CAMTEC III

Symposium on Fine-Scale Mechanical Characterisation and Behaviour

7th-8th April, 2014

The CAMTEC series

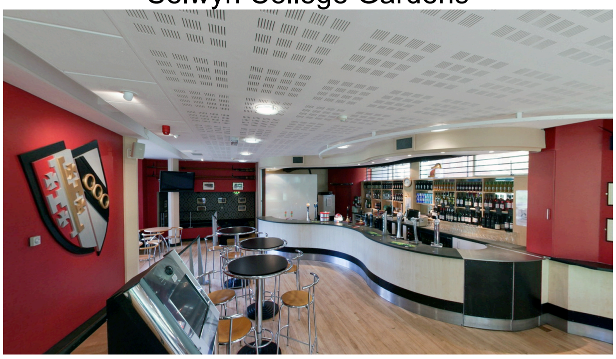
The first two Symposia in the CAMTEC series took place in April 2006 and March 2010, in Downing College, Cambridge. CAMTEC III will be held in April 2014, again in Cambridge. This time, the venue will be the Materials Science Department, now housed in a purpose-built building (opened in 2013) on the West Cambridge site. There is ample lecturing and exhibition space, with adjacent catering facilities. The Symposium will be chaired by Bill Clyne and co-chaired by James Dean, Bill Clegg (all of the Gordon Laboratory, Cambridge, UK), Johann Michler, Ivo Utke and Jeff Wheeler (all of EMPA, Thun, Switzerland).

Entrance to Materials Science Department

Format of the Meeting

All presenters have been personally invited, and attendance is also by invitation only. There will be about 70 delegates, including about 20 representatives of organisations participating in the Industrial Exhibition. There will be 27 talks (all of 20 minutes duration, including a few minutes for questions), divided into 5 themed sessions spread over the two days. In addition, there will be 19 posters, constituting a prominent element of the Symposium. These will be on display throughout the meeting, in the area where refreshment breaks and lunches will be taken and where the Industrial Exhibition will be held. Overnight accommodation, and the Symposium Dinner, will be provided in nearby Selwyn College. There will be a session of short talks by the poster authors. Four poster prizes will be awarded, on the basis of delegate voting. These prizes will be presented at the end of the Symposium

Selwyn College Gardens



Selwyn Bar

The Oral Programme

Monday 7th April

Time	No.	Presenter	Affiliation	Title
09.00-12.30	**Registration / Setting up of Exhibition and Posters (Materials Science Department)**			
12.30-13.30	**Lunch (Materials Science Department)**			
13.30-15.10	Session 1: **Micropillar Compression and other Novel Testing Procedures** (Chair: **Johann Michler**)			
13.30-13.50	**1**	Etienne Barthel	CNRS (St.Gobain)	*Uniaxial Compression of Silica Pillars: the Plastic Regime*
13.50-14.10	**2**	Gerhard Dehm	MPI Düsseldorf	*In Situ Electron Microscopy and Micro-Laue Study of Plasticity in Miniaturized Cu Bicrystals*
14.10-14.30	**3**	Helena van Swygenhoven	PSI Villingen / EPFL	*In-situ Laue Diffraction during Microcompression*
14.30-14.50	**4**	Karsten Durst	TU Darmstadt	*Nanoforming Behaviour and Microstructural Evolution during Nanoimprinting of Ultrafine-grained and Nanocrystalline Metals*
14.50-15.10	**5**	Fabio Di Gioacchino	Cambridge U.	*Lattice Deformation Measurements in Small-scale Testing using Digital Image Correlation*
15.10-15.50	**Tea / Exhibition / Posters**			
15.50-17.30	Session 2: **Temperature- and Time-Dependent Behaviour** (Chair: **Bill Clyne**)			
15.50-16.10	**6**	David Armstrong	Oxford U.	*Micro-mechanical Testing of Ion Irradiation Damage at Elevated Temperatures*
16.10-16.30	**7**	James Dean	Cambridge U.	*Assessment of a Conventional Indentation Methodology to Obtain Creep Parameters*
16.30-16.50	**8**	Mathias Göken	Erlangen U.	*Nanoindentation Testing of the Time and Temperature Dependent Mechanical Properties of Nanomaterials*
16.50-17.10	**9**	Nigel Jennett	NPL	*Indentation Creep Measurement*
17.10-17.30	**10**	Jeff Wheeler	EMPA Thun	*High Temperature Micro-Plasticity of Covalent Solids*
17.40-18.20	**Oral Presentation of Posters -** 2 minutes per poster (Chair: **Jeff Wheeler**)			
18.20-19.00	**Walk to Selwyn College (Cash Bar open from 18.00)**			
19.00-19.30	**Reception - Sparkling Wine and Soft Drinks (Old SCR in Selwyn College)**			
19.30-22.00	**Symposium Dinner (Hall in Selwyn College)**			
	Cash Bar open in Selwyn College until 23.00			

Tuesday 8th April

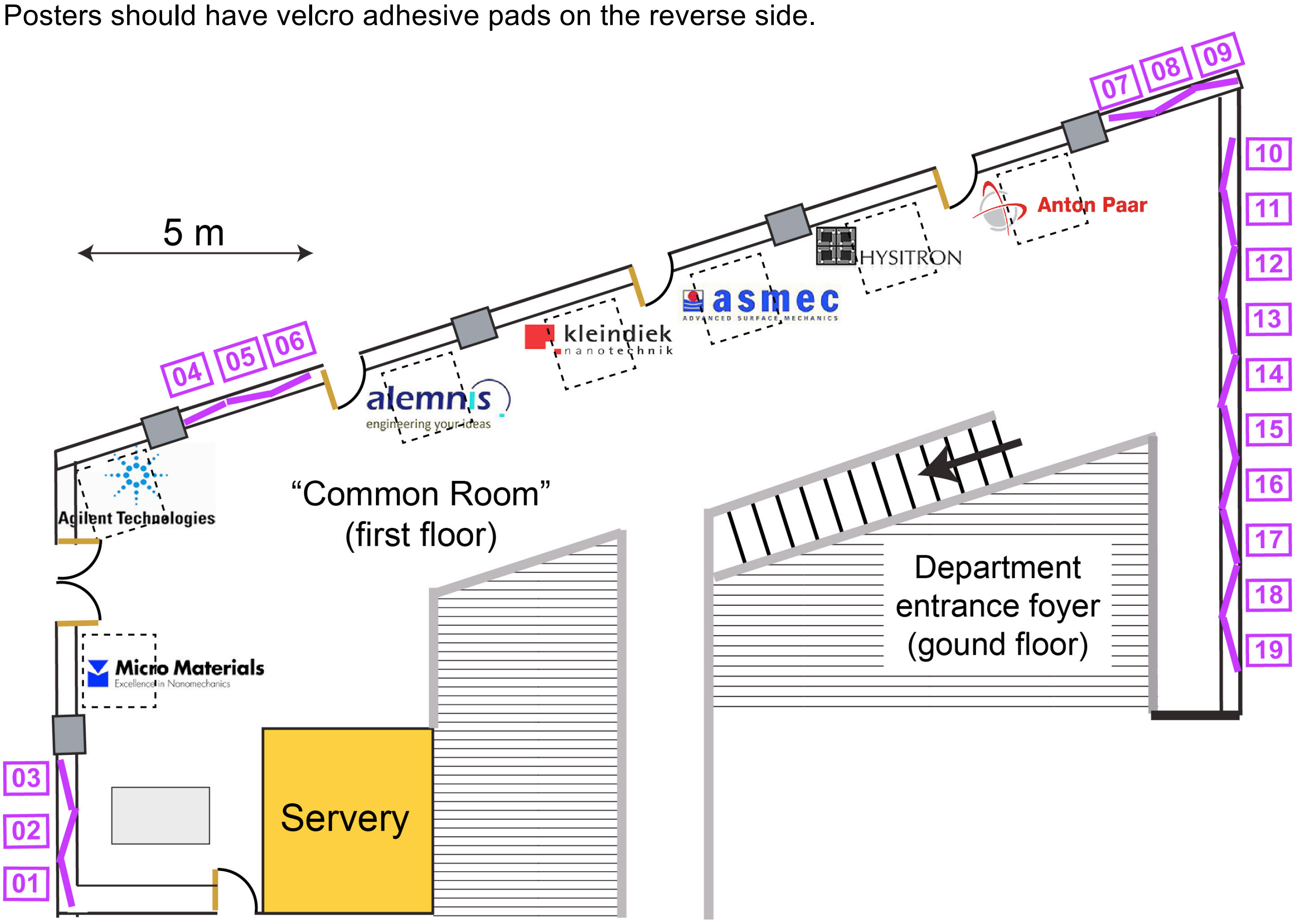
Time	No.	Presenter	Affiliation	Title
07.30-09.00		**Breakfast (Selwyn College) & walk to Materials Science Department**		
09.00-11.00		Session 3: **Deformation and Fracture Mechanisms** (Chair: **Ivo Utke**)		
09.00-09.20	**11**	Finn Giuliani	Imperial Coll.	*In Situ Deformation of Metallic Interlayers*
09.20-09.40	**12**	Sandra Korte-Kerzel	Aachen U.	*Deformation Mechanisms in the Intermetallic $Mg_{17}Al_{12}$ Reinforcement Phase in Magnesium at 25 - 300°C*
09.40-10.00	**13**	Oliver Kraft	Karlsruhe U.	*Fracture at Small Dimensions - from Nanowires to Hierarchical Materials*
10.00-10.20	**14**	Michelle Oyen	Cambridge U.	*Distinguishing Viscoelastic and Poroelastic Deformation Mechanisms*
10.20-10.40	**15**	Christophe Tromas	Poitiers U.	*Influence of Pre-Existing Dislocations on the Pop-in Phenomenon in MgO*
10.40-11.00	**16**	Gerold Schneider	Hamburg U.	*What can we learn from nature-made nanostructured hierarchical ceramic like composites?*
11.00-11.30		**Coffee / Exhibition / Voting for Poster Prizes**		
11.30-13.10		Session 4: **Extraction of Material Properties from Indentation Data** (Chair: **James Dean**)		
11.30-11.50	**17**	Russell Goodall	Sheffield U.	*Indentation Testing in High Throughput Alloy Development*
11.50-12.10	**18**	Guillaume Kermouche	ENSM St.Etienne	*About Direct Measurements of Stress-strain Curves from Instrumented Indentation and Repeated Micro-impact Results*
12.10-12.30	**19**	Jin Chong Tan	Oxford U.	*Fine-scale Mechanical Properties of Metal-organic Framework Thin Films and Single Crystals*
12.30-12.50	**20**	Guenhael Le Quilliec	Tours U.	*A Reverse Method Based Approach for Evaluating the Local Tensile Curves of Metallic Materials from Instrumented Indentation*
12.50-13.10	**21**	Ehrenfried Zschech	IZFP Dresden	*Pore Topology Effect on Mechanical Properties of Porous Organosilicate Glasses – A Nanoindentation and PALS study*
13.10-14.00		**Lunch (Materials Science Department)**		
14.00-16.00		Session 5: **Coatings, Multilayers & Interfaces** (Chair: **Bill Clegg**)		
14.00-14.20	**22**	Reinhold Dauskardt	Stanford U.	*Nanoscale Multilayers in Nanoscience and Bioscience Technologies*
14.20-14.40	**23**	Steve Bull	Newcastle U.	*Mechanical Design of Organic Light Emitting Diodes on Polymer Substrates*
14.40-15.00	**24**	Megan Cordill	Leoben U.	*Interface Characterization of Printed Circuit Boards using Four Point Bending and Nanoindentation*
15.00-15.20	**25**	Jon Molina	IMDEA Madrid	*High Temperature Mechanical Behaviour of Nanoscale Multilayers*
15.00-15.20	**26**	Richard Todd	Oxford U.	*Measurement of Interfacial Properties in Ceramics by Microcantilever Testing*
15.40-16.00	**27**	Ivo Utke	EMPA Thun	*Mechanical Characterization of ZnO/Al_2O_3 Multilayer Oxide Films prepared by Atomic Layer Deposition*
16.00-17.00		**Tea / Exhibition / Presentation of Poster Prizes / Group Photograph**		
17.00-18.00		**Transport to Rail Station**		
18.00-19.00		**Evening Meal (Selwyn College - Cafeteria service)**		

