

IBMM-95

**APPLICATIONS OF ION BEAM
ANALYSIS WORKSHOP**

(Incorporating the 2nd French-Australian
Workshop on Ion Beams)

LUCAS HEIGHTS, SYDNEY

1-3 February 1995

WORKSHOP HANDBOOK

(Program, Abstracts, List of Participants
and General Information)

Sponsored by:

Australian Institute of Nuclear Science & Engineering Inc.
Australian Nuclear Science & Technology Organisation
Commonwealth Scientific & Industrial Research Organisation -
Division of Exploration & Mining
The University of Newcastle - Department of Physics

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Workshop organised by

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and
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in conjunction with

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Division of Exploration & Mining
and
The University of Newcastle - Department of Physics

Workshop Manager

Margaret Lanigan (AINSE)

PROGRAM

WEDNESDAY 1 FEBRUARY 1995

PAPER NO.

0930-1030		Registration and coffee
1030-1045		Welcome and introduction Dr John Boldeman, Director Applications of Nuclear Physics, ANSTO
Session 1		Chairman: J Boldeman
1045-1120	1	Invited Paper Nuclear Microanalysis Applications in Basic Research for Nuclear Waste Management P Trocellier
1120-1140	2	Contributed Paper The Analysis of Contaminants Deposited on Automotive Catalytic Converters Using Proton Induced X-Ray Emission (PIXE) D Angove
1140-1200	3	Contributed Paper The High Throughput, High Precision AMS System at ANTARES A Smith
1200-1220	4	Contributed Paper A New Surface Analysis Facility - the RMIT Scanning Auger Nanoprobe J Gorman
1220-1330		LUNCH
Session 2		Chairman: J Frontier
1330-1405	5	Invited Paper Analysis of the Hydrogen Content in Metal Surface Layers Modified by Ion Implantation J D Meyer
1405-1425	6	Contributed Paper IBA in Geological and Mineralogical Applications S Sie

WEDNESDAY 1 FEBRUARY Cont'd

	PAPER NO.	
1425-1445	7	Contributed Paper The Use of Ion Beam Analysis of Quartz in Mineral Exploration J van Moor
1445-1505	8	Contributed Paper New Results by High Dose Implantation and High Power Ion Beams in Metals and Alloys A Pogrebniak
1505-1530		AFTERNOON TEA
Session 3:		Chairman: J O'Connor
1530-1605	9	Invited Paper Effective Characterization of Beam Conditions During Ion Assisted Processes J Ullmann
1605-1625	10	Contributed Paper Atom Transport and Stability in Irradiated Metal/Ceramic Interfaces A Balogh
1625-1645	11	Contributed Paper Low Energy, High Intensity Plasma Ion Sources and Their Application to the Solid State Surface Modification and Solar Cells Fabrication B A Mukashev

THURSDAY 2 FEBRUARY 1995

Session 4		Chairman: H H Anderson
0900-0935	12	Invited Paper Heavy Ion Elastic Recoil Detection Analysis (ERDA) J A Davies
0935-0955	13	Contributed Paper Space Charge Formation and Relaxation in Ion- Bombarded Polyimide Kapton J Costantini

THURSDAY 2 FEBRUARY Cont'd

0955-1015	14	Contributed Paper Amorphization Kinetics of Poly(Vinylidene Fluoride) on High-Energy Ion Irradiation E Dooryhee
1015-1035	15	Contributed Paper Materials Analysis Using RBS/HIRBS with Realistic Computer Simulation Q Yang
1035-1105		MORNING TEA
Session 5		Chairman: P Thevenard
1105-1140	16	Invited Paper RBS Investigation of Low Temperature Interdiffusion Kinetics for Thin Metal Films Used in Magnetic Recording Heads J E E Baglin
1140-1200	17	Contributed Paper RBS on a Heavy Marker as a Tool to Study Atomic Transport Induced by Ion Bombardment L Thomé
1200-1220	18	Contributed Paper An Overview of the Heavy Ion Time-of-Flight ERDA Facility on the ANTARES Tandem Accelerator N Dytlewski
1220-1240	19	Contributed Paper Pyrole, a User Friendly Software for Teaching IBA and Analysing Experimental Data J P Frontier
1240-1420		LUNCH BBQ at the Tandem Hall and tours of ANSTO facility
1420-1630		Laboratory Tours, North Ryde
1730-2100		Sydney Harbour Dinner Cruise. Departure: Man O'War Steps, Sydney Opera House

FRIDAY 3 FEBRUARY

	PAPER NO.	
Session 6		Chairman: J A Davies
0900-0920	20	Contributed Paper Improved ExB Filter Device for Low Energy ERDA Applications P Trocellier
0920-0940	21	Contributed Paper Heavy Ion Recoil Spectrometry of Barium Strontium Titanate Films P Johnston
0940-1000	22	Contributed Paper Detecting of Heavy Ions Using a Recently Developed Time-of-Flight Detecting System Z Fang
1000-1020	23	Contributed Paper Experimental Techniques in Elastic Recoil Time-of- Flight Spectrometry J W Martin
1020-1045		MORNING TEA
Session 7		Chairman: J Baglin
1045-1120	24	Invited Paper High Sensitivity Hydrogen Profiling of IC Metallizations Using the 6.4 MeV $^{15}\text{N}(p,\alpha\gamma)^{12}\text{C}$ Nuclear Reaction K Horn
1120-1140	25	Contributed Paper Indirect Measurements of Hydrogen: The Deficit Method for a Many-Component System T E Levine
1140-1200	26	Contributed Paper Nuclear Reaction Analysis of Nitrogen in Steels L S Wielunski

FRIDAY 3 FEBRUARY 1995 Cont'd

- | | | |
|-----------|----|--|
| 1200-1235 | 27 | Invited Paper
The $^{19}\text{F}(p,\alpha\gamma)^{16}\text{O}$ Resonant Nuclear Reaction and its Applications in Depth Profile Measurements
C Tan |
| 1235-1330 | | LUNCH |
| Session 8 | | Chairman: P Trocellier |
| 1330-1405 | 28 | Invited Paper
Damage Induced in Insulators by Electronic Processes Associated with Energetic Ion Beam or Cluster Beam
P Thevenard |
| 1405-1425 | 29 | Contributed Paper
Comparison of Ion Beam Mixing Effects in Metal-Si Composites with keV and MeV Beams
A Bhagwat |
| 1425-1500 | | FINAL DISCUSSION AND OVERVIEW OF WORKSHOP
P Trocellier, J O'Connor, D Cohen |
| | | CLOSE OF WORKSHOP
Professor Bob Breakspere, President of AINSE |

ABSTRACTS

NUCLEAR MICROANALYSIS APPLICATIONS IN BASIC RESEARCH FOR NUCLEAR WASTE MANAGEMENT

Patrick Trocellier

CEA-CNRS, Laboratoire Pierre Süe, Centre d'Etudes de Saclay
91191 Gif sur Yvette Cedex (France)

In a waste repository, the leaching mechanisms of solid surfaces appear to be the main factor of radionuclide release in the biosphere. Thus, it is important to know the evolution of the composition both for the leachant and the solid surface. Photon induced spectroscopies, electron and ion beams techniques have been used extensively for more than fifteen years to study the composition of nuclear glasses ceramics and spent fuel submitted to aqueous leaching.

