Register for the meeting at www.msm.cam.ac.uk/camtec

OPENING OF CAMBRIDGE MICRO-MECHANICAL TESTING CENTRE (CAMTEC)

Workshop, Tuesday 11th April 2006

AUSTIN WING OF MATERIALS SCIENCE DEPARTMENT, CAMBRIDGE

9.30-10.00	*Registration*
10.00-10.20	*Welcome and CAMTEC Tour*
10.20-10.30	*Walk to Downing College*

HOWARD BUILDING, DOWNING COLLEGE, CAMBRIDGE

10.30-10.45	*Coffee, with Poster Display (Ground Floor)*	
10:45-11:15	**Adrian Mann** (**abmann@rci.rutgers.edu**) Mat. Sci. & Eng. Dept., Rutgers Univ., USA	*Nanoscale Mapping of Mechanical Properties and Chemistry in Mineralized Tissues*
11:15-11:45	**Mark Spearing** (**spearing@soton.ac.uk**) Eng. Sci., Univ. of Southampton, UK	*Design of High Power Density MEMS: Materials and Testing Requirements*
11:45-12:15	**Oliver Kraft** (**oliver.kraft@imf.fzk.de**) University of Karlsruhe, D	*Size Effects on Deformation and Fatigue of Thin Films and Small Structures*
12:15-12:45	**Ivo Utke** (**ivo.utke@empa.ch**) EMPA Thun, CH	*In-situ SEM Fabrication, Manipulation, & Mechanical Investigations of Composite Nanowires*
12.45-13.30	*Lunch in West Lodge (Industrial Exhibition)*	
13.30-14.00	*Coffee in Howard Building (Poster Display)*	
14:00-14:20	**Lisa Taylor** (**Lisa.Taylor@pfizer.com**) University of Greenwich, UK	*Predicting Bulk Particle Breakage from Single Crystals*
14:20-14:40	**Gerold Schneider** (**g.schneider@tu-harburg.de**) TU Hamburg-Harburg, D	*Indentation Size Effect in Barium Titanate Single Crystals: Dislocations versus Domain Walls*
14:40-15:00	**Jon Molina** (**jmolina@ceit.es**) CEIT, San Sebastian, ES	*Adhesion Studies in Blanket & Patterned Thin Films using Cross-sectional Nanoindentation*
15:00-15:20	**David Moore** (**dfm1@eng.cam.ac.uk**) Eng. Dept., Univ. of Cambridge, UK	*Use of Scanning Profilometry for Mechanical Characterisation of MEMS Materials*
15.20-15.40	*Tea in Howard Building (Poster Display)*	
15:40-16:00	**Ulrike Wegst** (**wegst@mf.mpg.de**) Max Planck, Stuttgart, D	*Micrometre Scale Mechanical Testing in FIB and SEM*
16:00-16:20	**James Curran** (**jac64@cam.ac.uk**) Mat. Sci. Dept., Univ. of Cambridge, UK	*Fine Scale Mechanical Characterisation of Plasma Electrolytic Oxide Coatings*
16:20-16:40	**Russell Goodall** (**russell.goodall@epfl.ch**) Materials Dept., EPFL, Lausanne, CH	*Study of Creep Deformation during Nanoindentation*
16:40-17:00	**Bill Clegg** (**wjc1000@cam.ac.uk**) Mat. Sci. Dept., Univ. of Cambridge, UK	*Hardening in Multilayered Thin Films*
17.00	*Tea and Discussion (West Lodge)*	

INDUSTRIAL EXHIBITION PARTICIPANTS

Participants	Firm / URL
Peter Rogers (peter.rogers@mts.com) Michel Fajfrowski (michel.fajfrowski@mts.com)	MTS / www.mts.com
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Financial, Accommodation & Travel Arrangements

There will be no fee for the Workshop, but attendance will be by invitation only.

In addition to invited speakers and industrial exhibitors, about 30 further delegates will be attending, who are all welcome to contribute posters. The first 30 people to register (via the website) will be accepted and after that it will be indicated on the website that no further places are available.

