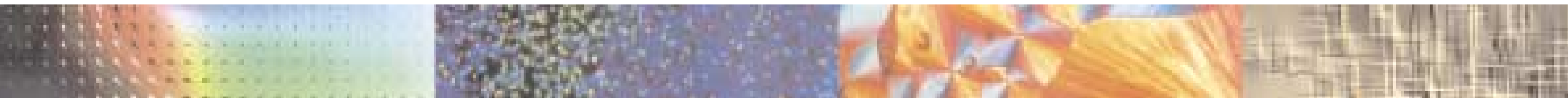
Oxford Materials

Research in Progress
2000 - 2001



Department of Materials

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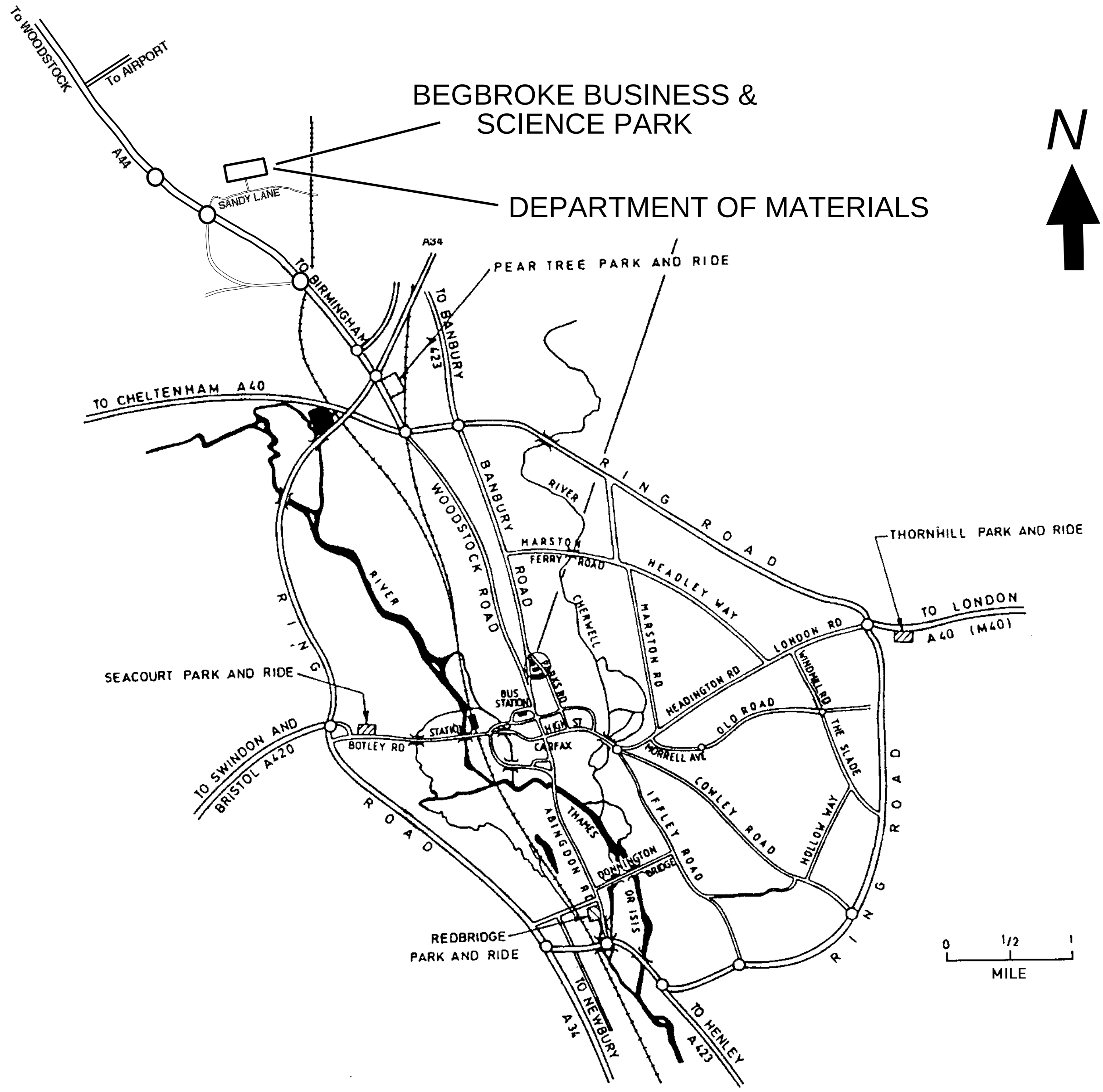
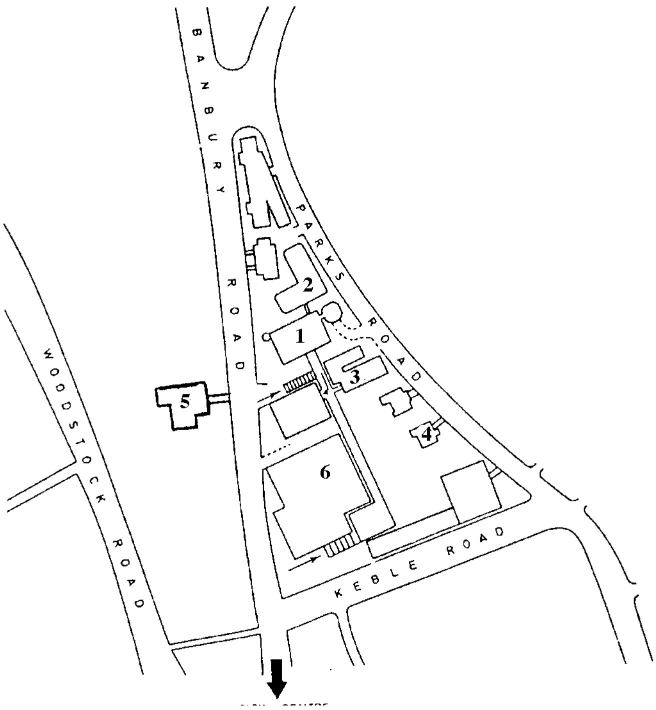
DEPARTMENT OF MATERIALS - SITES AND BUILDINGS





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Department Buildings

Holder Building (1)
Wolfson Building (2)
Hume-Rothery Building (3)
12/13 Parks Road (4)
21 Banbury Road (5)
Nuclear Physics basement (6)

Foreword from the Head of Department

Welcome to the Department of Materials at Oxford University. Our objectives are to produce world class graduate materials scientists and engineers, and to conduct world class research into the manufacture, structure, properties and applications of materials, for the benefit of the UK and world community. We were awarded top gradings for both teaching and research in the government's most recent assessment exercises, and we continue to make outstanding progress in the pursuit of our objectives. Major advances over the last three years include:

1. 3 new Professorships, in electron microscopy (David Cockayne), structural integrity (John Titchmarsh) and nanomaterials (to be appointed);
2. 2 elections to Fellowships of the Royal Society (David Cockayne and John Pethica) and 2 elections to the Royal Academy of Engineering (Richard Brook and Brian Cantor);
3. 2 promotions to personal professorships (Adrian Sutton and Andrew Briggs) and 8 promotions to readerships (Alfred Cerezo, Patrick Grant, Chris Grovenor, John Hutchison, Mike Jenkins, Amanda Petford-Long, Steve Roberts, John Sykes);
4. over £8m from the Joint Infrastructure Fund, to purchase cutting edge equipment for atomically engineered, nanoscale materials processing and analysis;
5. the launch of the new £22m Begbroke site, which greatly expands the Department's space, and sets up a unique combination of industry-linked materials research and spin-out science park; and
6. the establishment of a Faraday Partnership in aerospace and automotive materials

The Department was founded by Professor Hume-Rothery in 1956. At present, it consists of about 30 academics, 50 senior researchers, 35 technicians and secretaries, 60 postdoctoral researchers, 20 visitors, 75 research students and 120 undergraduates. The Department is part of an integrated Division of Mathematical and Physical Sciences at Oxford, which includes physics, chemistry and engineering departments, providing an ideal environment for interdisciplinary teaching and research. Fundamental developments in the physics and chemistry of materials can take place directly alongside applications in manufacturing processes and engineering design.

Materials teaching and research is increasingly important world-wide for economic prosperity. Per capita income and export earnings for a country are correlated with the size of its manufacturing industry, and the development and application of materials is a key enabling technology, as demonstrated by the government's Technology Foresight exercise. Industry depends critically on graduate employment and on innovations developed in collaboration with universities. The Department is developing strategic industrial alliances to meet industry's R&D, employment and continuing education needs.

This booklet describes the full range of our current research programmes within the Department. The Department of Materials at Oxford provides a vibrant and stimulating environment, and acts as an academic meeting point for materials scientists and engineers from all over the world. We are always pleased to discuss our research projects in more detail. We actively seek applications from new undergraduates, research students and research fellows, and we are keen to investigate further opportunities for collaboration and scientific exchanges. Please do not hesitate to contact us by letter, phone, fax or e-mail.

Oxford
October 2000

Professor G.D.W. Smith
Head of Department

Professors

Professor G.D.W. Smith, FRS	*Head of Department*
Professor B. Cantor, FREng	*Cookson Professor of Materials* *Head of Division of Mathematical and Physical Sciences*
Professor D.J.H. Cockayne, FRS	*Professor in Physical Examination of Materials*
Professor D.G. Pettifor, FRS	*Isaac Wolfson Professor of Metallurgy* *Director of the Materials Modelling Laboratory* *Director of Graduate Studies*
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Professor C.J. Peel	*Visiting Professor*
Professor J.B. Pethica, FRS	*Professor of Materials*
Professor A.P. Sutton	*Professor of Materials*
Professor M.J. Whelan, FRS	*Emeritus Professor*

Readers

Dr. C.R.M. Grovenor	*Deputy Head of Department*
Dr. P.S. Grant	*Director of Oxford Centre for* *Advanced Materials and Composites* *Director of Faraday Partnership* *in Aerospace and Automotive Materials*
Dr. M.L. Jenkins	*Director of Electron Microscopy Facilities*

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Taylor, R.N. (CASE: Nanox)
Wain, N. (EPSRC)

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Lee, H.K.(self-supporting)
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Hardwicke, R.
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Hutchinson, S.
O'Donnell, M.
Pullen, C.
Stowe, D.
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Whiteley, R.

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Fray, A. J.
Gigorov, A.V.
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Profiles of Academic Staff

Dr. Hazel Assender **Lecturer in Materials**
Linacre College

Research, both experimental and modelling, on a range of polymer and polymer composite materials. Particular areas of interest include crystallinity and morphology, hydration of polymers, the relationship between processing and microstructure and surface characterisation and modification. Modelling spans a wide range from atomistic modelling of crystal structure through mesoscale microstructural modelling and finite element continuum level simulations.

Dr. Roger Booker **Emeritus Reader in Electronic Materials**
Wolfson College **OCAMAC Senior Fellow**

Studies of semiconductor materials and devices. Group III-V's, Si and SiGe. Ingots, single and multiple layers, superlattices, quantum wells and dots. Single crystals, polycrystalline and amorphous layers. Composition, geometry, interfaces, defects, phase separation, atomic ordering, dopant distributions, diffusion. Relationship between growth methods, processing conditions, structures, optical and electrical properties. Lasers, detectors, MOSFETS, bipolar transistors.

Professor Clive Bradley **Visiting Professor**
Wolfson College

Electronic materials and systems, semiconductors, research and development management and strategy.

[Advisor to Sharp Laboratories of Europe Ltd.]

Mr. Richard Briant **Department Administrator**
St Cross College

Responsible for the departmental finances, buildings, personnel management and provision of technical and secretarial services. Focal point for research agreements and services to industry. Administrator of the Begbroke Business and Science Park.

Professor Andrew Briggs **Professor of Materials**
Wolfson College

Nanostructures and surfaces. Design and characterisation of nanocomposites for packaging. Semiconductor nanomaterials for electronics and quantum computation.

- Holliday Prize, Institute of Materials, 1984
- Metrology award for World Class Manufacturing, 1999
- Honorary Fellow of Royal Microscopical Society, 2000

Professor Richard Brook OBE FREng **Professor of Materials**
St Cross College

Processing and properties of ceramic materials.

[Currently Chief Executive at the Engineering and Physical Sciences Research Council.]

Dr. David Bucknall **Lecturer in Materials**

Structure and morphology of polymers. Effects of molecular architecture on polymer diffusion and structure. Influence of external fields in determining and controlling chain orientation and segregation behaviour. The structure and dynamics of polyrotaxanes. Nano-scale molecular devices derived from polyrotaxanes. Use of neutron reflection and ion beam depth profiling techniques for studying surfaces and interfaces.

Professor Brian Cantor FREng **Cookson Professor of Materials**
St Catherine's College **Head of Division of Mathematical and Physical Sciences**

Relationship between manufacture, microstructure and properties of engineering materials, in particular metallurgical alloys, metal matrix composites, wear and corrosion resistant coatings and thin film functional metals, ceramics and composites, particularly associated with the aerospace, automotive and packaging industries.

- Rosenhain Medal and Prize, Institute of Materials, 1993

Dr. Martin Castell **Royal Society University Research Fellow**
Wolfson College

Elevated temperature scanning tunnelling microscopy of oxide surfaces to identify atomic scale defects relevant to catalytic processes. Investigation of patterned oxide surfaces for use as templates in nanoelectronics. High resolution secondary electron imaging in the SEM of semiconductor nanostructures and devices to study local strain, dopant distributions, dopant diffusion and deactivation.

Dr. Alfred Cerezo **Reader in Materials**
Wolfson College

Investigations of solid state phase transformations on the atomic scale by a combination of high resolution microscopy and computer modelling. Development of atom probe microanalysis and its application to a range of materials.

- E.W.Müller Outstanding Young Scientist Award, Int. Field Emission Soc. 1988
- C.R. Burch Prize, British Vacuum Council, 1990.

Professor Jack Christian FRS **Emeritus Professor**
St Edmund Hall

Deformation of bcc metals and alloys, martensitic transformations, martensitic and epitaxial interphase interfaces, study of deformation twinning.

- Platinum Medal, Institute of Materials
- Acta Metallurgica Gold Medal
- Mehl Medal, AIME
- Gold Medal, Japan Institute of Metals

Professor David Cockayne FRS **Professor in the**
Linacre College **Physical Examination of Materials**

Development of electron optical techniques for investigating structure of materials; defects in crystalline material; structure of amorphous materials; refinement of structures including quantum dots and interfaces.

Dr. John Coyle FRSC **OCAMAC Senior Fellow**
Linacre College **Linacre College Industrial Fellow**

Role of technology and innovation in small and medium-sized enterprises, and the processes of forming beneficial links between such companies and the 'knowledge base' including university departments and other research laboratories.

Dr. Jan Czernuszka **Lecturer in Materials**
Trinity College

Interaction of biochemicals with ceramics. Formation of nanolaminates, composites and coatings at room temperature. Development of novel bone analogues, dry delivery systems and hierachically controlled structures.

- CBI / Toshiba Year of Invention, winner of University section, 1993

Dr. Rafal Dunin-Borkowski **Royal Society**
University Research Fellow

The application of off-axis electron holography in the field emission gun transmission electron microscope to the characterisation of magnetic and electric fields in nanostructured materials.

Professor Brian Eyre FREng. **Visiting Professor**
Wolfson College **Wolfson College Industrial Fellow**

Main areas of interest are electron microscopy studies of irradiation damage in metals and alloys and studies of the deformation and fracture processes of metals and alloys.

[Currently Chairman of CLRC, running the Daresbury and Rutherford Appleton Laboratories]

Dr. Patrick Grant **Reader in Materials**
Linacre College **Director of OCAMAC**
Director of Faraday Partnership

Advanced processing of materials, such as spray forming of metals, composites and coatings. Research has focused on the relationship between heat and mass flows and microstructures. On-line monitoring and numerical simulations are used to help understand the underlying process physics.

Dr. Geoff Grime **Lecturer in Materials**

Development of techniques for materials characterisation using focused MeV ion beams, in particular the design of high resolution focusing systems for high energy ions and data acquisition and processing systems. Application of these techniques to a wide range of scientific disciplines.

Dr. Chris Grovenor **Reader in Materials**
St Anne's College

Applied superconductivity and the processing of electronic materials. Most recent work has focused on understanding the fundamental limitations in the processing of high temperature superconducting materials and developing techniques for reliable preparation of HTS components.

Professor Sir Peter Hirsch FRS **Emeritus Professor**
St Edmund Hall

Electron microscopy of defects in crystals and modelling mechanical properties of crystalline materials in terms of dislocation processes. Recent interests include modelling the brittle-ductile transition and plastic properties of intermetallics.

- Royal Society : Hughes Medal 1973 and Royal Medal, 1977.
- Metals Society Platinum Medal 1976
- Wolf Prize in Physics, 1983
- Acta Metallurgica Gold Medal, 1997

Professor John Hunt **Professor of Materials**
St Edmund Hall

Modelling and understanding fundamental solidification processes. This has included work on eutectics, peritectics, cellular and dendritic growth. The fundamental understanding has been applied to casting processes. Recent work includes an experimental and theoretical study of twin roll casting.

- The Bruce Chalmers Award, TMS AIME, 1996.
- Rosenhain Medal and Prize, Institute of Materials, 1981.
- The C.H. Mathewson Gold Medal, TMS AIME, 1967

Dr. John Hutchison **Reader in Materials**
Wolfson College

Development of high resolution electron microscopy for structural characterisation of new materials including : CVD diamond films, quantum dots, inorganic fullerenes and complex oxides. Development of controlled environment electron microscopy for in-situ study of catalysts and of gas-solid reactions. Other interests include novel forms of carbon e.g. nanotubes.

- Glauert Medal, Royal Microscopical Society, 1975
- Vice President of the Royal Microscopical Society, 2000

Dr. Beverley Inkson **Royal Society University Research Fellow**

Mechanical properties and wear of nanostructured materials. Nanotribology. Mechanisms of deformation and failure in 'spatially confined' nanostructured systems including nanocomposites, multilayered devices and thin film coatings. Techniques used include nanoindentation, in-situ mechanical testing in electron microscopes, and 3D microstructural analysis by FIB.

Dr. Mike Jenkins — **Reader in Materials**
Jesus College — **Director of Electron Microscope Facilities**

Radiation damage, transmission electron microscopy, metal matrix composites, ceramic nanocomposites, phase stability under irradiation. Recent work has focused on fundamental mechanisms of radiation damage, especially displacement cascade processes, and mechanisms of embrittlement of pressure vessel steels.

Dr. Steven Kenny — **Lecturer in Materials**

Application of materials modelling at the atomistic level to the description of real materials. Development of ab-initio methods for the description of materials.

Dr. Oleg Kolosov — **EPSRC Advanced Fellow**
Wolfson College

Elastic and visco-elastic microstructure of materials, particularly ceramics, glasses, semiconductor quantum structures, polymer nanocomposites. Development of ultrasonic and scanned probe microscopy techniques for imaging and characterisation of surface and subsurface elastic and non-mechanical properties of materials on the micro and nanoscale. Nanoscale dynamics of ferroelectric domains.

- Metrology award for World Class Manufacturing, 1999

Dr. John Martin — **Emeritus Reader**
St Catherine's College — **OCAMAC Senior Fellow**

The relationship between the structure and the properties of metallic materials, particularly precipitation hardening, recrystallization and grain growth, fatigue and fracture.

- Sidney Gilchrist Thomas Medal and Prize, Institute of Materials, 1986.

Dr. Günter Möbus — **EPSRC Advanced Fellow**

Characterisation of materials on the atomic and nano-scale. Development of tomography techniques for 3D composition mapping and solving of crystal defect structures by high-resolution electron microscopy in combination with analytical TEM. Advanced studies on statistical image processing and dynamical simulations of electron diffraction.

Dr. Peter Northover — **Lecturer in Materials**
St Catherine's College

Non-ferrous and precious metallurgy and metalwork in ancient and historical contexts and their experimental reproduction; engineering metallurgy of the industrial revolution; very long term stability of microstructures.

Dr. Keyna O'Reilly **Lecturer in Materials**
The Queen's College

Solidification processing of advanced materials from laboratory scale simulations through to pilot scale processing plant, with particular interests in grain refinement and intermetallic phase selection. Also thermal analysis of phase transformations. Covering a wide range of materials including Al alloys, intermetallics, biomaterials, and solder alloys.

Professor Chris Peel **Visiting Professor**

Chief Scientist (Air) for Mechanical Sciences Sector, DERA looking after all aspects of aerospace technology. Specific expertise in aerospace structural materials.

