ION BEAM ANALYSIS OF METAL ION IMPLANTED SURFACES

by

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ABSTRACT

Ion implantation is an established method for altering the surface properties of many materials. While a variety of analytical techniques are available for the characterisation of implanted surfaces, those based on particle accelerators such as Rutherford backscattering (RBS) and nuclear reaction analysis (NRA) provide some of the most useful and powerful for this purpose. Application of the latter techniques to metal ion implantation research at Ansto will be described with particular reference to specific examples from recent studies. Where possible, the information obtained from ion beam analysis will be compared with that derived from other techniques such as Energy Dispersive X-ray (EDX) and Auger spectroscopies.

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INTRODUCTION

Ion beam analysis (IBA) offers a number of powerful diagnostic tools for the characterisation of modified surfaces. By means of Rutherford backscattering (RBS), nuclear reaction analysis (NRA) and elastic recoil detection (ERD), it is possible to determine the elemental composition of the near surface region as a function of depth. This capability makes them particularly useful for the analysis of thin films and implanted surfaces.

In 1989, Ansto's Applications of Nuclear Physics Program commenced a project on metal ion implantation (MII) research, primarily directed at engineering materials. The stimulus for this undertaking was the success achieved, in the years immediately preceding it, by several groups in developing high current metal ion sources. One such source, first described by Brown and co-workers[1] was that based on a metal vapour vacuum arc (MEVVA). This type of source, which appeared to offer both simplicity and reliability, is capable of generating ions of a wide range of metallic materials together with some semiconductors (i.e. Si and Ge) and non-metals (i.e. C). In addition, its high ion beam currents make it particularly suitable for high dose applications. For these reasons, the MEVVA ion source was selected for our work.

The initial phase of the MII project involved development of an ion implanter equipped with a MEVVA ion source. The completion of this phase in early 1991 marked the beginning of our applications oriented research which has included detailed studies on the MII of several systems. During this latter phase, IBA has been extensively used to determine implant distributions and to characterise surfaces subjected to post implantation testing. The purpose of the present note is to illustrate this work with particular reference to studies on specific systems.

EXPERIMENTAL

The implantation system and experimental procedure have been described in detail previously[2,3]. Specimens were polished to 1/4μ diamond and ultrasonically cleaned prior to implantation. The ion source was operated at ion beam currents (pulsed) of 40 - 100 mA, pulse repetition frequencies of 1 - 10 Hz and extractor voltages of 20 or 25 kV. The latter in conjunction with the average ion charge states[4] yielded mean ion energies in the range 40 - 60 keV. Ion doses ranged from 3.5×10^{14} - 1.5×10^{17} cm^{-2} for tungsten implanted GC and between 1×10^{15} - 2×10^{17} cm^{-2} type 2011 Al alloy implanted with Ti and Cr ions.

RBS spectra of implanted substrates were obtained using a 2 MeV $^4He^+$ incident beam, which was normal to the target, and a scattering angle of 169°. The oxidation of metal surfaces has been monitored by measuring the alpha particle yield from the reaction $^{16}O(^3He,\alpha)^{15}O$. A beam of 2.4 MeV ^3He ions was directed onto the targets which were inclined at 45° to the incident beam and the charged particle spectra measured at 90°. Further details of the oxygen measurements appear elsewhere[3]. Finally, elastic recoil detection (ERD) of the hydrogen distribution in specimens exposed to an rf plasma was performed using a 2.5 MeV ^4He beam.

RESULTS AND DISCUSSION

Recently, we have investigated the implanted of glassy carbon (GC) with tungsten ions focussing on both the wear properties and surface characterisation of the modified material. In an extension of this work, W implanted GC has been exposed to an rf hydrogen plasma for varying times. The hydrogen content of the exposed surfaces was then determined by ERD. For W implanted GC, the

hydrogen content after a 1 hour exposure was found to be 10 ± 10%. In contrast, C^+ implanted GC yielded a corresponding increase of 30 ± 15%. The lower hydrogen content in the tungsten modified material suggested the observed inhibition was due to a chemical effect which slowed the formation of hydrocarbons thereby reducing surface erosion.