This paper is devoted to review the abundant work which has been performed in "Laboratoire Pierre Süe, Saclay", concerning radioactive waste management basic research.

Ion millibeam and microprobe investigations of nuclear glass and uranium dioxide surfaces are described. We show how ion beam analysis could be used to identify the composition of alteration phases growing into hydrated layers and to assess the role of the main experimental parameters.

**THE ANALYSIS OF CONTAMINANTS DEPOSITED ON
AUTOMOTIVE CATALYTIC CONVERTERS
USING PROTON INDUCED X-RAY EMISSION (PIXE)**

Dennys E Angove¹, Grahame M Bailey²,
Philip E Johnson², Noel W Cant¹ and David D Cohen²

Automotive catalytic converters are used in petrol-engined vehicles to convert the noxious gases produced as a result of the combustion process into less harmful ones. These catalytic converters undergo a loss of activity whilst in service as a result of thermal damage and/or poisoning by contaminants present in their input exhaust stream. The origin of these contaminants is generally believed to be engine oil and/or fuel. A trial investigation was performed on samples cut from near the centre of the front face of 5 vehicle-aged three-way monoliths, 4 oxidation monoliths and 1 unused three-way monolith, included as a reference. The monolith samples were carefully selected to cover a BET surface area range from 0.1m²/g to 16.3m²/g. By way of comparison, the unused reference samples possessed a BET surface area of 29.3m²/g. The PIXE analyses utilised a beam spot diameter of 3mm and detected the active metals platinum (0.13-0.46wt%), palladium (0.04-0.45wt%) and rhodium (0.04-0.11wt%) with reasonable precision. The contaminants phosphorus (4.4-16.8wt%), lead (0.01-1.8wt%), zinc (0.07-4.3wt%), calcium (0.08-5.0wt%) and potassium (0.02-6.7wt%) were detected in significantly larger concentrations on those monolith samples exhibiting low BET surface areas, compared with those samples exhibiting higher BET surface areas. The analysis also detected increasing concentrations of silicon for a corresponding decrease in BET surface area. Silicon is a major constituent of the ceramic underlying the monolith washcoat. The 3mm beam overlapped the exposed ceramic ribs located at the side of each 1mm channel formed as a result of sample excision. Mathematical compensation for silicon contribution by these broken ceramic ribs does not offer an explanation for the observed high silicon concentration. Work to determine the origin of these high concentrations of silicon is continuing. An additional contaminant mapping study was also performed longitudinally along a monolith. Although this technique is still under developed, the results of this study suggest that monolith deactivation by poisoning occurs in the front 30mm of the monolith.

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THE HIGH-THROUGHPUT, HIGH-PRECISION AMS SYSTEM AT ANTARES

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E M Lawson, Quan Hua, Claudio Tuniz
Australian Nuclear Science and Technology Organisation
PMB 1, Menai NSW 2234, Australia

The ANTARES AMS facility at the Lucas Heights Research Laboratory is operational and routine AMS measurements of ^{14}C , ^{26}Al and ^{36}Cl have commenced. Measurement capabilities for ^{129}I and ^{10}Be are being established. The overall aim of the facility is to promote advanced programs in Quaternary science, biomedicine and nuclear safeguards. This paper discusses the equipment and methodology that has been developed to achieve high-throughput and high-precision capability which is essential to our research program. A high-intensity, multi-sample negative ion source and a system for rapidly modulating the injection energy of the ion beam are key elements of the accelerator facility. A dedicated ^{14}C beam line and a general purpose AMS beam line permit measurement of light to intermediate isotopic masses, whilst a heavy isotope beam line with electrostatic energy and time-of-flight analysis has been funded and is under development to extend our measurement capability to actinides.

A NEW SURFACE ANALYSIS FACILITY THE RMIT SCANNING AUGER NANOPROBE

J Gorman
Department of Applied Physics
Royal Melbourne Institute of Technology
GPO Box 2476V, Melbourne 3001

RMIT Applied Physics Department have recently purchased a Scanning Auger Nanoprobe as a result of a successful ARC Mechanism C submission.

This state of the art surface science tool is essentially a very clean scanning electron microscope able to carry out surface atomic electron spectroscopy, ie. elemental and chemical bonding analysis, on areas as small as 25 nm diameter and 1nm deep, on exterior and interior surfaces in both amorphous and crystalline materials without any specialised preparation.

The instrument chosen was the Fisons VG 310-F, valued at \$1,350,000.

The VG 310-F uses a high brightness warm field emission source which is 50 times brighter than LaB₆ filaments. The beam energy is typically variable from 25kV down to 1kV with associated spatial resolution of 10 nm to 150 nm (1kV) for elemental analysis. In addition the 310-F has high sensitivity with superior energy resolution. Secondary electrons produced in the sample from the ion beam allow an SEM type image to be obtained giving the operator the ability to accurately position the etched area anywhere on the sample. The etched area of the beam can be varied from approximately 10mm (x10) to 0.1mm (x1000) and the beam energy from 1 keV to 5 keV. The beam current is typically between 1 and 3 μ A. Software control of the sample stage and ion beam allows continuous azimuthal rotation of the sample during depth profiling significantly reducing ion beam shadowing and thereby increasing depth resolution.

The addition of an X-ray source has provided XPS as a supplementary technique, with a sensitivity of x1000 times that of older dedicated XPS instruments. A well equipped and flexible preparation chamber allows in situ cleaning, annealing, and fracturing of samples in a UHV environment. In addition thin films may be separated from their substrates and both surfaces analysed, without exposure to the atmosphere.

In carrying out materials analysis the above features provide new benefits. At *low beam voltage* the excitation volume is small, reducing scattering and increasing microanalytical accuracy. Specimen charging on insulators is easier to control, and beam damage to delicate samples is reduced.

High energy resolution enables direct determination of chemical states particularly in many binary compounds which enables the plotting of AES line scans and chemical maps. The addition of XPS increases the analysis capability for more complex compounds and for electron beam sensitive compounds.

High depth resolution chemical depth profiling can be carried out either by angle resolved emission or by sputtering with rotation which dramatically increases depth resolution.

Examples of analyses which have been carried out with the VG 310-F include: particles, trench bottoms, chemical state mapping and scattergrams, fracture surfaces, and depth profiling of multilayers, interfaces and thin films.

