

Materials and Engineering Science, Safety and Radiation Services

Materials & Engineering Science

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Materials & Engineering Science Facilities - General

Materials & Engineering Science currently conducts research and development projects in the following areas:

- Waste conditioning - development of multiphase crystalline titanate ceramic systems for the immobilisation of high level nuclear waste.
- Functionalised nanostructures – the development of processing techniques (e.g. sol-gel, atomic-layer deposition) for producing functionalised thin films and nanoparticles.
- Materials assessment - development of a systematic program of remnant life estimation of HIFAR aluminium structures and transfer of this technology to remnant life estimation of oil refining and coal-fired electricity generating plants under appropriate industry funding.
- Plasma processing of materials - the development of plasma processing techniques to modify material surfaces to enhance the value of industrial products.
- Materials characterisation - development and application of sophisticated techniques for the characterisation of the microstructures of metallic and ceramic materials.

Many of the facilities available in Materials & Engineering Science are also to be found in the laboratories of the AINSE member universities. However, at ANSTO a number of these facilities have the capability of being applied to the characterisation or processing of nuclear materials. Facilities which are unique within Australia include the Sinter-HIP installed in 1991, the ANSTO developed Plasma Immersion Ion Implantation system and a number of hot presses and high temperature testing machines which are unique in terms of their capacities or high temperature capabilities.

5050 Impedance Spectroscopy

Our Solartron 1260 impedance spectrometer allows electrical conductivity of materials to be measured using electrodes imbedded in or stuck to the surface of samples. Together with a multiplexer unit, up to eight samples can be monitored simultaneously. Typically the apparatus has been used to look at the curing of cement and geopolymer wasteform materials.

5060 JAF Calorimeter

The calorimeter is an isothermal conduction calorimeter used to measure heat of hydration in cements and heat of formation of geopolymeric materials.

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5100 Ceramics Powder Characterisation and Fabrication Facilities

Description

These facilities include a wide range of state-of-the-art equipment for measuring particle size, density, pore volume, pore size and surface area of ceramic powders fabricating ceramic powders by cold pressing, graphite die hot pressing and sintering and measuring density and pore size distribution of densified ceramics. Facilities are available for glove box fabrication of actinide containing ceramics.

Support Services and Facilities

Microstructural characterisation facilities
Hot pressing of materials
Cold and hot isostatic pressing facilities

Applications

The characterisation of powders, particularly those to be used in ceramic processing. Fabrication of ceramic components or materials

Mastersizer 2000
Malvern Zetasizer
ASAP 2400
Multipycnometer

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5105-5230 Ceramics Research Facilities

Description

These facilities may be used to characterise a wide range of the physical and chemical properties of colloidal precursors, ceramic powders or bodies. The available equipment includes:

- a SETARAM system for simultaneous differential thermal analysis/differential scanning calorimetry and thermogravimetric analysis
- a THETA 160 dilatometer
- an investigation of bulk solution and surface chemical processes occurring during the formation of colloids
- Atomic Layer deposition

The facility consists of

- BIORAD NIR FT-Raman and FT-IR vibrational spectrometers
- Wyatt Technologies static light-scattering and two NICOMP photon correlation spectrometers
- monolight systems UV/VIS/NIR/MIR spectrometer, equipped with fibre-optic probes

Support Services and Facilities

Mechanical testing and microstructural characterisation facilities.

Applications

These facilities can be applied to basic studies on the preparation and surface characterisation of ceramic powders and colloids in aqueous or non-aqueous media. In addition, the thermal response of powders and powder compacts can be studied in relation to thermal decomposition or sintering.

Availability

Service by ANSTO staff is available on an advance booking basis and subject to ANSTO program needs; the equipment is generally not available for operation by the researcher.

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5400-5425 Remaining Life Assessment Facilities - General

Description

A wide range of mechanical test equipment with associated furnaces for testing at elevated temperatures.

- Experimental facilities are as follows
- servo-hydraulic and screw driven machines for fracture toughness, fatigue and tensile testing at ambient and elevated temperatures
- impact testing machines for fracture toughness measurements
- small push pull fatigue machine
- a range of constant load creep machines for elevated temperature testing

Support Services and Facilities

Microstructural characterisation facilities (section 5610-5660).