Registration, a brief Introduction and a Tour of the CAMTEC facilities will take place in the Department of Materials Science, on the New Museums Site (www.msm.cam.ac.uk/mmc/directions/index.html)

The Workshop will be held in the Howard Building and the West Lodge / Maitland Room, Downing College, Cambridge (www.dow.cam.ac.uk/www_server/Conf.html#HOWARD), which is about 10 minutes walk from the Department.

Travel information, and the location of Downing College, can be found at:
www.dow.cam.ac.uk/dow_server/info/GetToCambridge.html
Visitors to Downing should report on arrival to the Porters' Lodge, which is located by the main entrance to the College in Regent Street. Car parking will be available in the College.

The cost of the Workshop itself, including lunch and refreshments, will be covered by the Organisers. It will be up to individual delegates to organise their own accommodation and travel. A list of nearby hotels is given below. Travel information, and the location of Downing College, can be found via websites at:
http://www.msm.cam.ac.uk/mmc/directions/index.html
http://www.dow.cam.ac.uk/dow_server/info/GetToCambridge.html

Information on accommodation can be found at http://www.visitcambridge.org/visitors/wheretostay.php

Regent Hotel (http://www.s-h-systems.co.uk/hotels/regent-hotel.html)
41 Regent Street, Cambridge CB2 1AB
Tel: (01223) 351470, Fax :(01223) 566562
Single £89, Double/Twin £99
The Regent Hotel is very close to Downing College.

Crowne Plaza Cambridge (http://www.cambridge.crowneplaza.com/)
Downing Street, Cambridge CB2 3DT
Tel: 0870 400 9180, Fax :(01223) 464440
Single £99- 275, Double £99-£275
The Crowne Plaza Hotel is adjacent to the Department of Materials Science, which is on the New Museums site (Pembroke Street).

Lensfield Hotel (http://www.lensfieldhotel.co.uk/)
53 Lensfield Road, Cambridge CB2 1EN
Tel: (01223) 355017, Fax :(01223) 312022
Single £65- 95, Double/Twin £98-£110

ABSTRACTS FOR ORAL PRESENTATIONS

Nanoscale Mapping of Mechanical Properties and Chemistry in Mineralized Tissues

A Mann

Materials Science & Engineering Dept., Rutgers Univ., New Jersey, USA

Hard tissues such as dental enamel and bone consist of mineral crystals packed tightly into an organic matrix. From a materials' perspective they can be thought of as nano-composites consisting of ceramic (defective hydroxyapatite) surrounded by polymer (proteins). Like any composite material changes in the matrix or the filler can affect the tissue's properties. For instance, variations in the size, composition and orientation of the apatite crystals can change the tissue's mechanical properties and increase the risk of fracture. This is seen in some diseases and conditions of the mineralized tissues where local changes in chemistry and microstructure dramatically modify the tissue's elastic and plastic properties (e.g. fluorosis of bones or carious enamel lesions). In this talk I will describe our ongoing research into how diseases and genetic mutations change the chemistry and structure of mineralized tissues and, hence, their nanoscale mechanical properties. Because of the complex microstructure of dental enamel and bone we have focused on understanding local variations in properties by using mechanical and chemical characterization to map properties over sections of tissue. The research has led to a better understanding of how treatments for dental caries work and the role of specific proteins such as osteopontin in bone mineralization.

