AECL-9381

ATOMIC ENERGY OF CANADA LIMITED

L'ÉNERGIE ATOMIQUE DU CANADA LIMITÉE

DEVELOPMENT OF ADVANCED CERAMICS AT AECL

Réalisation de matériaux céramiques avancés á l'EACL

B.J.F. PALMER, P.J. HAYWARD, S.R. MacEWEN, B.D. SAWICKA and S. SRIDHAR

Presented on 1986 November 4 to the
Second Workshop of the Canadian-University Council on Advanced Ceramics held in Toronto

Chalk River Nuclear Laboratories

Laboratoires nucléaires de Chalk River

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RÉSUMÉ

L'Énergie Atomique du Canada, Limitée (EACL) réalise depuis longtemps des matériaux céramiques pour les applications en fission et fusion nucléaires. Elle applique maintenant ses aptitudes en R et D multidisciplinaires sur les matériaux, dont ses aptitudes uniques en traitement des matériaux céramiques et évaluation non destructive, afin de réaliser des matériaux céramiques avancés pour les applications commerciales et industrielles. Le présent rapport donne un aperçu des moyens, installations et programmes associés à la réalisation des matériaux céramiques avancés à l'EACL.

Présenté le 4 novembre 1986 au Deuxième Atelier du Conseil des Universités Canadiennes pour les Matériaux Céramiques Avancés tenu à Toronto.

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ABSTRACT

Atomic Energy of Canada Limited (AECL) has a long history of developing ceramics for nuclear fission and fusion applications. AECL is now applying its multidisciplinary materials R&D capabilities, including unique capabilities in ceramic processing and nondestructive evaluation, to develop advanced ceramic materials for commercial and industrial applications. This report provides an overview of the facilities and programs associated with the development of advanced ceramics at AECL.

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1. INTRODUCTION

Atomic Energy of Canada Limited (AECL) is a founding industrial member of the Canadian University Industry Council on Advanced Ceramics. It is actively involved in commercial development of advanced ceramics for nuclear and non-nuclear applications. Work is focussed on process development and nondestructive evaluation of new ceramic-based materials.

2. CORPORATE CHARACTER

AECL is a federal Crown Corporation accountable to the Minister of Energy, Mines and Resources and has an independent Board of Directors.

The development of advanced ceramics is carried out by AECL's Research Company, which operates two laboratories -- Chalk River and Whiteshell. Both offer comprehensive facilities for multi-disciplinary R&D.

Chalk River, located 190 km (2.5 h) northwest of Ottawa, employs about 2000 people and was established in 1944. Whiteshell, established in 1963, employs about 1000 people and is located 105 km (1.5 h) northeast of Winnipeg.

The Research Company places considerable emphasis on developing and exploiting commercial technology in cooperation with Canadian industry. This is done through contract R&D, technology licencing, business spin-outs and contractual joint ventures. The Research Company has recently restructured to encourage innovation and facilitate business development.

3. PROCESSING

3.1 Programs

AECL has been developing ceramic materials and processes for over 35 years. It established the Canadian nuclear ceramics industry by developing powder production and pellet fabrication technology and transferring it to Canadian companies. In addition to developing nuclear fuel, which is a ceramic material, AECL has also done considerable work on the development of ceramic pressure tubes.

Currently, work on the development of ceramic materials and processes goes on at both Chalk River and Whiteshell. It is organized into the following programs:

1. Nuclear ceramics	(Chalk River)
2. Nuclear waste immobilization	(Whiteshell)
3. Nuclear waste encapsulation	(Whiteshell)
4. Fusion ceramics	(Chalk River)
5. Commercial/Industrial ceramics	(both sites)

which are outlined in the following paragraphs.

Nuclear ceramics:

The production of nuclear fuel is possibly Canada's largest and most established advanced ceramics industry (over 40 M$/year, not including the costs associated with raw materials). At AECL, ceramic processing work on nuclear fuel is directed towards developing processes for fabricating advanced nuclear fuels, and developing microstructures that facilitate improved in-reactor performance.

Considerable work has been carried out on developing processing technology for producing thoria-based fuels. Because, in eventual application, the thoria will be mixed with gamma-active U-233, processing must be done remotely, without hands-on intervention. Simple, reliable and controllable processes were developed for producing sols and gelling them into high-density ceramic spheres, and for producing powders and fabricating them into pellets. This program is now completed.

