CAMTEC II

Symposium on Fine-Scale Mechanical Characterisation and Behaviour

29th – 30th March, 2010

The CAMTEC series

As for the first Symposium in the CAMTEC series, which took place in April 2006, CAMTEC II will be held in Downing College, Cambridge. The venue will be the Howard Lecture Theatre, a purpose-built building opened in December 2009. There is 160 m^2 of exhibition space on the ground floor, with adjacent catering facilities, and an auditorium above, with over 100 seats and a raked floor. The Symposium will be chaired by Bill Clyne and the co-Chairs will be Bill Clegg (both Gordon Laboratory, Cambridge), Johann Michler and Ivo Utke (both EMPA, Switzerland).

Acknowledgement of Support

We wish to thank the following for their contribution to the success of this conference: European Office of Aerospace Research and Development, Air Force Office of Scientific Research, United States Air Force Research Laboratory <http://www.london.af.mil>.

In addition, the sponsorship provided by the following organisations is gratefully acknowledged: Agilent Technologies, Cambridge University Press, LOT Oriel and Wiley.

The following people, all of whom are based in the Materials Science Department in Cambridge, have contributed substantially to various aspects of the organisation of the Symposium: James Dean, Jorge Sobral, Brian Barber, Sandra Korte, Rebecca Martin and Rebecca Pritchard.

Format of the Meeting

All of the presenters have been personally invited, and attendance is also by invitation only. There will be about 80 delegates, including about 20 representatives of organisations participating in the Industrial Exhibition. There will be 22 talks (all of 20 minutes duration), divided into 5 sessions spread over the two days. In addition, there will be 32 posters, constituting a prominent element of the Symposium. These will be on display throughout the meeting, in the same place as the Industrial Exhibition. The meals and refreshment breaks (apart from the Symposium dinner) will also be in this location. There will be a session of short talks by poster authors. Five poster prizes will be awarded, on the basis of delegate voting. These prizes, which have all been provided by sponsors, will be presented at the end of the Symposium.

The Oral Programme

Monday 29th March

Time	**Presenter**	**Affiliation**	**Title**
11.00-12.40	**Registration / Setting up of Exhibition and Posters**		
12.40-14.00	**Lunch**		
14.00-15.20	Session 1: **Extraction of Properties & Characteristics** (Session Chair: **Johann Michler**)		
14.00-14.20	George Pharr	Tennessee	*Critical Issues in Making Small-Depth Mechanical Property Measurements by Nanoindentation with Dynamic Stiffness Measurement*
14.20-14.40	James Dean	Cambridge	*Quasi-static Nanoindentation of Metals to obtain Stress-Strain Constitutive Relations*
14.40-15.00	Christophe Tromas	Poitiers	*Slip line analysis around nanoindentation imprints in Ti3SnC2: a new insight into plasticity of MAX-Phase materials*
15.00-15.20	Michel Barsoum	Drexel	*Spherical Nanoindentation Stress-Strain Curves, Kinking Nonlinear Elastic Solids and Plastically Anisotropic Solids*
15.20-16.00	**Tea / Exhibition / Posters**		
16.00-17.40	Session 2: **Anisotropy, Layered structures, Thin Films and Surface Effects** (Session Chair: **Bill Clyne**)		
16.00-16.20	Helena Van Swygenhoven	PSI Villigen	*In-situ micro-compression during white beam Laue diffraction*
16.20-16.40	David Armstrong	Oxford	*Measuring Anisotropy in Young's Modulus using Micro-Cantilevers*
16.40-17.00	Adrian Mann	Rutgers	*Surface chemistry and plasticity - the Rehbinder effect revisited at the nanoscale*
17.00-17.20	Ehrenfried Zschech	IZFP Dresden	*Modifications in organosilicate glasses on a sub-100 nm scale studied with nanoindentation and FM-AFM*
17.20-17.40	Gerold Schneider	Hamburg	*Size-dependent Elastic/Inelastic Behavior of Enamel over Millimetre and Nanometre Length Scales*
17.40-18.40	**Oral Presentation of Posters** - 2 minutes per poster (Session Chair: **Bill Clegg**)		
18.40-19.15	**Free**		
19.15-19.45	**Champagne Reception** (Sponsored by **Agilent Technologies**) / **Exhibition** / **Posters**		
19.45-22.00	**Symposium Dinner** (After-dinner speaker: **Munawar Chaudhri**)		