Design of High Power Density MEMS: Materials and Testing Requirements

SM Spearing

Engineering Science Dept., Southampton Univ., UK

The speaker is involved in several programs to develop the technology for high power density micro-systems. Devices under development include; a micro-gas turbine engine, a micro motor-compressor, a micro-rocket, a micro-hydraulic transducer and various micro-chemical power devices. These devices are designed to operate at high power densities in order to perform energy conversion tasks for applications such as providing portable electrical power, small-scale propulsion or local actuation. Power densities are projected to be in the range of 10-100 W/cc, which is similar to that of large scale prime movers. The design and fabrication of such devices offers many challenges and opportunities in the fields of materials and structures. The major challenges arise from the very high stress levels (~ 1 GPa) required to achieve the necessary performance and, for devices such as the micro-gas turbine and micro-rocket, the high temperatures inherent to creating an efficient engine. The task is complicated by the need to achieve a good structural design within the constraints imposed by micro-fabrication processes. The major opportunities arise from the use of silicon and ceramic materials at small lengthscales. In particular, the use of micro-fabrication techniques offers the potential to control the processing-induced flaw size such that very high strengths can be obtained. In this presentation a brief overview of the projects will be given and the role of materials and the overall approach to structural design for this family of devices will be described. Key materials and mechanics issues will be presented, with particular emphasis on the ramifications of the devices' small size and the implications for mechanical testing requirements. Case studies for room and high temperature strength and how test data is then applied to structural design will be presented.

Size Effects on Deformation and Fatigue of Thin Films and Small Structures

O Kraft

Forschungszentrum Karlsruhe and Universität Karlsruhe (TH), Germany

Size effects on the deformation behaviour of metal films or small structures with typical dimension below 1 µm have been the subject of many detailed studies in the last decade. In these studies, a general trend that “smaller is stronger” has been observed, which has been attributed to the constraint on dislocation motion in a small volume and strain gradient effects. Most recently, size effects have been studied by uni-axial compression testing of small micro-pillars, which were prepared by using a focused ion beam technique. In our work, we have explored the question whether fatigue is also subject to a size effect in the sub-micron regime. For this, we have investigated the behaviour of thin films on substrates during repeated cyclic loading. We have studied the fatigue lifetime and damage formation in metal films with thicknesses between 100 and 3000 nm on compliant polymer substrates with typical total strain ranges between 0.1% and 1%. These experiments indicate clearly that the fatigue resistance and the fatigue mechanism depend on film thickness. In thinner films (< 500 nm), fewer and smaller extrusions were observed, in addition, to an extensive cracking along twin and grain boundaries. In contrast, the thicker films (> 1 µm) showed many extrusions/intrusions and cracks lying along the extrusions rather than along the boundaries. This clear change in fatigue damage behaviour with film thickness will be discussed in the light of existing models and simulations for the motion of dislocations in finite volumes. It is argued that fewer mobile dislocations are available, and, as a result, the decrease in film thickness and grain size leads to a reduction of the accumulated plastic strain within grains. Furthermore, the formation of extended dislocation structures, which have in bulk materials characteristic length scales of the order of 1µm, can no longer be established as confirmed by transmission electron microscopy. The insights gained from these studies, in particular from the observations of length scale dependent damage mechanisms, provide a basis for developing models for fatigue life prediction in thin films.

In-situ SEM Fabrication, Manipulation & Mechanical Investigations of Composite Nanowires

I Utke

Nanomechanics and Nanostructuring Group, EMPA, Thun, Switzerland

Focused particle beam fabrication offers the unique possibility to combine fabrication, manipulation, and mechanical testing of nanowires with the high resolution capability of scanning electron / ion microscopes. Our group at EMPA develops focused electron beam (FEB) induced deposition technology and nano-manipulation tools for use inside a scanning electron microscope. This talk summarizes state-of-the-art equipment and results at EMPA: FEB induced growth of metal/carbon nano-composite wires, FEB “soldering” of nano-wires to force sensors, and results on their density, tensile strength, and bending behaviour.