The Poster Programme

No	Presenter	Affiliation	Title
Micromechanical Testing of Soft and Biological Materials			
1	Riaz Akhtar	Liverpool U.	*Nanomechanical and Ultrastructural Variations in the Sclera*
2	Elmar Kroner	INM Saarbrücken	*Contact Experiments on Bioinspired Adhesives*
Micropillar Compression & other Novel Testing Procedures			
3	Jens Bauer	Karlsruhe U.	*Push-to-pull Tensile Testing of 3D Micro-architectures*
4	Ben Beake	Micromaterials	*Extreme Nanomechanics: Vacuum Nanoindentation to 1000˚C*
Temperature & Time Dependent Behaviour			
5	Gaurav Mohanty	EMPA Thun	*Strain Rate Jump, Load Relaxation and Creep tests on Nanocrystalline Ni Micropillars as a Function of Temperature*
Deformation and Fracture Mechanisms			
6	Philip Howie	Cambridge U.	*Deformation in B2 Intermetallics*
7	Andy Bushby	QMUL	*On the Size-Dependent Strength of Nano-structured Materials*
8	Claire Davis	Cambridge U.	*Plastic Flow in Laves phases*
9	David Mercier	MPI Düsseldorf	*Identification of the Critical Resolved Shear Stress Ratios of the Deformation Systems and Investigation of Slip Transfer in Ti-5Al-2.5Sn alloy*
10	Edmund Tarleton	Oxford U.	*Modelling the Size Effect in Micro-cantilever Beams*
11	Luc Vandeperre	Imperial Coll.	*Drafting Deformation Mechanism Maps from Temperature Dependent Indentation Data*
12	Claudio Zambaldi	MPI Düsseldorf	*Quantifying Slip and Hardening Properties of Crystals by Indentation*
13	Bill Clegg	Cambridge U.	*Deformation in Fine-grained CrN-based Coatings*
14	John Colligon	Manchester Met. U.	*Durability of MAX-phase Thin Films: Nano-impact and Elevated Temperature Nano-friction*
Extraction of Material Properties from Indentation Data			
15	Marco Sebastiani	Roma TRE	*A Novel Nanoindentation Method for the Analysis of Surface Free Energy on Micro-areas: Application to Patterned Surfaces and Micro-particles*
16	Noushin Moharrami	Newcastle U.	*Problems of Extracting Mechanical Properties of Porous Coatings using Nanoindentation*
17	Bill Clyne	Cambridge U.	*Software for the Extraction of Material Properties from Indentation Data (SEMPID)*
Coatings, Multilayers & Interfaces			
18	Joe Reed	Cambridge U.	*Indentation of Bilayer Systems with Finite Top Layer Thickness*
19	Johann Michler	EMPA Thun	*Mechanical Properties of Multilayer Thin Films: an Elevated Temperature Microcompresison Study*

Poster Display and Industrial Exhibition

The poster display and industrial exhibition will be in the common room area of the Department, which is immediately above Goldsmiths 1 (accessible via a short stairway), where the oral presentations will take place. (There is also lift access to the first floor level.) The common room is also the area where refreshments and lunches will be served. Seating will be available, including some on the outside terrace, which will be accessible if the weather is suitable. The layout of the posters and exhibition stands is shown below, with a nominal 2 m square (dotted areas) allocated to each Exhibitor. The individual poster boards (ie the velcro space within the borders) will measure 90 cm (horizontally) by 116 cm (vertically). They will therefore be suited to posters in portrait orientation. Posters should have velcro adhesive pads on the reverse side.



Poster prizes will be awarded, on the basis of delegate voting. Four prizes will be available, in the form of vouchers having values of £400, £300, £200 & £100. All delegates will be supplied with 5 coloured plastic disks, with the following values for voting: white - 5 points, yellow - 4 points, green - 3 points, blue - 2 points, red - 1 point. Votes will be cast by placing a disk in an envelope at the poster concerned. Delegates cannot vote for a poster for which they are an author or co-author. Posters will be ranked according to the total number of points allocated, and prizes presented to the first 4 in the list. Votes can be cast at any time ***up until 11.30am on Tuesday***.

Symposium Dinner

The dinner will be held in Selwyn College at 19.30, with a Reception beforehand at 19.00. Dress will be informal - lounge suit, with or without tie. The menu will be as follows:
Onion Soup / Forerib of Beef, Potatoes & Vegetables / Lemon & Almond Cake, Cream & Raspberries
There will be a vegetarian alternative for the main course of Stuffed Aubergines
Attendees with dietary requirements should ensure that these have been recorded via registration on the website. Delegates are welcome to bring partners to the dinner, in which case they should ensure that the organisers are aware of the arrangement. There will be no seating plan for the dinner.

Internet Access

Free Wifi access will be available to all delegates within the Materials Science Department. Individual passwords will be supplied in the welcome pack.

Attending Delegates

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Abstracts – Oral Presentations

Monday 7th April, 13.30

Uniaxial Compression of Silica Pillars: the Plastic Regime

E. Barthel, R. Lacroix, G. Kermouche & J. Teisseire {CNRS (St.Gobain)}

It has been known for quite a long time that silicate glasses experience plastic deformation at the micron scale. However, the micromechanics tools for a quantitative characterization of plastic deformation at the micron scale have become available only recently. Combining micro-Raman spectroscopy and microindentation, we have derived a constitutive equation for the plastic deformation of silica including strain hardening through densification [1]. Recently, we have developed micro-pillar experiments to probe a reasonably well defined stress state with significant shear. With optimum design of the experiment irreversible deformations of up to 30% were reached [2]. We show that the results completely support the constitutive relation we proposed earlier [1]. Our results also suggest that silica exceeds crystalline oxides in terms of yield stress over shear modulus ratio, with no sign of size effect. With strong plastic deformation, well defined rupture patterns are observed, which suggest ductile rupture at small scales and will be discussed in relation to the remarkable tensile strength of amorphous silica.