Work is currently focussed on developing special microstructures that would allow urania-based fuels to be used at higher powers for longer times, and on producing urania-based materials that are doped with stable elements to simulate the composition and phase-microstructure of high-burnup nuclear fuel.

Nuclear waste immobilization:

The recycling of used nuclear fuel involves the dissolution of irradiated fuel bundles in nitric acid solutions, followed by solvent extraction to recover reusable fuel materials and separate out highly radioactive fission product wastes. A major part of AECL's Nuclear Fuel Waste Management Program is directed towards developing suitable glassy or ceramic materials that could provide highly insoluble matrices that incorporate and immobilize these high-level wastes.

This program has successfully demonstrated the construction and operation of pilot-scale equipment for immobilizing high-level liquid wastes in glassy materials. The first stage of this process uses the Roto-Spray Calciner, designed and built at Whiteshell, to convert the liquid waste into a powder. This calcined powder is then mixed with glass-forming materials and the mixture is melted in an electric melter and poured into stainless steel cans. This process has been successfully run for about 10 000 hours and can produce glass at 10 kg/h. In addition, continuous gel and slurry-based processes have been developed for the conversion of high-level liquid wastes into calcined powders suitable for incorporation in borosilicate, aluminosilicate, sphene-glass and SYNROC waste forms.

The development of sphene glass-ceramics as potential nuclear waste hosts has been pioneered at Whiteshell. These new materials, which consist of discrete sphene ($CaTiSiO_5$) crystals in a matrix of aluminosilicate glass, exhibit significantly improved chemical durabilities and mechanical strengths compared to borosilicate glasses. Sphene glasses are being prepared by traditional (melting/crystallization) and sol-gel techniques. A 20 L high-temperature melter, featuring advanced refractory and electrode materials, and vacuum removal of the melt, is under development for producing glass-ceramic materials.

SYNROC B is a three-phase ceramic material designed for the immobilization of fuel recycle wastes. A program is underway to optimize the formulation of this material for Canadian applications and to simplify its production by substituting pressureless sintering for hot pressing.

Nuclear waste encapsulation:

The purpose of this program is to develop suitable containment vessels for various types of nuclear waste. Monolithic oxide ceramics are under consideration for this application. The experimental work focusses on evaluating the chemical durability of commercially-available ceramic materials.

Fusion ceramics:

Lithium-based ceramics are favoured by many to make up the blanket that will surround the plasma in an operating fusion reactor. In this blanket, fusion neutrons will react with lithium nuclei to produce tritium gas, which will be collected and purified so that it can be used to fuel the plasma. The blanket will also serve to capture the heat generated by the fusion reaction so that it can be used to generate electricity.

A comprehensive program is underway at Chalk River to develop fusion breeder blanket technology. This program is jointly funded by AECL and the Canadian Fusion Fuels Technology Project. The development of fabrication technology for lithium-based ceramics is a major element of this program. This fabrication work is directed towards producing ceramic materials with microstructures optimized for the production and removal of tritium gas under irradiation. Work is underway on producing high-purity lithium aluminate via powder and sol routes, starting from conventional and alkoxide precursors. Emphasis is placed on producing ceramic spheres by the powder agglomeration and sol-gel methods.

Commercial/Industrial ceramics:

AECL's program on commercial/industrial ceramics began about a year ago, and involves the coordinated efforts of both Chalk River and Whiteshell. The program addresses two tasks:

1. Develop and/or apply advanced ceramic materials in traditional AECL products, such as reactors and accelerators, and

2. Utilize AECL's technology, facilities and expertise to create new products and businesses based on advanced ceramic materials.

In addressing these tasks, strong emphasis has been placed on teaming our R&D and product development skills with the manufacturing, marketing and distribution skills of other companies. AECL would like to enter into contractual joint ventures to exploit many of these opportunities, and has investment funds available to make this possible.

Possible new applications of ceramics in traditional AECL products include:

- ceramic coatings in components subject to wear or corrosion,

- sensors that can operate in severe environments and permit improvements in reactor efficiency,

- new materials for high-performance mechanical seals,
- sodium heat engines (thermoelectric generators) for converting heat from small nuclear reactors into electricity,
- ceramic journals and bearings, and
- advanced CANDU fuel channels with ceramic components and/or ceramic pressure tubes.