Aluminium and its alloys are one of the most widely used group of metals. While generally exhibiting good corrosion resistant properties, specific alloys are susceptible to corrosion in some applications. For this reason, various protective treatments, including ion implantation, have been investigated as a means of improving the operational performance of these metals. In alloys of the 2000 series, copper is the main alloying element with percentage compositions by weight in the range 5.0 - 6.0%. Recently, we investigated the oxidation of this alloy implanted with Ti and Cr ions[3]. The oxygen uptake as a function of time at 500°C was monitored using nuclear reaction analysis. RBS analysis was also performed on both the as-implanted and oxidised specimens. Typical RBS spectra for Ti and Cr implanted substrates, prior to and after oxidation for 1 hour, are shown in Figs. 1 and 2 respectively. The main features of note here are the redistribution of particular alloying elements (Pb/Bi and Cu) and differences in the behaviour of the implant ions following high temperature oxidation. In order to distinguish between purely thermal effects and those associated with the oxidation process, Ti and Cr implanted specimens were vacuum annealed at 500°C for 1 hour. RBS spectra of the as- implanted and annealed surfaces are presented in Figs. 3 and 4 for Ti and Cr respectively. These spectra indicate that the redistribution of Cr, Cu and Pb/Bi apparent in the oxidised samples (Figs. 1 and 2) is largely due to thermal effects. Furthermore, comparison of the results in Figs. 3 and 4 with the spectrum of a vacuum annealed virgin sample of 2011 Al shown in Fig. 5 suggests that the redistribution of Cu and Pb/Bi occurred to a greater extent in the implanted material. This is attributed to the presence of ion beam induced damage in the implanted layer which promotes diffusion or segregation of particular alloying elements.

CONCLUSION

In the foregoing, we have described the application of IBA techniques to the characterisation of ion implanted surfaces. Either individually or in conjunction with other surface analytical methods, techniques such as RBS, NRA and ERD are capable of providing key information on the nature of modified surfaces. Our studies of 2011 Al oxidation and annealing offer a good illustration of this approach. In this instance, RBS measurements were combined with scanning electron microscopy and EDX analysis to distinguish between different oxidative and thermal processes from which we were able to propose a model for the structure of the oxidised implanted surface.

ACKNOWLEDGEMENTS

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REFERENCES

1. I.G. Brown, J.E. Galvin, B.F. Gavin and R.A. MacGill, Rev. Sci. Instrum., 57, 1069 (1986).
2. A. Hoffman, P.J. Evans, D.D. Cohen and P.J.K. Paterson, J. Appl. Phys., 72, 5687 (1992).
3. J.W. Chu, N. Dytlewski, P.J. Evans and D.K. Sood, Nucl. Instrum. Meth. B, B80/81, 289 (1993).
4. I.G. Brown, B. Feinberg and J.E. Galvin, J. Appl. Phys., 63, 4889 (1988).

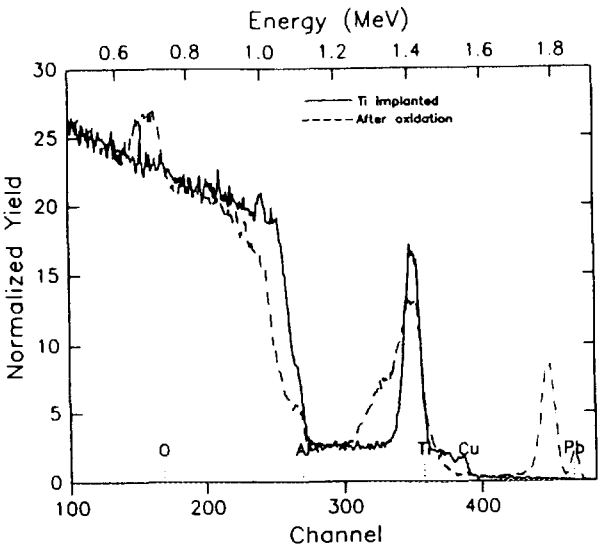

Figure 1 - RBS spectra of Ti implanted (dose = $2x10^{17}$ cm^{-2}) 2100 Al specimen prior to (solid line) and after 1 hour oxidation at 500°C (broken line).

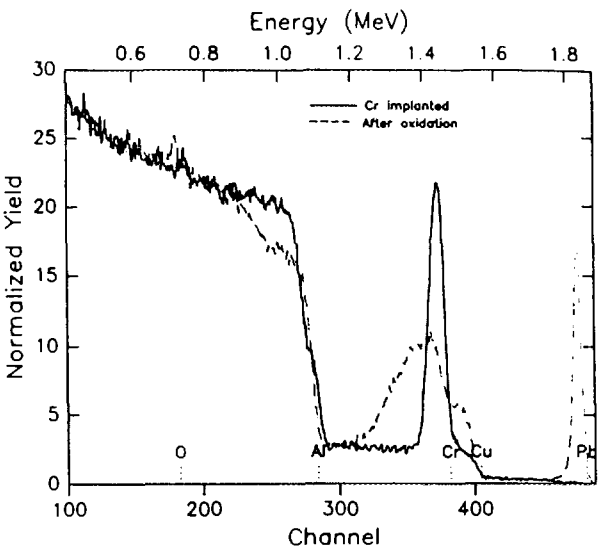

Figure 2 - RBS spectra of Cr implanted (dose = $2x10^{17}$ cm^{-2}) 2100 Al specimen prior to (solid line) and after 1 hour oxidation at 500°C (broken line).