**ANALYSIS OF THE HYDROGEN CONTENT IN
METAL SURFACE LAYERS
MODIFIED BY ION IMPLANTATION**

K Bethge, H Baumann, N Angert and K Neu
Presented by J D Meyer
Institut für Kernphysik
der Johann Wolfgang Goethe-Universität Frankfurt am Main

Hydrogen in metals is analysed by the nuclear reaction ${}^1\text{H}({}^{15}\text{N},\alpha\gamma){}^{12}\text{C}$. Titanium samples were implanted with 150-200 keV carbon, nitrogen and oxygen to investigate a possible correspondence between hydrogen and the implanted species. The implanted layers were analysed by RBS and a suitable (p,γ) reaction for the host matrix. Dependent on the implanted doses of the implanted species the hydrogen profiles are different. As an explanation we propose that chemical reactions between the implanted elements and titanium inside the bulk material cause the effect. The degree of saturation informing a special lattice, is different according to the many known phases which can be formed between C, O and Ti. The comparison of the results between implanted elements will be discussed.

IBA IN GEOLOGICAL AND MINERALOGICAL APPLICATIONS

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IBA applications in minerals research are steadily expanding, and the methods are now part of the standard repertoire in a number of areas. While macrobeam methods are still useful and unique in some applications, the predominant techniques are microbeam based, consistent with the fact that the most typical basic constituents of all geological samples are microscopic monomineralic grains. Microbeam approach is also essential in studies of zoning features, inclusions and experimental petrological samples. The relatively large (tens of microns) range of MeV ion beams also enabled studies of intact fluid inclusions. Most applications have been based on micro-PIXE, but RBS, ERDA and NRA are also gaining steadily. AMS is well established in geophysical and geomorphological applications based on cosmogenic radionuclides. Micro-AMS being developed in a number of laboratories will open up new areas of applications based on ultra trace element geochemistry and standard geochronology studies hitherto only possible by bulk techniques following mineral and chemical separation. In selecting applications it is important that one is aware of competing analytical techniques that may impact on IBA's viability, and whenever possible exploit their complementarity.

THE USE OF ION BEAM ANALYSIS OF QUARTZ IN MINERAL EXPLORATION

J C van Moort

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Simultaneous PIXE/PIGME analysis of quartz at ANSTO since 1990 indicates that quartz associated with a variety of gold and volcanic hosted mass of sulphide deposits is invariably very impure. The method can be extended into the field of rockgeochemistry, if sufficient sample purification is observed. Common impurities are Li, F, Na, Al, Cl, K, Ca, Ti, Mn, Fe, Zn, Ga, Ge, As, Rb, Zr, Ba, and Pb, often in concentrations not easily detected by other methods.

The application of ion beam analysis of bulk samples as an exploration tool was carried a step further in the study of quartz in the highly weathered plumbic-manganiferous and ferric surface gossans in the Broken Hill area. The area has suffered at least since Tertiary times intensive weathering. Samples were selected from the oxidised surface from the few remaining outcrops of the lodes, rocks above buried lodes and from barren gossans along strike and away from the lode line. The material was treated with aqua regia in order to break up the plumbic-manganiferous minerals and retreated with nitric acid and ammonium acetate.

The acid insoluble residue of the Broken Hill gossans consist of corroded quartz and jasper fragments, with minor amounts of sphene, anatase, rutile, garnet, zircon, barite and occasionally potash feldspar and mica. The Mn and F content of this acid insoluble residue and its Mn x F product can be used as indicators of mineralisation and the location of deeply buried deposits. These indicators are more reliable than previously used Pb, Zn and Mn content or elemental ratios like Pb/Fe of the bulk material, determined without acid treatment.

**NEW RESULTS BY HIGH DOSE IMPLANTATION AND HIGH
POWER ION BEAMS IN METALS AND ALLOYS**

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ABSTRACT NOT AVAILABLE

EFFECTIVE CHARACTERIZATION OF BEAM CONDITIONS DURING ION ASSISTED PROCESSES

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The knowledge of the ion beam conditions (charge states, energy distribution, current density) both in ion assisted deposition processes and in material characterization systems using ion beams is essential for a better understanding of various phenomena. However, in many cases these information are not known in detail.

Here, two systems for the characterization of ion beam conditions used in an ion assisted evaporation facility will be presented. The kind of ion flux component does influence the material formation and modification due to ion-solid interactions significantly. Therefore, information about the ion charge states are available by use of a differentially pumped mass spectrometer system with ion optics. Furthermore, with a combined Faraday-Cup (FC) and Retarding Field Analyzer (RFA) the ion current density and ion energy distribution, respectively, can be measured. The advantage of this simple, but efficient, analytical system lies in the possibility to collect the data from the hole beam cross section by computer controlled scans. This modular FC- and RFA-probe was designed for use in an ion assisted deposition process. However it can also be used for beam analysis in equipment using ions for material characterization. The set-up, operating mode, potential and limits of the ion beam probe mentioned above will be demonstrated. Furthermore, in connection with the obtained experiences, the importance of the knowledge of ion flux conditions will be discussed generally.

ATOM TRANSPORT AND STABILITY IN IRRADIATED METAL/CERAMIC INTERFACES

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D M Rück
Institute for Heavy Ion Research, Darmstadt, Germany
H Baumann and K Bethge
Institut für Kernphysik, J S Goethe Universität, Frankfurt, Germany

Temperature dependent diffusion properties and stability of metal/ceramic interfaces were studied using bilayer sample geometry. Ion beam mixing and radiation enhanced diffusion have been investigated in different metal/ceramic samples. Specimens (Cu/Al₂O₃, Au/Al₂O₃ and Au/ZrO₂) with film thickness of 60-70nm were prepared by vapor deposition and irradiated with 150 keV Ar⁺ ions in the range of 1.8-10¹⁶ to 1.5-10¹⁷ Ar⁺/cm². Sample temperature during irradiation was varied between 77K and 673 K. The mixing behavior was analysed using concentration depth profiles, measured by Rutherford Backscattering Spectroscopy (RBS). Additionally, samples were studied by high resolution scanning electron microscopy (HRSEM) and by photoelectron spectroscopy (XPS). The results show that mixing efficiencies for all elements scales linear with the Ar⁺ ion dose. Radiation enhanced diffusion is clearly separated from temperature independent mixing processes. The migration enthalpy for interdiffusion is determined for the different metal/ceramic systems. Additionally, different irradiation effects near to the interface, including mobility, defect trapping and chemical affinity will be discussed, according to their influence on the diffusion properties.