The development of new ceramic products and businesses falls into three classifications: contract R&D, spin-off products and new technology.

Contract R&D ranging from simple analyses to comprehensive multidisciplinary programs is available at both sites. All such work is done on a confidential basis.

A number of projects are underway that involve the adaptation (spin-off) of technology developed in our nuclear fuel and waste management programs to non-nuclear applications, including:

- the preparation of stabilized zirconia powders using the Roto-Spray Calciner,
- the production of thoria products such as high-purity crucibles,
- structural ceramics toughened with metal precipitates, and
- other proprietary projects.

AECL is also making use of its facilities and expertise to develop new ceramic technology and products for various proprietary applications, including:

- microporous ceramics,
- ceramic spheres,
- colloidal ferrites,
- "flexible" ceramic coatings,
- ion-conducting ceramics, and
- high-temperature furnace elements.

3.2 Expertise and Facilities

Chalk River and Whiteshell bring together over 50 scientists and engineers with an expressed interest in advanced ceramics and expertise in:

- ceramic process development
- nondestructive evaluation
- materials science
- electron microscopy
- colloid chemistry
- corrosion
- electrochemistry
- surface science
- chemical engineering

Currently about 16 man-years of engineering/scientific effort are spent on ceramic materials, not including work on irradiation performance. Technical effort is one to two times this.

Chalk River and Whiteshell maintain laboratories dedicated to the development of ceramic processes and the evaluation of ceramic materials. These clean and modern laboratories, many of which can handle toxic materials such as ceramic whiskers, are outfitted for work on:

- powder preparation
- powder drying and granulation
- fine grinding
- glass formulation
- ceramic forming
- sol-gel processing
- plasma processing
- powder characterization
- colloid characterization
- mechanical testing
- fracture analysis
- thermal endurance
- physical properties
- thermal analysis
- corrosion resistance
- microstructure
- ceramic machining
- wear testing

The two sites also maintain specialized analytical equipment that is essential to the development of advanced ceramic materials, including:

- scanning and transmission electron microscopes
- scanning Auger microscopes
- secondary ion mass spectrometers
- X-ray photoelectron spectrometers (ESCA)
- computerized image analysis system
- X-ray and neutron diffraction
- small-angle neutron spectrometer
- Raman and infrared spectrometers
- laser-doppler anemometer (Zetasizer)

4. NONDESTRUCTIVE EVALUATION

4.1 General

Chalk River provides a comprehensive capability in nondestructive evaluation. Longstanding programs develop and apply:

- neutron diffraction
- tomography
- ultrasonic testing
- eddy current testing
- gamma and neutron radiography
- X-ray diffraction

Work on ultrasonic and eddy current testing includes technique development and transducer optimization. Sophisticated computer modelling is utilized in the ultrasonic work.

4.2 Neutron Diffraction

For the past five decades, neutron scattering techniques have been used by the solid state physics community to study properties of condensed matter, primarily crystallography, magnetism and lattice vibrations. Over the past five years, the material science community has awakened to the potential of using neutron diffraction to solve problems of concern to materials science and engineering.

The scattering of thermal neutrons by crystalline solids is analogous to X-ray diffraction; there is a general scattered background and sharp Bragg peaks. From the position of the peaks one can determine inter-planar spacings, with a precision of the order of 10^{-4} angstroms. Since residual strains cause changes in inter-planar spacing, the position of Bragg peaks can be used to obtain information about macro- and grain-interaction stresses. The width of the peak gives information on particle size and micro-strains, and from the intensity of the peak one learns about crystallographic texture and density.

Since neutrons are expensive compared with X-rays, and their generation requires either a nuclear reactor or a spallation source, neither of which is portable, one may ask why use neutrons? The answer is penetration. The penetration of neutrons into metals and ceramics is from 1000 to 10 000 times greater than that of X-rays. Thus while X-rays probe the first few microns of the surface of a sample, neutrons provide information that is characteristic of the sub-surface, bulk volume of the component.