Predicting Bulk Particle Breakage from Single Crystals

LJ Taylor

Medway Sciences, University of Greenwich, Medway Campus, Chatham, Kent, ME4 4TB, UK
Pharmaceutical R&D, Pfizer Global Research and Development, Sandwich, Kent, CT13 9NJ, UK

During development of a new solid dosage form, the particle size of the active pharmaceutical ingredients (APIs) plays an important role in processing and dosage form properties. As a result, API is generally milled to meet stringent particle size specifications. Typically, pilot-scale milling trials are run to determine the most effective and efficient mill for each material. Such trials require relatively large quantities of bulk active, often at a stage of development when material is not readily available. The ability to predict the breakage propensity of materials using a small quantity of material would be beneficial. It is believed that both deformation and fracture mechanisms play an important role in particle breakage and thus in milling performance. One parameter that combines the effect of these two competing mechanisms, is the 'brittleness index'. This parameter is the ratio of material hardness (resistance to deformation) to toughness (resistance to fracture). Nanoindentation, a surface characterisation tool, was employed to measure the deformation and fracture properties of pharmaceutical materials from single crystals. These measurements can be used to determine the brittleness index for a range of APIs. When compared with size reduction ratios determined from pilot and full scale milling trials, an excellent correlation is observed between the single crystal and bulk behaviour. This correlation forms the basis of a method to guide mill selection.

Indentation Size Effect in Barium Titanate Single Crystals: Dislocations Versus Domain Walls

GA Schneider[1], T Scholz[1], MV Swain[2], F.J. Espinoza Beltrán[3], WJ Clegg[4] and F Giuliani[4]

[1] *Advanced Ceramics Group, Hamburg University of Technology, Germany*

[2] *Dept. of Biomaterials, Faculty of Dentistry, University of Sydney, Australia*

[3] *Centro de Investigación y de Estudios Avanzados del IPN, Unidad Querétaro, Querétaro, México*

[4] *Department of Materials Science, University of Cambridge, UK*

Nanoindentation tests in *a*- and *c*- domains of an {001} orientated barium titanate single crystal were performed in order to investigate the elastic, plastic and ferroeleastic response of the material. The indentation size effect was studied by using 4 different precisely calibrated conical tips with radii between 61 nm – 1.9 µm. The topography and the polarization vectors of the indented areas were imaged by both atomic force and piezoresponse force microscopy (PFM), respectively. FIB was used to prepare thin sections of areas underneath the indentations for TEM studies. The measured elastic modulus is independent of the indenter radius. After "pop-in" the $BaTiO_3$ shows plastic deformation with a constant mean pressure which increased with decreasing indenter radius. TEM micrographs show dislocations with {110} glide plains. These dislocations can also be visualized by topography images of edged surfaces. The indenter radius dependence of the hardness support the concept of geometrically necessary dislocations" proposed by Nix and Gao (1999) and its extension to spherical tipped indenters by Swadener et al (2002). The PFM images of indented in-plane domains revealed an almost quadratic arrangement of the domains around the indent, which can be explained by residual circumferential tensile stresses around a residual impression and was unambiguously correlated to the crystal orientation.

Adhesion Studies in Blanket and Patterned Thin Films using Cross-sectional Nanoindentation

J Molina[1], I Ocaña[1], D González[1], MR Elizalde[1], JM Sánchez[1], JM Martínez-Esnaola[1], J Gil-Sevillano[1], D Pantuso[2], B Sun[2], G Xu[2], B Miner[2], J He[2], J Maiz[2]

[1] *CEIT and TECNUN, P. Manuel Lardizabal 15, 20018 San Sebastián, Spain*

[2] *Intel Corporation, Hillsboro 97124 (OR), USA*

The thermo-mechanical robustness of interconnect structures is a key reliability concern for integrated circuits. The miniaturization process and the package/silicon interaction result in an increase of the thermal stresses whilst the use of new low-k materials, with degraded mechanical properties, makes it more and more difficult to predict their in-service behavior. Cross-sectional nanoindentation (CSN) was developed to characterize the interfacial adhesion in metallic and ceramic blanket films deposited on silicon substrates. Recently, this technique has been adapted to the study of adhesion failure in real interconnect structures, using a FIB (Focused Ion Beam) to prepare a sample test geometry better suited for this type of patterned films. The cracks are generated in the silicon cross-section by a Berkovich indenter and then propagate through the film stack, preferentially along the weakest interfaces. The method has been applied to test chips simulating a portion of the interconnect structure. The results obtained correlate well with adhesion energies obtained by four-point bending (4 PB) in blanket films. In addition, the CSN technique has proven useful to study local adhesion effects in patterned structures and to analyze crack propagation through the interconnect structure. The test has been modeled using a FE code and cohesive elements developed in house to improve the understanding of the crack path and its interaction with the different structures present in the stack.