[1] G. Kermouche, E. Barthel, D. Vandembroucq and P. Dubujet: *Mechanical Modeling of Indentation-induced Densification of Silica*, Acta Materialia **56** (2008) p.3222-3228.

[2] R. Lacroix, G. Kermouche, J. Teisseire and E. Barthel, *Plastic Deformation and Residual Stresses in Amorphous Silica Pillars under Uniaxial Loading,* Acta Materialia **60** (2012) 5555-5566.

Monday 7th April, 13.50

In Situ Electron Microscopy and Micro-Laue Study of Plasticity in Miniaturized Cu Bicrystals

G. Dehm, P. Imrich, C. Kirchlechner, C. Motz & JB Jeon {MPI Düsseldorf}

Grain boundaries are well known to enhance the strength of polycrystalline metals as they act as obstacles for dislocations. However, the blocking strength of individual grain boundaries may be very different depending on the orientation of grain boundary plane and the orientation of the slip systems in the adjacent grains. In addition some grain boundaries act as sinks, while others are sources of dislocations. In this talk we compare as a model system the deformation behavior of a general large angle grain boundary and a coherent twin boundary in a compression experiment of Cu micropillars. For comparison, the flow behavior of the adjacent single crystalline Cu micropillars is analyzed. In situ SEM, TEM and micro-Laue studies as well as DDD and MD simulations are performed to shed light on the dislocation processes. The huge differences in mechanical behavior of the bicrystalline pillars are discussed based on the experimental results.

Monday 7th April, 14.10

In-situ Laue Diffraction during Microcompression

H. Van Swygenhoven, C. Marichal, A. Irastorza, S. Van Petegem & C. Borca
{PSI Villigen & EPFL}

Micro-compression experiments are performed during in-situ Laue diffraction at the MicroXAS beamline of the Swiss Light Source to explore the sequence of activated slip systems in bcc single crystals. Diffraction patterns are obtained in transmission with a 5-23 keV X-ray beam with FWHM of 0.5-1 µm. Laue scans allow the mapping of the spatial distribution of strain gradients in the deformed pillars, providing information on local crystallographic orientations and on the activated dislocation slip systems. Additional examination by scanning electron microscopy allows identification of slip traces on the surface. Slip in W occurs on {110} planes even when slip traces are observed on {112} planes. For some orientations anomalous slip is observed. Slip in W is compared with slip in Mo, Nb and W6%Re, the character (sharp or wavy) of slip traces and the activated slip systems are discussed.

Monday 7th April, 14.30

Nanoforming Behaviour and Microstructural Evolution during Nanoimprinting of Ultrafine-grained and Nanocrystalline Metals

K. Durst {TU Darmstadt}

In this work, nanoindentation with a specially designed tip has been used as a tool for studying the local plastic flow of metals with varying grain sizes in a sub-mircon cavity during a nanoimprinting. Of main interest in this work was the flow of crystalline materials in submicron sized cavities during imprinting. The ring cavities, which have widths of 650 nm and 80 nm, were fabricated by focused ion beam (FIB) machining. Atomic force microscopy (AFM) and scanning electron microscopy (SEM) were used to evaluate the imprinted ring geometries. The microstructure after imprinting was investigated in detail by FIB cross-sections and electron back scatter diffraction (EBSD), as well as by using finite element analysis (FEA) of the forming process. SX-Ni showed the smallest extrusion height together with a sinking-in of the formed region. This is accompanied by strong orientation gradients up to 1.8 ° nm^{-1} below the cavities. The UFG samples exhibited the best formability, with a subgrain formation inside and around the cavities. The plastic flow is confined to the surface and a pile-up formation occurs. For the nanocrystalline material only a slight elongation of the grains inside the cavity was found, yielding moreover a smooth and homogeneous extruded geometry. These findings can be explained by the grain size / cavity width ratio as well as the yield strength and the work hardening behaviour of the materials.

Monday 7th April, 14.50

Lattice Deformation Measurements in Small-scale Testing using Digital Image Correlation

F. Di Gioacchino & WJ Clegg {Cambridge U.}

Diffraction techniques, such as electron backscatter diffraction and Laue X-ray diffraction, are often used to measure the lattice curvature resulting from any strain gradients produced by deformation. However, different strain gradients can give rise to the same lattice curvature and density of geometrically necessary dislocations. Here, a method for mapping deformation uniquely using high-resolution digital image correlation (DIC) is described for different small-scale specimen geometries.

Monday 7th April, 15.50

Micro-mechanical Testing of Ion Irradiation Damage at Elevated Temperatures

D. Armstrong, J. Gibson & S.G. Roberts {Oxford U.}

Understanding post-irradiation changes in mechanical properties is key in the development of advanced materials for nuclear fusion reactors, which will be subjected to higher levels of radiation damage than fission reactors (up to 100 displacements per atom as opposed 1-10 dpa). Ion irradiation can be completed more cheaply and rapidly than neutron irradiation, accelerating material development. Work has been carried out on the nano-mechanical properties of tungsten implanted with helium ions. Tests were carried out from 21˚C to 750˚C and strain rates from 0.001 s^{-1} to 0.1 s^{-1}, using a high temperature vacuum nanoindenter. The hardness of pure tungsten decreases strongly with increasing temperature, from ~6 GPa at 50˚C to ~3 GPa at 250˚C, after which it remains constant. He^+ implantation to levels of 600 appm produces an increase in hardness of ~4 GPa at 50˚C, which decreases to ~2 GPa by 750˚C. This decrease in hardness with temperature is thought to be due to the increased mobility of dislocations to bypass helium-vacancy clusters, which cause the hardening effect. Ion irradiation of tungsten alloys with He^+ and W^+ ions produces damaged layers ranging in depth from 200 nm to 3000 nm. The changes in mechanical behavior in these damaged layers have been measured by microcantilever bending. Simultaneous He^+ and W^+ irradiation has the most drastic effect, causing severe embrittlement, but it has been found that the effects of irradiation can be reduced by using nanostructured alloys.

Monday 7th April, 16.10

Limitations in a Common Methodology for the Extraction of Steady-State Creep Parameters during Indentation

J. Dean, J. Campbell & T.W. Clyne {Cambridge U.}

A technique for finding the steady-state creep stress exponent, *n*, from indentation data has become established in the indentation community. The method assumes i) that a steady-state exists beneath the indenter and ii) that spatial and temporal variations in stress, strain and creep strain rate can be defined with equivalent values. Under these assumptions, the gradient of the displacement-time curve during the indentation dwell period can be used to calculate *n*. The validity of this method is examined here by comparing indentation-derived values of *n* with those obtained using conventional creep testing methods (i.e. from compression specimens loaded uniaxially under constant stress). An indentation finite element model is also used to more rigorously explore the conditions of stress, strain and creep strain rate that prevail during indentation, to determine whether or not the assumptions are suitable, and to understand more clearly the effect of primary creep on the measured behaviour.

Monday 7th April, 16.30

Time and Temperature Dependent Mechanical Behaviour of Nanomaterials and Thin Films as tested with Nanoindentations and Bulge-Tests

M. Göken {Erlangen U.}

Studying the time and temperature deformation behavior of nanomaterials and thin films on the small scale is of utmost importance to understand the deformation mechanism of these materials. In the presentation, different approaches to measure such influences will be discussed and compared. Nanoindentations strain rate jump tests have been developed, which allow reliably measuring the strain rate sensitivity of ultrafine grained and nanocrystalline materials, which is generally enhanced and important to understand the ductility of these materials. In addition also bulge testing of thin films has been used to study such influences of very thin free-standing gold membranes. Strain-rate jump tests were implemented in a bulge test experiment to allow quantitative measurements of the strain-rate sensitivity of freestanding gold films, which showed a 5 times higher rate sensitivity than films bonded to a SiN_x substrate. This higher time-dependence was confirmed by creep tests. Subsequent observations of the surface of the freestanding films revealed a strong roughening, corresponding to the out-of-plane sliding of grains along at least one of their grain boundaries. The strain-rate sensitivity of ultrafine-grained aluminum (Al) and nanocrystalline nickel (Ni) has also been studied with an improved nanoindentation creep method. Using the dynamic contact stiffness, thermal drift influences can be minimized and reliable creep data can be obtained from nanoindentation creep experiments even at enhanced temperatures and creep segments of up to 10 h. The indentation creep behavior resembles a power-law behavior with stress exponents n being around 20 at room temperature. For higher temperatures, the stress exponents of ufg-Al and nc-Ni decrease down to around 5. These locally determined stress exponents are similar to the macroscopic exponents, indicating that similar deformation mechanisms are acting during indentation and macroscopic deformation.