There are many advantages that accrue from the penetrating power of the neutron. Access to transmitted beams, rather than only reflected beams as is the case with X-ray diffraction, provides the ability to determine the complete residual strain tensor. Surface preparation is not necessary, thus measurements can be made on components having curved or threaded surfaces and on samples with thick oxides or scales. The beam can pass through the walls of furnaces, cryogenic chambers, or pressure chamber, thus allowing measurements to be made under a variety of imposed conditions. One disadvantage to the technique is that the source is not portable, and thus the samples must be brought to the spectrometer.

The principal applications of neutron diffraction are for the measurement of:

- residual strain and strain gradients,
- crystallographic texture and texture gradients,
- the presence and distribution of minority phases, and
- temperature.

The experiments can be classified according to the cross-sectional area of the incident beam. With large-beam experiments, the entire sample, or at least a large part of it, is irradiated. The advantages are excellent statistics, since a very large number of grains contribute to the diffraction pattern, and a very high count rate, meaning that measurements can be made quickly. The restriction is that there should be no gradients in strain, texture or phase concentration, since the parameter determined is the bulk average over the whole sample volume. In many applications one does not want a sample average, but rather it is the spatial gradients that are required. In this case, one does a small-beam experiment, which employs narrow slits to define precisely a small volume at a known position in the sample. Figure 1 is a schematic of the small-beam setup. A beam of polychromatic (white) neutrons is extracted from the reactor core. A monochromatic beam, 5 cm x 5 cm in cross-section with a wavelength of about 2 A, is obtained by the use of a Ge monochromating crystal. This is the "large-beam". Slits, typically 1 mm wide and 5 to 10 mm high, in Cd masks, are used to define a narrow beam which usually passes through the sample and reaches the beam stop. If one were to collect all of the diffracted beam, the result would give the average over the total beam path, but this is not what is required. Therefore a slit is placed in the diffracted beam, thereby defining a small, diamond-shaped prism from which all of the diffracted information originates. The diffracting volume is ideally around 10 mm^3, however experiments have been performed at Chalk River with volumes as small as 1 mm^3. Gradients in residual strain or texture are then determined by translating the sample on computer-controlled X-Y-Z tables.

A triple-axis spectrometer at Chalk River is currently operating nearly full time applying neutron diffraction to engineering problems. Recent work includes:

- residual strains in stainless steel pipe,
- residual strains and texture in welded Zr-alloy pipe,
- crystallographic texture in Ti-alloy forgings,
- powder patterns from Al_2O_3,
- residual grain-interaction strains and thermal strains in Zircaloy-2, and
- residual strains in girth-welded 36" diameter pipeline steel.

The objective of the work on the pipeline steel was to determine the through-wall and axial variation of the axial component of residual strain in the vicinity of the girth weld. Figure 2 shows the through-wall distribution at the centerline of the weld. It is evident that high residual tensile strains exist halfway through the wall as a result of the welding procedure.

It is clear that neutron diffraction provides a powerful nondestructive tool for materials science, and will play an important role for both metals and ceramics.

4.3 Tomography

Chalk River has developed expertise and facilities for the nondestructive evaluation of materials and components by the computed tomography (CT) technique.

Computed tomography, also known as computer-assisted tomography (CAT), is a nondestructive, non-intrusive technique for generating quantitative cross-sectional images of an object. Tomography is related to radiography, but while more expensive, it has significant advantages over the latter. Radiography produces a distorted image of an object due to the superposition of features on the recording media; tomography yields distortion-free images that show the internal features in their correct spatial configuration, and with potentially much higher sensitivity.

The best-known application of CT scanning is in medicine where X-ray transmission tomography has come into widespread use over the past 15 years. The use of CT for industrial nondestructive examination is a relatively new but growing application. An introduction to industrial CT scanning and the Chalk River program can be found in reference 1.

The principle of CT scanning is presented in Figure 3. An object of interest is traversed by X-ray (or gamma-ray) beams in a plane; the object is rotated through consecutive angular positions at the same axial position until the desired number of incremental scans (usually 180 or 360, sometimes 1000, 2000 or more) have been obtained. After the scan is completed and the measured data are stored in a computer, the CT image of the cross-section is constructed by computer. The CT image is an exact map of X-ray (gamma-ray) attenuation, related to density, in the plane of measurement. The sensitivity to small differences in the X-ray absorption is extremely high, 10 to 100 times higher than with conventional radiography.

