal methods (\( f_i = 1 \)) based upon theoretical models describing the emission of secondary ions [for an overview see ref [1]]. The results obtained with these models are in general only valid for a given set of samples and experimental conditions. In optimal cases the uncertainties may be 10% to 20%, but for other samples may reach several orders of magnitude. Even those methods require well characterized calibration samples to check theoretical predictions.

(b) Semi Empirical Methods

Usually with these methods a simplified model of the secondary ion emission process is used for calculating the SIMS intensities [1,2]. The shortcomings of these approaches are compensated for by introducing parameters adjusted to the experimental results (fudge factor approach). From several review papers [1,2,3] it can be concluded that semi-theoretical methods provide quantitative SIMS analysis with uncertainties between 25% - 200%.

(c) Empirical Methods

In these methods the relation between \( I(A) \) and \( C(A) \) is determined by means of a calibration curve [1]. The curve is generated by measuring calibration samples containing element A in various concentrations. The slope of the calibration curve gives the absolute sensitivity factor \( P_A \) of element A in that particular matrix \( T \).
The relative sensitivity factor $P_{A,R}$ is defined similarly from a plot of $I(A)/I(R)$ versus the relation concentration $C(A)/C(R)$ where $R$ is an internal reference element, usually the matrix or a main component. For the determination of an unknown sample $X$ the calibration curve is used as an analytical curve. To be a valid procedure the experimental conditions must be kept constant and the matrices of $T$ and $X$ must be identical.

Sensitivity factors are strongly dependent on the matrix surface properties, particularly oxygen content. It has been shown [4] the ratios of matrix ions can serve as internal indicators for indexing matrix surface properties, resulting in the best cases of relative accuracies of 10% [5,6].

Instrument dependence of sensitivity factors has been investigated by Rudenauer et al [7]. By tuning different SIMS instruments so that the relative sensitivity factors for a particular samples of metallic glass coincided, these authors showed that the sensitivity factors of six further elements on different SIMS instruments agreed to within a factor of 1.7.

In summary, of the available methods for SIMS quantification it must be concluded that only empirical methods offer sufficient accuracy (10%) to make SIMS a quantitative tool for assessing ion implanted samples. The main drawback of these methods ie. the selection of standards of matching matrix composition, is insignificant for semiconductors as these matrixes are generally very well characterized.

Requirements for SIMS standard samples

The general requirements for a SIMS standard are (i) lateral homogeneity on a micron scale, (ii) composition stable over an extended period of time, (iii) assessment of elemental composition by one or more independent analytical techniques must be possible. Ion implanted standards are attractive for the following reasons (a) almost any element/matrix combination is possible, (b) monitoring the total implanted fluence allows quantitative ion implantation, (c) lateral homogeneity is ensured by adequate mastering of the ion beam, (d) a large range of concentrations can be obtained by varying the fluence, (e) from the projected range of the profile the sputtering rate can be estimated, (f) information on system performance (dynamic range, background intensity can easily be derived form the characteristic shape of the implantation profile. Furthermore Rutherford Backscattering Spectometry (RBS) and Neutron Activation analysis NAA [8] can be used as independent analytical techniques.

For quantification of SIMS date the integration method is generally used. The sensitivity factor $P_A$ is derived from the integrated count rate of the implantation profile. The basic assumption that SIMS intensities depend linearly on the concentration is generally valid well below 1 at%.

For practical work two approaches have been developed (a) implantation of the element of interest (or an isotope of this element) directly into the unknown sample standard addition method [9], (b) application of external implanted standards [10].

These approaches together with the effects of primary beam energy and primary ion mass will be discussed for a range of implants into silicon including In, As and B.
References


Angle Resolved Secondary Ion Mass Spectroscopic Studies of Ni₃Al.

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Introduction

In this work, shadow-cone enhanced sputtering yields are compared for the <100> and <110> azimuths of the {001} plane of a clean Ni₃Al fcc monocrystal sample. It is known from ion-scattering that a shadow-cone is formed behind a target atom (or source atom), when low energy primary ions are incident upon it, preventing the penetration of primary ions into the shadow region. For example, in figure 1, S is a source atom which shadows atom B lower in the surface. Since there is a void of primary ions over a considerable volume within the surface, this phenomenon produces a concentration of primary ion flux around the boundary of the shadow-cone, which can intersect with atoms on the surface and in lower monolayers (see reference atom R in figure 1). This enhances sputtering mechanisms due to a larger number of primary ion and surface atom collisions, producing a peak in the yield of sputtered particles from the surface. Observation of the sputtered particles as a function of the orientation of the incident angle, would then produce peaks and troughs depending on whether the primary ion beam is focussed onto surface atoms with respect to atoms in the very near surface region.

Experimentally, detection of one type of sputtered particle, secondary positive ions (Al⁺, Ni⁺), was made using Secondary Ion Mass Spectrometry (SIMS). The incident angle of the ion beam was varied, at a fixed azimuth, and yield measurements taken at a constant scattering angle of 120°. It can be seen from figure 1 that many source-reference focussing interactions could be possible for a 0-90° rotation of the ion beam about the <010> axis. Table 1 summarises the theoretical angles at which certain source-reference focussing interactions are possible, for the <100> and <110> azimuths. The interactions have been restricted to 1st and 2nd layers in <100>, (1st-3rd interactions were considered as making a negligible contribution to the total yield), but for <110>, 1st and 3rd, with 2nd and 4th layer interactions are possible because of the non-close-packed geometry of this azimuth.

Polar incident angle scans for the two azimuths, are shown in figures 2 and 3. It is noted that for each azimuth, peaks in the yield arise from a superposition of separate source-reference focussing interactions occurring at relatively similar angles. The condition where R lies on the
shadow-cone of S in figure 1, corresponds to an incidence angle where the peak height is 85% of the maximum, making atom positions within the lattice independent of thermal motions [3]. In the polar incident angle scan parallel to the <100> azimuth, the peak at 20° can be assumed to be a combination of the focussing interactions of Al with Ni in the 2nd layer (figure 1(b)), and Ni with Ni in the 2nd layer (figure 1(b)). Similar arguments apply to the rest of the peaks in <100> and to the incident angle scan parallel to the <110> azimuth, figure 3.

The geometry of other surfaces studied in previous papers using the shadow-cone approach [1,2], attempt to quantify the surface relaxation and reconstruction. However, the work presented here suggests that individual focussing interactions need to be isolated, so that retracing back the various focussing interactions to determine the positions of surface atoms maybe possible regardless of the large variety of focussing interactions occurring within a small angular spread. It seems, at the moment, that the angular resolution in this experiment was not adequate enough to enable an accurate geometrical analysis to be carried out on this surface. More work is required to interpret the low energy induced shadow-cone’s effect, both short range and long range, on the sputtering yield of clean monocrystal targets.

References

Table 1: Angles and focussing interactions for the <100> and the <110> azimuths.

<table>
<thead>
<tr>
<th>Azimuth</th>
<th>Glancing Angle (degrees)</th>
<th>Focussing Interaction Source</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;100&gt;</td>
<td>7</td>
<td>Al(1st layer) Al(1st)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>Ni(1st) Ni(1st)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>13</td>
<td>Al(1st) Al(1st)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>14</td>
<td>Ni(1st) Ni(1st)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>17</td>
<td>Al(1st) Ni(2nd)</td>
<td></td>
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<td></td>
<td>18</td>
<td>Ni(1st) Ni(2nd)</td>
<td></td>
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<td>Ni(1st) Ni(2nd)</td>
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<tr>
<td></td>
<td>60</td>
<td>Al(1st) Ni(2nd)</td>
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<tr>
<td></td>
<td>64</td>
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<tr>
<td>&lt;110&gt;</td>
<td>10</td>
<td>Al(1st) Al(1st)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>11</td>
<td>Ni(2nd) Ni(2nd)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>12</td>
<td>Ni(1st) Ni(1st)</td>
<td></td>
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<tr>
<td></td>
<td>17</td>
<td>Al(1st) Ni(1st)</td>
<td></td>
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<tr>
<td></td>
<td>18</td>
<td>Ni(2nd) Ni(2nd)</td>
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<td></td>
<td>19</td>
<td>Ni(1st) Al(1st)</td>
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<tr>
<td></td>
<td>44</td>
<td>Ni(2nd) Ni(4th)</td>
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<td></td>
<td>44</td>
<td>Al(1st) Al(3rd)</td>
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<td>45</td>
<td>Ni(1st) Ni(3rd)</td>
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<td>66</td>
<td>Al(1st) Ni(3rd)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>67</td>
<td>Ni(2nd) Ni(4th)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>68</td>
<td>Ni(1st) Al(3rd)</td>
<td></td>
</tr>
</tbody>
</table>

Note: Glancing angle refers to the angle from the plane of the surface.
POLAR INCIDENT ANGLE SCAN OF A1+, PLANE:<100>.

Figure 2: Polar incident angle scan for the <100> azimuth.

POLAR INCIDENT ANGLE SCAN OF A1+, PLANE:<110>.

Figure 3: Polar incident angle scan for the <110> azimuth.
ION ASSISTED DEPOSITION OF DIAMOND FILMS
CHARACTERISATION OF APPARATUS.
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1. Introduction

The production of synthetic diamonds from low pressure gases was first achieved in 1911 by Von Boltan. However, detailed investigation of diamond formed by vapour deposition techniques began primarily in the Soviet Union and the United States in the 1950's. More recently the Japanese have begun major studies of these techniques. Development has been such that there have been many review articles written on the progress of research into diamond thin films [1]. The motivation for the production of diamond films is their potential use as an advanced material. Diamond's many properties such as hardness and high thermal conductivity have lead to its use in cutting and grinding tools and to research into high speed electro-optic devices.

Present work in this field involves research into the area of ion assisted deposition (IAD) of diamond films [2]. It is the characterisation of apparatus used in the implementation of this method that we present here.

2. Experimental work

The experimental set up is illustrated in Fig.1. The apparatus consists of a 3 cm Kaufman type ion source mounted 21 cm (grid to cup dist.) below a 1mm diameter faraday cup and an e - beam evaporator. The faraday cup was used to measure current density in the centre of the beam. Film thickness was measured using a quartz crystal oscillator film thickness monitor and the thickness was verified using a Dektak profiler. (N.B. The ion source and the evaporator were tested independently).

3. Results and Discussion

From work done by Ogata et al [2,3] it has been found that diamond formation using IAD is not only dependent on the energy of the ions hitting the film but also on the ion-atom arrival rate ratio. Hence, if similar results are to be produced both the ion source and carbon source must be characterised.

3.1 Behaviour of the Carbon Source

Tests of evaporation rate control were made at two different power levels of the beam and the beam was rastered across the carbon source to allow even power dissipation. In accordance with the work done by Ogata et al [2,3] the evaporation rate aimed for was 1.3 Å/sec.

<table>
<thead>
<tr>
<th>Beam Power</th>
<th>Evaporation Rate</th>
<th>Control Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.875 W</td>
<td>Av. 4.0 Å/sec max. 7.8 Å/s</td>
<td>Rate hard to control and will not stay at low rate. (1100Å deposited).</td>
</tr>
<tr>
<td>0.8 W</td>
<td>1.0 – 1.5 Å/s</td>
<td>Some initial fluctuation of the evaporation rate, but settled down to become fairly constant. (350Å deposited).</td>
</tr>
</tbody>
</table>

From the results it can be seen that the lower power density used allowed more control over the carbon deposition rate.
3.2 Characterisation of Ion Source

Ogata et al [2,3] has also shown that ion energy and ion mass as well as ion-atom arrival rate ratio effects the formation of diamond. This work has shown that diamond formation occurs more readily for low ion energies [Fig.2] and also that diamond crystallization varies with ion mass.

The method used for characterising the ion source was similar to that used by D. Van Vechten et al [4] in characterising a similar ion source for nitrogen feed gas. The feed gas used here for characterising the ion source was neon.

The ion-atom arrival rate ratio is an important factor in the formation of diamond films. Hence, the variation of current density at the substrate is important if diamond crystals are to be formed. Major factors affecting the current density are substrate to ion source distance, beam current ($I_b$), gas pressure in the chamber, and beam energy (or voltage). The ion source to substrate distance was kept constant as we were interested in the source characteristics alone.

Fig 3(a) shows the dependence of current density at the substrate ($I_{fc}$) on beam current ($I_b$) is linear. It also shows that this linearity occurs for both energies and that current density increases slightly with a decrease in beam energy. This last observation would have to be verified by further experimentation.

Fig 3(b) shows the dependence of current density ($I_{fc}$) on chamber pressure for a fixed beam current ($I_b$) of 5mA. The decrease in $I_{fc}$ with increasing pressure is mainly caused by charge exchange occurring in the space between the ion source grids in the faraday cup [4].

4. Further work

Further characterisation of this system is being done for different ion energies and for different gases of atomic weight less than Neon.

References


Figure 1. Experimental set up for the characterisation of diamond film apparatus.

Figure 2. The relation between neon ion energy and transport ratio of Ne/C for crystalline growth of carbon films. Area of slash marks indicates the optimum conditions for crystallization. [3]

Figure 3(a) Faraday cup current vs beam current for a gas pressure of $2.5 \times 10^{-4}$ Torr.

Figure 3(b) Faraday cup current vs gas pressure in chamber for beam voltage = 250 V and $I_b = 5$ mA.
Structure analysis of O on Ni(001) and Ni₃Al(001) surfaces by low energy ion beams

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1. Introduction

Low energy ion scattering spectroscopy (LEIS) has been used to determine the structure of oxygen covered overlayers on both Ni(001) and Ni₃Al(001) surfaces in a direct and straightforward way. The intensities of backscattered projectile particles (noble gas and alkali ions) are measured as a function of the incident and azimuthal angles with respect to the surface, supplying a real space image of the adsorbed atom positions. On the basis of shadowing and blocking of the projectile particles the location of the oxygen atoms on the surface has been obtained.

2. Experimental

The experiments were conducted in two UHV LEIS systems. System A is a Leybold-Heraeus 3-dimensional angular resolved energy spectrometer operating in the constant retardation mode with energy resolution (E/ΔE) of 200. This system is equipped with a 3M ion gun. System B is a Vacuum Science Workshop angle resolved ion scattering system operating in the constant pass energy mode at 125 eV with 2% energy resolution. It contains a three-grid LEED optics. In both systems the sample can be rotated around two perpendicular axes through the sample surface to control angle of incidence and azimuthal orientation of the sample. Both vacuum chambers are pumped by a combination of turbomolecular and titanium sublimation pumps which achieve a base pressure of 1x10⁻¹⁰ mbar. The Li⁺ ion beam was only used in system B. The ion source in system B is a Colutron gun with a Wien filter as the mass selector.

3. Structure of the C(2x2)O-Ni(001) surface

Structure information about the oxygen binding site can be obtained from an examination of the incident angle dependence of the Li⁺-Ni scattering shown in fig. 1. The similarity in shape of the incident angle scans between the clean and C(2x2)O surface shows that the Ni surface is essentially unreconstructed by the adsorbed oxygen atoms. However, there are some important changes in the scans. All of these changes can be explained by the effects of oxygen adsorbed at fourfold hollow sites. The decrease in Ni intensity near focusing peak edge S₁₁ is a result of shadowing by oxygen atoms. Assuming the oxygen is located 0.9 Å above the surface, the first layer Ni atoms is exactly shadowed by oxygen at an incident angle of \( \alpha = 27° \). Taking into account the size of the oxygen shadow cone, first layer Ni atoms behind O atoms should begin to appear at angles of \( \alpha > 44° \), giving rise to the new edge labeled S₁O in fig. 1.

Computer simulations for Li⁺ scattering were performed on a two dimensional array of Ni and O atoms along the [100] azimuth for the first four monolayers. The height of the O above the first layer Ni atoms is sensitive to the expected critical angle \( \alpha_c \). Fig. 2 shows the calculated critical angle as a function of the O height at a scattering angle of 90°.
Curves are shown for the ZBL and the TFM potentials. The screening length used in TFM are the values used by O'Connor and Bieasack [1]. The angle at 80% of maximum intensity is taken as the critical angle [2]. The experiment yields the 0 height of 0.88±0.05 Å at the fourfold hollow sites. The value is in agreement with other recently reporteded results [3,4].

Fig. 1 Li⁺-Ni Scattering intensity as a function of the incident angle α for the clean and C(2x2)O surfaces along the [100] azimuth. Scattering edges are denoted schematically.

Fig. 2 The calculated critical angle αc along the [100] azimuth is shown as a function of the 0 height above the first Ni layer.

4. Oxygen adsorption on Ni₃Al(001) surface

The Ni₃Al alloy has the Cu₃Au (L1₂) structure (fig. 3) with a lattice constant of 3.56 Å [5]. Our results show that the (001) plane has a 50% Ni-50% Al termination on the outermost layer; while the second layer has a pure Ni. There was no evidence from the LEIS analysis of enrichment of the surface in either constituent in the clean and well annealed surface. We note also that upon annealing a clean Ni₃Al(001) surface in the absence of reactive gases, no significant variation of the Ni/Al intensity ratio was found for temperatures up to about 850°C. These data indicate that a surface cleaned and well annealed is compositionally stable between room temperature and 850°C.

Fig. 4 shows the oxygen uptake experiments as a function of exposure at a temperature of 700°C. The sample was cleaned by annealing between each exposure. With increasing oxygen exposures, the oxygen peak increases greatly, while Al and Ni peaks decrease. The net intensity decrease corresponding to scattering from the Ni atoms is much greater than that from the Al atoms. Finally, at the 0 exposure of 100 L, the Ni peak almost disappears in spite of the presence of a considerable Al peak. Although it is well known that neutralization of He⁺ ions during scattering at the surface may be influenced by adsorbed oxygen atoms, leading to an increase in He⁺ neutralization. However, the significant decrease of the scattered ion intensity from the Ni atoms can only be understood if the Ni atoms were preferentially shadowed by the oxygen atoms. The most probable explanation for this behavior is that the oxygen bonds directly to the surface Ni atoms and not to the Al atoms in the initial stage of oxygen adsorption. This model is also supported by analysis of both the azimuthal dependence of the He⁺ ions and the incident angle dependence of the O⁻
recoil ions. These results will be published later.

![Perspective view of a unit cell of the Ni₃Al structure.](image)

![He⁺ ion scattering energy spectra.](image)

5. discussions

Oxygen chemisorbed on the Ni(001) and the initial stage of oxygen adsorption on Ni₃Al(001) surfaces have been investigated by LEIS and LEED. For the C(2×2)O-Ni(001) surface, the results confirm the O atom is located above the fourfold hollow site with the O height of 0.08±0.05 Å. This is in agreement with a symmetric fourfold chemisorption position for the oxygen, although a recent electron diffraction study of Demuth et al. [6] suggests that the C(2×2)O overlayer structure is displaced nearly 0.3 Å from the hollow position towards the bridge position. The suggestion that the oxygen atoms are chemisorbed preferentially at the Ni sites of the Ni₃Al(001) surface seems strange, since the oxidative activity of Al is higher than that of Ni due to the well-known affinity of aluminium for oxygen. At present, the origin of the preferential oxygen chemisorption at the Ni sites is not understood well enough to describe how the alloy species interact with oxygen. However, as a possibility, it may be explained as a drastic modification of the electronic properties of the Al by its interaction with the Ni.

More detailed information about structure of oxygen adsorption such as the vertical position of the oxygen are not obtainable from the limited LEIS results presented here. Little information about adsorbate structure on Ni₃Al ordered alloys in the literature. Confirmation this model will require much further both theoretical and experimental work to examine it, in order to clarify the details of the O adsorption on Ni₃Al surfaces.

References:

Empirical Formulae for Proton Energy Straggling in Elemental Materials

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ABSTRACT

The straggling in energy loss is a very important parameter in the field of atomic collision in solids especially in ion beam analysis, and has been studied both theoretically and experimentally in the past few decades [1-5]. But experimental study on straggling was far from enough than that on energy loss.

Although there are much fewer experimental data for energy straggling than for stopping power, a lot of experimental data of proton energy straggling in most common elemental materials has still been collected under a literature search [3-31] (more than 1000 data points from about 30 papers for proton). With weighted least-square-fit calculations, we have obtained empirical formulae for proton energy straggling in various target materials while there are no comments on data accuracy. The formulae for fitted straggling are chosen as:

$$\frac{\sigma^2}{\bar{\Delta}^2} = \frac{1}{1 + A1 \cdot E^2 + A3 \cdot E^4}$$  (1)

where $E$ is proton energy in MeV/amu, and $A1-A4$ are coefficients determined by least-square-fit.

The results are shown in Fig.1-2 and Table 1. The straggling data from monatomic gas target have more reliability and accuracy than solid target because the inhomogeneity of solid thin films cause additional spread in energy loss spectrum, which is proportional to the square of stopping power [5]. It is seen from Fig.1 that the experimental data of gas target are in agreement qualitatively with theoretical results except for experimental results being obviously greater in the region of 100<E<500 KeV, which relate to large stopping area. For solid target, the data dispersed much more especially for light elements (such as C, Al and Si). For Bohr's theory gives the high energy limit of electronic straggling, the data greater than $\bar{\Delta}$ may have less reliability and have smaller weight in fitting.

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Table 1. The fitted coefficients in equation (1)

<table>
<thead>
<tr>
<th>Element (Zz)</th>
<th>A1</th>
<th>A2</th>
<th>A3</th>
<th>A4</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>1.076x10^{-2}</td>
<td>-1.045</td>
<td>4.457x10^{-3}</td>
<td>-1.910</td>
</tr>
<tr>
<td>Ne</td>
<td>1.704x10^{-2}</td>
<td>-1.041</td>
<td>4.697x10^{-3}</td>
<td>-1.991</td>
</tr>
<tr>
<td>Al</td>
<td>3.060x10^{-2}</td>
<td>-0.9257</td>
<td>9.937x10^{-3}</td>
<td>-1.945</td>
</tr>
<tr>
<td>Si</td>
<td>3.951x10^{-2}</td>
<td>-0.9048</td>
<td>9.947x10^{-3}</td>
<td>-1.974</td>
</tr>
<tr>
<td>Ar</td>
<td>1.112x10^{-1}</td>
<td>-0.6453</td>
<td>5.690x10^{-2}</td>
<td>-0.6570</td>
</tr>
<tr>
<td>Ni</td>
<td>1.123x10^{-1}</td>
<td>-0.6274</td>
<td>2.848x10^{-2}</td>
<td>-1.798</td>
</tr>
<tr>
<td>Cu</td>
<td>2.294x10^{-1}</td>
<td>-0.6237</td>
<td>5.887x10^{-3}</td>
<td>-1.598</td>
</tr>
<tr>
<td>Ge</td>
<td>1.902x10^{-1}</td>
<td>-0.6114</td>
<td>1.502x10^{-2}</td>
<td>-1.712</td>
</tr>
<tr>
<td>Se</td>
<td>1.565x10^{-1}</td>
<td>-0.6018</td>
<td>3.028x10^{-3}</td>
<td>-2.447</td>
</tr>
<tr>
<td>Kr</td>
<td>3.880x10^{-1}</td>
<td>-0.5883</td>
<td>3.082x10^{-2}</td>
<td>-0.6059</td>
</tr>
<tr>
<td>Ag</td>
<td>3.878x10^{-1}</td>
<td>-0.5698</td>
<td>4.084x10^{-3}</td>
<td>-1.440</td>
</tr>
<tr>
<td>Xe</td>
<td>4.048x10^{-1}</td>
<td>-0.5597</td>
<td>1.036x10^{-1}</td>
<td>-0.5441</td>
</tr>
<tr>
<td>Au</td>
<td>4.939x10^{-1}</td>
<td>-0.4538</td>
<td>4.494x10^{-2}</td>
<td>-0.7586</td>
</tr>
<tr>
<td>Pb</td>
<td>6.024x10^{-1}</td>
<td>-0.2210</td>
<td>3.985x10^{-2}</td>
<td>-1.420</td>
</tr>
</tbody>
</table>

In conclusion, we have analyzed the experimental data and obtained empirical formulae for proton straggling in most common elemental materials without comments on data accuracy. Equation (1) with A1-A4 listed in table 1 are fitted and applicable in the energy range of 10<E<10000 MeV/amu.

Fig. 2 Energy Straggling of proton and deuteron in solids vs energy.

--- fitted; -----------Lindhard.

(a) In C, Al and Si;
(b) In Ni, Cu, Ge and Se;
(c) In Ag, Au and Pb.

--- E (MeV/amu)
--- Q/π (σ/μm)
REFERENCES

ION BEAM ANALYSIS ON THE LUCAS HEIGHTS TANDEM

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Introduction

In September 1989 the Rutgers University FN Tandem Accelerator arrived in Australia and was placed on its stanchions on a concrete pad at Lucas Heights. It is hoped to have the machine up to voltage by mid 1990 and beam on target by the end of that year. The programs envisaged on this machine include Accelerator Mass Spectrometry (AMS), a Radiocarbon Dating Service, Ion Beam Analysis (IBA) techniques and some neutron irradiations and have been well documented in the lengthy proposals[1,2] that were written for the justification of this facility. The AMS aspects of this facility have been discussed elsewhere at this Conference by J.R. Bird and R. Davie and here we shall try to summarise the proposed IBA program in the short term.

The Proposed Beamlines

The joint proposal documents identified several topics, within the Ion Beam Analysis area, that we considered to be worth pursuing in the national interests, these included

- Ion beam analysis techniques such as PIXE, PIGME, NRA and ERD for industrial, environmental and biological studies.

- High intensity heavy and light ion beams for materials science applications, industrial uses such as thin layer activation, and ion beam irradiations for implantation and dynamic radiobiological studies.

- Intense, neutron fluxes for safeguards development, industrial applications and radiobiological studies.

- A small mission orientated basic Atomic and Nuclear Physics program.

In order to meet these goals 3 of the 4 beam lines on the FN Tandem will be devoted to IBA techniques. A schematic of the beamline layout is shown in Figure 1. The Accelerator Mass Spectrometry line, R5, is discussed elsewhere at this Conference.

L1, is the IBA line which is based around a 40 cm diameter scattering chamber containing the PIXE, PIGME, RBS and NRA techniques. With these techniques ANSTO should be able to provide a comprehensive elemental surface analysis service covering the majority of the periodic table. Heavy ion PIXE will compliment our low energy light ion work for elements above Si, heavy ion RBS will provide better depth and mass resolution for profiling heavy elements in a light matrix. Nuclear reactions such as $^1H(\bar{N},\alpha\gamma)^12C$ at 6.4 MeV for profiling hydrogen, or the $^{12}C(p,p')^{12}C$ and the $^{16}O(p,p')^{16}O$ reactions at energies between 5 and 8 MeV for C and O analysis provide good methods for determining light elements in a heavy matrix.
RI is the Ion Beam Irradiations Leg (IBR) and will also be used for techniques requiring the beam to be brought out into air through a thin window, such as thin layer activation (TLA) studies. TLA has been used extensively in industrial research as a sensitive method for monitoring absolute mass loss by corrosion, erosion or wear.

Work as early as 1976[3] showed that for an irradiation of 10 MeV protons at 1 µA for 1 hour sensitivities of 10 ppb for Ca, Ti, Cr, Ni, Cu, Ga, Ge, Zn, Se, Br, Rb, Y, Zr and Mo; and 100 ppb for Li, S, V, Fe, As, Sr and Nb were obtainable using the (p,n) reaction techniques of TLA.

Other selected activation reactions are useful for determining light trace elements in a heavy matrix where standard RBS techniques are generally poor. For example, \( ^{12}\text{C}(^6\text{Li},\alpha)^{13}\text{N} \) above 7 MeV has been applied to determine carbon in steel down to 30 µgg\(^{-1}\), and \(^9\text{Be}^+\) ions at energies between 7 and 14 MeV may be used via the \(^{10}\text{Be}(^9\text{Be},n)^{18}\text{F} \) and \(^{14}\text{N}(^9\text{Be},\alpha)^{18}\text{F} \) reactions to determine B and N concentrations down to a few 10's of µgg\(^{-1}\). The \(^{14}\text{N}(p,n)^{14}\text{O} \) activation reaction at 16 MeV may also be useful for measuring nitrogen (in protein for example). The decay of \(^{14}\text{O} \) produces a 2.31 MeV γ-ray with a half life of 71 seconds and for proton energies below 21 MeV no other reaction can yield \(^{14}\text{O} \).

The L2 microprobe leg has not been fully specified at this stage, but it is envisaged that the beam spot size will be large between 10 and 100 µm in diameter, will be swept over ~ 1 cm\(^2\) and used for heavy ion PIXE and RSB scanning of selected industrial samples.

In summary the Ion Beam Analysis techniques that are provided at ANSTO within the Applied Nuclear Physics Program Area are comprehensive, cover a very broad range of ion energies from as low as 100 keV to tens of 10 MeV using both the 3 MV Van de Graaff and the 8 MV FN Tandem accelerators. Our techniques are particularly applicable to thin films, polymers, metals, semiconductors and a variety of problems in environmental and biological areas.

References


Figure 1
ABSTRACT

Ion irradiation of polymeric films can significantly alter the physical and chemical properties of the near surface region of such materials. These changes are a direct consequence of the energy deposited by the slowing ions and include processes such as scission and/or crosslinking of polymer chains, the generation of free radicals and the loss of volatile molecular species during the collision cascade process. Properties such as solubility, wettability, electrical resistivity, hardness and optical density are examples of properties which can be modified by ion bombardment. Recent studies have shown that the refractive index of polymers can also be altered by ion irradiation. This raises the possibility of fabricating integrated optical waveguides in polymers.

This paper reports the results of a preliminary investigation of waveguiding effects in ion irradiated poly(methyl methacrylate) (PMMA). In particular, waveguiding behaviour (number of guided modes and attenuation factors) is reported for PMMA thin films irradiated with inert gas ions in the energy range 50–100keV, and for ion fluences in the range $1 \times 10^{12} - 1 \times 10^{16}$ ions/cm$^2$. The effect of the different ion irradiation conditions on guiding is examined in terms of the dominant energy deposition processes and a probable mechanism for the irradiation induced refractive index change is discussed.
Measurement of Hydrogen at Metal - Semiconductor Interfaces

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Abstract

The enhancement of metal adhesion to semiconductors by ion, electron and photon irradiation has long been noted\(^1,^2,^3\). Since this discovery, however, no clear explanation of the exact mechanism has been established. The models proposed so far include atomic mixing, electrostatic mechanisms due to charge distribution and new bond formation involving interface chemistry. Recently it has been shown that different mechanisms may apply to different systems\(^4\).

Substrates prepared under typical vacuum conditions have monolayer films of impurities e.g. water and carbon containing compounds\(^5\). Where such films exist, excitation by ions, electrons or secondary electrons is likely to induce chemical reaction between this film and either its interface with the semiconductor or the thin metal film. Any such impurity chemistry would likely involve Oxygen or Hydrogen with the formation or destruction of oxides, hydroxides etc. In this study we investigate the role of Hydrogen in the chemistry of metal-semiconductor interfaces.

Rutherford Backscattering Spectrometry, RBS, is a powerful technique for determining the mass of atomic constituents in a solid target. The energy lost by the incident beam in its collision with a target atom is dependent on the mass of the target atom. When the mass of the target atom is equal to or less than the mass of the incident particle then a large fraction of the incident energy is transferred to the lighter target atom which recoils from the target in a forward direction\(^6\). By
orienting the target at a grazing angle to an incident Hé\textsuperscript{++} beam, therefore, and placing a detector at a forward angle with a Hé\textsuperscript{++} filter, Hydrogen can be detected by forward recoil spectrometry (see fig.1).

In the present study films of Al and Au were evaporated onto semiconductors in a diffusion pumped chamber at 2-5x10\textsuperscript{-6} torr. Electron irradiation was carried out in an Auger Spectrometer at 6-8KeV at fluences between 10\textsuperscript{16} and 10\textsuperscript{17} cm\textsuperscript{-2} at low beam current to minimise heating. The concentration of Hydrogen is measured on irradiated and unirradiated samples and the results are correlated with adhesion change.

References

fig.1. Experimental Set-up for Forward Recoil Spectrometry.
SELENNUM IN THE BLOOD AND FOOD OF AUSTRALIANS

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Abstract

Selenium determination by neutron activation analysis via both short and long-lived selenium isotopes was performed on 98 samples of whole blood taken from Sydney workers. The average selenium concentrations were found to be 0.103 and 0.108 μg mL⁻¹ respectively. A similar analysis protocol was used to analyse 350 foods sampled Australia-wide in order to identify the major sources of selenium intake.

Introduction

Selenium has long been recognised as an essential trace element within the human system¹,² and this fact has been reflected in the data found within the compilation reports of Iyengar³,⁴. While there is a large amount of published data for levels of selenium in human blood and its components for many overseas countries, few Australian data exist. For this reason, the selenium content of the whole blood from 98 workers at Lucas Heights, Sydney, was studied using neutron activation analysis.

Earlier measurements⁵ in our laboratory have indicated that the average selenium content in the plasma of Australians is significantly lower than many Western countries including Japan, Canada and United States². For this reason, food samples typical of an Australian diet were analysed to identify the major sources of selenium intake.

Experimental

Blood and Food via ‾⁷⁷mSe

Whole blood samples were obtained from staff at Lucas Heights and were separated into red cells and plasma by centrifugation. Red cells were washed twice with isotonic tri-ammonium citrate solution, weighed into pre-cleaned (A.R. 8M nitric acid, high-purity demineralised water and A.R. ethanol) polythene irradiation containers and dried in a vacuum desiccator. Plasma samples were prepared using ultrafiltration⁶ and dried as for red cells. Haematocryt values obtained at sampling then enabled whole blood selenium values to be calculated.

Food samples, obtained as part of the National Health and Medical Research Council's (NH&MRC) autumn Market Basket Survey, were freeze dried, homogenised, and weighed into pre-cleaned polythene irradiation containers (food groups suitable for analysis via ‾⁷⁷mSe had been previously identified by a trial run).

A selenium laboratory standard, prepared by dissolution of 99.9% selenium metal (Johnson Matthey) in HNO₃, and the NBS Standard Reference Material, Bovine Liver (SRM 1577), were both similarly prepared in polythene.
Samples and standards were packed into polythene "rabbits" and individually irradiated in the XI76 self-service pneumatic tube of the Australian Nuclear Science and Technology Organisation's 10 MW reactor, HIFAR, for 15 seconds at a thermal flux of $2 \times 10^{13}$ n cm$^{-2}$ s$^{-1}$. After 20 seconds decay, they were counted for 20 seconds. Irradiation timing was controlled by an ND660 multichannel analyser system (15% Ortec Ge(Li)) which was also used to count the 162 keV photopeak of $^{77}$Se.

Blood and Food via $^{75}$Se

Different samples of whole blood, obtained from the same source as above, were individually freeze dried, homogenised and sealed into pre-cleaned (as for polythene) quartz ampoules. Freeze dried food samples were sealed into polythene irradiation containers. Selenium laboratory and Bovine Liver standards were also prepared in quartz or polythene as for respective samples.

Whole blood and food samples were irradiated (7 days and 24 hours respectively) with standards in HIFAR at a thermal flux of $5 \times 10^{12}$ n cm$^{-2}$ s$^{-1}$ and were counted after 28 days' decay on a 15% Canberra Ge(Li) detector coupled to a 4096 channel Canberra Series 40 multichannel analyser. Selenium in blood and food samples was determined via the 265 keV photopeak of $^{75}$Se.

Results and discussion

The results for the analysis of 98 whole blood samples for selenium are summarised in Table 1. The results (mean and range) for the analysis via $^{77}$Se and $^{75}$Se methods are in good agreement and show that there is little statistical difference between whole blood samples of females and males.

The results compare favourably with the median world value of 0.105 µg ml$^{-1}$ reported by Iyengar and Woittiez$^6$ although lower than other western countries such as Canada, USA and UK (180, 157 and 136 µg ml$^{-1}$ respectively)$^4$.

The results (range and mean) for the analysis of 350 food samples for selenium are summarised in Table 2. While the majority of foods were of Australian origin, some canned products (meat, salmon, sardines) were imported. Results show that the best source of selenium intake is fish followed by meat, poultry, peanuts, and grain products. Both fruit and vegetables contain negligible amounts of this trace element.

Table 1
Selenium in whole blood.

<table>
<thead>
<tr>
<th>Isotope used</th>
<th>No. of Samples</th>
<th>Sex</th>
<th>Mean (µg ml$^{-1}$)</th>
<th>S.D.(a) (µg ml$^{-1}$)</th>
<th>Range (µg ml$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>via $^{77}$Se</td>
<td>30</td>
<td>Male</td>
<td>0.103</td>
<td>0.013</td>
<td>0.080-0.130</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>Female</td>
<td>0.105</td>
<td>0.012</td>
<td>0.086-0.136</td>
</tr>
<tr>
<td></td>
<td>45</td>
<td>Both</td>
<td>0.103</td>
<td>0.013</td>
<td>0.080-0.136</td>
</tr>
<tr>
<td>via $^{75}$Se</td>
<td>42</td>
<td>Male</td>
<td>0.108</td>
<td>0.018</td>
<td>0.082-0.180</td>
</tr>
<tr>
<td></td>
<td>11</td>
<td>Female</td>
<td>0.108</td>
<td>0.021</td>
<td>0.076-0.145</td>
</tr>
<tr>
<td></td>
<td>53</td>
<td>Both</td>
<td>0.108</td>
<td>0.019</td>
<td>0.076-0.180</td>
</tr>
</tbody>
</table>

(a) Standard Deviation
Table 2
Selenium concentration in Australian foodstuffs
(ug g⁻¹ wet basis)

<table>
<thead>
<tr>
<th>Food</th>
<th>Range</th>
<th>Mean</th>
<th>Se Isotope</th>
</tr>
</thead>
<tbody>
<tr>
<td>White bread</td>
<td>0.04 - 0.15</td>
<td>0.08</td>
<td>long</td>
</tr>
<tr>
<td>Wholemeal bread</td>
<td>0.08 - 0.13</td>
<td>0.10</td>
<td>long</td>
</tr>
<tr>
<td>Infant cereal</td>
<td>0.06 - 0.12</td>
<td>0.09</td>
<td>short</td>
</tr>
<tr>
<td>Corn flakes</td>
<td>0.07 - 0.09</td>
<td>0.08</td>
<td>long</td>
</tr>
<tr>
<td>Wheat biscuits</td>
<td>&lt;0.01 - 0.18</td>
<td>0.18</td>
<td>long</td>
</tr>
<tr>
<td>White rice</td>
<td>0.01 - 0.05</td>
<td>0.03</td>
<td>long</td>
</tr>
<tr>
<td>Bran</td>
<td>0.09 - 0.24</td>
<td>0.16</td>
<td>short</td>
</tr>
<tr>
<td>Minced steak</td>
<td>0.06 - 0.19</td>
<td>0.12</td>
<td>short</td>
</tr>
<tr>
<td>Lamb</td>
<td>&lt;0.02 - 0.13</td>
<td>0.13</td>
<td>short</td>
</tr>
<tr>
<td>Pork</td>
<td>0.09 - 0.23</td>
<td>0.15</td>
<td>short</td>
</tr>
<tr>
<td>Chicken</td>
<td>0.14 - 0.23</td>
<td>0.18</td>
<td>short</td>
</tr>
<tr>
<td>Canned meat</td>
<td>0.09 - 0.14</td>
<td>0.12</td>
<td>long</td>
</tr>
<tr>
<td>Fish</td>
<td>0.22 - 0.34</td>
<td>0.29</td>
<td>short</td>
</tr>
<tr>
<td>Salmon</td>
<td>0.22 - 0.38</td>
<td>0.27</td>
<td>short</td>
</tr>
<tr>
<td>Sardines</td>
<td>0.25 - 0.78</td>
<td>0.57</td>
<td>short</td>
</tr>
<tr>
<td>Eggs</td>
<td>0.15 - 0.23</td>
<td>0.19</td>
<td>short</td>
</tr>
<tr>
<td>Liver</td>
<td>0.20 - 0.54</td>
<td>0.30</td>
<td>short</td>
</tr>
<tr>
<td>Full cream milk</td>
<td>&lt;0.01 - 0.03</td>
<td>0.02</td>
<td>long</td>
</tr>
<tr>
<td>Skim milk</td>
<td>&lt;0.01 - 0.03</td>
<td>0.02</td>
<td>long</td>
</tr>
<tr>
<td>Lactogen</td>
<td>&lt;0.01 - 0.01</td>
<td>0.01</td>
<td>long</td>
</tr>
<tr>
<td>Butter</td>
<td>&lt;0.02</td>
<td></td>
<td>long</td>
</tr>
<tr>
<td>Vegetables/Fruit</td>
<td>&lt;0.04</td>
<td></td>
<td>long</td>
</tr>
<tr>
<td>Infant dinner</td>
<td>&lt;0.02</td>
<td></td>
<td>long</td>
</tr>
<tr>
<td>Peanuts</td>
<td>0.06 - 0.21</td>
<td>0.14</td>
<td>long</td>
</tr>
<tr>
<td>Peanut Butter</td>
<td>0.05 - 0.11</td>
<td>0.08</td>
<td>long</td>
</tr>
</tbody>
</table>

References
ION BEAM INDUCED RESISTIVITY CHANGES IN GLASSY CARBON

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Introduction

Glassy Carbon (GC) is an interesting low density (1.5 g/cm³) form of carbon produced in a slow, controlled, high temperature degradation of certain polymer-type materials. The resulting material microstructurally consists of a tangle of graphite-like ribbons or microfibrils resembling the polymer chain configuration from which it was derived¹. It is moderately hard, chemically inert, thermally and electrically conducting, impermeable and biocompatible. Ion beam irradiation has been found to significantly enhance the wear resistance of GC²,³. When combined with the other properties of GC, a surface hardened GC may be a more attractive material for proposed bioengineering applications. In our attempts to understand the structural changes associated with this observed increase in wear, surface conductivity measurements have been conducted on ion implanted GC. Ion species included C, Xe and Si ions with doses ranging from 10¹⁴ to 10¹⁸ ions/cm².