**LOW ENERGY, HIGH INTENSITY PLASMA ION SOURCES AND THEIR
APPLICATION TO THE SOLID STATE SURFACE MODIFICATION
AND SOLAR CELLS FABRICATION**

B A Mukashev

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National Sciences of the Republic Kazakstan
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Three types of high intensity plasma ion source have been used for the homogeneous large square ion implantation and deposition into silicon into/on stainless steel, and titanium-aluminium alloys substrates. We have prepared and studied shallow phosphorus ion implanted doping layers in poly and monocrystalline silicon as well as TiO_2/Si system and titanium ion implanted metals and alloys. It is found that SIMS phosphorus atom distribution is very similar to the charge carrier dependence with depth after annealing samples at 850 and 930°C during 10 sec. Effective carrier lifetime has been increased and the electrical properties of p-n junctions have been remarkably improved by hydrogen passivation of the surface, deleterious impurities and grain boundaries. As a result efficiency of polycrystalline solar cells has reached 9.5% (without anti-reflection coating). It is shown that TiO_2/Si system is more effective as anti-reflection coating on comparison SiO_2/Si system.

Titanium ion implantation into stainless steel and titanium-aluminium alloys changes parameters of martensitic transformation and mechanical properties of steel as well as causes transition initial two phases ($\gamma + \alpha_2$) system to one phase (γ) alloys. It is a very interesting effect for further studies because under usual conditions phase transforms to the α_2 phase during heating up to 1000°C.

HEAVY ION ELASTIC RECOIL DETECTION ANALYSIS (ERDA)

J A Davies
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Using extremely heavy ion beams (eg. 200 MeV ^{197}Au), ERDA becomes a semi-universal technique capable of analysing simultaneously almost all elements up to mass 100 or even higher. The basic concepts will be presented and illustrated by several recent applications.

SPACE CHARGE FORMATION AND RELAXATION IN ION-BOMBARDED POLYIMIDE KAPTON

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The space charge formation and relaxation in polyimide Kapton thin films irradiated with heavy ions in the MeV/u range has been studied by *on-line* measurements of the space charge distribution in the bulk of the targets by using the pressure pulse method. The space charge relaxation either by diffusion under high internal field or by dielectric breakdown will be addressed in relation to the material modification, ie. degradation of the polymer to a more conducting carbonaceous phase in the ion tracks. Possible stress effects in the space charge formation will also be discussed.

AMORPHIZATION KINETICS OF POLY(VINYLDENE FLUORIDE) ON HIGH-ENERGY ION IRRADIATION*

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The crystalline fraction of irradiated semi-crystalline $(C_2H_2F_2)_n$ polymer is measured as a function of dose (or fluence) and energy loss (dE/dx) using GeV ion beams. The rate of scattering loss as a function of fluence provides the amorphization efficiency A .

For low energy transfers (dE/dx 58 keV/nm), A smoothly increases with increasing dE/dx . For higher values of dE/dx , A increases more rapidly. In this high dE/dx regime, the loss of crystal is not simply related to the overall mean absorbed dose.

The high densities of electronic excitations presumably affect the primary chemical modifications, which in turn prevail on further physical/chemical changes in the polymer.

In addition, an incubation effect is observed under electron, oxygen or neon irradiations and does not appear for heavier projectiles. We suggest that at low dE/dx , the disorder propagates from the amorphous interface into the crystallites. At higher dE/dx , the traversed region directly transforms into an amorphous track with no incubation.

*Ion irradiations performed at the GANIL accelerator, France.

MATERIALS ANALYSIS USING RBS/HIRBS WITH REALISTIC COMPUTER SIMULATION

Q Yang
Department of Physics
The University of Newcastle, NSW 2308, Australia

Rutherford Backscattering Spectrometry (RBS) is a powerful tool for materials analysis. Experimental parameters can be optimised depending upon specific requirements. The advantages of RBS and its extension using heavy projectiles (HIRBS) have been explored with the help of computer simulations. A realistic simulation program for RBS/HIRBS simulation has been developed, which includes energy straggling, lateral spread, multiple scattering and nonlinear detector resolution as well as geometrical effects. Also, it allows thorough optimisation of experimental parameters for material analysis. The reliability of the simulation has been verified and demonstrated by comparing with experiments.

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**RBS INVESTIGATION OF LOW TEMPERATURE INTERDIFFUSION
KINETICS FOR THIN METAL FILMS USED IN
MAGNETIC RECORDING HEADS**

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IBM Almaden Research Centre
650 Harry Road, San Jose, CA 95120, USA; and
J K Howard, K R Coffey and M A Parker
IBM Storage Systems Division, San Jose, CA 95193, USA

Multilayered films including Ta-Permalloy ($\text{Ni}_{80}\text{Fe}_{20}$) play a key role in magnetic recording sensors, where it is important to preserve the integrity of the thin Permalloy layer under thermal conditions of processing, and in subsequent service.

An investigation of the kinetics of the thermal interdiffusion of these materials in the temperature range 250-500°C has highlighted the special ability of RBS analysis to identify the rapid (but sparse) population by Ta of grain boundaries of the magnetic layer, even at low temperatures. The effect is unexpected, and it could possibly degrade the performance of the magnetic layer. The RBS signature for this process is clear, enabling the quantitative evaluation of structural features such as grain size upon layer stability. Although qualitative support from species-selective TEM may emerge, other standard analytical approaches that depend on sputter-profiling had not previously been able to recognise this process. (In fact, Ta has been regarded as a diffusion barrier in this system). The diffusion measurements will be presented and discussed.

*Permanent address: Institute for Physics, University of Sao Paulo, PO Box 20516, 01452 Sao Paulo, SP, Brazil.

RBS ON A HEAVY MARKER AS A TOOL TO STUDY ATOMIC TRANSPORT INDUCED BY ION BOMBARDMENT

Lionel Thomé

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Irradiation of a solid target with energetic ions is known to induce atomic displacements depending on many parameters, such as the nature of the target, the mass and energy of the irradiating ions, the temperature. We present an original methodology developed to study the atomic transport induced by ion bombardment, based on the use of heavy marker atoms introduced by vacuum deposition or ion implantation beneath the surface of the investigated target. Nuclear microanalysis techniques, particularly Rutherford backscattering (RBS), are used to monitor the modifications of the marker profile due to irradiation.

Results obtained in two different experimental situations are discussed. The ion-beam-mixing phenomenon occurring in solids irradiated with low or medium energy (<10 keV/u) ions, for which nuclear collisions is the dominant damage process, is first examined. Then, the case of very high energy (>1 MeV/u) ion irradiation, leading to a dramatic plastic deformation phenomenon due to ion electronic energy loss, is reviewed.

AN OVERVIEW OF THE HEAVY ION TIME-OF-FLIGHT ERDA FACILITY ON THE ANTARES TANDEM ACCELERATOR

N Dytlewski

**Australian Nuclear Science and Technology Organisation
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The development of the Time-of-Flight facility at ANSTO has progressed from where initially, a count rate of 10 Hz was considered good, to now, where count rates up to 1000 Hz are obtained, causing distortions in the data due to pulse pileup and false triggers. A VME based data acquisition system and on-line graphical display using risc instruction set DEC-5000 workstations have been developed, as well as graphical control of the accelerator system from ion source to target end station. Data analysis is done using the PAW software developed at CERN. A description of this facility will be presented.