Tuesday 30th March

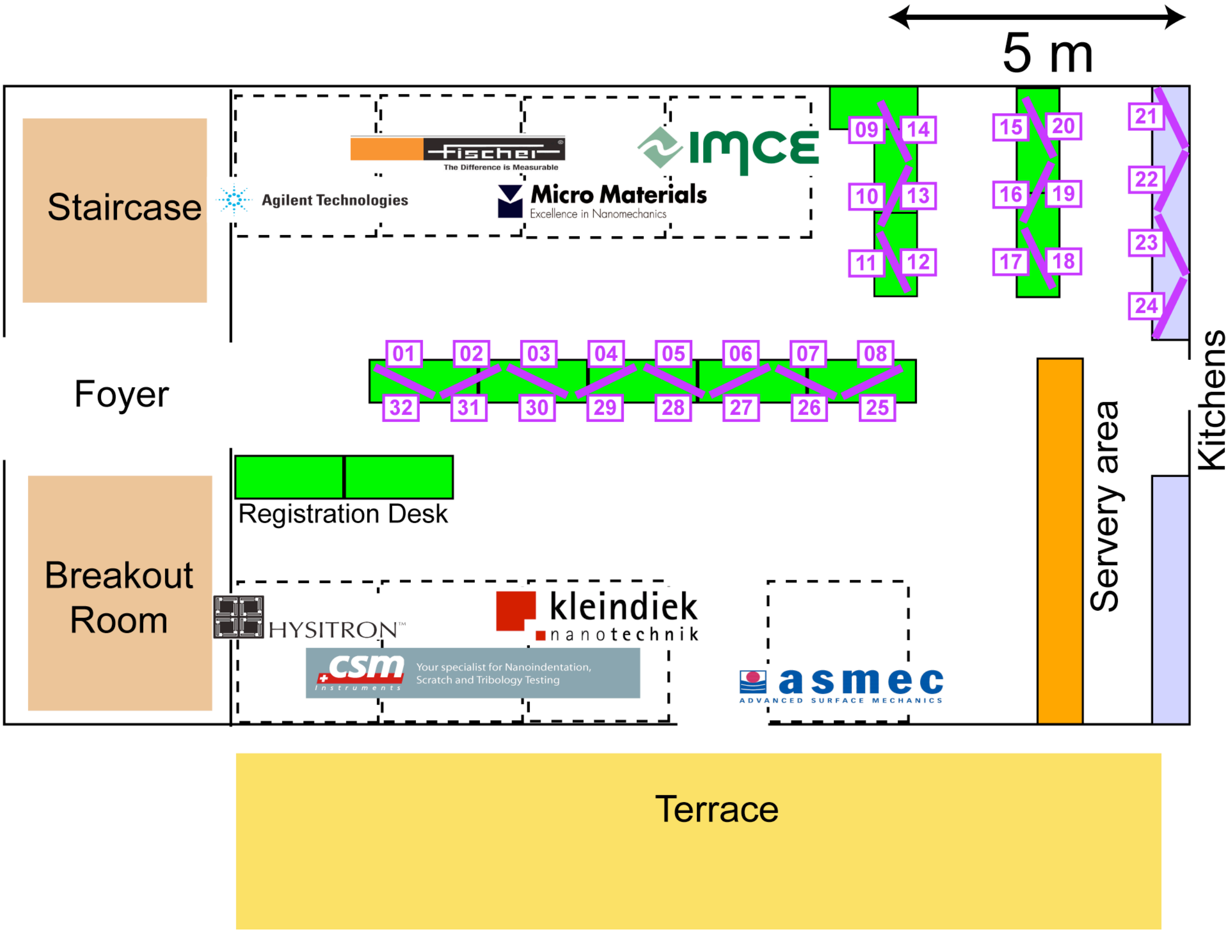
Time	**Presenter**	**Affiliation**	**Title**
07.30-09.00	**Breakfast**		
09.00-10.40	Session 3: **Scale Effects** (Session Chair: **Ivo Utke**)		
09.00-09.20	Easo George	Oak Ridge	*Spherical nanoindentation as a complementary technique to micropillar compression for characterizing size and dislocation density effects*
09.20-09.40	Steve Bull	Newcastle	*Size effects in the mechanical response of thin coatings on glass*
09.40-10.00	Andy Bushby	QMUL	*Indentation size effects: is smaller harder or just more difficult?*
10.00-10.20	Sandra Korte	Cambridge	*Size effects in microcompression of hard materials*
10.20-10.40	Rejin Raghavan	EMPA	*Size effects in diverse structural states of a Zr-based bulk metallic glass*
10.40-11.20	**Coffee / Exhibition / Voting for Poster Prizes**		
11.20-12.40	Session 4: **Damage Development and Fracture** (Session Chair: **Bill Clegg**)		
11.20-11.40	Gerhard Dehm	Leoben	*Deformation and fracture studies of thin films using miniaturized mechanical tests*
11.40-12.00	Jon Molina Aldareguia	IMDEA	*Deformation and failure mechanisms of fibre-reinforced composites studied by in-situ mechanical testing and computational micromechanics*
12.00-12.20	Oliver Kraft	Karlsruhe	*In Situ Investigation of Plasticity and Fracture at Small Scales*
12.20-12.40	Richard Todd	Oxford	*Fracture of ceramics at the microscale*
12.40-14.00	**Lunch**		
14.00-15.20	Session 5: **Soft and Biological Materials** (Session Chair: **Bill Clyne**)		
14.00-14.20	Heinz Sturm	BAM	*Using a SFM / ESEM Hybrid for the Analysis of Vibrating Surfaces*
14.20-14.40	Michelle Oyen	Cambridge	*Nanoindentation measurements of hydraulic permeability*
14.40-15.00	Yang Tse Cheng	Kentucky	*Obtaining Viscoelastic Properties from Instrumented Indentation*
15.00-15.20	Ulrike Wegst	Drexel	*Spherical nanoindentation to measure the elastic modulus and indentation yield point on biological materials*
15.20-16.00	**Tea / Exhibition / Presentation of Poster Prizes / Group Photograph**		