Electrical conduction in crystalline graphite occurs mainly in the sheets of sp² bonded carbon atoms. At room temperature the resistivity along the basal plane has been measured to be as low as 40 micro-ohm-cm compared to values along the c-axis of 0.3-0.5 ohm-cm⁴. Introduction of defects into the basal plane has a great effect on the conduction in graphite⁴,⁵. For example, ion irradiation of graphite with C and Ne results in an increase of its resistivity by an order of magnitude⁵. These measurements show that the electrical conductivity can be used to monitor disorder in graphite based systems, and it was with this background in mind that we undertook DC conductivity measurements of ion beam irradiated Glassy Carbon.

Results

According to the manufacturer, the GC used in this study was heat treated to 2500°C and has a resistivity of 4.5x10⁻³ ohm-cm. The dose dependence of the resistance of irradiated Glassy Carbon (R) as compared to unirradiated carbon (R₀) is shown in Figure 1. As can be seen from the figure ion beam irradiation has been found to increase the resistance of GC by up to five orders of magnitude. It should be noted that these changes are much larger than those reported for ion beam irradiated graphite. The results have been plotted as a function of the calculated defect density, D*, estimated by

\[
D^* = \frac{\text{dose} \times (\text{vacancy production per ion})}{\text{volume of the affected area}}
\]

where vacancy production per ion and the depth of the affected region were estimated by TRIM⁶. The displacements per atom (dpa) is derived from D* by dividing by the atomic density of GC of 7.5 x 10²² carbon atoms/cm³. This method of displaying the data was chosen because it "normalizes" the data to take into account both ion mass and energy so that if knock-on damage is
the dominant factor governing the ion beam induced increase in the resistance, then the dependence of \( R/R_0 \) on \( D^* \) should be the same for all ions studied.

Displacements per Atom (dpa)

\[
\begin{array}{c}
10^8 \quad 10^7 \quad 10^6 \quad 10^5 \quad 10^4 \quad 10^3 \\
10^2 \quad 10^1 \quad 10^0 \quad 10^{-1} \quad 10^{-2} \quad 10^{-3}
\end{array}
\]

110 keV Si\(^{+}\)

320 keV Xe\(^{+}\)

50 keV C\(^{+}\)

R/\(R_0\)

\[
\begin{array}{c}
10^1 \quad 10^2 \quad 10^3 \quad 10^4 \\
10^5 \quad 10^6 \quad 10^7 \quad 10^8
\end{array}
\]

\[
\begin{array}{c}
10^{21} \quad 10^{22} \quad 10^{23} \quad 10^{24} \quad 10^{25} \quad 10^{26}
\end{array}
\]

Defect Density (Vacancies/cm\(^3\) )

Fig. 1. The resistance of Glassy Carbon (R) as compared to its unirradiated value (\(R_0\)) as a function of calculated defect density. The solid lines are a guide to the eye.

For all ions studied there is little or no change in the resistivity below damage levels of about 0.2 dpa. Above this level there is a very marked increase in the resistance. However the changes are more rapid for Si than for Xe or C ions. This may be due to the formation of amorphous SiC surface layers.

Discussion

The value of 0.2 dpa for the onset of changes in the resistance is to be compared with the critical dose reported for increased wear resistance of 0.5 dpa for He, C, N, O, F\(_2\) and Ne irradiation\(^2\). It is also similar to the damage level at which the material undergoes an ion beam induced transformation as measured by Raman spectroscopy at 0.21 dpa\(^8\).

Interestingly, whilst the resistance appears to saturate for Xe implantation above damage levels of about 13 dpa, in the case of C implantation the resistance shows a maximum at this damage level and a decrease towards the unirradiated value at higher doses. This decrease could be evidence for an ion beam induced increase in order in the system (i.e partial graphitization) as has previously been observed in Raman and conductivity investigations of ion beam irradiated amorphous carbons\(^7\).
There are two possible causes for the observed large increased resistance of ion beam modified GC. The first of these is the introduction of defects into the graphite planes which make up the ribbons in the GC structure by the ion beam. As is the case with graphite, defects either in the form of c-axis dislocations or point defects would impede conduction. The introduction of defects in the ribbons of the GC structure has also been used to explain the increase in wear resistance observed after ion implantation.

However, a second alternative explanation for the observed increases in electrical resistance may be an ion beam induced change from sp² (with its one delocalized electron per carbon atom) to sp³ type bonds (with no delocalized electrons) within the modified layer. The formation sp³ bonds would strengthen interplaner bonding and could explain the increase in wear resistance observed.

The temperature dependence of the conductivity in GC has been used previously to gain information about the nature of the transport mechanism. In particular, evidence has been presented to indicate that the conduction in GC is one-dimensional, i.e. along the ribbons. We now plan to measure the temperature dependence of the conductivity in ion beam modified GC. If the results of this study show no fundamental changes in the transport mechanism (i.e. still one-dimensional conduction), then the introduction of defects by the ion beam is the most likely cause for the increases in resistance observed. However, if the conduction transport mechanism has been altered so that conduction is no longer one-dimensional then the possibility that the ion beam has modified the ratio of sp² to sp³ bonds will have to be considered.

References


DETECTION OF TRACE COPPER LEVELS IN POLYMERS BY RBS

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Introduction
A problem encountered by cables composed of a copper conductor surrounded by a polymer insulator is that in time copper migrates into the polymer. This eventually leads to a degradation of the polymer and subsequent breakdown. The cost of replacing cables which are buried within a duct can be quite high. Additives, known as chelators, can be placed into the polymer to inhibit the migration of the copper and hence prevent breakdown. Because the breakdown process occurs slowly over a period of years, it is necessary to simulate the migration over a much shorter period. Parameters which are important include temperature, pressure and moisture. In order to evaluate the effectiveness of the additives, a technique is needed for measuring copper levels (concentrations of order 100 ppm) in the surface region (typically 1 micron). Rutherford Backscattering Spectrometry is ideal for this purpose. Since the polymers consist primarily of hydrocarbons with small amounts of oxygen, it is relatively easy to detect copper which is heavier than each of these elements.

Method
Polymer samples were clamped between copper plates in a "sandwich" arrangement and conditions of temperature, pressure and moisture were varied. Cycle times varied from one week to eight weeks. Some samples had no additive, others had one of a number of compounds designed to inhibit the movement of copper into the polymer.

Rutherford backscattering measurements were performed on the samples using 2 MeV helium ions. Two surface barrier detectors were used. One was at a back angle of 160° to give good mass resolution and the other was at a glancing angle of 100° to give depth resolution. Beam current integration
is difficult with polymer samples because they are insulators. A rotating vane with a 200 Å gold layer on a glassy carbon substrate was placed in the chamber so as to intercept the beam twice each second. RBS counts from this were scattered only into the back angle detector and the gold peak was well clear of the copper peak. The area of this peak was then used to normalise the beam current. Alternatively the carbon plateau could be used to monitor current for samples of the same polymer type. With beam currents of order 50 nA, sufficient data could be collected for each sample in about 15 minutes.

Glancing Angle RBS Spectra
Polymer Sample RF 11

Figure 1 shows glancing angle spectra (log vertical scale) for a polyethylene sample with no additives. Point 1 is where the copper plate was clamped and has the least copper and polymer oxidation. Copper migrates through the polymer at the point of stress relief and presumably oxygen availability at points 2 and 3 (dark band on specimen). The copper concentration here is about 1% and its depth is several hundred nm. After saturation of the initial area, some copper migrates at points 4 and 5. (Copper concentration =0.1%).

Figure 2 is for a sample which contains a polymeric additive and which has been handled the same way as the previous sample. It can be seen that there is virtually no evidence of copper at either the stressed area, point 1, or the unstrained area, point 2. The additive is clearly acting as an inhibitor to the migration of the copper.
Conclusion

Additives can be developed which prevent the migration of copper into the polymer and hence lifetime of the cable can be increased. Rutherford Backscattering Spectrometry provides a quick and reliable method of determining the concentration and depth of any copper in the top micron of the polymer.
Ion Beam Induced Damage and Element Loss in Biological Tissue

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Abstract
Specimen damage and element loss induced by 3 MeV proton and 2 MeV alpha particle bombardment of thin sections of freeze dried kidney tissue and embedding medium have been studied. Yields of ions scattered from H and C, and characteristic X-rays were measured versus accumulated charge, for current densities in the range from $8 \times 10^{-15}$ to $1.1 \times 10^{-11}$ A/$\mu$m$^2$. Structural damage, discolouration and loss of S, Cl, C and H have been observed. Implications of this study for ion beam microanalysis of biological tissues are discussed with particular reference to the results for H loss.

1 Introduction:
X-ray microanalysis of biological tissue involves the use of thin fragile specimens which can be damaged in various ways by an MeV microbeam. Structural damage, specimen or element movement, and shrinkage of an area of interest, or mass loss from the tissue matrix will affect concentration measurements, and discolouration and melting of the tissue makes identification of internal structure difficult. The damage incurred will become more severe as microbeam focusing improves towards sub-micron dimensions, increasing the loading parameter (C/$\mu$m$^2$) upon which beam damage depends. In order to maintain the integrity of elemental concentrations and distributions within the specimen the damage induced by the incident beam must not cause elemental loss and must be decreased to be below the resolution of the microbeam. In electron microscopy the limit in determining biological structure is the damage produced in the specimen due to the action of the irradiating beam [1], not the resolution of the beam.

2 Experimental Methods:
During irradiation of the specimen, particles scattered at 60° and 135° to the incident beam direction were detected by Silicon Surface Barrier charged particle detectors, and X-rays were detected by a Si(Li) X-ray detector. Beams roughly focused to 10 $\mu$m in diameter were rapidly and uniformly scanned over rectangular areas of the specimen. The microprobe data collection system was configured to monitor beam related effects by recording accumulated charge pulses on the same tape as that used to record the X-ray, Rutherford backscattered and Rutherford forward scattered events. Energy spectra for characteristic X-rays and scattered particles were then obtained from the stored data, and plots of yield versus accumulated charge were then created.

Concentration of elements was inferred from the characteristic X-ray yields for Na, P, S, Cl and K and target mass from the yield of bremsstrahlung continuum X-rays between the Cl and K$_\alpha$ X-ray peaks (Ar K$_\alpha$ X-rays are not observed in biological tissue microanalysis). H and C content were determined from the yield of beam particles forwards scattered and back scattered respectively, from H and C nuclei.

3 Results:
A 6 $\mu$m thick freeze dried cryosection of rat kidney in a hydrocarbon embedding medium for frozen tissue specimens (O.C.T.), was irradiated by a 2 MeV alpha particle beam with a current density of $1.8 \times 10^{-13}$ A/$\mu$m$^2$. No Na loss was observed whereas a decrease in S and Cl X-ray yields was observed. Then for the same specimen but for a current density of $1.5 \times 10^{-14}$ A/$\mu$m$^2$, no loss of Na, S or Cl was observed. Tissue structural damage and shrinkage for a given accumulated charge were also less for the latter case. For the

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data presented here, only H loss was continually observed, sometimes C loss was, and only rarely were any of the elements heavier than Na lost.

Beam current densities from $3 \times 10^{-13}$ to $1.1 \times 10^{-11} \text{ A/\mu m}^2$ for 3 MeV protons, and from $8 \times 10^{-15}$ to $1.8 \times 10^{-13} \text{ A/\mu m}^2$ for 2 MeV alpha particles were used to irradiate both the freeze dried kidney tissue and its embedding medium. The data obtained from scattered beam particles off H which includes the elastic recoil of H atoms from the specimen, were least squares fitted by two exponentials,

$$Y(d) = A \exp(-d/T_f) + B \exp(-d/T_s)$$

where $A$ and $B$ are constants, $T_f$ is the fast characteristic dose, $T_s$ is the slow characteristic dose and $d$ is the irradiated dose ($\text{C/\mu m}^2$). An example of such a fit to the decaying yield of scattered beam particles off H has been shown previously [2]. The results for the fast and slow characteristic doses have been plotted against current density in fig.s 1 and 2. The data obtained from scattered beam particles off C were adequately fitted by one exponential and a constant. It was found that C loss did not always occur when H loss was observed and therefore there were fewer characteristic doses obtained [4]. Therefore the results for H loss will mainly be discussed in this paper.

4 Discussion:

In fig.s 1 and 2, both alpha particle data and proton data are plotted together as are the results from the two specimens irradiated. Both the fast and slow characteristic doses for H loss increase approximately linearly with current density on a log-log scale, which implies that the rate of H loss increases for decreasing current density. This is not an intuitively apparent result. Sternglass [3] found that the yield of secondary electrons per second decreased significantly at higher specimen temperatures, and attributed this to the increased vibrations of the atoms about their equilibrium positions which decreased the mean free path of the secondary electrons. For higher densities (and hence for higher temperatures) the amplitude of atomic and molecular vibrations may have increased so that the mean free path for free or ionised H atoms was decreased. A consequence of this effect is the decreased escape probability of the H atoms from the specimen at higher current densities, i.e. there must be a concomitant increase in the recombination probability for H atoms as the current density increases. This may be explained by the number of broken bonds formed per unit time; such that for higher current densities a greater number of bonds will be broken per unit time, and hence for each free or ionised H atom there will be a greater number of locations to which it can recombine prior to diffusing out of the specimen than for lower current densities.

As the current density decreases the rate of loss of H from the pool of 'fast H lost' is increased. This result is clarified by the variation observed in the proportion of 'fast H lost' $A/D$, which indicated a decrease in the proportion of fast H lost for decreasing current density [4]. Therefore although the rate of H loss increased with decreasing current density, the size of the pool of 'fast H lost' was also decreasing.

5 Conclusion:

During ion beam microanalysis of freeze dried kidney tissue and its embedding medium, H and C loss were monitored according to the yields of forward and backscattered beam particles, and were found to be described by the sum of two decaying exponentials and the sum of one decaying exponential and a constant, respectively. The fast and slow characteristic doses describing the H loss differed by at least an order of magnitude, suggesting two clearly different pools of H with differing radiation sensitivity, for both the kidney tissue and its embedding medium. Both characteristic doses were also shown to increase with increasing current density.

6 References:


Figure 1: The fast characteristic dose $T_f$ as a function of current density $J$ for H loss from specimens bombarded by proton and alpha particle beams.

Figure 2: The slow characteristic dose $T_s$ as a function of current density $J$ for H loss from specimens bombarded by the proton and alpha particle beams.
CARBON–14 DATING WITH THE 14UD ACCELERATOR

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1. Introduction

The recent carbon-14 dating of the Turin Shroud has given a high public profile to the technique of accelerator mass spectrometry (AMS). Its major advantage over conventional radioactive-decay counting, which was crucial to the successful measurement of the Shroud’s age, is that it permits the use of very small samples (less than 1 mg of carbon).

At the ANU’s 14UD accelerator we have developed a carbon-14 AMS capability which uses essentially the same hardware as the successful chlorine-36 system. Graphitisation of samples has been carried out either in the Radiocarbon Laboratory or in the Department of Biogeography and Geomorphology. Present accuracy for a recent sample containing ~1 mg of carbon is 3% and is limited by system reproducibility rather than by counting statistics.

2. The Carbon-14 AMS System

A description of the principles of AMS is presented in another abstract to this conference (R.F. Davie et al.) so only features specific to \(^{14}\)C will be presented here. The graphitised samples are pressed into a 2mm diameter dimple in the centre of a "reflected geometry" sample holder for insertion into the ion source. Typical outputs from these samples are 10\(\mu\)A of \(^{12}\)C—ions, which is only 30% less than the output from a piece of commercial graphite. Accelerator terminal voltages of 7.00, 6.46 and 6.00 MV are employed when accelerating \(^{12}\)C, \(^{13}\)C and \(^{14}\)C respectively. At these voltages, \(4^+\) is the dominant charge state of the carbon ions after they have passed through the stripper foil in the high voltage terminal. These voltages are chosen so that all three species have the same magnetic rigidity after acceleration, thereby doing away with the need to adjust the various magnetic elements of the high energy beam transport when changing from one beam to another. A typical measurement sequence consists of measurements of 30s each of the \(^{12}\)C and \(^{13}\)C beam currents,
followed by a 10 minute counting interval for $^{14}\text{C}$. Because of the large $^{12}\text{C}$ currents provided by the ion source, it is necessary to attenuate the beam during the $^{12}\text{C}$ phase of the measurement sequence in order to reduce the loading of the accelerator. This is achieved with a beam chopper immediately after the ion source which discards all but 1/80 of the beam.

Carbon-14 ions are counted individually by an ionisation chamber which measures the total energy and the rate of energy loss of each ion that enters it. Typical count rates are $\sim 100 \text{s}^{-1}$, most of which are $^{12}\text{C}^3+$ and $^{13}\text{C}^3+$ ions which have undergone charge-changing collisions in the high-energy section of the accelerator. Generally, the total energy measurement and only one of the energy loss measurements are enough to identify a $^{14}\text{C}$ ion uniquely.

3. **Representative projects**

The following problems are representative of those being tackled with the $^{14}\text{C}$ AMS system.

(i) **Dating of paleosol layers in loess-paleosol sequences from the Loess Plateau in China.** Dating of these layers by conventional $^{14}\text{C}$ decay counting has proved difficult because of contamination by modern organic materials which percolate down from the intensive agricultural activity above. Techniques for chemical extraction of the different organic fractions in the sediments are being developed by the Radiocarbon Laboratory with the aim of identifying a fraction which comes only from the original organic material incorporated in the sediment. Often, the resulting samples are too small to date by decay-counting, and AMS is then the only means of measuring the $^{14}\text{C}$ in the various fractions.

(ii) **Dating of human bones from the Lake Mungo area.** Most bones from this area have been burnt and subsequently degraded by environmental processes. Hence they contain none of the collagen which is usually used for dating bones. In collaboration with the ANU's Department of Biogeography and Geomorphology, we have been exploring the possibility of dating this material by looking at different organic fractions with AMS.

(iii) **Dating of groundwater from the Kalgoorlie area.** This groundwater has a very low carbon content, and hence AMS can greatly reduce the amount of water which is required to obtain enough carbon to provide information on its age.
4. **Conclusion**

A carbon-14 AMS system is now in routine operation on the 14UD accelerator. It offers a modest precision of ~3% for samples that are >10% modern.
Introduction

Conventional ion implantation is a line of sight process where ion beams strike a surface, penetrate it and then come to rest below the surface. One of the major uses of ion implantation is in reducing wear between surfaces. In many cases these are planar surfaces, but in some situations they may be spherical or complex in shape. It is therefore necessary to manipulate the sample so that the beam reaches every part and a uniform dose is implanted over all the surface.

There are two conventional approaches to implanting large area samples—electrostatic scanning of the beam or mechanical scanning of the sample. Each has its advantages and disadvantages, although mechanical scanning is more common. Sometimes a combination of both techniques is used.

Our implanter is a research tool and the size, shape and composition of samples varies from one run to another. Samples which are implanted include rectangular planar surfaces up to 30 x 50 mm, spherical balls up to 40 mm diameter and rods 10 mm diameter and 100 mm in length. On some occasions ion implantation is performed with the sample heated to temperatures up to 350°C or cooled to liquid nitrogen temperatures.

It is difficult to incorporate all features into the one sample manipulator. This paper describes a novel device with rotational and translational degrees of freedom which is controlled by a PC type computer. It has the additional option that the sample may be operated at a potential of -50 kV to enable the maximum ion energy to be increased from 50 to 100 keV. This feature is described separately at this conference.

Equipment

Our implanter produces a beam which is typically a vertical strip 3 mm wide and 30 mm high. One project involves implanting the surface of a sphere about 40 mm in diameter. The requirement is to implant about two thirds of the area— all of the top hemisphere and the part of the lower hemisphere nearest to the equatorial zone. This requires two rotational degrees of freedom— one to rotate the sphere on its axis and the other to
change the tilt angle. In addition it is highly desirable to have a translational movement so that the sample can be shifted out of the beam and the beam current monitored and adjusted if necessary at various stages during the implantation.

The manipulator is designed to fit within a 40 cm diameter chamber with 20 cm diameter conflat flanges. The chamber is pumped to a vacuum of 100uPa. Three rotary feedthroughs are mounted on one flange. One of these drives a screw thread which contains a carriage which enables the entire sample holder and beam monitoring equipment to be translated at right angles to the beam by up to 150 mm. The rotary drives allow 360° of axial rotation and 120° of tilt variation and are linked to the carriage through a system of gears and drive shafts. All three feedthroughs are controlled by stepper motors which are in turn controlled by a PC computer.

Because of the option to bias the target to -50 kV to increase beam energy, the stepper motors (at ground potential) are linked to the sample holder by gears which are made from high density polythene, capable of withstanding voltages in excess of 50 kV in vacuum. The carriage is insulated from the ground potential flange by high density PVC.

The computer program is designed to continuously rotate the sample about its axis and to step the tilt angle so as to give uniform dose per unit area. This means slow stepping in the equatorial zone and much more rapid stepping in the polar region. From time to time the program translates the sample holder out of the beam to monitor the current and its uniformity and then moves it back in again. The program integrates total sample dose.

Although primarily designed to handle spherical samples, the manipulator is sufficiently versatile to handle cylindrical and planar samples and a variety of other complex shapes. Sample heating and cooling options exist, although these then exclude the use of the -50kV bias.

Reference
1) M.J.Kenny and L.S.Hielunski, A 50 kV Target bias system, this Conference.
ION BEAM INDUCED PHASE CHANGES IN GaAs

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ABSTRACT

Ion bombardment of GaAs has been studied to investigate damage production and the competition between defect production and dynamic annealing processes. The type of disorder produced is found to depend strongly on sample temperature and ion dose-rate. An irradiation regime has also been identified in which dynamic annealing processes can lead to the recrystallisation of pre-existing amorphous layers.

EXPERIMENTAL

Single crystal (100) GaAs samples were irradiated with 100 keV Ar ions to doses of up to \(2 \times 10^{15}\) ions cm\(^{-2}\) at temperatures in the range \(-196^\circ C\) to \(125^\circ C\). The ion dose-rate during irradiation was held at a constant value in the range \(0.04 \mu A\cdot cm^{-2}\) to \(2.7 \mu A\cdot cm^{-2}\). During irradiation the sample reflectivity was monitored in-situ using a HeNe laser. The sample reflectivity has been found to be a sensitive indicator of the residual damage levels (Johnson et al 1987). High resolution RBS-C was performed on selected samples.

RESULTS AND DISCUSSION

Figure 1 summarises the damage build-up as a function of implantation dose and temperature for a dose-rate of \(-1.2 \mu A\cdot cm^{-2}\). The reflectivity trace (figure 1a) has been normalised such that the crystalline value is 0 and the amorphous level is 1. At an irradiation temperature of \(25^\circ C\) the reflectivity trace shows a rapid build-up of damage, with amorphous phase formation occurring at a dose of \(1 \times 10^{14}\) ions cm\(^{-2}\). RBS-C measurements taken for small dose increments over this interval show that the damage build-up to amorphisation is directly proportional to ion dose. For an irradiation temperature of \(125^\circ C\) a small increase in reflectivity is observed, but RBS-C analysis is not sensitive to the level of damage produced, even for the highest irradiation dose.

A more interesting damage regime is seen to occur for an irradiation temperature of \(75^\circ C\). In this case the reflectivity trace indicates crystalline disorder (similar to the \(125^\circ C\) case) for doses up to \(8 \times 10^{14}\) ions cm\(^{-2}\). Beyond this dose there appears to be a rapid increase of damage until amorphisation occurs. RBS-C measurements clearly show that for doses below \(8 \times 10^{14}\) ions cm\(^{-2}\) the material is crystalline and that there is
a sudden collapse to the amorphous phase, which then rapidly extends to the sample surface for doses above this value. This behaviour is qualitatively described by the model of Vook and Stein (1969), which postulates that there is competition between defect production and annealing. For any given sample temperature/dose-rate combination there exists an equilibrium defect concentration where the defect production rate is balanced by the annihilation rate. Vook and Stein suggest that the crystalline structure of the material can only support a critical level of these defects, above which there is a spontaneous collapse of the lattice to the amorphous phase. The model predicts that as the dose-rate increases, this critical value will be reached for correspondingly lower doses and that for low enough dose-rates the equilibrium defect level will never reach the critical value for collapse to the amorphous phase. Figure 2 illustrates the reflectivity traces obtained by varying the ion dose-rate at a temperature of 75°C. It can be seen that for the lowest dose-rate amorphisation does not occur, whilst as the dose-rate increases, amorphisation occurs for a correspondingly lower irradiation dose. This behaviour is in agreement with the predictions of the Vook and Stein model.

For samples irradiated at low temperatures defect migration and dynamic annealing effects are minimised and the amorphisation process is best described by the model of Morehead and Crowder (1970) in which each ion produces an amorphous region about its track and complete amorphisation results from the overlap of these individual amorphous zones. Correlation of measured amorphous layer thicknesses with Monte Carlo simulations of the spatial distribution of the nuclear energy deposition for implants at -196°C show that the lattice converts to the amorphous phase for a deposited energy density of $3 \times 10^{20}$ keV·cm$^{-3}$.

Dynamic annealing effects during 1.5 MeV Ne irradiation have also been observed to result in the recrystallisation of pre-existing surface amorphous layers in GaAs. This recrystallisation is seen to occur at substrate temperatures as low as 75°C (Johnson et al 1988,1989).

REFERENCES
FIGURE 1 a) Reflectivity traces for damage build-up during 100 keV Ar irradiation at temperatures of 25°C, 75°C and 125°C. b) RBS-C spectra (2 MeV He) showing damage corresponding to 100 keV Ar doses of $2 \times 10^{14}$ cm$^{-2}$ at 25°C and 125°C.

FIGURE 2 Reflectivity traces obtained during 100 keV Ar irradiation of GaAs at a substrate temperature of 75°C, showing the damage build-up as a function of ion dose-rate.
A CORONAL J-POINT EXTENSION SYSTEM FOR LOW ENERGY OPERATION
OF THE VAN DE GRAAFF ACCELERATOR

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In recent times there has been a need to run the 3 MeV Van de Graaff Accelerator at terminal voltages ranging from 200 keV to 600 keV; considerably lower than those for which the corona stabilisation system was designed. The limiting factor was that of being able to draw adequate corona current at low terminal voltage to ensure reliable terminal voltage stabilisation, see Figure 1.

Corona current flows between the high voltage terminal, the tank insulating gas, a set of sharp needle points and a tetrode valve to ground. Changes in this current result in more or less charge residing on the terminal and appearing as voltage.

Various methods of achieving adequate corona current were investigated and were reported in the NTA Conference 1985¹. Briefly, the three techniques investigated included using more needle points, a lower insulating gas pressure or driving the points much closer to the terminal.

For reasons previously reported, the latter method was chosen and as a trial, careful measurements were taken between the needle points and the high voltage terminal, and an extension shaft constructed to reduce the gap between the terminal and points. The points fully in gap was reduced to ~ 30 mm with a subsequent improvement allowing accelerator operation down to 80 kV whereas the standard configuration allowed stable operation down to approximately 600 kV if the needle points were in good condition. Unfortunately, the points could not then be fully withdrawn for operation at higher terminal voltages. Either way of operating resulted in the time consuming and costly opening/closing of the tank and subsequent labour requirement.

Following this trial it was decided to design and construct a new corona points mechanism. All work, including the pressure vessel design considerations were carried out "in-house". The new mechanism consists of an extended drive shaft, 125 mm longer than the previous and an extended high pressure tubular housing in which the shaft rides, see Figure 2. Modifications were also made to the gearbox that drives a 10 turn potentiometer from which a meter reading indicating points position is obtained. Some minor electrical adjustment of this mechanism was also necessary to align the new system.
The new corona points mechanism has now been installed for several months and has proven reliable in operation. For a typical corona current of 80 \( \mu \text{A} \), terminal voltages in the range 68 kV to 3 MeV present no difficulties, see Figure 3. This simple modification has expanded considerably the range of terminal voltages possible and has thus provided a more versatile accelerator capable of being used in a broader range of experiments including high energy implants and accessibility to resonant nuclear reactions below 600 keV.

Reference

Fig 1 CORONA STABILISER SYSTEM
Fig 2 CORONA POINTS ASSEMBLY

Fig 3 CORONA POINTS

- $80 \mu$Amps
- $100 \mu$Amps
ION-BEAM-INDUCED EPITAXIAL CRYSTALLIZATION OF Ge_xSi_{1-x}/Si HETEROSTRUCTURES

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ABSTRACT

Ion irradiation at elevated temperatures is shown to promote epitaxial crystallization of amorphous Ge_xSi_{1-x} layers both from a (100) crystalline alloy of the same composition and from a (100) Si substrate. It is also shown that strained-layer configurations can be grown by ion-beam-annealing and that the commensurate-incommensurate transformation occurs at approximately the same critical composition (for a given thickness) as measured for layers grown by molecular beam epitaxy.

INTRODUCTION

Ion irradiation at elevated temperatures can induce epitaxial crystallization of amorphous Si layers from an underlying crystalline substrate [1]. This process has many advantages over conventional thermal annealing; it is a low temperature process, occurring at temperatures as low as 150 °C compared to the ~500 °C required for thermal annealing, the recrystallized material is confined to the irradiated area, enabling selective crystallization of specific regions, and it can be used to crystallize layers which have different crystallization temperatures, enabling epitaxial multi-layer structures to be fabricated.

In this study, ion-beam-annealing of Ge_xSi_{1-x}/Si structures is examined, with a view to fabricating strained-layer structures for optoelectronic device applications.

EXPERIMENTAL

Crystalline Ge_xSi_{1-x} alloy layers, with x in the range from 0.1 to 0.8, were grown to thicknesses in the range from 75 to 100nm on (100) Si substrates by molecular beam epitaxy (MBE). Samples were subsequently implanted with ^28Si ions to produce amorphous layers which extended either part way through the alloy layer or completely through the layer and into the underlying Si substrate. Epitaxial crystallization was then induced at 275 °C by irradiating with 300 keV ^28Si ions. The extent of crystallization was monitored by high depth resolution Rutherford backscattering and channelling analysis (RBS-C), for which channelling was with respect to the <001> axis, using a scattering angle of 100°. Samples were also examined by transmission electron microscopy (TEM).
RESULTS AND DISCUSSION

The RBS-C spectra in fig. 1 demonstrate the effect of ion-beam-annealing a thin amorphous Ge<sub>0.2</sub>Si<sub>0.8</sub> layer in contact with a crystalline alloy of the same composition (i.e., the amorphous layer is around half the alloy layer thickness). The spectra show that the amorphous layer crystallizes epitaxially during ion irradiation, with the 79nm amorphous layer being completely crystallized following a fluence of 8x10<sup>15</sup> Si.cm<sup>−2</sup>.

![Energy vs. Depth Graph](image)

Figure 1. RBS-C spectra of a thin amorphous Ge<sub>0.2</sub>Si<sub>0.8</sub> layer, before and after ion-beam-annealing. (---) initial amorphous layer, (.....) 4x10<sup>15</sup> Si.cm<sup>−2</sup>, and (- - -) 8x10<sup>15</sup> Si.cm<sup>−2</sup>.

TEM analysis of the recrystallized layer reveals that it contained a high density of radiation induced defects, mainly in the form of intermediate defect complexes (rods and elongated loops oriented in ⟨011⟩ directions), and dislocation loops and lines. Such analysis also revealed that the layer remained commensurate with the underlying Si substrate following ion irradiation; it is a strained layer, consistent with MBE grown material [2].

The ion-beam-annealing behaviour of a completely amorphous Ge<sub>0.5</sub>Si<sub>0.5</sub> alloy layer is illustrated in fig. 2. In this case the initial amorphous layer extended throughout the alloy layer and ~37nm into the underlying Si substrate. The spectra clearly show that ion-beam-annealing promotes epitaxial crystallization of both the amorphous Si and Ge<sub>0.5</sub>Si<sub>0.5</sub> alloy layers, crystallizing the amorphous Si layer and approximately half of the amorphous alloy layer following a fluence of 1.2x10<sup>16</sup> Si.cm<sup>−2</sup>. The thickness of the recrystallized alloy layer exceeds the critical thickness for commensurate growth for MBE grown layers [2]; TEM analysis confirms that the ion-beam-annealed sample is also incommensurate with the Si substrate.
Figure 2. RBS-C spectra of a thick amorphous layer of Ge$_{0.5}$Si$_{0.5}$, before and after ion-beam-annealing. (—) initial amorphous layer, and (.....) after irradiation with 1.2x10$^{16}$ Si·cm$^{-2}$.

CONCLUSIONS

It has been demonstrated that ion-beam-annealing can be used to promote epitaxial crystallization of amorphous Ge$_x$Si$_{1-x}$ alloy layers from both a crystalline alloy of the same composition and from a crystalline Si substrate. Furthermore, during ion-beam-annealing the commensurate-incommensurate transformation has been shown to occur at a critical thickness consistent with that determined for MBE grown layers.

ACKNOWLEDGEMENTS

The Commonwealth Special Research Centres Scheme, the Australian Research Council and the Victorian Government are acknowledged for their financial support. Mr.R.Loccisano and Mrs.S.Lane are acknowledged for their technical assistance. Dr.A.Pogany for his assistance with the interpretation of the TEM data. We also wish to thank Dr.J.Bean of AT&T Bell Laboratories for providing the MBE grown material.

REFERENCES


A new treatment of the momentum distribution of swift ions in inhomogeneous media has been developed and applied to the scattering of swift ions from solid surfaces. The method involves a full quantum mechanical treatment of the ion motion using a Hamiltonian which includes contributions from the unperturbed ion, the unperturbed electrons in the solid and the perturbation which arises from the interaction of the ion and the surface. The ion is described by a wave packet so as to retain a semi-classical picture of the ion motion.

This treatment of ion motion in the homogeneous case leads to the usual stopping power formulation [1]

\[ - \frac{dE}{dz} = - \frac{(Q e)^2}{\pi^2 V} \int_0^\infty d\omega \int d^3k \frac{1}{k^2} \text{Im} \left( \frac{1}{e(k, \omega)} \right) \omega \delta(\omega - k \cdot V) . \]

In the case of ion scattering from a surface, the solution is complicated by a lack of knowledge of the ion-surface interaction potential, particularly the contribution of the image potential.

Development of the theory indicates that the inhomogeneity of the surface introduces an uncertainty into the surface normal component of ion momentum and a coupling between the surface-normal and surface parallel components of the momentum distribution. This coupling will have significant influence on the scattering of ions from surfaces, particularly in low angle grazing circumstances, such as those in which a skipping motion was recently reported [2].


NUCLEAR MICROSCOPY OF TECHNOLOGICAL MATERIALS AT OXFORD

by

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Abstract

The first dedicated nuclear microprobe analytical facility has recently commenced operation in Oxford. The facility includes a specialized microprobe, designed by drawing on the pioneering work of the Melbourne microprobe group, for analysis of single crystal materials of technological importance. A scanned, focused, high energy proton or alpha microprobe (2-3 MeV) allows the techniques of Proton Induced X-Ray Emission (PIXE), Nuclear Elastic Backscattering Spectrometry (BS), Channeling Contrast Microscopy (CCM) and Secondary Electron Microscopy to simultaneously image the structural and elemental composition of materials. Quantitative information about the materials may be obtained from the images. The new microprobe has been applied to study Fe substituted single crystal high temperature superconductors, \( Y_1Ba_2(Cu_{1-x}Fe_x)_3O_{7-\delta} \), where the crystals were from 50 \( \mu m \) to 500 \( \mu m \) across. The new microprobe has also been used to measure the crystal quality of heteroepitaxial GaAs films grown in 50 \( \mu m \) and 25 \( \mu m \) square wells on <111> Si substrates.

Introduction

During the past year, a specialized nuclear microprobe has been constructed within the Oxford Nuclear Microprobe Analytical Unit that is dedicated to analysis of single crystal samples. The new Oxford nuclear microprobe has the capability of producing simultaneous PIXE [1], (R)BS/CCM [2-5] and secondary electron maps of samples to a lateral resolution of sub-micron. These techniques are known collectively as ‘Nuclear Microscopy’. The new microprobe incorporates several unique facilities [6] including a versatile data acquisition system and large area custom built x-ray and charged particle detectors. Also, the probe forming lens system consists of a new type of magnetic quadrupole lens triplet that is practically free from parasitic aberration that would otherwise degrade the resolution of the probe. The sample is mounted on a precision eucentric goniometer and a channeltron electron detector provides simultaneous imaging of the sample surface features from the secondary electron emission.

The present work makes use of a 2 or 3 MeV \( H^+ \) analysis beam focused to a diameter of 1 \( \mu m \). At this energy the proton nuclear elastic backscattering (BS) cross section for \( O^{16}(p,p)O^{16} \) is generally an order of magnitude greater than the Rutherford cross section, which gives much greater sensitivity for detection of O compared to RBS analysis with traditional \( \sim 2 \) MeV He\(^+\) beams. The BS spectra in the present work have been analysed using empirical cross sections, details of which are given in reference [6].

Poly - crystalline High Temperature Superconductors

Measurements of the crystal quality of \( Y_1Ba_2Cu_3O_{7-\delta} \) crystals, of a size 1–3 mm along the a- and b-axes, with conventional ion channeling techniques, by use of unfocused beams, have been reported in the literature [7,8]. However measurement of the crystal quality of typical crystals, which are usually less than 1 mm in size, with unfocused beams is extremely difficult. Previous workers [9] have used 5 \( \mu m \) D, II and He focused probes to measure the composition uniformity of superconducting \( YBa_2Cu_3O_{6.5+\delta} \) pellets over 150x150 \( \mu m^2 \) regions. The present work shows CCM analysis of 50 \( \mu m^2 \) subregions of sub-millimetre sized crystals. The crystals were of composition

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Y₁Ba₂(Cu₁₋ₓFeₓ)₃O₇₋₅, grown in alumina crucibles by the method of spontaneous nucleation from a nonstoichiometric flux bath [10]. The crystals typically display both mirror smooth regions and spiral-like growth regions [11]. A fit to the BS spectrum (see figure 2) obtained from a randomly aligned x = 0 crystal gave a composition of Y:Ba:Cu:O = 1:1.9:3.1:8.5, in good agreement with a previous result obtained from electron probe analysis, Y:Ba:Cu = 1:2:1:3.