Dr. Amanda Petford-Long **Reader in Materials**
Corpus Christi College

The correlation of microstructural and magnetic or optical properties of thin films with applications in information storage. The main characterisation tool is TEM, including in-situ techniques to study magnetisation mechanisms (Lorentz microscopy) in magnetic thin films, and crystallisation kinetics (in-situ TEM annealing) in phase-change optical films and optical nanocomposite films.

Professor John Pethica FRS **Professor of Materials Science**
St Cross College

Surface and nanometer scale properties of materials. Study of mechanical properties using nanoindentation and of surface atomic structure and transport processes using scanning tunnelling microscopy. Development of atom resolved AFM and force spectroscopy of single bonds.

- Sabbatical Chair, Sony corporation R&D, Japan, 1993-4
- Rosenhain Medal and Prize, Institute of Materials, 1997

Professor David Pettifor FRS **Isaac Wolfson Professor of Metallurgy**
St Edmund Hall **Director of Materials Modelling Laboratory**

Development and application of electron theory to understanding and predicting the properties of materials, in particular metals, alloys and covalently bonded semiconductors and ceramics.

- Royal Society Armourers and Brasiers' Medal 1999
- William Hume-Rothery Award, TMS 1995.
- Hume Rothery Prize, Institute of Materials, 1990.

Dr. Steve Roberts **Reader in Materials**
St Cross College

Mechanical behaviour of brittle materials, especially their response to surface deformation and the brittle-ductile transition. Studies aim at linking modelling at the defect and dislocation level with experimental studies of well-characterised materials.

Dr. Oleg Salata **OCAMAC Senior Research Fellow**

Organic Electroluminescence: novel emissive and charge transporting materials, their characterisation and optimisation. Design and fabrication of organic light emitting devices(OLEDs), modelling of device characteristics. Effects of material properties, chemical structure and thin film morphology on device performance and stability. Advanced fabrication techniques for OLEDs and OLED based displays . Engineering of organic-inorganic interfaces for improved OLED performance.

Dr. Peter Schumacher **EPSRC Advanced Fellow**

Mechanisms and kinetics of heterogeneous nucleation in metallic alloys and the mechanisms by which grain refiner additions operate, including direct transmission electron microscope observation of the devitrification of metallic glasses to observe discrete nucleation events.

- Cook Ablet Award, Institute of Materials, 2000

Dr. Jeremy Sloan **Royal Society University Research Fellow**
Wolfson College **(joint with Department of Chemistry)**

Synthesis and low dimensional crystal growth behaviour of low dimensional materials formed within single and multi-walled carbon nanotubes. Synthesis and characterisation of inorganic fullerene-like structures. Physical properties determination.

Professor George Smith FRS **Professor of Materials**
Trinity College **Head of Department**

Phase transformations, atom probe analysis. Studies of the role of alloy elements and trace additions on the microstructure, heat treatment and properties of steels and non-ferrous alloys. Atomic scale studies of heterogeneous catalysts.

- Rosenhain Medal and Prize, 1991.
- Sir George Beilby Medal and Prize, 1985.

Professor Adrian Sutton **Professor of Materials Science**
Linacre College

Modelling of materials at the atomic and microstructural levels. Computational materials synthesis. STM and surface structure of Mott-Hubbard insulators. Mechanical and electrical properties of nanocontacts. Interfaces. Dislocations. Microstructural evolution due to phase transformations.

Dr. Ian Stone **Senior Research Fellow**

Processing-microstructure relationships in alloys and metal matrix composite systems. Evolution of microstructure during the spray forming process, grain-growth in the semi-solid state, deformation behaviour of semi-solid alloys. Characterisation of the spatial distribution of reinforcements in composite materials and its effect on their processing and properties.

Dr. John Sykes **Reader in Materials**
Mansfield College

Corrosion of metals. Conversion treatments for aluminium, protection by organic coatings, studies of coating breakdown. Passivity, chloride-induced pitting, corrosion of steel in concrete.

Dr. Glyn Taylor **Senior Research Fellow**
Linacre College

Mechanical properties of metallic materials, especially the deformation of single crystals. Growth of single crystals for deformation studies, bcc metals and alloys containing oxide or nitride dispersions, intermetallic compounds including γ-TiAl and various B2 compounds. Relating yield stress and strength to the properties of dislocations. Measuring elastic constants.

Professor John Titchmarsh **RAE Research Professorship in Microanalysis and Structural Integrity**
St Anne's College

Techniques for electron microscopy materials analysis: electron energy loss spectroscopy and X-ray analysis. Mechanical properties, precipitation and segregation in nuclear reactor alloys. Surface engineering. Extraction of information using chemometric techniques.

Dr. Richard Todd **Lecturer in Materials**
St Catherine's College

Mechanical properties of ceramics and metals. Most research revolves around oxide ceramics, thermal residual stresses, neutron and X-ray diffraction, and superplastic metals. Current interests include the processing and mechanical properties of alumina matrix nanocomposites, residual stresses in thermally sprayed coatings, characterisation of cold worked microstructures using diffraction peak profile analysis, and projects on the superplastic forming and diffusion bonding of commercial alloys.

Dr. Drahosh Vesely **OCAMAC Senior Fellow**
Wolfson College

Light and electron microscopy, electron beam damage and spectroscopy are used to study crystallographic morphology of spherulitic structures, nucleation and crystallization, stabilization, degredation, electrical conductivity, fluorescence, diffusion, permeability, solubility and mechanical properties of polymeric compounds.

Dr. Tiffany Walsh **Glasstone Research Fellow**
Linacre College

Modelling interfaces between hard and soft matter at atomic and mesoscopic levels. Efficient implementation of perturbation theory in periodic systems. Development of techniques to accurately calculate weak interactions between molecules and metal surfaces. Modelling adhesion of polymers to inorganic substrates. Modelling of self-assembled monolayers on inorganic surfaces.

Dr. Paul Warren **Lecturer in Materials**

Nanostructured materials. Non-equilibrium processing combined with structural and chemical characterisation on the nanometre scale using a range of microanalytical techniques. Materials under investigation range from conventional precipitation hardened aluminium alloys through to novel amorphous, nanocrystalline and nanoquasicrystalline alloys.

Professor Mike Whelan **Emeritus Professor**
Linacre College

Transmission electron microscopy of materials, transmission electron diffraction of thin specimens (theory and application to crystal lattice defect observation). Reflection electron diffraction of surfaces (theory and applications to molecular beam epitaxial growth).

- Distinguished Scientist Award, Microscope Society of America, 1998
- Hughes Medal, Royal Society, 1988
- C.V. Boyes Prize, Institute of Physics, 1965

Dr. Angus Wilkinson **Royal Society University Research Fellow**
Corpus Christi College **Lecturer in Materials**

Mechanics at the microscopic scale, both experimental and modelling. Dislocation modelling of fatigue and fracture processes. Development of SEM based diffraction methods (ECCI and EBSD) for imaging lattice defect distributions and measuring local internal strain distributions.

Dr. Peter Wilshaw **Lecturer in Materials**
St Anne's College

Characterisation of the electrical and mechanical properties of defects in semiconductors. The development of improved semiconductor substrates. Development of novel structures and materials for field emitters to be used in field emitter displays. Improved biomaterials for prostheses.

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This figure shows how the deformation of nanomaterials can be analysed in 3D by focused ion beam (FIB) microscopy.

The top image shows a Cu-Al multilayer, deposited on an Al_2O_3 substrate by MBE, which has been deformed by nanoindentation, and then cross-sectioned using a focused beam of Ga^+ ions. Computer reconstruction of a series of such 2D sections through the nanoindentation site, enables the deformation of each of the individual subsurface layers to be mapped in 3D for the first time. The lower image is a 3D map of the total multilayer thickness around the indentation site, clearly showing the 3 pile-up zones occurring after indentation with a 3-fold Berkovich indenter tip.

Dr. Beverley Inkson and Timothy Steer

A. Structure and Mechanical Properties of Metals

I - INTERMETALLICS

Mechanical Properties of γ -TiAl

N. Bird, Dr. S. Jiao, Professor Sir Peter Hirsch, Dr. G. Taylor*

Single crystals of γ - Ti 54.5% Al with various orientations have been deformed over a range of temperatures, and the dislocation structures analysed by TEM. Mechanisms are being developed to explore the yield stress anomalies observed for slip on 1/2<110]{111}, 1/2<112]{111} and <011]{111} systems, and why the yield stress for <011]{111} slip is reversible with temperature, but that for 1/2<110]{111} slip is not. (*Argonne National Laboratory, Argonne IL,USA) (Funded by EPSRC and DERA Pyestock)

3D microstructural characterisation of intermetallics

Dr. B.J. Inkson, Dr. M. Bobji, Dr. P. Threadgill, Professor H. Clemens***

The 3D microstructures of TiAl and FeAl intermetallics, processed by rolling and friction welding, are being determined by a new technique of 3D FIB analysis, combined with TEM and EBSD. This enables the grain shapes and orientations in 3D to be directly correlated with local mechanical properties across non-uniform microstructural features. (*The Welding Institute, Cambridge, **GKSS Germany)

Growth of single crystals and deformation characteristics of binary RuAl and other B2 alloys

S.J. Pak, Dr. M.L. Jenkins, Dr. G. Taylor

RuAl has a high melting point and the vapour pressure of Al when the alloy is molten is sufficiently high to make the growth of single crystals difficult. A floating-zone crystal growth apparatus is being constructed for operation at above ambient pressure in an attempt to grow good quality crystals of RuAl and other B2 intermetallic compounds. The mechanical properties of the crystals will then be studied by a variety of methods and dislocation structures characterised by transmission electron microscopy.

Mechanical properties of lamellar TiAl and effects of purity and composition

*Dr. G. Taylor, Professor S. Naito**

Polysynthetically twinned crystals may be formed during growth of TiAl binary alloys depending on composition and growth conditions. A series of binary alloys with very high purity has been made for the study of mechanical behaviour and yield stress values over a range of temperatures. (*Kyoto University, Japan)

Mechanical properties of CoTi based alloy single crystals

L. Zhang, Dr. M.L. Jenkins, Dr. G. Taylor

CoTi crystals show a yield stress anomaly characteristic of certain intermetallic compounds. The peak-stress temperature and strength of the stoichiometric binary alloy are relatively low. The addition of isostructural CoZr or CoHf is expected to increase these parameters significantly. The mechanical properties of the ternary alloys will be measured and the associated dislocation structures characterised by electron microscopy.

Mechanical properties of RuAl and (Ru,Ni)Al alloys

Dr. A.L.R. Sabariz, Dr. G. Taylor

The ruthenium aluminium system forms an intermetallic compound with the B2 structure at 50 % Al. In the main Ni can be substituted for Ru without change of structure and the solid-solution hardening adds considerable strength to the material. The mechanical properties are being studied by compression tests and transmission electron microscopy.

Transition metals in TiAl

Dr. K. Xia, Dr. B.J. Inkson, Professor B. Cantor*

Microstructures of γ + $\alpha 2$ + B2 Ti - Al alloys are being examined as a function of processing conditions and ternary alloying additions at the atomic scale using high resolution chemical and structural electron microscopy analysis techniques.(*University of Melbourne, Australia)

Simulation of weak-beam images of defects in γ-TiAl

Dr. S. Jiao, Professor D.J. H. Cockayne, Professor Sir Peter Hirsch*

Weak beam images of 1/2[112> dislocations in γ-TiAl are being simulated using the CUFOUR programme with a view to distinguishing between possible alternative structures of these defects. (*Argonne National Laboratory, Argonne IL,USA)

Dislocation dipoles in γ-TiAl

Professor Sir Peter Hirsch

Faulted dislocation dipoles are a destructive feature of the deformation of γ-TiAl at low temperatures. A new model is being developed to explain the formation of these defects.

II - NANOCRYSTALLINE MATERIALS

Metallic nanocrystalline materials

M. Abraham, Dr. A. Cerezo, Professor G.D.W. Smith

Electrodeposition is being investigated as a possible method for the production of single phase, multiphase and alloy nanocrystalline materials. Apart from its simplicity, this method has the advantage that it can produce large quantities of material without the need for subsequent consolidation. However, the alloy compositions need to be chosen to produce a microstructure which resists rapid grain growth during service. Nickel- and iron-based alloys produced by electrodeposition are being characterised by X-ray diffraction, TEM and field-ion microscopy/atom probe techniques. (Funded by EPSRC in collaboration with Carpenter Technology)

FIB characterisation of surface damage generated by nanoindentation, scratch testing and abrasion

T. Steer, H. Wu, P. Boon, Dr. M.S. Bobji, Dr. B.J. Inkson

Surface plastic deformation in multilayers and nanocomposites, generated by nanoindentation, scratch tests and abrasion, are being systematically characterised by high resolution electron microscopy and 3D FIB. (Funded by EPSRC and The Royal Society)

Microstructure and mechanical properties of thin films and nanowires

Dr. B.J. Inkson, Dr. M.S. Bobji, Professor E. Arzt, Dr. G. Dehm*, Dr. Th. Wagner*, Dr. O. Kraft**

High strength multilayered thin-films, grown on ceramic substrates by molecular beam epitaxy (MBE), are subjected to controlled static (indentation) and cyclic (thermal and stress) mechanical deformation. The resulting evolution of the thin film microstructures is quantified by 3D FIB and electron microscopy down to the atomic level. (*Max-Planck-Institut fur Metallforschung, Stuttgart, Germany) (Funded by The British Council, DAAD and The Royal Society)

Nanoscale deformation of materials quantified by TEM nanoindentation

Dr. M.S. Bobji, Dr. B.J. Inkson, Professor J.B. Pethica, R.C. Doole

A novel nanoindenter is being built to enable the impact and deformation of nanostructured materials to be observed in real time inside a TEM down to the atomic level. Mechanisms of deformation will be correlated to the applied load, indenter morphology, substrate microstructure and chemistry. (Funded by EPSRC and The Royal Society)

III - MECHANICAL PROPERTIES OF STRONG SOLIDS, METALS AND ALLOYS

Deformation of single crystals of Nb-Zr-O alloys and Nb-Zr-N alloys

Professor J.W. Christian, Dr. P. Manyum, Dr. G. Taylor

Single crystals of niobium zirconium alloys are being oxidised at low pressures in an ultra-high vacuum furnace to produce a zirconia dispersion. The size of the precipitate is controlled by a subsequent anneal at ~1600°C. Mechanical properties are being studied by differential tensile tests and the particle-matrix structure and dislocation-particle interactions observed by transmission electron microscopy. Similar experiments on Nb-N solid solutions are being carried out also at deformation temperatures well below ambient.

Diffusion bonding of zirconium alloys

P.S. Hill, Dr. R.I. Todd, Dr. N. Ridley**

Diffusion bonding is a solid state joining method for metallic alloys. Advantages include minimal distortion of the product and the ability to join thick sections. Diffusion bonding is already used extensively for titanium alloys and duplex stainless steels. The aim of this project is to extend this useage to zirconium alloys, which are employed in nuclear power generation and chemical plant. Preliminary experiments have demonstrated the feasibility of diffusion bonding zirconium, and the research is now concentrating on establishing the mechanisms of bond formation, so that modelling can be used to predict bonding conditions and surface treatments suitable for commercial application. (*University of Manchester) (Funded by EPSRC and Rolls Royce & Associates Ltd.)

Microstructure and mechanical properties of 7xxx and 8xxx alloys

Dr. I.G. Palmer, Professor B. Cantor

Electron microscopy, heat treatment and mechanical testing methods are being used to investigate the microstructure and static and cyclic mechanical properties of 7xxx and 8xxx alloys. The results are being used to optimise the materials for applications in airframe and other components. (Funded by DERA Farnborough)

Mechanical properties of semi-solid slurries

H.S. Kim, S.B. Park, Dr. I.C. Stone, Professor B. Cantor

Viscometry methods are being developed in order to measure the mechanical response of semi-solid slurries. The results are being used to determine semi-solid constitutive laws, and are being applied to a variety of metallurgical manufacturing processes, particularly for the Al alloy automotive components. (In collaboration with KIST)

Fundamentals of cyclic deformation and fatigue crack initiation

Dr. A.J. Wilkinson, Dr. S.G. Roberts, Dr. J.W. Martin

Single crystal specimens are being tested in fatigue under constant plastic strain amplitude conditions. The dislocation microstructures produced are being examined using electron channelling contrast imaging, a novel SEM technique, and their evolution is being modelled by computer simulation. The objective is an understanding of dislocation patterning and subsequent initiation and early growth of fatigue cracks.

Measurement and development of residual stresses in coatings

*S. Saunders, Dr. R.I. Todd, Dr. J. Lord**

We are developing a robust method for measuring residual stresses in coatings both during deposition, and as they develop during simulated service. The method is based on the measurement of the curvature produced in coated substrates by the residual stresses. Although the basic method is well established, there is considerable uncertainty surrounding the assumptions used in interpreting the results. We are investigating these systematically using both commercial coating compositions, and model materials which can be selectively removed following deposition so that the effect of the deposition process itself on the stresses in underlying layers can be ascertained. The work is currently concentrating on thermally sprayed coatings, but the methodology developed may also be applied to other coating techniques. (*National Physical Laboratory) (Funded by EPSRC and NPL)

Mechanical properties of the bond coat in thermal barrier coatings

R.O. Howells, Dr. R.I. Todd, J. Wigren, P. Bengtsson**

The mechanical properties of air plasma sprayed NiCoCrAlY bond coats are being investigated, and the results are being used to model the thermal residual stresses which accrue during deposition and subsequent thermal cycling. Direct measurements of the thermal residual stresses are being made for comparison by measuring the curvature of coated substrates during thermal cycling. (*Volvo Aero Corporation)

Carbide cracking and the brittle-ductile transition in ferritic steels

M. Coates, Dr. S. Noronha, Dr. A.J. Wilkinson, Dr. S.G. Roberts

The effects of brittle carbides on the fracture behaviour and brittle-ductile transition in ferrite is being studied experimentally, and the evolution of dislocation arrays around crack tips and these particles is being modelled. The objectives are to understand the basic processes leading to cleavage fracture in steels, and hence to underpin the FEM-based models used in safety codes (Funded by EPSRC in collaboration with AEA Technology, HSE and NII)

Physical properties of Li-Mg alloys

Professor J.W. Christian, Dr. G. Taylor, Dr. M.E. Siedersleben, Professor S. Naito***

Thermal expansion coefficients and the values of elastic constants C11, C12 and C44 are being studied over the temperature range 4-300K for alloy compositions between 40 and 70at% magnesium. (*Honsel Werke Reichmetalle, Germany ; **Kyoto University, Japan)

Growth of short fatigue cracks

*Dr. A.J. Wilkinson, Dr. J.W. Martin, Dr. P.A.S. Reed**

The growth rates of short fatigue cracks are being measured and correlated with crack path through the local microstructure. Experiments on Al-Li alloys showed that cracks grow on {111} slip planes that minimise crack twist deflections at grain boundaries. The crack retardation is largest at boundaries where the minimum twist deflection is relatively large. Further experiments are planned for a nickel based superalloy at room and elevated temperatures. (*Materials Research Group, University of Southampton).

Microstructure-property relationships in commercial superplastic 7475-Al alloys

*Dr. D. Whittle, Dr. R.I. Todd, Dr. N. Ridley**

The microstructural features controlling flow stress and cavity nucleation during superplastic forming of commercial 7475-Al alloys are being investigated. The microstructures of material deformed using both conventional uniaxial deformation and sheet forming of cones are being examined using FEG-SEM and EBSD. The influence of back pressure on cavitation is also being revisited. (*University of Manchester) (Funded by British Aerospace Military Aircraft & Aerostructures)

Mechanical properties of squeeze cast Al alloy composites

M. Fuller, Dr. P. Schumacher, Professor B. Cantor

Squeeze casting is being used to manufacture Al alloy near net-shape components containing local reinforcements of various types. A combination of mechanical testing and numerical modelling is being used to determine the composite mechanical properties and to optimise the resulting material. (Funded by EPSRC and Lucas)

3-D Imaging of fatigue cracks using x-ray micro-tomography

Dr. A.J. Wilkinson, Dr. G.R. Davis, Prof. J.C. Elliott**

X-ray micro-tomography is being used to study the three dimensional geometry of fatigue cracks. The change in size and shape of small natuarally initiated fatigue cracks in Al-Li alloys is being measured as a function of number of applied load cycles. In situ loading is to be used to study the premature closure of both long and short fatigue cracks. (*Biophysics in Relation to Dentistry, Queen Mary and Westfield College, London)(Funded by EPSRC through JREI scheme)

Residual stress and property development in plasma sprayed bond coats during thermal cycling and oxidation

H. Thompson, Dr. R.I. Todd

Recent work in the Department has measured the residual stresses in air plasma sprayed NiCoCrAlY coatings as they develop during the first thermal cycle after spraying. This project is extending the work to investigate the development of the residual stresses and changes in bond coat properties during continued cycling and oxidation.