The Poster Programme

No.	Presenter	Affiliation	Title
1	Dave Armstrong	Oxford	*Applications of Micro-Cantilever Testing*
2	Ian Ashcroft	Loughborough	*An Investigation of the effect of moisture on the mechanical properties of polymers by depth sensing indentation*
3	Ben Beake	MML	*Elevated temperature (to 750˚C) and high strain rate nanomechanics for extreme applications*
4	Gottfried Bosch	Fischer	*ESP – Enhanced Stiffness Procedure applied on soft or hard µm-coatings*
5	Munawar Chaudhri	Cambridge	*Nanohardness of high purity Cu (111) single crystals: the effect of indenter load and prior plastic sample strain*
6	Thomas Chudoba	ASMEC	*Micro wear inestigations of DLC coatings with nanometre resolution in normal and lateral direction*
7	John Colligon	Manchester Metro	*Influence of mechanical properties on deformation behaviour of TiFeN, TiN and TiFeMoN films on Si in nano-scratch tests*
8	Georgios Constantinides	Cyprus	*Indentation Testing of Porous Materials*
9	James Dean	Cambridge	*Using Indentation to Measure Residual Stresses in Surface Layers*
10	Brian Derby	Manchester	*Ultra-High Strength Nanoporous Silver Produced by Inkjet Printing*
11	Quynh H. Duong	CSM	*Investigation of creep and time dependent properties of elastomers and polymers*
12	Nicola Everitt	Nottingham	*Isothermal contact nanoindentation in a controlled environment up to 750°C*
13	Mark Gee	NPL	*Micro- and nano-tribology experiments with a novel in-situ test system*
14	Russell Goodall	Sheffield	*Characterisation of Free Volume in Amorphous Metals by Nanoindentation*
15	Griselda Guidoni	Saarbrucken	*Do the individual pillars of a structured compliant surface communicate with each other?*
16	Ude Hangen	Hysitron	*Thin film interfacial adhesion studies by use of nanoindentation*
17	Philip Howie	Cambridge	*Compression Splitting of Micropillars*
18	Nigel Jennett	NPL	*Enhanced Yield strength of materials - the "Thinness Effect"*
10	Stephan Kleindiek	Nanotechnik	*Kleindiek nanotools for fine scale mechanical characterization*
20	Johann Michler	EMPA	*In-situ electron backscatter diffraction (EBSD) during the compression of micropillars*
21	Charlie Parkinson	GSK	*Erosion and the Role of Fluoride: A Nanomechanical Study.*
22	Andreas Schneider	Saarbrucken	*Correlation Between Critical Temperature and Strength of Small-Scale BCC Pillars*
23	Norbert Schwarzer	Saxonian Institute	*Pharr's Concept of the Effectively Shaped Indenter Extended to Creep and Viscous Material Behaviour*
24	Jim Smith	Micro Materials	*Small-scale Polymer Analysis using Sample Oscillation*
25	Greg Swadener	Aston	*Influence of crystal orientation on scratch testing with a spherical indenter*
26	Jin Chong Tan	Cambridge	*Relating Mechanical Properties and Chemical Bonding in Hybrid Framework Materials*
27	Philipp Thurner	Southampton	*Cantilever-based Nanoindentation for the Characterization of Thin Films and Soft Materials*
28	Christophe Tromas	Poitiers	*Nanohardness cartography: influence of the dendritic/interdendritic chemical composition on the mechanical properties of a MCNG superalloy*
29	Ivo Utke	EMPA	*SEM integrated force and resonance measurements of focused-electron-beam-induced Cu/C nanowire deposits*
30	Luc Vandeperre	Imperial	*The relation between microstructural scale and the influence of porosity and second phases on hardness*
31	Jeff Wheeler	EMPA	*AFM Observation of Diamond Indenters after Oxidation at Elevated Temperatures*
32	Ping Xiao	Manchester	*Deformation and fracture behaviour of silicon carbide under indentation*

Poster Display and Industrial Exhibition

Both the poster display and the industrial exhibition will be held on the ground floor of the Howard Lecture Theatre, immediately below the auditorium. The layout is shown below. All of the poster boards (ie the velcro space within the borders) measure 116 cm (horizontally) by 90 cm (vertically). They will therefore be suited to posters in landscape orientation. Posters should have velcro adhesive pads on the reverse side.



Poster prizes will be awarded, on the basis of delegate voting. The following prizes are available:

1 - LOT Oriel Prize (£250 cash)
2 - Wiley Book Voucher Prize (£250 value)
3 - Wiley Journal Subscription Prize (£250 value)
4 - Agilent Book Voucher Prize (£200 value)
5 - CUP Book Voucher Prize (£200 value)

Each delegate will be supplied with 5 coloured plastic disks, having the following values for voting:

White:	5 points
Yellow:	4 points
Green:	3 points
Blue:	2 points
Red:	1 point

Votes are cast by placing a disk on a stack at the poster concerned. Voting for a poster which has the voter as an author will not be permitted. Posters will be ranked according to the total number of points allocated, and prizes presented to the first 5 in the list (at 15.30 on Tuesday 30th March). It would be appreciated if all poster authors, or a nominated substitute, could be present at that time. Votes can be cast at any time from the beginning of the Symposium ***until lunch-time on Tuesday***.