CCM images of a typical x = 0 crystal, with the c-axis of a central mirror smooth region aligned with the analysis beam, are shown in figure 1. By extracting BS spectra, shown in figure 2, from the stored data array, the mirror smooth regions (region A in fig.2) were found to have a proton channeling $X_{\text{min}}$ of 20.8% and therefore consist of good quality crystal. Part of the the spiral-like growth region (region B) had $X_{\text{min}}$ of 53% and part (region C) had a yield equal to that of the randomly aligned crystal. The spiral-like growth region was therefore assumed to consist of polycrystalline material, some of which was near the alignment of the smooth regions.

By taking the ratio of the x-ray yield from aligned and randomly oriented x=0.05 and x=0.075 samples. The Fe $X_{\text{min}}$ is above the expected value except when measured along the <111> axis. (Above) A 50x50 µm² secondary electron image of the x=0.05 sample.
Aligned  Random

Sec. Elect.  0.0-0.8 \( \mu m \) GaAs (BS)

100 \( \mu m \)

Figure 4: Simultaneous BS and secondary electron CCM images of GaAs epilayers grown in wells on a patterned \(<111>\)Si substrate. The BS maps are labeled with the approximate depth which corresponds to the energy window used to produce the maps.

Figure 5: (Left) BS spectra from subregions of the maps in figure 4. The bar labeled \( A \) delineates the energy window used to produce the maps in figure 4. The simulation is for a 2.7 \( \mu m \) GaAs layer on a Si substrate, which was the nominal sample structure. Beam energy was 2 MeV.

x=0.075 crystals, it was possible to calculate a x-ray \( \chi_{\text{min}} \) for each element in the samples. These \( \chi_{\text{min}} \), shown in figure 3, reveal that the Fe is not completely substitutional when observed down the \(<100>\) axis, but is closer to being substitutional when observed down the \(<111>\) axis. Further work is in progress to attempt to understand the reasons for these results.

Heteroepitaxial GaAs on Si

Semiconductor devices that take advantage of the best properties of Si and GaAs by combining Si and GaAs devices on a single chip require successful integrated growth of these two materials. One major problem with obtaining high quality epitaxial GaAs layers on single crystal Si substrates is the 4% lattice mismatch. However, a MBE GaAs growth process [12] has been developed that produces good quality GaAs films grown in small wells cut through a surface oxide layer on a \(<111>\)Si substrate. Conventional broad beam ion channeling analysis of the crystal quality of the GaAs film is not possible owing to the small scale of the wells which are from 20 to 50 \( \mu m^2 \) square.

CCM images, shown in figure 4, for a sample with the \(<111>\) axis aligned with the analysis beam, clearly show the contrast caused by the significantly lower yield from the epi-GaAs in the...
wells compared to the poly-GaAs on the oxide layer. The contrast vanishes when the sample is randomly oriented to the analysis beam. From BS spectra extracted from the stored data array, the epi-GaAs in the wells was found to have a $\chi_{\text{min}}$ of approximately 22% for proton channeling and is therefore of reasonable crystal quality.

The crystal quality as a function of position across a period of the sample, shown in figure 6, revealed that the GaAs crystal quality was uniform across a well to the limit of the spatial resolution. The $\chi_{\text{min}}$ was 100% in the poly-GaAs over the oxide region surrounding the smaller wells, however was only about 75% in the poly-GaAs over the oxide surrounding the larger wells. This suggests that there is some degree of preferred orientation of the poly-GaAs in these regions, however further work with complementary techniques is required to determine the reason for this result.

Conclusion

Nuclear Microscopy has been used to image single crystal samples using contrast from PIXE, BS, CCM and secondary electrons with a scanned 2 or 3 MeV proton microbeam. The techniques, when combined with a sophisticated computerised data acquisition system, have provided quantitative ion channeling measurements of the crystal quality of regions as small as a few tens of microns in size. Further work on these materials is presently being done with the Melbourne microprobe.

Acknowledgements

We acknowledge C. Chen* of the Clarendon Laboratory, University of Oxford, for growing the YBa$_2$(Cu$_{1-x}$Fe$_x$)$_3$O$_{7-x}$ crystals and the assistance of Dr Linda T. Romano of the Metallurgy Department, University of Oxford, with the analysis of the high $T_c$ material and interpretation of the results. We acknowledge Dr Yung C. Kao of the Texas Instruments Research Laboratories, Dallas, Texas, U.S.A., for providing the GaAs/Si heterostructures. None of this work would have been possible without the assistance of the Oxford Microprobe group, in particular Drs Geoff W. Grime and Frank Watt.

References


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DIFFRACTION ANALYSES OF ZIRCONIA - TOUGHENED CERAMICS

by

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Abstract

The zirconia in zirconia-toughened ceramics can exist in cubic, tetragonal, orthorhombic, monoclinic, and other related forms\(^1,2\). It is important to establish the phase composition of the ceramics, not least because the toughening mechanism depends on having zirconia in the metastable tetragonal form\(^3\). Neutron powder diffraction appears to be the ideal technique for the required analyses, and its application will be described here.

The validity of the neutron diffraction technique has been demonstrated in test measurements, first on weighed mixtures of rutile (TiO\(_2\)) and corundum (Al\(_2\)O\(_3\))\(^4\), then on weighed mixtures of the cubic, tetragonal and monoclinic polymorphs of zirconia\(^5\).

The application of neutron powder diffraction to determine the form of zirconia in zirconia-toughened alumina (ZTA) provides a simple illustration of the power of the technique, and the reasons neutrons are preferred over X-rays.

The application to partially stabilised zirconia (PSZ) will also be described. This is a far more complex system, in that as many as five distinct phases need to be considered in the analysis. Despite the complexity, encouraging results have been obtained\(^6,9\). The aim in this talk will be to relate the phase composition, as determined by neutron diffraction, to physical properties as diverse as sample volume to fracture toughness.

References

\[9\] R.H.J. Hannink, C.J. Howard, E.H. Kisi and M.V. Swain, \textit{ibid.}
SIMULTANEOUS PROFILING of $^{14}\text{N}$ and $^{15}\text{N}$

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Introduction

The conventional method of profiling nitrogen concentration by NRA is to use the reaction $^{14}\text{N}(d,\alpha)^{12}\text{C}$. The cross section is only about 1 mb sr$^{-1}$, but since $^{14}\text{N}$ is 99.64% abundant, adequate yields can be obtained at a deuteron energy of 1.2 MeV when the nitrogen concentration exceeds $5 \times 10^{14}$ ions cm$^{-2}$. The Q value is 13.57 MeV and two $\alpha$-particle groups are given off. The ground state $\alpha$-particle has energy 9.9 MeV and that corresponding to the first excited state has energy 6.7 MeV. This latter $\alpha$-particle group is about seven times more intense than the former and is the one usually used for nitrogen profiling. It is well separated in energy from other particles due to $(d,p)$ and $(d,\alpha)$ reactions and much higher in energy than the backscattered deuterons whose yield is prolific. Even though count rates are low for atomic concentrations below 1%, there is an almost complete absence of background counts in the 6-7 MeV region. A problem with this profiling technique is in the radiation background from deuteron induced neutron producing reactions. This radiation level is usually above that which would be acceptable for typical ion beam analysis facilities without substantial shielding.

Nitrogen $^{15}$ is only 0.36% abundant in nature, but is more easily profiled using the reaction $^{15}\text{N}(p,\alpha)^{12}\text{C}$. This has a Q-value of 4.26 MeV and a cross section of order 30 mb sr$^{-1}$ at 1 MeV. The ground state $\alpha$-particle has energy 4.2 MeV. This is well above the energy of particles from competing reactions or RBS and hence is easily observed. The high cross section means that concentrations as low as $2 \times 10^{14}$ atoms cm$^{-2}$ can be profiled. However the low abundance means that it is only practical to use this reaction when $^{15}\text{N}$ is specifically introduced into the sample.

There are circumstances under which it is desirable to use both $^{14}\text{N}$ and $^{15}\text{N}$ in a sample under different conditions. Examples of this include nitrogen leaching from the surface or sub-surface, diffusion under different conditions of temperature and pressure and uptake and distribution of nitrogen based fertilisers as a function of time.

Profiling both nitrogen isotopes using the above reactions involves two measurements and inherent errors of normalisation with regard to beam current, solid angle and depth resolution. However by use of the reaction $^{15}\text{N}(d,\alpha)^{13}\text{C}$ both isotopes can be profiled simultaneously and the.
normalisation errors are eliminated. The reaction Q-value is 7.69 MeV and the \( \alpha \)-particle corresponding to ground state transitions has energy 5.8 MeV for a 1.2 MeV deuteron energy.

**Experiment**

As part of a program investigating the diffusion and retention rate of nitrogen in ion implanted steel, a bright mild steel sample was implanted with 25 keV \(^{15}\text{N}\) ions at a dose of \(1.2 \times 10^{17}\) ions cm\(^{-2}\). Dry \(^{14}\text{N}\) gas was bled into the implant chamber to give a dynamic pressure during implantation of \(5 \times 10^{-4}\) torr.

The sample was then transferred to a scattering chamber with surface barrier detectors at 108\(^\circ\) and 161\(^\circ\) to the incident beam. Profiling was first carried out with a 1.0 MeV proton beam by measuring the \( \alpha \)-particle spectrum from the reaction \(^{15}\text{N}(p,\alpha)^{12}\text{C}\) and then using exactly the same experimental arrangement, the beam was changed to 1.2 MeV deuterons and the \( \alpha \)-particle spectra from the two reactions \(^{14}\text{N}(d,\alpha)^{12}\text{C}\) and \(^{15}\text{N}(d,\alpha)^{17}\text{C}\) were measured simultaneously.

The cross section for the \(^{15}\text{N}(d,\alpha)^{15}\text{N}\) reaction has been measured previously by Davies et al\(^1\) using a frozen gas technique. Our measurement of the \(^{15}\text{N}\) content by the two reactions using the same geometry provides an indirect measurement of the cross section which is consistent with their data.

**Discussion**

Figure 1 shows the high energy end of the 108\(^\circ\) spectrum with the \( \alpha_1 \) peak due to \(^{14}\text{N}\) and the \( \alpha_2 \) peak due to the \(^{15}\text{N}\).

![Profiling of Nitrogen-14 and -15 with deuterium](image)

*Figure 1*
In this situation, the $^{15}\text{N}$ is from implanted ions and the $^{14}\text{N}$ is from the surrounding partial atmosphere. The depth scale corresponds to 10 nm per channel. The expected depth for 25 keV nitrogen ions is less than 30 nm, so diffusion has taken place for both $^{14}\text{N}$ and $^{15}\text{N}$. The measured retained $^{15}\text{N}$ dose is consistent with the implanted dose and it therefore follows that it is possible to obtain a nitrogen content in the steel in excess of the nominal implanted dose.

In situations where $^{15}\text{N}$ is the only isotope used, profiling would be done by the $^{15}\text{N}(p,\alpha)^{12}\text{C}$ reaction because of its high cross section and low background radiation. However, in circumstances where both isotopes are present in significant quantities the two can be profiled simultaneously and if the two have been introduced under different physical conditions or at different times, then the contributions of each can be clearly identified.

This measurement has clearly shown how to differentiate between implanted and absorbed nitrogen ions. The advantages of profiling two isotopes with one ion beam and elimination of normalisation errors are obvious.

Reference

ION BEAM MODIFICATION OF THE SURFACE WEAR CHARACTERISTICS OF DIAMOND.

G.C. Anderson and S. Prawer,


Abstract

Recent reports\(^1,2,3\) have suggested that ion implantation of diamond may significantly enhance its wear resistance. In an attempt to reproduce and extend these results natural diamond held at high temperature (800°C) has been subjected to implantation with various ion species. Scratch and indentation tests showed that the surface of diamond can indeed be toughened by ion beam irradiation.

Abrasive wear tests were used to assess the modifications to the surface wear characteristics of the diamond. During wear tests the diamond was removed at specific intervals for interferometric analysis. As interferometry is very sensitive to variations in surface topology, any variation in wear rate between implanted and unimplanted diamond is easily determined.

Backscattering spectroscopy has been used to analyse the microstructural effect of ion implantation in an attempt to determine whether a relationship exists between ion beam induced damage and enhanced abrasion resistance.

RHEED and Raman Spectroscopy have shown that even after high dose implantation the diamond is still single crystalline and has not undergone polycrystallisation, graphitisation or amorphisation due to the ion irradiation.

References


TOWARDS PRACTICAL USES OF ION BOMBARDMENT IN CERAMICS

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Abstract

Recent work at RMIT and in laboratories elsewhere, is reviewed to show that ion bombardment techniques of surface modification of ceramics are getting close to practical/industrial uses. Some properties altered are hardness, compressive stress, fracture toughness, flexural strength, friction, wear, adhesion of metallic films onto ceramics and sinterability of powders. A promising area of application — solid lubricant films is discussed in detail.

1. INTRODUCTION

Surface modification of ceramics by ion bombardment techniques is being used to create improved and novel mechanical [1—3], optical, electrical [4] and chemical properties [5]. This paper reviews recent progress made towards achieving some practical uses of ion bombardment in ceramics with emphasis on the control of surface mechanical properties.

2. PHYSICAL PROCESSES INVOLVED

The mechanical properties of ceramics are crucially determined/limited by surface conditions such as flaws, defects and microstructure. Ion beam induced alterations in surface properties are brought about by one or more of the following factors — microstructure of implanted layer, residual (compressive) stress, lattice damage and defect—impurity interactions. Implantation parameters such as energy, ion dose, ion species and substrate temperature can be varied to produce surface microstructurcs which can be crystalline or amorphous. Heat treatment can be applied after implantation to produce further changes in the microstructure and the phases present in the implanted layer, in order to optimise its beneficial properties [6].

3. EXAMPLES OF MODIFICATION OF PRACTICAL PROPERTIES

3.1 Microhardness, compressive stress and fracture toughness all undergo changes correlated clearly with the presence of three regions as shown in Fig.1 from the work of Burnett and Page [2]. As the 300 keV, Ti ion dose is increased all these properties peak at similar doses corresponding to the dose at which amorphization first occurs. 'Region I' behaviour corresponds to the presence of a radiation—damaged, but still crystalline implanted layer—hardening is attributed principally to radiation hardening effects. 'Region II' behaviour corresponds to the initial formation and subsequent growth of a subsurface amorphous layer, which is much softer than the crystalline host material. 'Region III' (absolute softening) corresponds to doses which are high enough to establish a true surface layer of amorphous (softer) phase. The ion beam induced compressive stress is relieved at the onset of the amorphous phase transformation. Fig.2 shows the implantation temperature dependence of relative hardness with 300 keV Ni ion dose implanted in sapphire [1]. The critical ion dose for the observed softening has been shown to be correlated with that for amorphisation. This critical dose at 300K is about two orders of magnitude higher than that at 100K due to dynamic annealing of ion induced defects. The existence of a softer amorphous phase produced by ion bombardment has been shown [1—3] to play a significant role in almost all mechanical properties of several oxide and non—oxide ceramics.
3.2 Flexural Strength (Bend Testing) Because of its brittleness, the strength of a ceramic component is easily reduced by mechanical contact events and a toughening of ceramics surface is highly desirable. Hioki et al [1] have shown that ion implantation is very effective in improving the resistivity to the contact–induced degradation of ceramics. The degradations of flexural strength associated with Vickers indentation were studied and the results are given in Fig.3, for sapphire plates (1mm thick, 7mm wide, 25 mm long) unimplanted and implanted with 400 keV Mn ions to a dose of $5 \times 10^{17}$ ions/cm$^2$ at 100 K. For the unimplanted sapphire, Vickers indentation with a load as low as 0.49 N reduces the strength to about one half of the unindented value. On the other hand, for the implanted sapphire, almost no degradation is observed by the indentation with the same load, indicating the complete suppression of radial crack initiation.

3.3 Friction and Wear of a ceramics surface moving in contact with a metal is reduced remarkably after ion implantation. The data of Hioki et al [1] is shown in Fig.4 as the friction coefficient $\mu$ as a function of sliding for SiC unimplanted and implanted with Ar ions. The data were obtained by the pin-on–disk test. Testing conditions included no lubricant, room temperature, sliding velocity of 0.05 m/s and an ambient atmosphere of relative humidity of 50 to 70%. As seen in Fig.4 the implantation of Ar ions to the SiC disk results in a remarkable reduction in $\mu$. It is suggested that the observed reduction in $\mu$ is related to the formation of an amorphous layer by ion implantation, and that the low friction state maintains until the surface amorphous layer is worn away.

3.4 Metal Film – Ceramic adhesion undergoes a remarkable increase as shown by the early work of Sood et al [7]. Kohzaki et al [8] have demonstrated that the improved adhesion obtained by Ar ion bombardment of a Nb film deposited on a SiC substrate, can reduce the relative amount of wear (measured by a pin & disc machine) to almost a zero level even at rather high loads (Fig.5).

3.5 Solid Lubricant Films by Ion Beam Assisted Deposition (IBAD) of silicone oil have been shown by Hioki et al [9] to be a very cheap and viable alternative to direct ion implantation. Their process is shown schematically in Fig.6. The base pressure of the system was 1µ Torr. The ceramic disc samples were mounted on a sample holder which was cooled with liquid nitrogen. A reservoir for the silicone oil was heated in the chamber, and a shutter in front of the oil reservoir was opened when the reservoir temperature reached a given value ranging from 60 to 120°C. Simultaneously, Ar ions at an acceleration energy of 1.5 MeV irradiated the samples to a fixed dose of $5 \times 10^{16}$ ions/cm$^2$. During the ion irradiation, the reservoir temperature was kept constant. The coated film thickness was varied mostly by changing the oil reservoir temperature.

Friction measurements (obtained by using a pin and disc apparatus) on such a film of about 0.27µm thickness deposited on silicon nitride disk are shown in Fig.7. For the uncoated disk, the coefficient of friction, $\mu$ is 0.85 and the fluctuation of $\mu$ is quite large. On the other hand for the coated disc, $\mu$ as low as 0.01 is obtained and the fluctuation of $\mu$ is also very small. This film is also very durable in that the sliding state with $\mu$ as low as 0.04 to 0.1 was seen to last up to about 0.1 million cycles. This indicates an exceptionally adherent film.

3.6 Enhanced Sinterability could be achieved in practice by using ion implantation to increase the atomic diffusivity by upto seven orders of magnitude in the ion beam induced ceramic phase. Sood et al have shown [6] such an enhancement in Fig.8 which shows RBSC spectra on a c-axis $\text{Al}_2\text{O}_3$ sample implanted with 100keV Zn ions at about 77°K to a dose of $6 \times 10^{16}$ Zn/cm$^2$; and subsequently annealed at 800°C for 3h in Ar. The as–implanted amorphous layer is about 80nm thick. After annealing, the layer remains amorphous as confirmed by
the RHEED pattern. Zn has undergone rapid diffusion within the amorphous layer and has attained a nearly flat top distribution. Note the arrows marking a–c interface and Zn surface within which Zn is confined. There is no detectable loss of Zn. From the observed increase in FWHM of Zn, we estimate an effective diffusion coefficient of $1.6 \times 10^{-15}$ cm$^2$/s at 800°C. This may be compared with a value $\sim 10^{-22}$ cm$^2$/s obtained by extrapolation to 800°C of self (Al ion) diffusion coefficients measured, previously in crystalline Al$_2$O$_3$.

REFERENCES


Fig.1  For Ti$^+$ implanted into sapphire, the curves show the variations with dose of (●) 25 g Knoop microhardness, (▲) integrated stress (cantilever bending method), and (▼) radial crack trace length (expressed as the ratio of the unimplanted crack size $C_u$ to the implanted crack size $C_i$) for 200 g Vickers indentations. Note that all three of these parameters peak at similar doses, $\sim 9 \times 10^6$ Ti$^+$ cm$^{-2}$, corresponding to the dose at which amorphization first occurs. The scatter shown in the microhardness results is ±σ (σ is the standard deviation).

Fig.2  Relative Knoop hardness as a function of ion dose for sapphire implanted with 300 keV Ni$^+$ ions at 100 or 300 K.
Fig. 3. Strength degradations by Vickers indentation for sapphire plates unimplanted and implanted with 400 keV Mn⁺ ions to $5 \times 10^{17}$ ions/cm² at 100 K.

Fig. 4. $\mu$ as a function of sliding cycle for SiC disk in sliding contact with SUJ2 steel under a normal load of 9.2 N. (a) Disk: unimplanted. (b) Disk: implanted with 800 keV Ar⁺ ions to $1 \times 10^{16}$ ions/cm² at 300 K.

Fig. 5. Relative amount of wear after 40 h test for Sn, Nb/SnC, and Ar⁺ ion-irradiated Nb/SiC as a function of the normal load.

Fig. 6. The system for silicon oil vapor deposition and Ar⁺ ion irradiation.

Fig. 7. $\mu$ vs. sliding time for Si$_3$N$_4$ disks uncoated and coated with a 0.27 µm-thick carbonaceous film prepared by silicone oil vapor deposition and 1.5 MeV Ar⁺ ion-irradiation. The pin material is SUJ2.

Fig. 8. RBSC spectra from a c-axis Al$_2$O$_3$ sample implanted with Zn ions, before and after anneal at 800°C for 3 h.
PITFALLS IN THE USE OF ION BEAMS
FOR QUANTITATIVE MATERIALS ANALYSIS

by

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Abstract

The Catania Ion Beam Handbook Workshop in 1974 and Baglin's subsequent RBS round-robin at the Karlsruhe IBA Conference in 1975 showed clearly that our oft-claimed accuracy of a few % for RBS was rarely achieved. Even among the most experienced practitioners of ion beam analysis, discrepancies in excess of 20% were found. Since then, the major factors responsible for these discrepancies have been identified and gradually eliminated; with care, an absolute accuracy of 1-2% is now achievable.

In this presentation, we will discuss some of the pitfalls to be avoided in quantitative ion beam analysis: problems in current integration, charge exchange along the beam line, deviation from Rutherford scattering, single/plural scattering effects, energy corrections in using solid state detectors, and finding suitable reference standards. We will also consider briefly how these effects depend on the energy and atomic number of the analysing beam.
ION BEAM ANALYSIS OF THIN FILM PHOTOVOLTAICS

by

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Abstract

More widespread use of solar photovoltaics is dependent upon perfecting methods of producing inexpensive thin film devices. Our research program has concentrated upon electrodeposition of II–VI semiconductors, notably CdTe, and has resulted in thin film solar cells with sunlight conversion efficiencies (13%) at least as good as single crystal CdTe [1], even though the starting materials are no purer than analytical grade reagents (99% purity) [2].

Because these II–VI semiconductors are direct band gap absorbers, their solar conversion and optoelectronic properties are achievable with micron thick material and with interfaces of atomic dimensions formed on it. Modern surface analytical techniques have provided an essential part of the armoury needed to provide a precise knowledge of the composition of the materials, the distribution of major and minor constituents within the material matrix and the effects of chemical and compositional changes which occur during processing [3]. However, the use of energetic ion beams on these materials can produce effects such as preferential removal of one or more species, implantation, knock–on phenomena, surface roughening and redeposition of sputtered elemental species each of which can confuse data interpretation. These problems will be addressed in this talk. Two examples are now discussed.
For composition analysis of compounds, depth profiling was used to remove outer surface contamination and to expose successive layers of the compound for analysis by XPS, AES or SIMS. In such analyses, a precise knowledge of the materials sputter rate was needed so that depths were accurately known, the uncertainty in depth resolution or interface width was required and whether there was any preferential sputtering must be determined. Hence, ion beam etch rates of CdS and CdTe films have been quantified and the atomic concentration ratios of Cd : S or Cd : Te determined by XPS or AES illustrated the preferential sputtering which occurred upon ion etching. Further, the high energy AES electron beam was shown to produce sample changes under irradiation when there was residual water vapour in the system [4]. A continuous ion beam sputter was necessary to avoid a build-up of oxide/hydroxide layers.

Trace impurity analyses of these semiconductor films were needed and SIMS was used routinely to determine doping profiles and the presence of unwanted impurities. Thus comparative SIMS data showed how electrodeposited CdTe films can be prepared in purer form than crystals of CdTe purchased from commercial suppliers as 5N pure [5], how thin film CdTe after etching with Br₂/methanol retained Br in the grains and at the junction interface of the substrate [3] among other data. To ensure that the qualitative information was reliable when the data were obtained on different samples at different times demanded not only that the obvious precautions must be observed (constant primary ion type, energy, current density, secondary ion lens settings, sample environment and geometry) but also that less obvious precautions must be observed, e.g. minimization of cross-contamination (memory) effects, minimization of fringe neutral effects, minimization of crater edge effects, etc. Recently, we have been able to learn the extent of the fringe neutral effects which provide a low level undesired signal from an area concentric with the analysis
area and which are caused by energetic, unfocussed, high energy neutral atoms. The extent of these neutrals is dependent on the ion gun type and these data were obtained using a PHI Model 04-303 ion gun incorporating an in-line electron impact source. Molybdenum cover plates were used with circular apertures of various sizes to measure the $^{98}$Mo$^+$ secondary ion signal originating from impact of the fringe neutrals when the primary ion beam passed through the centre of the aperture. A similar Mo plate was placed over the aperture and the total secondary ion signal from the ion beam plus its fringe neutrals was also recorded. The ratio of the secondary ion signals from primary ions and from neutrals striking an area greater than 3 mm away from the beam centre was 1.6%. The fringe neutrals distribution as a function of distance from the beam centre was determined. To reduce this fringe neutral contribution, it was necessary to bypass any front sample mounting hardware such as cover plates, screws, etc., and to etch a large crater c. 9 mm x 9 mm during initial profiling into the bulk and then reduce the analysis area to the inner 3 mm x 3 mm, when collecting data representative of the films bulk composition. The secondary ion signal due to the fringe neutrals would thus originate from an area at a similar depth and similar composition as that of the inner primary ion beam.

References


INTRODUCTION

Although ion beam irradiation of semiconductors has been studied for many years, a comprehensive picture of the damaging process is still emerging. Early models of ion damage in Si were concerned with finding the parameters affecting the production of an amorphous surface layer rather than describing the accumulation of defects within an essentially crystalline matrix.

The limitations of these models become apparent when one considers their lack of any sophisticated treatment of time-dependent effects: i.e. competition between defect production (ion dose rate) and annealing (temperature). In addition, the models do not say anything about what kind of defects might exist in damaged (but not amorphous) material.

Recent work on ion irradiation of amorphous surface layers over crystalline substrates in Si has revealed some intriguing phenomena. By balancing the ion dose rate and temperature, the amorphous layer may recrystallise (low dose rate/high temperature), or extend into the underlying crystal below (high dose rate/low temperature) [1]. Surprisingly, this behaviour appears to be successfully modelled by describing the crystallisation regime as simple annealing by beam-generated point defects (with the production rate scaling with Monte-Carlo calculations of linear displacement density) [2], and the amorphisation regime resulting from accumulation of divacancies at the amorphous-crystalline interface [3].

A convenient simplification for these theories results from the existence of a perfect sink for defects: the interface itself. The models can therefore deal with material which is either amorphous, or crystalline with some concentration of defects. Indeed, TEM examination after the irradiations does not show any evidence for defects in the crystal near the interface [4].

These studies, therefore, deal with a somewhat artificial situation, but by understanding the dynamic processes at work, a clearer picture of amorphisation in crystalline material may emerge. It is significant, then, that a recent study of keV Xe implantation into Si finds that the parameters affecting the suppression of amorphous layer formation in Si seems to be consistent with the data for suppression of ion-induced amorphisation of surface layers. This suggests that clustering of divacancies may provide the nucleation for producing amorphous material in crystalline Si.

Given the above possibilities for describing damage processes in Si, it is interesting to consider similar processes in GaAs. Curiously, there do not seem to be any investigations of ion beam damage in GaAs as a function of dose rate and temperature. Despite the added complexity of defects and damage in GaAs compared to Si [5], it may yet be possible to find similar relations.

RESULTS

Figure 1 shows the depth profile of displaced atoms, \( N_d \), as measured by RBS, from irradiation of (100) GaAs with 100keV Ar ions to a dose of \( 5 \times 10^{15} \) Ar/cm\(^2\) for temperatures from 25°C to 100°C. At room temperature, the concentration of displaced atoms in the first 700Å is coincident with that from a randomly-oriented crystal, suggesting that this surface layer contains amorphous material. As the temperature is increased, the level of damage decreases sharply, and at 75°C there is no deep damage observable by RBS, with the only apparent damage being at the surface.
Fig. 1 Normalised displaced atom profiles in GaAs after implantation of 100keV Ar ions to a dose of 5 x 10^{14} Ar/cm^2 at 25 (solid line), 50 (dashed line), 75 (dot-dash) and 100°C (solid).

Fig. 2 Total integrated number of displaced atoms from fig. 1 plotted against temperature. The data suggests a transition at a temperature of ~70°C at this dose and dose rate.

Figure 2 shows the total integrated disorder from figure 1 as a function of temperature. The data indicates that, at this dose and dose rate, a transition temperature exists (here ~70°C), above which an amorphous layer will not form. This suggests, by analogy with the model of damage in Si, that at this temperature the rates of defect production and annealing are closely balanced. Figure 3 shows the effect of dose rate at 75°C for a dose of 1 x 10^{15} Ar/cm^2. Reduction of the dose rate by a factor of six reduces the integrated disorder by an order of magnitude.

DEFECT CONSIDERATIONS

Figure 4 shows the damage build-up for 100keV Ne irradiation at 50°C. Note that, although the data suggests that the top ~1000Å are amorphous after a dose of 1 x 10^{16} Ne/cm^2, at half this dose there appears to be a thick (~700Å) layer of defects. TEM studies have shown that although amorphous layers can be produced at room temperature, elevated temperature implants of Ar can produce dense networks of dislocation lines and loops in GaAs [6]. However, at room temperature, an amorphous layer appears to form quickly at a dose of ~3 x 10^{14} Ne/cm^2, where changing the dose by a factor of 5 increases the number of displaced atoms by a factor of 50.

Clearly, then, any models attempting to predict the state of GaAs after ion implantation will need to accommodate complex defect clusters, at least in the case of light to medium mass ions.

Fig. 3 Damage profiles for 100keV Ar at 75°C at beam currents of 0.1 and 0.6 µA into 0.388 cm^2. At this temperature increasing the dose rate by a factor of six increases the integrated disorder by an order of magnitude.

Fig. 4 Damage profiles for 100keV Ne implantation into GaAs at 50°C. The data is for doses from 1 x 10^{14} to 1 x 10^{16} Ne/cm^2 increasing by a factor of two at each step.
MICROBEAM DAMAGE

It has been previously shown that the large dose and dose rates encountered in microbeam analysis can result in significant damage to Si and GaAs targets [7]. It is difficult to analyse such damaged material due to the extremely small lateral dimensions of the damaged areas. Here we use the aligned yield, or $X_{\text{min}}$, as an indication of damage rates in GaAs under microbeam irradiation with 2MeV He.

Figure 5 shows $X_{\text{min}}$ as a function of He dose for an initially undamaged sample. The irradiations were carried out at room temperature with an 8μm diameter beam spot. The beam was scanned in a square region with side dimensions of 15, 30, 60 and 100 μm. Clearly, dose rate effects are operating here, with the smaller scans producing significantly larger levels of damage. The difficulty of interpreting this data lies in the uncertainty of the scan pattern. Although the time-integrated dose is uniform over the scanned region, the time evolution is uncertain, and it is impossible to rule out high order Lissajous figures. Nevertheless, the data appears to be reproducible. Figure 6 compares the damage for a 15 x 15μm scan with that seen in Si under identical conditions. Clearly, the GaAs is more sensitive to radiation damage for the parameters in this example. In addition, the data shows that the damage in Si is negligible for doses up to ~50 mC/cm². This 'incubation' dose allows reliable data to be obtained from Si targets up to this dose, prior to the onset of large amounts of damage. GaAs shows no such tendency under any of the parameters studied thus far, with a linear increase of damage starting at arbitrarily small doses. What is suggested, however, is that the damage rates in GaAs may be minimised by either reducing the dose rate or, preferably, heating the sample during the analysis.

References

THE EFFECT OF SOME IMPURITIES ON THE ANEALING BEHAVIOUR OF ION-IMPLANTED SILICON
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Abstract

The annealing behaviour of ion-implanted <100> silicon containing various low-melting point, low-solubility impurities has been studied. The analysis techniques used include, Rutherford backscattering and channeling, transmission electron microscopy and time-resolved reflectivity measurements. Various annealing regimes have been identified corresponding to different implanted concentrations. For "low" concentrations, complete epitaxial regrowth and incorporation of the implanted impurity onto substitutional silicon lattice sites is observed. For concentrations that exceed the equilibrium solid solubility of the impurity in crystalline silicon, impurity segregation is seen at the amorphous-crystalline interface. For higher concentrations, an impurity induced amorphous to polycrystalline phase transformation is observed that is characterised by a thermal activation energy of 1.5-2.5 eV depending on the impurity.

The annealing behaviour of <111> silicon implanted with low dose impurities is also discussed. In this case, the presence of impurities appears to assist the crystallisation process.

Introduction

Ion-implantation is an important technique for the doping of silicon. It enables accurate control of the doping profile and has led to microelectronic devices with smaller feature size than was possible previously by conventional doping methods. Considerable damage is caused to the silicon lattice by the process of ion-implantation, and must be removed by annealing for the efficient operation of any electronic device. The annealing behaviour of silicon implanted with electrically active impurities is, therefore, an important research area.

In this study we have investigated the behaviour of a range of impurities (Ga, In, Bi, Sn, Pb, and Si) at annealing temperatures in the range 450-650°C for implant doses of 5x10¹⁴ - 2x10¹⁶ impurities.cm⁻².

Experimental

Silicon (<111> and <100> orientation) was implanted in a Whickham 200 keV ion-implanter with various doses of Ga, In, Bi, Pb, Sn and Si at energies of 100-150 keV. The samples were held at -196°C during implantation and inclined at an angle of ~80° to the normal to prevent ion-channeling. The implanted samples were first analysed using Rutherford backscattering and channeling (RBS) with 2 MeV He²⁺ to determine the exact implant dose and the peak concentration. The samples were then annealed in air on
a hot block at various temperatures (450-650°C). During annealing, the regrowth kinetics were monitored using time-resolved reflectivity (TRR) with a HeNe laser operating at 632.8 nm. The samples were then analysed again using RBS and selected samples thinned for plan or cross-section transmission electron microscopy (TEM).

**Results and discussion**

At low doses of Pb, Ga and In ($5 \times 10^{14} - 10^{15}$ ions.cm$^{-2}$), the amorphous layers produced by ion-implantation are seen to recrystallise to give near perfect crystal but the implanted impurities segregate to the surface, whereas similar doses of Sn and Bi do not segregate.

At higher concentrations (>1 atomic percent), recrystallisation of the amorphous layer is interrupted by a sudden phase transformation to fine grain polycrystalline silicon stimulated by the presence of the impurity. This phase transformation has been studied in some detail and a model has been proposed, in which the incubation time before transformation, $T_C$, can be characterised by an Arrhenius, relation of the form:

$$T_C = T_0 \exp \left( \frac{E_a}{kT} \right)$$

where $k$ is Boltzmann's constant and $T_0$ is a constant; $E_a$ is the thermal activation energy of impurity diffusion in amorphous silicon. The study of this phase transformation has been extended to cover Sn, Bi, Pb and Ga. It is found that all of these low-melting point, low-solubility impurities behave in a similar way to In and are characterised by activation energies of 1.5-2.6 eV (c.f. $E_a$ for In is 1.9 eV).

The intrinsic recrystallisation of $<111>$ Si does not result in perfect crystal; instead the regrown layer contains $<511>$ twins. The effect of low concentrations of impurities (<1 atomic percent) on the formation of these twins has been examined for the same implanted species as above. The TRR spectra show a more abrupt amorphous-crystalline interface than is seen with intrinsic regrowth and the RBS spectra show a lower yield in the regrown region for all impurities, indicating better quality epitaxial growth. The mechanism for the reduction in the number of twins is under investigation.

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References


ION–BEAM INDUCED EPITAXIAL CRYSTALLIZATION OF METAL SILICIDES

by

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Abstract

Epitaxial crystallization induced by high energy ion irradiation of amorphous NiSi2 and CoSi2 layers at 520°C is reported and compared with thermal annealing. Planar epitaxy was observed for both the ion–beam and thermal annealing regimes though levels of post–anneal disorder exceeded that of as–grown samples.

Introduction

Epitaxial metal silicides such as NiSi2 and CoSi2 have numerous applications in integrated–circuit devices. Though metal silicides are generally radiation resistant, implantation through or into such layers degrades the silicide crystalline quality and subsequent annealing is required to reduce implantation–induced disorder. Amorphous NiSi2 and CoSi2 layers on crystalline substrates recrystallize epitaxially at ~100°C [1,2]. Recently, epitaxial crystallization induced by Si ion irradiation of amorphous NiSi2 has been observed at 450°C [3]. Ion–beam induced epitaxial crystallization (IBIEC) [4] has been applied to the regrowth of amorphous Si, GaAs, and most recently the heterostructures NiSi2/Si [3] and GeSi/Si [5], at temperatures well below that required for significant thermally–induced solid phase epitaxial growth (SPEG). As an annealing method, this technique offers advantages of lower processing temperatures and stringent spatial control of the recrystallized volume. Furthermore, the application of IBIEC to heterostructures offers interesting technological possibilities when the crystallization/amorphization kinetics of the constituent materials differ. For the present report, ion–beam induced epitaxial crystallization of amorphous metal silicides was further investigated and compared with thermal annealing.

Experimental

Epitaxial NiSi2 and CoSi2 layers of thickness ~100 nm were formed on (111) Si substrates by metal deposition and thermal reaction [6]. Channeling minimum yield (\(\chi_{\text{min}}\)) values for as–grown samples measured directly beyond the metal surface peak were 0.11 and 0.06 for NiSi2 and CoSi2 layers, respectively. To create amorphous surface layers ~1/2 the thickness of the original layer, samples were implanted at ~−196°C with 40 keV \(^{28}\text{Si}\) ions to a dose of 2X10\(^{15}\) /cm\(^2\). To recrystallize such layers, samples were mounted on a hot stage and irradiated with 1.5 MeV \(^{20}\text{Ne}\) ions at a dose rate of 7X10\(^{12}\) ions/cm\(^2\)sec. Thermally–induced recrystallization during ion irradiation was monitored by mounting control samples in unirradiated positions on the hot stage.

The silicide thickness, stoichiometry and crystalline quality were measured with Rutherford backscattering spectrometry combined with channeling (RBS/C) using 2 MeV He ions and a scattering angle of 1100°. All channeling measurements were with respect to the <111> Si direction.
Discussion

Figure 1 shows RBS/C spectra, detailing only the Ni signal, of amorphous NiSi₂ layers irradiated at 52°C with 1.5 MeV Ne ions. Epitaxial recrystallization of the amorphous surface layer from the underlying crystalline layer is evident. Similar observations were apparent for CoSi₂. Growth rates at 52°C were 3.3 ± 0.7 and 1.6 ± 0.4 nm/10¹⁶ ions/cm² for NiSi₂ and CoSi₂, respectively and \( \chi_{\text{min}} \) values for recrystallized samples were ~0.3 and ~0.5, respectively. Compared to samples thermally annealed in air at ~100°C, \( \chi_{\text{min}} \) values were ~1.5 and ~2.5 times greater for ion-beam annealed NiSi₂ and CoSi₂ samples, respectively. Note the presence of Ne-irradiation induced disorder at depths greater than the original amorphous/crystalline interface. The level of such disorder was dose independent for the dose range used in the present study. As-grown samples irradiated with 1.5 MeV Ne ions exhibited a similar level of disorder throughout the silicide layer. Such observations suggest the Ne-irradiation induced disorder is not nucleated at the amorphous/crystalline interface. However, misfit dislocations at the Si/silicide interface may serve as initial nucleation sites. Similar observations have been reported for IBIEC of amorphous NiSi₂ layers at 45°C with 300 keV Si ions [3] wherein the rate of nuclear energy deposition at the Si/silicide interface was an order of magnitude greater than that in the present study and consequently the substrate was rendered amorphous immediately below the Si/silicide interface. At present, the nature of such disorder is under investigation with transmission electron microscopy.