Characterisation of cold worked microstructures in aluminium alloys

*N.J. Akram, Dr. R.I. Todd, Professor F.J. Humphreys**

The main aim of the project is to use X-ray diffraction peak broadening to characterise cold worked microstructures which are too dense and complex to be resolved in the TEM. Previously developed analysis methods will be adapted and tested using simple alloys with microstructures which can be characterised by alternative methods. Complementary techniques such as measurement of the thermoelectric effect will also be included in the investigation. (*UMIST) (Funded by EPSRC and Alcan International Ltd.)

The dependence of mechanical properties of microalloyed steel strip on microstructural changes induced during the rolling process.

W. Hicks, Professor J.M. Titchmarsh

The project will employ TEM and microanalysis, concentrating on correlating the number densities, sizes, distributions and compositions of small second phase precipitates with mechanical properties measured at Corus (South Wales) who are part-funding the research.

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This figure show (a) HRTEM image of a 2x2 KI crystal in a 1.4nm diameter SWNT; (b) structural representation of (a) (inset: end-on view); (c) Reconstructed image of a 3x3 KI crystal in a 1.6nm SWNT; (d) Structural representation of (c) (inset: end-on view).

Recently, we showed that 2x2 and 3x3 atomic layer thick KI crystals can be formed in SWNTs as a function of their diameter. These crystals frequently have reduced co-ordination (e.g. the K and I atoms in the 2x2 crystal are 4:4 coordinated) and exhibit lattice distortions compared to the bulk halide. An enhanced HRTEM image-restoration technique developed by Angus Kirkland and Owen Saxton at Cambridge University makes possible an atom-by-atom reconstruction of these crystals, the first time that crystallography has been attempted on such a scale.

Dr Jeremy Sloan

B. Non-Metallic Materials

I - CERAMICS AND COMPOSITES

Processing and properties of nanophase ceramic coatings

S. Jiansirisonboom, Dr. P.S. Grant, Dr. S.G. Roberts

Alumina/SiC nanocomposite powders are being manufactured in-house using both freeze drying and sol-gel routes. Powders are then consolidated into alumina/SiC coatings by vacuum plasma spraying. The coating microstructure is being investigated by a combination of transmission electron microscopy, X ray diffractometry and nuclear magnetic resonance, as a function of the manufacturing conditions. The microstructural features are then being used to explain coating behaviour in comparative mechanical tests with monolithic alumina coatings. (Funded by Thai Government)

The influence of heat treatment on the strength of ceramic nanocomposites

H. Wu, Dr. S.G. Roberts

Annealing increases the strength of alumina/silicon carbide ceramic nanocomposites. To investigate this, we are using acoustic and Hertzian indentation techniques to investigate the effects of heat treatment on the defect population and strength in these materials before and after surface machining.(Funded by EPSRC)

Surface mechanical properties of alumina/SiC nanocomposites

J.L. Ortiz Merino, Dr. R.I. Todd

Previous work has shown that the addition of a small volume fraction of nanoscale SiC particles to a conventional alumina matrix can significantly strengthen the alumina grain boundaries. One manifestation of this effect is a dramatic reduction of the surface grain pullout during surface abrasion which is prevalent in unreinforced alumina. This leads to improved surface finish and wear resistance. The reason for the grain boundary strengthening is being investigated by studying the response of alumina matrix nanocomposite surfaces to abrasion as key microstructural features (grain size, particle size, particle location, particle properties) are varied systematically. (Funded by the Mexican Government)

Nanocomposite ceramics for technical applications

P. Boon, Dr A.M. Cock, Dr. S.G. Roberts, Dr. R.I. Todd, Dr. B.J. Inkson, Professor J.M. Titchmarsh

The project is aimed at use of alumina - silicon carbide ceramic nanocomposites in applications where wear and abrasion resistance are important. The project is focussed on the surface mechanical properties of sintered ceramic nanocomposites. There are two main aims - (a) to understand the mechanisms of their improved properties over normal alumina ceramics; (b) to produce materials usable in real industrial applications. This project is in collaboration with Morgan Matroc.

Composites based on synthetic opal

*Dr. J.L. Hutchison, Professor L.M. Sorokin**

Novel composites have been prepared by filling the regular voids in synthetic opal by guest materials such as tellurium, InSb, GaAs, etc.. The opal is a cubic-close-packed lattice of SiO2 spheres, and it has been found that the guest materials may be present as a single-crystalline, 3-D networks, giving unusual properties. (*In collaboration with the Ioffe Physical-Technical Institute, St Petersburg, Russia, supported by the Royal Society)

Thermal shock of alumina-SiC nanocomposites

S. Maensiri, Dr. S.G. Roberts

We are examining the thermal shock resistance and failure mechanisms in sintered alumina - silicon carbide "nanocomposite" ceramics. (Funded by the Thai Government)

High temperature properties of float glass

*M. O'Donnell, Dr. S.G. Roberts, Dr. P.D. Warren**

This "Part II" project is investigating the high temperature flow and fracture properties of float glass. It aims to understand the mechanisms by which glass gets damaged during the float glass process. (In collaboration with *Pilkingtons Glass)

Ultrahard multilayered ceramic coatings

*T. Steer, Dr. B.J. Inkson, Dr. K. Cooke**

Mechanisms of improvements in mechanical strength achieved by grain size reductions and lamellar spacing of nitride and boride ceramic multilayers are being quantified using indentation, cross-sectional TEM studies and HREM. (*Teer Coatings Ltd.) (Funded by EPSRC)

Abrasion mechanisms of alumina

Dr. B.J. Inkson, Dr. G. Möbus

The abrasion mechanisms of alumina are being studied at the atomic scale. High resolution cross-sectional electron microscopy studies of abraded surfaces are being carried out to resolve the structure of deformation twins and dislocation core structures.

Residual stresses and mechanical properties in oxide matrix nanocomposites

N. Wain, Dr. R.I. Todd

Preliminary results have shown that MgO/SiC nanocomposites have greatly improved strength and toughness compared to unreinforced MgO. The project aims to identify the mechanisms involved, with a focus on the large thermal residual stresses, in excess of the yield stress, which are to be expected in this system. The work will be extended to investigate the importance of such effects in other oxide matrix systems such as alumina/SiC.

II - BIOMEDICAL MATERIALS

Bone biomaterials bonding

*Dr. J.T. Czernuszka, Professor J.J. O'Connor**

The micromechanical properties of the biomaterial/long bone interface are being measured. This allows us to measure externally the incorporation rates of the biomaterial. (*Oxford Orthopaedic Engineering Unit)

In situ formation and electrodeposition of active coatings

S. Wilkinson, Dr. J.T. Czernuszka

Electric fields are used to regulate the precipitation rates of sparingly soluble solids. Biologically active coatings have been fabricated and we are now extending the process to other systems. (Funded by EPSRC)

Mechanical properties of biocomposites

Dr. J.T. Czernuszka

Composites based on natural systems are being made and their dynamic mechanical and fracture response determined. New models of how this class of materials deform are being formulated.

An improved bone-implant interface

E. Briggs, M. Karlson, Dr. E. Palsgard*, Dr. P.R. Wilshaw*

A new coating for metal implant prostheses is being developed. This entails bonding a layer of porous alumina to the metal surface and filling the pores with a bioactive material such as bioactive glass. It is hoped that in this way the strength of the interface between the bone and implant will be improved whilst the mechanical properties of the implant are maintained. (*Centre for surface biotechnology, Uppsala University, Sweden)

Modelling phospholipid monolayers at the alveolar interface

Dr. I. Gentle, K. Gunton*, Dr. D.G. Bucknall*

Phosphatidylcholine molecules play an important role in the action of natural lung surfactants by supplying lipids to the alveolar monolayer. The exact mechanism for this process remains uncertain. Using self-assembling layers in a Langmuir-Blodgett apparatus, surface pressure, neutron reflectivity and Brewster angle microscopy studies of the compression-expansion cycles of these systems is being used to investigate the physiological behaviour within a lung. (*Department of Chemistry, Queensland University) (Funded by Australian Government)

In vitro approaches to bone formation

Dr. J.T. Triffitt, Dr. J.T. Czernuszka, S. Wilkinson*

Processes are being developed that encourage bone formation on a laboratory scale. The control and manipulation of osteoblasts is of the utmost importance. (*Nuffield Department of Orthopaedic Surgery) (Funded by EPSRC and in collaboration with MRC Bone Research Lab.)

Tissue Engineering and three-dimensional scaffolds

E. Sachlos, Dr J.T. Czernuszka, Professor Z.F. Cui, Professor B. Derby***

A three dimensional printing method is being developed to promote the alignment, proliferation and differentiation of cells. The project will examine various cell types. (* Dept of Engineering Science, ** UMIST)

Studies on the precipitation of calcium phosphate

Dr. P. Fewster, Dr P. Kidd * Dr. J.T. Czernuszka*

The kinetics of calcium phosphate precipitation are being determined and the products analysed. X-ray diffraction techniques and modelling are being used to determine the phases present, their proportions, morphologies and preferred orientations. Comparison with other techniques will be made throughout. (*Philips Research Labs)

Design and fabrication of ceramic: biochemical: polymer composites

Dr. J.T. Czernuszka, Professor E. Bres, Professor W. Hosseini***

Additions of biochemicals, such as amino acids or lipids, either to the growth medium or onto the surface of polymeric substrates influence strongly the morphology and crystallographic orientation of deposited ceramics. This is being used to create tailored composites and structures.(*University of Lille ; **University of Strasbourg)

Macro-assembled spheres of apatite

T. Pongsaanutin, S. Wilkinson, Dr. J.T. Czernuszka

Lipid spheres are being coated with apatite which are then deposited on to metallic surfaces. We have hierarchical control of the macro-assembly on 5 length scales.(Funded by Wellcome Trust and Thai Government)

Nanolaminated composites

Dr. J.T. Czernuszka

Biochemicals are reacted with inorganic salts to form layered structures compromising alternating monomolecular sheets of biochemicals and ceramic monolayers. These materials possess novel ferro-electric, elastic and optical properties.

III - POLYMERS

The hierarchical structure of spider silk

*Dr. J.Y.J. Barghout, Dr. J.T. Czernuszka, Professor C. Viney**

Spider silk microstructures on the scale of 1 nanometer to 1 micrometer are being characterised by transmission electron microscopy / diffraction. Molecular modelling is being used to identify particular amino acid sequences that can account for the observed microstructural features, which in turn are associated with specific mechanical properties. (*Heriott-Watt University) (Funded by EPSRC)

The response of poly(vinyl alcohol) to humidity

J. Spindura, Dr. H.E. Assender

The control of water solubility in poly(vinyl alcohol) is achieved industrially using heat and chemical treatments, the effect of which are not fully understood. The uptake of water into the polymer prepared as films and fibres, and its effect on crystallinity and morphology as well as the effect of heat/crosslinking treatments upon this process is being examined.

Mesoscale modelling of processing rubber toughened polymers

K. Porfyrakis, Dr. H.E. Assender

A mesoscale model for predicting the microstructure of a rubber toughened polymer after typical processing treatments such as extrusion and injection moulding. The distribution and deformation of rubber toughening additives will be modelled using calculations of flow history during processing made from finite element models.

Polymer adhesives with good interfacial bonding

Dr. H.E. Assender

A novel system is under development to enhance interfacial bonding in a polymeric adhesive system. The research includes characterisation of nanoscale interfacial defects, investigation of diffusion and demixing in polymer blends, encapsulation technology and mechanical testing of joints. (In collaboration with DERA)

Real time studies of polymer interfaces

Dr. D.G. Bucknall, Dr. S.A. Butler, Professor J.S. Higgins***

This project is developing the methodology, techniques and apparatus required to conduct neutron reflection experiments in real time. The technique has been applied to investigate the diffusion of oligomers and plasticisers into polymers and to study the subsequent dissolution of the polymer films. This is the first time that such measurements have been successfully carried out in real time, providing new information and insight into the processes involved. (*Department of Chemistry, Cambridge University; **Department of Chemical Engineering, Imperial College) (Funded by EPSRC)

Structure and dynamics of polyrotaxanes

*Dr. D.G. Bucknall, Professor H.W. Beckham**

Polyrotaxanes are a novel polymer which consist of macrocycles treaded onto the polymer chain. The incorporation of these rings on the chain can have a dramatic effect on the physical properties as observed by its structure and dynamics. Using a combination of solid state NMR, quasi-elastic and small angle neutron scattering, and X-ray diffraction the molecular basis for these physical changes are being investigated. (*Department of Textile and Fiber Engineering, Georgia Institute of Technology) (Funded by NATO)

Effects of molecular architecture on polymer interdiffusion

Dr. D.G. Bucknall, Dr. N. Clarke, Dr. J.H.G. Steinke**, Professor J.S. Higgins****

Although the idea of polymer diffusion via reptation is well understood for linear polymers, the same is not so true for non-linear polymers. This project is studying the effect of molecular architecture on the diffusion process for a set of chemically identical polymers, and using this model system to interpret the behaviour under the framework of the reptation model. (*Department of Chemistry, Durham University; **Department of Chemistry, Imperial College; ***Department of Chemical Engineering, Imperial College)

Oxidative degradation of polymers

T. Burman, Dr. H.E. Assender, Dr. D. Vesely

The mechanism of oxidation is investigated from the point of view of formation and diffusion of free radicals. Dispersion, solubility and diffusion of anti-oxidants is correlated with Oxidation Induction Time test for different antioxidants. Evaporation and degradation of anti-oxidants, as well as oxidation rates of polyolefins in different halogen environments are investigated. The main aim is the explanation of the mechanism in which the oxidation results in loss of mechanical properties. (Funded by EPSRC and National Power)

Structure and properties of silk

*J. Trancik, Dr. J.T. Czernuszka, Professor C.Viney**

A variety of spider and insect silks are being characterised by transmission electron microscopy/diffraction. The aim is to correlate microstructure to mechanical properties, in the context of silk evolution.(*Heriott-Watt University) (Funded by The Rhodes Trust)

Mechanical properties of polymer interfaces

Dr. R. Matthews, Dr. D. Vesely

Molecular interactions and entanglement at interfaces of homo-polymers and polymer blends are investigated and correlated with interfacial fracture. Stress distribution at interfaces with different microstructure are studied by finite element analysis with the aim to establish the conditions for the formation of microcracks. The results are applied to the mechanical behaviour of polymer welds, polymer blends and composites. (Funded by National Power)

Thin film properties of macrozwitterions under the influence of an electric field

Dr. D.G. Bucknall, Professor R.W. Richards, Dr. L.R. Hutchings**

Macrozwitterions have slightly different properties in thin film compared to the parent unfunctionalised homopolymer, due to segregation of the oppositely charges end groups. The molecular structure and orientation can be drastically altered by application of external fields when in the melt. This project is studying the effects of applying an external field to the polymer morphology within these thin films, not only to understand the orientation behaviour in restricted geometry but also to be able to manipulate the film properties. (*IRC in Polymer Science and Technology, Durham University)

Development of ion-permeable membranes

P. Chandrapalan, Dr. H.E. Assender

Various chemical and physical treatments may be applied to ion-permeable membranes to modify their performance. Such developments are attempted and charaterised in terms of the extent and nature of the modification. (Funded by EPSRC and National Power)

Microstructure of cured unsaturated polyester (UP) matrices

*D. L. Morgan, Dr. H. Assender, S. Clarke**

The microstructure and crosslinking of a styrene fumarate copolymer formed during the cure of UP resins is investigated dependant upon resin compositions, cure system/conditions and degree of cure. The role of post-curing treatments is also under investigation.

Diffusion and solubility in polymers

Dr. D. Vesely

Accurate measurement of diffusion rates, solubilities and concentration profiles are used to establish thermodynamical parameters, which can explain the observed mechanism of diffusion process. Polymer solvents, as well as compatible polymers with upper and lower critical solubility temperatures are investigated. Two component phase diagrams are compared with three component phase diagrams, in which the third component is a solvent or a compatibilizer. The results are used to advance our understanding of the formation of microstructrure in immiscible, miscible and compatibilized polymer systems.

Diffusion in composite materials

Dr. D. Vesely

Diffusion of compounds through inhomogeneous polymeric materials is investigated from the point of view of diffusion rate and solubility. The size and distribution of the second phase is taken into account for the calculation of the diffusion path and for the permeability. Several diffusion mechanisms are considered and compared with the experimental results.

Creep, fatigue and environmental stress cracking of polymers

Dr. R. Matthews, Dr. D. Vesely

Crack propagation under different types of loading of polyethylene samples in an oxidative environment is investigated. The response of the polymer to stress concentration is critically dependent on the physical and chemical state of the microstructure. The conditions for acceleration or deceleration of the crack propagation are being established. (Funded by National Power)

Gloss of polymers

K. Porfyrakis, Dr. H.E. Assender

The gloss of a material is a measure of the specular reflectance of light from the surface. The gloss depends upon the roughness of the surface. AFM examination of a range of polymer surfaces is being applied to allow quantitative analysis of the roughness to link the topography of a surface to the measured gloss. Related scanning force microscopy techniques, such as UFM, are used to monitor the morphological origin of the roughness.

Luminescent Conjugated Polyrotaxanes

Dr. H.L. Anderson, Dr. D.G. Bucknall, Dr. F. Cacialli**, Professor R.H. Friend***

Conjugated polymers have many potential applications, particularly as organic semi-conductors and electroluminescent display materials. We are developing a way of improving the luminescence, stability and processability of these polymers by insulating with threaded macrocyclic rings to form polyrotaxanes. This work represents the first use of rotaxane formation to control the optoelectronic properties of a conjugated polymer chain, by isolating, insulating and encapsulating it. These new materials will provide fundamental insights into the behaviour of conjugated polymers by controlling inter-chain separation, so blocking short range inter-chain processes. (*Dyson Perrins Lab., Oxford University, **Cavendish Lab, Cambridge University)

Electric Field Induced Orientation of Zwitterionic Telechelic Polymers

Dr. D.G. Bucknall, Dr. L.R. Hutchings, Professor R.W. Richards**

Zwitterionic telechelic polymers are ionomers with oppositely charged end-groups, which in solution can cluster into aggregates or behave as single chains depending on the polarity of the solution. We have been using electro-optic Kerr birefringence to understand the complex solution properties of these novel polymers. Due to the presence of the permanent dipoles on the chain ends orientation effects are highly sensitive to aggregation behaviour allowing different field alignment effects to occur. The segmental orientation of the chain therefore acts to produce an optical switch. (*IRC in Polymer Science and Technology, Durham University)

Nano-structures derived from polyrotaxanes

Dr. D.G. Bucknall, Professor H.W. Beckham, H.L. Anderson***

Polyrotaxanes are polymers which have been threaded by macrocyclic rings, which can have a dramatic effect on the properties of the polymer. We are exploring the use of polyrotaxanes as a synthetic route to forming molecular scale devices which can mimic the behaviour of switches, magnetic memory disks and circuit wiring. Polyrotaxanes have potential to be exploited in a number of these molecular scale devices by manipulation of the polymer and macrocycle chemistry. (*Department of Textile and Fiber Engineering, Georgia Institute of Technology, **Dyson Perrins Lab, Oxford University)

Mechanisms of Glass Transition of Polymer Thin Films

T. Kanaya, I. Tsukushi**, Dr. D.G. Bucknall*

Polymer thin films show some interesting but unusual features. One of them is the glass transition temperature, Tg, of these thin films is very different from that of the bulk. We are studying the behaviour of polymer thin films to understand this behaviour within the context of a new glass transition mechanism recently proposed. (*Institute of Chemical Research, Kyoto University; **CIT, Chiba, Japan)

Unstable polymer-polymer interfaces

Dr. D.G. Bucknall, Dr. M. Sferrazza, S. Hutchinson*

The width of an interface between immiscible polymers is determined by the Flory-Huggins parameter. However, when measured using techniques such as neutron reflectivity the interfacial width measured is larger than expected due to thermally excited capillary waves. The amplitude of these capillary waves and therefore the magnitude of the contribution to the measured interfacial width is logarithmically dependent on the film thickness. We are studying the behave of thin films where the film thickness is of order of the chain dimensions (geometrically confined), and therefore similar to the capillary wave amplitude. This can lead to dewetting occurring. As expected this can be altered by altering the interaction between the polymers and the substrate, surprisingly though the unstable films become metastable when mechanically confined. This Part II project aims to understand the mechanism of dewetting and the role played by capillary waves in mechanically confined thin films. (*Department of Physics, Surrey University)

Microstructure of polymeric materials

Dr. D. Vesely

The structures of amorphous and crystalline polymers are studied by light and electron microscopy. New techniques, which overcome and/or utilize the electron beam damage are developed. These techniques, which include microdiffraction, STEM dark field imaging, mass loss measurements, selective staining and chemical analysis are used to obtain more information on the molecular arrangement in amorphous and crystalline polymers. The aim of this work is to understand the effect of micro-structure on the mechanical properties of polymer systems and composites.