Internet Access

Free Wifi access will be available to all delegates via the “Eduroam” system. Information about how this system works is available at www.cam.ac.uk/cs/wireless/eduroam/. In addition, there will be 4 hard-wired connections available in the “Breakout Room” (see diagram above) for delegates to connect to their laptops. Again, there will be no charge for this access.

Attending Delegates

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Seating Plan for Dinner

	Helena van Swygenhoven	Adrian Mann	Marilyn Pharr	George Pharr	Gail Clyne	Bill Clyne	*Munawar Chaudhri*	*Joanne Clegg*	Bill Clegg	Heike Zschech	Ehrenfried Zschech	
Michel Barsoum												Lindsay Greer
	Oliver Kraft	Ulrike Wegst	Andrew Wallwork	James Dean	Neil Seagrave	Ivo Utke	Gerhard Dehm	Sandra Korte	Tony Skinner	*Michelle Oyen*	*Johann Michler*	

Brian Derby	Gottfried Bosch	Phil Mallard	*Nigel Jennett*	Philipp Thurner	Quynh Huong Duong
Ian Ashcroft	*Steve Roberts*	Luc Vandeperre	Rhys Jones	Christophe Tromas	Claire Leppard
Elizabeth Johnson	Ping Xiao	Stephan Kleindiek	Greg Swadener	Keith Dawes	Andreas Schneider
David Armstrong	Michael Berg	Richard Todd	Russell Goodall	Andrew Smith	*Jeff Wheeler*
Nicola Everitt	Joern Ritterbusch	Jim Smith	Franziska Kairat	Torsten Kirst	Norbert Schwarzer
Ben Beake	Richard Wellman	Heinz Sturm	Mark Gee	Rejin Raghaven	Gerold Schneider
Steve Bull	Gregory Favaro	Jin Chong Tan	John Gearing	Jon Molina	*Caroline Humphrey*
Joris Bracke	Phillip Howie	Griselda Guidoni	Easo George	Lisa Taylor	Giles Aldrich-Smith
Andy Bushby	Denise Hoban	David Morgan	Steve Goodes	Krish Narain	*Georgios Constantinides*
Paul Cave	John Colligon	Robert Stearn	Finn Giuliani	James Curran	Charles Parkinson
Thomas Chudoba	Yang Tse Cheng	Winfried Staib	Ude Hangen	Pedro Portella	Hans-Peter Vollmar

(Italics = Dietary Requirement)

Abstracts – Oral Presentations

Monday 29th March, 14.00

Critical Issues in Making Small-Depth Mechanical Property Measurements by Nanoindentation with Dynamic Stiffness Measurement

George Pharr
University of Tennessee, Knoxville

Dynamic stiffness measurement (DSM), often referred to as continuous stiffness measurement (CSM), is a technique used commonly in nanoindentation to measure hardness and elastic modulus continuously as a function of depth as the indenter is loaded into the specimen. To apply the technique, a small, sinusoidal oscillation is added to the primary loading signal, and the amplitude of the resulting displacement oscillation at the same frequency is measured by means of a lock-in amplifier. For elastic contact, the ratio of the load to displacement oscillation amplitudes provides a measure of the contact stiffness, which can then be used to determine the hardness and elastic modulus by standard methods of analysis. However, we have recently realized that for materials with a high modulus-to-hardness ratio, e.g., soft metals, CSM techniques can produce significant errors in the measured properties at depths of indenter penetration of 100 nm or even greater. In this presentation, the origin of these effects is documented by means of experiments conducted in a soft copper single crystal, and a model is developed that allows the effects to be quantified. By correcting the data in accordance with model and performing measurements at smaller displacement oscillation amplitudes, the errors can be significantly reduced. The errors are particularly important in characterizing the indentation size effect in metals.