![RBS/C spectra](image-url)

Figure 1 RBS/C spectra, detailing only the Ni signal, of NiSi₂ layers irradiated at 52°C with 1.5 MeV Ne ions to doses of 0 (\( \triangle \)), 2.5 (\( \bullet \)), 4.8 (\( \circ \)) and 9.7X10¹⁶ (\( \square \)). A random spectrum (\( \triangle \)) is also included.
Conclusions

Ion-beam induced epitaxial crystallization of metal silicides has been further investigated. Amorphous NiSi2 and CoSi2 layers were recrystallized at a temperature of 520°C with 1.5 MeV Ne ions though Ne-irradiation induced disorder was evident within the silicide layer. Further studies of the residual disorder and the temperature dependence of the IBiEC rate are now in progress.

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References


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VERY HIGH RESOLUTION POWDER DIFFRACTOMETRY

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ABSTRACT

Diffraction from polycrystalline materials, using either X—radiation or neutrons, has been used for many years for the determination of unit cell parameters and for phase identification. Following the invention of the Rietveld method [1] refinement of known crystal structure became possible yielding accurate values of structural and thermal parameters.

For the determination of the structure of crystals, that is, the positions of the atoms in the unit cell, single crystals were required. It was accepted that for some substances suitable crystals could not be found or grown.

The powder pattern contains, in principle, all the information available from a single crystal pattern (except for the separation of centric and non—centric space groups) however, the instrumental resolution has been insufficient to separate reflections from different sets of crystal planes. It is essential for the application of modern methods of structure solution that unambiguous intensity data is obtained for a substantial number of reflections.

The experimental situation has changed significantly with the construction and operation of spallation neutron sources, and synchrotron sources of X-radiation. It is now possible to obtain powder diffraction profiles whose widths of the order of one minute of arc [2]. This degree of resolution permits unambiguous identification of sufficient reflections in a system of moderate complexity for ab—initio structure solution to succeed.

These technique are of major use in studies of multi—phase systems in which the products are a consequence of the way in which the system is prepared, and cannot be obtained separately. Our present interest is in SYNROC, which contains major phases whose structures are known, and minor, but not insignificant phases, whose structures are unknown. To evaluate the role of these minor phases in the retention of high level nuclear waste it is essential that the arrangement of atoms in the unit cell of the crystals is known.

These new methods will be of great value in studies of engineering ceramics and mineral systems, both of which are important to Australia. It is quite unrealistic to assume that all the experimental facilities can be made available within this country. Materials Science, like nuclear physics, has become a "suitcase" science. It is essential that a mechanism is established for Australian scientists to use international facilities.

The current work on SYNROC is in progress at the DESY Synchrotron Laboratory, W. Germany; the ISIS spallation neutron source at the Rutherford—Appleton Laboratory, UK; and the LANSCE spallation neutron source at Los Alamos National Laboratory, USA.

We thank these laboratories for making instruments available to us, and thank ANSTO and DITAC (through the Australia/FRG Science and Technology Agreement) for funds necessary to make use of them.
References

[1] A profile refinement method for nuclear and magnetic structures


[3] Ab-initio structure determination by high resolution powder neutron
diffraction at the spallation neutron source, ISIS; the crystal structure
of ferric arsenate.

[4] The ab-initio structure determination of α-CrPO₄ by synchrotron
powder X-ray diffraction. J.P. Attfield, A.W. Sleight and A.K.
THERMAL NEUTRON SCATTERING AS A PROBE OF ATOMIC STRUCTURE -
A BRIEF OVERVIEW

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Thermal neutron scattering can be a very powerful non-destructive
technique for probing static and dynamic structure at the atomic level -
often in conjunction with other techniques, using x-rays, electrons etc.
A brief illustrated overview of the Australian use of neutron scattering
to study materials will be given.
NEW ION BEAM FACILITIES AT ANU

by

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and

Microelectronics & Materials Technology Centre, Royal Melbourne Institute of Technology

Abstract

Introduction

The Department of Electronic Materials Engineering within the Research School of Physical Sciences at ANU was established late in 1988 to carry out research into the near-surface preparation, modification, processing and characterisation of mainly semiconductor materials. Ion beam modification and analysis facilities feature strongly in the research programs of the new department. This paper outlines the new and upgraded ion beam facilities which will be available for such programs.

Riber SIMS Apparatus

A Riber SIMS system MIQ 256 was installed at ANU in February. This machine has the following broad features and specifications:

- base pressure $3 \times 10^{-11}$ torr
- oxygen and argon ion beam sources
- spot size 0.8μm
- scanning and elemental mapping features
- elemental depth profiling to 1ppm or better sensitivity.

The main applications are for elemental profiling in ion implanted materials, thin film structures and semiconductor multilayers such as III-V compound semiconductors.

MeV Ion Implanter

A purpose-designed MeV ion implanter (model 5SDH-4) has been purchased from NEC (Wisconsin) and will be installed in January 1990 at ANU. The broad specifications of the machine are as follows:

- Tandem accelerator with 1.7MV terminal potential
- 4 chains to allow 1.5mA column current
- SNICS Cs sputter source for negative ions of most elements
- 90° injection providing $\Delta M/M$ of > 240
- Uniform electrostatic beam scanning over 15 x 15cm²
- typical on-target beam currents of 300μA Si⁺⁺ at 3MeV or 100μA of Au⁺⁺ at 3MeV.
Typical applications of this machine will involve materials modification and buried compound formation in materials, deep doping of semiconductors, damage and amorphization studies in semiconductors, ion beam induced epitaxial crystallization and ion beam isolation of GaAs structures.

**Ion Beam Analysis**

The Van de Graaff accelerator at ANU is currently being recommissioned to undertake routine ion beam analysis to support the programs indicated above. A precision 2 axis goniometer has been built for channeling analysis. Initially, Rutherford backscattering and channeling and forward scattering/recoil analysis will be available using up to 2.1 MeV He$^+$ and H$^+$ ions.
MICROANALYTICAL PROBLEMS IN INDUSTRY AND THE SPMP

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The scanning proton microprobe, sometimes known as a nuclear microprobe or high energy ion microprobe, is a quantitative microanalytical tool of wide application to science and industry. This paper discusses its evolution as a scientific instrument and its role in microscopic analysis.

Sensitivity and Quantitative Accuracy
Any technique available for elemental analysis with a charged particle beam is in theory available to the scanning proton microprobe, which can equally employ focussed beams of other charged particles. In general the microprobe beam is cleaner (better defined with less stray radiation); so backgrounds can be lower. However sensitivity and quantitative accuracy may be limited by statistics and generally the sensitivity limits will be in the range 1 to 10 ppm. This sensitivity is still two to three orders of magnitude better than that of the electron microprobe (EMP). Other sensitive microprobes, such as the (sputter) ion microprobe and the laser microprobe are destructive and do not have the quantitative accuracy of the proton microprobe. They must be regarded as complementary instruments.

Spatial Resolution
The sensitivity and quantitative accuracy of nuclear techniques of analysis, referred to above, led Bird and associates at Lucas Heights (1) and Pierce and associates at Harwell (2) to collimate their accelerator beams and these collimated probes attained resolutions of about 100 micron. A major advance was made at Harwell by Cookson et al. (3), who focussed such a collimated beam down to 3.5 micron and built the first scanning proton microprobe (SPMP). Generally 100 pA has been recognised as a basic beam current for elemental microanalysis and, for such a current, a resolution of 0.5 micron has recently been reported at Oxford (4). Thus resolution has improved by a factor 7 over a period of nearly two decades.

Relatively simply calculations show that the major limitations on effective resolution lie not in the lens performance, but in the magnetic rigidity of the beam (unavoidable) and in the low brightness of available ion sources compared with electron sources. Brighter sources are under investigation (5,6,7) but, for the present, elemental analysis will continue to be restricted to resolutions close to 1 micron. The rewards to be gained with a brighter beam are seen when object and aperture diaphragms are stopped down. The resolution then depends ultimately on the mechanical and electromagnetic stability of the microprobe line and, with operation in STIM mode (see later), a resolution of 50 nm has been achieved at Melbourne (8).

Power and Versatility
The development of Total Quantitative Scanning Analysis (TQSA) on the SPMP enabled quantitative analysis of all elements in scanning mode with complete retention of data (9). As well as giving the SPMP a power unavailable to the EMP and other scanning instruments, this advance led to the development of several new techniques of quantitative analysis, specimen imaging and investigation of specimen-beam interactions.

Fig. 1 lists the basic interactions utilised in a SPMP, some of the available imaging methods, many of which give quantitative information, and some further developments, many of them recent.
Fig. 1 The principal interactions for elemental microanalysis in a SPMP, the principal imaging techniques and some major developments that greatly expand the role and power of the SPMP. SEI is secondary electron imaging, STIM is scanning transmission ion microscopy, CCM is channeling contrast microscopy and CSTIM is channeling scanning transmission ion microscopy.

Applications
A wide variety of industrial and scientific problems can be handled by the SPMP and Table 1 lists some of those tackled by the SPMP at MARC. A medical example(10) and a biological example(11) are given in these proceedings. The technique of channeling contrast microscopy(12) is a powerful one for examining the crystal structure of a specimen and examples of its use are also given(13). Scanning transmission ion microscopy(14,15) is a technique for mapping the areal density of a specimen with extremely high efficiency (the beam is run directly into a detector behind the specimen at 0 degrees). Hence extremely low beam currents are required and it is possible to improve the spatial resolution by an order of magnitude. The present limit is 50 nm (8), but another order of magnitude may be achievable. This development is exciting in its own right but also because of the further developments which spring from it. One of these is high resolution stereo imaging(16) which is made possible by the extremely high efficiency and hence good statistics of STIM. Another such development is STIM tomography(17). An example of this powerful new technique is also given in these proceedings(18). These images are all quantitative and furnish accurate 3 dimensional information on the density distribution within the solid unsectioned specimen. Very recently STIM and CCM have been combined to yield a very high efficiency high resolution technique for mapping defects with thin crystals (19).

The technique of automonitoring of elemental losses and matrix losses (20) has now been supplemented by STIM to yield high resolution information on the interaction between the beam and the specimen, also discussed in these proceedings (21). Finally techniques developed for high resolution STIM can also be applied to study effects of precisely localised single ionising events in semiconductors or biological cells, or precisely directed ions may be used in combination with etching techniques to carry out microscopic machining of selected materials, as demonstrated by Fischer et al. (22).

The SPMP is a powerful and versatile instrument for elemental microanalysis, but with new developments it is becoming also a high resolution microscope yielding much new information on the properties of materials and even offering possibilities for microscopic engineering.
TABLE 1

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<td>Materials Analysis</td>
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<td>in metal foils and in metal slag</td>
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<td>PIXE, RBS</td>
<td>Identification of surface damage to material</td>
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<td>and crop pollination and development</td>
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<td>Identify cells and tissues participating in</td>
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<td>RBS, RFS,</td>
<td>Identify mutant cells from trace metal</td>
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<td></td>
<td>PIXE</td>
<td>content. Measure drug uptake by single cells</td>
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We wish to acknowledge ARC support and the contributions from our many colleagues who were responsible for much of the development described.

References
(1) B.K. Mak et al. Nature 211 (1966) 738
(2) T.B. Pierce et al Nature 211 (1966) 66
(7) P.M. Read et al. Nucl. Instr. and Meth. B30 (1988) 293
(10) B.J. Kirby et al. these Proceedings (1989)
(11) G.S. Bench et al. these Proceedings (1989)
(13) D.N. Jamieson, these Proceedings (1989)
(14) J.C. Overley et al. Nucl. Instr. and Meth. 218 (1983) 43
(16) G.S. Bench et al. to be published
(18) G.S. Bench et al. these Proceedings
(19) M. Cholewa et al. to be published
(21) M. Cholewa et al. these Proceedings
(22) B.E. Fischer, Nucl. Instr. and Meth. B30 (1988) 284
STIM TOMOGRAPHY

Micro Analytical Research Centre, School of Physics, University of Melbourne, Parkville, Victoria, 3052, Australia.

ABSTRACT

Scanning Transmission Ion Microscopy (STIM) when used in conjunction with computed tomography is a powerful technique for imaging internal structure within a specimen. STIM has been used to produce a tomographic reconstruction of a double barreled pipette. Spatial resolution was shown to be 0.37 μm, limited by the pixel size rather than the beam spot size which was 0.2 μm.

INTRODUCTION

When a scanning MeV Ion-Microprobe is used to produce and record PIXE [1] or STIM [2] images, a map of the the 2-dimensional distribution of elements or areal density is produced. However, one would frequently like to map the 3-dimensional shape of objects to provide detail on internal structures without having the need to undergo detailed target preparation.

Additionally one would like to know the depth distribution to obtain accurate PIXE concentrations. To obtain elemental areal concentration of a sample with PIXE microanalysis, used in conjunction with STIM [3] or Rutherford back scattering, the sample is assumed to have uniform density and composition through any given point in the incident beam direction and emitted x-ray path.

While for thin targets, errors from these assumptions can be negligible, for thicker targets they can become significant. In order to correct for these effects, the density distributions along the ion beam and x-ray path are required. One possible way to determine these is to use computer assisted tomography (CAT) on STIM images taken with a range of target orientations.

CAT [4] is essentially a technique for calculating cross-sectional images of an object from a set of projections. As STIM can obtain a spatial resolution of 50 nm [5], and is ideal for making projection images of samples, it is suited for micron and sub-micron tomography. From the areal density projections, produced by STIM images, CAT reconstructions will be able to accurately determine the density of a sample.

This paper will illustrate the use of STIM tomography on a double barreled pyrex pipette of outer diameter 50 μm, displaying a spatial resolution of 0.37 μm, in this case limited by the relatively large pixel size used to allow for target precession.

RESULTS

The basic problem of CAT is to reconstruct a section image from multiple 1-dimensional projections. A simple approximate method of constructing a section image from multiple projections is merely smear (backproject) each projection across the image plane [7]. The resulting image is blurred by a function proportional to 1/r , where r denotes the distance from the image point. This blurring is easily corrected by simple Fourier filtering. This reconstruction technique is known as filtered backprojection [8].

90 projections taken every 2 degrees were read into a version of the program SEMPER (Copyright Synoptics LTD 1985,1986) and an image formed using the backproject algorithm. Figure 1 shows the resulting image. The image size is 80 by 80 μm spread over 220 by 220 pixels. There are 32 grey levels spanning a linear scale. The general outline and shape of the pipette can be seen, but contrast is poor due to the 1/r distribution.

Using SEMPER the 90 projections were used to form a filtered backprojection image. The method is summarised briefly below.

The 1-dimensional Fourier transform of each projection was taken using a fast fourier transform.For every projection in fourier space each coefficient was multiplied by a correction function

\[ c(f) = |f| w(f) \]  

(1)
where $f$ is the frequency and $w(f)$ is a window function. The inverse transform of the product was then taken for each projection and the corrected projections were backprojected to a reconstruction plane.

Figure 2 shows the reconstructed image, using (1) where

$$w(f) = \begin{cases} 1 & \text{if } |f| < 111 \\ 0 & \text{if } |f| \geq 111 \end{cases}$$

The image size is 73 by 73 $\mu$m spread over 200 by 200 pixels with a pixel size of 0.37 $\mu$m. The double barrelled pipette structure is clearly seen. The pipette is not circular but somewhat ellipsoidal in shape. The minimum and maximum diameters are 51 and 57 $\mu$m respectively. The width of the outer wall is 6.5 $\mu$m while the width of the partition is 4.5 $\mu$m. The partition is seen to pull away from the inner wall at the bottom of the image. The resulting wedge shaped gap is resolvable down to 2 pixels indicating a spatial resolution of better than 0.73 $\mu$m.

A horizontal slice, through the centre of the pipette, corresponding to row 111 of the 220 rows of pixels was extracted from figure 2. From this slice a graph of density versus position for the right hand edge of the pipette is plotted in figure 3. Fitted to these data was an error function with a FWHM of 0.31 $\mu$m. This indicates that as the spatial resolution cannot be less than one pixel it is limited by the pixel size and is 0.37 $\mu$m. Additional slices for various rows and columns of data in the image were also taken and confirmed that the spatial resolution was limited by the pixel size.

The density of the pipette calculated from the reconstructed image was 2.20 ± 0.16 g/cm$^3$. This compares favourably with the density of pyrex which is 2.23 g/cm$^3$ as given by TRIM (Transport and Range of Ions in Matter) [6].

CONCLUSION

STIM is essentially a non-destructive technique, beam currents are around 0.1 fA and images are formed from less than two million events. Thus it can serve as a useful tool to study the 3-dimensional structure of an object. Spatial resolutions down to 50 nm mean STIM tomography could be a useful tool for many applications, one of which [9] shows the distribution of catalytic particles in a foam.

REFERENCES

A HIGH CURRENT METAL VAPOUR VACUUM ARC ION SOURCE FOR ION IMPLANTATION STUDIES

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INTRODUCTION

A vacuum arc is sustained by material evaporated from the cathode during the discharge. Evaporation occurs at a multiplicity of small, localised regions of high current density \((10^5 - 10^9 \text{A cm}^{-2})\) known as "cathode spots". These are highly mobile and move randomly over the cathode surface. Vapour is emitted from these spots at velocities of up to \(1000 \text{ m s}^{-1}\). A fraction of the atomic particles removed from the cathode in this process are ionised to produce positive ions, some of which possess sufficient energy to reach the anode against the prevailing electric field gradient.

The metal vapour vacuum arc (MEVVA) ion source utilises the plasma production capabilities of the vacuum arc. By combining the latter with an extraction stage, it is possible to produce an intense beam of metal ions. This arrangement was first described by I. Brown and co-workers of Lawrence Berkeley Laboratory\(^1\) who have also been responsible for much of its subsequent development.

The composition of the vacuum arc cathode determines the particular ions obtained from the MEVVA source. In contrast, other types of ion sources capable of producing metal ions often require feed material to be in gaseous or compounded form. Use of the latter may lead, during ionisation, to the formation of undesirable, reactive products which degrade source performance. The absence of such deleterious effects in MEVVA ion sources suggests they may have considerable applications in the field of ion implantation. In particular, their high beam current and their potential for generating broad beams make them suitable for the implantation of large surface areas such as might be encountered in the treatment of some metal products. It is this application which is currently being investigated by ANSTO.

EXPERIMENTAL

The main features of the MEVVA ion source are shown in Figure 1. In addition to the cathode and annular anode, a third electrode is incorporated into this design for triggering purposes. A high voltage pulse \((-10 \text{ kV})\) applied between the trigger electrode and the cathode leads to breakdown on the surface of the alumina insulator. This produces a small burst of plasma which initiates the main discharge.

Some of the ions produced in this discharge flow through the anode and the 3 extraction grids to form an extracted ion beam. Extraction potentials of up to 70 kV are routinely achieved with the arrangement shown in Figure 1.
For most cathode materials, vacuum arcs and hence, MEVVA ion sources produce ions with a distribution of charge states. Brown et al. have used time-of-flight spectra to determine these distributions for a large number of elements and several binary compounds. Some typical examples are listed in Table 1.

![Schematic diagram of MEVVA ion source](image)

**Figure 1** - Schematic diagram of MEVVA ion source

**Table 1 - MEVVA Charge State Distributions**

<table>
<thead>
<tr>
<th>Cathode</th>
<th>Observed Ion</th>
<th>Ion Fractions, %</th>
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<tbody>
<tr>
<td>C</td>
<td>C⁺</td>
<td>100</td>
</tr>
<tr>
<td>Al</td>
<td>Al⁺, Al₂⁺, Al₃⁺</td>
<td>56, 39, 5</td>
</tr>
<tr>
<td>Ti</td>
<td>Ti⁺, Ti₂⁺, Ti₃⁺</td>
<td>6, 82, 12</td>
</tr>
<tr>
<td>Cu</td>
<td>Cu⁺, Cu₂⁺, Cu₃⁺</td>
<td>44, 42, 14</td>
</tr>
<tr>
<td>W</td>
<td>W⁺, W₂⁺, W₃⁺, W₄⁺, W₅⁺</td>
<td>8, 34, 36, 19, 3</td>
</tr>
<tr>
<td>TiC</td>
<td>(Ti²⁺, Ti³⁺) (C⁺, C²⁺)</td>
<td>40, 10</td>
</tr>
</tbody>
</table>

The Rutherford backscattering spectrum for a sample implanted with a MEVVA ion source is shown in Figure 2. In this case, the surface of a aluminium/zinc alloy was bombarded with tungsten ions extracted at 50 kV. The implanted tungsten layer is clearly evident in the spectrum.
DISCUSSION

The MEVVA ion source is a fairly recent development in ion source technology. Despite this, it already forms the basis for significant research programs in the USA, Russian and Japan. In the latter, Nippon Steel Corporation (NSC) has recently acquired a complete implanter system which incorporates a MEVVA ion source. This system is now fully operational and the construction of a second unit is presently underway. Available information suggests that NSC is particularly interested in implantations with composite cathodes (i.e. TiO₂).

A project concerned with the development of MEVVA-type ion sources for implantation of metals and ion beam analysis of materials was commenced by ANSTO in late 1988. To date, a MEVVA ion source has been constructed and an implantation system is nearing completion. It is anticipated that the facility will be in operation by the end of 1989.

REFERENCES

ION SCATTERING ANALYSIS OF CaF\textsubscript{2}/Si(111)

by

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Abstract

Thin epitaxial insulators have great potential for application in SOI devices, 3D integrated circuits or to lattice match III-V semiconductors onto Si substrates. CaF\textsubscript{2} is a favoured insulator for epitaxial growth on Si since it is lattice matched (0.6% at RT) and high quality films can be grown by Molecular Beam Epitaxy on Si substrates. These films always grow in B-type epitaxy, where the epitaxial layer is rotated by 180° about the <111> axis normal to the surface. High energy ion channeling measurements indicate very good crystallinity near the film surface although increased scattering from Ca occurs within 20nm of the film-substrate interface. This interfacial disorder and associated tensile strain in the films has been related to nucleation of interfacial defects to accommodate misfit strain developed during high temperature deposition\textsuperscript{*}. The aim of this work is to investigate the growth of CaF\textsubscript{2} on Si(111) and re-examine the high energy scattering results.

A low energy ion scattering technique, CAICISS, (coaxial impact-collision ion scattering spectroscopy)\textsuperscript{2} has been used with LEED to determine the structure of thick CaF\textsubscript{2} films deposited on Si(111). In this technique 2 KeV He\textsuperscript{+} is scattered from a surface with scattering angle 180° and detected by a time-of-flight spectrometer. CaF\textsubscript{2} films 1-100ML thick were deposited by MBE onto a heated Si(111) substrate and analysed during or after deposition by CAICISS and LEED. Films were then deposited from a boron nitride crucible at 1275°C onto 7x7 Si substrates at temperatures between RT and 700°C. Figure 1 shows a CAICISS TOF spectrum from a CaF\textsubscript{2} film in which peaks due to the scattering of He off Ca, Si and F are clearly resolved.

The deposited CaF\textsubscript{2} films always exhibited a 1x1 LEED pattern and ion scattering angular spectra characteristic of good epitaxial growth of CaF\textsubscript{2}. For example, figures 2a, b and c show graphs of the intensity of the Ca surface peak in the <112> azimuth as a function of the angle of incidence of the ion beam to the surface of (a) bulk CaF\textsubscript{2} and thick CaF\textsubscript{2} epitaxial films grown at (b) 700°C and (c) RT. The intensity minima at 54°, 90° and -54° to the [112] direction in figure 2a are due to shadowing of Ca atoms along the [110], [111] and [001] channeling directions respectively. The intensity peaks on either side of these dips are due to focussing of ions onto deeper atomic layers. Thick films of CaF\textsubscript{2}, grown at 700°C (fig 2b) are seen to give the same ion scattering spectra as that for bulk CaF\textsubscript{2} (figure 2a), indicating that high quality single crystal films can be grown under these conditions. CAICISS allows the underlying Si substrate to be observed through a CaF\textsubscript{2} film. An incidence angle scan for the Si surface peak from the substrate underlying a 1ML film (figure 2d) has a similar shape to that found for Ca but the peak positions are reversed about the 90° incidence angle. This shows that CaF\textsubscript{2} and Si have identical structures but the CaF\textsubscript{2} epitaxial layer is rotated 180° with respect to the substrate, i.e. B type epitaxy.
CaF$_2$ films of medium thickness (15 minutes growth at 700°C) show a quite different incidence scan (figure 3a) to that from the thickest films (figure 2b). The large peaks at 36° and 73° in the incidence scan in figure 2b are reduced while new peaks at 60°, 78° and 120° are seen. Since the angular scans for the medium and very thick films differ and the very thick film corresponds to a bulk CaF$_2$ structure, it is likely that figure 3a corresponds to a near interface structure which is overgrown with a bulk-like CaF$_2$ layer. This structure was identified by annealing the thick CaF$_2$ epitaxial layers. Figure 3b shows a CAICISS angle of incidence scan for the medium thickness film after 8 minutes annealing at 750°C. The anomalous peaks are even higher than in the spectrum for the unannealed layer (figure 3a). This scan can, however, be modelled very well by assuming that the underlying layer is CaSi$_2$ (figure 3c). This is justified since, if F is lost from the film during deposition, as is found during annealing, then the Ca metal rich layer will react with the Si substrate to form epitaxial CaSi$_2$, in much the same way as thermal formation of CaSi$_5$. CaSi$_2$ is known to give the 2x2 LEED pattern observed and grows on Si(111) with a bulk CaSi$_2$ structure in B-type epitaxy. Figure 3c shows the simulation of a bulk CaSi$_2$ structure which is terminated by Ca atoms. The agreement with the peak positions in the spectrum of the annealed film (fig 3b) is excellent. The as-deposited film (figure 3a), which contains features of figures 2b and 3b, then corresponds to a mixture of CaF$_2$ and CaSi$_2$. Formation of CaSi$_2$ only occurs near the substrate since, if deposition of CaF$_2$ is continued, Si supply from the substrate to the film surface will be presumably limited by diffusion through the film. Thicker films of CaF$_2$ will then grow in a bulk-like structure.

Epitaxial layers of CaF$_2$ have been grown by MBE on Si(111) substrates and analysed by CAICISS and LEED. Annealing experiments and modelling of the CAICISS angular scans indicate that the thickest films showed bulk CaF$_2$ structure with B type epitaxial growth. Medium thickness films were similarly found to contain both CaF$_2$ and CaSi$_2$. The presence of CaSi$_2$ would give rise to anomalous Ca yields seen in high energy channeling.

REFERENCES


Figure 1 A CAICISS time of flight spectrum for 2 KeV He$^+$ scattered from a CaF$_2$ epitaxial layer deposited on Si(111). The ion beam was incident at 12° to the surface in the [-1-12] azimuth. Surface peaks corresponding to Ca, Si and F occur at flight times of 5170 ns, 5600 ns and 5800 ns respectively.
Figure 2. The CAICISS yield from the Ca surface peak as a function of the angle of incidence of 2 keV He\(^+\) with respect to the sample surface for (a) bulk CaF\(_2\), (b) a thick epitaxial CaF\(_2\) film on Si(111) grown at 700\(^\circ\)C, (c) a thick epitaxial CaF\(_2\) film on Si(111) grown at RT, and (d) the CAICISS yield from a Si substrate coated with a 1ML CaF\(_2\) film.

Figure 3. The CAICISS yield from the Ca surface peak as a function of the angle of incidence of 2 keV He\(^+\) with respect to the sample surface for (a) an epitaxial CaF\(_2\) film deposited onto Si(111) at 700\(^\circ\)C for 15 min, (b) the above film after annealing at 750\(^\circ\)C for 8 minutes, (c) a numerical simulation of ion scattering from Ca terminated CaSi\(_2\).
APPLICATION OF OUT-OF-PLANE MEIS TO SURFACE STRUCTURE ANALYSIS

by

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Abstract

Multilayer relaxation of clean Fe(lll) surface has been studied by using medium energy ion scattering with both in- and out-of-plane geometries. One of the advantages of out-of-plane scattering is that it gives out greater range of choice in scattering geometry, which can be critical in high index surface analysis. When compared with a full crystal Monte Carlo simulation, the results indicate an oscillatory relaxation surface structure with a -29% of first layer (ΔD₁₂) contraction and a 6% of second layer (ΔD₂₃) expansion.

1. Introduction

The essence of the medium energy ion scattering technique for surface structure analysis is to find out two low index crystallographic directions for channeling and blocking to highlight the surface properties being measured. Greater flexibility is achieved if the restriction of limiting these directions (channeling and blocking) to the same plane defined by ion incidence direction and surface normal is relieved [1]. This can be critical in high index surface analysis. In the present study, an out of plane scattering measurement, in which the analyzer was tilted away from the incidence plane, has also been applied to maximizes the sensitivity of the technique for surface relaxation analysis.

2. Experiment and Results

The measurements were performed in a UHV chamber coupled to a 120 keV accelerator. The Fe(lll) sample was mounted on a two rotation axis goniometer with three independent translations. The cleaning process included an initial anneal at 700°C for two hours and subsequently at 800°C for 20 minutes before every set of measurement. The backscattered ions were analyzed by a compact electrostatic analyzer [2] which was mounted on a goniometer with two independent rotational axes. In the present study, all the experiment data have been analyzed by a full crystal Monte Carlo program [3]. The fit between experiment data and simulation results was carried out by a R-factor analysis which was similar to that used in LEED analysis [4].

In-plane scattering: The incidence beam was aligned in the <100> channeling direction and the scattered ions were detected around the <111> blocking direction in the (011) scattering plane (fig.1a). The minimum in the angular scan of the surface peak shows a 2.5° shift from the bulk <111> direction which is determined by the bulk blocking minimum.

Out-of-plane scattering: This scattering geometry was chosen to maximize the sensitivity of the measurements to the first interlayer spacing displacement (fig.2a). The scattered ion beam was measured about <111> direction while retaining the incident beam in another <111> channeling direction in which all the subsurface atoms are shadowed by the upper layer atoms. The surface blocking curve (fig.3) shows a almost 5 degrees (in β) shift of surface minimum form the bulk dip. The more sensitive angular change make the estimate of the relaxation more precise.
Fig. 1. (a) Top view: schematic picture of (0-11) scattering plane side view indicating the channeling <100> and blocking <111> direction. Bottom view: surface blocking curve for the clean Fe(111) surface in the (0-11) scattering plane, the squares are experimental data and the solid line is a Monte Carlo simulation with the best fit parameters $\Delta D_{12} = -29\%$, $\Delta D_{23} = 6\%$ and a Debye temperature 260K. (b) Contour plot of the $R$ factor as a function of first two interlayer spacings relaxations comparing the experiment results with the calculation in the (0-11) scattering plane.

Fig. 2. (a) Top view: out-of-plane scattering geometry with the <111> channeling and <1-11> blocking direction. Bottom view: surface blocking curve form out-of-plane scattering measurement (squares) using 55kev proton. The solid line is a Monte Carlo simulation using the best parameters $\Delta D_{12} = 29\%$ and $\Delta D_{23} = 6\%$. (b) Contour plot of the $R$ factors from out-of-plane scattering measurement and calculation. The numbers on the lines are $R$ factors as a function of first and second interlayer spacings displacements.

Fig. 1b is the contour plot of simulation and experimental results in the (011) scattering plane. It is clear from the best fit area that the sensitivity of the measurements are almost the same for the first two interlayer spacings displacement because both of layer atoms arc involved in the scattering process. Since the second layer atoms are as same as the first layer atoms exposed to the incident ion beam, the geometry is sensitive to the average of first and second interlayer spacing relaxation. The contour results from out-of-plane scattering measurements and calculations are shown in fig. 2b. In this geometry, the backscattering yield from the interaction of ion and the third or deeper layer atoms is not
significant, the first layer relaxation determination has been then further improved since the application of the out-of-plane geometry enabled to obtained the best blocking geometry, in which the second or deeper layer atoms are blocked by the surface atoms in this scattering geometry.

The final results of best parameters of surface relaxations can be illustrated from the total contour summing the R factors from two scattering geometries (fig.3). The best parameters of surface relaxations were obtained from the cross center of the contour: $\Delta D_{12} = (29\pm7)\%$, $\Delta D_{23} = (7\pm5)\%$.

\[ \text{Fig. 3 Total contour plot with the average R factor including (0-11) and out-of-plane scattering results.} \]

### 3. Conclusion

Multilayer relaxation surface structure of Fe(111) has been measured using in and out-of-plane MEIS. The results show a first interlayer spacing contracted by $(29\pm7)\%$ and a second interlayer spacing expanded by $(6\pm5)\%$ which are listed in the table 2 together with other LEED and MEIS experiment works. From table 2, our results are in excellent agreement with the MEIS measurements [19].

#### Table 2. Relaxation of clean Fe(111) surface

<table>
<thead>
<tr>
<th>$\Delta D_{12}$</th>
<th>$\Delta D_{23}$</th>
<th>Technique</th>
<th>Reference</th>
<th>date</th>
</tr>
</thead>
<tbody>
<tr>
<td>$-15.4\pm4$</td>
<td>------</td>
<td>LEED</td>
<td>[17]</td>
<td>1981</td>
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<tr>
<td>$-16.9\pm3$</td>
<td>$9.8\pm3$</td>
<td>LEED</td>
<td>[18]</td>
<td>1986</td>
</tr>
<tr>
<td>$-27.0\pm5$</td>
<td>$4.0\pm5$</td>
<td>MEIS</td>
<td>[19]</td>
<td>1986</td>
</tr>
<tr>
<td>$-29.0\pm7$</td>
<td>$6.0\pm5$</td>
<td>MEIS</td>
<td>present</td>
<td>1989</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>work</td>
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</tbody>
</table>

One of the aims of this work was to demonstrate the usefulness of out-of-plane geometry scattering which can enhance the sensitivity of the technique. An out-of-plane geometry can give out greater range of choices in scattering geometry - allowing the choice of scattering conditions which best highlight the properties being measured.

### References

REIONIZATION IN LOW ENERGY He-Pb SCATTERING
by
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Abstract

The scattering of 50-2000 eV He⁺ off Pb has revealed two interesting features. Over a limited range of energies, at particular scattering angles, two distinct peaks in the energy spectra are observed which will be referred to as the high and low energy peaks. The high energy peak is observed only below a critical incident energy which depends on the scattering angle. The low energy peak is only observed at energies above a different threshold energy. There is an overlap region of incident energies at which the ion yield is initially dominated by the high energy peak. With increasing incident energy this peak gradually disappears and the low energy peak dominates. An intriguing feature of these two peaks is their energy width which for the high energy peak is much less than that expected for scattering off a real surface and is equal to the analyzer resolution. The low energy peak has a much larger energy width than would be expected for the combined effects of analyzer resolution, thermal broadening and scattering.

Introduction

There have been numerous studies of the rate of energy loss of projectiles in solids but it has been difficult to separate individual loss processes in most systems. In Low Energy Ion Scattering (LEIS) using inert gas ions, discrete losses can be identified as a relative unique scattering event leads to the detection of scattered ions. The earliest observation of low energy discrete loss was reported by Heiland and Taglauer for the scattering of Ne off Ni [1]. In that experiment a loss was observed which they attributed to the reionization of Ne ions which had been neutralized on the approach to the surface. Interest was renewed in the study of discrete energy losses when studies of the quasi-resonant charge exchange between He and Pb revealed that the oscillatory component of energy loss could be related to the reionization of He in the vicinity of the Pb surface [2]. In a recent study of the limits of the quasi-resonant process it was revealed that for distances of closest approach of greater than 0.884Å all oscillatory charge exchange processes stopped [3]. If there were discrete losses then they must result from other processes.

More recent experiments on the reionization of inert gas projectiles [4-12] have identified the process which leads to a discrete loss at scattering angles from 7° to 90° and at energies greater than 200eV. The observations have shown that curve crossing processes between the ground state of the He ion and the target lead to the energy loss. The energy thresholds determined experimentally agree well with calculations. In the following work it will be seen that while the energy loss measured is discrete, it is also a function of energy, leading to the conclusion that the transition occurs predominantly at the distance of closest approach and that the loss is a measure of the potential energy difference between two states.
The experimental system is a Leybold-Hereaus ion scattering system which is pumped by a combination of a turbomolecular pump and a titanium sublimation pump which achieve a base pressure of $5 \times 10^{-11}$ mB. The energy analyzer has a geometrical energy resolution of 1.25% and once combined with a retardation factor (2.5, 8 and 25) energy resolutions of 0.5%, 0.16% and 0.05% are attainable. In the work to be reported here the energy resolution of 0.5% has been used. The energy analyser is mounted on a goniometer which has two degrees of rotational freedom about the target surface.

The target was a 99.999% Pb polycrystalline sample mechanically polished to a flat clean finish before introduction to the system. The sample was mounted on a goniometer which has three translational degrees of freedom and two rotational degrees of freedom. As the experiments involved small scattering angles a further cleaning stage was undertaken with the sample being subjected to an intense Ar ion beam at an incidence angle of 5° to the surface to both clean and flatten it. Analysis by ISS after this procedure yielded a surface free of adsorbates to the limit of detection.

Data acquisition was performed by a LeCroy 3500 computer interfaced by CAMAC modules to the apparatus. The data acquisition, control of the ion gun and manipulation of the target by stepper motors were all under computer control. The beam current was monitored with a beam current digitizer to enable beam current normalization.

**Results and Discussion**

In the following presentation of results all energies are referenced to the energy of the narrow high energy peak. It is expected that the highest energy peak will have suffered some inelastic energy loss but that will not be considered in the following analysis.

A sequence of scattered ion spectra for increasing projectile energies (75 to 175 eV) of He scattered off Pb at a scattering angle of 32° (fig. 1) reveals the characteristics of a narrow high energy peak and a broad low energy peak. The growth of the low energy peak at the expense of the high energy peak is evident. In this figure the vertical zero offset of each spectrum represents the projectile energy and for ease of illustration the spectra have been normalised to have the same integrated yield in the high energy peak. The same behavior has been observed at scattering angles of 42° and 22° but over a different range of projectile energies. The low energy peak is observed when the distance of closest approach is less than 1.1 Å. The high energy peak vanishes when the distance of closest approach is smaller than 0.89 Å. These values are just outside the distance of closest approach at which the quasi-resonant charge exchange process is found to cease [3].

It can be seen in fig. 1 that the energy difference between the two peaks increases with projectile energy and this is more clearly illustrated in fig 2. The maximum loss observed at all three angles studies is approximately 6 eV. Since the measured loss increases with projectile energy, the reionization processes described in previous studies are inappropriate to explain these results. This interaction occurs at distances of closest approach just outside the range of the observed quasi-resonant charge exchange process and as the maximum loss of 6 eV is much less than the ionization or excitation energies of He, the preliminary explanation for this process is that the loss involves a transition between the 5d levels of Pb and the ground state of He.
In the model for quasi-resonant charge exchange the ground state of He and the 5d levels of Pb mix at an inter atomic separation of about 2.7 Å and as the inter atomic separation decreases, the energy levels for the two states (He*\textsuperscript{-}Pb and He-Pb\textsuperscript{*}) diverge. It is a transition between these two potential energy curves that is proposed to explain these observations. The increasing loss with higher energy results from an increasing separation between the levels as the particle separation decreases, and the broadening in the low energy peak results from a range of potential energy transitions over the trajectory. After the collision the projectile may be reionized when the levels mix on the exit. Thus this process can be regarded as the precursor to the quasi-resonance process.

\begin{equation*}
\text{He off Pb}
\end{equation*}

\textbf{ENERGY LOSS}

\begin{equation*}
32 \text{ DEGREES}
\end{equation*}

\textbf{32 DEG SCATTERING ANGLE}

\textbf{Fig. 1} Ion scattering spectra for He*\textsuperscript{-} off Pb with a scattering angle of 32 degrees. The level of the baseline indicates incident energy.