Applications

These facilities can be applied to the measurement of the mechanical properties of materials, particularly metals, alloys and ceramics and the study of thermal shock resistance of refractory materials. Additional facilities are available for metallographic examination and sample preparation for SEM, TEM etc.

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5450 High Temperature Mechanical Testing Facilities - General

Description

The ANSTO's High Temperature Materials Group has the following expertise and facilities in the area of high temperature mechanical testing and thermal shock testing of predominantly brittle materials:

5455 High Temperature Strength in Bending

Test to measure the high temperature modulus of rupture of ceramic and refractories. Four point bending test is to be conducted on bending bars, 3.5 x 5 x 50mm³ and 15 x 25 x 120mm³ in size for ceramics and refractories respectively.

5460 High Temperature Single End Notched Beam

Test to measure the high temperature fracture toughness (K_{Ic}) of materials. Three point bending test is to be conducted on notched or pre-cracked bending bars, 3.5 x 5 x 50mm³ and 15 x 25 x 120mm³ in size for metals/ceramics and refractories respectively.

5465 Creep in Bending

Test to measure the creep resistance of high temperature materials. Four point bending test is to be conducted on bending bars, 3.5 x 5 x 50mm³ and 15 x 25 x 120mm³ in size for metals/ceramics and refractories respectively.

5470 Hot Crushing Strength

To measure the hot crushing strength of refractories. The specimen can either be a rod (30-50mm in diameter, 50-70mm in height) or a block (30-50 x 30-50 x 50-70mm³).

5475 Compressive Creep

To measure the compressive creep resistance of refractories. The specimen can either be a rod (30-50mm in diameter, 50-70mm in height) or a block (30-50 x 30-50 x 50-70mm³).

5480 Thermal Shock Testing (rapid heating)

To measure the thermal shock resistance of ceramic and refractories. Three techniques are available for the thermal shock test. For ceramics a small disk, 20mm in diameter and 0.5mm in thickness, is rapidly heated by a laser beam in the centre of the disk. For refractories a ring-type specimen, 20 and 100mm in ID and OD respectively and 10mm in thickness, is rapidly heated by an electrical heating element on the internal surface of the ring. For conductive materials such as carbon electrodes a disk specimen, 100-150mm in diameter and 10-15mm in thickness, is heated in the centre by an arc-discharge technique.

5485 Young's Modulus (Grindo-Sonic)

Test to measure the elastic properties of metals, ceramics and refractories. Specimen can either be a bar (3.5 x 5 x 50mm³ for metal/ceramic and 10 x 24 x 100mm for refractories) or a disk (100-150mm in diameter and 10-15mm in thickness).

5490 Small Scale Tensile or Bending (Mini-tester)

Test to measure the stress-strain behaviour of small metallic components in tension or fracture and adhesion of thin films on ductile substrates (tension/bending). Capability also of in-situ optical examination during loading.

Support Service and Facilities

Extensive facilities for cutting, drilling and diamond grinding of specimens are available.

Applications

The assessment of the high temperature mechanical properties and thermal shock resistance of materials can be used either as feedback information to optimise new materials or as database for the reliability evaluation of structural components in critical applications.

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Microstructural Characterisation Facilities - General

Description

A comprehensive range of equipment for determining the microstructures of materials particularly metals and ceramics. Experimental facilities are as follows

5610 JEOL 2010F

Transmission electron microscope fitted with a Scanning Image Observation Device (a STEM), bright field and high angle dark field detectors, a hollow cone illumination device, a Gatan Imaging Filter (GIF 2001) and an EmiSpec E Vision EDX system.

5615 JEOL 2000II TEM

Fitted with a pole piece that allows $\pm 60^\circ$ tilt and a high take-off angle (70°) Link Isis EDX System.

5620 JEOL JSM 6400

Scanning electron microscope, Noran Voyager Series IV EDS system with stage automation.

5625 JEOL JSM 6300 SEM

Noran Voyager Series IV EDS system with stage automation.

5630 XRD - PANalytical X'pert Pro X-ray diffractometer

The PANalytical X'Pert Pro Diffractometer is used for the identification and quantification of unknown phases in a mixture as well as the acquisition of data for crystal structure studies. In conjunction with the heating and cooling stages, it can also be used for phase transformation studies. It comprises a X'Celerator detector, multi purpose stage, 45 position sample changer, Cu anode ceramic X-Ray tube, Data Collector and Highscore software. Also available is an Anton Parr HTK16 hot stage (to 600°C in vacuum or 1200°C in air), Anton Parr HTK 450 cryo furnace (-190°C to 450°C) and a capillary spinner."