Monday 29th March, 14.20

Quasi-static Nanoindentation of Metals to Obtain Stress-Strain Constitutive Relations

James Dean†, Jeff Wheeler§, Giles Aldrich-Smith* & Bill Clyne†
†University of Cambridge, §EMPA Thun & *AWE

Depth-sensing indentation is commonly employed for material characterisation. Young's moduli can be determined from the unloading portion of the load-displacement curve, while hardness values can be obtained from peak loads and residual indentation areas. Less reliably, claims are often made that properties such as fracture toughness, creep parameters and viscoelastic characteristics can also be obtained. In many ways, the most significant and promising challenge at present is that of obtaining constitutive relationships from nanoindentation data (for materials, particularly metals, which exhibit extensive plasticity). Many previous attempts have been made to do this, but in general there is little or no consensus about the reliability of different procedures or about their optimization. In this presentation, a methodology is presented for extracting (rate-independent) constitutive equations from indentation data (load-displacement-time datasets and residual indent shapes obtained by AFM). The procedure involves iterative comparisons with FEM predictions corresponding to a series of constitutive relations. The main sources of error are identified and quantified, notably the effects of creep and, to a lesser extent, interfacial friction. It is shown that account must be taken of such effects in order to converge robustly upon unique constitutive relations. As outlined in a recent publication [1], focussed on a copper sample exhibiting linear work hardening, it is estimated that this methodology allows the yield stress to be established with a precision of about ±10% and the work hardening rate to approximately ±25%.

[1] J Dean, JM Wheeler & TW Clyne, *Use of Quasi-Static Nanoindentation Data to Obtain Stress-Strain Characteristics for Metallic Materials*, Acta Mater, in press (doi:10.1016/j.actamat.2010.02.031).

Monday 29th March, 14.40

Slip line analysis around nanoindentation imprints in Ti_3SnC_2: a new insight into plasticity of MAX-Phase materials

Christophe Tromas, P. Villechaise, V. Gauthier-Brunet & S. Dubois

University of Poitiers

Nanoindentation is a very interesting technique for the determination of plastic deformation mechanisms. Compared to a uniaxial deformation test, the stress field generated by indentation offers areas in compression, traction or shear, and reaches locally very high values. This is thus an original method to probe all the possible slip systems in a material. The observation by Atomic Force Microscopy (AFM) of slip lines around the indents can then provide information on the deformation behavior in terms of individual dislocations.This technique has been used in this study to investigate the plasticity of the new MAX-phase material Ti_3SnC_2. $M_{n+1}AX_n$-phases (n=1, 2 or 3) materials are a class of nanolaminated ternary carbides or nitrides, with a hexagonal structure, where M stands for an early transition metal, A for a group A-element and X for carbon or nitrogen. MAX-phase materials present a unique combination of metal and ceramic properties. Their high technological potential is largely due to their specific mechanical properties, associating damage tolerance and easy machinability.Nanoindentation has been used first to characterize Ti_3SnC_2 hardness. As the sample was not a pure phase sample, the nanohardness cartography technique has been used, to correlate hardness with the tested phase and with the grain orientation. A strong indentation size effect (ISE) has been observed, and it has been shown that MAX-phase hardness is probably often underestimated to due specific grain boundaries deformation processes.In a second time, surface deformation around the indents has been observed by AFM. For several grains, buckling around the indent has been observed, in agreement with the kink bands deformation process often reported for MAX-phases. For other grains, slip lines have been revealed by Atomic Force Microscopy (AFM). The corresponding slip systems have been identified by electron backscatter diffraction (EBSD). First and second order pyramidal slip systems are shown to be active for some grain orientations, as well as dislocation interactions and cross slip. This study shed new light on deformation mechanisms of MAX-phase materials, inasmuch as their plasticity was believed to be strictly restricted to dislocation activity in basal planes.