\textbf{Fig. 2} Energy difference between the two peaks vs. incident energy.

\textit{References}

Polarity Determination of Epitaxial Structures of CdTe on GaAs by Channelling Technique.

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1. INTRODUCTION
The interaction of energetic ions with solids is used in materials modification and analysis in modern research and technology. Rutherford Backscattering (RBS) and Nuclear Reaction Analysis (NRA) in combination with ion channelling are very powerful techniques in thin layer analysis due to very good mass and depth resolution and capability to detect, identify and provide depth profiles of crystal defects such as interstitial atoms, dislocations and dislocation loops [1-5]. In the case of epitaxial and heteroepitaxial structures, interface quality and strain in the epitaxial layer can be determined by the channelling technique [6]. In compound semiconductors the face polarity is an important parameter affecting chemical and physical properties. Different methods have been used to determine the polarity of the (111) CdTe surface including chemical etching, x-ray diffraction and electron diffraction [7-9]. Recently the ion channeling technique, convergent beam electron diffraction (CBED), characteristic x-ray emission under various electron channelling conditions (ALCHEMI) and x-ray diffraction have been proposed as methods for compound semiconductor polarity determination [10-12].

2. THE CHANNELLING METHOD.
The (111) face in CdTe is a polar face. The (111)A face is terminated by a triply bonded Cd atom and the (111)B face is terminated by a triply bonded Te atom (see Fig. 1 (a) and (b)). The atomic stacking sequence in the crystal is related to the (111) surface polarity and the surface polarity can be determined from RBS spectra measured at about the critical angle \( \psi_c \) in any (111) planar channeling direction [10].

Let us assume that the ion beam is channelled in one of the inclined (111) planes as indicated on Fig. 1 and let us define angle \( \alpha \) as the angle between the beam direction and the normal to the surface (111) axis. Let us assume that we will be changing the angle \( \alpha \) only and that \( \alpha = \alpha_c + \psi_c \) corresponds to the planar channeling minimum. When \( \alpha = \alpha_c + \psi_c \), the first close interaction of the ion beam entering the (111) channel will predominantly be with crystal atoms of one kind. From Fig. 1 it is clear that in this case the ion beam will be first scattered dominantly by Cd atoms in (111)A face crystal or by Te atoms in (111)B face crystal. The result will be opposite when \( \alpha = \alpha_c - \psi_c \).

3. EXPERIMENTAL
Commercially available (111) CdTe epitaxial layers on 50mm diameter sapphire have been analysed using a 3MV particle accelerator as a source of 2 MeV He ions. The samples were mounted on a three axis goniometer in the vacuum chamber. The ion beam diameter was about 1mm and sample translations were used to avoid significant beam damage. The RBS channelling analysis was performed first in a direction normal to the sample surface to identify crystal orientation and measure crystal quality. The next step was to search for (111) planar channelling in one of (111) planes forming a 19.47°
angle with the normal to the surface (111) axis. Finally the sample tilt angle was reduced by about $2^\circ$ from the value corresponding to the selected direction within the (111) plane and a scan across this (111) plane was made by increasing the sample tilt angle in $0.1^\circ$ steps. During each scan step the RBS spectrum was recorded as well as the goniometer setting.

4. RESULTS AND DISCUSSION

The RBS spectra of CdTe on sapphire recorded during a scan across the (111) plane are shown on Fig. 2. The best (111) planar channelling corresponds to the lowest counting rate in the low energy part of the spectrum (channels 160-170) and it takes place for $\alpha = 21.5^\circ$. The spectra shown on the top and the bottom of Fig. 2 correspond to $\alpha = \alpha_0 + \Delta\alpha$ and $\alpha = \alpha_0 - \Delta\alpha$, respectively. These two spectra are compared on Fig. 3. From Fig. 3 it is clear that for $\alpha = 21.8^\circ$ the channelled ions interact first predominantly with Cd atoms and in the opposite case of $\alpha = 21.2^\circ$, the first interaction takes place mostly with Te atoms. From these results we conclude that this sample has (111)A face. Our results agree with independent polarity determination of the same sample by x-ray diffraction technique [12].

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**Fig. 1.** (110) plane views of CdTe crystals with (111)A face (a) and (111)B face (b). The Te atoms are represented by full circles and Cd atoms by open circles. The Te (111) atomic planes are represented by solid lines and the Cd planes by dashed lines. The thick solid lines represent Cd-Te atomic bonds. The (111) planar channelling direction used in polarity determination is indicated by the arrow.

**Fig. 2.** RBS spectra collected during angular scan across (111) plane from CdTe layer on sapphire. The best planar channelling is observed for $\alpha = 21.5^\circ$. 
5. CONCLUSIONS

Polarity determination by (111) planar ion channelling is a very useful nondestructive direct technique. In contrast to x-ray diffraction, it can be applied to very thin epitaxial layers (less than 100nm). It is applicable to any compound semiconductor material when elements involved can be resolved by RBS.

6. ACKNOWLEDGMENTS

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REFERENCES

HIGH ZINC CONCENTRATION IN SPIDER FANGS

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Abstract

We have used a scanning proton microprobe to investigate the distribution of elements in the fang of the white tailed spider, Lampona Cylindrata. The results indicate a high zinc concentration of 0.13 ±0.02g/g in the fang.

Method

A white tailed spider was anaesthetized and its fang removed and left to dry for three weeks. The fang was then mounted on a standard microprobe stainless steel target holder. The tip of the fang was subsequently scanned with a 3 MeV hydrogen beam of spot size 3μm, over a 272 x 328 μm scan area until 0.2μc of charge was collected. A STIM scan was then performed on the fang with a spot size of less than 0.5μm to map the areal density of the sample. PPM concentrations for the elements zinc and chlorine were calculated by normalising the resultant PIXE image to the areal density image.

Results

Figure 1 is the resultant zinc distribution from the PIXE scan. Figure 2 shows the areal density distribution from the STIM scan, a duct running down the middle of the fang can be seen, and a portion of the tip of the fang is seen to be missing. Pixel by pixel zinc concentrations were calculated using a modified version of the program thick [1]. The composition of the fang was initially chosen to approximate cuticle, of which the exoskeleton of many insects and spiders are composed. This has the approximate composition C₆₃H₆₀O₂N [2]. This estimated composition should be quite a good approximation since stopping powers and mass attenuation coefficients are fairly insensitive to changes in composition of biological material [3].

Once the concentrations of the elements had been obtained, corrections were made to the composition, to accommodate any previously ignored major elements. The composition of the fang was adjusted to allow for the fact that the zinc concentration was around 0.15±0.02 g/g, and the pixel by pixel concentrations were recalculated. The process of iteration was continued until the change in the concentration was less than 0.01 g/g. Figure 3 shows the resulting zinc concentration. The concentration is higher on the inside edge of the fang, indicating that it is perhaps forming some form of support in the fang. The black spots are where an x-ray event has been detected but no areal
density has been recorded. They result from anomalous x-ray events and from target movement during the PIXE irradiation. Ignoring these black regions, the average zinc concentration in the fang is 0.13 ±0.02 g/g.

Considering the nature of the fang, it seems likely that the zinc accumulation is associated with mechanical properties - such as hardness, rigidity or fracture resistance.

References


CORRECTED ZINC CONCENTRATION

227 um

0.001 mg/g

>0.22 g/g

227 um

32 GREYSCALES USED
SUBSTRATE ORIENTATION DEPENDENCE DURING ANNEALING OF AMORPHOUS LAYERS PRODUCED BY STOICHIOMETRIC ION IMPLANTATION

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ABSTRACT

The crystallization of intrinsic amorphous layers produced by stoichiometric implantation of Al and O ions in α-axis oriented Al2O3 has been studied. The amorphous phase transforms directly to α-Al2O3 by the motion of a well defined planar interface. The interface velocity shows an Arrhenius behaviour with anneal temperature. The crystallized phase and the activation energy are compared with that of c-axis sapphire. The crystallization of top-surface layer (non-epitaxy) was also observed. Substrate orientation is shown to play a dominant role in deciding the mode of crystallization as well as the kinetics of transformation.

INTRODUCTION

The near-surface modification of α-Al2O3 by ion implantation and subsequent annealing is known to create novel optical, chemical, mechanical and electrochemical properties. Previous studies on ion implanted α-Al2O3 with several ion species, e.g. In, Zn[1], showed a complex dependence on ion species, dose, anneal temperature and substrate orientation. In this work we report on a study of the crystallization of intrinsic amorphous layers produced by stoichiometric implantation of Al and O ions in α-axis oriented α-Al2O3 substrate.

EXPERIMENTAL

Single crystals of α-axis Al2O3 (<1210> orientation) were used in this work after pre-annealing at 1400°C in an oxygen environment for 5 days to remove any residual surface damage incurred during surface polishing. Following the recipe of the Oak Ridge group[2] a stoichiometric implant, with a dose of 4 x 10^16 Al+/cm² at 90 keV, and of 6 x 10^16 O+/cm² at 55 keV, at the liquid nitrogen temperature was used to produce the amorphous phase of Al2O3 free of any impurities. The ion energies were selected to give similar projected ranges for both species. The isothermal annealing was performed in flowing high purity argon gas at 600°C, 800°C and 900°C for time periods ranging from half an hour to 10 hours. Rutherford backscattering and ion-channeling measurements (RBS/C) and RHEED were employed to study the crystallization behaviour.

RESULTS AND DISCUSSION:

The RBS/C results in Fig.1a show that a stoichiometric implant at LN2 produces an amorphous layer (confirmed by RHEED) about 1300Å thick on α-Al2O3. The aligned yield in the implanted region clearly reaches the random value in Al sublattice. After annealing at 900°C for 0.5h the amorphous layer of 690Å thick was measured in Fig.1b. The crystallized layer is epitaxial with the underlying α-Al2O3 substrate, and grows by the motion of an abrupt amorphous/
\( \alpha \)-phase interface. The aligned yield in the surface region of up to 690Å depth still reaches the random value in the Al sublattice after annealing. A plot of the regrown layer versus anneal time is indicated in Fig.2. Two regions, in which the crystallized thickness varies linearly with annealing time, can be clearly identified for all of annealed temperatures at 600°C, 800°C and 900°C. From these measurements, the velocity of the interface can be determined. The crystallization velocity slows down in region II after an initial rapid growth in region I. Fig 3 shows growth velocities as a function of inverse of anneal temperature \( T \). The data are roughly fitted with activation energies of \( Q_I = 1.48 \text{ eV} \), and \( Q_{II} = 0.75 \text{ eV} \) for the two regions of epitaxial regrowth. From a detailed comparison with the Oak Ridge data [2] on epitaxial regrowth (via an intermediary \( \gamma \)-phase) of intrinsic amorphous layers on c-axis \( \text{Al}_2\text{O}_3 \), we conclude that substrate orientation plays a dominant role in deciding the mode of crystallization as well as kinetics of transformation since the activation energies are much different from 3.6eV for c-axis data [2]. The data from RHEED results tell us that the crystallization behaviour of top-surface layer, which is less than 200Å thick, is more complex. Fig.4 shows the formation of \( \kappa \)-phase (Fig.4a) after annealing at 600°C for 10h, and of \( \gamma \)-phase (Fig.4b, Fig.4c) at 800°C for 7h and 900°C for 4h. This is different from the epitaxial crystallization at the a-c interface and may be random on surface crystallization.

Further study is in progress to confirm these phenomena and understand the mechanism for the recrystallization of amorphous \( \text{Al}_2\text{O}_3 \), which is not very clear so far.

REFERENCES


Fig. 2. The dependence of the crystallized thickness on the annealing time at indicated temperatures.

Fig. 3. An Arrhenius plot for $a$-axis $\text{Al}_2\text{O}_3$ samples by stoichiometric implantation of $\text{Al}$ and $\text{O}$ ions at LN$_2$.

Fig. 4. RHEED patterns obtained from the annealed samples. $\kappa$-phase was shown after annealing at 600°C for 10h (a), and $\gamma$-phase was shown respectively at 800°C for 7h (b) and at 900°C for 4h (c).
S.I.M.S. ANALYSIS OF SEMICONDUCTOR STRUCTURES

by

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Abstract

Introduction

Secondary ion mass spectrometry (SIMS) is a very sensitive method of elemental profiling of dopants and impurities in semiconductors. The new Riber SIMS MIQ 256 system at ANU has been used for a number of semiconductor profiling applications, some of which are described in this paper.

Ion Implanted Silicon

Ion implanted profiles of several species have been measured including B, P, As, In, Ga, Sb and Sn in both crystalline and preamorphised silicon. Channeling tails have been observed for B and diffusion and segregation effects have been measured for In, Ga and Sb following thermal annealing. Depth scales have been determined using an Alpha Step profilometer to measure sputter etch pit heights.

Impurities in Gallium Arsenide Structures

Profiling of Al, Si, S and Cr in VPE, MOCVD, MBE and ion implanted GaAs and AlGaAs layers has been carried out using both O$_2^-$ and Ar$^+$ ion beams to determine doping uniformity and segregation effects. Ion implanted standards have been used to provide absolute concentrations.

Sn-alloyed ohmic contacts to GaAs have also been studied using SIMS to give concentration versus depth profiles.

Ion Implanted Silicides

Boron and phosphorus have been implanted into amorphous NiSi$_2$ and profiled using SIMS both before and after thermal crystallisation. Little diffusion and segregation was observed.

Oxygen Profiling

Oxygen has been profiled in silicides, silicon and gallium arsenide. In the case of silicides, oxygen is found to retard epitaxial growth. In both silicon and gallium arsenide oxygen was first ion implanted and then the substrates annealed to form buried insulating layers. The oxygen concentration profiles were measured with SIMS and correlated with electrical and other properties.
A 50 kV TARGET BIAS SYSTEM

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Introduction

Ion implanters typically use an extraction voltage in the range 50 to 80 kV to obtain beams from the ion source. Many applications of ion implantation, particularly metallurgical uses and advanced semiconductors with deep buried layers require higher energies to increase the depth of penetration of the ions into the substrate. This necessitates a further stage of acceleration.

A number of options exist for this secondary acceleration:

(i) Acceleration after initial extraction, but before the analyser magnet
(ii) Acceleration after the analyser magnet
(iii) Negative bias of the target chamber

Each of these techniques has advantages and disadvantages. Acceleration prior to the analyser magnet means that the magnet must handle the higher energy and hence is considerably larger and more expensive. Acceleration after the analyser magnet means that the analyser magnet and power supplies must all be operated at a high potential (50 - 100 kV). This in turn means that the ion source is at a potential of 50 kV with respect to the magnet and 100 - 150 kV with respect to ground. If the whole of the target chamber is raised to negative potential, it requires large insulators, an insulated pumping system and a safety cage surrounding the high voltage components.

Equipment

Under conditions of high vacuum, voltage gradients of 50 kV can be maintained over a few mm whereas in air this voltage requires at least 50 mm. We have developed a novel system where the target chamber body and the vacuum pumping equipment are maintained at ground potential. However the target holder and associated scanning equipment are insulated from ground by using high density PVC. The negative bias is obtained from a power supply and is fed into the chamber through a high voltage vacuum insulator.

A significant problem encountered with a negative bias of this kind is the release of secondary electrons from the sample. To overcome this problem, the sample holder is surrounded by a large screen cage mounted
inside the chamber and supported by high voltage insulators. A second high voltage power supply and feed through insulator are used to maintain the cage at a negative potential of several thousand volts relative to the target. Secondary electrons emitted from the target then see this negative potential and are trapped within the cage. The power supply for this screen is capable of 60 kV, to enable the full 50 kV bias to be applied and at the same time provide adequate suppression of the secondary electrons.

The screen has shaped entry and exit ports for the beam and target manipulator. The sample holder can be moved at right angles to the beam and contains a hole which can be aligned with the beam entry port and a similar beam exit port, to enable the beam to pass through to a grounded Faraday Cup, for regular beam monitoring during an implant.

The high voltage power supplies are interlocked with the vacuum to ensure that they cannot be turned on until the pressure is sufficiently low to enable the voltage gradients to be maintained in the chamber. The high voltage is mechanically and electrically interlocked to prevent contact with high voltage when the power is turned on.

The universal sample manipulator used in conjunction with this high voltage system is described elsewhere at this Conference.\[1]\)

This relatively simple approach to target bias enables the energy range of the implanter to be extended with minimum cost. A similar approach in Ion Beam Analysis enables target voltage to be ramped in search of a resonance. This is of significance in searching for the depth of a layer burned below the surface.

Reference

1) R.A. Clissold and D. Stevenson. Universal Ion Implantation Manipulator. this Conference.
The ANSTO/ANU $^{36}\text{Cl}$ Accelerator Mass Spectrometry Measurement Program


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1. Introduction

An accelerator mass spectrometry program for measuring the long lived environmental radio-isotope $^{36}\text{Cl}$ has been developed jointly over the last three years by Applications of Nuclear Physics, ANSTO and the Department of Nuclear Physics, ANU. The $^{36}\text{Cl}/\text{Cl}$ measurements are carried out on the ANU 14UD accelerator. The principles of operation of the measurement system have been described elsewhere [1,2], and will not be repeated here.

Our measurements of the $^{36}\text{Cl}/\text{Cl}$ ratio have exhibited a long term reproducibility of $\sim 3\%$ and background levels of $\sim 2 \times 10^{-15}$. These performance figures can be compared with $^{36}\text{Cl}/\text{Cl}$ ratios in typical environmental samples which are in the range $\sim 20 - 1000 \times 10^{-15}$. Our AMS group continues to be the only group worldwide which makes absolute measurements of $^{36}\text{Cl}/\text{Cl}$ ratios, without normalization to a standard. That we are able to do this can be partially attributed to the fact that we measure all three Cl isotopes in the same detector system [1], instead of measuring the two stable isotopes immediately after the analysing magnet. A recent measurement of a standard prepared by Kuni Nishiizumi** yielded a $^{36}\text{Cl}/\text{Cl}$ ratio of $511(23) \times 10^{-15}$, in excellent agreement with the nominal $^{36}\text{Cl}/\text{Cl}$ ratio of $498 \times 10^{-15}$. In addition to the experimental program, we have also been involved in theoretical work aimed at furthering our knowledge and understanding of $^{36}\text{Cl}$ within the Australian environment [3,4,5].

2. The $^{36}\text{Cl}$ Measurement Program

Chlorine-36 is a naturally occurring radioactive isotope with a half life of 301 ka. Both stable chloride ($^{35,37}\text{Cl}$) and $^{36}\text{Cl}$ are natural components of recharge waters into groundwater aquifers. Furthermore, the highly soluble chloride ion has simple geochemistry. These attributes, together with variations in the $^{36}\text{Cl}$ contents of environmental chloride, make $^{36}\text{Cl}$ an ideal isotope for the study of salt and water movement within the environment.

During the past year measurements were made on approximately 300 environmental samples. These samples were associated with several measurement programs which are

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currently in progress, in collaboration with groups at CSIRO, BMR, ANSTO, ANU and other state and federal research bodies.

2.1 $^{36}$Cl Studies of Groundwaters in the Murray Basin

The Murray Basin is a very important agricultural region of Australia, which is suffering from many problems associated with soil and groundwater salinity and changing groundwater tables [6]. Salinity within the Murray Basin has been identified as Australia's most critical environmental problem and is estimated to be responsible for the loss of agricultural production within the Basin in the order of $200$ million per annum through adverse effects on plant growth and land degradation [6]. Chlorine-36 has shown itself to be very useful as a natural environmental tracer of salt and water movement within the Murray Basin, and we are involved in several studies aimed at furthering our understanding of this important area.

2.1.1 $^{36}$Cl Studies of the Murray Mallee

This is a major project aimed at studying the dynamics of the Victorian and South Australian Mallee region of the Murray Basin to the south of the Murray River. Here we are using the variations in $^{36}$Cl/Cl ratios in rainfall to indicate areas of the Basin which are providing recharge waters to the aquifer systems underlying the Basin. This study has already identified several new areas of recharge, which is important information for the management and conservation of this groundwater resource. In addition, several groundwater discharge zones exhibit unexpectedly low $^{36}$Cl/Cl ratios. A preliminary report on this work has been prepared and accepted for publication [7]. A major extension of this preliminary study of the aquifer system is currently in progress. In addition, a separate study of the discharge zones is planned, to further investigate the observed low $^{36}$Cl/Cl ratios.

2.1.2 $^{36}$Cl Studies of Murray Basin Soil Profiles

Chlorine-36 was introduced into the atmosphere during the 1950s, as a result of neutron capture on $^{35}$Cl in seawater during atmospheric nuclear explosions. The subsequent pulse of $^{36}$Cl in rainfall can be used as a tracer within groundwater systems to identify water that fell later as rain. This has prompted studies of $^{36}$Cl in several soil and groundwater depth profiles, near Naracoorte in South Australia, aimed at estimating the rate of recharge into the groundwater systems.

This study is of considerable practical significance as, due to with the high level of irrigation in this area, restrictions are placed upon the amount of groundwater that can be used for irrigation. Therefore, a detailed assessment of groundwater availability within the area, with particular emphasis on the magnitude of both diffuse and point recharge to the unconfined aquifer, is necessary for successful management of this important resource.

2.2 $^{36}$Cl Studies of Salt Lakes

We have undertaken a major study of $^{36}$Cl in Australian salt lakes with an aim to determining the origin of salts within the lakes. Preliminary results from this study indicated a greater total $^{36}$Cl budget than we had originally anticipated and we are currently investigating possible mechanisms for the production and supply of the $^{36}$Cl. In addition
to the studies of Australian salt lakes, we have made a series of measurements on salts from the Qarhan Salt Playa, China, aimed at investigating the origin of the Playa.

2.3 \textsuperscript{36}Cl Studies of the Great Artesian Basin

The Great Artesian Basin of Australia was the first groundwater basin where the decay of \textsuperscript{36}Cl within the groundwater was used as a dating tool. The aim of the present study is to use \textsuperscript{36}Cl both as a dating tool and as a natural environmental tracer to extend our knowledge of the groundwater dynamics of this major groundwater resource.

2.4 \textsuperscript{35}Cl in Rainfall

The \textsuperscript{36}Cl content and \textsuperscript{36}Cl/Cl ratio of rainwater is an important fundamental parameter in many of the studies outlined above. Therefore, a program of collecting and analysing rainfall samples in regions of interest has been initiated. Because of the very low levels of chloride in much of the rainfall, special collection, handling and analysis techniques have been devised to ensure that sample integrity is preserved.

REFERENCES


Introduction

The amorphization of silicon under ion bombardment is an area which has stimulated research for many years. Early experimental results\(^1,2\) and the models derived from them\(^3-7\) fall into two categories. The first, suited to light ion bombardment and higher substrate temperatures\(^6\), involves a critical energy density model\(^4,5\). This model consists of ion damage which builds up with ion fluence until the defect density reaches a critical value at which the lattice collapses into the amorphous phase. The second category\(^3\) is more suited to heavier ions at lower bombardment temperatures\(^6\). In this regime each ion entering the target creates an amorphous zone around its path, these zones overlap to eventually amorphize a whole region of the lattice. Although this work could often not adequately explain the microscopic details of amorphization, it did identify the parameters influential to the process. Ion energy, mass and dose all associated with defect production were found to be important to the process as was the substrate temperature.

More recent studies of keV and MeV heavy ion induced amorphization have revealed strong dose rate effects\(^8\), as well as the crucial role that defect mobility and annihilation play in inhibiting amorphous phase production\(^8,10\). Bombardment at elevated temperatures was found not only to inhibit amorphous phase production but in some cases to recrystallise previously amorphized material\(^8,11\). These results were explained in terms of defect production processes competing with defect annihilation at elevated temperatures. When defect production and annihilation rates are almost balanced the models mentioned above are not adequate to explain the observed behaviour. This paper examines some of the complexities associated with amorphization by keV ions in this regime.

Observations and Discussion

Figure 1 depicts Rutherford backscattering channeling spectra illustrating the dependence of the amorphization process on implant temperature. All the spectra are for 100 keV Xe\(^+\) implanted into <100>
silicon to a nominal dose of $2 \times 10^{15}$ ions/cm$^2$. The current was kept constant at about 0.1$\mu$A for all of the samples. As the temperature is decreased the amount of residual disorder is seen to increase. A critical temperature ($T_c$) is finally reached between 210° & 240°C where the defect annihilation cannot keep up with the production of defects at these implant conditions and an amorphous layer forms. The formation of the amorphous layer is indicated by the channeling spectrum reaching the same height as that of a randomly oriented sample.

Close to $T_c$ a strong dependence on dose rate was found to exist as shown in figure 2. The data shown is the critical temperature for amorphization for a series of $<100>$ silicon samples implanted with 100 kev Xe$^+$ to a nominal dose of $2 \times 10^{15}$ ions/cm$^2$. The dose rates for these implants were varied over two orders of magnitude resulting in the critical temperature value changing by about 100°C. The increase in $T_c$ associated with the higher currents suggests that the rate of energy deposition is important to the amorphization process. Results similar to these have also been reported for MeV irradiations$^{12}$. Work involved with the $T_c$ dependence of buried amorphous layers$^{[8]}$ suggest that secondary defects produced within collision cascades, such as divacancies help control the amorphization process; the dose rate dependence here could well be linked to such defect production and annihilation processes.

A marked dose dependence is also observed in the temperature region close to $T_c$. Figure 3 shows the amount of damage present for various doses of 80 keV Sb ions implanted into $<100>$ silicon at 240°C. As the implants were performed close to $T_c$, significant dynamic annealing is taking place. The sample suddenly amorphizes at a dose of $4 \times 10^{14}$ ions/cm$^2$ after which the amorphous layer grows in thickness with increasing dose. Lowering the temperature of the implants to 230°C resulted in the amorphous transition occurring at a lower dose. This behaviour suggests that at temperatures close to $T_c$, an amorphous layer is difficult to nucleate within a crystalline lattice. Once the layer has nucleated at defective regions, this area acts as a sink for defects, causing the amorphous region to increase in thickness with increasing dose.

**Conclusion**

From the results presented in this paper it is clear that substrate temperature, ion dose and dose rate are all important parameters in controlling the amorphization process during ion implantation. A competition between defect production and annihilation are also shown to not only control the growth of an amorphous layer but also its nucleation from crystalline material.

Further work is being undertaken to help determine the microscopic interactions occurring during amorphization. From this data it is hoped that semi-universal curves can be constructed to predict amorphization under different implant conditions.
Figure 1: <100> Si implanted with 100 keV Xe ions to a nominal dose of 2x10^15 ions/cm^2 for the implant temperatures of 210°C (---), 240°C (-----), and 270°C (----). The dotted curve is a random spectra. The dose rate for the implants was kept constant at about 0.1μA/cm^2.

Figure 2: The maximum temperature at which amorphous layers were observed (critical temperature, Tc) plotted as a function of dose rate for 100 keV Xe ions implanted into <100> Si to a nominal dose of 2x10^15 ions/cm^2.

The Special Research Centres Scheme and the Australian Research Council are thanked for financial support.

References
ZINC ION IMPLANTATION INTO <0001> SAPPHIRE

by

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Abstract

Ion implantation of 100 keV Zn$^+$ ions into the surface of <0001> single crystal sapphire slices was used to produce layers of amorphous Al$_2$O$_3$ up to approximately 90 nm thick and having an abrupt interface with the underlying single crystal. These implants were performed at liquid nitrogen temperature and at approximately 7° off axis to avoid any problems of self annealing or channeling effects. The samples were implanted with doses of 1, 3 and 6 E16 ions/cm$^2$ with approximately one third of the sample masked to retain an unimplanted area. These samples were isothermally annealed in a flowing Argon atmosphere for various times, up to 24 hours, and at various temperatures ranging from 600°C to 1190°C.

At each stage in the anneal sequence (including as implanted) the samples were analysed using Rutherford Back-Scattering and Channeling (RBS-C) and Reflection High Energy Electron Diffraction (RHEED) to study the recrystallization behaviour of the Al$_2$O$_3$, and the diffusion, segregation or surface loss of the Zinc. These techniques were used as their non-destructive nature allowed analysis of the samples at each stage of an anneal sequence.

For the lower dose cases, after the 800°C 1 hr anneal, RBS-C and RHEED showed the presence of columnar crystallites of 2—5 nm in size comprising expitaxially oriented α—Al$_2$O$_3$ as well as γ—Al$_2$O$_3$ with (111) planes parallel to the substrate (0001) surface. The well defined planar interface has not moved, indicating no epitaxial regrowth. This does not change greatly with increasing time (up to 24 hr), and is in contrast to the previous results for intrinsic amorphous Al$_2$O$_3$ [2]. Also the Zinc has become substitutional within the crystallites. The 900°C anneal shows similar behaviour but at a faster rate.

For the high dose (6 E16 ions/cm$^2$) however no recrystallization was observed at either 800°C or 900°C. The Zinc diffused within the layer and some was lost at the surface. The Zinc diffused within the layer and some was lost at the surface. The Zinc peak took on an almost flat-top profile in the amorphous layer. Diffusion coefficients were estimated. There was no evidence of phase segregation of Zinc or formation of any oxide of Zinc at the surface.

A tentative model for the recrystallization of amorphous Al$_2$O$_3$ with the lower dose Zinc impurities is proposed. Work is continuing towards TEM analysis to better determine the crystallites orientation for both phases, if possible.
References


A DATA COMPRESSION ALGORITHM FOR NUCLEAR SPECTRA

by

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Abstract

There are many applications of nuclear spectroscopy where large numbers of spectra are stored and transmitted over communication links. The total amount of data can lead to problems with storage space or transmission time. A technique is described which significantly reduces the space required to store nuclear spectra without losing any information content.

Nuclear spectra are histograms, typically of 1000 to 8192 bins, with each bin, or channel, being a simple counter of 24 or 32 bits.

Spectra normally consist of a number of roughly Gaussian peaks but the width of the peaks varies dramatically with the type of spectrometer in use. Two types of spectra which provided the impetus for the present work were gamma-ray spectra from high resolution Ge spectrometers and high-resolution alpha-particle spectra. Both these types of spectra consist of narrow peaks containing quite high numbers of counts. Between the peaks is a slowly varying continuum in which there are many fewer counts per channel.

Obviously, a large part of the data storage area for these spectra is simply used to store leading zero bits. This is also true for many other types of nuclear spectra. The information content could be considered to be just the number of bits required to store each number. Because of the slowly varying nature of the data, regions of approximately \( N \) counts/channel could be stored in \( \log_2 N \) bits with a count indicating the number of channels using that number of bits.

The algorithm comes from a simple variation on this idea.

Assume 32 bits/channel of data storage. The spectrum can be divided up into regions which have an upper bounds of \( 2^{4n} - 1 \) where \( n = 0, 1, 2, 3, 4, 5, 6, 7 \) or 8. In a region where \( n = m \) the data can be stored in \( 4m \) bits (m nibbles), provided that the data is preceded by a count and the value of \( n \). This will produce a nett compression of data provided that \( (8-m) \times (the \ count \ of \ channels) \) is greater than the overhead in storing the count and the value of \( n \).

The algorithm breaks into three phases:

a. At the beginning of the spectrum or after a region of data has been output one needs to determine the number of nibbles required for the next section of spectrum. This is done by examining the next four channels and finding the

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minimum number of nibbles required for those. After four channels if more nibbles become necessary then the overhead of a nibble region change has already been saved.

b. Then having established the number of nibbles required, one looks at successive channels until either: (i) the spectrum ends; (ii) more nibbles are required or; (iii) there is a saving by going to a smaller number of nibbles. Each of these conditions ends the current region of data and that region can be output. Conditions (i) and (ii) are straightforward. Condition (iii) requires looking ahead for enough channels to ensure that the overhead of a change of region is at least compensated for.

To allow a reduction of 1, 2, 3, 4, 5, 6, 7 or 8 nibbles from the current size then 2, 2, 2, 2, 3, 3, 5 or 9 successive channels respectively must be compatible with the reduced number of nibbles. This places the limit on the number of channels that need to be held in memory at a time. If memory space is limited only 9 channels need be remembered, the rest can be output as the searching continues. In practice storage in 7 nibbles was not used to allow the number of nibbles to be encoded in 3 bits.

c. Allowance must be made for the circumstance where the end of spectrum is reached during the first phase, i.e. the number of nibbles required must be assessed when there are less than 4 channels.

Decoding of the spectra is straightforward as each group of data is preceded by a channel count and the number of nibbles used. The encoded data is simply expanded with zeroes in the high order bits.

Results for application of the compression algorithm to some spectra acquired at the Australian Radiation Laboratory are given in Table 1.

The technique is simple, requires little computer time for encoding and decoding and has achieved an 80% reduction in data volume for those applications to which it has been applied.

Table 1

<table>
<thead>
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<th>Type of sample</th>
<th>Radionuclides</th>
<th>Size channels</th>
<th>Typical Compression ratio</th>
</tr>
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<td>Cs-137</td>
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<td>4096</td>
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<tr>
<td>Air filters</td>
<td>Am-241 and fallout</td>
<td>8000</td>
<td>0.15</td>
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</table>
An Investigation into the Minimisation of Parasitic Aberrations in the Fringe Field region of a Magnetic Quadrupole Lens.

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Abstract

Previous measurements, [1][2][3], have indicated the presence of significant duodecapole and 20-pole field components in the fringe field region of magnetic quadrupole lenses. Any duodecapole field will contribute to the fifth order aberrations of an ion optical system.

The form of the fringe field is determined primarily by the longitudinal profile of the pole tip (ie. how it is terminated), and by any saturation of the iron in the pole tip. Previous workers [3] have investigated the shaping of the pole tips to minimise contamination of the fringe field by higher order multipoles.

At Melbourne we are currently conducting a more detailed analysis of the fringe field profiles. The Melbourne quadrupole design currently uses abruptly terminated pole pieces. We intend to investigate different methods of pole termination.

The magnetic scalar potential, \( \Phi \), of a magnetic lens, which maintains quadrupole symmetry, may be written:

\[
\Phi(r, \theta, z) = \sum_{n=2,6,10...}^{\infty} A_n(r, z) \cos(n\theta + \alpha_n)
\]

where

\[
A_n(r, z) = \sum_{m=n,n+2,n+4...}^{\infty} C_{n,m}(z) r^m
\]

The condition that \( \Phi \) obey Laplace’s equation implies that:

\[
C_{n,m}(z) = \frac{-1}{m^2 - n^2} C_{n,m-2}'(z)
\]

where the primes denote differentiation with respect to \( z \). So, the \( C_{n,m} \) components are uniquely determined by the form of the \( C_{n,n} \) component.

We can also write:

\[
\Phi(r, \theta, z) = \sum_{n=2,6,10...}^{\infty} \sum_{m=n,n+2,...}^{\infty} \Phi_{n,m}(r, \theta, z)
\]

where

\[
\Phi_{n,m}(r, \theta, z) = C_{n,m}(z) r^m \cos(n\theta + \alpha_n)
\]
In the central region of a quadrupole \( \Phi \) is independent of \( r \), i.e. the \( C_{n,m} \) are constants. \( \Phi \) then reduces to the two dimensional multipole expansion:

\[
\Phi(r, \theta) = \sum_{n=2,6,10...}^{\infty} C_n r^n \cos(n\theta + \alpha_n)
\]

In the fringe field region, the \( \Phi_{2,4} \) component contributes to the third order aberrations of the optical system, while both the \( \Phi_{2,6} \) and \( \Phi_{6,6} \) components contribute to the fifth order aberrations. The \( C_{2,6} \) and \( C_{2,4} \) terms are uniquely determined by the form of the \( C_{2,2} \) component. The \( C_{6,6} \) is independent of \( C_{2,2} \), and is determined by the boundary conditions (i.e. the quadrupole geometry and excitation).

Field measurements are conducted with a Bell Hall effect probe, having a sampling area of \( \approx 1 \text{mm}^2 \). The Hall probe is rotated about the magnetic axis by a computer controlled rotary stage. Thus, measurements of \( B_r \) vs. \( \theta \) are obtained. Fourier analysis of this data gives us the \( A_n(r, z) \) coefficients for each scan. Repeating the scan at various radii, \( r \), and fitting a polynomial expansion in \( r \) to the \( A_n(r, z) \), gives us the \( C_{n,m}(z) \) coefficients. These can then be used by the computer program OXRAY[4][5] to numerically determine the aberration coefficients of the lens.

Saturation of the magnetic field at the ends of the pole pieces is expected to be a major determining factor in the form of the duodecapole \( (\Phi_{6,6}) \) component. This will be investigated by determining the form of the duodecapole component over a wide range of quadrupole excitation. From previous measurements [1][2], we expect the saturation effects to become apparent at quite low values of excitation.

There is a two fold purpose to shaping the pole ends:

1. to obtain a form of the quadrupole profile, \( \Phi_{2,2} \), which will yield a minimum contribution of the \( \Phi_{2,4} \) and \( \Phi_{2,6} \) components to the third and fifth order aberrations.

2. to minimise the duodecapole component, \( \Phi_{6,6} \), which contributes to the fifth order aberrations of the lens.

We intend to present the results of a complete analysis of the fringe field data for the current Melbourne quadrupole design. From this data it should be possible to determine the role saturation plays in determining the fringe field profiles. We also hope to present preliminary data on the influence of simple “radiusing” of the pole tips on the fringe field.

References

LEAD IMPLANTATION INTO SAPPHIRE

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ABSTRACT

Lead ions of 155 keV energy have been implanted into hot (350°C) and cold (-196°C) single crystals of α-Al₂O₃ (sapphire) with doses of 1E16 and 3E16 ions/cm². Phase formation in as-implanted samples has been investigated. The structure and properties of the amorphous implanted layer have been characterized by optical absorption spectrophotometry, electrical resistivity measurements, Reflection high energy electron diffraction, (RHEED), and Rutherford backscattering and channeling.

INTRODUCTION

Ion bombardment of ceramics produces inhomogeneous defect creation in a non-equilibrium thermodynamic situation. Ion bombarded ceramics are thus polyatomic solids with complicated structures and bonding. Modification of ceramic structures alters their mechanical, optical, electrical, and chemical properties. This paper presents experimental results on the modification of both the structure and properties (electrical, optical and chemical) of α-Al₂O₃.

EXPERIMENTAL CONDITIONS

Single crystals of c-axis (<0001> oriented) α-Al₂O₃ (sapphire) of optical quality were pre-annealed at 1400°C in oxygen for five days to ensure damage-free surfaces. The lead ion implants were carried out at -196°C and at 350°C with 155 keV ion to doses of 1E16 and 3E16 ions/cm² using the 200keV Whickham high current implanter at R.M.I.T. Four analytical techniques were used:-

i) Optical absorption measurement using a Hitachi U-3200 spectrophotometer in the wavelength range of 185 - 910 nm to examine the nature of colour centres produced by ion beam induced damage.

ii) Electrical resistivity measurements using a four-point probe method

iii) RHEED at incidence angles ranging from approximately 0° to 3°.

iv) Rutherford backscattering and channelling (RBSC) with detectors at scattering angles of 170° and 110° to determine the depth distribution of implanted Pb atoms and of ion damage to the Al and O sublattices.

RESULTS AND DISCUSSION

Optical absorption measurements in Figure 1 show the effect of implantation dose on the build up of F - centres (oxygen vacancy with two trapped electrons) and F⁺ - centres (oxygen vacancy with one trapped
electron). The absorption spectra reveals only a broad peak centred about 5.1 eV. This peak is most evident for the higher dose curves. F - centres occurring at 6.1 eV, and F+ - centres at 5.4 eV and 4.8 eV have previously been reported under neutron bombardment [1,2] and ion bombardment [2] techniques. These peaks have not been resolved in this case suggesting that any vacancies which have been produced by displacement collisions may have clustered to form lattice defects of greater complexity, hence the broader singular peak. This correlates with the build up of implantation-induced lattice damage [2,3]. For the lower doses, increasing temperature seems to decrease absorption indication some in situ annealing, whereas for higher doses, increasing temperature also increases absorption. Further studies are needed to clarify the effect of implantation temperature on optical absorption.