The digitally constructed CT image is usually presented in visual form on a video screen, using either gray or various color scales. The video image can be photographed to provide a permanent record. Color scales are preferred since they can show a larger dynamic range of densities than gray scales and allow more features to be presented in one visual CT image.

A CT scanner consists of:

- an X-ray or gamma-ray source,
- a detector or array of detectors,
- signal amplifying and digitizing electronics,
- mechanical mechanisms to move the source/detector and/or the object,
- a high-speed computer to control the scan and reconstruct the image, and
- a display system.

There are four different types of geometrical conditions possible, and accordingly, four types of scanners (named somewhat misleadingly "generations"). First generation scanners, which use a collimated pencil beam and a single detector, have non-restricted geometries and flexible scan parameters (resolution and contrast), but are slow in collecting data, and therefore are used only in laboratory conditions. To make the scan time shorter, the CT scanners can be equipped with a number of detectors and use a divergent or "fan" beam. These are higher generation scanners with more restricted geometry and usually a limited choice of scan parameters. Medical scanners are usually fourth generation machines.

The suitability of the CT technique for the study of advanced ceramic materials has been demonstrated (e.g. [2-4]), and it is clear that the CT method will play a key role in both the development of reliable engineering ceramic materials and the quality control of ceramic product. Although the characterization of some ceramics, especially those in the green state, can be achieved using medical scanners, the use of such scanners for this application has several drawbacks:

- fixed and/or limited spatial and density resolutions, which are optimized for typical medical requirements and not for industrial requirements (e.g. the contrast is usually not sufficient to detect small density gradients in ceramic materials and the spatial resolution is not sufficient to detect small inclusions or voids),

- fixed geometries that are optimized for a patient and not for industrial objects,

- X-ray sources which are polyenergetic and therefore can lead to severe beam-hardening artifacts; the effect of beam hardening is more pronounced for dense materials, like ceramics, in comparison to a human body, and proper corrections are required if high-contrast images are to be measured,

- X-ray energies which are usually too low for studies of denser materials like sintered ceramics, and

- a high cost which is, in a large part, connected with the requirement for a design that provides fast scans and assures patient safety and convenience.

To take full advantage of the CT technique for industrial applications, particularly advanced ceramics, a new approach is needed. This is the mandate of the tomography group at Chalk River. The group consists of a team of scientists and engineers who develop the CT technique for industrial applications and offer a variety of services to industry, including:

- performing service scans,

- consulting and problem solving on applications related to tomography and radiography,

- collaborative scientific research to investigate new applications of CT and related techniques, and

- building CT scanners optimized for specific problems.

The group currently operates two prototype CT scanners that are available for service inspections. Both machines have flexible scan parameters and are suitable for imaging a variety of industrial objects and materials. The scanners employ various gamma-ray sources and are suitable for measuring green and sintered ceramics. Source selection for a given inspection is based on the object size and density, and the type of defects to be detected. The obtained CT images are free from beam-hardening artifacts (the exact corrections are introduced for polyenergetic sources) and provide accurate numerical values of the absolute densities. The CT images are displayed and analyzed on an image processing system that has extensive software and allows:

- adjustment of the gray or color scales used to display images,

- region-of-interest calculations, such as statistics or histogram equalization,

- construction of three-dimensional images from a series of two-dimensional images,

- determining the absolute linear attenuation coefficients and plotting density profiles through the data set,

- adding images (e.g. to increase contrast), and

- subtracting images (e.g. for differential comparisons).

As an example of a CT scan on a ceramic material, CT images of two ceramic tiles are presented in Figure 4 [4]. The tiles are densified alumina, with the nominal density of 3.93 g/cm^3. The CT images of square, 146 mm by 146 mm, cross-sections were measured. The statistical noise of the images, 0.5 to 1% from pixel to pixel, was low enough to detect three low-density regions in the first tile (A, B and C, with porosities 1, 3 and 2% below the tile average, respectively), and, in the second tile, two extended low-density regions intercrossed by cracks and high-density "ribbons", and long-range density gradients as small as 0.2%. The images in Figure 4 are presented using a gray scale that spans only a few percent around the average tile density.