Monday 7th April, 16.50

Indentation Creep Measurement

N. Jennett & X. Hou {NPL}

Quasi-static instrumented indentation is well established and has been standardised since 2002 in BS EN ISO14577. This standard requires that static property measurements are obtained by means that minimise the effect of creep on the result. Creep is, however, very prevalent in materials, even metals at room temperature, and more so as temperatures are increased. In particular, the drive for light-weighting of transport is requiring the structural use of materials that creep more (such as Al and polymer based composites). In such components, creep often becomes the lifetime limiting parameter and strategies are being sought to limit this, such as the use of nanoparticle/clay additives to modify the viscosity of polymers. Indentation is increasingly being used as a creep measurement method. ISO14577, however, only contains a very simplified method for assigning a value to the creep of a material. We report work from The European Metrology research Programme projects “MeProVisc” and “T3D”, and describe better methods for measuring the creep, viscosity and visco-elastic properties of materials, and the foundation for valid indentation measurements at elevated temperatures. We also report probably the longest stable indentation creep measurements ever made, obtained using the NPL indentation femto-creep facility. We have measured the indentation creep rate of a 9 mN Berkovich/CVD diamond contact for over 300 ks. We found it to be stable and linear after ~100 ks and recorded a constant 20 fm s^{-1} displacement rate over the next 238.2 ks (a total of only 4.9 nm).

Monday 7th April, 17.10

High Temperature Micro-Plasticity of Covalent Solids

J.M. Wheeler & J. Michler {EMPA Thun}

At ambient temperature and pressure, most of the semiconductor materials are brittle: this is the case of the III-V compound semiconductor indium antimonide (InSb). Traditionally, use of confining pressure via indentation or a hydrostatic confining medium has been required to study the plasticity of such brittle materials. However, previous work has demonstrated that sample miniaturization can prevent the onset of cracking and allow plastic deformation. Recent advances in *in situ* instrumentation have enabled micro-compression techniques to extract temperature- and time-dependent deformation parameters. Due to its well-characterized ductile to brittle transition with temperature for bulk deformation, InSb is a model system for determining whether thermally activated deformation mechanisms at the micro scale correspond to macro scale behaviour. Here, strain rate jump micro-compressions at elevated temperature have been performed *in situ* in the SEM to measure and observe the deformation of InSb and other III-V semiconductors at the micro scale above and below the transformation temperature.

Tuesday 8th April, 09.00

In Situ Deformation of Metallic Interlayers

F. Giuliani {Imperial Coll.}

There are many interesting mechanical properties that can be achieved with metal/ceramic multilayers, especially if they are defined more broadly to include structures such as MAX phases. However, the deformation mechanisms, such as hysteresis, can be complex. To simplify the problem, in this work we have concentrated on the effect of a single metal interlayer within a ceramic bi-crystal. The samples were produced by sputtering niobium on to sapphire substrates and then diffusion bonding the two coated crystals together. This allowed the production of sapphire micropillars containing a ~50-200 nm niobium layer. These were then loaded *in situ* within a microbeam Laue set up and *in situ* within an SEM. By varying the layer thickness, the measured strength of the pillar could be varied, along with the magnitude of the hysteresis. This hysteresis was tracked by the load-displacement trace, along with the movement and elongation of diffraction spots associated with the interlayer.

Tuesday 8th April, 09.20

Deformation Mechanisms in the Intermetallic $Mg_{17}Al_{12}$ Reinforcement Phase in Magnesium at 25 – 300˚C

S. Korte-Kertzl & H. Mathur {Aachen U.}

Microstructures reinforced by complex intermetallic phases can display superior mechanical properties, such as an improved creep resistance, making them attractive for demanding structural applications in the automobile and aerospace industries. Much of the work in understanding the beneficial, or often detrimental, effects of intermetallic phases on the deformation mechanisms has been done either by post-deformation analyses of alloys or by studying single phase intermetallics. However, due to their brittleness, much of the work on pure intermetallics is confined to temperatures much higher than the operating temperatures of the alloy. In order to investigate the plasticity of the reinforcing phase at the relevant temperature from room to operating temperature and the cooperative deformation of an intermetallic skeleton and the matrix, nanomechanical testing can be used in conjunction with electron microscopy. In this study, $Mg_{17}Al_{12}$ was investigated, which represents one of the major intermetallic phases found in magnesium alloys containing aluminium. By means of high temperature nanoindentation, atomic force microscopy and transmission electron microscopy, the hardness and changes in deformation and dislocation structure have been studied. A transition in deformation mechanism and more rapid decrease in hardness with temperature appears near the critical temperature at which the alloys exhibit a loss of creep strength. In order to allow a meaningful correlation of the properties of the intermetallic phase and the alloy in which it is used, some initial in-situ deformation experiments at room temperature have also been conducted. These allow the observation of the initial stages of plasticity and transmission of deformation from the soft magnesium matrix into the intermetallic.

Tuesday 8th April, 09.40

Fracture at Small Dimensions - from Nanowires to Hierarchical Materials

O. Kraft, J. Bauer, C. Ensslen, S. Boles, R. Schwaiger, H. Riesch-Oppermann & R. Manig {Karlsruhe U.}

Size effects at length scales below 1 μm have been the subject of many studies in past decades. These studies have shown that, with a reduction in size, strength increases, often accompanied by a decrease in toughness. This is also seen in more recent investigations on the deformation behavior of nanowires made from different materials. In this talk, recent results from the nanomechanical testing of Si nanowires, with diameters from 30 to 500 nm will be presented. It will be shown that the strength of such nominally defect-free nanowires may reach the regime of theoretical strength. Nevertheless, one feature of these experiments is a strong scatter in the measured strengths, which cannot be attributed to uncertainty in the measurements. Therefore, it can be argued that the strength of nano-scale structures is of a statistical nature, which can be described by applying Weibull statistics. Moreover, we present an approach to take advantage of the mechanical size effects by designing hierarchical materials with nanometre-size building blocks. These materials are manufactured by using 3D laser lithography, with the goal of combining high strength with low density. Polymeric templates are patterned and coated with ceramic films by atomic layer deposition. The structures are then characterized with respect to their deformation behavior. The specific strength of these artificial cellular materials exceeds the ones of all natural and engineering foams with a density below 1 g cm^{-3}.

Tuesday 8th April, 10.00

Distinguishing Viscoelastic and Poroelastic Deformation Mechanisms

M. Oyen {Cambridge U.}

Hydrogels have applications in drug delivery, mechanical actuation, and regenerative medicine When hydrogels are deformed, load-relaxation arising from fluid flow/poroelasticity and from rearrangement of the polymer network/viscoelasticity is observed. The physical mechanisms are different, in that poroelastic relaxation varies with experimental length-scale, while viscoelastic does not. Here, we show that poro-viscoelastic load-relaxation is the product of the two individual responses. The difference in length-scale dependence of the two mechanisms can be exploited to uniquely determine poro-viscoelastic properties from simultaneous analysis of multi-scale indentation experiments, providing insight into hydrogel physical behavior.

Tuesday 8th April, 10.20

Influence of Pre-Existing Dislocations on the Pop-in Phenomenon in MgO

C. Tromas, A. Montagne & V. Audurier {Poitiers U.}

In magnesium oxide (MgO), as in many other materials, the first stage of plastic deformation during a nanoindentation test is characterized by a strain burst or "pop-in", which is associated with a sudden nucleation of dislocations. In this study, the question of the nature of this nucleation event, homogeneous or heterogeneous, is addressed. In particular, the role played by preexisting dislocations is studied. For characterizing the dislocation structures associated to the pop-in phenomenon, original methods, such as slip line analysis, but also nano-etching, have been developed. This last method consists of revealing the emergence points of the dislocations by nanometre size etch pits, observed by AFM. To investigate the effect of pre-existing dislocations, a controlled density of dislocations has been introduced by cleavage prior to indentation. A double etching technique has then been used to reveal and identify both of the preexisting dislocations and those nucleated during the pop-in.

Tuesday 8th April, 10.40

What can we learn from Nature-made Nanostructured Hierarchical Ceramic-like Composites?

G. Schneider {Hamburg U.}

Enamel is chosen as a prototype material for a nature-made hierarchical composite and its mechanical properties are presented from the nano- to the macro-scale. The experimental methods applied include nanoindentation, micro- and macro-beam tests as well as micro- and macro-pillar tests. It turns out that, with increasing length scale, the elastic modulus, hardness and strength are decreasing, whereas the toughness and damage tolerance are increasing. The reasons for these length scale effects are discussed in comparison to engineering ceramics. Based on these considerations, we started to develop self-assembled nanoparticle composites. First results of their microstructures and mechanical properties are presented.