PYROLE, A USER FRIENDLY SOFTWARE FOR TEACHING IBA AND ANALYSING EXPERIMENTAL DATA

Philip Trouslard

Presented by Jean-Pierre Frontier

Institut National des Sciences et Techniques Nucléaires

Laboratoire van de Graaff

C.E.N. Saclay, 91191 Gif sur Yvette Cedex, France

When performing Ion Beam Analysis, some calculations are currently carried out, such as particle energy loss inside a filter, energy-depth relationship, energy of a particle emitted by a specific nuclear reaction, scattering energy, etc. Physicists are often led to write a specific computer code, solving their own problem. The context is quite different with teaching activities. Students, indeed, are concerned with solving themselves classical calculations with regard to interactions between matter and particles. Nevertheless, considering a number of different examples would take too much time. For this reason, a software has been carried out in our laboratory. PYROLE is a user friendly software concerning hydrogen and helium energetic ions in the Me V range, designed to be used as a tool-box; a number of different options is proposed, according to each question to be answered separately, such as:

- Range and stopping power table printing
- Kinematic factor
- Rutherford cross section
- Thin film thickness by RBS
- Thickness computing from energy loss
- Energy loss in thin film
- Straggling
- Nuclear reaction (Q value, particle energy before and after a Mylar foil)

PYROLE is currently used by the students during the training sessions. It also appears that PYROLE is often used by the scientists themselves, before and during their experiments.

Some examples will be given concerning analyses carried out in our laboratory.

**IMPROVED EXB FILTER DEVICE FOR LOW ENERGY
ERDA APPLICATIONS**

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91191 Gif sur Yvette Cedex, France

ABSTRACT NOT AVAILABLE

HEAVY ION RECOIL SPECTROMETRY OF BARIUM STRONTIUM TITANATE FILMS

W B Stannard, P N Johnston, S R Walker, I F Bubb and J B Scott
Royal Melbourne Institute of Technology
Melbourne, Australia
D D Cohen and N Dytlewski
Australian Nuclear Science and Technology Organisation
Menai, Australia

Ferroelectric films are being developed for use in DRAMs. However some commercially promising materials suffer fatigue problems that are likely to be related to stoichiometric changes.

Heavy ion recoil spectrometry employing 77 and 98 MeV ^{127}I ions has been used to analyse thin films of barium strontium titanate (BST) at the new heavy ion recoil facility at ANSTO, Lucas Heights. The technique employs a Time of Flight and Energy (TOF-E) detector which enables the determination of separate energy spectra for individual or small groups of elements. Depth profiles for all major elements and carbon have been obtained. Analysis reveals depletion of oxygen in the BST layer next to the platinum electrode and interdiffusion of strontium and oxygen through the layers.

Work supported by the Australian Research Council and Australian Institute of Nuclear Science and Engineering.

DETECTING OF HEAVY IONS USING A RECENTLY DEVELOPED TIME-OF-FLIGHT DETECTING SYSTEM

Zewei Fang

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A timing detector suitable for detecting fast particles has been developed recently. The secondary electrons produced by the energetic projectiles after passing through a thin carbon foil are transferred isochronously to the multichannel plate electron multipliers to form a timing signal. In this timing detector design, no permanent magnet is employed and the use of grids has been minimised.

The operational characteristics of this novel timing detector has been studied by experimental testing and computer simulation. It has been shown from computer simulation that the timing uncertainty introduced by this timing detector is 35ps (FWHM) under the optimal operating condition.

A Time-Of-Flight detection system based on this timing detector has been developed with a flight path length about 1m and a detector solid angle of 0.4mstr. A stepper motor controlled 3-axis goniometer within a UHV chamber has been employed to perform the heavy ion RBS/channelling analysis. Computer software has been developed to control the stepper motors as well as to collect the spectrum. Automatic channelling search can be performed by minimising the yield within a given energy window for various target positions.

The TOF system has been tested and calibrated using 6-15MeV C ions generated from the 14UD heavy ion accelerator at ANU, Canberra. A number of semiconductor materials including P implanted Si, Te or Se implanted GaAs and $\text{Si}_{1-x}\text{Ge}_x\text{Si}$ strained layer superlattice samples have been analysed. The experimental results have indicated, as expected, the enhanced mass and depth resolutions for the heavy ion RBS using 0.5-1MeV/amu C or Cl as the analytical beam. Enhanced detection sensitivity for heavy elements has been observed as the result of using TOF detector as the energy analyser. Further improvement on the detector performance is anticipated as the timing signal detection process is optimised.

EXPERIMENTAL TECHNIQUES IN ELASTIC RECOIL TIME-OF-FLIGHT SPECTROMETRY

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An elastic recoil time of flight spectrometer has been built on the 10 MV tandem accelerator at the Australian Nuclear Science and Technology Organisation for the major purpose of materials characterisation. The spectrometer, consisting of two electrostatic timing signal generators and an ion implanted surface barrier detector, is capable of using ion beams with masses of 12-130 amu and energies of 20-100 MeV. Resolution studies have shown that timing, mass and depth resolutions of 300ps, 4-5amu and 12Å for surface gallium recoils respectively, are achieved using a higher mass and energy beam, with the predominant ion used being 84 MeV I¹¹⁺. Recent studies using the spectrometer have included metal-nitride structures and transition metal implanted superconductors. This paper will discuss some recent results in view of resolution considerations as well as material characterisation.

J W Martin, D D Cohen, N Dytlewski, D B Garton, H J Whitlow and G J Russell,
Nucl. Instr. and Meth. B 94 (1994), 277-290.

HIGH SENSITIVITY HYDROGEN PROFILING OF IC METALLIZATIONS USING THE 6.4 MeV $^{15}\text{N}(p,\alpha\gamma)^{12}\text{C}$ NUCLEAR REACTION

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The presence of hydrogen in very large scale integrated circuit metallizations has been examined as a possible contributing factor in the formation of stress-induced voids in integrated circuit metal lines. Using the 6.4 MeV $^{15}\text{N}(p,\alpha\gamma)^{12}\text{C}$ nuclear reaction, hydrogen profiles have been measured on a series of layered structures consisting of aluminium-copper alloy metallizations deposited on boro-phospho-silicate glass and capped with a variety of commercial passivation materials. Employing a compact combination of material and electronic shielding, and also off-resonance yield correction, the hydrogen measurements were conducted with detection sensitivities of 60 ppm and depth resolutions of several hundred angstroms. The hydrogen profiles reveal differences in the concentrations and depth distributions of hydrogen within the layered structures which depend on the composition and type of passivation material used. A description of the compact, background suppression system and off-resonance yield corrections used in these measurements is presented, as well as the results of the hydrogen profiling measurements which indicate that, a) the amount of hydrogen present in the metallization depends on the type of passivation material employed, b) that there is no correlation between the hydrogen content within the metallization layer and the degree of compressive stress present in the overlying passivation layer, and c) that the introduction of hydrogen into the metallization layer occurs during fabrication.