5635 XRD - Siemens D500 X-ray diffractometer.

5640 XRD – Scintag X-ray diffractometer

with primary beam monochromator, P_{Si} detector and provision for glancing angle X-ray diffraction.

5650 MSL-400 NMR Spectrometer

equipped with fast MAS, slow MAS and static probes

5655 CXP-300 NMR Spectrometer

equipped with MAS probes, static probes and a full array of probes for NQR analyses

5660 CXP-90 NMR Spectrometer equipped with fast and slow MAS capabilities

Support Services and Facilities

Specimen preparation facilities, including a Gatan Model 600 Dual Ion Mill, a Gatan Precision Ion Polishing System and facilities for ion beam thinning of radioactive specimens.

Applications

Routine examination of metallic and ceramic specimens. The microscopes are suitable for the determination of lattice site preferences by ALCHEMI and for microanalysis of precipitates, including carbides, in metals.

Crystal structure determinations can be carried out by X-ray diffractometry at temperatures up to 1200°C.

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5705-5710 Plasma Surface Engineering - General

Description

Experimental facilities for surface treatment using the Plasma Immersion Ion Implantation technique. Samples and tools up to 500 -2 cm in area can be treated at temperatures from 150-500 °C, with ion energies up to 45 keV.

Support Services and Facilities

Plasma diagnostics include a wide range of electrical probes and optical emission spectroscopy. A pin-on disc tribometer and stylus profilometer are available for wear testing and surface roughness measurements.

Extensive facilities for microstructural characterisation and mechanical testing of treated materials are available within the Advanced Materials program (Section 5500 & 5600).

Applications

The Plasma Immersion Ion Implantation technique can be used to modify metallic surfaces by direct implantation and/or diffusion. In particular, nitrogen is used for increasing hardness and wear resistance.

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5750 Instrumental Indentation Facility

Description

A Nano Indenter Model IIs from Nano Instruments Inc for making instrumented indentations to probe near-surface material properties. Vickers, Berkovitch and spherical indenter tips are available. Conversion of data to hardness measurements is standard, while elastic moduli can be obtained under suitable conditions.

A new option is now available allowing the elastic modulus to be measured throughout the loading curve.

Support Services and Facilities

Some support for data interpretation.

Applications

Hardness measurements of treated or coated surfaces as a function of depth.

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5760 Scanning Probe Microscope

Description

The Scanning Probe Microscope from Digital Instruments makes available high resolution surface imaging through a variety of modes - contact AFM, intermittent mode AFM, scanning tunnelling microscopy, lateral force microscopy and magnetic or electrostatic force microscopy. Maximum scan area is 100mm x 100 μ m with an inspection area of approximately 100mm² disc. Samples may be in air or under liquid. Some image analysis is also possible.

Applications

It can map and quantify sample-tip force interactions such as friction, magnetic fields and electrostatic forces at sub-micron dimension, especially for samples which are unsuitable for imaging under vacuum conditions.

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5770 Spectroscopic Ellipsometer

Description

The Spectroscopic Ellipsometer facility is a GESP5 made by SOPRA. It performs ellipsometric measurements as a function of wavelength from 250nm to 900nm. The incident and reflected angles are also adjustable. From this data the optical properties of samples can be determined. Extensive data analysis routines are available to model the ellipsometric data and hence extract further information about the properties/structure of the measured sample.

Applications

Measurement of optical properties of bulk material - real and imaginary refractive index verses wavelength.
Optical response of multi-layer structures - determination of layer thickness, porosity, interface structure, etc.

Support Services and Facilities

Support and instruction on data modelling.

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5800 Computer Aided Engineering Design Facilities

Description

Design and analysis facilities based on SGI work station and PC, using the CAD software package "Microstation", the Finite Element Analysis program "NISA II" and "ABAQUS", and the Boundary Element Analysis code "BEASY".

Applications

Design and analysis of engineering and structural components.

Support Services and Facilities

Prototype manufacture using CAM program is possible in conjunction with Engineering workshops, for additional cost.