IV - PACKAGING MATERIALS

Deformation and fracture of polymer-glass nanocomposites

Dr. O. Kolosov, Dr. Y. Tsukahara, Professor G.A.D. Briggs, Dr. H.E. Assender*

Fracture properties of polymer-glass nano-composites on submicrometre level are studied using surface brillouin spectroscopy and ultrasonic force microscopy. (*Funded by and in collaboration with The Toppan Printing Company)

Deposition of novel glassy barrier layers for packaging applications

G. Erlat, Dr. C.R.M. Grovenor, Professor R. Howson and Dr. B.M. Henry

Reactive, dual target sputtering is being used to deposit Al(X) oxynitride films of carefully controlled composition in order to investigate the feasibility of improving barrier layer properties by the formation of highly dense oxide films. The permeation of water vapour and oxygen through these barrier films is being measured and correlated with microstructural observations. Of particular interest is to develop an understanding of the permeation mechanism of water vapour through these composite samples. (Funded by and in collaboration with The Toppan Printing Company)

Development of a prototype gas transmission microscope

Dr. H. Norenberg, Professor G.D.W. Smith, Professor G.A.D. Briggs

A gas transmission microscope is being developed to study gas permeation through thin barrier films with high spatial resolution (at the micron level). This instrument will be used to correlate the microstructure of the barrier layers directly with the local transmissivity. (Funded by and in collaboration with The Toppan Printing Company)

Polymer surfaces

Dr. V. Bliznyuk, Dr. H.E. Assender, Dr. O.V. Kolosov, Professor G.A.D. Briggs

The topography, adhesion and mechanical properties of polymer surfaces will be examined by combination of number of techniques to a series of model systems. Techniques will include SPM, SBS and XPS. The surface will be considered as a precursor to an interface, and various surface treatments and deposition routes will be examined. (Funded by The Toppan Printing Company)

Prediction and measurement of crystallinity at a polymer surface

K. Kirov, Dr. H.E. Assender

An algorithm has been developed for the quantitative comparison of ATR-FTIR data with transmission FTIR data, to enable the quantitative measurement of the surface properties of polymers. The crystallinity and molecular orientation towards the surface of a polymer is measured after a range of thermal treatments and mechanical deformation of the material. (Funded by The Toppan Printing Company)

Characterisation of glassy barrier films on PET substrates

Dr. B.M. Henry, G. Erlat, Dr. C.R.M. Grovenor, Dr G. Grime

The microstructure of PET/glass films is being studied in order to correlate the gas transmission properties with defects produced during fabrication and handling. High resolution TEM and SEM, Atomic Force Microscopy and microanalysis techniques such as SIMS and the scanning proton microprobe are being used to study the structure and composition of the glass layer. Observations of defects such as local cracking, porosity and impurity contamination are proving valuable in explaining the barrier layer properties of these composite packaging materials. (Funded by and in collaboration with The Toppan Printing Company)

Design and characterization of layered nanocomposite materials

*Professor G.A.D. Briggs, Professor G.D.W. Smith, Professor A.P. Sutton, Dr. C.R.M. Grovenor, Dr. O.V. Kolosov, Dr. H.E. Assender, Dr. D. Vesely, Dr. Y. Tsukahara**

Layered nanocomposite materials are used to provide enhanced gas barrier properties for food packaging and other applications. We study the structure of such materials and the mechanisms of gas permeation, with a view to understanding their properties and designing materials with enhanced performance. Individual projects include characterization by electron microscopy and secondary ion mass spectroscopy, imaging and measuring nanoscale mechanical properties and local gas transmission, polymer surfaces and interlayers, and theoretical modelling of oxide film formation. (* Toppan Printing Company Ltd) (Funded by The Toppan Printing Company)

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This figure shows a 3-D reconstruction of an atom probe analysis through a magnetic multilayer stack.

Dr. A. Cerezo

C. Electronic Materials and Devices

I - SUPERCONDUCTING MATERIALS

During the last few years very exciting advances have led to the development of new oxide materials which superconduct at temperatures up to 160K. The Department of Materials has been working for the past 10 years on fabricating and characterizing bulk and thin film materials in collaboration with other University Departments and Industry. The aim of this work is to develop reliable processing techniques for materials fabrication, to understand the fundamental relationships between microstructure and properties and to investigate the potential of these materials for commercial exploitation.

Development of practical conductors and devices from high temperature superconducting ceramics

Dr. J.C. Moore, Dr. C.R.M. Grovenor, Dr. M. McCullock, Professor D. Dew-Hughes**

Several fabrication routes are being used to produce from superconducting ceramic materials prototype conductors capable of carrying significant currents at liquid nitrogen temperatures and much larger currents at lower temperatures. Current work is concentrating on the development of spray pyrolysis and dip coating deposition techniques. The mechanisms of growth and the composition and microstructure of the films are being investigated as a function of deposition parameters, and related to the superconducting properties. The ways in which these conductors may be fabricated into useful devices, such as magnets motors and fault current limiters, are being investigated and demonstrator devices produced and tested. Superconductor systems based on Bi, Tl and Hg are all under investigation. (*Department of Engineering Science) (Funded by EPSRC)

Microstructural characterisation of superconducting materials

Dr. J. Moore, T. Gladstone, S. Speller, C.J. Salter, Dr G Grime, Dr. C.R.M. Grovenor

Superconducting ceramic samples fabricated in bulk and thin film form are being characterised by X-ray diffraction and electron microscopic techniques. Of particular interest is the determination of the phase distribution and alignment, grain boundary structure and chemistry, and impurity phase chemistry in materials prepared both within the University and by a number of collaborators, and the correlation of these features with critical current measurements. High resolution and analytical TEM, XRD texture analysis, electron microprobe proton microprobe and orientation imaging microscopy techniques are being used. (Funded by EPSRC)

Fabrication of thin films of superconducting ceramics

S. Speller, Dr. A. Jenkins, Dr. C. Stevens*, Dr. C.R.M. Grovenor, Professor D. Edwards*, Professor D. Dew-Hughes**

Sputtering and post annealing processes are being used to deposit thin films up to 3” in diameter of Tl-based superconducting ceramics and buffer layers. The mechanisms of growth and the composition and microstructure of the films are being investigated as a function of deposition parameters, and related to the superconducting and microwave properties. Vicinal substrates are also being used to achieve off axis growth for specific device designs. (*Department of Engineering Science) (Funded by EPSRC and in collaboration with Department of Physics, Kings College London, Department of Engineering Science, Oxford, Department of Metallurgy, University of Cambridge)

Microwave device fabrication from superconducting thin films

Dr. A.P. Jenkins, Dr. C. Stevens*, S. Speller, Professor D. Dew-Hughes*, Dr. C.R.M. Grovenor, Professor D. Edwards**

Prototype passive microwave components (filters, resonators, mixers and correlators) are being fabricated in 2 inch diameter TlBaCaCuO thin films on a variety of substrates, and their performance compared with superconducting properties to optimise preparation processes. World class surface resistance values are routinely achieved, and a range of novel device types are being studied particularly for applications in digital TV systems. More fundamental properties of thin films containing an array of defects are also being investigated. (*Department of Engineering Science) (Funded by EPSRC and in collaboration with WSIL Ltd, BBC, Hymatic Ltd., Birmingham University, Portsmouth University, Kings College London and Department of Engineering Science)

Properties of metallic substrates for superconducting tapes

R. Whiteley, T. Gladstone, Dr. J. Moore, Dr. C.R.M. Grovenor, Dr J.M. Sykes

The thermal/mechanical properties of nickel, silver and silver alloy substrates are being investigated to identify the most promising material for use in Tl-1223 superconducting tapes. High quality cube textured Ni and <110> textured Ag substrates are being produced, and oxide buffer layers growth by a novel application of electrodeposition and by sputtering. The crystallographic relationship between the metal substrate and the superconducting phase is a central aspect of this work, and is analysed by texture X-ray diffractometry. (Funded by EPSRC and in collaboration with Advent Research Materials Ltd.)

II - SEMICONDUCTOR MATERIALS

Secondary electron mapping of doped regions in semiconductors

Dr. M.R. Castell, Dr. A.J. Wilkinson, Dr. P.R. Wilshaw

The secondary electron (SE) signal in an SEM is used to produce 2-dimensional maps of doped regions in silicon and III-V semiconductors. SE images of cross-sections of doped heterostructures and laser devices reveal the type and extent of doping. Quantitative information about the observed contrast has been obtained experimentally. A model has been proposed and is being developed to account for the effect.

Semiconductor quantum dots

Dr. G.R. Booker, Dr. P. Klipstein, Professor R.J. Nicholas*, Dr. P. Möck***

InSb quantum dots embedded in either InAs or GasSb are grown by MOVPE and assessed by TEM, atomic force microscopy, photoluminescence, Raman spectroscopy and electrical measurements. Conditions are optimised to give high luminescent emission in the near infra-red and mechanism are determined.(*Clarendon Laboratory, Oxford; **Physics Department, University of Illanois at Chicago) (Funded by EPSRC)

Dopant profiling in silicon by chemical etching, TEM and AFM

K.D. Yoo, Dr. C.D. Marsh, Dr. G.R. Booker, Dr. C.R.M. Grovenor

One dimension and two dimension dopant profiles are being determined in implanted and annealed bulk silicon wafers and MOS transistors by using selectrive chemical etching in combination with AFM and TEM techniques. The method is being developed for application to shallow p-n junction devices.

Polysilicon emitter bipolar transistors

Dr. C.D. Marsh, Dr. G.R. Booker, Dr. C.R.M. Grovenor, Dr. G. Nash, Dr. J.F.W. Schiz*, Professor P. Ashburn*, Dr. P. Osborne**, Professor D.J. Roulston****

Polysilicon layers are deposited on silicon wafers, implanted with arsenic and annealed. The effects of incorporated fluorine on interfacial oxide break-up and polysilicon layer regrowth are determined using TEM, SIMS and electrical measurements. Application to high performance polysilicon emitter bipolar transformers. (*Electronics Department, Southampton University; **Mitel Inc., Swindon; ***Electrical Engineering Department, Waterloo University, Canada)

Investigation of strain distributions in semiconductors

Dr. A.J. Wilkinson

A techniques for probing local elastic strain fields using electron back scatter diffraction patterns has been developed. It is being used in conjunction with electron channelling contrast imaging to characterise local strain and defect distributions in semiconductor materials and devices. (Funded by The Royal Society)

Scanning Infrared Microscopy - development and applications

Dr. G.R. Booker, Dr. P.R. Wilshaw, Dr. Z.J. Laczik, Dr. P. Torok**

A high performance polarising scanning infrared microscope operating in transmission and reflection is being used to investigate defects in semiconductor specimens, e.g. dislocations and precipitate particles (*Department of Engineering Science)

Quantum wires and dots

Dr. J.L. Hutchison, S. Gledhill, N. Allsop, Professor P.J. Dobson, R.N. Taylor, Dr. O. Salata, Dr. G. Wakefield***

We are assessing methods of making semiconducting dots and wires with dimensions less than 10 nm. This project is a coordinated optical, electronic and structural assessment of these new materials. (*Department of Engineering Science; **Nanox Ltd.)

In-situ doped polysilicon layers

Dr. C.D. Marsh, Dr. G.R. Booker, A.I. Abdul-Rahim, Professor P. Ashburn**

TEM structural studies of in-situ doped polysilicon layers deposited on silicon wafers using a UHV cluster tool system are being performed and the results correlated with the deposition conditions and electrical measurements. (*Southampton University)

Improved Si substrates for microwave applications

Dr. K. Mallik, Dr. S. Senkader, Dr. R. Falster, Dr. P.R. Wilshaw*

This project is to invesitgate materials issues that affect the speed of integraed circuits processed on silicon or solicon based structures and to develop strategies for producing improved materials. Novel methods are being investigated and microwave test structures will be fabri cated. (*MEMC, Italy)

Microstructure of ion beam irradiated silicon for high power semiconductors.

Dr. C.D. Marsh, Dr. C. Johnston, Dr. G.R. Booker, Professor J.M. Titchmarsh*

TEM structural studies of proton and He irradiated silicon are being performed. Proton and He irradiation are being investigated to control carrier lifetimes and the structural results are being correlated with electrical measurements. This work has application to silicon power devices. (*AEA Technology)

Impurities and dislocations in Si wafers

Dr. S. Senkader, A. Giannattassio, Dr. R. Falster, Dr. P.R. Wilshaw*

The interaction between impurities such as O, N and H and defects such as dislocations and precipitates is being studied. In particular the diffusivity and effect of H on diffusivity in the range 350-700°C is being investigated together with the locking of dislocations by impurities and the mechanism by which precipitates weaken wafers. One of the aims of the project is to understand impurity-defect interactions so that stronger Si wafers may be manufactured. (*MEMC, Italy)

Physical embodiment of qubits

M. Austwick, Professor G.A.D. Briggs

We shall investigate the physics of candidate systems for embodying qubits in a solid-state quantum logic gate.

III - MAGNETIC MATERIALS

Growth and characterisation of magnetic multilayer systems for magnetic and magneto-optic recording

Dr. A.K. Petford-Long, Dr. J.L. Hutchison, K. Kang

The structure and composition profile of thin multilayer films with applications as magneto-optic recording are being studied on the atomic scale using HREM, STEM microanalysis and atom-probe microanalysis, for correlation with the magnetic properties. The magnetic domain structure is being characterised using Lorentz microscopy in the 4000EX TEM. The films are being grown by UHV evaporation or magnetron sputter deposition. (Funded by The Royal Society)

MBE growth of spin-valve structures and exchange-biased layers

Y-S. Choi, Dr. A.K. Petford-Long, Dr. R.C.C. Ward, M.R. Wells**

The MBE system in the Clarendon Laboratory is being used to grow epitaxial spin-valve structures and exchange-bias films, so that the exchange-biasing mechanism (vital to the operation of modern hard-disk read-heads) can be studied in the absence of features such as grain boundaries. The magnetisation reversal of the films is being characterised using Lorentz electron microscopy and their microstructure is being analysed using HREM and composition mapping. (*Clarendon Laboratory, Oxford)

Microstructure and magnetic structure of spin-valves and exchange-couples

Dr. A.K. Petford-Long, Dr Y Wang, Dr. H. Laidler, Professor K. O'Grady**

The microstructure and magnetisation reversal mechanisms of spin-valve devices and exchange-couples with applications in information storage technology are being studied at high spatial resolution using electron microscopy, for correlation with their giant magnetoresistive properties. (*Univ. of York) (Funded by EPSRC and Seagate Technology)

Studies of patterned magnetic thin films

Dr. R. Langford, N. Owen, Dr. A.K. Petford-Long

Thin magnetic films grown by sputter deposition and by molecular beam epitaxy are being patterned to form arrays of magnetic antidots. Their magnetic domain structure and magnetisation processes are being studied by Lorentz microscopy for correlation with microstructure. The films have applications as high density storage media (Funded by EPSRC)

Effect of pinning layer material in spin-valve materials

A.R. de Morais, Dr. A.K. Petford-Long, Dr. A. Cerezo

The effect of different pinning layer materials on the magnetic and transport properties of NiFe-based spin-valve structures is being studied. The films are being grown by sputter-deposition and electron microscopy techniques are being used to assess their properties. (In collaboration with Nordiko Ltd.)

Surface engineering of layered films for magnetic sensor applications

*M.W. Ormston, Dr. A.K. Petford-Long, Dr. D.G. Teer**

Magnetic layered films are being grown by magnetron sputter deposition and their interfaces are being modified in a controlled way by ion-bombardment to assess the usefulness of this technique for improving the magnetic and transport properties of the films. (*Teer Coatings Ltd) (Funded by EPSRC and Teer Coatings Ltd.)

Spin-tunnel junctions based on magnetic layered films

Dr. A.K. Petford-Long, Dr. D. Ozkaya, Dr. P. Shang, Dr. T.C. Anthony, Dr. J.A. Brug**

Spin-tunnel junction devices are magnetic layered systems which exhibit giant magnetoresistance. The aim is to develop these systems for applications as magnetic field sensors and/or magnetoresistive memory elements. (*Hewlett-Packard Labs.) (Funded by Hewlett-Packard Labs. and EPSRC via a collaboration with the Univ. of Cambridge and Univ. of Plymouth)

IV - DISPLAY MATERIALS

Non-lithographic definition of sub-micron field emitter structures

J.Kang, Dr. Y-G. Li, Dr. P.R. Wilshaw

At present most triode type vacuum microelectronic devices are fabricated using sub-micron resolution lithography. Such processing is costly and difficult to achieve over the large areas required for field emitter displays. This project involves the use of specially fabricated materials which contain sub-micron features suitable for field emission without the need for lithographic processing. (Funded by EPSRC)

Large band gap field emission displays

D. Stowe, Dr. J. Kang, Dr. P.R. Wilshaw

Field emission from large band gap semiconductors such as diamon and glas with low or negative electron affinity is being studied. An experimental technique has been developed whereby electrons are injected into the semiconductor conduction band using an SEM. In this way the problems associated with the back contact are overcome allowing investigation of emission from the front surface.

Development of electroluminescent display based on organolanthanides

S. Capecchi, Dr. A. Mosley**, Dr. V. Christou***, Dr. O.V. Salata*

Organolanthanides are promising electroluminescent materials that can be used in flat panel displays. Their fundamental advantages such as pure green and red emission colours as well as possible utilisation of the triplet excitations in light generation make these materials favourite candidates for the future displays. Advanced light-emitting and charge-transporting materials are under constant development at ICL, University of Oxford. Organic light-emitting devices based on those novel materials are optimised for the highest possible performance and then transferred to the CRL Ltd to be used in the manufacturing of display panels. (*Opsys Limited; **CRL Ltd.; ***ICL, Department of Chemistry) (Funded by Opsys Ltd.)

Carbon nanotube field emission displays

Dr. J. Kang, Dr. P.R. Wilshaw

Field emitting structures based on carbon nanotubes embedded in an alumina matrix are being investigted. These show potential for large area dislay devices with a gate structure simply integrated into the cathode emitting region.

Material issues in degradation of OLEDs

Dr. D.-G. Moon, Dr. V. Christou**, Dr. O.V. Salata*

Although reports on the long operational lifetime of the OLEDs are not uncommon, most of them are referred to the TPD/Alq3 based devices. It is expected that organolanthanide based devices should posses an improved stability. However, no experimental information is yet available to support this claim. The objective of this project is to establish the influence of environmental factors like oxygen and moisture as well as charge transport and heat dissipation on the degradation of organolanthanide based OLEDs. Established molecular structure-stability dependencies will allow us to design more robust molecules.(*Department of Engineering Science; **ICL, Department of Chemistry)(Funded by Opsys Ltd.)

Lanthanide Dendrimers as multifunctional materials for OLEDs

Dr. J. Pillow, Dr. M. Halim*, Dr. P. Burn**, Dr. I.D.W. Samuel***, Dr. V. Christou****, Dr. O.V. Salata*

Dendrimers or starburst molecules have been applied recently to both light emitting and charge transporting layers in OLEDs. It is possible to design dendrimeric molecules that can direct both charge and energy to the light-emitting moiety. If this light-emitting moiety is a lanthanide ion then a pure emission is expected. The objective of this project is to find out the “design rules” of the efficient electroluminescent molecules possessing bipolar transport abilities. We are trying to establish relations between molecular structure and behaviour of dendrimers in electroluminescent devices.(*Opsys Limited; **DPL, Department of Chemistry; ***Department of Physics, Durham University; ****ICL, Department of Chemistry) (Funded by Opsys Ltd.)