**INDIRECT MEASUREMENTS OF HYDROGEN:
THE DEFICIT METHOD FOR A MANY-COMPONENT SYSTEM**

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We have developed a simple technique for determining hydrogen concentration from the measurement of the Rutherford backscattering signal of remaining species. This technique uses the surface heights of various RBS signals and applies the surface energy approximation in the form of a linear matrix equation. For an n-component system, n-1 equations are comprised of a modified surface height equation and the nth row is the conservation equation, thereby producing a system of n equations in n unknowns. We have developed this method for use with homogenous hydrogen-containing thick films, the depth of which is unprobable by the analysis beam. We apply this technique to *in situ* analysis of ion beam induced densification of sol-gel zirconia thin films, there hydrogen is the most volatile species during irradiation. Results are confirmed through the direct measurement of hydrogen using forward recoil energy spectrometry. Attendant errors are discussed with an emphasis on stopping powers and Bragg's rule.

NUCLEAR REACTION ANALYSIS OF NITROGEN IN STEELS

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Tribological properties of steels are substantially modified by nitriding processes. Nitrogen can be introduced by different techniques, from traditional chemical nitriding to plasma immersion ion implantation or ion implantation using high energy ion implanters or low energy ion guns. The concentration depth profile of nitrogen in steel is strongly dependent on physical and chemical parameters of the nitriding process: temperature, time, nitrogen pressure, nitrogen ion current density, ion energy and of course steel composition. Nuclear reaction analysis can measure nitrogen concentration and nitrogen depth profiles as a function of physical and chemical parameters used. The results can be compared with results of hardness and other tribological tests.

Usually nitrogen is detected and profiled using the $^{14}\text{N}(\text{d}, ^4\text{He})^{12}\text{C}$ nuclear reaction. This reaction can be used in laboratories with small accelerators (1MV); however laboratory neutron radiation background is high when using this reaction and in practice this limits the use of this reaction to laboratories with adequate neutron radiation shielding.

An alternate method for nitrogen profiling is the use of $^{14}\text{N}(^3\text{He}, \text{p})^{16}\text{O}$; however the low cross section and low depth resolution limits use of this reaction.

In this work we propose the use of nuclear reaction $^{14}\text{N}(^3\text{He}, ^4\text{He})^{13}\text{N}$ in the broad resonance region (^3He energy about 43.65 MeV). The high cross section about 2.5mb/sr provides good sensitivity. This reaction can be used to profile nitrogen in steels up to 2500nm depth with good depth resolution and can be used in laboratories without neutron radiation shielding. Experimental results will be shown for different materials tested.

THE $^{19}\text{F}(p,\alpha\gamma)^{16}\text{O}$ RESONANT NUCLEAR REACTION AND ITS APPLICATIONS IN DEPTH PROFILE MEASUREMENTS

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In this paper, we review briefly the $^{19}\text{F}(p,\alpha\gamma)^{16}\text{O}$ resonant nuclear reaction and its application. Depth profiles of fluorine in $^{19}\text{F}^+$ implanted SnO_2 films, MCT, CdTe, $\text{Pb}_{1-x}\text{Sn}_x\text{Te}$, $\text{KTa}_{1-x}\text{Nb}_x\text{O}_3$ (KTN), LiNbO_3 crystal materials, Ta, Nb, Mo refractory metals and $\text{Ni}_{0.8}\text{Fe}_{0.2}$ alloy have been accurately measured using $^{19}\text{F}(p,\alpha\gamma)^{16}\text{O}$ resonant nuclear reactions at $E_R=872.1$ keV and 340.46 keV. In order to extract the range distribution of implanted fluorine from the experimental excitation yield curve, a proper convolution calculation method is presented, from which the range distribution parameters, such as the average projected range R_p , the projected range straggling ΔR_p , and the skewness of the range distribution SK, were obtained. It shows that the $^{19}\text{F}(p,\alpha\gamma)^{16}\text{O}$ resonant nuclear reaction is an ideal technique for fluorine depth profile measurements in these heavy materials. However, careful subtraction of interfering resonances must be made if one uses the strong resonance at $E_R=872.1$ keV. The narrow resonance at $E_R=340.46$ keV is well isolated from other resonances. Therefore this reaction can be used to obtain depth profiles of fluorine with much less background and higher depth resolution. But the sensitivity is lower than that by use of the resonance at $E_R=872.1$ keV.

**DAMAGE INDUCED IN INSULATORS BY ELECTRONIC PROCESSES
ASSOCIATED WITH ENERGETIC ION BEAM OR CLUSTER BEAM**

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A Brunel, S Della Negra, Y Le Beyec
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Single crystals of MgO, Al₂O₃, LiNbO₃ were bombarded with GeV ions at GANIL or 20 MeV C₆₀ cluster beam. The damage resulting from the high electronic stopping power (up to 60 keV/nm) was characterized by both Rutherford backscattering ion channelling (RBS-C) and optical absorption measurements.

The surface swelling induced by the defect creation has been measured with a profilometer. Depending on the target an electronic stopping power threshold for surface swelling was observed, the step height increased with the damage for electronic stopping power higher than the threshold.

COMPARISON OF ION BEAM MIXING EFFECTS IN METAL-Si COMPOSITES WITH keV AND MeV BEAMS

A Bhagwat

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Ion mixing with keV beams in metal-Si systems can be understood as either collisional or cascade type of mixing. Recently, there has been a lot of interest in studying the effects of MeV beams in materials. An ideal way to understand the interaction of MeV ions in materials appears to be in the field of ion mixing, especially in metal-Si system, where keV beam mixing has been well studied and documented.

We have utilised MeV heavy ions to bring about ion mixing in Sn-Si, Au-Si, Ge-Si and Cu-Si bilayers. Various values of energy deposited at the interface of metal-Si system were employed. Silicide formations with MeV beams appear to have a distinct advantage over the keV beam mixing process: the energy deposited within the thin metal films is a well defined quantity with less lateral straggling. The kinetics of silicide formation have been studied by Rutherford Backscattering technique. Unirradiated and annealed samples were also analysed for comparison and completeness. The results are compared with keV beam mixing processes reported in literature.

Our investigations reveal that although the defects created with MeV and keV beams appear to be similar, the extent of beam mixing with ion dose varies with a negative slope. This may probably be due to radiation annealing effects, which have also been seen with MeV/GeV ions in metals. Some overview of the ion mixing phenomena will be presented.