Monday 29th March, 15.00

Spherical Nanoindentation Stress-Strain Curves, Kinking Nonlinear Elastic Solids and Plastically Anisotropic Solids

Michel Barsoum

University of Drexel

Recently we postulated that plastically anisotropic solids belong to the same class we designated as kinking nonlinear elastic, KNE. The signature of KNE solids is the formation of fully reversible, hysteretic, stress-strain loops, on repeat loadings. This full reversibility is due to the formation of incipient kink bands, that are comprised of two, nearly parallel, dislocation walls of opposite polarity that are attracted to each other; when the load is removed they annihilate. The energy dissipated per cycle is usually substantial and scales with the stress squared. Recently we have also shown that repeated spherical nanoindentations results - when converted to nanoindentation stress strain curves - on the same location is a technique that is ideally suited to characterize KNE solids. Examples on Ti_3SiC_2, mica, graphite, sapphire, $LiNbO_3$, ZnO among others are presented. We will also show that these results are in good agreement with a microscale model we developed, based on the growth and shrinkage of incipient kink bands to explain this intriguing phenomena.

Monday 29th March, 16.00

In-situ micro-compression during white beam Laue diffraction

Helena Van Swygenhoven, J. Zimmermann & S. Van Petegem

PSI Villigen

In-situ microcompression during Laue diffraction is preformed on single crystal Mo pillars obtained via directional eutectic growth (samples from E. George, ORNL). Such pillars showed a whisker type behaviour where the strength of the pillar approaches the theoretical shear strength and no particular size dependence is observed (Acta Mat 56(2008)4762). However, when these pillars are pre-deformed prior to compression, or FIB treated, the strength measured in a micro-compression test decreases and a large scatter is observed (Acta Mat 57(2008)503). To address the smaller is stronger effect during micro-compression the role of the initial microstructure and defect content in metallic pillars micro-compression is performed during Laue diffraction on as-prepared, pre-deformed and the FIB surface treated Mo pillars. The experiments show that FIB surface treatment and pre-deformation introduce streaking of the diffraction peaks, whereas the as-prepared Mo pillars exhibit very narrow diffraction peaks as expected for perfect single crystals. In-situ compression experiments performed in load control as well as in displacement control allow the correlation of special characteristics of the stress-strain curves with plastic events and changes in the microstructure. The role of the defects introduced by FIB and pre-deformation on the mechanical behaviour of Mo pillars will be discussed.

Monday 29th March, 16.20

Measuring Anisotropy in Young's Modulus using Micro-Cantilevers

David Armstrong, Angus Wilkinson & Steve Roberts
University of Oxford

Focused ion beam machining was used to manufacture micro-cantilevers 30 µm by 3 µm by 4 µm with a triangular cross section in single crystal copper at a range of orientations. Micro-cantilevers were also milled within a single grain in a polycrystalline copper sample. Electron backscattered diffraction was used to identify the direction of the long axis of the cantilevers. The cantilevers were tested using an AFM/nanoindenter, using two different testing methods, so as to explore the effects of the flexibility of the “fixed” beam end on testing and analysis. A method using multiple loading points allowed determination Young’s modulus and of the range of geometries of the beams within which simple encastré beam theory could be used. A method using a single loading point was used allow a comparison of Young's Modulus on loading and unloading. The experimentally measured values of Young's modulus and their variation with orientation were found to be in good agreement with the values calculated from the literature data for bulk copper. The method therefore has the potential to be used for determination of (crystallographically anisotropic) Young’s moduli in very small samples.