Transport properties and electronic structure of organolanthanides

Dr. G. Maliaris, Dr. I.D.W. Samuel**, Dr. V. Christou***, Dr. O.V. Salata*

Knowledge of type and mobility of charge carriers in organic materials is important for design of effective OLED. It is also important to know the values of energy barriers to the charge injection from electrodes as well as HOMO-LUMO energy offsets at the organic-organic interfaces. Modelling of I-V-B characteristics, time of flight measurements, XPS and electrochemical methods are applied in order to get complimentary values of mobilities and energy levels. (*Department of Materials Science and Engineering, Cornell University; **Department of Physics, Durham University; ***ICL, Department of Chemistry) (Funded by Opsys Ltd. and Cornell University)

Studies of field emitter/phosphor interactions for display applications

Dr. M. Huang, Dr. M. Ushirozawa, Dr. A. Cerezo, Professor P.J. Dobson, Professor G.D.W. Smith*

Field emission microscopy and atom probe microanalysis techniques are being combined to study the degradation mechanisms in field emitter displays. (*NHK, Japan) (In collaboration with NHK, Japan and Nanox Ltd., Oxford)

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This picture shows the new squeeze-casting and rheocasting equipment currently being installed in the processing area of Begbroke Business and Science Park.

Dr. Keyna O'Reilly

D. Processing

Spray formed Ni alloys

M. Hedges, T. Rayment, Dr. I.C. Stone, Professor B. Cantor, Dr. P.S. Grant

The mechanism of refined, equiaxed grain evolution in spray forming, and the role of insoluble nitride, carbides and microporosity, are being investigated by a combination of manufacture of Ni preforms by spray forming under different processing conditions and microstructural characterisation by EPMA and phase extraction/XRD. The development of Ni superalloys which exploit the unusual solidification conditions in spray forming is also being explored in order to enhance high temperature strength and creep properties (Funded by EPSRC, Rolls-Royce, Allvac and Osprey Metals Ltd.)

Arc sprayed self lubricating coatings

H. Llewellyn and Dr. P.S. Grant

Various grades of steel coatings containing a dispersion of graphite and boron nitride particulate are being manufactured by arc spraying. Coating microstructure is being characterised by microscopy and coatings are being wear tested in a special rig to assess their application in the off-shore oil industry. (Funded by EPSRC and Spraylube Coatings Ltd)

Spray formed automotive tooling

S. Hoile, P. Jones, Dr. Z. Djuric, Dr. S. Duncan*, J. Betts**, Dr. P.S. Grant*

Electric arc spraying of liquid steel droplets onto shaped substrates is being investigated for the rapid manufacture of dies for stamping/pressing tools for a wide range of applications. Lead times are several times faster than for conventionally machined dies and tooling. Research focuses on closed loop feedback control of residual stresses, simulation of shape evolution and microstructural characterisation. (*Department of Engineering Science, **Ford Motor Co) (Funded by EPSRC, Sprayforming Developments Ltd, Sulzer Metco and Ford Motor Co.)

Low pressure squeeze cast alloys and composites

P. Simmons, Dr. P. Schumacher, Professor B. Cantor

A series of potential alloys for piston applications are being squeeze cast at low pressures to investigate structure and properties, and to develop lower cost squeeze casting methods. (Funded by EPSRC and Federal - Mogul)

Laser imaging and other diagnostics in spray processes

T. Rayment, Dr. P.S. Grant

A particle image velocimetry (PIV) system powered by twin Nd-YG lasers, an Ar ion laser based phase Doppler anemometry (PDA) system and a state-of-the-art far infrared 50Hz infrared thermal imaging system are being used in conjunction with ultra fast video and photography to investigate the velocity and trajectory histories of molten metal droplets in a variety of spray processes. As well as novel developments in imaging techniques, the process measurements are related to operating variables and resulting sprayed microstructure. (Funded by EPSRC)

Rapid steady state solidification of 6xxx series Al alloys

G. Sha, Dr. K.A.Q. O'Reilly, Professor B. Cantor

Phase selection in model and commercial 6xxx series Al alloys is being investigated using electron beam surface melting in order to simulate the high steady state growth velocities experienced in modern continuous casting techniques. (Funded by Alcan International)

Metallurgical soldering and bonding

A.D. Brand, Dr. K.A.Q. O'Reilly

Thermal and mechanical properties of metallurgical soldering and bonding materials are being investigated as a function of microstructure. The effect of initial microstructure, the thermal stability of the microstructure and its effect on the mechanical properties are being studied in a range of lead containing and lead-free materials. (Funded by EPSRC and Alpha Fry)

Freeform fabrication of ceramics and metals using ink-jet printing

N. Reis, K. Seerden, Dr. P.S. Grant, Dr. B. Derby*, Dr. J. Evans***

It is possible to construct rapid prototpes of complex engineering structures slice by slice using 3-dimensional printing technology. We are developing ceramic suspensions in low melting point waxes for drop-on-demand ink-jet printing of 3D shapes. (*University of Manchester; **Queen Mary and Westfield College) (Funded by EPSRC)

Microstructural studies of Pb-Sn solder/PCB interactions

C. Langham, Dr. C.R.M. Grovenor, Dr. S. Gupta**

The stability of copper PCB tracks coated with commercial Ni, HASL, OSP and Pd protective coatings in contact with molten Sn-Pb solder is being studied in order to compare the performance of these barrier layers in wave soldering operations. The replacement of the standard solder by lead-free formulations is also being studied. (*Motorola ECID)

The downstream processing of twin roll cast aluminium alloys

*Dr. M. Yun, Dr. C. Gras, Professor J.D. Hunt, Dr. K.M. Gatenby**

The solidification conditions experienced during twin roll casting result are very different from those in conventional direct chill (D.C.) cast ingots. This project is concerned with how these differences affect the final properties of twin roll cast aluminium sheet after downstream processing. (*Alcan International) (Funded by EPSRC and Alcan International)

Coupled eutectic growth in twin roll cast aluminium alloys

*T. Cockfield, Dr. M. Yun, Professor J.D. Hunt, Dr. K.M. Gatenby**

A range of aluminium alloys of eutectic composition are to be twin roll cast to investigate the extent of the coupled eutectic growth region. Alloys of this type are of interest because the large amount of eutectic can possibly improve the recrystallisation behaviour and therefore the mechanical properties of the twin cast roll sheet. (*Alcan International) (Funded by EPSRC and Alcan International)

Twin roll casting of stainless steel and other ferrous alloys

Dr. M. Yun, Professor J.D. Hunt

The twin roll casting of ferrous alloys is of great commercial interest, to date the majority of research has concentrated on casting vertically downwards. This technique has a number of disadvantages such as control of metal head and metal constraint. The project intends to cast horizontally which alleviates these problems. (Funded by EPSRC and Kvaener Metals)

The grain refinement of twin roll cast aluminium alloys

Dr. M. Yun, Professor J.D. Hunt, D. Bristow, Dr. R. Cook**

Grain refiners are traditionally added to aid casting and improve the castability of aluminium alloys. This project is investigating the relative efficiency of different grain refiner additions i.e. TiB2, TiC, during the twin roll casting of commercial aluminium alloys. (*London and Scandinavian Metallurgical Co. Ltd.) (Funded by EPSRC and London and Scandinavian Metallurgical Co. Ltd.)

Continuous casting of copper base alloys

*Dr. M. Yun, Professor J.D. Hunt, R. Bloomer**

The purpose ot this project is to investigate the twin roll casting of copper based alloys and to compare the sheet with material produced by other continuous casting routes. (*Mason Precision Strip)(Funded by EPSRC and Mason Precision Strip)

Twin roll casting of superplastic SPZ Zn-Al

*Dr. M. Yun, Professor J.D. Hunt, T. Rance**

This material is traditionally ingot cast prior to a complex thermomechanical processing route which produces the very fine grain size necessary for superplasticity. Twin roll cast Zn-22wt%Al has been produced which exhibits superplasticity in the as-cast condition. Trials are underway to maximise this effect by investigating both the casting conditions and possible heat treatments. (*Custom Metalforms Ltd.)(Funded EPSRC and Custom Metalforms Ltd.)

Spray formed Al alloys

Dr. I.C. Stone, Dr. I.G. Palmer, Dr. P.S. Grant

A 80kg Al spray forming plant is currently being installed in a dedicated laboratory. Research focuses on the manufacture of Al-Li, Al-Zn-Mg and Al-Si alloys by spray forming and their downstream processing. At each process stage, the microstructure is investigated by SEM/TEM/EPMA and XRD, and microstructure related to final properties. (Funded by EPSRC and Joint Infrastructure Fund and in collaboration with Universities of Cranfield and Southampton, Imperial College. British Aerospace Military, GKN Westland Helicopters Ltd and DERA)

Second generation Ti MMCs

Dr. K.H. Baik, Dr H. Peng, Dr. P.S. Grant, Dr. F Dunne, Professor B. Cantor*

New Ti alloy fibre reinforced MMC formulations are being manufactured by plasma spraying, sputter coating and hot uniaxial and isostatic pressing, and being assessed by detailed microstructural examination and residual stress chcracterisation (Funded by EPSRC, Rolls-Royce plc and DERA).

Manufacture of Ti MMCs

Dr. T.W. Kim, Dr. F.P. Dunne, Dr. Z.X. Guo**, Professor B. Cantor*

The manufacture of Ti MMCs is being controlled by modelling and experimental validation to understand the development of porosity, interface reaction, fibre fracture and other defects during hot consolidation.(*Department of Engineering Science; **Queen Mary Westfield) (Funded by EPSRC, Rolls-Royce and DERA)

Improved processing of wrought Al alloys by modelling and control of grain refinement

Dr. P. Cizek, Dr. K.A.Q. O'Reilly, Dr. P. Schumacher, Dr. A.L. Greer, Dr. R. Hamerton**, D. Bristow****

The performance of Al-Ti-C grain refiners in wrought Al alooys is being characterised to understand the nucleation mechanism and to develop quantitative modelling of the refiner action capable of application to a range of alloys and processing conditions. (*University of Cambridge; **Alcan International; ***London Scandinavian Metallurgical Company)(Funded by EPSRC, LSM and Alcan)

Melt spun amorphous and nanocrystalline Al and Fe alloys

Dr. Y. Zhang, Dr. P.J. Warren, Dr. A. Cerezo, Professor B. Cantor

Amorphous and nanocrystalline Al and Fe alloys are being fabricated by melt spinning to form crystalline/quasicrystalline/amorphous composites for detailed TEM and FIM/AP investigations of microstructure. (Funded by EPSRC and in collaboration with Carpenter Technology and DERA Farnborough)

Residual stresses in Ti MMCs

Dr. S. Gungor, Professor C. Riuz*, Dr. J. Durodola**, Professor P. Bowen***, Professor B. Cantor*

Residual stresses in Ti MMCs are being investigated by modelling and measurements using novel moiré interferometry and slitting methods. The resulting creep, fatigue and damage mechanics are being measured and modelled to allow lifting of components.(*Department of Engineering; **Oxford Brookes; ***Birmingham University) (Funded by EPSRC, Rolls-Royce and DERA)

Grain refinement tolerance to Fe

*Dr. E.D. Manson-Whitton, Dr. K.A.Q. O'Reilly, M.R. Jarrett**

Control of intermetallic phase selection, together with modified quenching procedures after extrusion, is being investigated to allow increased productivity of extrusions while maintaining, or improving, surface quality of the product and increasing tolerance to Fe impurity.(*Luxfer Group)(Funded by Teaching Company Scheme)

Grain refinement in Mg alloys

*Dr. B. Davies, Dr. K.A.Q. O'Reilly, Dr. P. Schumacher, J.F. King**

Metallic glasses, sedimentation and the entrained droplet technique are being used to investigate the mechanisms of grain refinement in model Mg alloys by Zr-containing particles.(*Luxfer Group) (Funded by Luxfer Group Ltd.)

Multicomponent alloys

K-B. Kim, Dr. P.J. Warren, Professor B. Cantor

Soldification of multicomponent alloys often results in complex microstructures consisting of a mixture of stable and metastable phases. These microstructures or individual constituent phases can exhibit a range of unusual and desireable properties such as magnetic, elastic or structural. This project is working near the centre of several different five and six component phase diagrams, outside the regime currently accessible by thermodynamic modelling.

Novel manufacturing routes for Al products

C.D.J. Manson-Whitton, Dr. P. Schumacher, Dr. K.A.Q. O'Reilly, Dr M. Clinch, Dr. W. Hepples*, Dr. H. Holroyd**

Novel manufacturing technologies are being investigated for the manufacture of Al alloy products.(*Luxfer Group) (Funded by The Royal Commission for the Exhibition of 1851 and Luxfer Group Ltd.)

Nanocrystalline and quasicrystalline nanocomposite Al alloys

Dr. P.J. Warren, Dr.Y. Zhang, Professor B. Cantor, Dr. A. Cerezo

Al-base nanocomposite materials containing high volume fractions of quasicrystalline dispersoids are being produced by rapid solidification techniques. The nanoscale microstructures formed during quenching are strongly dependant upon the thermal history of the melt due to cluster formation in the liquid. (Funded by EPSRC)

Carbon fibre reinforced magnesium

*M. Russell-Stevens, Dr. K.A.Q. O'Reilly, Dr. P. Schumacher, P. Schultz**

Microstructural investigation of long fibre carbon fibre reinforced magnesium alloys manufactured by squeeze casting, as a function of casting parametrs. (*Leichtmetall Kompetenzzentrum Ranshofen) (Funded by EPSRC)

Direct chill casting of Al and Mg alloys

*Dr. E.D. Manson-Whitton, Dr. B. Davis, Dr. K.A.Q. O'Reilly, M.R. Jarrett**

A one tonne direct chill (DC) caster has been installed in the department and is being used to investigate the effects of alloy composition, processing parameters and grain refinement practice on the microstructures and properties of Al and Mg alloys.(*Luxfer Group)

Squeeze casting and semi-solid processing of Al and Mg alloys

*C.D.J. Manson-Whitton, Dr. B. Davis, Dr. I.C. Stone, Dr. P. Schumacher, Dr. K.A.Q. O'Reilly, Prof. B. Cantor, M.R. Jarrett**

An UBE 350 tonne squeeze casting and semi-solid processing machine is being installed in the department and will be used to investigate the effects of alloy composition, and processing parameters on the microstructures and properties of squeeze cast and semi-solid processed Al and Mg alloys.(*Luxfer Group)

The downstream processing of twin roll cast aluminium alloys

*Dr. M. Yun, Dr. M.E. Keeble, Professor J.D. Hunt, Dr. K.M. Gatenby**

The solidification conditions experienced during twin roll casting result are very different from those in conventional direct chill (D.C.) cast ingots. This project is concerned with how these differences affect the final properties of twin roll cast aluminium sheet after downstream processing. (*Alcan International) (Funded by EPSRC and Alcan International)

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STEM composition maps showing a composite particle formed by a quasi-peritectic solidification reaction in 6xxx series Al alloy.

Gang Sha and Dr. Keyna O'Reilly

E. Phase Transformations, Surfaces and Interfaces

I - PHASE TRANSFORMATIONS

Phase transformations of copper precipitates in ferritic matrices

Dr. M.L. Jenkins, Professor G.D.W. Smith, Professor A.P. Sutton, Professor J.M. Titchmarsh, X. Wang

High-resolution electron microscopy (HREM) has shown that small coherent bcc copper precipitates in thermally-aged or irradiated iron alloys and some steels transform initially to a 9R structure by a martensitic mechanism, and then subsequently transform further to an fcc structure, probably by a diffusive mechanism. These transformation mechanisms are being elucidated by further HREM and theoretical studies.

Quantitative analysis of crystallisation processes in amorphous alloy films

G. Ruitenberg, Dr. A.K. Petford-Long, Professor P.J. Dobson, R.C. Doole, Dr. C.N. Afonso***

In-situ TEM crystallisation experiments are being carried out on amorphous Sb-based alloy films with applications as ultra-fast phase-change optical storage media. The results are allowing quantitative data about the crystallisation kinetics to be obtained. The effects of the relaxation state of the amorphous structure on crystallisation kinetics are also being studied in both alloy and oxide films. (*Department of Engineering Science; **Institute of Optics, CSIC, Madrid, Spain) (Funded by EPSRC and Royal Society)

Model alloys (ferritic steels), precipitation as a function of composition and ageing treatments using HREM techniques

Professor J.M. Titchmarsh, Dr. M.L. Jenkins, S. Lozano-Perez

The embrittlement of pressure vessel steels is being investigated through a systematic investigation of the influence of alloying element content and heat treatment. High resolution imaging and microanalytical electron microscopy methods are use to identify precipitation and segregation in a series of model alloys. These results will be correlated with the variations in mechanical properties and related to models of toughness changes in neutron irradiated pressure vessel steels. (Funded by EPSRC) (In collaboration with AEAT and INSS)

Structure and crystallisation kinetics of optical nanocomposites

J-P. Barnes, Dr. G.T. Fei, Dr. A.K. Petford-Long, Dr. R. Serna, R.C. Doole,*

The effect of metal particle size and morphology on the ultrafast non-linear optical properties of nanocomposite materials is being studied. In-situ TEM annealing is being used to understand the kinetics of the crystallisation process. (*Institute of Optics, CSIC, Madrid, Spain)(Funded by EPSRC, British Council and Chinese Government)

Cyclic phase transformations

I. de Ardo, Dr. K.A.Q. O'Reilly, Professor B. Cantor

The effect of temperature cycling is being investigated on the melting, solidification and solid state phase transformations in pure metals, alloys and amorphous materials using a novel calorimetric technique and TEM to determine kinetics of phase transformations.

Heterogeneous nucleation in Al-Si alloys

B. McKay, Dr. P. Schumacher, Dr. K.A.Q. O'Reilly

Two novel techniques employing sedimentation and metallic glasses are being used to investigate the nucleation mechanisms operating when various grain refiner additions are made to Al-Si alloys. Refiner potency and the effects of alloying elements and processing conditions are also being evaluated. (Funded by EPSRC and in collaboration with London Scandinavian Metallurgical Company)

Modelling microstructure in multicomponent alloys

M. Shin, Professor J.D. Hunt

Diffusion based models will be used to predict microstructure in multicomponent alloys. The work will extend an existing model which treats two components. The model will predict the path of the liquid, microsegregation, the non equilibrium freezing range and will treat three and four phase reactions. The programme will initially use the NPL MTDATA database to provide thermodynamic data.(Supported by Alcan International and in collaboration with NPL)

Experimental studies and modelling of cellular and dendritic growth

A. Chilton, Professor J.D. Hunt

The transition between cellular and dendritic growth is being studied experimentally and models will be developed to predict the transition. the models will be based on an existing axisymmetric cell/dendrite model. (Supported by Alcan International)

Nucleation kinetics of potent heterogeneous site

Dr. P. Schumacher

Heterogeneous nucleation of commercial grain refiner particles (TiB2, TiC) is being studied as additions in amorphous Al alloys. Nucleation substrate surfaces are investigated in TEM and their properties measured in entrained droplet measurements. A kinetic adsorption model is being developed to predict quantitative nucleation rates. (Funded by EPSRC)

The development of a high temperature single pan scanning calorimeter.

Dr. H. Dong, Professor J.D. Hunt

A novel scanning calorimeter is being built to measure enthalpy up to temperatures of 1650 C. The heat flow within the calorimeter will be modelled numerically to improve the accuracy of the measurements. (Funded by EPSRC and in collaboration with NPL)

Modelling columnar and equiaxed growth

D.J. Browne, Professor J.D. Hunt

A three dimentional casting model is being developed to descibe columnar and equiaxed growth.

Calorimetry of twin-roll cast alloys.

E.C. Kurum and Professor J.D. Hunt

A novel sensitive and accurate scanning calorimeter has been developed within the research group. A calorimetric study of commercial twin roll cast alloys will be carried out to determine the phases and the fraction solid formed as a function of temperature.

II - OXIDATION AND CORROSION

The study of thick corrosion layers on archaeological metals using controlled laser ablation in conjunction with an external beam microprobe

M. H. Abraham, Dr. G.W. Grime, M.A. Marsh, Dr. J. P. Northover

The variation with depth of the composition of corrosion layers on buried metal objects can provide the archaeologist with valuable information relating to the burial conditions of the object. In some cases these layers can be very thick (up to 1mm) and so normally, destructive methods such as sectioning are used to characterise the layers. The technique developed here uses a micro-focused high power pulsed Nd:YAG laser to ablate the corrosion layer in a series of controlled steps, while monitoring the composition of the exposed surface using PIXE and RBS in the external beam facility of the Oxford Scanning Proton Microprobe. The region of the surface removed by the laser beam is typically less than 500mm diameter and so, by comparison with other sampling techniques, the effect on the appearance of an object is minimal.