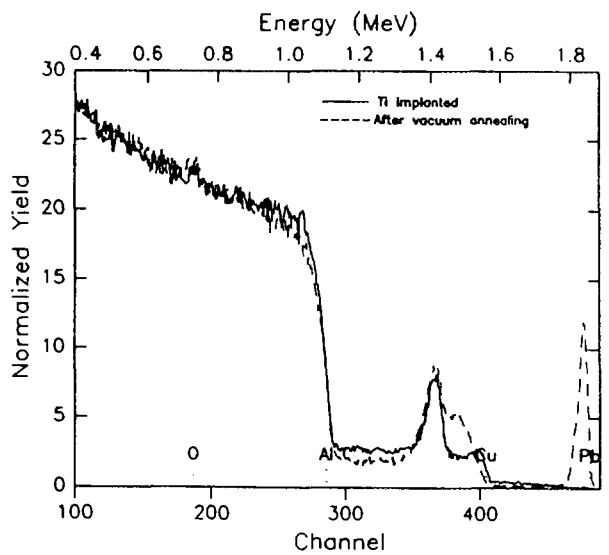

Figure 3 - RBS spectra of Ti implanted (dose = $1x10^{17}$ cm^{-2}) 2100 Al specimen prior to (solid line) and after 1 hour vacuum anneal at 500°C (broken line).

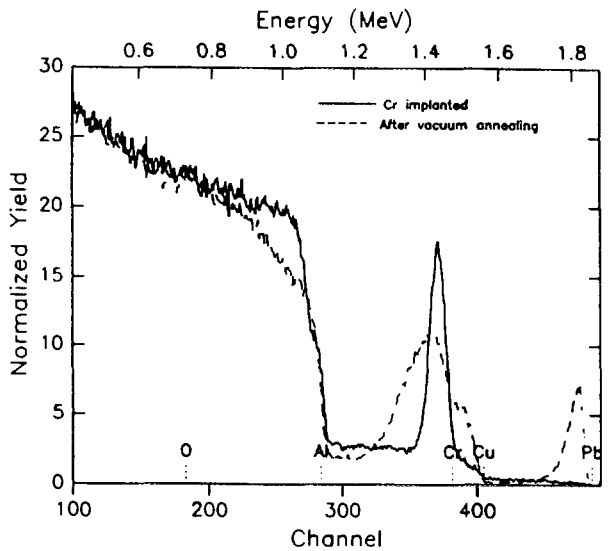

Figure 4 - RBS spectra of Cr implanted (dose = $1.5x10^{17}$ cm^{-2}) 2100 Al specimen prior to (solid line) and after 1 hour vacuum anneal at 500°C (broken line).

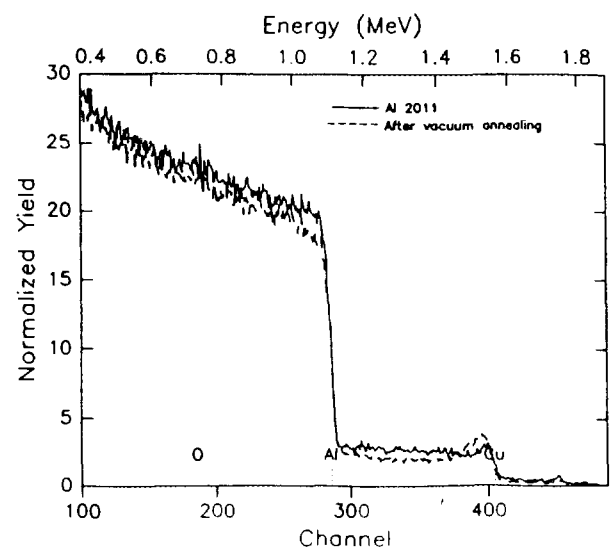

Figure 5 - RBS spectra of polished virgin 2100 Al specimen prior to (solid line) and after 1 hour vacuum anneal at 500°C (broken line).