Tuesday 8th April, 11.30

Indentation Testing in High Throughput Alloy Development

R. Goodall & P. Mahoney {Sheffield U.}

Indentation is a highly effective method for probing the mechanical properties of small volumes of material, and is therefore highly suited to rapid screening tests in the development of new materials, where sample volume is frequently limited. This presentation will provide an account of some experiences of the use of indentation testing at micro- and nano-scale in *Accelerated Metallurgy*, a European project aiming to increase the speed of development of new alloys by the use of a combinatorial approach. This includes the use of indentation in a high throughput mode, applied to metallic specimens produced via a combinatorial approach, in order to determine more well-understood properties such as hardness and modulus, and the further exploration of the use of indentation to investigate a wider range of properties, including creep and fracture toughness. A number of case studies will also be presented of indentation investigations of the mechanical properties of new alloy systems and advanced metallic materials, including Bulk Metallic Glasses.

Tuesday 8th April, 11.50

About Direct Measurements of Stress-strain Curves from Instrumented Indentation and Repeated Micro-impact

G. Kermouche, C. Langlade & J.M. Bergheau {ENSM St.Etienne}

Since the pioneering work of Tabor in the 1950s, the measure of the stress-strain curves of materials from indentation measurements is one the main source of scientific papers dealing with indentation. One may distinguish two types of methods: the first type, called direct methods, try to use analytical relations relating the indentation data - such as the Hardness, the viscoplasticity index etc - to the indentation representative stress and strain. The second are based on the use of reverse finite element analyses to fit the indentation data. These are clearly more precise, but they require a very accurate description of the indented material mechanical behaviour and of the test conditions, to be efficient enough. In this talk, we will discuss direct methods and, more specifically, the use of analytical solutions we developed some years ago. A simple method will be proposed to solve the crucial problem of the contact area determination. Application to different kinds of materials will be shown. In the last part, we will present an extension of this work to identify the dynamic stress-strain curve using repeated micro impact testing.

Tuesday 8th April, 12.10

Fine-scale Mechanical Properties of Metal-organic Framework Thin Films and Single Crystals

J.C. Tan, B. Van de Voorde, R. Ameloot & D. De Vos {Oxford U.}

We have investigated the mechanical properties of metal-organic framework (MOF) [1] thin-film coatings grown by means of a novel electrochemical method, which allows fast deposition in environmentally friendly solvents. For the first time, 3 unique variants of copper-based MOF coatings have been electrochemically synthesised [2]. To interrogate the mechanical characteristics of these coatings (~1 μm thickness), both nanoindentation and nanoscratch experiments have been carried out. The indentation of polycrystalline thin films enables us to establish the (average) Young's moduli and hardness behaviour of the coatings, which are then corroborated with single-crystal elasticity measurements [3]. Analysis of the crystal lattice of the MOF materials obtained from X-ray diffraction provides further insights into structure-property correlations at the molecular level [1]. Effects of the interfacial adhesion properties of MOF thin films on their scratch and wear resistance has also been studied.

[1] J.C. Tan & A.K. Cheetham, *Mechanical Properties of Hybrid Inorganic-Organic Framework Materials: Establishing Fundamental Structure-Property Relationships*, Chem. Soc. Rev. **40** (2011) p.1059-1080.

[2] B. Van de Voorde, R. Ameloot, I. Stassen, M. Everaert, D. De Vos, and J.C. Tan, *Mechanical Properties of Electrochemically Synthesised Metal–Organic Framework Thin Films*, J. Mater. Chem. B, **1** (2013) p.7716-7724.

[3] J.C. Tan, B. Civalleri, C.C. Lin , Valenzano, L., Galvelis, R., Chen, P.F., Bennett, T.D., Mellot-Draznieks, C., Zicovich-Wilson, C.M. & Cheetham, A.K., *Exceptionally Low Shear Modulus in a Prototypical Imidazole-Based Metal-Organic Framework*, Phys. Rev. Lett. **108** (2012) art. No. 095502.

Tuesday 8th April, 12.30

A Reverse Method Based Approach for Evaluating the Local Tensile Curves of Metallic Materials from Instrumented Indentation

G. Le Quilliec, G. Mauvoisin, G. Inglebert, M. Drissi-Habti, H-P. Lieurade
P. Macquet & L. Jubin {Tours U.}

In this presentation, we propose an approach to estimate the local stress-strain characteristics of metallic materials from instrumented indentation data. This approach is based on a reverse method, which implies to first perform a set of numerical indentations. Contrary to inverse methods, the generated analytical reduced model can then be used to instantaneously evaluate the desired local properties from experimental instrumented indentation curves. In order to improve the estimation accuracy, it is able to take into account the elastic deformations of the indenter occurring during the test. It is also able to take into account the defect in shape of the actual indenter. This proposed general approach has been applied in the particular case of linear hardening behaviour. This approach was then successfully validated by comparison with experimental tensile test results. Finally it has been used to estimate the local tensile curves in various projects, such as the local characterisation of welded joints to be treated by high frequency hammer peening.

Tuesday 8th April, 12.50

Pore Topology Effect on Mechanical Properties of Porous Organosilicate Glasses – A Nanoindentation and PALS study

E. Zschech, A. Clausner, M. Kraatz, M. Gall, M. Butterling, R. Krause-Rehberg & S. Mahajan
{IZFP Dresden}

Managing chip-package interaction (CPI) is a key effect to consider for high performance and reliability of leading-edge microelectronic products. Weak mechanical properties, e.g. low Young's modulus and fracture toughness, of porous organosilicate glasses (OSG) used as insulating materials between on-chip interconnects are serious reliability concerns. Therefore, new types of self-assembled organosilicate glass (SA-OGS) films were developed based on sol-gel templating and spin-on deposition. Significantly improved mechanical properties were achieved for these OSG materials by tuning the sol-gel chemistry to form (a) organic bridges between network silicon atoms, (b) polymer chains across interfaces, and (c) periodic pore arrangement. Based on previous studies, depending on silica, surfactant and solvent compositions, domains with different pore arrangements – cubic, hexagonal or disordered pores – were found in SA-OSG films. In this talk, we will demonstrate the relationships between porosity, pore topology and elastic modulus based on experimental studies at several OSG films, supported by Monte Carlo simulation. Nanoindentation (NI) data demonstrate that the elastic modulus of SA-OGS films is significantly higher than that for chemical-vapor-deposited CVD-OSG films with with the same chemical composition of the skeleton and the same porosity, but with a broad pore size distribution. Films with a wide range of *k*-values (about 3.0 to 1.8) and porosity (0 to ~50%) were characterized using NI and positron annihilation lifetime spectroscopy (PALS). The pore size is well-controlled to about (3.5 ± 0.5) nm, slightly increasing for films with lowest *k*-value (1.8). The existence of a strong and dense sealing layer (< 20 nm) was identified which increases the robustness of the self-assembled, mechanically strong thin films.

Tuesday 8th April, 14.00

Nanoscale Multilayers in Nanoscience and Bioscience Technologies

R. Dauskardt {Stanford U.}

Reliability integrating new multi-functional films in emerging nanoscience, energy and bio technologies requires a new understanding of their adhesion and mechanical behavior. We describe our research by selecting several examples involving nanoscale multilayers with application in a number of such emerging technologies. Specifically, we discuss thin-film micromechanical testing metrologies and the relationship between composition, molecular design, and film structure on resulting mechanical and fracture behavior. We consider the effects of environmentally assisted fracture in moist and chemically active environments together with the behavior of multilayers in photovoltaic devices operating in solar UV environments. Finally, we consider the multilayer structure of the top stratum corneum layer of human skin and show how its biomechanical function can be influenced by exposures and treatments to reduce skin damage.

Tuesday 8th April, 14.20

Mechanical Design of Organic Light Emitting Diodes on Polymer Substrates

S. Bull {Newcastle U.}

In many current applications coatings are being developed which do not exist in bulk form and cannot be examined by conventional mechanical tests. This raises problems in the mechanical design of devices based on such materials, as no property data are available to include in design calculations. One particular example is the semiconducting layers in organic light emitting diodes (OLEDs), which may consist of evaporated films of organic molecules. The elastic properties of these coatings are often essential for design of devices, particularly if they are deposited on compliant substrates which allow bending during manufacture or service (e.g. in flexible electronics). Whereas it is possible to make good measurements of elastic properties on stiff substrates such as silicon there are serious issues with the reliability of data from coatings on compliant substrates such as the PET used for plastic electronics. This presentation will outline the development of a simple analytic model of the extraction of the contact modulus of a coating from nanoindentation data obtained from a coating/substrate system and analyse the reliability of the data produced. The data will then be used to optimise the design of a multilayer OLED on a compliant substrate subject to bending through the development of an analytic bending model.