Study of anti-corrosive paints by scanning acoustic microscopy and scanning Kelvin probes

Dr. J.M. Sykes and Prof. G.A.D. Briggs

The process of coating breakdown and adhesion loss is being examined by combined use of scanning acoustic microscopy and scanning Kelvin probe. The influence of pigment type and other coating variables is being determined.

Corrosion protection of metal packaging by organic coatings

M. Doherty, Dr. J.M. Sykes and Dr. H.E. Assender

The influence of barrier and other properties of polymer coatings on corrosion of food cans and other packagin is being examined. Permeation and electrochemical measurements are supplemented by adhesion studies using scannign acoustic microscopy and potential mapping by scanning Kelvin probe.

III – SURFACES AND INTERFACES

Atomic structure and stability of metallic multilayered systems

*Dr. G. Möbus, Dr. B.J. Inkson, Dr. T. Wagner**

Thin films and multilayers of various representative metal-metal (fcc-fcc, bcc-bcc, hcp-fcc) systems are examined by combinative electron microscopy techniques. Atomic structure, interdiffusion, and stability of the interfaces are studied. Systems include W-Nb, Cu-Al, and Cu-Ti. Analytical and high-resolution electron microscopy are applied using the JEM3000F FEGTEM and JEM 4000EX. (*MPI fuer Metallforschung Stuttgart, Germany)

OLED interface engineering using ultra-thin inorganic layers

Dr. O. Renault, Dr. O. Kolosov, Dr. O.V. Salata*

Typical OLED device contains a number of interfaces both organic-organic and organic inorganic. These interfaces quite often control the charge balance and hence device efficiency. Various deposition techniques of ultra-thin inorganic layers are used to create tunnelling layers located at different interfaces. Morphology of the resulting layers is studied using AFM and related methods and is linked both to the deposition conditions and device performance.(*Department of Engineering Science) (Funded by Opsys Ltd.)

IV - SURFACE REACTIONS AND CATALYSIS

Controlled atmosphere analytical electron microscopy

R.C. Doole, Dr. J.L. Hutchison

A 400 kilovolt analytical transmission electron microscope has been extensively modified and equipped with special controlled-atmosphere specimen stage. A number of projects of chemical and materials interest are now being undertaken, e.g. the study of catalysts under their working environments.

In-situ observation of solid-state reactions

Dr. J.L. Hutchison, Dr. M.J. Sayagues de Vega, Dr. F. Krumeich***

The controlled environment TEM is being used in a study of oxidation and reduction reactions of Nb, W and Mo. (*University of Seville; **ETH Zürich) (Funded by British Council and NATO)

One-dimensional crystal growth inside single-wall carbon nanotubes

*Dr. J.L. Hutchison, Dr. J. Sloan, G. Brown, Professor M.L.H. Green**

Crystals of various salts and metals grown within single-wall carbon naotubes are effectively 1-D wires, with a range of interesting physical properties which arise from their unique configurations, We are exploring ways of growing these structures, which are characterisaed by HREM, EDX and EELS. Their physical properties are also under investigation. *(Inorganic Chemistry Laboratory)

Catalytic atom probe

Professor G.D.W. Smith, Dr. A. Cerezo, T.J. Godfrey

A specially adapted atom probe, incorporating a gas reaction cell, is being developed in order to permit the atomic scale study of catalytic reaction processes. (Funded by EPSRC and in collaboration with Johnson Matthey and Omicron Surface Science Ltd.)

Tableau of ring patterns formed by Fourier transforming a series of images of amorphous carbon taken in a field emission gun transmission electron microscope. Each image in the series was obtained with a different angle of the incident illumination, with the central image obtained slightly underfocus. Computer simulations are fitted to the experimental ring patterns to measure the aberrations of the microscope objective lens. Accurate values for these aberrations are required in order to use techniques such as focal series restoration to improve the directly interpretable resolution of images obtained in the microscope to below one Angstrom (0.1 nm).

Dr. Rafal Dunin-Borkowski and Dr. J.L. Hutchison

F. Characterisation

I - SCANNING TUNNELLING AND ATOMIC FORCE MICROSCOPY

Ultrasonic heterodyne force microscopy for studies of dynamics of nanoscale non-mechanical properties

*Dr. O.V. Kolosov, Dr. B. Huey, Professor G.A.D. Briggs, Professor O.B. Wright**

The physical principles, theoretical background and technical realization of the novel principle of mechanical heterodyne force microscopy is developed. This novel Scanned Probe Microscopy approach enables measurements and mapping of time-dependent physical properties of studied object (e.g. electrical, optical, thermal, etc.) with the nanometre scale spatial resolution and sub-nanosecond time sensitivity. (*Dept. of Applied Physics, Hokkaido University, Japan,) (Funded by the Japanese Society for Promotion of Science and British Council)

Nanostructures on the SrTiO3 (001) Surface

Dr. M.R. Castell

Atomically resolved scanning tunneling microscopy of the SrTiO3 (001) surface reveals that certain treatments give rise to two types of self assembled nanostructures. The one dimensional structure type consists of perfectly straight lines that run in <100> directions and have a minimum separation of 2.4 nm. The other structures are dots that on closest packing form 2.4 nm x 1.6 nm arrays. It is proposed that both structure types are formed through nano-crystalline growth of cubic SrO on a TiO2 terminated surface containing ordered TiO2 defects. Further characterization and metal island growth on these surfaces is currently being carried out.

Direct measurement of interatomic potentials

Professor J.B. Pethica, Dr. P. Hoffmann, L. Giberti, Dr. S.P. Jarvis, Dr. H. Tokumoto**

A new AFM allows the direct mechanical measurement of short range interaction potentials between tip and surface. Strong interactions can be accessed without mechanical instability. (*JRCAT, Tsukuba, Japan) (Funded by EPSRC and Paul Instument Fund)

Growth and characterization of quantum silicide and nitride islands

Professor G.A.D. Briggs, Dr. M.R. Castell, Dr. S.D. Kenny, Dr. C. Norenberg, Dr. M. Martin Fernandez, R. Oliver, Professor A.P. Sutton

The growth and properties of silicide and nitride islands are studied, with a view to discovering materials systems that may be useful for quantum structures. Islands are grown in our variable temperature STM, and examined in situ. We study both metallic and semiconducting islands. We investigate factors that affect their shape and size distributions, the phases that are present, and the surface structures and electronic properties. Atomistic and multiscale modelling enables the structure of surfaces and interfaces to be elucidated, and the distribution of island types and sizes to be accounted for.

Ultrasonic Force Microscopy characterization of Focused Ion Beam implantation and damage

Dr. B.D. Huey, Dr. R. Langford

The spatial extent of Gallium ion implantation and/or damage from FIB is measured using UFM. This allows characterization of the FIB system, in particular to identify proper implantation parameters for a given damage threshold. UFM sensitivity and resolution are also investigated using the wide variety of implantation profiles available with the FIB.

STM & AFM of metals on oxides at variable temperature

Professor J.B. Pethica, Dr. C.E.J. Mitchell, Dr. R.G. Egdell*, Professor J.S. Foord***

A new variable temperature STM and XPS system is used to study catalytic oxides and metal islands thereon. Surfaces under investigation include SnO2 , WOn and tungsten bronzes, ZnO. (*Inorganic Chemistry Laboratory; **Physical Chemistry Laboratory) (Funded by EPSRC)

Atomic resolution AFM

Dr. P. Hoffmann, L. Giberti, S. Jeffery, Professor J.B. Pethica

A new AFM designed and built in-house, gives real space surface atomic resolution of forces and force gradients, using sub-Å oscillation amplitudes. It is shown that atomic resolution is correlated with dominance of short range interactions. (Funded by EPSRC and Paul Instrument Fund)

AFM of aqueous bio-systems

C. Ramanujan, Dr. P. Hoffmann, Professor A.B. Watts, Professor J.B. Pethica*

We have recently used a new AFM to resolve the solvation layers in water. The low amplitude AFM technique is now being applied to image the structure near peptides inserted at low density in lipid layers. (*Dept. of Biochemistry)

Investigation of the physics of dynamic tip-surface interaction at ultrasonic time-scale

Dr. B. Huey, Dr. O.V. Kolosov, Prof. G.A.D. Briggs

Dynamic interactions between a tip and a sample surface are studied by performing ultrasonic, waveguide and heterodyne force microscopy experiments in different controlled environments. (Funded by EPSRC)

Forces on single atoms in STM

*Professor J.B. Pethica, Dr. P. Hoffmann, L. Giberti, Dr. J. Nieminen**

STM images change with tip-surface separation as a result of forces present. Experiment and MD modelling are combined to determine forces at individual atoms. Metals and halogen adsorbates are presently studied. (*University of Tampere, Finland)

Room temperature manipulation of surface atoms

*Professor J.B. Pethica, Dr. P. Hoffmann, Dr. R.G. Egdell**

Individual bromine atoms can be placed at specified sites on a copper surface and nanostructures written at room temperature. The mechanisms of atomic manipulation and their possible specificity are under investigation. The study is being extended to variable temperature. (*Inorganic Chemistry Laboratory)

Semiconductor metallization in variable temperature STM/STS

Dr. M.R. Castell, Professor G.A.D. Briggs, Dr. C. Norenberg, Dr. M. Martin Fernandez

Technologically important refractory metals (Ti, Co, etc) will be deposited from UHV evaporators onto semiconductor surfaces in variable temperature STM. The crystallographic characteristics of the contacts will be investigated by STM, RHEED/LEED and AES, and the physical characteristics of the resulting barriers will be measured by STS.

Ferroelectric domain characterization

Dr. B.D. Huey, Dr. O.V. Kolosov, Professor G.A.D. Briggs

Ultrasonic force microscopy, electrostatic force microscopy, and piezoresponse imaging are applied to investigate the piezoelectric characteristics of individual domains in ferroelectric thin films. Using this combination of techniques, domain switching can be directly measured at frequencies ranging from Hz to 100 MHz. (Funded by US-NSF and Marshall Aid Commission)

New force microscopy techniques in ambient

C.Ramanujan, S. Jeffery, Dr. P. Hoffmann, Professor J.B. Pethica

A new, high resolution AFM which functions in liquids has been developed. We have successfully observed normal and lateral stiffness fluctuations due to individual molecular layering. The unusual shear properties of the final liquid molecular layers adjacent to a solid are being studied.

Study of nanoscale material properties using ultrasonic-based scanning force microscopy techniques

Dr. B. Huey, Dr. O.V. Kolosov, Professor G.A.D. Briggs, Dr. H.E. Assender

Ultrasonic, waveguide and heterodyne force microscopy are used to investigate the nanoscale mechanical structure of materials of different origin, including semiconductor quantum dots, polymer composites and metallic compounds.

Experimental development of heterodyne force microscopy

Dr. B. Huey, Dr. O.V. Kolosov, Professor G.A.D. Briggs

Implementation of Heterodyne Force Microscopy (HFM) is in progress. Experimental research seeks to determine the capabilities and limitations of the technique for the study of time-dependent processes on a nanometer scale and to realise the HFM potential for the time sensitivity on the sub-nanosecond time scale. (Funded by EPSRC)

SPM of semiconductor quantum structures

Dr. O. Kolosov, Dr. B. Huey, Dr. N. Mason, Dr. M.R. Castell, Dr. C.D. Marsh, Dr. R.S. Williams**, Dr. T.I. Kamins**, Professor G.A.D. Briggs*

The growth and nanoscale mechanical properties of groups iii-v and group iv semiconductor buffer layers, superlattices and quantum dots are studied by various SPM techniques. (*Clarendon Laboratory, Department of Physics; **Hewlett-Packard Laboratories) (Funded by EPSRC)

II - FIELD-ION MICROSCOPY AND ATOM PROBE MICROANALYSIS

Scanning atom probe

Dr. M. Huang, O. Dimond, Dr. S. Jordan**, Dr. A. Cerezo, T.J. Godfrey, Professor G.D.W. Smith*

The requirement of a sharp needle specimen can limit the type of materials that can be analysed in the atom probe, and is especially problematic in the area of thin films. We are testing a new type of atom probe system, first proposed by Nishikawa (Kanazawa, Japan) in 1993, which uses a micron-sized extraction electrode to allow analysis of microtips formed in thin film materials. This new instrument will allow atomic-scale microanalysis of electronic materials and layered metallic films. (*Omicron GmbH) (**Kindbrisk Ltd.) (Funded by BRITE/EURAM and in collaboration with Omicron GmbH, Kindbrisk Ltd. and Seagate Ltd.)

Atom probe analysis of information storage materials

A Georgalakis, Dr. A. Cerezo, Dr. A.K. Petford-Long, Dr. P. Clifton, Dr. D.J. Larson**

Thin metallic layered films with applications in information storage are being grown by sputter deposition and atom probe tips are being fabricated from these layers by FIB milling. The layer composition and interface nature are being studied using both three-dimensional atom probe analysis, in parallel with HREM studies of the crystal structure of the films. Experimental analysis of interface changes with thermal annealing is being compared with simulations of the interdiffusion process. (*Seagate Technology) (Funded by EPSRC and Seagate Technology)

Atom probe microanalysis techniques

Dr. A. Cerezo, T.J. Godfrey, S. Jordan, Professor G.D.W. Smith*

The combination of field-ion microscope and atom probe techniques allow us to image the surface of materials with atomic resolution and perform microanalysis of sub-nanometre regions or layers within the specimen. We are continuing the development of the Position Sensitive Atom Probe (PoSAP), which can provide a full 3-dimensional reconstruction of atomic-scale composition variations in materials. The latest generation of this instrument includes an optically coupled multi-hit detector system, and uses a reflectron lens to increase mass resolution to m/Dm=600 full-width at half maximum. A new instrument is currently being built to extend the technique to the study of catalysis. (*Kindbrisk Ltd.)(Funded by EPSRC and JREI and in collaboration with Kindbrisk Ltd., Omicron Surface Science Ltd. and Johnson Matthey plc.)

Early stages of precipitation in 6XXX automotive sheet

D. Vaumousse, Dr. P.J. Warren, Dr. A. Cerezo

The thermal response of 6XXX series aluminium sheet materials, as used in the automotive industry, is very sensitive to time at room temperature prior to ageing during the paint-bake process. The compositional variations during the early stages of clustering and precipitation are being studied on the atomic scale using three dimensional atom probe microanalysis. (Funded by EPSRC and Alcan International)

Solute distribution at grain boundaries in low-carbon steels

N. Maruyama, Dr. P.J. Warren, Professor G.D.W. Smith

A combination of TEM and 3DAP techniques are being used to study the atomic distribution of interstitial and substitutional solutes at grain boundaries in low carbon steels, as a function of boundary parameters and steel heat treatment. (Funded by Nippon Steel Corporation)

Thermal ageing of steels

Dr. S. Hirosawa, Dr. A. Cerezo, Professor G.D.W. Smith

The atomic-scale changes which take place in the microstructure and composition of pressure vessel steels during long term thermal ageing are being investigated by three-dimensional atom probe techniques. (Funded by Rolls Royce Power Engineering)

3D Reconstruction of atom probe data

O. Dimond, Dr. P.J. Warren, Dr. A. Cerezo*

Software for accurate 3-D reconstruction of data from the position-sensitive atom probe (PoSAP) is under continuous development. Algorithms include statistical functions for the detection of the early stages of clustering in alloys, and the use of Fourier transforms to permit detailed crystallographic reconstruction. (*Omicron GmbH)(Funded by Kindbrisk Ltd. and Omicron GmbH)

Atomic changes during the tempering of steel

*R. Hardwicke, Dr. A. Cerezo, Professor G.D.W. Smith, Prof. G. Krauss**

The atomic-scale redistribution of solute elements which takes place during the tempering of alloy steel is being studied by three-dimensional atom probe techniques, with the objective of understanding how solutes affect the strain ageing behaviour. (*Colorado School of Mines)

III - ELECTRON DIFFRACTION AND TRANSMISSION MICROSCOPY, SCANNING ELECTRON MICROSCOPY, X-RAY MICROSCOPY AND MICROANALYSIS

The Department has a comprehensive range of electron optical instruments for structural and chemical characterization on the atomic level. In addition to a number of routine transmission and scanning electron microscopes, there are several state-of-the-art instruments for:

High resolution electron microscopy (HREM)

The Department's 400 kilovolt JEOL 4000EX(II) electron microscope, commissioned in 1989, has a point-to-point resolution of 0.16nm with an information limit approaching 0.12nm; this is currently better than any other instrument in the UK.. The technique of structure imaging is being used to elucidate disorder on the atomic scale in a wide range of variety of crystalline materials. This instrument is equipped with parallel EELS (electron energy-loss spectroscopy) and an on-line TV system.

Analytical electron microscopy (AEM)

A Philips CM20, a modern 200 kV AEM with full analytical facilities, was installed in 1990. A unique feature of this instrument is an energy-dispersive X-ray system (EDX) with simultaneously usable twin detectors. One detector is a standard thin Be window type, capable of quantitative analysis of elements down to Na (Z=11). The other is a windowless detector capable of analysing for light elements down to B (Z=5).

High resolution analytical electron microscopy

A JEOL 2010 high resolution electron microscope has been installed in 1994. This instrument combines high spatial resolution (down to 0.19 nm) with a two nanometre diameter electron probe for nano-diffraction or convergent beam diffraction. An energy-dispersive X-ray analysis capability and an on-line TV imaging system make this a very versatile instrument. It is being used in a wide range of projects, with particular emphasis on microstructural characterization of nanocomposites.

Field-emission-gun high resolution electron microscopy (FEG-TEM)

Early in 1999 the Department installed and commissioned the UK's first 300 kilovolt field-emission-gun high resolution electron microscope. This instrument, a JEOL 3000F, is fully equiped with a comprehensive range of advanced analytical facilities, including light-element sensitivity EDX, parallel electron energy loss spectroscopy (PEELS), energy-filtered imaging (GIF), an electrostatic biprism for electron holography, a high performance CCD camera and a piezoelectric, drift correcting specimen stage. With a capability of microanalysis and electron diffraction from areas down to <0.4nm in diameter, and a spatial resolution of 0.16nm, this instrument is being used in a wide range of applications, involving new nanostructured materials.

In-situ high resolution analytical electron microscopy

A 400 kV high resolution electron microscope (JEOL 4000EX) has been extensively modified so that it can be equipped with either a gas environmental cell for in-situ studies under controlled atmosphere conditions, or with a low-field objective pole-piece for studies of magnetic materials. The unique gas environmental cell facility is capable of better than 0.3nm resolution whilst the specimen is surrounded by gas and held at elevated temperature. The instrument also includes x-ray microanalytical and electron energy loss spectroscopic (PEELS) facilities, together with an on-line TV imaging and recording system. With the low-field objective pole-piece inserted magnetic specimens can be studied under a controlled applied field or at elevated temperatures. Recent additions to the instrumentation enable magnetisation configurations to be mapped quantitatively.

Scanning transmission electron microscopy (STEM)

The VG HB501 STEM has a high brightness field emission gun (FEG) and facilities for microanalysis from selected areas of 1nm in diameter. Microanalysis techniques include energy dispersive x-ray spectroscopy (EDS) and electron energy loss spectrometry (EELS). The beam size and stability gives sub-monolayer sensitivity for grain boundary segregation analyses. External computer control has been implemented for a flexible approach to mapping and linescan techniques. The gun and specimen chambers are all ultra-high vacuum (<1×10^{-10}mbar). The instrument is used primarily for combined structural and chemical analyses of interfaces.

Scanning electron microscopy (SEM)

The JEOL JSM-840F field emission scanning electron microscope (FEG-SEM) was installed for the purpose of obtaining images of crystal defects in bulk materials using the electron channelling contrast imaging (ECCI) technique developed in the department. This machine can produce both images of single dislocations and electron channelling patterns (ECP) and is being used to investigate sub-surface dislocation arrays and networks in partially relaxed epilayer materials and also to study defect distributions around crack tips. A JEOL JSM-6300 scanning electron microscope has also been installed for electron diffraction experiments. This has a LaB_6 gun which gives a higher beam current but also a larger probe size. An electron back scatter diffraction (EBSD) system allows automated mapping of local crystal orientation.