Use of Scanning Profilometry for Mechanical Characterisation of MEMS Materials

DF Moore and M Boutchich

Dept. of Engineering, University of Cambridge, Trumpington Street, Cambridge, UK

Flexing micro-beams are important in microelectromechanical systems (MEMS) technology for sensors, actuators and for packaging applications. This talk briefly describes a convenient way to determine the elastic modulus and other mechanical properties of thin films on silicon, with particular reference to 3 micrometre thick silicon nitride films and multlilayer films based upon it, and assesses the prospects for future micromechanical MEMS test beds.

Micrometre Scale Mechanical Testing in FIB and SEM

UGK Wegst

MPI for Metals Research, Stuttgart, Germany

A novel method that allows the micromechanical testing in tension, compression and bending of samples with a length of a few tens of micrometres and a few micrometers or less in diameter will be presented and its application illustrated. It uses a focussed ion beam (FIB) and/or a scanning electron microscope (SEM) system as an in situ laboratory for sample preparation, fixation and testing. Advantages of this method are that samples from larger objects can be prepared site-specifically, and that testing in tension is possible without damage due to gripping, because the samples are affixed by metal 'tapes' deposited using the FIB. Forces are measured with a piezoresistive atomic force microscope (AFM) tip attached to a micromanipulator for high precision positioning. The displacement is determined from FIB or SEM micrographs taken during the test. Since the cross-sectional area of the tested sample can be determined by FIB-sectioning before and after the test, the method is particularly well suited for the testing of porous samples and those with an irregular cross-sectional shape. The technique is remarkably versatile and applicable to micrometre scale testing of biological and man-made specimens.

Study of Creep Deformation During Nanoindentation

R Goodall[1] and TW Clyne[2]

[1] *Lab. for Mech. Metallurgy, Inst. of Mats., Ecole Polytechnique Fédérale de Lausanne (EPFL), CH-1015 Lausanne, CH*

[2] *Dept. of Materials Science, University of Cambridge, Pembroke Street, Cambridge CB2 3QZ, UK*

The increasing capabilities of nanoindentation equipment have allowed measurement of a large range of properties with these devices. Amongst these, creep parameters are being increasingly reported in the literature. However, the accuracy of the methods commonly used is unproven. In this work, previously used nanoindentation methodology is applied to measurement of the stress exponent, n, for about 15 different materials, for which the creep behaviour has already been well characterised through conventional testing. The agreement between the measured values and the literature data is in general found to be very poor. The reasons for this are explored, including the assumptions made in the development of the indentation methodology, and the nature of the deformation occurring during creep indentation. A possible factor is that non-steady state deformation mechanisms, such as primary creep, may be exerting a significant influence on the observed indentation behaviour. A simple procedure is proposed to characterise the creep behaviour of materials during nanoindentation, and allow simple comparison between different materials, despite the highlighted uncertainties.

Hardening in Multilayered Thin Films

WJ Clegg

Dept. of Materials Science, University of Cambridge, Pembroke Street, Cambridge CB2 3QZ, UK

It is shown that the substantial increases in hardness of approximately 30 GPa that have been reported are not generally observed. Cross-sectional transmission electron microscopy of TiN/NbN multilayers with wavelengths from 2 to 25 nm shows that flow occurs by dislocation motion across the layers and by the compaction of layers if they are porous. Even where dense the hardness of the multilayers tested here is approximately 6 GPa greater than predicted by a mixtures rule using the hardnesses of the monolithic components. Furthermore there is no significant variation in hardness with layer thickness. It is tentatively suggested that the high values previously reported were associated with high internal stresses.

ABSTRACTS FOR POSTER PRESENTATIONS