Electrical resistivity measurements produced similar results on all implanted and un-implanted samples. Accurate resistivity figures could not be obtained, however the sheet resistivity exceeded $1 \times 10^8$ ohm per sq.

Figures 2 and 3 show RBS measurements with $\theta = 110^\circ$ conducted on Al$_2$O$_3$ implanted at -196 C and at 350 C respectively, both at doses of $1 \times 10^16$ Pb+/cm$^2$. In Figure 2, up to a depth of 670 Å, the aligned spectrum is coincident with the random spectrum suggesting an amorphous surface layer in sapphire. [4]. This result is supported by the corresponding RHEED pattern which shows no sign of surface crystallinity. Figure 3 and its corresponding RHEED pattern both indicate that a crystalline surface is observed at higher temperatures. The width of the damage zone is larger than that in Fig. 2.

RBS and RHEED results were also obtained for Al$_2$O$_3$ implanted at -196 C and at 350 C but at doses of $3 \times 10^16$ Pb+/cm$^2$ (not shown here). For ion implants at -196 C, RBS and RHEED both suggest an amorphous surface layer, (similar to Fig. 2) but at 350 C, RBS measurements and RHEED patterns give conflicting views of the surface structure. The presence of spots in the RHEED pattern indicated some degree of single crystal alignment whereas RBS indicated an amorphous or possibly a polycrystalline surface. Further investigations are required to clarify these apparent inconsistencies.

The nature of the defects produced by ion implantation can be determined by transmission electron microscopy and thus this technique will be used as a complementary technique to those already employed. TEM studies which are to follow will be more conclusive and may clarify any discrepancies.

ACKNOWLEDGEMENTS

The author would like to thank Daryl Duckworth, Steve Johnson, Richard Thornton and Wei Zhou for their assistance with ion implantation and RBS.

REFERENCES

Fig. 1
Optical Absorption for sapphire unimplanted and implanted at various doses and temperatures with 155 keV Pb ions.

Fig. 2
RBS spectra ($\theta = 110^\circ$) for sapphire implanted with Pb ions at -196°C and a dose $1 \times 10^6/cm^2$. RBS - random and $\alpha$ aligned. Corresponding RHEED pattern at $= 0^\circ$.

Fig. 3
RBS spectra ($\theta = 110^\circ$) for sapphire implanted with Pb ions at 350°C and a dose of $1 \times 10^6/cm^2$. RBS - random and aligned. Corresponding RHEED pattern at $= 0^\circ$. 
A STUDY OF HIGH DOSE NITROGEN IMPLANTATION IN THE SURGICAL IMPLANT ALLOY, Ti-6Al-4V.

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ABSTRACT

As implanted and annealed samples of Ti-6Al-4V surgical implant alloy were studied. Rutherford Back Scattering (RBS) measurements showed an implanted surface thickness of 1100 A. During thermal annealing a laterally non-uniform layer of TiO₂ was formed. The oxide layer was found to be greater than 1600 A by simulation of RBS spectra. Scanning Electron Microscope (SEM) examination revealed that a laterally non-uniform oxide layer was formed with preferential oxidation near the grain boundaries, perhaps due to higher diffusion rates along grain boundaries.

INTRODUCTION

Titanium alloys are a group of materials whose wear properties can be extensively improved through ion implantation [1]. Nitrogen implanted Ti-6Al-4V surgical implant femoral component is receiving increased use because of its excellent wear resistant properties [2,3]. Ion implantation creates a surface layer with improved wear resistance, which is continuous with the underlying material. The aim of this work is to study systematically

(i) the metallurgical aspects of high dose nitrogen implantation in Ti-6Al-4V alloy and
(ii) to understand the physical behaviour of the implanted surface in relation to its modified microstructure.

In this paper we present a preliminary investigation based on the RBS measurements and SEM studies of as implanted and annealed samples of Ti-6Al-4V.

EXPERIMENTAL

Annealed and polished samples of polycrystalline Ti-6Al-4V alloy were implanted with nitrogen ions using the Whickham high current implanter at R.M.I.T. The samples were implanted with 80keV nitrogen ions in the dose range from 1 x 10¹⁶ to 1 x 10¹⁷ ions/cm² at room temperature with a beam current density of 1.2 mA/cm².

Post implantation annealing was performed in high purity Ar gas at 800°C for 3 hours. RBS with detectors at scattering angles of 170 and 110 was used to analyse the depth profile and nitrogen distribution in the implanted layer. SEM was used to study the topographic features of the samples.

RESULTS AND DISCUSSION

Figure 1 shows the RBS data on as implanted and virgin sample. The as implanted spectrum shows the characteristic reduction in Ti at the edge corresponding to the concentration profile of the nitrogen ions in the layer. There are no appreciable counts from nitrogen (due to a large background of Ti counts), at channel number 125. However the reduction of the Ti yield up to channel number 300, (a depth of 1100 A) is clearly seen.

The RBS spectrum of the annealed sample is shown in figure 2, along with the 'RUMP' simulation of a 1600 A thick titanium oxide layer on the annealed sample. Comparison of the spectra shows an oxide layer greater than 1600 A present on the sample. At the oxygen part of the spectrum the simulation is in good agreement with the annealed sample but not at the Ti part. This might have come from either a non-stoichiometric or non-uniform oxide on the surface. In order to examine the oxide topography, SEM analysis was performed on the samples.
Figures 3a and 3b show SEM micrographs of annealed and as implanted samples. Because of higher secondary electron emission from the oxide, the image appears to be bright wherever oxide is present. Such white regions are absent in the as implanted sample. In figure 3a the oxide formation is laterally non-uniform and appears to be preferentially located near the grain boundaries. This type of oxidation may be due to relatively faster atomic transport along grain boundaries, hence grain boundary oxidation [4].

Although oxidation of the samples was inadvertent, and work is in progress to avoid it, it is certain that oxidation is influenced by implantation.

ACKNOWLEDGEMENTS

We wish to thank Mr. P. BOND and Mr. D. DUCKWORTH for their assistance in the implantation work.

REFERENCES


Fig. 1 RBS spectra of Ti-6Al-4V (—virgin) and nitrogen implanted (o) Ti-6Al-4V alloy.

Fig. 2 RBS spectra of annealed Ti-6Al-4V with stimulation of 1600Å TiO₂ on Ti-6Al-4V.

Fig. 3a Scanning Electron Micrograph of nitrogen implanted and annealed Ti-6Al-4V. Light phase present are oxides formed during annealing.

Fig. 3b Scanning Electron Micrograph of as implanted Ti-6Al-4V.
IMPLANT INDUCED ELECTRICAL ISOLATION OF GALLIUM ARSENIDE


INTRODUCTION

It has been shown recently that GaAs can be electrically isolated using ion bombardment of species such as H, He, O and F. Development of this technology is of utmost importance in discrete microwave device technology. In an earlier paper we established that He implantation at elevated temperature is an effective way of isolation. In this paper we show that properties of the isolated layer are strongly dependent on whether the active layer is grown epitaxially or prepared by implantation and activation.

EXPERIMENTAL

Samples used for these experiments were of the structure $n^+\text{--}n$-semi insulating GaAs. The layer thicknesses of $n^+$ and $n$ regions were $0.2\mu$ and $0.6\mu$, respectively. This structure is typical for microwave applications. Samples were prepared by two different processes, namely vapour phase epitaxy (VPE) using sulphur as dopant and implant activated silicon dopant (CX).

Both types of wafers were implanted with He$^+$ ions of energy 60 Kev. The ion dose was varied over the range of $10^{11} - 10^{16}$ atoms/cm$^2$. The sample temperature during implantation was also varied over the range $25^\circ\text{C}$ to $300^\circ\text{C}$.

The sheet resistivity and mobility at room temperature were measured for all samples. For a selected batch of samples, temperature-dependent resistivity was measured over the temperature range 300K–100K.

RESULTS AND DISCUSSION

The dose dependence of resistivity for VPE and CX samples is presented in figures 1 and 2 respectively. The results at three different implant temperatures are incorporated. The dose dependence of resistivity is dramatically different in the two cases. For CX samples, a small dose of $10^{12}$/ cm$^2$ is adequate to achieve maximum isolation. In fact, the resistivity starts to drop at higher doses. For VPE samples, on the other hand, the higher the dose the better is the isolation. In terms of sample temperature during implantation, the optimum range is 100–200°C. This is about the same for both types of samples.

The mobility of these samples was measured at room temperature. The mobility of VPE samples was formed to exhibit very low values $\sim 10–100$ cm$^2$/ v·s at a $10^{16}$/cm$^2$ dose and at the optimum implant temperature. For CX samples the mobility was much higher $\sim 1000$ cm$^2$/v·s for optimum isolation conditions ($\sim 10^{12}$/cm$^2$ dose and $\sim 100^\circ\text{C}$ implant temperature). This clearly indicates that isolation is achieved by different mechanisms in the two cases. For VPE samples implantation creates complex defect clusters that not only reduce the number of carriers by trapping but also impedes the flow of carriers. A 10–100 cm$^2$/v·s mobility indicates a transport mechanism different from normal semiconducting behaviour, probably phonon-assisted hopping. On the other hand,
CX — samples clearly retain the normal semiconductor transport mechanism. The high resistivity is due to efficient removal of charge carriers through creation of deep levels in the material. We suspect the previous history of implantation and activation done on these samples must have left some defect types that are related to these trap centres.

To obtain further insight into the mechanisms, temperature-dependent resistivity measurements were performed on these samples and results for CX samples are shown in Fig 3. At lower doses, the temperature dependence is initially log-linear, going over to constant value at lower temperatures. This behaviour is characteristic of normal semiconductor behaviour and the transition indicates a change from intrinsic to impurity related conduction. The unusual low temperature required for this transition to occur indicates very low carrier concentration. At higher implant doses, we observe the breakdown of the normal semiconductor transport mechanism and the onset of hopping conduction with an activation energy of ~ 0.1 ev.

CONCLUSION

The results of electrical measurements have clearly demonstrated that different mechanisms are responsible for isolation in VPE and CX samples. For VPE samples progressive creation of damage related complex defect clusters impedes the flow of the carriers and leads to eventual breakdown of normal semiconductor transport behaviour. For CX samples, creation of deep traps effectively removes all charge carriers at fairly low doses when the defect concentration is low. Creation of deep traps appears to be related to the past history of the sample (implantation and annealing).

ACKNOWLEDGEMENT

The authors gratefully acknowledge the funding received from the Special Research Centres Scheme and Australian Research Council to undertake this research project.

REFERENCES


Figure 1. Dose and implant temperature dependence of sheet resistivity for VPE samples.

Figure 2. Dose and implant temperature dependence of Sheet resistivity of implant activated (CX) samples.

Figure 3. Variation of sheet resistivity with temperature for CX samples implanted at different doses.
FLUORINE IN AUSTRalian COALS:
A NUCLEAR PHYSICS TECHNIQUE

by

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Abstract

This report describes the apparatus, basic principles, results and suggested further work on fluorine determination in Australian coal. This work was undertaken using the $^{19}\text{F}(p,\alpha\gamma)^{16}\text{O}$ reaction with a 2.5 MeV proton beam from the 3 MV accelerator at the Australian Nuclear Science & Technology Organisation, Lucas Heights. To keep the possibility of contamination of the sample to an absolute minimum, a stainless steel UHV target chamber with differential vacuum pumping was used.

Thick pressed powdered coal samples were used for this work and the resulting gamma rays from the reaction were measured with a Ge(Li) detector mounted at 0° with respect to the incident proton beam. Results are compared with standard values and published ones. Finally suggestions for applications and further work are given.

It is shown that the technique of using the $^{19}\text{F}(p,\alpha\gamma)^{16}\text{O}$ reaction yields data which gives excellent agreement with published values of fluorine levels in pressed powder samples. These data are reproducible and do not show systematic errors.

The method used in this work is particularly useful as an UHV target chamber was used. This allowed minimum possible fluorine contamination of the samples that might otherwise occur in less clean vacuum systems. As the proton beam penetrates only a short distance into the sample, surface contamination of a sample would be easily detected. No such contamination was found in this work.

Values for fluorine in some Queensland coals have been given in this report. They range from 70 µg g$^{-1}$ to 572 µg g$^{-1}$. 
A COMPARATIVE STUDY BY SIMS AND RBS–C OF FORMATION OF 
\( \beta \)-SiC IN SILICON BY CARBON ION IMPLANTATION

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ABSTRACT

We use high dose implantation of carbon ions in (100) Si at liquid nitrogen temperature to produce amorphous alloys of Si and C. Post implantation annealing is employed to produce \( \beta \)-SiC phase. The depth profiles of C in Si are measured by SIMS and RBS techniques.

INTRODUCTION

High dose carbon ion implantation at elevated temperatures has been employed [1] to produce buried layers of \( \beta \)-SiC in single crystal silicon. In continuation with our previous study of room temperature implantation of C in Si [2], the present paper presents our results on C implantation at liquid nitrogen temperature to produce amorphous alloys of Si and C which are subsequently annealed at temperatures up to 1350°C.

EXPERIMENTAL

Single crystal wafers of Si with (100) orientation were implanted with C ions using RMIT Whickham high current implanter. Implantation parameters were: ion energy 40 keV, dose from 1E16 to 1E18/cm sq. and target held at liquid nitrogen temperature, with beam current density up to 30\( \mu \)A/cm sq. Prior to annealing up to 350°C in Ar ambient, the implanted sample was capped with a 3000 Å film of SiO\(_2\) (sputter deposited) to avoid oxidation of the surface. This layer was dissolved away in HF after annealing.

RESULTS AND DISCUSSION

Some of the RBS–C results are presented in Fig.1a which shows that an amorphous surface layer of composition 42% C peak concentration is formed after ion implantation with 40 keV C to a dose of 1E17 ions/cm sq. SIMS data in Fig.2 does not show any significant amount of mass 40 (SiC) ions indicating absence (also confirmed by TEM data not presented here) of any SiC phase in as-implanted sample. After annealing at 1350°C for 30 minutes, the RBS–C data (Fig.1b) shows considerable narrowing of the dip near silicon edge, increase in the amount of Si at the surface and movement of C atoms — all indicative of probable formation of a buried layer of SiC. Further work is in progress and the results will be presented at the conference.

REFERENCES

2. X.V. Trinh et al, Proc. 5th Australian Conf. NTA, November 1987, p.101
Fig. 1 RBS–C spectra obtained on a (100) Si sample implanted with 40 keV C ions (1E17/cm sq.), a) before annealing, b) after annealing at 1350°C, 30 minutes.

Fig. 2 SIMS depth profiles from an as-implanted sample, Dose 1E18 C/cm sq.
PHYSICAL CHARACTERISATION OF MERCURY CADMIUM TELLURIDE (HgCdTe) FILMS GROWN BY MOCVD.

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Structural properties of \( \text{Hg}_x\text{Cd}_{1-x}\text{Te} \) epitaxial layers on GaAs were measured using a variety of surface analysis techniques such as Reflectance High Energy Electron Diffraction (RHEED), Auger Electron Spectroscopy (AES), Rutherford Backscattering Spectrometry (RBS), Transmission Electron Microscopy (TEM) and Energy Dispersive X-ray analysis (EDX). These heterostructures have been of considerable interest in recent years due to their potential for opto-electronic device applications, including infra-red detectors and imaging arrays[1].

The HgCdTe epitaxial layers were grown on GaAs (100) wafers of 50mm diameter in a Cambridge Instruments Quantax 226 Metal Organic Chemical Vapour Deposition (MOCVD) system[2]. This process involves the use of dimethylcadmium, diethyltelluride and elemental mercury. Thermal growth of the MOCVD layer took place in a reactor cell in a flow of hydrogen gas. Successive layers of CdTe and HgTe were deposited. The wafer was then annealed to obtain a uniform HgCdTe layer. This was then capped by thin HgTe layer.

RBS measurements, using 2 MeV \( \text{He}^+ \) ions, were performed on the samples. Figure 1 shows a RBS spectra of HgTe/HgCdTe/GaAs both random and channelled. The results indicate epitaxial growth of HgCdTe on GaAs. No evidence of diffusion of Hg or Cd into the GaAs substrate was obtained[3].

Energy dispersive X-ray analysis and transmission electron microscopy show that both the composition and the crystallinity of the HgCdTe epitaxial layer are uniform across the wafer.

In conclusion, results from several surface analysis techniques will be discussed. Some electrical properties are also examined.
References

1: J.D. Murphy, SPIE, Vol 659 Materials Technologies for IR detectors (1986), 2

Acknowledgements

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STUDY OF THE NEAR SURFACE DAMAGE IN SILICON
CREATED BY P$_2^+$ AND P$^+$ IMPLANTATION

by

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ABSTRACT

Damage created by molecular ions is usually described approximately as the
sum of the damage created by the individual ions which constitute the molecular
species. Early studies indicated that there are molecular effects when the mass and
energy of the incident ions were in the regime to create spike effects.$^{[1,2]}$ Due to the
spike effect, one P$_2^+$ molecule implanted at LN$_2$ temperature would cause 10%–20% 
more damage than two P$^+$ ions within the energy range 20–30 keV/atom.$^{[2]}$ That
damage enhancement vanishes when the ion energy exceeds 60 keV/atom since the
defect density in the individual collision cascade is too dilute to induce spike effects.
Recently, some molecular effects were observed within the regime where spike
effects do not occur.$^{[3,4]}$. These observations were considered to relate to multiple
collision effects$^{[5]}$, in which target atoms are considered to collide with all moving
molecular fragments. The damage enhancement caused by the multiple collision
effect was relatively small since it existed only when two phosphorus ions were
moving together. In this study, a difference in the damage structure of silicon
bombarded by P$^+$ and P$_2^+$ is observed in the near surface region where the two
fragments of P$_2^+$ can be considered to travel together.

<100> Silicon was bombarded at LN$_2$ temperature with 90keV P$^+$ at a dose 
of 2x10$^{14}$/cm$^2$ and beam current 1.0$nA$. Equivalent implantation parameters were
chosen for single phosphorus ions, implantation of 180keV P$_2^+$ at a dose of
1x10$^{14}$/cm$^2$ and beam current 0.5$nA$, for comparison. Rutherford backscattering
(RBS) spectra in Fig.1 show the effect of both P$^+$ and P$_2^+$ implantation. In both
cases an amorphous layer was formed which extended from the surface to a depth
~145nm. During subsequent annealing at 550°C, the epitaxial regrowth behaviour
of the P$^+$ and P$_2^+$ implanted samples is different. This is highlighted by the RBS
spectra in Fig.1, which show that the amorphous layer created by P$^+$ implantation
crystallized from both the surface and substrate, but only substrate regrowth
occurred in the P$_2^+$ implanted samples. Like most LN$_2$ temperature implanted
samples, the time–resolved reflectivity (TRR) of P$_2^+$ implanted sample shown in
Fig.2(a) is well understood and the a/c interface position could be calculated from
the spectrum. The TRR spectrum of P$^+$ implanted sample shown in Fig.2(b) is
more complex since it corresponds to two moving a/c interfaces. Assuming that the


Fig. 1 High resolution RBS–channelling spectra of $P^+$ and $P_2^+$ implanted samples, before and after $550^\circ C$ annealing.

Fig. 2 TRR spectra of $P^+$ and $P_2^+$ implanted samples annealed at $550^\circ C$, dotted line is calculated from the model in Fig. 3.

Fig. 3 The position of rear and front $\alpha/c$ interfaces of $P^+$ implanted sample. Dotted line shows the thickness of the buried $\alpha$–Si. For $P_2^+$ implanted sample, only rear $\alpha/c$ interface moves.
annealing behavior of the rear \( x/c \) interface is exactly the same as that in \( P_2^+ \) implantation case, we can calculate the position of the front \( x/c \) interface from Fig.2(b). The results are shown in Fig.3 which shows reasonably good agreement with the experimental data. The TRR spectrum from the \( P^+ \) implanted sample, annealed at 550°C for 150 sec, indicated that there was a buried amorphous layer \(~55\) nm thick and a single crystalline silicon layer \(~25\) nm thick on the top. This result is in consistent with the RBS measurement (Fig.1 (b)) and cross-sectional TEM observations.

The damage structure near the surface of the samples implanted by \( P^+ \) and \( P_2^+ \) was also studied by Reflection High Energy Electron Diffraction (RHEED) observations. Both samples showed amorphous rings, although recrystallization from the surface is seen in the \( P^+ \) implanted sample. The RHEED results imply that there could be single crystalline islands immersed in the surface amorphous region. During heating, these islands would grow transversally and combine each other to form a continuous crystalline layer which could be the seed for the epitaxial growth from the surface.

The difference in the damage structure near the surface region created by \( P^+ \) and \( P_2^+ \) implantation was observed, it indicates that the multiple collision effect could be responsible for the removal of the crystalline islands seen for atomic ion implantation. More detailed investigations for various ion energy, dose, dose rate are in progress.

REFERENCE

TRACE ELEMENT DISTRIBUTION IN NATIVE GOLD

by


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Abstract

The HIAF proton microprobe has been used to study the trace element distribution in gold from alluvial sources and ore samples, to investigate the possibility of identifying characteristic distributions associated with different types of geological environments. Preliminary results look promising but improvements in the detection limits are required for a clearer picture.

Introduction

The presence of trace elements in native gold from both placer and bedrock deposits was demonstrated by Warren and Thomson [1] by spectrographic techniques. They concluded that observed trace element variations are controlled mainly by the characteristics of the metallogenic provinces rather than the type of deposit. Further corroborating data were obtained [2-4], raising the possibility of using trace elements in alluvial gold grains to establish their provenance through genetic associations and to identify multiple sources. However, the early spectrographic studies were beset by problems of contamination due e.g. to mineral inclusions. Multiple sources could not possibly be identified because a large number of grains were required for each analysis. Later electron probe studies avoided these problems, enabling one to distinguish traces in solid solution, but the method does not have the required sensitivity (>100 ppm). In the following, we report on a preliminary study of the use of the proton microprobe in trace element analysis of gold from a number of sources.

Method

Alluvial samples were obtained from six prospects in the Elliott Bay area in the southwest of Tasmania, and from three other areas of different geological environments, namely ultramafic (19 Mile Creek), dolomite (Jane River) and skarns (Lea River). Ore samples from two massive sulfide deposits (Rosebery and Que River) and a Au-Cu-Bi mineralization (Warrego, N.T.) were also included in the study. The alluvial gold grains are typically less than 0.5 mm in size. These are set in araldite, ten grains to a single mounting, and arranged so that the probability of cross contamination is minimized during polishing. Bedrock samples are prepared by simply polishing the rock, after mounting in araldite for convenience of handling. A thin coating of carbon was applied to allow beam current integration and prevent charge build-up on the specimen during analysis.

The analysis was carried out on the HIAF proton microprobe. Most data were obtained with a 20 micron beam of 3 MeV protons, and with a 100 micron thick Al absorber in front of the Si(Li) detector. On alluvial samples analyses were carried out at the centre of the grain whenever possible, to avoid possible effects of leaching or accretion at the grain edges during
fluvial transport. Several traverses across selected grains were carried out to examine this effect. A total of 128 spot analysis (15 to 30 minutes each) representing 69 grains were carried out. In addition "pure" Au and Ag standards were also analyzed.

Results

Figure 1 shows a typical spectrum obtained from the gold grains, showing the dominant Au and Ag peaks. The Au L lines presented a problem in the detection of many elements. Pb and Bi can only be observed reliably through their L gamma lines. The low energy tail of the Au lines severely limits the detection sensitivity for elements adjacent below e.g. Pt, Os and Ir. This problem is compounded by the fact that there is a time dependence on the intensity of the "tail"; it grows steadily from the time the Si(Li) detector is turned on. Hg, with peaks higher in energy than Au, can be fitted unambiguously. The tails of the Ag K lines interfere with the detection of Pd. More significantly, Pd is also interfered with by the pile-up peaks from Au, as can be seen in fig. 1. Au ranges from 69 to 98 weight percent, and Ag from 1.4 to 30%. Elements observed include Hg, Bi and Cu at about 0.1% concentrations, and other base metals (Fe, Pb and Zn) at <0.1%. Traces of Pd, As and Mn were also measured. The Mn numbers are unreliable because of the high absorption by the filter used. Examples of the results obtained for Cu and Hg are shown in the correlation diagrams in figures 2 and 3.

Discussion

The most obvious relationship observed is that between Au and Ag, especially in the alluvial samples from the Elliott Bay Area. Virtually complete solid solution between Au and Ag exists between the limits of Au observed (68-100%), which is an indication that any trace elements in the grains are not present in concentrations significant enough to perturb the linear Au/Ag relation. Hg is present in the Elliott Bay samples at significant levels (0.27-0.5%), and shows a positive correlation with Au content. The Cu content ranges from 100 to 3400 ppm, also showing a positive correlation with Au (fig. 2). Such correlation has been observed previously [5]. The other base metals Fe, Zn show a generally positive correlation with Au, but Pb does not show any apparent trend. A positive correlation between Ag and Pb was observed, although with the severe interference problem mentioned above the results should be viewed with caution at the present stage.

Alluvial samples from the other areas are distinguished from each other and from the Elliott Bay samples by their Cu, Hg, Pd and Bi concentrations. The 19 Mile Creek samples, associated with an ultramafic complex, show anomalous Cu concentration (0.2-0.5%). An exceptionally high content of Hg was observed for the Jane River samples. The Lea River samples show high contents of Bi (0.2%) and Pd (0.13%). Grains from this area show zoning with increasing Au, Fe, Cu and Hg towards the rim.

Gold grains in the ore samples from Warrego are characterized by high levels of Au (>98%) and corresponding anomalous concentrations of Bi, Fe, Cu and Zn. The high level of Bi is to be expected since the ore contains about 0.13% Bi.

Several ore types from Rosebery demonstrate that the composition of the gold grains varies substantially, although all are characterized by high Hg (fig. 3) (0.3-0.5%) and Fe (0.08-0.6%) and lower Au and Cu. The Que River deposit is characterized by high Hg (0.9%) and anomalous Zn (2.4%) content. The latter is consistent with the inferred close stratigraphic relationship between Au and Zn in the deposit, and may possibly represent the presence of an intermediate Au-Zn compound.

In summary, the results of this study demonstrated that there are characteristic differences between different deposit types and styles of
mineralization; between deposits of the same type (Rosebery and Que River) and between different mineralogical associations within a particular deposit. Alluvial samples show decreasing Ag content with distance from the source.

The present results were obtained within the present limitations on the sensitivity of detection of certain trace elements due to interference problems. Improvements in the detection sensitivities can be achieved with better resolution detectors with less "tail" problem, or ideally with a wavelength dispersive spectrometer. The present results however demonstrate the diagnostic potential of trace element composition in gold exploration.

References


Fig. 1 A typical spectrum obtained from the gold grain, showing the dominant Au and Ag peaks, and the fitted spectrum with the pile-up peaks indicated.

Fig. 2 Correlation diagram for Cu vs Au for the samples studied.

Fig. 3 Correlation diagram for Hg vs Au for the samples studied.
The general purpose nuclear reaction code GENUREAC was developed as a tool for the analysis of nuclear reactions of the type \( A(a,b)B \) with \( Q > 0 \), where \( a \) is the incoming particle at incident angle \( \alpha \), \( b \) is the outgoing particle at exit angle \( \beta \), and \( A \) and \( B \) are the initial and final reaction nuclides. The special cases of Rutherford backscattering (RBS) and elastic recoil analysis (ERA) may be treated by setting \( Q \) equal to zero, and \( a=b \) for RBS and a heavier than \( b \) for ERA. In particular, the code was designed to include straggling and multiple scattering effects and is capable of predicting yield spectra for most ions in any matrix, and for depth profiling.

The target thickness is divided into \( N_x \) slices and the total yield at each energy is obtained by summing the yield calculated for each thin slice. The code evaluates the kinematics at depth \( x \) in the target according to the equations of Ref.[1], and the stopping powers for the input particle and mean foil energy from the theory of Ref.[2]. The depth profile for each slice \( x \) and the cross-section at each energy \( E \) and angle \( \phi=\alpha+\beta \) are evaluated in the two FORTRAN functions FND(X) and XSECT(E,PHI) respectively. These vary with each material used and must be supplied to the code by the user.

The unbroadened theoretical yield spectrum is then calculated, followed by the energy straggling through both the absorber foil, if present, and the target using either the Bohr, Symon or Payne-Tschaler equations of Ref.[3] depending on the energy loss being <20%, <50% or >50%. The target and foil roughness is incorporated. For the target, where it is assumed that the bumps on the surface produce an energy spread, this is added in quadrature to the energy straggling term. The Symon straggling may be evaluated from either the full integral or an approximation using one of three different values for the mean energy in a ratio of stopping powers approximation to the full integral [4] (option set by user).

The geometric straggling in the target due to finite beam width and detector slit size is calculated, as is the multiple scattering component in both the target (for particles \( a \) and \( b \)) and the foil (\( b \) only). All straggling components plus the detector resolution are then added in quadrature to give the total straggling.

The experimental data is mapped into 500 channels for comparison with theory using a 3SD Gaussian for each channel, and may be renormalised to align the front edge and fit the total area (with the experimental value) if required.
A plotting option (set by the user) allows output of all or any of the following graphs:
1) yield vs outgoing particle energy for both theory and experiment,
2) scattering cross-section of the reaction vs incident particle energy,
3) depth distribution of yield,
4) yield vs incident particle energy for series of incident ion angles.

Some examples of nuclear reactions A(a,b)B:
(i) NR : $^{18}_{0}\text{O}(p,\alpha)^{13}_{5}\text{N}$ reaction with $Q=3.98$ MeV, $a=858$ keV proton incident at an angle of 40°, $b=\alpha$ particle exiting at an angle of 40°, thickness of tantalum oxide target = 2296 Angstrom.

Figure 1 a): Yield vs alpha energy (keV) for tantalum oxide target. The theoretical and scaled experimental yields are given in counts per channel as a function of alpha energy in keV with the solid line being the fit to the experimental data using GENUREAC.

b): Scattering cross-section of oxygen vs proton energy (MeV)

(ii) RBS and proton straggling in Mylar foil of 23½µm thickness with $Q = 0$, $a=b=\text{proton}$. Protons backscatter off a thin (200 Angstrom) gold layer and then pass through the Mylar absorber foil.

Figure 2 a): Yield vs proton energy (MeV) for proton straggling in the Mylar foil.
The results are plotted for 3 different energy losses ($\Delta E/E$) to show the broadening of the yield spectrum with increasing incident particle energy and fixed foil thickness, and for 3 different straggling options (Bohr, Symon approximation with $\langle E \rangle = \frac{1}{2}(E_1+E_2)$ and Symon full integral) for comparison. It can readily be seen that the spectrum broadens with increasing input particle energy, and that the Symon full integral gives the best fit to experiment if ($\Delta E/E > 50\%$).

b): Ratio of total to Bohr straggling vs fractional proton energy loss to show that the ratio can exceed unity.

Curves 1 and 2 are for the Symon approximation straggling option with mean energy set to $E_1$ and $\langle E \rangle$ respectively, while curve 3 is for the Symon full integral. Again the full integral straggling option gives the best fit to experiment.

c): Ratio of total to Bohr straggling vs proton energy (MeV) for a series of foil thicknesses to show the approach to unity as a function of increasing input particle energy.

In summary, the code GENUREAC is a user friendly nuclear reaction analysis (NRA) code capable of handling RBS and ERA as specific cases. The code includes energy straggling and multiple scattering effects, and so accurate depth profiles can be extracted from experimental spectra.

References
Figure 2a)  
PROTON STRAGGLING IN MYLAR

23 µm  
1.991 MeV  
(ΔE/E) = 27%

YIELD (ARBITRARY UNITS)

1.3 1.4 1.5 1.6

Figure 2b)  
TOTAL STRAGGLING/BOHR (O / Q0)

1.0 1.2 1.4 1.6 1.8 2.0

FRACTIONAL PROTON ENERGY LOSS (ΔE/E)

Figure 2c)  
PROTON STRAGGLING IN MYLAR

5 10 20 50 µm

PROTON ENERGY (MeV)

1.210 MeV  
(ΔE/E) = 80%

Bohr  
Symon (E)  
Symon

Expt

PROTON ENERGY (MeV)
DESIGN AND FABRICATION OF A SEMICONDUCTOR PARTICLE DETECTOR

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Abstract

Radiotherapy applications often require accurate measurements of dose rate and intensity across a target area. In the majority of cases an ionization chamber is used to gather information about the emission. However, a solid state detector such as a silicon junction diode has a number of advantages over an ionization chamber. This article describes the fabrication and operation of a silicon pn junction diode suitable for relative dosimetry applications.

In biomedical applications, it is critical that the intensity and uniformity of the radiating field accurately determined. This is often done using an ionization chamber, which can be time consuming and expensive. A silicon pn junction is cheap to produce, and can simplify data collection by being incorporated in an array formation of a number of devices. This would allow information across a large area of the field to be obtained in one measurement.

There are several advantages in using a semiconductor detector compared to an ionization chamber (Tsoulfanidis, 1983). These include:

a. physically smaller device (allowing greater spatial resolution)
b. lower mean ionization energy
c. much higher stopping powers
d. superior energy resolution
e. lower fabrication cost

For the particular problem of mapping the intensity and uniformity of a high energy photon field considered here, the size and cost of the device were two important considerations. An ionization chamber may be used for absolute calibration of the semiconductor detector, allowing subsequent use of the relative dosimetry measurements of the detector.

When an ionizing photon (or particle) passes through a pn junction diode, a number of electron-hole pairs are generated. The charge generated is collected at the p and n terminals by virtue of the electric field existing in the depletion region associated with the pn junction. The charges which contribute to the signal are the minority carriers which are collected from within the effective ionization volume of the diode. This includes the depletion width plus the semiconductor p and n regions which lie within a minority carrier diffusion length from the depletion region.
The silicon pn junction diode detector was fabricated using standard silicon processing techniques. For the base material p-type silicon was chosen as the low doped substrate as the detector response of this material to high energy photons is more linear compared to a detector fabricated using an n-type substrate (Rikner, 1983). In this particular case, only photons with energy higher than 0.1 MeV were to be detected, so the junction did not have to be located near the surface. This is in fact an advantage as the influence of lower energy radiation, including optical radiation, is reduced or eliminated.

The starting material was Boron doped 17-23 ohm.cm Silicon. A field oxide was grown and patterned to act as a mask for the phosphorus pre-deposition and drive-in. The oxide layer grown during the phosphorus drive-in was then patterned to expose the central contact area. Aluminium contacts to both the top and bottom of the wafer were then deposited by evaporation. The bottom contact was protected while the top contact was patterned. The Al contacts were sintered, and individual diodes were cleaved and bonded to a transistor header with wire bonds and epoxy.

Individual diodes were tested on a curve tracer and showed excellent I-V characteristics, with breakdown voltages exceeding 350V, and <1μA leakage current at this voltage. One of the major disadvantages of using semiconductor detectors is the possibility of sensitivity loss due to radiation damage. No degradation was observed or expected here, as the total radiation flux was low.

The fabricated diode produced an output pulse of the same shape, but with slightly better amplitudes than a commercial detector (Ortec CA 14-25-100). A Phillips model SL-25 linear accelerator was used for the irradiation of several diodes with 6 MeV photons at various doses. The diodes gave a linear response over the range of dose, indicating they may readily be used for relative dosimetry measurements.

References


Accelerator Mass Spectrometry

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1. Introduction

Accelerator mass spectrometry (AMS) is a technique for measuring very small isotopic ratios. Typically, AMS is sensitive down to a few parts in $10^{15}$ with an accuracy of the order of 1 percent. Although AMS is widely used for analysis of long lived radioisotopes, it can also be used to study stable isotopes, as it does not depend upon the detection of radioactive decay.

The primary advantages of AMS over conventional mass spectrometry are the rejection of molecular ions and the separation of isobars, allowing clean identification of the isotope of interest. Further, AMS requires sample sizes which are only about a milligramme. For many long lived radioisotopes this is orders of magnitude smaller that the sample size used in decay counting. For example, in analysing $^{36}$Cl the AMS system discriminates against the mass-36 molecular ions $^{12}$C$_3$ and $^{18}$O$_2$, as well as the isobar $^{36}$S, each of which may be several orders of magnitude more intense than the $^{36}$Cl of interest. In addition, AMS can measure a $^{36}$Cl/Cl ratio of $10^{-13}$ from a 5 mg chloride sample to an accuracy of 10% in 1 hour. To measure this ratio by decay counting of the same sample would take 5 years ($t_{1/2}$=301 ka) in an ideal counter with no background and 100% efficiency; in practice, such a measurement would probably be impossible.

The first mass spectrometry measurements of $^{14}$C using a tandem accelerator were carried out in 1977 by groups at the University of Rochester [1] and McMaster University [2]. During the 12 years since this first use of the technique the range of isotopes that can be measured by AMS has been expanded to include $^{10}$Be ($t_{1/2}$ = 1.6 Ma), $^{26}$Al ($t_{1/2}$ = 720 ka), $^{36}$Cl ($t_{1/2}$ = 301 ka), $^{41}$Ca ($t_{1/2}$ = 103 ka) and $^{129}$I ($t_{1/2}$ = 16 Ma) as well as stable isotopic ratios and trace element analysis of B, Cr, P, Ge, As, Pt, Ir, Au and Os.

2. Principles of Accelerator Mass Spectrometry

Accelerator mass spectrometry relies upon a number of features of the tandem accelerator to attain its sensitivity for isotopic ratios down to $\sim 1 : 10^{15}$. The samples are put into the accelerator ion source in solid form and negative ions are produced by sputtering the sample with cesium. This sputtering process can in some cases provide considerable discrimination between isobars. For example, $^{14}$C readily forms a negative ion, whereas $^{14}$N$^-$ is unstable. These negative ions then undergo an initial acceleration to an energy which is typically between 20 and 200 keV, depending upon the details of the accelerator system being used. The beam is then put through an initial magnetic mass analysis to

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select the ions of interest.

After the initial mass analysis the beam is injected into the accelerator and accelerated towards the high voltage terminal, which is typically held at a potential of between 2 MV and 15 MV, depending upon the element of interest and the accelerator. At the high voltage terminal the ions in the beam are stripped of their outer electrons by passing them through either a thin carbon foil or a gas cell. It is this stripping process which breaks up the molecular ions within the beam. This breaking of the molecular ions is a very important step in the mass spectrometry system, as without it the number of molecular ions in the beam would be many orders of magnitude greater than the number of ions of interest, making mass spectrometry of the atomic ions impossible.

The stripping process leaves the ions in a positive charge state so they undergo a further acceleration away from the high voltage terminal. Following this acceleration the beam is put through another magnetic mass analysis, which separates ions with the mass of interest from the molecular fragments. In addition, the use of an electrostatic analyser or Wien filter is common to remove ions with the same magnetic rigidity as the primary beam, but with different energy, which are produced through interactions with the residual gas in the accelerator tube.

Finally, the ions are put into a heavy-ion detector which identifies the ions of interest and discriminates against isobars and other background events. This detector is typically a gas ionization chamber, with a segmented electrode plane to allow several measurements of the ion's energy loss, or a time-of-flight detector. As with the breaking of the molecular ions, the heavy ion detector plays a very important role in the system, since the count rate of the isobars and other unwanted events can be many orders of magnitude greater than the count rate of the ion of interest.

3. Applications of Accelerator Mass Spectrometry

The first application of accelerator mass spectrometry was the measurement of $^{14}$C in archaeological and environmental samples. AMS has made an enormous impact in this field, due primarily to a reduction in sample size by about a factor of 1000, which has enabled much greater selectivity in the choice of the sample to be analysed [3,4]. This selectivity has enabled the dating of objects that were either too small or too rare or valuable to be analysed by decay counting. An example of the former is the dating of individual seed grains from archaeological sites, while an example of the latter is the much publicized dating of the Turin Shroud [5].