Electron probe microanalysis (EPMA)

The department made a successful joint proposal with Department of Earth Sciences to the 1997 Joint Research Equipment Initiative for a high-specification microprobe. The instrument chosen was a JXA 8800RL electron probe microanalyser with four wavelength-dispersive X-ray spectrometers, for high-volume, automated microchemical measurements. This instrument is particularly suitable for light element analysis and X-ray mapping. It was installed in October 1999 and is presently undertaking acceptance tests. The microprobe will contribute essential microchemical information to a series of research projects investigating metallic, ceramic, composite, superconducting, biomedical and sedimentary materials. The overall objective of the research is to apply state-of-the-art microprobe techniques in an integrated way to synthetic and natural materials. The scientific and technological impact will range from the development of improved efficiency aeroengine components and new prosthetic bone implants to phases synthesised at ultra-high (earth's core) pressures and marine sediments related to global change and the environment.

Focused Ion Beam system (FIB)

The department has recently installed an FEI FIB2000 TEM system that will be used for micromachining with a spatial resolution down to 12 nm, and for sample preparation of TEM and atom-probe samples from specific sites. The system includes gas injectors for enhanced etching of metals and insulators, plus deposition of Pt.

Diffuse and inelastic scattering in RHEED

Dr. S.L. Dudarev, Professor L.-M. Peng**, Dr. Z. Mitura***, Professor M.J. Whelan*

Methods are being developed in order to understand diffuse scattering from rough crystal surfaces and investigate the effect of inelastic scattering on RHEED intensity. (*UKAEA Fusion, Culham Science Centre; **Chinese Academy of Science; ***University of Krakov, Poland)

Characterisation of RHEED oscillations

Dr. Z. Mitura, Dr. S.L. Dudarev**, Professor M.J. Whelan, Professor L.-M. Peng****

The phenomenon of reflection high energy electron diffraction oscillations is being investigated theoretically. Special attention is being paid to the phase of oscillations as this quantity may be both precisely measured experimentally and determined computationally. (*University of Krakov, Poland; **UKAEA Fusion, Culham Science Centre; ***Chinese Academy of Science)

Integrated quantitative HREM analysis and molecular modelling

Dr. G. Möbus

A program package for numerical structure retrieval from HREM images, using iterative simulation and digital image matching, is extended and maintained with respect to: (i) improving user-friendliness and conversion to a public-domain product; (ii) including three-dimensional structural coordinates in extension to projected atomic column positions; (iii) a methodology is developed which allows interface and dislocation structure models to be characterised simultaneously by an energy value (from semi-empirical potentials)and by an experimental R-factor (from matching simulated to experimental HREM-images). (Funded by EPSRC)

The structure of copper precipitates in age-hardening steels

Dr. M.L. Jenkins, Professor J.M.Titchmarsh, X. Wang

High-resolution and analytical transmission electron microscopy is being used to study the structure and chemistry of Cu-rich precipitates in age-hardening martensitic and maraging stainless steels, which appear similar to those found in irradiated pressure-vessel steels and model alloys. The potential of energy-selected imaging (using the Gatan imaging filter on the Jeol 3000F FEGTEM)is being explored.

Disorder in complex oxides

*Dr. J.L. Hutchison, Dr J. Sloan, Dr. M.-J. Sayagues de Vega**

Disorder in a variety of complex oxide structures which include layered bismuthates, non-stoichiometric rutiles and tungsten oxides is being investigated by high resolution techniques using the Oxford JEOL 4000EX and 3000F ultra-high resolution instruments. (*Univertsity of Seville) (Funded by British Council and NATO)

Preparation of TEM specimens for high resolution electron microscopy

Dr. R.M. Langford, Dr. Y.Z. Huang, S. Lozano-Perez and Dr. A.K. Petford-Long

Tehcniques are being developed to prepare site-specific TEM cross-section and plan-view specimens. In particular, the effect of different milling parameters and methods to manipulate the specimens in-situ for broad-beam ion milling are being investigated.

In-situ TEM studies of magnetic domain structure

Dr. A.K. Petford-Long, R.C. Doole

Facilities are being developed for Lorentz microscopy of magnetic materials using a 400kV TEM. Facilites developed so far allow the effects of temperature and applied fields on the magnetic domain structure to be studied in situ using heating, cooling and magnetising stages, with the additional capability of observing active magnetoresistive elements in situ. The range of facilities is being further extended. (Funded by The Royal Society)

Determination of gradual partial coherence of hetero-interfaces using quantitative HREM

*Dr. G. Möbus, Dr. B.J. Inkson, Dr. M. Hytch***, Dr. A. Trampert**, Dr. T. Wagner**

High-resolution electron micrographs of a large variety of hetero-interfaces (metal-metal, metal-ceramic, semiconductors) are digitally analysed. A novel combination technique measures local amplitude and phase spectrum of lattice fringes with high spatial resolution. Presence of strain and misfit dislocations are detected as a function of interface distance. A classification scheme for hetero-interfaces is developed from the measurements. (*Max-Planck-Institut fuer Metallforschung, Stuttgart, Germany,**Paul Drude Institut, Berlin, Germany; ***CNRS, Paris-Vitry, France)

Investigation of carbon nanotubes produced by novel synthetic methods

*Dr. J.L. Hutchison, Dr. J. Sloan, Prof. N.A. Kiseler**

We are investigating the structure of carbon nanotubes prepared by various synthetic routes with the aims of controlling tube dimensions, and understanding growth mechanisms. (*Insitutie of Crystallography, Russian Academy of Sciences)

Behaviour of carbon in controlled environment electron microscopy

*Dr. J.L. Hutchison, Dr. A.P. Burden**

The behaviour of various forms of carbon in the gas-reaction cell is being investigated. The interaction of the electron beam with the volume of gas above the specimen produces a plasma which in turn generates new structures - including fullerenes. The effects of different gases and other parameters are being studied with a view to understanding the mechanisms of fullerene growth. (*Rutherford Appleton Laboratory)

Polyhedral and cylindrical metal chalcogenides

*Dr. J.L. Hutchison, Dr. J. Sloan, Professor R. Tenne**

Closed polyhedral structures of the layered materials WS2, MoS2 and other chalcogenides have recently been discovered. They are in the form of concentric, polyhedral shells, somewhat similar to the "buckyball" and "fullerene" carbon cage compounds. Their formation and structures are being investigated by high resolution electron microscopy. Their possible use as high-performance solid lubricants is being investigated. (*Weizmann Institute, Israel) (Funded by UK-Israel Research Fund)

Development of an aberration-corrected electron microscope for high resolution analysis and imaging

Professor D.J.H. Cockayne, Professor J.M. Titchmarsh, Dr. J.L. Hutchison, Dr. R. Dunin-Borkowski, Dr. C.J.D. Hetherington

As part of a major research grant, the Department has secured funding which enables us to work closely with an electron microscope manufacturer in developing the next generation of high performance electron microscopes. The new instrument will include a field-emission-gun, aberration correctors and various advanced detectors which will provide analysis and spatial resolution capabilities a the 1 Å level. The instument will be used for atomic-scale investigations of a range of new materials.

Electron back scatter diffraction

Dr. A.J. Wilkinson

Electron back scatter diffraction patterns are used to measure the orientations of individual grains and grain boundary misorientations in polycrystals. The method is being applied to characterise crystallographic textures in a variety of materials systems: Al-Li alloys, Ni-based superalloys, steels, metal-ceramic interfaces, and superconductors. New analysis methods are being developed for the measurement of small angle misorientations developed during plastic deformation and for the measuremnet of local elastic strain tensors. (The Royal Society)

Development of Spectroscopic Tomography for 3D Composition Mapping of Nanomaterials.

Dr. G. Möbus, Dr. B.J. Inkson, Professor J. Titchmarsh, Professor D. Cockayne, Dr. R. Langford, R. Doole, U. Eigenthaler, Dr. R. Brydson**, Dr. M. Hytch***, Dr. A. Trampert****.*

Energy filtered transmission electron microscopy (EFTEM) and EDXS-mapping are used to obtain compositional maps over very large ranges of angular tilt. Tomographic reconstruction and display algorithms are developed to account for the specific properties of EELS and EDXS based data sets. Specimen preparation techniques are optimised towards high tilt capabilities. Applications comprise nanoparticles, nanocomposites, multilayered metal and semicondutor structures. (*Max-Planck-Institut fuer Metallforschung, Stuttgart, Germany, **University of Leeds, ***CNRS Paris-Vitry, France, ****Paul-Drude-Institut, Berlin, Germany)

Development of transmission electron microscopy for studies of crack tips

Professor J.M. Titchmarsh, Y. Huang, S. Lozano-Perez

Environmentally Assisted Cracking and Irradiation Assisted Stress Corrosion Cracking are phenomena which occur in power generating plant and are difficult to predict or control. The influence of precipitation, intergranular segregation, lattice defects and alloy chemistry on crack nucleation and growth will be investigated by developing the techniques to prepare TEM specimens containing crack tips and characterising microstructures. Correlation between crack tip microstructure, applied stress and environmental parameters will be made. (In collaboration with Rolls Royce, AEAT and INSS)

Structure of amorphous materials

*Professor D.J.H. Cockayne, Dr W. McBride, Dr D Nguyen Manh, Dr. R. Dunin-Borkowski, Professor D. McKenzie**

The structure of amorphous thin films and small volumes of amorphous materials is being investigated using energy selected electron diffraction combined with atomistic modelling. Refinement procedures are being developed which will allow differentiation between alternative structural models. (*University of Sydney)

Quantum dot structures

Professor D.J.H. Cockayne, Professor G.A.D. Briggs, C. Lang, Dr J. Zou, Dr L. Xiaozhou*, Dr. R. Leon**, Professor C. Jagadish****

The geometry and composition of quantum dots in semiconductor materials is being investigated with a range of electron optical techniques including HREM, energy filtered EM, and image simulations. (*University of Sydney; **California Institute of Technology; ***Australian National University)

Nanobeam analysis of strained layers with highest spatial resolution

Dr. G. Möbus, Dr. C.J.D. Hetherington, Dr. R. Dunin-Borkowski, Dr. J.L. Hutchison

Convergent beam electron diffraction is applied using the highly focused beam of the FEGTEM JEM-3000F to obtain local information on strain and composition of multilayered structures. The diffraction information is complemented by and compared to energy loss signals obtained with the same nanobeam. An automatised beam propagation mode to scan two-dimensional areas of interest on the specimen will be implemented.

Development of an advanced FIB system for micromachining applications

Dr R M Langford and Dr A K Petford-Long

Novel techniques based on the use of a focused ion-beam system are being developed. These are primarily aimed at micromachining of a range of materials systems such as magnetic devices, optoelectronic devices and embossing heads. The aim is to develop techniques for the fabrication of structures with high depth:width aspect ratio and curved side-walls. (Funded by EPSRC)

3D microstructural characterisation of deformed nanostructures using FIB

Dr. B.J. Inkson, T. Steer, Dr. M. Bobji, Dr. G. Möbus, Dr. O. Kraft, Dr. T. Wagner**

A new technique of characterising nanostructures in 3D by serial FIB sectioning and 3D computer reconstruction is being developed. 3D microstructual maps of deformation under indents, grain shape around deformation sites and crack profiles are being determined and related to the local mechanical properties determined by nanotesting (Funded by the Royal Society, EPSRC, DAAD and The British Council) (* MPI fuer Metallforschung, Germany).

Electron probe microanalysis of multicomponent materials

Dr. M.L. Jenkins, C.J. Salter, Dr. C.R.M. Grovenor, Dr. J.T. Czernuszka, Dr. P.S. Grant, Dr. E. Young, Dr. E. McClelland*, Professor R.K. O'Nions*, Professor B. Cantor*

A state of the art electron-probe X-ray analyser (JEOL JXA 8800RL) has been installed for composition analysis and mapping of a wide variety of complex multicomponent metals, ceramics, composites, biomaterials and minerals.(*Department of Earth Sciences) (Funded by JREI)

Subnano-Probe Performance Optimisation for FEG-TEM/STEM

Dr. G. Möbus, Dr. R. Dunin-Borkowski, Dr. C.J.D. Hetherington, Professor J.M. Titchmarsh, Professor D. Cockayne, Dr. J.L. Hutchison

Optimum conditions of beam formation for nanoanalysis and STEM imaging will be examined around a newly developed software package for dynamical propagation of subnano-beams. The project comprises: (i) STEM performance tests by matching of simulated to experimental beam profiles; (ii) prediction of future modes of imaging and analysis in STEM with and without Cs-correction; (iii) evaluation of modes and limitations for "single atomic column" EELS.

Materials for a quantum logic gate

J. Brook, Dr. J. Sloan, Professor G.A.D. Briggs

We shall use high resolution electron microscopy and statistical analysis to characterise candidate materials for a quantum logic gate.

Fabrication of site-specific 3-dimensional atom-probe specimens

Dr R M Langford, Dr M Kowalewski, Dr A K Petford-Long, Dr A Cerezo, Dr P Clifton and Dr D J Larson**

The FIB is being used to develop methods to prepare site-specific 3-dimensional atom-probe specimens from flat samples such as magnetic multilayer films, so that the magnetic and transport properties can be directly compared with the 3-dimensional morphology. (*Seagate Technology) (Funded by EPSRC and Seagate Technology)

Multivariate analysis of EDS and EELS data

Professor J.M. Titchmarsh

The generation of large data sets by EDX and EELS imaging and spectroscopy is now routine using modern analytical TEM methods. However, conventional processing of data cannot separate small signals from artefacts and noise and cannot always detect correlations between signals. Multivariate analysis methods are being developed for routine handling of large data sets to improve the extraction of information from analytical EM data. (In collaboration with AEAT and INSS)

Tilt- and through-focus series image reconstruction techniques for super-reconstruction electron microscopy

Dr. R. Dunin-Borkowski, Dr. J.L. Hutchison, Dr. J. Sloan, Dr. A.I. Kirkland, Dr. W.O. Saxton**

We are developing numerical techniques for reconstructing exit-waves from crystals to enable us to extract both the amplitudes and phases of diffracted beams. In this way the useable information in lattice imaging from the JEOL 3000F instrument can be extended out as far as 1Å. In the case of complex oxide structures the positions of the oxygen atoms are clearly revealed by this technique. (*University of Cambridge) (Funded by EPSRC)

3-D microstructural analysis using a FIB system

Dr R M Langford, Prof J M Titchmarsh, Dr B J Inkson, Dr G Mobus, T Steer

A focused ion-beam system is being used to mil a set of cross-sections through a chosen area. The images obtained are being used to reconstruct the 3-dimensional microstructure. The suitability of this technique to a range of different systems and the errors associated with the milling and the 3-D reconstruction are being assessed.(*Sheffield-Hallam University) (Funded by the EPSRC)

IV - NUCLEAR MICROSCOPY WITH THE SCANNING PROTON MICROPROBE

The Scanning Proton Microprobe (SPM) was established in its present form in the Nuclear and Astrophysics Laboratory in 1988 following several years of development in the then Nuclear Physics Department, and became a part of the Materials Department in 1997. The instrument uses a focused beam of 1 - 3 MeV protons (or alpha particles or oxygen ions as required) to carry out elemental microanalysis and mapping. At present the facility uses the techniques of *proton-induced x-ray emission* (PIXE) and *Rutherford backscattering* (RBS) to obtain elemental compositions over the whole periodic table with a detection limit in the range of 1 - 100ppm and a spatial resolution of 1µm. RBS also has the possibility of determining depth profiles or surface film thickness in suitable samples. In addition to these main techniques, other interactions are exploited for more specialised applications. These include *ion beam induced charge* (IBIC) for mapping the active regions of semiconductor devices, *elastic recoil detection analysis* (ERDA) for measuring near surface (<500nm) depth profiles of light elements (H, D, He etc.), *total reflection PIXE* (TRPIXE) for measuring metal concentrations in surface monolayers and *scanning transmission ion microscopy* (STIM) for mapping the thickness/density variations in thin (<30µm) samples. In crystalline materials most of these interactions can be used in conjunction with channelling to investigate lattice properties and defects.

The high energy ions are produced by a small tandem Van de Graaf accelerator and the beam is either focused to a 1µm diameter spot in a high vacuum target chamber (using a novel high precision quadrupole triplet lens) or brought into air through a thin foil to allow analysis at 250µm resolution of large objects or objects incompatible with vacuum. A programme of instrumentation development aimed at enhancing the capability of the technique and producing a beam of 0.1µm diameter is running in parallel with collaborations in wide range of analytical projects.

Measuring small strains in graded Si(1-x)Ge(x)/Si layers using ion beam rocking

Dr. D.G. de Kerckhove, Dr. M.B.H. Breese, Prof. P.J.M. Smulders**, Prof. D.N. Jamieson***, Dr.* G.W. Grime

Using an additional beam deflector the scanning system of the proton microprobe can be modified to generate a beam deflection in angle instead of position which allows the system to be used to produce high-resolution angle resolved channelling maps of crystalline materials. This system is being used to measure very small strain in thick (several microns) graded composition alloys of silicon germanium grown by plasma assisted chemical vapour deposition. Such materials, with their curved off-normal planes, have been proposed as alternatives to conventional crystal extraction systems for bending and extracting 100 MeV - GeV protons in medical accelerators and high energy accelerators such as those found at CERN, Geneva. In order to bend protons effectively, these layers must be fully strained. Proton beam rocking provides the means for measuring strain and any relaxation as a function of depth by using Rutherford backscattering spectrometry (RBS). Detailed simulations of the channelling behaviour of protons in the 1 to 10 MeV energy range are used to interpret results. (*Department of Physics, University of Surrey; **Materials Science Centre, Groningen University, The Netherlands; ***School of Physics, University of Melbourne, Australia)

Determination of hydrogen and metals on mineral surfaces

Dr. R.A. Wogelius, Dr. G.W. Grime, Dr. D.G. Fraser***

The SPM has been used in two studies of chemical interactions on mineral surfaces. 1) ERDA using a beam of 7.5MeV O16 ions has been used to investigate the depth distribution of hydrogen on clean mineral crystal surfaces hydrated under high temperature and pressure. 2) Total reflection PIXE (using a beam a glancing incidence) has been used to determine the concentration of metals reacted onto the surface of mineral crystals at sub-monolayer concentrations. The sensitivity for Zn on olivine surfaces was significantly better than using TRXRF with synchrotron radiation. (*University of Manchester Department of Earth Sciences; **University of Oxford Department of Earth Sciences) (Funded by NERC)

Direct-write microlithography in polymers and glass using MeV ion microbeams

Dr. D.G. de Kerckhove, M. H. Abraham, I. Gomez-Morilla, Dr. G.W. Grime

The low scattering of MeV ions in solids means that the ~1 micron spatial resolution of a focused proton beam is maintained over a long range (typically 60 microns in silicon at 2 MeV). High aspect-ratio structures have been fabricated in PMMA and photo-sensitive glass. Grooves 1 micron wide and 100 microns deep can be formed, and more recent work has produced miniature (100 microns diameter) gear wheels and turbines with angled blades in PMMA. Structures have also been formed in photo-sensitive glass, and preliminary results show that it is possible to form buried waveguides in glass.

Metal uptake by hyperaccumulating plants

Professor J.A.C. Smith, Dr. G.W. Grime*

Certain plants native to metal rich soils have the ability to sequester high levels of metals in their tissues, and these are being investigated with a view to their use in bio-remediation of polluted soils. MicroPIXE is being used in a study of the transport mechanism and storage sites of metals in a range of plants known to be hyperaccumulators. In the nickel hyperaccumulating plant Alyssum lesbiacum it was found that the nickel is stored in modified leaf hairs on the leaf surface. (*University of Oxford Department of Plant Sciences) (Funded by NERC)

Development of a 0.1 micron Scanning Proton Microprobe beamline

Dr. G.W. Grime, Dr. I. Rajta, Dr. M.B.H. Breese*, M.A. Marsh, Dr. D.G. de Kerckhove

A new beamline is under development which is planned to have a beam diameter of 0.1 micron with sufficient beam current to carry out PIXE, RBS and STIM analysis. The increased resolution will be achieved with a combination of a new design of high demagnification magnetic quadrupole triplet lens and careful engineering to ensure that the system is mechanically and electrically stable to the high tolerances required. The expected beam current at 0.1 micron beam resolution is 30pA of 3MeV protons. At the very low beam currents required for STIM and IBIC the beam diameter is predicted to be 10 - 30nm. (*Department of Physics, University of Surrey) (Funded by the Wellcome Trust)

V - RADIATION DAMAGE

Displacement cascade processes

Dr. M.L. Jenkins, Dr. M.A. Kirk, Dr. C. Abromeit**, Dr. H. Fukushima****

Displacement cascade processes in various materials are being studied by diffraction-contrast, high-resolution and analytical electron microscopy techniques. The "disordered zone" technique is being used to study cascades in the ordered alloys Ni3Al and Cu3Au. Studies are also being extended to high-Tc superconductors and to intermetallics. Some of the work involves in-situ heavy-ion irradiations in the Argonne IVEM-Tandem Facility. (*Argonne National Laboratory; **Hahn Meitner Institut, Berlin; ***Hiroshima University) (Funded by British Council and U.S. D.O.E.)