LIST OF PARTICIPANTS

LIST OF PARTICIPANTS

Invited Papers	PAPER NO.
Dr J E Baglin Almaden Research Center, USA	1
Dr J A Davies AECL Chalk River Research Laboratories, USA	12
Dr K Horn Sandia National Laboratories, USA	24
Dr J D Meyer Institut für Kernphysik der Johann Wolfgang Goethe-Universität, Germany	5
Dr C Tan Shandong University, China	27
Dr P A Thevenard Université Claude Bernard, France	28
Dr P Trocellier CEA Laboratoire Pierre Sue, France	1, 20
Dr J Ullman Technische Universität Chemnitz-Zwickau, Germany	9
Contributed Papers	
Mr D Angove, Macquarie University, Australia	2
Dr A G Balogh Fachbereich Materialwissenschaft, Fachgebiet Dünne Schichten, Germany	10
Dr A Bhagwat Nuclear Science Centre, India	29
Dr J-M Costantini Commissariat à l'Énergie Atomique, France	13
Dr E Dooryhee Centre interdisciplinaire de recherches avec les ions lourds, France	14
Dr J-P Frontier INSTN, France	19

Contributed Papers cont'd	PAPER NO.
Dr P Johnston Royal Melbourne Institute of Technology, Australia	21
Dr T Levine Cornell University, USA	25
Dr J W Martin The University of New South Wales, Australia	23
Professor B Mukashev Physical Technical Institute, Kazakstan	11
Dr A Pogrebnyak Sumy Institute of Surface Modification, Ukraine	8
Dr S Sie Division of Exploration & Mining, Commonwealth Scientific & Research Organisation, Australia	6
Dr A M Smith ANSTO, Australia	3
Dr L Thomé Centre de Spectrométrie Nucléaire et de Spectrométrie de Masse, France	17
Dr J C van Moort The University of Tasmania, Australia	7
Dr L Wielunski Division of Applied Physics, Commonwealth Scientific & Research Organisation, Australia	26
Q Yang The University of Newcastle, Australia	15

Other

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Okayama University of Science, Japan

Dr W Stannard
Royal Melbourne Institute of Technology, Australia

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Gesellschaft für Schwerionenforschung-Darmstadt, Germany

GENERAL INFORMATION

IBMM-95
Applications of Ion Beam Analysis Workshop
(Incorporating the 2nd French-Australian Workshop on Ion Beams)

Lucas Heights, Sydney, Australia, 1-3 February 1995

FIRST ANNOUNCEMENT AND CALL FOR PAPERS

A workshop on Applications of Ion beam Analysis will be held at the Australian Nuclear Science and Technology Organisation (ANSTO) at Lucas Heights, Sydney from Wednesday to Friday 1-3 February, 1995, immediately prior to the **IBMM-95 Conference in Canberra**. The Workshop is sponsored by the Australian Institute of Nuclear Science and Engineering (AINSE).

The number of Workshop participants will be **limited** so positions will be filled on a first come first served basis.

Aims: Ion beam analysis techniques offer a suite of powerful diagnostic tools for characterising thin films and modified surfaces. The workshop will review developments and current status on use of light and heavy, high and low energy ion beams for analysis emphasising the following areas:

- Fundamental ion beam research and secondary effects of ion beams
- Materials sciences, geological and industrial applications
- Environmental, archaeological and life science applications
- Data bases and computing codes for use in accelerator research
- Stopping powers and cross sections for IBA
- Recent technological developments using ion beams
- High energy heavy ion scattering and recoil

There will be invited speakers in several of these key areas

Venue: The AINSE Theatre, outside the main gate at ANSTO, Lucas Heights Research Laboratories (20km SE of Sydney)

Accommodation: (a) **Kingsway Hotel**, on the beach at Cronulla (approximately 20 mins by bus from the Workshop venue and within easy access to Sydney city centre by train (40 mins) for sightseeing). Delegates who stay at the Kingsway Hotel will be transported by bus to the workshop every day.
Cost A\$100/ room only (single/double/twin) per night.

(b) **Stevens Hall**, at Lucas Heights adjacent to the workshop venue. This is budget priced accommodation. There is no public transport available to or from Stevens Hall for private evening social activities or visits to Sydney city.
Cost A\$55/ room (single/double/twin) per night including light continental breakfast.

Social Functions: To allow international guests and visitors to Sydney to enjoy the delights of our magnificent Sydney Harbour, a dinner cruise has been arranged for Thursday 2 February 1995.
Cost: A\$60 per person.

Papers and Abstracts: If you wish to offer a paper (20 mins) presentation you should prepare a title and no more than 25 line abstract on an A4 page in 12 point text with 3 cm margins and send it to Dr. David Cohen (address below) before 1 November 1994 for consideration by the organisers.

Proceedings for this Workshop will be produced. Participants giving an oral presentation will need to prepare a four page, camera ready, summary of their talk by the date of the Workshop, 1 February 1995.

Costs: No extra costs are envisaged at this stage for registration at the workshop. However, participants are totally responsible for their own accommodation costs (A\$100/ night), living expenses, social programs and all other costs not associated with the Workshop.

NOTE: A A\$100 holding deposit is required, in advance, by 1 November 1994 to obtain bookings at the Kingsway Hotel and Stevens Hall. Payment **must** be included with your registration form.

Payment: All international registrants should pay by a bank draft in Australian dollars national registrants may pay by cheque. All bank drafts and cheques should be made payable to; **IBA Workshop IBMM-95**. Payment in any other currency will not be accepted.

Deadlines:
1 November 1994, Receipt of Workshop registration (no cost)
1 November 1994 Receipt of title of paper and 25 line abstract if offering a presentation.
1 November 1994, Receipt of A\$100 holding deposit for accommodation.
1 November 1994, Receipt of A\$60/ person for payment of Sydney Harbour Cruise (including dinner)

Further Information: Dr David Cohen
Australian Nuclear Science and Technology Organisation
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For information concerning registration and accommodation please contact:

Mrs Margaret Lanigan,
Workshop Manager,
AINSE, PMB1,
MENAI, NSW, 2234, Australia.
Ph: +61 (02) 717 3376; fax +61 (02) 717 9268.