Tuesday 8th April, 14.40

Interface Characterization of Printed Circuit Boards using Four Point Bending and Nanoindentation

M. Cordill & R. Schöngrundner {Leoben U.}

Adhesion of copper foils to pre-preg layers in printed circuit boards (PCB) is important in order to manufacture reliable products. During fabrication, layers of Cu foils and Pre-preg are subjected to different mechanical and thermal loads. It is critical to understand the Cu-Pre-preg interface because it is prone to delamination during the fabrication and testing processes. In order to measure the interfacial strength of the Cu-Pre-preg interface, four-point-bending was implemented with a specially designed sample geometry that isolates the Cu-pre-preg interface so that it would be in the middle of the bending beam. The special geometry also reduces the bending flexibility to aid the extension of the interface crack. With four-point-bending, the adhesion energy as a function of pre-preg type, copper foil treatment and reflow heating cycles can be studied. Additionally, nanoindentation measurements across the Cu/pre-preg interface have been made to characterize the interface properties as a function of the reflow cycles. It has been found that the number of reflow cycles has the largest influence on the adhesion energy, decreasing as the number of reflow cycles increases. With this new knowledge, the reliability of the PCB can be improved and the lifetime of the products extended.

Tuesday 8th April, 15.00

High Temperature Mechanical Behaviour of Nanoscale Multilayers

J. Molina {IMDEA}

Nanoscale multilayers, made up of alternating layers of two materials with layer thickness in the nm range, have been the subject of an increasing number of studies in the past 10 years due to their exceptional high strength at room temperature. Their unique properties are mainly a result of the high density of interfaces, which change the standard mechanisms of plastic deformation and fracture, when the layer thickness is below ~100 nm. However, little is known about their high temperature strength due to the lack of appropriate testing techniques adequate for thin films up to date. Recent progress on the development of high temperature nanoindentation has opened the door to test the high temperature strength of nanoscale multilayers. In this talk, we will address two specific systems: Al/SiC and Cu/Nb. The first one constitutes an example of a metal-ceramic multilayer, where the combination of metallic and ceramic layers at the nanoscale has demonstrated a good combination of strength and toughness. The second example, combining metals of different crystal structure, corresponds to an extensively studied metallic multilayer, which has shown a superior He solubility and the ability to prevent the degradation of material properties under irradiation. We will show that the high temperature strength is very dependent on interface structure and layer thickness. This implies that the multilayer structure can be tuned to achieve optimum high temperature strength.

Tuesday 8th April, 15.20

Measurement of Interfacial Properties in Ceramics by Microcantilever Testing

R. Todd, A. Norton, N. Yahya, G. Otiento, G. Hughes, D. Armstrong, S. Falco & N. Petrinic {Oxford U.}

Results demonstrating that strength and toughness measurements on the microstructural scale can be used to distinguish between the properties of grain boundaries with different doping in polycrystalline ceramics (alumina, silicon nitride and others) and to investigate interfacial behaviour in MWCNT-reinforced materials. These properties determine the macroscopic behaviour of ceramics, but have not been directly measurable before. The problems with making absolute measurements using this technique are also described along with possible solutions. These issues include moisture-assisted subcritical crack growth, notch bluntness and implantation and residual stresses from the FIB machining of the specimens.

Tuesday 8th April, 15.40

Mechanical characterization of ZnO/Al2O3 Multilayer Oxide Films prepared by Atomic Layer Deposition

I. Utke, R. Raghavan, M. Bechelany, D. Frey & J. Michler {EMPA Thun}

We report on a comprehensive structural and nanoindentation study of nanolaminates of Al_2O_3 and ZnO synthesized by atomic layer deposition (ALD). By reducing the bilayer thickness from 50 nm to below 1 nm, the nanocrystal size could be controlled in the nanolaminate structure. The softer and more compliant response of the multilayers, as compared to the single layers of Al_2O_3 and ZnO, is attributed to the structural change from nanocrystalline to amorphous at smaller bilayer thicknesses. It is shown that ALD is a unique technique for studying the inverse Hall-Petch softening mechanism related to grain size effects in nanomaterials. The talk will also present initial results obtained from tensile test experiments and SEM integrated compression experiments.

Abstracts – Poster Presentations

Poster 1

Nanomechanical and Ultrastructural Variations in the Sclera

R. Ahktar, P. Paoletti, B. Geraghty & M. Papi {Liverpool U.}

The sclera is dense, fibrous, viscoelastic connective tissue, which extends from the cornea to the optic nerve. Myopia is characterised by scleral weakening and axial elongation. We have previously shown that there are macroscopic variations in the biomechanical properties of the sclera across its different regions. Under uniaxial tension, there is an increase in tangent modulus from the posterior region of the sclera towards the limbus (anterior region). However, little is known about how this variation in stiffness is related to localised structure organisation within the sclera. We have now used atomic force microscopy (AFM)-based indentation using PeakForce Quantitative Nanomechanical Mapping (QNM) to characterise the nanomechanical properties and ultrastructure of the posterior, equatorial and anterior regions of the porcine sclera. PeakForce QNM, an extension of pulsed-force AFM provides real time calculation of elastic modulus at each surface contact point. QNM data were fit with the Derjaguin-Muller-Toporov (DMT) model. We found an increase in elastic modulus from the posterior to the anterior regions, which correlated with an increase in collagen fibril diameter. Dissipation also increased with fibril diameter. The lower dissipation in the posterior region suggests it is more resistant to creep as would be expected with a smaller fibril diameter. This may be related to the elevated stresses imparted by intra-ocular pressure in the posterior region.

Poster 2

Contact Experiments on Bioinspired Adhesives

E. Kroner {INM Saarbrucken}

Bioinspired adhesives, usually consisting of patterned elastomeric surfaces, have been investigated in detail during the last decade. However, the setups for these experiments were not adapted to the special boundary conditions necessary for testing patterned surfaces, i.e. adhesion tests were performed with spherical probes rather than with flat probes and controlled alignment. We have built an adhesion measurement setup, which is especially designed for contact experiments with flat probes. Adhesion measurements were performed on arrays of micropillars as well as on single macroscopic elastomer pillars to investigate both the tilt angle dependent adhesion and the deformation mechanisms of the pillars. For example, flat tip pillars showed a high tilt angle dependent adhesion, while pillars with spherical tips showed no tilt angle dependency. Mushroom shaped pillars only exhibited a tilt angle dependent adhesion, if the pillar tip did not form complete contact with the probe at high tilt angles and low preload. We also showed that the probe geometry plays a crucial role for adhesion tests on patterned surfaces. The deformation of the pillar tips, the pillar stems and the backing layer was investigated by data analysis of adhesion measurements from pillars with different aspect ratio. The contribution of the deformation mechanisms to the overall deformation was identified and compared to theoretical models.

Poster 3

Push-to-pull Tensile Testing of 3D Micro-architectures

J. Bauer, C. Lange & O. Kraft {Karlsruhe U.}

It has been shown that cellular materials with specifically designed micro-architecture can be fabricated applying 3D laser lithography and atomic layer deposition. The resulting truss structures consist of polymer cores with a typical diameter of 0.5-1.0 µm coated with thin alumina layers. The conjunction of both structural design and size dependent material strengthening effects enables outstanding ratios of strength to weight. However, mechanical testing in that size regime is not trivial. In this paper, we present a novel technique of push-to-pull tensile testing for such micro-architected structures as well as single structural elements such as beams and struts. Specimen and push-to-pull structure are integrally manufactured in one single lithography step, avoiding time-consuming preassembly of specimens and related experimental scatter. The push-to-pull mechanism allows for performing tensile tests with a nanoindentation setup equipped with a flat punch tip. First results are presented, from which the strength of alumina layers with thickness ranging from 10 to 200 nm is deduced.

Poster 4

Extreme Nanomechanics: Vacuum Nanoindentation to 1000˚C

B. Beake, M. Davies & A. Harris {Micromaterials}

To reliably test mechanical properties at very high temperatures without any indenter or sample oxidation an ultra-low drift high temperature vacuum nanomechanics system with a maximum test temperature of 1000˚C has been recently developed [1]. Elevated temperature mechanical and tribological properties are much more relevant for practical wear situations than corresponding measurements at room temperature. However, high temperature nanomechanics and tribology is highly challenging experimentally. To overcome these challenges we have developed our NanoTest Xtreme instrument with:

(1) active heating of the sample and the indenter with patented thermal control method (no thermal drift during the high temperature indentation so that measurements can be performed as reliably as at room temperature)

(2) horizontal loading (to avoid convection at the displacement sensor)

(3) patented hot stage design (greater stability for drift-free high temperature measurements in vacuum).

This technology pitch briefly describes the development of the new NanoTest Xtreme system and its use at high temperature on a wide range of metals, alloys, ceramics and coatings for applications in the aerospace, nuclear and wear resistant coatings sectors. The new capability is unique and provides a step-change in the ability to do high temperature nanoindentation tests at above 750˚C.