The Chalk River CT group is currently working on the specification for a CT scanner specifically designed for advanced ceramics. Such a scanner should assure the resolution and detectability required for the development and production of ceramic materials, be fast in the accumulation of data, have a convenient geometry, and be reasonably inexpensive. Inquiries on this scanner, as well as on applications of CT scanning, are invited.

5. SUMMARY

AECL has a long history of developing ceramics for nuclear waste containment, nuclear fuel, reactor materials and fusion breeder blankets. Long-term ceramic programs continue in these areas, spinning off new ceramic technologies for other advanced ceramic applications.

AECL has unique capabilities in both ceramic processing and nondestructive evaluation. We have well equipped laboratories for work on processing and characterizing ceramic materials, and we have a comprehensive capability in nondestructive evaluation, including two rare techniques -- neutron diffraction and tomography. These two techniques could make substantial contributions to the development of advanced ceramics, but have yet to be widely exploited.

AECL is keen to team its resources with industry to develop advanced ceramic processes and products. This can be done through contract R&D, technology licencing, business spin-outs and contractual joint ventures.

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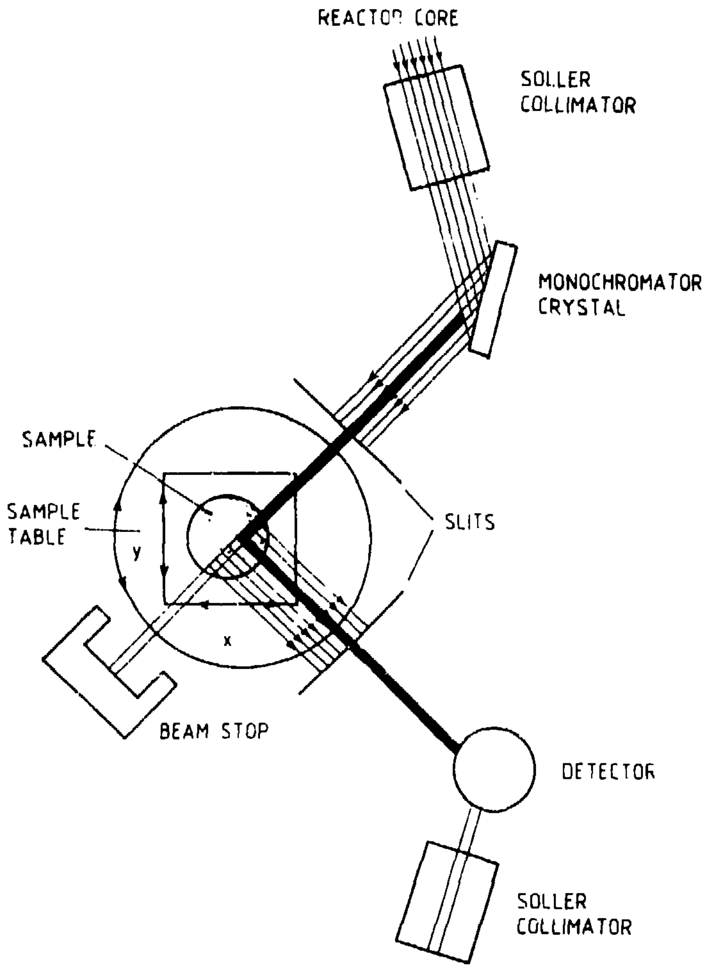

Figure 1: Schematic of residual strain measurement using slits to define a small beam.