ACCOMMODATION

The Kingsway Hotel
20 Kingsway Street, Cronulla NSW 2230
Telephone: +61 (0)2 527 3100
Fax: +61 (0)2 523 9541

Lucas Heights Motel
New Illawarra Road, Menai NSW 2230
Telephone: +61(0)2 717 3437
Fax: +61(0)2 717 9282

TRANSPORT

Timetable for coach transport for delegates staying at the Kingsway Hotel

Wednesday 1 February

0845 Depart from Kingsway Hotel
Drop off at Lucas Heights

1700 Depart from Lucas Heights
Drop off at Kingsway Hotel

Thursday 2 February

0815 Depart from Kingsway Hotel
Drop off at Lucas Heights

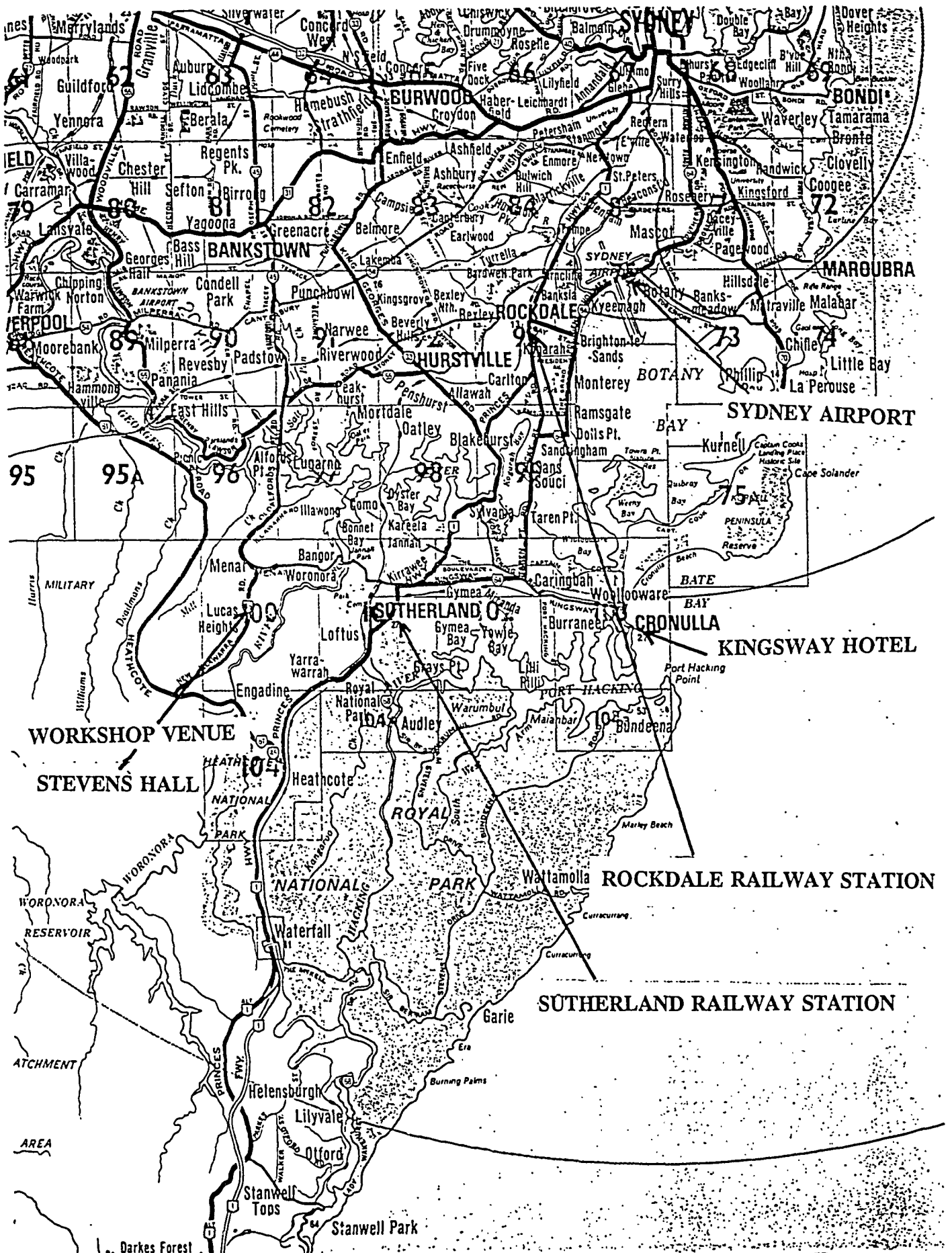
1430 Depart from Lucas Heights
Drop off at Sutherland Railway Station and Kingsway Hotel
(For delegates who are not participating in the
technical tour of CSIRO laboratories at North Ryde)

Friday 3 February

0815 Depart from Kingsway Hotel
Drop off at Lucas Heights

1530 Depart from Lucas Heights
Drop off at Kingsway Hotel

LOCATION MAP





AUSTRALIAN INSTITUTE OF NUCLEAR SCIENCE AND ENGINEERING (AINSE)

AINSE is pleased to support the Applications of Ion Beam Analysis Workshop. This is an important area of interest to its members.

AINSE is a consortium of Australasian universities in partnership with ANSTO, which has been set up to collaborate in the use of the national facilities at the Lucas Heights Research Laboratories. The facilities include two nuclear reactors, a number of particle accelerators, ion implantation facilities, a supercomputer, and many other facilities and services. Access to these facilities is provided through a program of grants and postgraduate research awards. Funds are provided by the consortium members to cover the costs of using the facilities and services, for minor equipment and materials, and for travel and accommodation for periods of attachment at Lucas Heights. AINSE also acts as a peak body on behalf of its members in obtaining and administering major infrastructure grants. A number of national conferences and workshops on nuclear science and technology topics are held every year and subsidies are available to enable participants from member universities to attend. Applications for grants close on September 30 and for Postgraduate Research Awards on December 31.

Researchers from member universities of AINSE who wish to find out more about the benefits of their university's membership should contact their representative.

University of Adelaide	A/Prof Gerald Laurence	Royal Melbourne Institute of Technology	Prof Dinesh Sood
University of Auckland	Prof Ralph Cooney	University of South Australia	Prof Roger Smart
Australian National University	Prof Trevor Ophel	Southern Cross University	Prof Peter Baverstock
Central Queensland University	Prof Bob Breakspere	Swinburne University of Technology	Dr Eddie Bakshi
Curtin University of Technology	Prof Brian O'Connor	University of Sydney	Prof Don Napper
Flinders University	Dr Peter Teubner	University of Tasmania	Prof Pip Hamilton
Griffith University	Dr Evan Gray	University of Technology, Sydney	Prof Joe Unsworth
James Cook University of North Queensland	Prof Len Lindoy	Victoria University of Technology	Prof Paul Clark
La Trobe University	Prof David Kelly	University of Western Sydney	Dr Robyn Crumby
Macquarie University	A/Prof Trevor Tansley	University of Western Australia	Prof Brian Stone
University of Melbourne	A/Prof Ron Cooper	University of Wollongong	Prof Peter Fisher
Monash University	Dr Trevor Hicks	ANSTO Program areas:	
Murdoch University	A/Prof John Webb	Biomedicine & Health	Prof Helen Garnett
University of New England	Prof Grant Harman	Applications of Nuclear Physics	Dr John Boldeman
University of New South Wales	Prof Hans Coster	Advanced Materials	Dr Adam Jostsons
University of Newcastle	Prof Ronald MacDonald	Environmental Science	Dr Wally Zuk
University of Queensland	Prof Jim O'Donnell	Nuclear Technology	Dr George Malosh
Queensland University of Technology	Prof Brian Thomas	Engineering	Mr Ken Horlock

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