[1] The NanoTest Xtreme. www.micromaterials.co.uk

Poster 5

Strain Rate Jump, Load Relaxation and Creep tests on Nanocrystalline Ni Micropillars as a Function of Temperature

G. Mohanty, J.M. Wheeler, J. Wehrs, R. Raghavan, L. Philippe, J. Michler {EMPA Thun}

This presentation will focus on micropillar strain rate jump, load relaxation and creep tests on nanocrystalline Ni micropillars (grain size ~30 nm) as a function of temperature from RT to 100˚C. Micro-pillar compression studies were carried out in-situ in the SEM to observe deformation of the pillars during testing. Strain rate sensitivity exponent, creep stress exponent, activation volume and activation energy values were determined. The decrease in strain sensitivity exponent and activation volume (from m = 0.017, V = 16b^3 at room temperature to m = 0.03, V = 10b^3 at 100˚C in micro-pillar compression) was found to be consistent with trends from bulk testing as reported in literature. Possible rate-controlling deformation mechanisms will be discussed in light of these extensive experimental results and activation parameters.

Poster 6

Deformation in B2 Intermetallics

P.R. Howie & W.J. Clegg {Cambridge U.}

Compounds with the B2 (CsCl) structure exist across a wide range of intermetallic systems. This makes them ideal subjects for the study of compositional and electronic effects on mechanical properties. In this work, a number of B2 intermetallics of the first row transition metals are synthesised and their hardnesses and moduli measured using nanoindentation and micropillar compression. Correlations between mechanical properties of the compound and electronegativity differences between the constituent atoms are explored.

Poster 7

On the Size-dependent Strength of Nano-structured Materials

A. Bushby {QMUL}

Nanomechanics experiments have firmly established the existence of genuine size effects in the strength of crystalline solids that deform by dislocation plasticity. The yield or plastic flow stress observed in different loading geometries is often considered to have a different dependence on length scale, depending in part on the presence or absence of a strain gradient. The internal microstructure of the material may also be considered to introduce a length-scale that determines the strength, such as the grain size, dislocation density or precipitate spacing. Here we show that by careful consideration of all the length-scales determining the flow stress of the material in different materials systems, the strength may be described by a simple model determined only by the shear modulus, G, Burgers' vector, b, bulk strength, σ_0, and the effective length-scale, l_{eff}. The model implies that all of the observed size dependencies of strength are driven by the constraints on stress and dislocation curvature according to the space available, that is, size itself is the underlying strengthening mechanism. Using these principles, new material systems based on controlled nano-structure can be developed to exploit "length-scale engineering".

Poster 8

Plastic Flow in Laves Phases

C. Davies, P.R. Howie & W.J. Clegg {Cambridge U.}

Laves phases have attracted attention as alternative high temperature materials because of their good creep resistance at high temperatures (Sauthoff, 2000). Laves phases are brittle, so that, apart from some indentation studies, the work to date has studied deformation at higher temperatures, generally above the ductile-brittle transition temperature. However, it is often the lower temperature flow behaviour, or rather lack of it, that limits the use of such materials, by limiting the toughness, although it is known that the flow behaviour depends on the composition of the Laves phase. Four cubic (C15) Laves phases were selected: $NbCr_2$, $NbCo_2$, $MgCu_2$ and $HfMo_2$. The elements in the different compounds have similar radius ratios but significantly different electronegativities. Micropillar compression was used to suppress fracture (Ostlund et al., 2011; Korte and Clegg, 2012) and enables plastic flow to be studied in a controlled manner. Micropillars were made by focused ion beam milling and compressed in a nanoindenter a flat punch. The yield stresses were measured and slip systems were identified using scanning electron microscopy. The detailed nature of the slip patterns, and of dislocation motion, is established using transmission electron microscopy.

Poster 9

Identification of the Critical Resolved Shear Stress Ratios of the Deformation Systems and Investigation of Slip Transfer in Ti-5Al-2.5Sn alloy

D. Mercier {MPI Düsseldorf}

The modeling of the mechanical response of polycrystalline metals requires the quantification of crystal plasticity hardening parameters as an essential part of the understanding of the micromechanical properties of grain boundaries. In the present work, the nature of single crystal plasticity of Ti-5Al-2.5Sn (wt%) near α alloy is quantified by a combination of crystal lattice orientation mapping, instrumented sphero-conical nanoindentation, and measurement of the resulting surface topography, based on a nonlinear optimization [1]. With this method, the adjustable parameters of the constitutive description are identified by iteratively improving the match of a corresponding crystal plasticity finite element simulation of the indentation. Moreover, bi-crystal deformation experiments are very informative to analyze the slip transfer at grain boundaries. This approach is implemented by carrying out indentations close to individual grain boundaries whose inclinations were measured by focused ion beam milling. The experimental data including pop-in behavior is analyzed in the light of possible slip transmission parameters [2]. Finally, experimental topographies are compared to simulated bi-crystal indentations using the previously calibrated single crystal constitutive law to assess the micromechanical effect of the grain boundaries.

[1] Zambaldi C. et al., J. Mater. Res. **27** (2012) p.356–367.
[2] Luster J. et al., Metall. Mater. Trans. **A26** (1995) p.1745-1756.

Poster 10

Modelling the Size Effect in Micro-cantilever Beams

E. Tarleton {Oxford U.}

Discrete dislocation plasticity was used to simulate end-loaded cantilevers to interpret the behaviour observed in the experiments. The model allowed correlation of the simulated dislocation structure to the experimental load displacement curve. There are similarities between the predictions of this model and those of earlier discrete dislocation plasticity models of pure bending. However, there are notable differences, including a strong source density dependence of the size effect that cannot be explained by GND arguments, and the effect of the cantilever stress distribution on the locations of the soft pile-ups.

Poster 11

Drafting Deformation Mechanism Maps from Temperature Dependent Indentation Data

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Despite the more complicated interpretation of indentation experiments over for example to more recent micro-pillar compression, the relative ease by which the indentation experiments can be a carried out means that indentation can more rapidly generate a wider set of data. Therefore for ceramics, indentation techniques continue to offer a convenient way to investigate plasticity and other deformation mechanisms. Indeed, combining indentation measurements from different temperatures can be used to draft out a deformation mechanism map. This will be illustrated using ZrB_2 as an example where combining nano-indentation data at moderate temperatures with self-indentation up to 2000˚C gave good agreement with the limited other information available in the literature and further high temperature testing has shown that the estimated parameters are in fact quite reasonable.

Poster 12

Quantifying Slip and Hardening Properties of Crystals by Indentation

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The anisotropic plasticity of crystals is of paramount importance for the mechanical performance of metallic materials. However, the critical resolved shear stresses (CRSS) and hardening parameters for widely used alloy constituents have only rarely been identified. This is due to the experimental effort that is associated with the classical way of determining these parameters: single crystals need to be grown, cut in specific orientations and then tested individually in tension or compression. We propose a novel high throughput method [1] to assess the active deformation mechanisms in ductile crystals by indentation. Individual grains of known orientations and sufficient size are indented by a spherical or conical indenter. The load-displacement curves, as well as the surface topographies, of the indents are measured. By three-dimensional crystal plasticity finite element simulation of the indentation process the load and topography can be simulated with good accuracy. Thus the constitutive parameters of the crystal can be identified by nonlinear optimization with an objective function based on the orientation dependent indent topographies. Examples of HCP and BCC phases will be presented.

[1] C. Zambaldi, Y. Yang, T.R. Bieler, D. Raabe (2012) *Orientation Informed Nanoindentation of Alpha-titanium: Indentation Pileup in Hexagonal Metals Deforming by Prismatic Slip*, JMR **27**, 356, http://dx.doi.org/10.1557/jmr.2011.334 or http://czam.de/publications

Poster 13

Deformation in Fine-grained CrN-based Coatings

W.J. Clegg, S. Liu, X. Zeng & J. Michler {Cambridge U.}

Very fine-grained CrAlN/Si_3N_4 hard coatings have a superior wear resistance to more conventional CrN-based hard coatings. To investigate this, direct measurements have been made of the hardness, *H*, yield stress, *Y*, and Young modulus, *E*, of a very fine-grained coating and compared with other CrN-based coatings. It is shown that, although the hardness is increased somewhat compared with conventional CrN-based coatings, the ratio of *H*/*Y* is consistent with that predicted by an expanding cavity type model. This suggests that effects associated with increases in the Young modulus do not occur. Observations of the compressive failure behaviour show that cracking is catastrophic in the conventional coatings, but occurs in a much more benign fashion where the material is fine-grained, suggesting that it is the fracture resistance that dominates the wear resistance of these coatings.