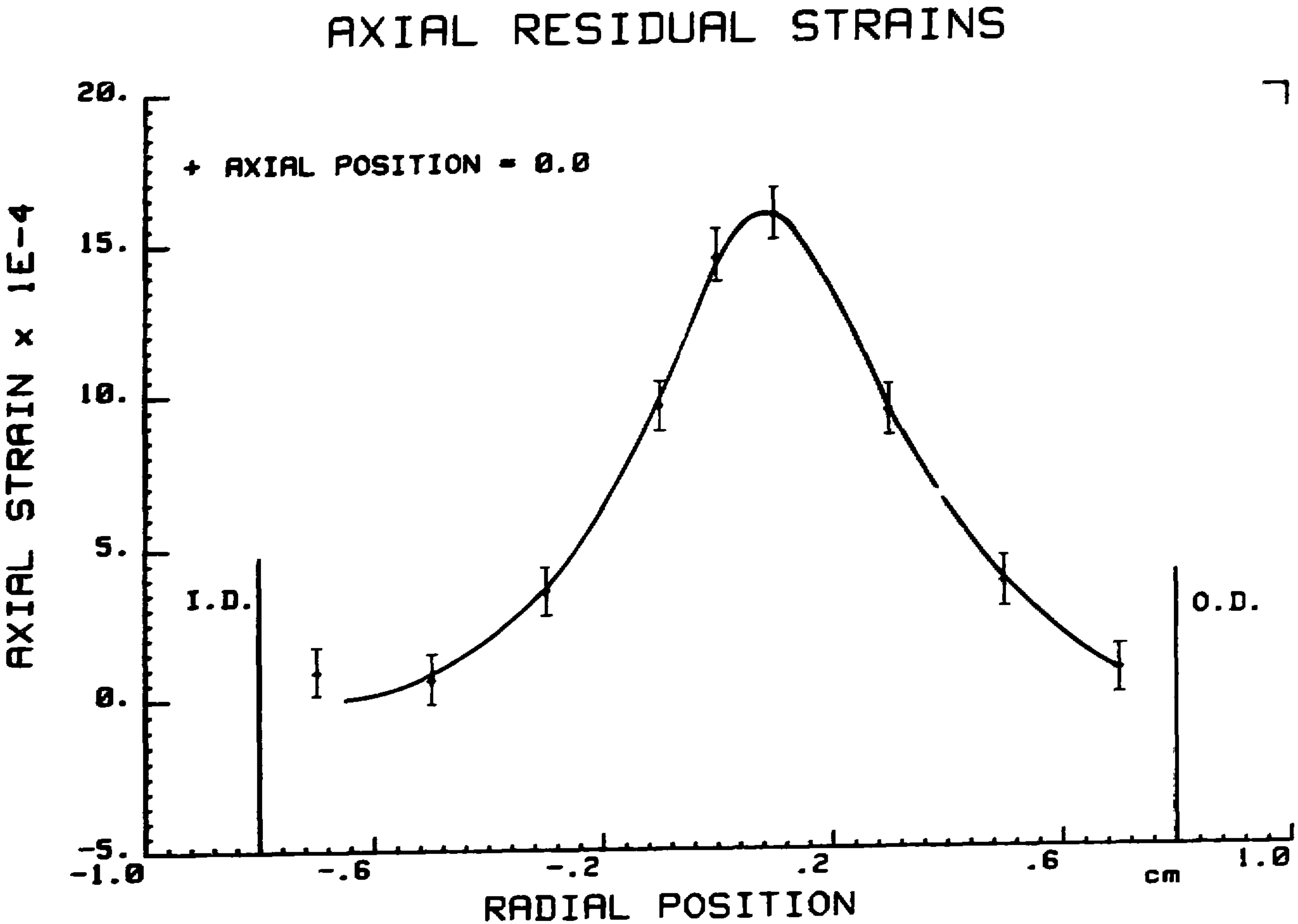

Figure 2: Residual strains in pipeline weld.

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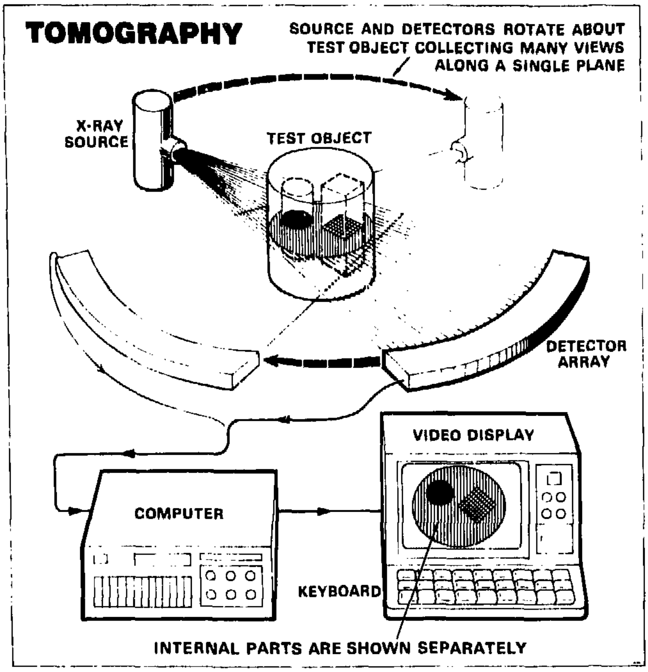