The use of $^{14}$C and other naturally occurring long lived isotopes is becoming increasingly important with the current concern about global climatic change due to the greenhouse effect and the depletion of the atmospheric ozone layer. A vast body of data on the Earth's climatic history is stored in the isotopic composition of environmental features ranging from manganese nodules and corals to glacial moraines, sediments and polar ice. Accelerator mass spectrometry is playing a major role in the recovery of this environmental history.

Chlorine-36 is highly soluble in water and has a long half life of 301 ka. The combination of these attributes makes it an ideal isotope for the study of hydrologic systems. Applications in this field include the dating of groundwater [6] and the use of $^{36}$Cl as a
natural tracer to study the movement of salt and water within the environment [7]. These applications are particularly important within the Australian context both because of our major environmental problems of soil salinity, groundwater salinity and changing water tables and because of the reliance of much of arid Australia on groundwater resources for both agricultural and domestic water supplies.

Beryllium-10 bonds strongly to clays and soils and is, therefore, very useful as a tracer of soil movement. As such, it has found a wide range of applications in studies of soil erosion and sedimentation [8]. Beryllium-10 has also been used as a tracer of the surface sediment layer in the study of island arc volcanos [9] and of manganese nodules which hold a record of conditions in the deep ocean over periods of many millions of years [10].

Iodine-129 has received less attention than the other isotopes mentioned above. This has largely been because of the lack of facilities which have been able to measure \(^{129}\text{I}\), and to date most studies have been carried out by the Rochester group [11] and June Fabryka-Martin [12,13]. Applications include the study of hydrology [12,13], marine sediments [14] and oil deposits [15]. There has also been a considerable amount of work done on studies of the isotope aluminium-26 [16].

In addition to the radioactive isotopes discussed above, accelerator mass spectrometry has been used for trace element analysis of Pt, Ir, Au and Os in environmental samples [17,18] and a wide range of impurities in semiconductor samples [19]. Furthermore, accelerator mass spectrometry has enormous potential in fields requiring artificial tracers of the elements C, Al, Si and Cl [20].

4. Accelerator Mass Spectrometry in Australia

Over the past 3 years an AMS system based upon the 14UD accelerator at the Australian National University has been developed by ANU and ANSTO as a joint project. The system was first developed for the measurement of \(^{36}\text{Cl}\) [21]. We now measure this isotope routinely and have an extensive program of environmental studies of \(^{36}\text{Cl}\) with a particular emphasis on the movement of salt and water within the Australian environment, in collaboration with BMR, CSIRO and other state and federal research bodies. We have also developed a measurement system for \(^{14}\text{C}\) which has started to make routine measurements during the last year. In addition, the capabilities to measure other isotopes, including \(^{10}\text{Be}\) and \(^{129}\text{I}\), are currently being developed. Our accelerator mass spectrometry system for \(^{36}\text{Cl}\) and \(^{14}\text{C}\) samples has a capacity of approximately 300 samples per year. Our research and development programs for \(^{36}\text{Cl}\), \(^{14}\text{C}\) and \(^{10}\text{Be}\) are described in separate contributions to this conference (references [22], [23] and [24], respectively).

ANSTO has recently purchased a FN tandem accelerator from the University of Rutgers. This accelerator has been transported from America and is currently being installed at Lucas Heights. It is planned to use the accelerator for a wide range of nuclear physics applications including an extensive program of accelerator mass spectrometry. The initial aim of the AMS program will be to provide a high quality commercial \(^{14}\text{C}\) dating service. The program will then be expanded to include the isotope \(^{10}\text{Be}\), which has important applications in the study of soil erosion. The expansion of the facility to other radioactive and stable isotopes will proceed, as resources allow, according to demand.
REFERENCES

PIXE and ART

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ABSTRACT

Extracting the proton beam from the Lucas Heights Van de Graaff accelerator has enabled us to apply the Proton Induced X-ray Emission (PIXE) technique to fragile or large objects which could not be analysed in vacuum.

We are using this method to characterise pigments used in early Australian paintings. Pictures to be analysed are placed at the end of the beam line and irradiated in air. A Si(Li) detector in close proximity to the sample detects the emitted X-rays which are then sorted and displayed as a characteristic spectrum. By keeping the target current low and analysing for short periods of less than 2 minutes, sample damage is not observed.

Methods of analysis are discussed with some results of preliminary measurements on well provenanced Australian paintings.

1. INTRODUCTION

The Bicentennial year witnessed a large growth of interest in the history of Australia and it's culture. In Sydney alone the past 12 months has seen the opening of the Powerhouse, the soon to be opened Maritime Museum and large extensions to the Australian Museum and the Art Gallery of NSW. This growth needs a solid scientific base and support. Each institute has a conservation laboratory whose charter is the preservation and restoration of the objects of our heritage. Whether it be a silver and gold inkstand from the goldrush period or a feathered headdress from the Islands, each object can have its unique problems in understanding the material problems associated with its preservation. In this context an important step in many investigations is the chemical analysis of the object, for example the composition of papers, glass, leather, coinage or metal alloys. This information can provide details of provenance as well as the technology employed in their manufacture. Because of the value of museum
collections, sampling is undesirable. Thus non-destructive methods of analysis become important for such studies.

A similar picture exists with respect to paintings and other art works. Non-destructive analysis of the pigments used in the production of a picture can provide guidelines for its conservation and restoration and also clues to its authenticity.

2. ANALYSIS OF PIGMENTS

Considerable information on an artist's technique can be gained by scientific analysis of the pigments of a painting. Visual inspection under different light sources can be revealing. For example, an ultra violet light examination may show evidence of retouching through contrasting fluorescence of the retouched areas. Infra red examination may show underdrawings or preliminary sketches. Further testing may be non-destructive or may involve the removal of microscopically small samples from the work, depending on the information required.

In this paper we discuss the application of external beam Proton Induced X-ray Emission (PIXE) analysis to in-situ analysis of paintings as a means of chemically characterising the pigments used in the production of the picture.

3. PIXE ANALYSIS

For pigment analysis we require a technique which has good sensitivity and preferably is non-destructive. When allied with a short measurement time and the ability to analyse small areas, PIXE becomes a method which has potential for pigment characterisation. It has been applied by Tuurnala et al. [1,2] and also Bird [3] from Lucas Heights has studied several commercially available pigments with a view to analysing water colours.

Figure 1 shows the external beam facility used at Lucas Heights for painting analysis. This has been fully described by Garton and Clayton [4]. In brief, the proton beam is extracted into air through a thin (12 micrometres) Kapton foil. The beam will have a range in air of some 10 cms. Large objects such as paintings can be positioned close to the foil and a standard PIXE analysis performed. Facilities are available to blow cold air or helium onto the exit foil and target. This, with the use of small beam currents, typically less than 5 nA, prevent heat damage to the samples.
4. DISCUSSION

To date, our work has concentrated on a pilot study on Australian artists. We aim to develop a database of information on techniques and pigments used by early Impressionists. An example of such data is shown in figures 2 and 3. Figure 2 shows the spectrum of lead white from the Tom Roberts painting "Washing Day Kallista". There is also a large amount of Zinc present. This can be contrasted with figure 3. This is the spectrum from a lead white pigment in a 17th century European landscape. In this figure there is no zinc. This change came about because of changes in the manufacturing process during the 18th and 19th Centuries as detailed by Fleming[5].

5. CONCLUSION

PIXE can be utilised in study of artists pigments. Its multielemental capability and short analysis time make it a useful tool for rapid survey of a picture. If necessary, information gained with this technique could be augmented by sampling from selected spots for further analyses.

Acknowledgement

We thank the Art Gallery of NSW who supplied the Tom Roberts painting discussed here.

References


Figure 1  The external beam system.

Figure 2  Lead white spectrum from Tom Roberts picture.

Figure 3  Lead white spectrum from 17th century landscape.
PROGRESS ON THE CSIRO-AMS FACILITY

by

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Abstract

An AMS (Accelerator Mass Spectrometry) system is being constructed at the CSIRO HIAF laboratory at North Ryde. The system is designed to enable cosmogenic isotope based chronology, and eventually will be developed to enable ultra trace measurements in the mineralogical samples.

Introduction

The HIAF laboratory was commissioned in 1983 with the mission of developing applications of accelerator based analytical methods in minerals research in aid of the minerals industry [1]. An essential requirement in order to achieve this is the development of the proton microprobe, completed in 1985, to enable applications of ion beam analysis on mineralogical samples, the fundamental constituent of geological samples [2,3]. Recognizing the significance of accelerator mass spectrometry not only in geochronology, but also in the in-situ detection of ultra traces (< 1 ppm) [4], an AMS system was also planned at HIAF. The following reports on the progress of the construction.

Design Consideration

The research interest of the Division lies mainly in the applications of 10Be and 36Cl dating to problems relevant to exploration and mining. The scope of published work on applications of 10Be [5], ranging from soil transport and erosion studies, rock exposure ages on to petroleum dating offers exciting prospects in research of ore and petroleum genesis as aids in exploration. Similarly, the information from 36Cl as tracers and chronometer of groundwater can be an important supplement in hydrogeochemical methods exploration, and water management in arid areas such as typically found around mines. In addition to the direct requirements in the minerals industry, HIAF in general is accessible by other areas of research, and thus the AMS facility will also be available for 14C dating.

Of more direct interest to minerals research is the AMS capability to detect very low levels (ppb) of trace elements. Thus far the method has been applied with submillimeter sputtering beam, which is not adequate for most measurements requiring monomineralic analysis [4]. A microprobing sputter source must be developed for successful applications of AMS as trace analyzer.

The HIAF-AMS project will proceed in two stages, with the first resulting in standard AMS capability, viz. 10Be, 14C and 36Cl dating on "bulk" samples. The second stage involves the development of the microprobing Cs sputter source. The AMS system incorporates the existing beam transport system at HIAF, with the addition of a beam line for the detection system, and a separate injector.
Progress

A schematic of the HIAF beam transport system, including the AMS system, is shown in fig. 1. The AMS injector system is based on a double focusing, 90 degree analyzing magnet with a radius of 30 cm and beam product \((ME/q^2)\) of 7, and a momentum resolution of 6500. A 90 deg electrostatic analyzer will be installed in the future, to improve the rejection ratio of adjacent isotopes.

The beam from the modified HICONEX sputter source is focussed by an einzel lens to form a waist at the magnet's object point. The beam is further transported to the original low energy cup through an opening in the "old" injector by means of another einzel lens. Distances are chosen to match the emittance of the AMS injector system to the acceptance of the original system. The "cone" of the HICONEX source, which holds the sample to be sputtered, is modified to permit focussing of the Cs beam at the upstream side, and into a smaller area. The sample is loaded into a "well" in the cone (1 mm diam and 1 mm deep). This modifications reduces the amount of sample required and improves the source emittance. Analyzed beams of \(^{12}\text{C}\) and \(^{28}\text{Si}\) have been obtained with intensities at least comparable to that from an unmodified cone (5 \(\mu\)A and 1 \(\mu\)A respectively). Initial tests to produce BeO\(^{+}\) from oxide powder gave poor yield (\(<100 \text{nA}\)). The sample was prepared from natural BeO mixed with fairly coarse Cu powder (100 mesh). Further tests will be conducted with finer powder to improve the heat and electrical conductivity of the mixture. These tests were conducted with the source in the old injector. In the new injector, this has not been reproduced, due most likely to alignment problems of the magnet. This problem is currently being rectified. The magnet box is electrically insulated from the rest of the beam line to permit "bouncing", i.e. energy modulation of the incident beam to maintain the same magnetic rigidity.

The beam passes through the usual two stages of acceleration with gas stripping at the terminal. There is currently no terminal pumping, which some believe to be essential for reducing the effect of fractionation through the accelerator, but there is provision for its installation. After the acceleration the beam is analyzed by the 90 deg analyzing magnet (b.p. 16). The more abundant isotopes will be measured in the Faraday cup at the image point of this magnet. A multi Faraday cup system with 4 independently adjustable cups has been designed and will be implemented as part of the second stage development. This will allow detection of the abundant isotopes without the need to alter the analyzing magnet setting. In the present system, the analyzing magnet will have to be adjusted for measurements of each isotope. To facilitate such switching, a computer controlled beam transport is being implemented, which will permit automation of the operation.

The rare isotope will be further transported into the detector beam line through a 22.5 deflection using switching magnet. A 12 deg electrostatic spherical analyzer with 4 m radius and 11 mm gap feeds the beam into a gas proportional counter. The counter is a conventional counter with a Frisch grid, operated with isobutane at pressures in the 10-40 mbar range. The anode is segmented into three parts, each 5 cm long. The window is 6 mm in diameter, made out of multiple layers of thin films of VYNS, capable of withstanding up to 200 mbar pressure difference. Tests with 6 MeV beams of \(^{12}\text{C}\), \(^{16}\text{O}\) and \(^{28}\text{Si}\) \((q=3+)\) have been carried out and resolutions of 120, 150 and 220 kev were obtained for the dE anode signal. The results are consistent with a 100 keV electronic resolution, with the remainder attributable in terms of the loss distribution width. The obtained
resolution is more than adequate to separate the C isotopes. At 6 MeV, the expected separation for $^{14}$C from the spurious $^{13}$C and $^{12}$C (injected as CH$^-$ or CH$_2$) is about 200 and 400 keV respectively. For $^{10}$Be measurements such resolution is more than adequate, but for $^{36}$Cl it may be inadequate to discriminate against $^{36}$S. A time of flight system is being considered as an alternative detection system.

Summary

The first stage of the CSIRO-AMS facility is virtually completed and is undergoing tests. It will enable $^{10}$Be, $^{14}$C and possibly $^{36}$Cl measurements. Further development will be directed towards the heavy isotope detection and a microprobing sputter ion source for detection of ultra tracts in mineralogical research.

References


Fig. 1 A schematic of the HIAF beam transport system, including the AMS System
ACCELERATOR MASS SPECTROMETRY OF BERYLLIUM–10

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1. Introduction

Beryllium–10 is a naturally-occurring radio-isotope with a half-life of 1.6 million years which is produced in the atmosphere by cosmic ray spallation of nitrogen and oxygen nuclei. Its half-life is ideally suited to its use as a tracer in erosion and sedimentation studies in the Australian context, but this same half-life makes it very difficult to detect by radioactive decay counting. However, the techniques of accelerator mass spectrometry (AMS) are not constrained by the half-life, and AMS may be used to detect $^{10}$Be quantitatively in small environmental samples (~ 1g) containing as few as $10^7$ atoms of the isotope. The AMS system at the ANU's 14UD accelerator, which was developed initially for chlorine-36 measurements and has subsequently added carbon-14 to its capabilities, has now been adapted to allow the measurement of beryllium-10.

2. Accelerator Mass Spectrometry of $^{10}$Be

2.1 Production of negative ions and acceleration

A description of the principles of AMS is presented in another abstract to this Conference (R.F. Davie et al.). Consequently, only considerations which are specific to $^{10}$Be will be discussed in the present abstract.

Beryllium does not have a stable negative ion, whence some form of molecular ion is required for injection into the accelerator. The BeO$^-$ ion was chosen since beryllium oxide is a convenient form in which to prepare the sample and the BeO$^-$ ion is produced with modest intensity (~ 0.5 μA) from the sputter ion source of the 14UD accelerator. However, the use of a molecular ion carries a certain penalty. If a carbon foil is used to strip electrons from the ions in the terminal of the accelerator, then the Coulomb repulsion of the resulting positively-charged beryllium and oxygen ions gives rise to an energy spread in the final energy of the beryllium ions. This energy spread is considerably greater than the energy acceptance of the system and leads to a substantial loss of intensity. The alternative is to use a gas stripper. The gentler collisions in the gas break up the molecule,
greatly reducing the "Coulomb explosion" effect and the consequent energy spread. Average charge states are, however, lower when a gas stripper is used and hence a higher terminal voltage is required to optimise the fraction of beryllium ions in the $3^+$ charge state. To date, measurements have been carried out at a terminal voltage of 8 MV, but a somewhat higher voltage may be required to maximise the $3^+$ charge state fraction.

2.2 Detection of beryllium-10 ions

The major technical difficulty to be overcome in the detection of $^{10}$Be is an intense flux of boron-10 ions that also reach the detector. Because $^{10}$B and $^{10}$Be have the same mass, no combination of magnetic and/or electric analysis elements can discriminate between them, and it appears to be difficult to reduce the boron contamination chemically to the ppb level which would be required to reduce the $^{10}$B flux to a rate which the ionisation counter could handle comfortably. Hence, the detection system must be able to cope with $^{10}$B fluxes as high as $10^7$ particles per second. To accomplish this, one exploits the difference in range between $^{10}$B and $^{10}$Be due to their different nuclear charges; $^{10}$Be travels further in matter than does $^{10}$B of the same energy. Other laboratories have used foils or small gas cells to stop the $^{10}$B ions before they reach the ionisation chamber which is used to identify and count the $^{10}$Be ions. We have been able to simplify the detection system by using the front portion of the ionisation chamber as the stopping region for the $^{10}$B ions and the back region as the $^{10}$Be counting region. This has the advantage of minimising the number of foils through which the $^{10}$B ions must pass before being counted. An added bonus is that the $^{10}$Be flux can be monitored continuously via the ionisation current from the front portion of the counter. This greatly facilitates tuning of the accelerator for maximum $^{10}$Be transmission.

One additional complication is that at the energies employed here, the $^1\text{H}(^{10}\text{B}, ^7\text{Be})^4\text{He}$ reaction induced by $^{10}$B on any hydrogen in the window or gas of the detector is a source of $^7$Be ions in the back section of the detector. Consequently, a Havar window was used rather than the traditional mylar, and the detector gas was changed from isobutane to freon-14 (CF₄).

3. Preliminary results

Development work is proceeding on two fronts; development of the AMS system and development of the chemical processing. The former is almost complete. The latter is being pursued at Lucas Heights and involves adding $\sim1\text{mg}$ of "dead" beryllium carrier to kg of soil in order to extract the $^{10}$Be in the soil. It has been shown that beryllium oxide can be produced in sufficiently pure form that reasonable currents of BeO$^-$ can be obtained
from it and now questions of extraction efficiency and contamination during processing are being addressed.

4. Conclusion

Accelerator Mass Spectrometry of beryllium-10 at the ANU has been developed to the point where quantitative measurements of the Be-10 content of environmental samples can be performed on a more or less routine basis. It is anticipated that application to environmental problems will commence in 1990.
ON-LINE ANALYSIS IN THE AUSTRALIAN COAL AND MINERAL INDUSTRY

by

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Abstract

Major improvements have taken place in coal and mineral processing in the last 15 years. A significant part of this improvement has resulted from the development of on-line sensors that continuously analyse the material being processed which has led to better control of the process. Developments in on-line instrumentation by the CSIRO Division of Mineral and Process Engineering at Lucas Heights are reviewed. These include on-line determination of ash in coal using dual energy $\gamma$-ray transmission and pair production techniques; systems for in-stream analysis of mineral and coal slurries; on-line determination of moisture in coal and coke; and specialised electronics for plant testing of the analysis techniques.

1. INTRODUCTION

Mineral and coal processing operations can be controlled more economically if information on important process variables is obtainable rapidly. Much of this information can be provided by nuclear and electrical techniques. The CSIRO Division of Mineral and Process Engineering, Lucas Heights, has developed a range of techniques, plant tested them in industrial plants, and licensed them to two Australian companies, the Australian Mineral Development Laboratories (Amdel) and Mineral Control Instrumentation Ltd. (MCI), both of Adelaide, South Australia. The considerable initiative shown by these companies and their close collaboration with CSIRO has led to the setting up of Australia as a world leader in the supply of industrial on-line analysis equipment. Although Australia earns significant export dollars from sale of this equipment, Australia earns far more from the increased yield of minerals and coal resulting from the application of the on-line analysers to improve control of beneficiation operations.

2. ASH CONTENT OF COAL

Two types of ash gauges are in widespread routine use in Australian coal operations: (a) gauges, based on photoelectric absorption, in which dual energy gamma-ray transmission (DET) measurements are made directly on coal on-line on conveyors(1) and (b) gauges, based on pair production (PP) gamma-ray interactions, and high flow sample by-lines(2). Both are manufactured and marketed by MCI. The former is called the Coalscan Model 3500 through-belt ash monitor, and the latter the Coalscan Model 4500 by-line ash monitor.

Coalscan ash monitors are mainly used to improve coal mining and preparation operations. The Coalscan ash monitors have gained rapid acceptance by the Australian coal industry since the first one was installed in 1983. The total coal scanned by Coalscan monitors in Australia is now roughly one third of the total yearly tonnage of black coal mined in Australia. Coalscan ash monitors are increasingly being used overseas, particularly in the USA. There are now 85 of these Coalscan ash monitors installed or on order throughout the world, 45 in Australia and 24 in USA. The economic benefit gained from use of the Coalscan on-line ash monitors in most cases results from control of mining and plant operations closer to the required specification ash content. External consultants have estimated that improved productivity resulting from use of Coalscan ash monitors has led to total benefits of $158 million to Australia over a five year period(3). Total sales of Coalscan ash monitors are about $13 million.
3. MINERAL AND COAL SLURRIES

3.1 Metalliferous mineral slurries

Amdel was licensed to manufacture and market systems based on radioisotope sources and scintillation detectors in 1973. These analysis systems are now installed in about 50 mineral concentrators throughout the world. In the early 1980s, Amdel incorporated solid state detectors into probes for use for on-stream analysis, and used these for the residue streams with low concentrations of valuable minerals. Recently, CSIRO has developed a solid state detector probe, with the detector cooled thermoelectrically. This probe is more robust than the liquid nitrogen cooled probe, and overcomes the need for weekly replenishment of the liquid nitrogen. The Peltier-cooled detector probe was first plant tested at Broken Hill in mid 1989.

3.2 Coal slurries

The gamma-ray transmission techniques widely used in the metalliferous mineral industry to determine solids weight fraction via bulk density of the slurry are inaccurate when applied to coal slurries of variable voidage. To overcome this problem, a new technique has been developed(4) which is based on probes immersed directly into plant process streams or into high flow rate sample by-lines, similar in concept to those used for the on-stream analysis of mineral slurries. CSIRO has licensed Amdel to manufacture and market this coal slurry analysis system. A three-stream system has been used at Oaky Creek Coal in the Bowen Basin, Queensland, since 1986, and a five-stream system was installed at Curragh at the end of 1988.

Oaky Creek Coal metallurgists have recently published results comparing manual and automatic control of the flotation banks of their coal preparation plant(5). They conclude that automatic control results in a more consistent product ash with a 10% increase in yield at constant ash. This is equivalent to an increase in coal production of 75,000 tonnes per year for their 3 million tonne per year plant.

4. MOISTURE IN COAL AND COKE

CSIRO has developed nuclear and non-nuclear techniques for the on-line determination of moisture. These include techniques based on the measurement of capacitance, microwave transmission, and neutron transmission. For many on-belt moisture measurements the capacitance and microwave techniques are favoured. However the capacitance and microwave methods are not suitable for use on materials of high electrical conductivity (e.g. coke) or on many steel-cord conveyor belts. Neutron techniques are preferred in these applications.

4.1 Capacitance and Gamma-Ray Backscatter

In this technique, moisture is correlated with radiofrequency susceptance and conductance, determined using an under-belt capacitance sensor in which a fringing electric field interrogates a layer of coal on the conveyor belt directly above the sensor. Variation in the density and thickness of this layer is compensated for by using a matched under-belt gamma-ray backscatter gauge. A prototype gauge was field tested by CSIRO on the coking coal product conveyor at Stockton Borehole colliery, Teralba, New South Wales in late 1985(6). MCI has been licensed to manufacture and market the capacitance gauges, and at March 1989 had installed 31 commercial units in industrial plants, of which approximately half were in the coal industry.

4.2 Microwave Transmission

Moisture measurement by microwave techniques is based either on the high real or imaginary part of the complex dielectric constant of water compared to the dielectric properties of the majority of dry materials. Microwave transmission techniques have the advantage of providing a bulk analysis that is not sensitive to the vertical segregation of moisture that may
occur in material carried on conveyors. A prototype non-contacting microwave gauge has been developed for on-belt measurement of moisture (7). An industrial prototype has been plant tested on the product conveyor at Howick Coal Washery, Singleton, NSW, since April 1989.

4.3 Fast Neutron and Gamma-Ray Transmission

The on-line determination of coke moisture is particularly important in the steel industry for controlling the net weights of dry coke and iron ore fed to blast furnaces. The potential benefits from improved control of the iron ore to coke ratio include stabilisation of blast furnace operation, a reduction in blast furnace hot metal temperature variability and a reduction in hot metal silicon content and its variation.

A prototype fast neutron and gamma-ray transmission (FNGT) gauge for the conveyor belt determination of coke moisture has been developed by CSIRO (8) and field tested at BHP, Newcastle since June 1988. This gauge permits direct on-belt moisture measurement and overcomes many of the limitations of conventional thermal neutron moisture gauges. MCI has been licenced to manufacture and market the FNGT moisture gauges.

5. ELECTRONIC INSTRUMENT DEVELOPMENT

Highly sophisticated and stable electronics is required to test the above techniques in the harsh industrial environment of mineral concentrators and coal washeries. Most of the electronic circuitry being developed is now being constructed in modular form using the Eurocard format. A range of standard modules has been constructed, so that each new application requires at most one or two new modules. The backplane wiring between the standard modules is customized for each application. This system of standard circuit and software modules allows rapid and economical development of new instruments which are well documented and easy to service. The transition from a laboratory prototype to a rugged industrial instrument can be achieved in a short time with very little redesign.

On-line measurement techniques using nuclear methods place very stringent demands on the nuclear counting electronics. To meet these demands a fast industrial nuclear counter module called SIROFINC has been developed. It offers almost an order of magnitude improvement in speed and stability over conventional circuitry. Each SIROFINC module is built on a double height Eurocard which contains all the circuitry (except preamplifier) required for one scintillation detector, and up to 15 modules can be accommodated in a single system. All functions are computer controlled.

6. REFERENCES

PROTON MICROPROBE STUDIES OF ILMENITE MEGACRYSTS FROM KIMBERLITES

by

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Abstract

1. Introduction

Diamonds are formed at depths of more than 150 km in the earth's mantle, and carried to the surface during volcanic eruptions of special rock types, such as kimberlite or lamproite. The diamond content of such rocks is typically a few ppm. Exploration for diamonds therefore is heavily based on the recognition of more abundant mantle-derived "indicator minerals" (Cr-pyrope garnet, Mg-rich ilmenite, chromite) in samples of soil and stream sediments. Electron microprobe analyses of indicator minerals give the major-element composition of individual grains. These data are used during exploration and during evaluation of prospects, to assess the probability that particular samples are derived from diamondiferous rocks. However, this method is commonly ambiguous, because minerals of similar major-element composition also be be derived from a range of barren rock types.

Since 1987, HIAF has carried out a program of proton microprobe analysis of trace elements in indicator minerals, aimed at adding another level of discrimination to this exploration method. Much of the material being analyzed consists of heavy-mineral concentrates from diamondiferous and barren sources, to provide empirical discriminants. However, we are also analyzing the minerals in fragments of mantle rocks (xenoliths and megacryysts)-carried to the surface by kimberlites, and the mineral inclusions enclosed within diamonds. These studies provide insights into rock-forming processes in the mantle, including those responsible for the formation of diamond, and help us to interpret compositional features seen in the concentrate samples.

2. Methods

The analytical methods used in this work have been reported elsewhere [1,2] and a detailed treatment of the HIAF hardware and software is given by Ryan et al. [3-5]. Mineralogical applications of the proton microprobe, especially those reported here, require large numbers of accurate, high-precision analyses. The HIAF hardware [6] has been specifically designed to allow high sample throughput, with high beam currents for low detection limits, flexible sample handling and minimum cycling times for sample changes. The software development has concentrated on ease of use and reliability of data treatment, with special attention given to the development of stable background algorithms for the reproducible treatment of low-statistics peaks [3,5]. For most of the PIXE microanalysis reported here, a 8 nA 3MeV proton beam was used, with a beam spot size of 20 μm. A 200 μm Al X-ray absorber is usually selected to attenuate major-element lines. The typical integrated charge was 3 μC, corresponding to an acquisition time of 6 minutes per grain.
3. Ilmenite Megacrysts

Many kimberlites carry abundant megacrysts (>2 mm diameter) of magnesian ilmenite ((Fe,Mg)TiO3), which is resistant to weathering and transport, and serves as an indicator mineral. The origin of the ilmenite, and its genetic relation to the kimberlite, is a matter of considerable debate. Our trace-element studies of ilmenites from individual kimberlites typically show well-defined interelement trends (Fig. 1). These are easily interpreted in terms of fractional crystallization of single batches of magma, in which crystallizing phases are removed from the magma (for example, by sinking) to form cumulate rocks. In a detailed study of ilmenites and coexisting silicate phases from the Monastery Mine kimberlite we have shown [7] that Nb behaves incompatibly; it is progressively concentrated in the liquid phase during crystallization of the magma. Hence the Nb content of the ilmenites increases steadily throughout the fractionation sequence, while the contents of other elements, such as Ni, Zr, Ga and Ta, vary according to the nature of the coprecipitating phases. In effect, the ilmenite serves as a monitor of the evolving composition of the magma. Breaks in the Ni-Zr and Ni-Nb curves (Fig. 1) reflect initial crystallization of zircon (Z+), the end of pyroxene + garnet crystallization (S–) and renewed crystallization of olivine (O2+).

In a broader study of ilmenite suites from 20 African kimberlites, we have found trends generally similar to those shown in Fig. 1, but with clear differences from pipe to pipe. In general, each kimberlite appears to contain ilmenites derived from a single batch of magma. This result is an important argument in favour of a genetic relationship between the megacryst-forming magma and the kimberlite that brought ilmenites to the surface. It also has significant implications for exploration strategies; it may be possible to tell from analysis of an ilmenite concentrate how many kimberlite bodies are to be found within an exploration area.

Many non-diamondiferous alkalic basalts carry magnesian ilmenites that overlap the major-element compositional range of kimberlite ilmenites, and these pose a serious exploration problem. However, preliminary studies of basaltic Mg-ilmenites suggest that most of these have low contents of Ni, Zr and Nb compared to kimberlitic ones of similar Mg content, and that the interelement relationships are different as well. These results suggest that the basaltic ilmenites are formed either from different magma types, or under different P-T conditions, or both.

4. Conclusion

Trace-element studies of heavy-mineral concentrates and xenolith material are providing tools for the discrimination of barren and diamondiferous source rocks, and for recognizing the presence of multiple sources within an exploration area. Studies of mantle-derived xenoliths and macrocryst suites provide information on mantle processes, including those important for diamond formation. Ultimately, understanding of the mantle processes that control the formation of diamond, kimberlite and other diamondiferous rocks will lead to better exploration models for locating primary deposits of diamonds. Proton-microprobe studies are making a significant contribution to that understanding.

References

fig. 1 Plots of Ni and Zr vs. Nb in ilmenite megacrysts from the Frank Smith kimberlite pipe, South Africa. Nb increases continuously during fractionation of the magma; inflections in the curves reflect changes in the coprecipitating minerals. Z+, beginning of zircon crystallization; S−, cessation of pyroxene+garnet+olivine crystallization; O2+, beginning of second olivine crystallization.
THE ACCURACY OF STANDARDLESS PIXE MICROANALYSIS
IN GEOSCIENCE APPLICATIONS

by

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Abstract

1. Introduction

The proton microprobe is an ideal tool for quantitative non-destructive in-situ microanalysis of mineral grains. Combined with proton induced X-ray emission (PIXE), it can provide trace element detection sensitivities of a few ppm in most mineral grains, and sub-ppm analyses in light matrices. Therefore, it has a key role to play in geoscience research in extending the range of established analytical instruments, such as the electron microprobe (EMP), by paying particular attention to the accurate analysis of trace elements with concentrations all the way down to the limits of detection.

A single pass PIXE data analysis scheme is used which usually incorporates the major element composition of the sample determined by prior EMP microanalysis [1]. This composition is used to calculate the yields and relative intensities of all the X-ray lines for all anticipated elements [2]. In cases where self-absorption and secondary fluorescence are not severe, a representative composition is used to calculate yields for the analysis of a series of mineral grains of similar composition. The calculated relative intensities are used in the least-squares fit to the spectrum. The most important feature of the fit is the use of a fixed background evaluated from the spectrum using the Statistics-sensitive Non-linear Iterative Peak-clipping (SNIP) algorithm [3]. This background treatment takes particular care to treat statistical fluctuations to provide a reliable approximation to the continuum background under peaks, even in regions of the spectrum with low counts. The important aspects of each analysis step, and its integration into a suite of programs, are detailed in ref. [5,9].

2. Standards comparisons

The accuracy of the method was tested against accepted bulk analyses of glasses prepared from standard rocks BCR-1, AGV-1 and GSO-1 from the US Geological Survey [4]. Table 1 shows the results of 3 MeV PIXE analyses of the glass BCR-1. The PIXE results are in general agreement with the accepted values.

3. Monte Carlo test

This test aims to determine whether peak areas are accurately determined as the concentrations of trace elements are reduced to the detection limits. We have used high statistics parent spectra (of total counts A and B) to generate spectra of varying total counts (aA and βB) by Monte Carlo methods. The total counts are selected so that the channel-by-channel sum of the two spectra form a linear combination (aA+βB=1). Hence, the deduced peak area of any element in the sum spectra should display a linear trend with a or β. If an element occurs at a high level in one parent spectrum and is below the detection limit in the other, then the linearity of the trend tests the ability of the spectrum treatment to cope with peak areas as the detection limit is approached.
(6.3±0.9 and 6.9±0.5 ppm, respectively). The garnet contains Ga (3.4±0.3 ppm) and Zr (6.2±0.3 ppm) which are below detection in the olivine (<0.6 ppm). Therefore, these four elements constitute good candidates for the linearity tests. Fourteen spectra, corresponding to 10 μC each, were generated by the Monte Carlo method [3] for fractional olivine content α ranging between 0 and 1 (fig. 1). The results of this test are shown in fig. 1. The straight line shown for each element is drawn between the analyzed concentrations of the element in the two parent spectra. The trends display remarkable linearity to sub-ppm levels. The linear regression values for Ni and Zn (~0.5 and ~0.1 ppm) are consistent with the detection limits obtained from the analysis of the 72 μC olivine spectrum (<0.7 and <0.6 ppm, respectively).

4. Comparison with electron microprobe data

The PIXE results can be normalized to match the EMP analysis of some major element, typically Fe; the factor by which the PIXE results are scaled provides an alternative measure of the accuracy of our PIXE analysis method. The scaling factors encountered in routine analysis are shown as histograms in fig. 2. The broad distribution, summarizing 4800 analyses for which EMP values were available, corresponds to the use of integrated beam current to determine the beam fluence. The narrow distribution corresponds to more recent data from 15 analyses on a range of minerals which made use of the beam-chopper to measure beam fluence. These minerals included grains of garnet, orthopyroxene, clinopyroxene, low and high chromium spinels and a variety of sulfides. The width of the distribution of scaling factors in fig. 2 using integrated beam charge (standard deviation of 9.5%) indicates that significant errors in current integration can result. On the other hand, use of the beam chopper reduces the width of the distribution markedly (standard deviation 3.3%). It should be noted that the uncertainty of the EMP analyses also contributes to this spread. We have adopted a value of 2% as representative of EMP analysis uncertainty. The mean of the chopper-normalized scaling factors is 1.01, which is an indication of the absolute accuracy of our PIXE method for mineral analysis using the chopper.

5. Conclusions

The data presented here show that absolute standardless PIXE analysis of minerals can be done to an accuracy of the order of 3% (standard deviation) using a chopper. The data also suggest that the bulk of our existing data, which are normalized to EMP Fe, are of similar accuracy. The Monte Carlo tests suggest that this reliability can be expected for quite different types of spectra from a wide variety of samples.

References

Table 1

<table>
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<td>Zn</td>
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Result of the analysis of a sum spectrum from 20 spot analyses, normalized to EMP Fe concentration. Total integrated charge Q = 60 μC. All values are in ppm except where indicated as weight %. Quoted uncertainties are analytical precision.

* Accepted values [13].
* Minimum detection limit (99% confidence level).

fig. 1 Result of analyzing 14 Monte Carlo generated spectra, which represent 10 μC each, displayed as a function of the fraction of olivine in the spectrum (expressed in %). The straight lines connect the values obtained from the analysis of the parent spectra (73 μC garnet and 72 μC olivine).

fig. 2 Histograms of the ratio of EMP analyzed Fe to PIXE analyzed Fe for (i) 4800 analyses using integrated beam charge (broad distribution, standard deviation 9.5%), and (ii) 150 analyses using chopper beam fluence measurement, scaled by 20 (hatched area, mean 1.01, standard deviation 3.3%).
LOW LEVEL U AND TH DETERMINATION IN AUSTRALIAN DUNE SYSTEMS

BY

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Abstract

Introduction. The application of thermoluminescence (TL) dating in archaeology and Quaternary geomorphology requires the measurement of annual radiation dose rates in the relevant field context in order to determine the rate at which energy is being delivered to the mineral being used in the TL age determination. The radiation dose is determined by the amount of uranium, thorium and potassium present and there is a small contribution from cosmic rays (Prescott and Hutton 1988).

Stimulated by the very time-consuming processes involved in obtaining a dose rate by chemical analysis and/or alpha particle counting, we have developed field scintillometry, following Løvborg et al (1979). In this technique, a sodium iodide scintillation counter in a hole at the field site records the total gamma dose from the surrounding soil. With a 76 mm x 76 mm diameter "low background" crystal and housing, used in conjunction with an "URTEC" scintillometer, we record all detected events with a deposited energy between 0.5 and 2.78 MeV. This "total count" is proportional to the environmental gamma ray dose. The first results of this programme were reported at the 1987 meeting (Prescott and Hutton 1987) and a complete account of the dose rate measurements has been published (Prescott and Hutton 1988).

In addition to the total count we record the counts in windows corresponding to the 1.46 MeV gamma ray from $^{40}$K, the 1.76 MeV gamma ray from $^{214}$Bi in the uranium series and the 2.62 MeV gamma ray from $^{208}$Tl in the thorium series. These allow the direct determination of total K, U and Th under field conditions, assuming secular radioactive equilibrium. Alternatively, the scintillometer results can be used to identify possible disequilibrium by comparison with the other methods.

The present contribution describes comparisons between gamma-scintillometry and neutron activation analysis conducted at ANSTO, Lucas Heights. We have demonstrated the close agreement between XRF and DNA/NAA results for total thorium and uranium at a previous Conference in this series (Hutton et al 1985).

The Measurements. The instrument was originally calibrated for both total dose and individual elements against the standard Th, U and K pads at CSIRO Mineral Physics, North Ryde, NSW. It was subsequently checked against the "bore-hole" standards of the SA Department of Mines and Energy at Glenside, S.A. These are primarily designed for calibration of instruments used to search for ore-grade deposits. One of the purposes of the present study was to check the scintillometer performance at low elemental concentrations, since the overwhelming majority of sites of
interest for TL dating in Australia have very low concentrations of the relevant elements. This was done by comparing scintillometer dose rates with those calculated from conventional chemical analyses: XRF, DNA and NAA, and from thick source alpha counting, at a range of field sites. We now have data from upwar ds of 200 sites. For most of these, samples were obtained at different depths in a soil profile, and many sites were sampled more than once.

Table 1 shows the comparative data. With very few exceptions, all within counting errors, the agreement is very satisfactory. This indicates that, in the dune systems sampled, radioactive disequilibrium is rare. We believe that this is because the U and Th are mostly in resistate minerals such as zircons. The table includes data from our site with the greatest activity (PJIES/75), sampled at a depth of 0.75m in the Cleland Hills, NT. Our site with the lowest activity, the Dwarf Kabali dune of the Cooloola National Forest, Queensland, is not included in the table since the concentrations are so low as to be at the limit of detection for all but scintillometry. This and nearby sites are in dunes of almost pure quartz sand. The site SQ2/2 at Steetley Quarries, near Mount Gambier, S.A., sampled at 2m, is almost pure Bryozoal limestone and has very low activity also. Other sites of interest are from Lake Amadeus where the dunes are largely blown gypsum; Roonka, a Holocene dune comprising quartz and variable amounts of clay; and the calcareous dunes from the South East of S.A., LeFevre Peninsula and Yorke Peninsula, where a sequence of stranded beach dunes span the time period 700,000 years to the present. These calcareous dunes are frequently consolidated and their characteristic feature is their relatively uniform composition.