Poster 14

Durability of MAX-phase Thin Films: Nano-impact and Elevated Temperature Nano-friction

J. Colligon, J.F. Smith, V.M. Vishnyakov, M.I. Davies, A.J. Harris & B.D. Beake
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MAX-phase materials, especially Cr_2AlC, have been suggested as potential new high temperature coatings for turbine blades, which may also act as solid lubricants. There is therefore much interest in their elevated temperature friction and wear behaviour and durability to repetitive contact. In this study $Cr_{54}Al_{20}C_{26}$ and also, TiN, TiFeN and TiFeMoN films were deposited on silicon wafers in a dual ion beam sputtering system and their mechanical properties determined by nano-indentation, nano-scratch and nano-impact techniques. The films were chosen, not only for the intrinsic interest in their performance as high temperature materials in tribological applications, but also because macro-scale data on related systems and room temperature tribological data already exist for comparison. Their stability and tribological behaviour as a function of temperature were assessed using a new experimental capability for elevated temperature nano-scale friction measurement. Frictional data, obtained using a WC-Co probe at 25, 400 and 750°C, shows an increase in surface roughness at 750°C, which is significant for all but the MAX film and one of the TiFeMoN films. The mean friction was not particularly affected, but the variability in friction along the scratch track was different. In addition, there was a non-linear behaviour of the friction coefficient with temperature and a maximum in friction coefficient was observed at 400°C. The decrease in friction at 750°C was associated with the formation of lubricating surface oxides. The MAX-phase coating exhibited lower surface roughening than the majority of the other films, presumably due to its reduced oxidation (oxidation onset ~700°C compared with ~ 550°C for TiN-based films). Nano-impact testing enabled assessment of the toughness and resistance to fatigue fracture of the films under repetitive loading. The weak bonding between the layers in the MAX-phase gave rise to layered deformation behaviour in the nano-impact test, which was completely different to the response in other films.

Poster 15

A Novel Nanoindentation Method for the Analysis of Surface Free Energy on Micro-areas: Application to Patterned Surfaces and Micro-particles

M. Sebastiani, R. Moscatelli, F. Vallerani, E. Bemporad & F. Carassiti {Roma TRE}

Surface Free Energy (SFE) is an important material property that plays a significant role in determining the functional behaviour of a large variety of micro/nano-devices, e.g, micro and nano-particles for drug delivery, micro and nano-electromechanical systems and nano-patterned surfaces for tissue engineering. However, measurement of SFE on small areas is not an easy task, due to the limits in terms of spatial resolution of the techniques currently available in the literature. In this work, a new method is presented for the analysis of surface free energy on a small area. The methodology is based on the analysis of the snap-in force detected before the contact between a spherical tip and a substrate, during a conventional nanoindentation experiment. An analytical model is developed to evaluate the SFE from the measured snap-in force, being known the radius and SFE of the indenter. The model has been also extended to the case of a spherical substrate, in order to make it suitable for the analysis of SFE on micro-particles. The new technique is tested and validated on different bulk materials, namely Molybdenum PP and PMMA. Obtained results compare well with independent measurements by conventional contact angle measurements. In addition, the experimental sensitivity to variations of surface chemistry and/or nano-scale roughness is analysed. Finally, the method is applied on nano-patterned PMMA surfaces and on PMMA micro-beads and preliminary results are presented.

Poster 16

Problems of Extracting Mechanical Properties of Porous Coatings using Nanoindentation

N. Moharrami & S. Bull {Newcastle U.}

Extracting mechanical properties using nanoindentation has been studied by researchers for many years. When using any indentation technique to assess the mechanical properties, factors such as surface roughness and porosity have a significant effect on the observed Young's modulus and hardness values. In this study, the nanoindentation response of three porous coated samples, copper-tin (Cu-Sn), pure copper (Cu) and tin (Sn), deposited by electroplating from an ionic liquid, have been measured. As the coatings were porous, the measured modulus and hardness results show large scatter. The Young's modulus values were about 30% of the bulk values for Cu, Sn and Cu-Sn alloy seen in previous studies, due to the low density of the coatings, as seen in Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM) images. The porosity effect on the indentation elastic response of the coatings was simulated using Object Oriented Finite 2 (OOF2) element analysis. This work highlights the effects of the coating structure on the mechanical response and the importance of the surrounding porous features on nanoindentation test results, when compared to those of fully dense coatings.

Poster 17

Software for Extraction of Material Properties from Indentation Data (SEMPID)

T.W. Clyne, G.N. Wells, G. Aldrich-Smith & J. Dean {Cambridge U.}

There are now well-developed methodologies [1, 2] for obtaining plasticity and creep characteristics from indentation data, using inverse iterative finite element methodologies (FEM). Future developments may allow these to be extended to more complex responses, such as those exhibited by multi-layered and coated systems, super-elastic deformation and shape memory effects. However, even for relatively simple properties, such as the yield stress and work-hardening rate of a bulk material, there are significant barriers to widespread usage of these methodologies. These partly arise from the limited availability of FEM packages, but there are also issues relating to the most appropriate experimental data on which to focus and the best type of algorithm to employ for efficient iterative convergence on "best-fit" values of the parameters being sought. For creep characterisation, these problems are compounded by there being more variables, particularly since both primary and secondary creep are likely to affect indentation responses. This presentation concerns a suite of packages being developed to facilitate wide and user-friendly utilisation of these methodologies. The software is self-contained and does not require access to, or even familiarity with, FEM packages (although it does have FEM computational capabilities). It incorporates recommendations concerning the indentation measurements to make and guidelines for iterative optimisation of agreement between experiment and prediction. It's not essential for the indentation to be carried out on a very fine scale and, indeed, the volume of material being interrogated must be large enough for its response to be representative of the bulk. Furthermore, very shallow penetration often leads to complications from surface roughness or surface contamination layers. Calibration specimens are provided for validation of the procedures being employed. The procedures are limited in this developmental version to transversely isotropic systems and the properties obtained are those in the through-thickness direction. During this developmental stage, collaboration is being sought for implementation testing and user feedback.

[1] Dean, J, Wheeler, J.M, & Clyne TW, *Use of Quasi-Static Nanoindentation Data to Obtain Stress-Strain Characteristics for Metallic Materials*, Acta Materialia **58** (2010) p.3613-3623.

[2] Dean, J, Bradbury, A, Aldrich-Smith, G & Clyne, TW, *A Procedure for Extracting Primary and Secondary Creep Parameters from Nanoindentation Data*, Mechanics of Materials **65** (2013) p.124-134.

Poster 18

Indentation of Bilayer Systems with Finite Top Layer Thickness

J. Reed, J. Dean & T.W. Clyne {Cambridge U.}

Progress has been made recently [1, 2] on obtaining plasticity and creep characteristics from indentation data, using inverse iterative finite element methodologies. This presentation is focussed on extending these methodologies, for study of plasticity, to bi-layer systems in which the thickness of the top layer ("coating") is not much greater than the indenter penetration depth, so that the properties of the under-layer ("substrate"), as well as those of the coating, affect the response. Specimens were produced from rods of pure copper and 304 stainless steel, using electro-discharge machining to remove thin disks of one material and bonding them to thick disks of the other. Top layer thicknesses were 150 and 300 µm. These specimens were indented using a large (2 mm) diameter WC sphere, with penetration depths of up to about half of the top layer thickness. Experimental indent profilometer data were compared with model predictions, both in the form of the penetration depth (for a given load) and the shape of the depression and its surrounding area. This was done for a series of loads, providing a comprehensive set of comparisons. Yield stress and work hardening rate values (for both materials) were inferred from indentation data obtained with thick samples and were found to be consistent with corresponding values obtained with thin coatings. A cross-check was also made with values of these parameters obtained by conventional (compressive) testing, although in that case it was only possible to induce plastic strains of up to about 20% before barrelling became significant (whereas indentation-induced local plastic strains ranged up to about 100%). In general, the methodology looks to be viable and some sensitivity information is presented to help guide such procedures.

[1] Dean, J, Wheeler, J.M, & Clyne TW, *Use of Quasi-Static Nanoindentation Data to Obtain Stress-Strain Characteristics for Metallic Materials*, Acta Materialia **58** (2010) p.3613-3623.

[2] Dean, J, Bradbury, A, Aldrich-Smith, G & Clyne, TW, *A Procedure for Extracting Primary and Secondary Creep Parameters from Nanoindentation Data*, Mechanics of Materials **65** (2013) p.124-134.

Poster 19

Mechanical Properties of Multilayer Thin Films: an Elevated Temperature Microcompresison Study

J. Michler, R. Raghavan & J.M. Wheeler {EMPA Thun}

The size dependence and effects of constraint on plasticity of soft metals such as Cu and Al sandwiched between alternating layers of hard and immiscible metals and / ceramics such as W and TiN were also studied by microcompression experiments. Mutilayers of different material combinations were deposited for this purpose by magnetron sputtering. Variable strain rate microcompression experiements were performed inside the scanning electron microscope at different temperatures. Estimates of flow stress as a function of temperature and deformation mechanisms were interpreted by Hall-Petch analysis, interfacial diffusion and abnormal grain growth.