Mechanisms of embrittlement in reactor pressure vessel steels

Dr. M.L. Jenkins, Professor J.M. Titchmarsh, Professor Sir Peter Hirsch, Dr. R. Dunin-Borkowski, Dr. M.A. Kirk, X. Wang*

Electron microscopy of heat-treated and irradiated pressure vessel steels and model alloys is being carried out to identify the mechanisms by which these materials become embrittled during neutron irradiation, with particular emphasis on (i) the precipitation of copper-rich particles, and (ii) identification of the matrix component of hardening.(*Argonne National Laboratory, Argonne IL, USA) (in collaboration with Argonne National Laboratory)

Investigations of the stability of precipitates under cascade-producing irradiation

Dr. M.L. Jenkins, Professor G.D.W. Smith, Professor J.M. Titchmarsh

The stability of various precipitate phases in copper, nickel and iron matrices under heavy-ion irradiation is being investigated using high-resolution electron microscopy (in collaboration with the Hahn-Meitner Institute, Berlin)

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The primary goal of this work was to predict the key properties of magnetic tunnel junctions which are used in magnetic RAMs, read heads and sensors. They consist of an insulating layer sandwiched between two ferromagnetic layers. First-principles density-functional calculations of the atomic and electronic structure of cobalt/alumina/cobalt tunnel junctions were performed in order to elucidate the main features determining the spin-dependent tunneling. It was found that the local density of states at the Fermi energy decays exponentially with distance from the interface into the alumina, the average decay length being larger for the majority-spin electrons than for the minority-spin electrons. This explains the observed positive spin-polarization of the tunneling current in cobalt/alumina junctions. Phys. Rev. B 62, 3952 (2000)

Dr Ivan Oleinik, Dr Evgueni Tsymbal and Professor David Pettifor

G. Modelling and Simulation

A materials modelling laboratory was set up in 1992 on the top floor of 21 Banbury Road. It houses a suite of Hewlett-Packard and Silicon Graphics workstations which have sole access to a very powerful HP Exemplar S-class computer. The laboratory acts as the focus for all computational modelling within the Department of Materials. It is a world-leading facility in that the research spans the entire spectrum from quantum mechanical atomistic simulations through the microscopic scale to macroscopic continuum modelling. The work of the laboratory supports and complements the in house experimental programmes and has close links with industry. In particular, the Exemplar S-class computer was bought with matching funds from Hewlett-Packard's Convex Division, Information Storage Group, and Corporate Laboratories. The Silicon Graphics Power Challenge was bought with funds from BNFL.

Electron transport and electromigration in nanoscale conductors

Professor A.P. Sutton, Dr. T.N. Todorov, Dr. J.T. Hoekstra, Dr. J.M. van Ruitenbeek***

The electronic conductance of nanometre scale metallic wires has been calculated as they are pulled to fracture in a molecular dynamics simulation. These simulations have revealed the dramatic effect of mechanical instabilities and atomic rearrangements on the conductance. We are now considering the reverse question - the effect of the flow of current on the mechanical properties and stability of the wires. Starting from a tight-binding model, used to model both mechanical and electronic properties, we calculate the change in the density matrix caused by the flow of current, which then yields simultaneously the additional current induced forces on individual atoms and the local current flowing in each bond, allowing a direct comparison of the two. Separately, we wish to model local heating by the current. Our goal is to model current-induced mechanical effects such as electromigration and local heating, and to compare our results with experiments on real nanowires, carried out at Leiden. (*Dept. Physics, Queen's University Belfast; **Kammerlingh Onnes Laboratorium, Leiden) (Funded by EPSRC and Leverhulme Trust)

Modelling diffusional phase transformations in the presence of elastic interactions

Professor A.P. Sutton, Dr. A. Cerezo, D. Mason

Earlier work has shown the necessity of using a vacancy mechanism of diffusion to model diffusional phase transformations in metallic alloys that occur at the 1-10 nanometre scale. Only when the correct mechanism of diffusion is used at the atomic scale will morphologies, kinetics and mechanisms of growth and coarsening be described correctly. In this work elastic interactions are being introduced to model transformations in alloys containing misfitting atoms. The elastic interactions are described by lattice Green's function methods we are developing. (Funded by EPSRC)

Simulations of the electronic and atomic structures of oxide surfaces

Dr. S.L. Dudarev, Professor A.P. Sutton, Dr.* G.A.D. Briggs, Dr. D.T. Goddard**

A new simulation programme is being applied to model surface reconstructions of oxide surfaces, to assist the interpretation of STM images. The oxides display strong electron correlations, e.g. NiO and UO2, and they are representative of a large class of materials that display important catalytic properties. (*UKAEA Fusion, Culham Science Centre; **Research and Development, BNFL Springfield) (Funded by BNFL)

Studies of amorphous carbon films by electron diffraction and modelling

*Professor D.J. H. Cockayne, Dr. W. McBride, Dr. D. Nguyen Manh, Professor D.G. Pettifor, Professor D.R. McKenzie**

Analytic bond-order potentials (BOPs) are being developed and applied to modelling the growth of amorphous carbon films for comparison with in-house electron diffraction and EELS measurements on films grown at the University of Sydney. (*University of Sydney)

Dynamical Ising model simulations of phase separation

Dr. S. Hirosawa, Dr. A. Cerezo, Dr. J.M. Hyde, Professor G.D.W. Smith*

Monte Carlo simulations based on the dynamical Ising model are being used to study the early stages of phase separation in simple alloys. The model is able to simulate spinodal decomposition in Fe-Cr, nucleation and growth in Cu-Co and Fe-Cu, and simultaneous ordering and clustering in Ti-Al. The results of the simulations are compared with experimental measurements of atomic-scale composition variations, as determined by the position-sensitive atom probe (PoSAP). (*AEA Technology, Harwell) (Funded by Rolls Royce Power Engineering)

MBE growth of spintronic materials

*Professor D.G. Pettifor, Dr. I. Oleinik, Professor H. Wadley**

Analytic bond-order potentials (BOPs) are being developed for modelling the MBE growth of spintronic materials such as Mn in GaAs films. (*University of Virginia) (Funded by DARPA).

CVD growth of carbon-nitride films

Professor E.G. Wang, Professor Lianmao Peng*, Dr. Gang Lui*, Dr. I. Oleinik, Professor D.G. Pettifor*

A variable time-step kinetic Monte Carlo model will be developed to model the CVD growth of carbon-nitride films in conjunction with an experimental study. (*Centre for Condensed Matter Physics, Beijing, China) (Funded by Royal Society - CAS joint research project)

Modelling adhesion between polymers and inorganic substrates

Dr. T.R. Walsh, Professor A.P. Sutton

A new strategy for modelling adhesion between polymers and inorganic substrates has been developed. It is being applied to polymer adhesion issues of interest to the semiconductor industry.

Mapping of magnetisation distributions in thin layered films

Dr. A.K. Petford-Long, Dr. P. Shang

We have developed a method for quantitative mapping of the magnetisation in thin magnetic specimens at a high spatial resolution. The method is being used to study the magnetisation distribution in thin films and layered systems. (Funded by Hewlett-Packard Labs.)

Modelling short fatigue crack growth through polycrystals

H.-K. Lee, Dr. A.J. Wilkinson, Dr. S.G. Roberts

We are developing models for short fatigue crack growth based on dislocation mechanics descriptions of crack - plastic zone - grain boundary interactions. The effects of grain size, grain orientation, and grain boundary misorientation distributions are being incorporated through a Monte-Carlo scheme allowing the microstructure induced statistical variations in short fatigue crack growth behaviour to be analysed.

First Principles tight binding package for general applications

*Dr. S.D. Kenny, Professor A.P. Sutton, Dr. A.P. Horsfield**

A multi-application, first principles, LCAO density functional program is being developed for use inside the Materials Modelling Laboratory. (*Fujitsu European Centre for Information Technology) (Funded by Fujitsu)

Effects of directional bonding on deformation and fracture of intermetallics

Professor D.G. Pettifor, Dr. D. Nguyen Manh, Professor V. Vitek,*

The influence of directional bonding on the deformation and fracture of intermetallics is being investigated at the atomistic level in order to understand why some intermetallics are brittle, whereas others are ductile. (*University of Pennsylvania)

First principle studies of transition metal sulphides

H.M. Sithole, P.S. Ntaohae*, H.R. Chauke*, Professor P.E. Ngoepe*, Dr. D. Nguyen Manh, Professor D.G. Pettifor*

The electronic structure, equation of state and structural properties of iron and noble metal sulphides are being predicted using first principles density functional theory. (*Materials Modelling Centre, University of the North, South Africa) (Funded by Royal Society - FRD collaborative project)

Modelling the magnetoresistive behaviour of spin-valve devices

Professor D.G. Pettifor, Dr. E. Tsymbal, Dr. A.K. Petford-Long, Dr. J.A. Brug, Dr. T.C. Anthony*, Dr. C. Moorhouse**

The magnetoresistive behaviour of metallic and tunnelling spin valve structures and devices is being modelled within a Tight Binding description of the electronic structure. The theoretical insights and predictions are correlated closely with the experimental programme both at HP Laboratories and Oxford. (*HP Laboratories, Palo Alto) (Funded by HP Laboratories)

Modelling the structure of amorphous oxide films grown by vapour deposition

*Professor A.P. Sutton, Dr V. Burlakov, Professor G.A.D. Briggs, Dr. Y. Tsukuhara**

A new Monte Carlo technique has been developed and applied to the growth of SiOx films by vapour deposition. The technique enables the structures of network forming oxide films to be modelled, and provides information about radial and bond angle distribution functions as well as porosity. The technique is now being extended to other amorphous oxides and amorphous elements grown by vapour deposition.

Atomistic and microscopy simulations for three-dimensional misfit-dislocation networks of heterointerfaces

A. Levay, Dr. G. Möbus, Professor G. Tichy*, Professor V. Vitek***

Molecular statics relaxations of metallic thin films on rigid substrates are combined to multislice simulations of high-resolution electron microscopy. The aim is to examine the visibility of the misfit dislocation networks in various directions of projection and to decide about real structural features and electron diffraction artefacts. The first model system comprises hexagonal and trigonal networks in Nb thin films. (*Eötvös University, Budapest, Hungary; **University of Pennsylvania, USA)

Fundamentals of brittle-ductile transitions

Dr. S. Noronha, Dr. S.G. Roberts, Professor Sir Peter Hirsch, Dr. A.J. Wilkinson

Cleavage failiure in the Brittle-transition of steels is being treated in terms of a model in which the cleavage is initialed at a microcrack situated in teh stress field ahead of a macroscopic crack. The plastic zone around the microcrack is modelled by computer simulations of dislocation arrays around the micro-crack-tips. Dislocation shielding plays an important part in determining the fracture stress. The model predicts a fracture stress independent of yield stress, in accord with experiments. (Funded by EPSRC, AEAT and HSE)

Modelling of growth of aerosol metal oxide nanoparticles

*Professor A.P. Sutton, P.J.D. Campbell, Dr. D.T. Goddard**

A new approach is being developed to the mathematical description of growth of metal oxide nanoparticles. The purpose of the study is to understand how the distribution of nanoparticles in their size and shape evolves in time and how the nature of this evolution depends on the process conditions. (*Research and Development, BNFL Springfield) (Funded by EPSRC and BNFL)

Modelling secondary electron emission from surfaces with inequivalent terminations

V. Jones, Professor A.P. Sutton, Dr. M.R. Castell

Objects with more that one type of surface termination, where there is a difference in the workfunction or potential of the terminations, give rise to "patch fields". The influence of these fields on secondary electron emission will be modelled. (Funded by EPSRC)

Modeling the evolution of cascades in fusion reactor materials

*Professor A.P. Sutton, T. Hudson, Dr. S.L. Dudarev**

Fusion reactor materials are subjected to neutron irradiation with energies in excess of 14MeV. These neutrons produce cascades of damage which evolve over time into defects and defect clusters. The temporal evolution of the cascade damage will be modelled by Monte Carlo simulations, with particular attention to long-range elastic interactions. (*UKAEA Fusion, Culham Science Centre.)

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An imported German crucible (Hessian Type), circa 1680-1710, used in the first University chemistry laboratory, at the Old Ashmolean (now the Museum of History of Science, Broad Street, Oxford).

Dr. Peter Northover and Chris Salter

H. Materials Science Based Archaeology

The Materials Science Based Archaeology Group is concerned with the investigation of all aspects of the metallurgical process, from smelting to metal finishing, and from the first use of alloys in the 5th/4th millennia BC to the Industrial Revolution. The themes of the research can be broadly labelled as archaeological and metallurgical. In archaeology the research derives from post-excavation and museum-based projects involving the characterization of the products and residues of past metallurgical processes. The results are used to explore the place of metals in ancient economies and societies, how they were made, used, traded and re-cycled, how their properties were understood, and what processes were associated with their deposition and survival in the archaeological record. This work is supported by experiments designed to relate this material to the process variables which shaped its formation. These experiments also form a link with the metallurgical objectives of the group. These are to acquire a deep knowledge of the physical and mechanical metallurgy of the metals used in the past, so that we can see how they were understood in the past. The results can be surprising and demand novel research, for example to determine why some alloys have an exceptional ductility. This work also links directly with other areas of metallurgy by extending to $6x10^3$ years the time range available for studying a variety of room temperature phenomena from corrosion to precipitation, and with results applicable in such diverse fields as electronic packaging and the storage of nuclear waste.

Non-ferrous and precious metallurgy in the European Iron Age

Dr. J.P. Northover, P. Nagy, P. Ramsl**, C. Zingerle***

The study of copper-based and precious metal alloys from excavations and hoards of the pre-Roman Iron Age is leading for the first time to an understanding how these metals were made and traded in a period when iron had become the dominant utilitarian metal. Material is now available to illustrate how production on individual sites was organised and what techniques were used together. Attention is also focused on cemeteries to look for associations between gender and status of individuals and the technical quality of artefacts buried with them. (*Abteilung Ur-und Frühgeschichte, Universität Zürich, Switzerland; **Institut für Ur-und Frühgeschichte, Universität Wien, Austria)

Application of microprobe and metallographic techniques to numismatic problems

Dr. J.P. Northover, Dr. D.M. Metcalf, Dr. C.E. King*, Dr. L. Treadwell**

The Cameca SEMPROBE is used to study the copper-, silver- and gold-based alloys used for a variety of coinages. Current projects involve Roman base-silver coinages of the later 3rd century AD, the silver and base-metal coinages of medieval England, the coinage of the pre-Roman Iron Age, Indo-Greek and Islamic coinages. Very large databases of analyses have been and are being assembled in all these areas and attention is now directed to new methods for interrogating these. (*Ashmolean Museum, Oxford)

Early metallurgy in the Upper Euphrates Basin

Dr. J.P. Northover, Dr. K. Prag, Dr. G. Philip***

Microanalysis and metallography have been used to characterise the metalwork from a number of major excavations in the Upper Euphrates basin. The sites straddle political and economic boundaries of the 3rd millennium B.C. during the period in which bronze became the main utilitarian metal. The results have given us a new understanding of the way in which bronze became part of the metal economy and have also focused our attention on the great importance of recycling in these early cities. (*University of Manchester; **Department of Archaeology, University of Durham).

Application of the scanning proton microprobe to the analysis of ancient bronze

Dr. J.P. Northover, Dr. G.W. Grime, M.H. Abraham

The requirements of museum collections have stimulated this project in non-destructive and minimally destructive analysis of ancient bronze. The aim is to use a laser to mill sub-millimetre diameter windows in the patina on selected bronzes and then use the SPM to analyse the metal as it is exposed, with the X-ray mapping facility employed to make basic metallographic observations. An experimental programme will develop the best mode of operation and assess the quality of the analyses in comparison with sample-based analysis.

Investigation of the relationship between slag inclusion compositions and welding practice

C.J. Salter, B.J.J. Gilmour

A study of the changes in slag inclusion and metal compositions that occur during the forge welding of iron. In particular those changes seen in phosphoritic/non-phosphoritic composite iron artefacts. (In collaboration with The Royal Armouries, Leeds)

Anglo-Saxon Ferrous Technology

C.J. Salter, G. Hey, B.J.J. Gilmour, K. Penn***, T. Mallin*****

A systematic survey of the ferrous artefacts from a number of East Anglian and Midland sites is being carried out to determine range and distribution of various Anglo-Saxon black-smithing skills and technologies. (*Oxford Archaeological Unit; ***Norfolk Archaeological Unit; ****Cambridgeshire Archaeology)

Study of the products of the experimental reproduction of the iron-working process at Bryn Y Castell and Crawcwellt Sites, Gwynedd

*C.J. Salter, P. Crew**

A series of iron smelting an smithing experiments have been carried out to reproduce the metal and other iron-working debris from these important Iron Age sites. Presently, this material is being studied, an attempt to fully understand the chemistry, microstructure and mechanical properties of the different types of iron and steel produced. (*Snowdonia National Park Study Centre, Maentwrog, Gwynedd)

Study of possible Bronze Age copper smelting debris from the Great Orme, Gwynedd, Wales

*C.J.Salter, Dr. J.P. Northover, S. Jones**

Although there is extensive evidence of Bronze Age mining activity in Britain, as yet no evidence of prehistoric copper smelting has been discovered on the British mainland. A small quantity of slag-like from contexts dated to circa 1580 BC are being studied to determine its nature and mode of origin. This will include experimental reproduction. (*Gwynedd Archaeological Trust)

Effects of cremation on copper alloys

Dr. J.P. Northover

The effects of high temperatures on copper alloys in oxidising, neutral and reducing atmospheres is being studied by optical metallography and by experimental replication. The results are used to determine the placement of grave goods in cremation pyres to assist in interpreting the burial rites. (In colloaboration with Wessex Archeology)

Effects of conservation techniques in bronze

Dr. J.P. Northover, N. Norman, M. Sahlstedt***

A combination of optical and scanning electron microscopy, electron microprobe analysis and PIXE/RBS spectrometry will be used to compare the effects of different conservation methods of a group of Bronze Age tools and weapons. Particular attention will be paid to the preservation of surface detail during cleaning and treatment, and to the distribution of inhibiting species in the corrosion and patina after treatment.(*Department of Antiquities, Ashmolean Museum; **Conservation Institute, Gothenburg University)

The characterisation of Islamic steels

*Dr. B.J. Gilmour, C. J. Salter, Dr. J. Allan**

A long-term project to develop the characterisation of high carbon and alloy steels from the medieval Islamic world using a range of metallographic and microprobe techniques. (*Department of Eastern Art, Ashmolean Museum)

The development of iron and steel structural and railway use in the 19th century.

*Dr J.P. Northover, T. Swailes**

Rails and structural iron and steelwork surviving from the nineteenth century are often well dated and provenanced to specific ironworks. They offer an ideal means of studying developments in the capabilities of, successively bloomery iron, puddled iron and Bessemer steel for such products. A successful pilot project on rail steels is being linked to a programme of mechanical testing of 19th century structural ironwork at UMIST to link mechanical properties, including fracture behaviour, to microstructure. (*Dept. of Civil Engineering, UMIST)

The use of high resolution scanning Auger microscopy to characterise internal corrosion in archaeological bronze

Dr J.P. Northover, Dr E. Paparazzo*, Dr D. Baer**, Dr S. Lea***

The mechanisms of long-term corrosion processes of buried bronze surfaces are beginning to be well understood. Much less clear are those involved in sub-surface and internal inter- and transgranular corrosion in the same objects. The approach used here is to maximise the resolution obtainable with scanning Auger microscopy in mapping corrosion species at grain boundaries in bulk samples. Resolutions of the order of 10nm have been obtained in elemental mapping; the interpretation of these maps is now being developed. (*ISM-CNR, Frascati, Roma, Italy; **Pacific Northwest National Laboratory, Richland, WA, USA)

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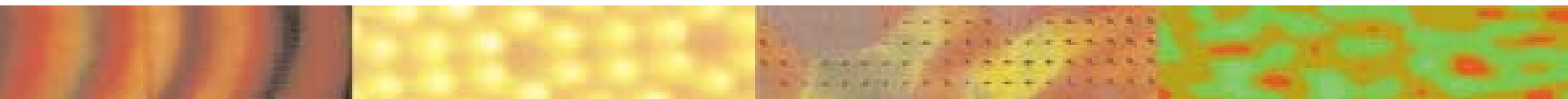

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