Conclusion The principal conclusion that we draw from these data is that field scintillometry is a very effective method, not only for finding total gamma-dose rates, but for individual element analyses. In most circumstances it is capable of providing in an hour or two in the field, data that would take two or three person-weeks to obtain in the laboratory. In extreme cases, near the limit of detection, it is clearly the preferred method. To guard against the possibility of disequilibrium some alternative method should also be used. The method is also difficult to interpret when the sampling site is highly stratified because the scintillator integrates the gamma ray flux from within a spherical volume of about 0.3m radius. It therefore gives a concentration of the individual elements averaged over this volume. On the other hand this is a positive advantage when one wants to know the total gamma ray dose rate, as in thermoluminescence dating.

Acknowledgements The work was supported by AINSE, by the ARC and by the Research Committee of the University of Adelaide. Particular thanks are due to L. Thomas who prepared the samples and carried out many of the laboratory measurements.

References

<table>
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N.B. 1 s.d. counting error is 0.5 μg/g Th and 0.2 μg/g U for range 1–5 μg/g.

* By XRF analyses at CSIRO Division of Soils, Adelaide.
New Britain Sources

Natural glass including obsidian and tachylite has been found in New Britain, but it is not always suitable for manufacturing stone tools. Previous analyses of natural glass artefacts have shown that people preferred obsidian from two main source areas: Talasea and Mopir (Specht 1981; Specht and Hollis 1982; Bird et al. 1988). Obsidian from the Talasea region has been used by people for at least 20,000 years (C. Gosden, pers. comm.). Later in the Holocene it was transported over long distances, to the Highlands of New Guinea (Watson 1986) and eastern Malaysia (Bellwood and Koon in press) in one direction and to Fiji (Best 1987) in another. Obsidian from Mopir, Cape Hoskins, is distributed less widely, but may have been of greater importance at particular times in the past (Green 1987). To help explain such preferences for obsidian, the aim of this project has been to discriminate between the main source areas as well as between source volcanoes within the Talasea region, using PIGME-PIXE.

In the Talasea region over 50 discrete obsidian exposures have been recorded in a range of topographic settings, and the flaking quality of the obsidian is highly variable. Seven obsidian sources have been tentatively identified, all with flaked artefacts and each associated with a separate volcano: Mt. Kutau (Schleuther), Mt. Bao, Mt. Humugari, Mt. Gulu, Garala (Schaumann Island), Mt. Baki and Mt. Hamilton. Good quality obsidian was formed at each of these sources, except Mt. Hamilton where it is of rather poor quality and very rarely flaked. It is not known how many flows have erupted at each source but obsidian occurs within in situ deposits of rhyolite and also in secondary contexts, within laharic flows and as blocks buried in ash or lying loose on the surface. Element concentrations were measured for samples of obsidian from each of the known sources.

Measurements

Measurements of element concentrations were made by ion beam analysis using a 2.5 MeV proton beam from the Lucas Heights Van de Graaf accelerator (Duerden et al. 1980). X-rays and gamma-rays were detected simultaneously in an automated system designed for unattended operation with a large number of samples. System parameters were the same as those for an extended study of Melanesian artefacts (Bird et al. 1988) so that the new data could be readily compared with earlier results. Data processing was also carried out using x-ray and gamma-ray fitting routines. The results were transferred to a computer file which was then used for conversion to element concentrations, plots, listings and statistical cluster analysis. Particularly important is the variability introduced by the measuring system and this can only be understood from such an extended study.

Some of the experimental factors which contribute to the variability of the
results include:

(i) Sample charging. A heated carbon fibre filament is mounted close to the sample so that electrons are drawn to the beam spot to compensate for the positive charge of the proton beam. This prevents observation of continuum x-rays but, during long unattended runs, problems may still result in unsatisfactory x-ray spectra.

(ii) Surface roughness. Samples are mounted so that a 1mm diameter area in the smoothest region faces the proton beam. But it is not always possible to position the samples precisely at right angles to the beam, and this affects self-attenuation of low-energy x-rays. Results for Si are particularly affected. A sum of oxides of major elements is calculated and if it is not within 100 ± 10% the measurement is rejected.

(iii) Surface composition. Ion beam analysis is sensitive to a surface layer approximately 20 microns thick. The results are thus affected by inclusions and contaminants. If any anomalies are observed that sample is remounted and reanalysed.

Results and Discussion

Analyses were conducted on archaeological artefacts, ethnographic obsidian sources and visible obsidian exposures associated with source volcanoes. Statistical analyses of PIGME-PIXE results indicate that obsidian from several sources was used in the past and that there are differences between obsidian flows from source volcanoes in the Talasea region. By comparing and combining element ratios from several previous PIGME-PIXE analyses it is possible to detect variation in instrumental measurement and also to account for some anomalies in listed elemental compositions.

More than 20 elements are detected in the PIGME-PIXE measurements but only 12-14 are determined with sufficient accuracy for the measuring conditions and short runs used here. Of these Ti, Fe and Zr show little variation amongst the geographic group of samples. However, the use of Fe or Zr ratios helps to minimise systematic variations which can occur particularly in the PIXE spectrum. The current characterisation programme has employed eleven elements to produce nine element ratios: Al/Na, F/Na, Mn/Fe, Zr/Fe, Y/Zr, K/Fe, Ca/Fe, Rb/Fe, Sr/Fe. These have been used in several non-parametric methods of cluster analysis.

On the basis of elemental composition of obsidian from currently visible source exposures, at least three source groups may be clearly distinguished within the Talasea region: 1. Kutau and Bao, 2. Pilu, Voganakai and Garua, i.e. Mt. Hamilton, and 3. Baki, also on Garua (Figure 1). The Garala (Schauraann) source overlaps with Baki and Pilu, but further samples need to be run. It is also possible that, with more refined measurements, the Garua (Mt. Hamilton) source will separate from the Pilu-Voganakai group. Analysis of artefacts has suggested a distinct group (ZZ), thought to indicate a West New Britain source on the basis of composition (Bird et al. 1988), but ZZ corresponds to none of the obsidian sources analysed here.

In order to further refine techniques for discriminating source volcanoes and obsidian flows within the Talasea region we propose to increase the number of obsidian exposures sampled for each recognised source volcano and also to measure more precisely a wider range of trace elements. The latter should be possible by modifying the perspex "pinhole" filter and widening the beam diameter to increase the total beam dose.
References


Figure 1: Correspondence analysis of West New Britain obsidian sources.
THORIUM-RADIIUM DATING OF FLOODPLAIN SEDIMENTS

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Abstract
The history of the Magela floodplain has been derived principally using a chronology based on radiocarbon dating of organic detrital remains embedded in sediment. Very little wood or charcoal has, however, been found in the freshwater sediments that make up the surface of the floodplain. Alternative dating methods have, therefore, been sought.

Dating of these sediments has been investigated using the thorium-radium method because a significant radioactive disequilibrium has been observed in the uranium series between $^{230}\text{Th}$ and $^{226}\text{Ra}$. Radiocarbon dating was also carried out to check on the validity of the assumptions inherent in the method. Good agreement was obtained between results using the two methodologies. Sedimentation rates of about 0.2 mm/year were deduced. The thorium-radium method should be applicable at other sites where suitable organic remains are not found.
MONITORING AUSTRALIAN FOODSTUFFS FOR TOXIC AND ESSENTIAL TRACE ELEMENTS USING NEUTRON ACTIVATION ANALYSIS

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Abstract

A total of nine toxic elements are listed in current Australian legislation for maximum permitted levels in foodstuffs. In addition, the National Health and Medical Research Council (NH&MRC) have published data for the recommended dietary intake of seven essential elements. This paper examines the use of neutron activation analysis to monitor the levels of these toxic and essential elements in 350 food samples gathered around Australia as part of the NH&MRC's Market Basket Survey.

Introduction

Current Australian legislation specifies the maximum permitted levels of nine toxic elements Cu, Zn, As, Se, Cd, Sn, Sb, Hg and Pb. in foods as well as specific food items. In addition, the National Health and Medical Research Council (NH&MRC) have published data for the recommended dietary intake of seven essential elements Na, Mg, K, Ca, Fe, Zn and Se. Recent investigations have examined the compliance of Australian foods with both of these requirements by measuring their concentrations using the advanced analytical techniques of radiochemical and instrumental neutron activation analysis. Elements that were insensitive to NAA were analysed by inductively coupled plasma mass spectrometry (ICP-MS). This paper reports the results for those elements sensitive to INAA as well as those for Cu, Cd and Pb measured by ICP-MS.

Experimental

Australia-wide sampling of representative foods from the diets of Australians posed a serious problem but was solved when the NH&MRC gave us permission to join the autumn sampling program of the Market Basket (Noxious Substances) Survey. This survey covered each of the seven State capitals where 50 individual foods were purchased by State health authorities in each of three localities in each city. These were forwarded to the School of Home Science at East Sydney Technical College for processing before chemical analysis. Equal amounts of the
three samples of each food from each city were blended during preparation to yield an individual food composite. Foods normally cooked were cooked without salt and then blended. Samples were placed in clean screw-cap polystyrene containers, frozen and sent to the laboratory.

All samples were stored frozen until ready for analysis. They were then either dried in an air oven at 100°C or lyophilized according to the nature of the material. The samples were blended further using the brittle fracture technique [1].

Standard INAA procedures as previously described [2] were used to measure those elements sensitive to this technique. When sodium concentrations permitted, selenium analysis were performed via the short-lived $^{75}$Se [3].

Since ICPMS cannot handle solid samples directly, samples were dissolved. Because both sulphate and chloride can interfere with some element determinations, both $\text{H}_2\text{SO}_4$ and HCl were avoided and samples were wet ashed with $\text{HNO}_3$ alone. Samples were mixed with 1.5 ml Aristar $\text{HNO}_3$ in 25 ml polycarbonate, screw-cap, vials whose threads were wound with Teflon tape. These were placed in 250 ml, screw-cap jars for secondary containment. Ten samples were simultaneously microwaved for 20 min using a power setting of 80 W. Samples were diluted to volume to maintain total acidity below 5% and total dissolved solids at 0.1%. These conditions reduce the occurrence of acid attack and sample blockage of the sampler cone in the ICPMS.

Results and discussions

Three hundred and fifty food samples were analysed for Na, Cl, Mg, K, Ca, Cr, Mn, Fe, Cu, Zn, Se, Cd and Pb. Results for Se have been discussed elsewhere [3,4] while As and Hg data were obtained by radiochemical NAA and are not examined in this paper. All results were calculated as ug g$^{-1}$ on a wet basis of the food prepared for consumption. While the majority of foods were of Australian origin, canned products (meat, salmon, sardines) are often imported.

All toxic elements reported in this paper comply with Australian regulation with the exception of Cd. Copper concentrations ranged from 0.1 to 160 ug g$^{-1}$ which were less than legal limit of 10 ug g$^{-1}$ for most food with limits for ovine livers set at 200 ug g$^{-1}$. Zinc levels fell in the range 0.4 to 65 ug g$^{-1}$ well below the limit of 150 ug g$^{-1}$. Selenium figures for fish and offal of 0.78 and 0.54 ug g$^{-1}$ approached the legislated maximum of 1 ug g$^{-1}$ but fruit and
vegetables were a factor of thirty less than this limit. Maximum levels for lead in foodstuffs are set at 1.5 ug g\(^{-1}\) for most foods although regulations allow for higher limits for bran, wheat germ, molluscs and meat and tomato products in tinplate containers (2.5). No foods exceeded these limits. However, some of the grain and meat products tested for cadmium exceeded the maximum legislated values of 0.2 and 0.05 ug g\(^{-1}\), respectively.

The total intake of essential elements present in each food item contained in a number of hypothetical human diets was calculated. These diets were compiled by the Nutrition Section of the Federal Department of Health for a nine-month old infant, a two-year old child, a twelve and half-year old male, a similarly aged female, an adult male and an adult female. Results were compared with the recommendations of the NH&MRC for each of these age groups.

Daily intake figures for Na (750 - 3100 mg) were higher than the maximum recommended for all groups (580 - 2300 mg) with the exception of adult females (1900 mg). In contrast, the average daily intake for K (1200 - 3200 mg) fell within the recommended ranges (470 - 5460 mg) for each population group. Zinc and Fe followed similar trends to Na with only the daily intake for adult females conforming to recommended values (12 - 16 mg).

The Food and Nutrition Board in the US have listed recommended daily intake figures for 14 essential elements. We have measured a number of these not included in the NH&MRC list. The level of Cl, Cr and Mn intake for all age groups fell within or on the maximum recommended figures of 400 - 5100 mg, 20 - 200 mg and 0.7 - 5.0 mg.

References
Beam Induced Damage in Organic Targets

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This work seeks experimental evidence and understanding of radiation damage, its nature and its dependence on dose, dose rate and scanning frequency [1]. Complex and strange results have been observed.

Charge Particle Microbeams

For the high current densities used in particle microbeams radiation and thermal damage may be so severe that changes in the target are visible under the microscope. Heating effects were described in great detail by Talmon and Thomas [2]. In this treatment of the specimen temperature calculation the authors ignored radiation heat loss and considered only heat loss through conduction. The temperature rise in such a case, is given by

\[ T_{\text{max}} - T_0 = \frac{q_a r^2}{4kd} (1 + 2 \ln \left(\frac{R}{r}\right)) \]

where \( T_{\text{max}} \) and \( T_0 \) are the maximum temperature in the centre of the beam and the conducting grid temperature respectively, \( q_a \) is the fraction of energy loss per unit area, \( k \) is the thermal conductivity of the specimen, \( d \) is the average sample thickness, \( r \) is the radius of the beam and \( R \) is the separation between scan centre and surrounding holder.

Intense beams with diameter of \( \sim 6 \mu \text{m} \) and current of up to 1 nA were applied by us to \( \sim 1 \mu \text{m} \) thick elvimide nylon \( (C_{12}H_{22}N_2O_2)_n \) foil mounted over a 10 mm diameter hole in the target holder. The estimated temperature rise for a 6 \( \mu \text{m} \) diameter steady 3 MeV proton beam was 95°. If the same beam is scanned over 100x100 \( \mu \text{m}^2 \) the temperature will be reduced to 61° only, assuming infinite scan frequency. Cookson [3] recently estimated the minimum frequency necessary to observe the temperature reduction.

Different scan frequencies for a continuous scan were applied. The measurements were made with frequencies of 10, 100 and 1000 Hz for the same scan size, beam current and same irradiating particles. Both forward and backward detectors were used simultaneously to monitor the concentration of major targets elements as determined from the yields of scattered particles. We observed no influence of scanning frequency on changes in elemental concentration. Fig.1(a-d) shows the data for a 3 MeV proton beam. Indication of a dose per unit area dependence for elemental losses comes from comparison of data for different scan areas. However Fig.2 indicates also that loss of Hydrogen in nylon is lower for higher dose per unit area. This could be explained by the fact that higher surface temperature blocks the escape of Hydrogen. The experimental yields were fitted by the sum of three exponentials using \( \min \chi^2 \).

The data on elemental yields carried information only on the irradiated area and even these provided insufficient statistics to determine fine structural changes. Therefore the effective foil thickness
Fig. 1
H, C, N and O concentration as a function of charge for (a) steady ~6 μm dia. 3 MeV proton beam on ~1 μm thick nylon foil, (b) 20x20 μm² scan, (c) 50x50 μm² scan and (d) 100x100 μm² scan.

Fig. 2
H concentration as a function of charge per unit area for (b) 20x20 μm², (c) 50x50 μm² and (d) 100x100 μm².

Fig. 3
STIM image of 1 μm thick nylon foil shows effects produced by previous high intensity irradiation with 2 MeV α⁺ beam scanned over 24x24 μm².
after irradiation was mapped by STIM (Scanning Transmission Ion Microscopy) [4]. Data taken with STIM after irradiation of a nylon foil with a 2 MeV alpha beam scanned over ~24x24 μm² are shown in Fig.3. Data were smoothed with a gaussian of 0.3 μm fwhm. The 2.37 μC scan region is thicker then the surrounding area. Three or four rings can be seen surrounding the scan area. The thinnest region in the STIM scan occurs just outside the 24x24 μm² scan, in a border of width about 3 μm. The difference between the valleys and peaks in the rings is typically 0.16 μm. The peaks are thicker than undisturbed nylon far away from the scanned area and the valleys thinner.

X-ray Microbeam

Intense x-ray microbeams from the Light Source at Brookhaven National Laboratory may reach intensities up to 10⁹ photons/μm²s of monoenergetic photons. During a typical analysis time of 100 s, the calculated energy deposition for 10 keV x-rays in 1 μm thick nylon foil would be ~ 10⁶ J/g. If one compares the average energy deposition for 3 MeV H⁺ and 10 keV x-ray beams in order to obtain the same MDL, the energy deposition is ~ 10³ less for x-rays. For 1 nA current of 3 MeV H⁺ beam focussed to a 2 μm diameter on a 1 μm thick nylon and with (R/r)=100 the temperature rise would be ~ 110°C. For the same flux (2×10⁹ photons/μm²s) of 10 keV x-rays the temperature rise would be ~ 0.1°C only. Use of the white x-ray spectrum should increase this to ~ 1°C.

A white beam from NSLS was focussed and collimated to a spot of 80 μm diameter on a target foil of 1.5 μm thick nylon. With only ~ 10⁷ photons/μm²s a hole was developed after about 300 s. The use of Au coated nylon foils did not lessen target damage, suggesting that the damage was not thermal but mostly ionization induced.

Conclusions

The results with proton and x-ray microbeams show clearly that there are limits on how large a flux of particles can be applied over a small area. Thermal, ionization and structural damage are characteristic parameters for each organic material and the differences in resistance could be large. Elemental losses and distortions of the target might introduce errors in precise quantitative and even qualitative analysis, especially if damage is not uniform over the scan area. It is essential to obtain more precise information about influence of damage on quantification of the data received. The STIM technique is ideal for such a task.

REFERENCES

1. M.Cholewa & G.J.F. Legge  
2. Y.Talmon & E.L. Thomas  
   *J. Microsc.*, 111 (1977)151
3. J.A.Cookson  
Distribution of Cu, Fe and Zn in Brindled and Normal Mouse Kidney Tissue

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Abstract
A proton microprobe has been utilised to determine the distribution and relative concentration of Cu, Fe and Zn in 10 day old normal and brindled mouse kidney tissue. High Cu levels were measured in the brindled mutant kidney confirming previous whole tissue results. Differential staining techniques were used to distinguish between proximal and distal tubules within the kidney cortex of mutant male mice, and Cu accumulations were demonstrated in the proximal tubule, whereas the Cu levels in the distal tubule were found to be normal. The distributions of Fe and Zn were found to be similar for normal and mutant kidney tissue. Accurate determination of the Cu distribution in the mutant kidney will provide further information on where the defective reabsorption of Cu is occurring, and may lead to a better understanding of the homologous human condition.

1 Introduction:
Cu is a multifunctional element, essential for the activity of at least ten metalloenzymes, each with important roles in the body. The dietary requirement for Cu arises from various physiological processes which are Cu dependent, such as i) erythropoiesis, ii) development of connective tissue, bone and of the central nervous system, iii) immunocompetence or the body's resistance to infection, and iv) pigmentation in the body [1].

A genetically induced defect affecting a specific step in Cu transport is far more amenable to systematic study than an overall Cu-deficiency. One profound inherited disorder of Cu metabolism in man is Menkes' disease. In 1962 Menkes reported a new familial disease characterised by progressive cerebral degeneration, abnormal spirally twisted hair, a failure to grow and thrive and an X-linked inheritance [2].

In 1972 Danks recognised that a Cu deficiency could describe the phenotype, and then showed that this was the case in Menkes' disease [3,4]. The study of Menkes' disease was greatly augmented by the discovery of mottled mice as an animal model [5,6] with distinct similarities in the disturbances of Cu transport and also with mutations affecting the X-chromosomal genes at the same gene locus. Brindled mice are one of the series of differing severities of the mutation in mottled mice which survive from 14 to 16 days post partum.

Measurements of whole kidney tissue Cu levels in brindled mice have shown an average Cu concentration of about 3 times higher than for normal mice [7,8]. A high resolution elemental microanalysis of thin sections of brindled and normal mouse kidney tissues has been carried out in order to determine the distribution and relative concentration of Cu in these tissues and how these are affected by the mutation.

2 Experimental Methods:
The mice were killed by cervical dislocation and the kidneys were excised from 4 suckling ten day old normal mice and from 5 ten day old brindled male mice. The kidney tissues were cut in half, placed in an Al specimen holder and then rapidly frozen in liquid nitrogen-cooled propane. The time interval between excision of the kidney tissue from the mouse and freeze fixation of the tissue was kept to a minute or two, to minimise the redistribution of ions in the tissue. The integrity of the in vivo elemental distributions in the tissue must be maintained throughout the different stages of sample preparation and during irradiation by the ion beam. Cryoprotectants or chemical fixatives were not used. The frozen tissue was then cryosectioned at −20 ± 1

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Four micron thick sections were obtained to optimise the X-ray yield for trace elements and to provide a uniform anatomical profile through the tissue section. Normal procedure entailed sectioning five serial sections, so that the first, third and fifth could be stained conventionally, the second and fourth then being prepared for analysis. Enzyme histochemical techniques were also used for differential staining of the kidney tubules, which enabled identification of the proximal and distal convoluted tubules [9].

The second and fourth frozen hydrated sections were freeze dried under high vacuum conditions, and then stored in a desiccator over silica gel. The elemental microanalyses of the freeze dried kidney tissue sections were carried out using a 3 MeV proton beam focused to a beam spot diameter of usually 2 to 3 \( \mu m \). Beam currents averaged 200 pA and were scanned rapidly and uniformly over areas ranging from 100 \( \times \) 100 \( \mu m^2 \) to 240 \( \times \) 200 \( \mu m^2 \), for irradiation times varying from 7 to 16 hours. X-rays were detected by an energy dispersive Si(Li) detector, digitised and stored on magnetic tape in coincidence with the X, Y scan coordinates. A high energy window was used at 5 keV providing a bremsstrahlung yield to which the Fe, Cu and Zn X-ray yields were normalised, enabling relative concentrations of these metals to be compared from one specimen to another.

3 Results:
Numerous sections of both normal and mutant mouse kidney were irradiated in order to analyse the distributions of the trace metals Fe, Cu and Zn. In one case a 150 pA beam of 3 MeV protons was scanned over an area of 100 \( \times \) 110 \( \mu m^2 \) for 16 hours, where fig.1 shows the schematic of the irradiated mutant kidney tissue section with the characteristic X-ray yields of K, Fe, Cu and Zn. The section was self supporting over the scanned area, although there was some relative movement of the tubules due to the impinging beam, producing gaps in the tissue next to two tubules arrowed in the schematic in fig.1. Fe is particularly high in tubules 1 and 3 and fairly uniform elsewhere, Cu is also high in tubule no.1, but less so for tubule no.3, and Zn is widely distributed. Frequency distributions for the relative trace metal concentrations for each tubule cross-section were obtained [9] and the distribution of Cu is shown in fig.2.

The normal Cu distribution shown in fig.2 extends up to a relative concentration (Cu/bremsstrahlung) of 0.23, where the lower portion of the mutant Cu distribution extends up to much higher concentrations than those for the normal distribution – those concentrations > 0.42 have been grouped into one interval since the maximum extends up to 1.6. From the use of two enzyme stains [9] for a series of 5 irradiations of mutant kidney tissue, 17 tubule cross-sections out of 72 were identified as distal tubule cross-sections (those labelled 2, 4, 5 and 6 in the schematic in fig.1 were found to come from distal tubules). All of these distal tubules had concentrations within the range of the normal distribution. The frequency distributions of the relative concentrations of Fe and Zn were found to be very similar for both mutant and normal kidney tissue [9].

4 Discussion:
The primary result of this work is that the higher range in concentration of the mutant brindled Cu distribution is attributed to having come from cross-sections of the proximal tubule. Further work for this project would entail either differential staining or examining ultrastructural detail to distinguish different segments of the proximal tubule and correlate these segments with associated Cu levels. If different segments can be shown to have different Cu transport pathways, then this may help to localise the defective step or steps in Cu utilisation in the brindled mouse.

5 Conclusion:
A Cu concentration in brindled mouse kidney tissue about 5 times the normal level has been observed, supporting earlier work. The proton microprobe microanalyses of thin sections of kidney cortex have revealed accumulations of Cu which were shown to be localised to within the proximal tubule of the brindled mouse kidney tissue. In addition, a non-uniform Fe distribution in both normal and mutant tissue was observed.

6 References:


Figure 1: Schematic of irradiated area of mutant kidney tissue, and maps of the K, Fe, Cu and Zn characteristic X-ray yields.

Figure 2: Distribution of the concentration of Cu in the normal and mutant suckling mouse kidney (light = mutant, dark = normal).
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AUSTRALIAN FACILITIES
FACILITIES AVAILABLE THROUGH ANSTO AND AINSE

ANSTO has a wide range of facilities available for research purposes. These are designed to enable full application of Ion Beam Analysis (IBA) techniques as well as other reactor based methods. IBA techniques such as PIXE, PIGME, RBS and NRA are available on the 3 MV Van de Graaff accelerator. This is capable of accelerating protons, helium ions and selected heavy ions (Ne, N). Measurements are supported by extensive computer programs for data analysis and interpretation. Large databases of nuclear parameters are available. A milliprobe with beam diameter between 20 and 50 micrometers can be used for scanning small samples.

1. **PIXE** (Proton Induced X-ray Emission) is a multielement trace analysis technique which has had wide application at ANSTO. Typical uses include detection at parts per million concentrations of elements (Si to U) in aerosol filters, archaeological artefacts and samples taken for ambient environmental monitoring and occupational exposure.

2. **PIGME** (Proton Induced Gamma ray Emission) is a useful nondestructive method for measuring low concentrations of light elements such as Li, F, Na, Al and Mg. It can be performed simultaneously with PIXE or RBS measurements.

3. **RBS** (Rutherford Backscattering) is based on a simple model for scattering particles. At ANSTO it has been applied to surface studies, particularly of semiconductor materials and polymer coatings on metals. Useful for profiling heavy elements in a light matrix.

4. **NRA** (Nuclear Reaction Analysis) has been used for the determination of hydrogen, carbon, nitrogen and oxygen in a variety of materials. It is generally considered complementary to RBS profiling light elements in a heavy matrix such as hydrogen, carbon and nitrogen in steels.

5. **SNIF**: Standard Neutron Irradiation Facility: One leg on the 3 MV Van de Graaff has been dedicated to neutron irradiation work. Various nuclear reactions are used [Be(d,n), Li(p,n) etc.] to produce standard, well characterised neutron beams for irradiation and neutron damage studies. Currently, we can supply $3 \times 10^8$ neutrons cm$^{-2}$ s$^{-1}$ with energy from 10 keV to 10 MeV with absorbed neutron doses of up to 10 K rad h$^{-1}$.

6. **Moata Reactor**: 100 kW maximum power, $1.2 \times 10^{12}$ neutrons cm$^{-2}$ s$^{-1}$ maximum thermal neutron flux. Two irradiation facilities exist in maximum flux regions for small volume irradiations. Positions in the external reflector ($5 \times 10^{11}$ neutrons cm$^{-2}$ s$^{-1}$) and thermal columns ($10^{10}$-$10^{11}$ neutrons cm$^{-2}$ s$^{-1}$) are available for larger volume irradiation.

7. **Uranium Analysis**: Using delayed neutron method. For 10 g samples, sensitivity 0.5 µg natural uranium. 50 samples/hour. 48 hour turnaround service on demand.
8. Neutron Activation Analysis: 0.2 to 0.5 g samples, encapsulated in disc geometry can be irradiated in batches of 35. Counting and data processing of activated samples is available. Typical elemental analysis include Th (0.5 ppm), Rare Earths (0.2 ppm). About 30 elements can be assayed at or below the 1 ppm level.

9. Fission Track Analysis: Fission track dating packages and fission track mapping assemblies can be irradiated in the maximum flux region of Moata. Typical uses are mapping the distribution of uranium in mineral grains or granites or uranium ores. Measuring the ages of opaline grains to aid oil exploration.

10. Neutron Radiography: A well collimated beam, diameter 33 cm, strength $4 \times 10^5$ neutrons cm$^{-2}$ s$^{-1}$ available on Moata, together with dark room facilities. Commercial quality neutron radiographs can be produced in an exposure time of 7 minutes for a film size of 20 x 25 cm. Typical uses to date include - examination of gas cooled turbine blades, aircraft components, pyrotechnic devices.

11. 200 kV - 1.3 MV Van de Graaff Electron Accelerator: Produces both d.c. and pulsed electron beams. d.c.: 300 μA pulsed: 10 mA/3 μs pulse Comes with a PDP11/23 dual floppy disk drive data acquisition system capable of digitising down to 100 ns transient pulses.

12. 600 kV Febetron: 3 ns pulsed electron source capable of delivering 10 J in a single pulse. Also comes with a fast data acquisition system which digitises 10 ns pulses and stores them on a PDP11/23 with dual floppy disk drive.

13. High Energy IBA: This will include activation and high energy nuclear reactions and heavy ion reactions at terminal voltages between 3 and 8 MV or ion energies substantially above 3 MeV. Typical uses include C and N in steels and H in polymer coatings.

14. Accelerator Mass Spectrometry (AMS): A FN Tandem accelerator is being installed at ANSTO laboratories at Lucas Heights to concentrate on all aspects of Accelerator Mass Spectrometry. The accelerator has been specially designed to provide the highest possible accuracy and sensitivity for $^{14}$C and $^{10}$Be measurements. Up to 50% of the time on the accelerator will be made available for AMS.

ANSTO has supplied considerable resources to an ANSTO/ANU joint venture for AMS. Special clean rooms are under construction at ANSTO for the preparation of samples. Currently $^{35}$Cl, $^{10}$Be and $^{14}$C can be done through the joint ANSTO/ANU collaboration.

For Australian university groups these facilities can also be made available through AINSE.

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ANSTO ISOTOPE TECHNOLOGY PROGRAM
and
AUSTRALIAN FEDERAL POLICE

Facilities for Thermal Neutron Activation Analysis

1 Irradiation Facility (HIFAR)
1 X-6 neutron flux \( \sim 5 \times 10^{12} \text{n.cm}^2\text{s}^{-1} \)
2 X-176 neutron flux \( \sim 5 \times 10^{13} \text{n.cm}^2\text{s}^{-1} \)
(Fast access facility - short irradiation only)

2 Gamma Ray Spectrometers
1. Intrinsic Germanium Planar Detector
   Active Area: 500 mm\(^2\)
   Thickness: 5 mm\(^2\)
   Energy Resolution: 378 eV @ 6.4 keV
   568 eV @ 122 keV

2. Intrinsic Germanium Coaxial Detector
   Energy Resolution: 1.8 keV @ 1.332 MeV
   Efficiency: 18%

3. Intrinsic Germanium Coaxial Detector
   Energy Resolution: 1.8 keV @ 1.332 MeV
   Efficiency: 15%

4. Intrinsic Germanium Coaxial Detector
   Energy Resolution: 1.8 keV @ 1.332 MeV
   Efficiency: 18%

5. Lithium Drifted Germanium Closed End Coaxial Detector
   Energy Resolution: 2.3 keV @ 1.332 MeV
   Efficiency: 13%

Facilities for Radiochemical Analysis

1 Double Laboratory Suite
1 Ten Fume Cupboards
2 Laminar Flow Cupboard

2 Counting Equipment
1 Low Resolution Gamma-Ray Spectrometer (3" NaI(Tl) Detector)
2 Quad Alpha Spectrometer
Analytical Services Offered
Facilities for thermal neutron activation and radiochemical analysis are offered to outside organisations on a limited and selective basis.

Further Information
Mr J. J. Fardy,
Advanced Analytical Methods Project,
ANSTO Isotope Technology Program,
Lucas Heights Research Laboratories,
Private Mail Bag 1
MENAI NSW 2234
The CSIRO HIAF facility is an analytical laboratory based on a 3 MV Tandetron tandem electrostatic accelerator, commissioned primarily to develop applications of Ion Beam Analysis (IBA) in minerals research and industry. The laboratory is designed to enable applications of the complete suite of IBA (PIXE, RBS, NRA) as well as Accelerator Mass Spectrometry (AMS).

The IBA techniques can be performed with microbeams from the HIAF microprobe. It is based on an electrostatic "Russian quadruplet" lens, which facilitates focusing of heavy ion beams, e.g. F19 for hydrogen profile measurements and other heavy ions for e.g. selective X-ray excitation, in addition to the usual H and He4 for PIXE and RBS. For H beam ample beam currents are obtained with resolution down to 5 microns. The system features:

- a normal viewing optical system, colinear with the beam, featuring a reflecting objective and a 150X total magnification. Samples are viewed at the beam side; an essential feature for mineralogical applications.
- an XYZ micromanipulator for the sample stage.
- rapid sample change through a vacuum lock.
- Si(Li) detector with externally selectable set of filters to optimize detection sensitivity.
- a particle detector at back angles for RBS and resonant RBS.
- Ge detector and close geometry NaI detector for NRA.
- a semi-focusing WDS spectrometer.
- UHV standard.
- a complete suite of software for PIXE and RBS analysis, including a general spectra manipulation program.

At present the AMS system will enable measurements of light cosmogenic radioisotopes (Cl4, Be10), using a HICONEX sputter source. The AMS system features:

- a separate injection system to enable optimization of the source and accelerator acceptance matching, as well to minimize operational interference with the IBA measurements.
- a 12 degree electrostatic analyzer in addition to the 90 degree and 22.5 degree magnetic analysis.
- a gas proportional telescope counter for particle identification.

A microprobing Cs sputter source is currently being developed to enable in-situ measurements with a spatial resolution better than 50 microns. Future developments include a time-of-flight spectrometer for detection of heavy elements.

Contact: Dr. S.H. Sie 887-8648, 887-8666 Fax:887-8909
An Accelerator Mass Spectrometry (AMS) capability has been established on the 14UD accelerator in the Department of Nuclear Physics at the Australian National University.

The technique has been established for the following isotopes:

**Chlorine-36**  
This naturally-occurring radio-isotope is proving very useful as a tracer of water and salt movement in the environment. It can be detected with a sensitivity of 1 part in $10^{15}$ (atoms of $^{36}$Cl to atoms of stable chlorine). Levels in groundwater are typically 20-200 parts in $10^{15}$. Samples containing as little as 5mg of Cl$^-$ can be measured. Preparation of samples entails conversion to AgCl and purification to remove sulphur.

**Carbon-14**  
The applications of $^{14}$C to the dating of archaeological artifacts are well known, but it may also be a useful tracer and has been widely used in oceanography and hydrology for example. The chief advantage offered by AMS over conventional radioactive-decay measurements is a substantial reduction in sample size. Samples containing 1mg of carbon can be measured. At present it is possible to determine the $^{14}$C content of samples down to $\sim$1% modern.

**Beryllium-10**  
Beryllium-10 attaches readily to soil and sediment particles and is therefore a useful tracer for erosion and sedimentation studies. Most rock and soil types contain very low levels of stable beryllium, and hence a carrier is usually added to extract the beryllium-10 in the form of BeO. Two milligrams of BeO is sufficient for a measurement.

This project is an ANU/ANSTO collaboration, and further information on applications, sample preparation and the possibilities for other isotopes may be addressed to Keith Fifield(ANU) or Raoul Davie(ANSTO) at

Department of Nuclear Physics  
Research School of Physical Sciences  
Australian National University  
GPO Box 4, Canberra, ACT 2601  
Telephones:  
(062) 49 2095 (LKF)  
(062) 49 2091 (RFD)  
(062) 49 2083 (Sec)  
Fax:  
(062) 49 0748
UNIVERSITY OF NEWCASTLE - Department of Physics

100kV Accelerator - 2 beam lines mass analysed.

- beam line A medium energy ion scattering
- beam line B recoil implantation target chamber equipped with SIMS for depth profiling
- can be used for direct ion implantation.

2 x Low Energy Ion Scattering

- +ve and -ve energy analysis
- angle resolving goniometer
- 5 kV ion gun

- target manipulator
- heating and cooling stages + 2 degrees rotational freedom

Auger Microprobe Spectrometer

- 3 μ beam spot
- photoelectron spectrometer

SIMS System

- 100 μ spot size - fast profiling

Surface Profilometer

- Alpha - step

Contact

Prof. R J MacDonald Telephone: (049)685 236/237
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Dr B V King (049)685 387
Department of Physics
University of Newcastle
NEWCASTLE, NSW 2308
The nuclear techniques specific to this laboratory are ancillary to the thermoluminescence dating programme. In the present context the item of interest is a very low background field scintillometer, calibrated to record K, U and Th concentrations at ppm level and total dose rates in mGy.a\(^{-1}\).

**contact**

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Adelaide, 5001.  
FAX (08) 224 0464
MICRO ANALYTICAL RESEARCH CENTRE
SCHOOL OF PHYSICS, THE UNIVERSITY OF MELBOURNE

MARC is set up to:

- provide a microanalytical service to industry
- undertake projects or consultations for industry
- undertake collaborative research with industry and research institutes
- undertake basic research and development in microanalysis, ion optics, instrumentation and data handling
- manufacture and export microanalytical equipment

Facilities for non-destructive trace elemental mapping and depth profiling

5 MV Pelletron Accelerator is coupled to a Scanning Proton Microprobe of 1 micron lateral resolution instrumented for:

- PIXE, RBS, RFS and NRA analysis
- Channeling Contrast Microscopy (CCM)
- High resolution (50 nm) Scanning Transmission Ion Microscopy (STIM)
- Channeling STIM (CSTIM)
- 2-dimensional and 3-dimensional STIM tomography
- Secondary Electron Microscopy
- Transmission and Reflection Light Microscopy and Photomicrography
- Total Quantitative Scanning Analysis (TQSA) with
- High Resolution 3-dimensional Colour Graphics
- Clean Room Facilities for Specimen Handling

Areas of Application

- Geology and Mineralogy
- Metallurgy and Materials Analysis
- Semiconductors
- Forensic Science and Industrial Science
- Chemistry and Environmental Science
- Agriculture and Forestry
- Botany and Biochemistry
- Zoology and Genetics
- Cytology and Haematology, etc

Microprobe Development

UHV microprobe components and assemblies are developed and marketed world wide. Completely instrumented microprobes with full hardware and software are exported and installed.

Other Facilities

The 5 MV Pelletron Accelerator is available for other applications
Ultrahigh resolution electron microscopy, electron and x-ray diffraction and electron microprobe analysis available through NAMAC and other departmental facilities.

Contact

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Ion Beam Facilities and Contacts

1. IMV Tandetron Accelerator

Under normal operation provides 600keV to 3MeV He+ and He++ ions at beam currents (on target) up to 400nA

- Routine Rutherford backscattering and channeling analysis: various goniometers and manipulators are available

- Helium-induced X-ray analysis can be performed using Si-Li and Ge-Li detectors

- Analyses available on request

   Contact person: Mr Robert Short (03) 660 2692

2. Sputter Profiling Using Auger Electron Spectroscopy

Routine sputter profiling can be performed on most targets under UHV conditions using Auger Electron Spectroscopy. Automatic data analysis facilities are available providing a direct readout of elemental composition vs. depth.

Analyses available on request.

   Contact person: Mr Peter Paterson (03) 660 2136

3. Ion Implantation

Two high current ion implanters are available for implanting most elemental species. Energy range 10-200keV. Odd shaped samples or components can be treated.

Service work available on request.

   Contact person: Mr Pier Bond (03) 660 2450