Use of CAD/FEA/BEA analysis software and computers

CAD/FEA/BEA Technical assistance

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5900 Hot Pressing of Materials

Description

Induction heated hot-pressing of shapes up to 125mm dia x ~ 100mm high can be carried out from ceramic powders in graphite dies. A maximum temperature of 1900°C and a maximum load of 20 tonnes may be applied. The atmosphere is nitrogen or argon.

Datalogging facility is available. The powders may be consolidated into a pellet by uniaxial pressing prior to hot-pressing. In the case of most ceramics boron nitride powder coating is applied to prevent the reaction between graphite die and the powder.

Support Services and Facilities

All material characterisation facilities available, including particle size and surface area, see sections 5100, 5200 & 5600.

Applications

Hot-pressing can be used to remove porosity to make dense ceramics or metals from powders.

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5910 Scanning Laser Dilatometer

Description

This equipment allows the researcher to continuously monitor the dimensions of components fabricated from polymers, ceramic, glass, refractory or metallic materials from -40°C to 1500°C in air atmospheres. Either single large samples can be measured (up to 250 mm), or a maximum of eight samples simultaneously (≤ 20 mm).

Cylindrically shaped samples are most suitable, but prefabricated components can also be monitored. The edges of the shadows cast by the samples are detected and processed to give a real time measurement with a sensitivity at the micron level. This is a non-contact form of dilatometry (cf. push-rod type dilatometry).

Weight changes which occur during heat treatment of large single samples (< 2000 g) can be monitored concurrently using an inbuilt load cell.

The furnace chambers are the largest in ANSTO's Materials ($300 \times 300 \times 300$ mm) and allow normal batch heat treatment of large samples to also be performed.

Support Services and Facilities

Ceramic samples can be prepared from component powders by uniaxial pressing or cold isostatic pressing (see Sections 5100 & 5920). Metallic samples can be machined to any desired size. Prefabricated components need no further treatment.

Applications

The scanning laser dilatometer can be used to determine thermal expansion coefficients of a wide range of ceramic and metallic samples and prefabricated components. Phase transformations which are accompanied by a change in volume of the sample can be detected. Oxidation of metallic components resulting in increasing size and/or oxidation products spalling from the surface of the sample(s) can also be examined in real time. The sintering kinetics of both metal and ceramic powder compacts can be determined.

5911 Heat treatment in air ($< 1500^{\circ}\text{C}$)

5912 Heat treatment in air ($> 1500^{\circ}\text{C}$)

5913 Heat treatment in vacuum/inert atmosphere ($< 1500^{\circ}\text{C}$)

5914 Heat treatment in vacuum/inert atmosphere ($> 1500^{\circ}\text{C}$)

5915 Weight change with temperature (max 1000g sample)

5916 Infra red thermal imaging camera

Can be used to capture thermal images of materials being processed in the temperature range -20°C to 3000°C . Thermal profiles and spot temperatures for each image pixel can be retrieved. Video can be taken of processes in which thermal processes change with time.

5917 Molten glass/ceramic viscometry

Using a rotary viscometer with a platinum bob viscosity of glass/ceramic melts up to 1700°C can be performed

5918 Melt quenching facility

Materials can be quenched from a range of temperatures (up to 1700°C in air)

5919 Portable laser IR thermometer with Emissivity measurement

The Mikron Quantum I Portable Laser IR Thermometer offers accurate temperature measurements in applications while conventional radiation pyrometers suffer from large systematic errors. This state-of-the-art improvement is based on direct measurement of actual target emissivity and radiance.. Materials can be quenched from a range of temperatures (up to 1700°C in air)

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5920 Cold and Hot Isostatic Pressing Facilities

Description

Powders of ceramics and metals can be (cold) isostatically pressed (CIP) in rubber bags using two CIP machines. The maximum sample size for the larger CIP is 300mm diameter and 750mm length. The maximum sample size for the smaller CIP is 75mm diameter and 300mm length. The maximum pressure that can be applied for the larger CIP is 200 MPa and for the smaller CIP is 400 MPa. Metals, ceramics and composites can be hot isostatically pressed (HIPed) up to maximum temperature of 2000°C and at a maximum pressure of 200 MPa. It is also possible to sinter in the same cycle prior to HIPing. There are two HIP machines available. The maximum sample size for the larger HIP is 300mm diameter and 500 mm length. The maximum size for the smaller HIP is 70mm diameter and 110mm length.