Figure 3: A CT scanner measures X-ray (or gamma-ray) attenuation in a single plane of an object from many different views. The data are stored in computer memory. The computer then reconstructs an image that is a map of X-ray attenuation, which is related to density, and shows the internal features of the cross section in their correct spatial configuration.

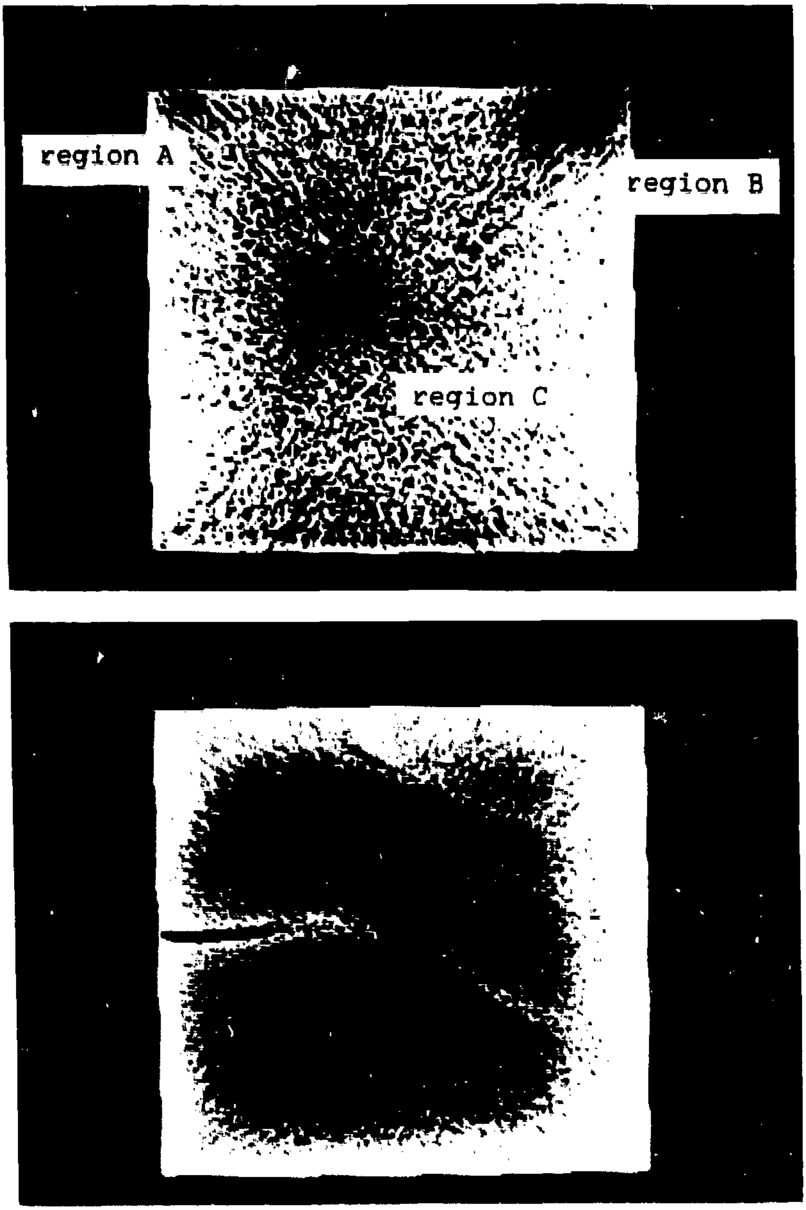

Figure 4: CT scans of two alumina tiles, 146 mm square. The images are presented in a gray scale that spans a density range from about 3.70 to 3.93 g/cm^3. White represents densities of 3.93 g/cm^3, or above; black represents densities below 3.7 g/cm^3.

ISSN 0067 - 0367

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