Support Services and Facilities

Ceramic or metal powders have to be encapsulated in a metal can before HIPing. The necessary facilities are available.

Extensive facilities for powder processing, fabrication of parts, sintering and heat treatment are available (Section 5100 and 5300).

Also facilities for microstructural characterisation and mechanical testing of processed materials are available (Section 5600 & 5450 respectively).

Applications

HIPing can be used to remove closed porosity in metal castings and ceramics, thereby producing higher quality castings or ceramics. Diffusion bonding of similar or dissimilar materials can be carried out. Superalloys (eg aircraft turbine blades) may be rejuvenated in the HIP. Consolidation by HIPing produces a fine microstructure and a very dense material with superior mechanical properties.

5921 Encapsulation

5925 Hot Isostatic Press LARGE (<1750 deg)

5930a Hot Isostatic Press LARGE (>1250 deg<1750 deg)

5930b Hot Isostatic Press LARGE (>1750 deg)

5935 Hot Isostatic Press SMALL (<1750 deg)

5940 Hot Isostatic Press SMALL (>1750 deg)

5945 AIP Cold Isostatic Press - Small

5950 AIP Cold Isostatic Press- Large

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5960 Cold Crucible Melting (CCM) of Materials

Description

CCM is a method of melting materials (metals and ceramics) in a segmented, water-cooled copper vessel, in air or controlled atmosphere in an induction coil. This is done with direct material charge to crucible contact, without the need for refractory lining. The copper crucible is made up of water-cooled segments or "fingers". The magnetic field produced by the coil, in effect, passes through the crucible to induce heat in the material charge causing it to melt. The field also intensively stirs the liquid material pool. A thin layer of metal remains frozen against the bottom and walls of the crucible forming a skull. The low velocity boundary layer in the liquid adjacent to the skull, together with the skull itself and the interface (joint) between the skull and the crucible all serve as thermal resistances, reducing the heat conducted from the hot liquid into the cold crucible.

The melting can be done in air or controlled atmosphere, which can consist of any gas required, e.g. Ar, N₂, O₂, mixtures such as Ar/H₂, N₂/H₂ etc. We can also maintain the atmosphere at pressure ranging from about 10⁻³ torr to 2 bar. The temperatures we can reach is dependent on the material being melted and can reach temperatures >2000C. Crucibles sizes range from 50mm to 100mm diameter.

Support Services and Facilities

All materials characterisation facilities are available.

Applications

CCM can be used for high melting point materials such as ceramics, refractory metals and alloys. Also as there is no reaction with the crucible, it can be used where material contamination needs to be minimised.

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Radiation (Biology, Chemistry and Physics)

The facilities and services described in this section include those provided by

ANSTO - Safety & Radiation Services

6000 Hot Spot Irradiators

6100 Gamma Sources – Physical Science

Description

One small shielded gamma facility with dose rate of approx. 0.2 kGy/h is available. A series of lead shields provide a range of lower dose rates. Access to samples under irradiation is possible through access holes in the upper shielding plug. The unit is equipped with an automatic meniscus sensing and tracking mechanism controlled by a dedicated computer to enable the acquisition of dilatometric data. Temperature control of irradiations is possible.

Applications

Radiolytic product studies, dosimetry, radiation damage, crosslinking, grafting and polymerisation work. Kinetic investigations.

6200 Deep Level Transient Spectrometer

Description

A deep level transient spectrometer, usually operating in the capacitance mode. Based on the Miller exponential correlator method. Used to characterise deep level centres in semiconductors. The spectrometer is run by Radiation Standards, part of the Safety .

Support Services and Facilities

Optical DLTS; which is particularly suited for the trap of high resistivity material such as CdTe and semi insulating GaAs. C(V) and N(X) profiler with associated computer based facilities for data manipulation. Resistivity measurements using 4 point probe and associated standards. Vacuum system for the evaporation of metallic (Au, Al and Pd) contacts.

Applications

Electronic characterisation of high purity semiconducting materials such as Ge, Si and GaAs, used to fabricate nuclear radiation detectors. The characterisation and possible identification of centres introduced into such materials during crystal growth furnace annealing, or other high temperature cycle. The study of damage to the crystal lattice caused by prolonged exposure to radiation.

7400 Gamma Spectrometer

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