Monday 29th March, 16.40

Surface chemistry and plasticity - the Rehbinder effect revisited at the nanoscale

Adrian Mann
Rutgers University

Reports of surface chemistry affecting mechanical behavior, in particular plasticity and hardness, have been in existence for 70 or more years, but it has always been highly controversial. The chemo-mechanical effects reported by Rehbinder in the 1940's were challenged by others almost from their first publication. Later controversies surfaced in the 1970's and 1980's when the importance of the zeta potential in determining plasticity was debated. Much of the controversy can be traced to the use of micro-scale methods, such as microindentation, to make mechanical measurements. With these methods the chemical effect can often be lost in the measurement error. It is also likely that the extreme sensitivity of surface chemistry to changes in the ambient environment (for instance humidity and temperature) produced large variations in the observed effects. With the advent of advanced nano-scale mechanical characterisation tools (nanoindentation) very small volumes close to surfaces can now be studied with unprecedented sensitivity and spatial resolution. By combining these methods with highly controlled surface chemistry we have found that there are two distinctly different chemical effects that play a role in modifying the observed mechanical behavior: (1) the chemistry of the surface layer modifies the geometry of the sample and contact giving a perceived, but not genuine, change in mechanical properties; (2) the behavior of dislocations is affected by the surface chemistry and associated surface stress which causes a genuine change in the material's plasticity. Examples of both cases will be presented and the physical origins of the effects will be explained by adapting current models for the mechanics of nanocontacts and the forces on dislocations in confined volumes.

Monday 29th March, 17.00

Modifications in organosilicate glasses on a sub-100nm scale studied with nanoindentation and FM-AFM

Ehrenfried Zschech†, Kong Boon Yeap†, Reinhold Dauskardt§, Taek Soo Kim†, Dmytro Chumakov†
†IZFP Dresden, §Stanford University

Ultra low-k (ULK) materials, particularly organosilicate glasses (OSG) that insulate on-chip metal interconnects are locally modified during processing in leading-edge semiconductor manufacturing. UV curing of porous OSG is applied to remove porogen from the thin films and to optimize dielectric and mechanical properties.The mechanical properties of OSG films and particularly process-induced local changes of the material were studied with nanoindentation, and at carefully prepared cross-sections with Force-Modulation AFM (FM-AFM) analysis. During monochromatic UV curing of porous OSG films, a monotonic slope of the Young's modulus superposed by periodic oscillations was measured. Since the oscillations are caused by standing waves, the local Young's modulus depends on the UV curing energy, the film thickness and the reflectivity of the material beneath the UV cured OSG film. Film thickness and wavelength for monochromatic UV curing can be optimized in such a way that the fracture toughness of the ULK material at interfaces is increased [1]. The potential and the limits of the analytical techniques (nanoindentation, FM-AFM) for the detection of local changes of the elastic properties on a scale well below 100 nm will be discussed.

[1] T. Kim, D. Chumakov, E. Zschech, R. H. Dauskardt, "Tailoring UV cure depth profiles for optimal mechanical properties of organosilicate thin films," Applied Physics Letters, 95, 071902, 2009.

Monday 29th March, 17.20

Size-dependent Elastic/Inelastic Behavior of Enamel over Millimetre and Nanometre Length Scales

Gerold Schneider

University of Hamburg

The microstructure of enamel, like most biological tissues, has a hierarchical structure which determines their mechanical behavior. However, current studies of the mechanical behavior of enamel lack a systematic investigation of these hierarchical length scales. In this study, we performed macroscopic uniaxial compression tests and the spherical indentation with different indenter radii to probe enamel's elastic/inelastic transition over four hierarchical length scales, namely: "bulk enamel" (mm), "multiple-rod" (10's µm), "intra-rod" (100's nm with multiple crystallites) and, finally "single-crystallite" (10's nm with an area of approximately one hydroxyapatite crystallite). The enamel's elastic/inelastic transitions were observed at 0.4-17 GPa, depending on the length scale, and were compared with the values of synthetic hydroxyapatite crystallites. The elastic limit of a material is important, as it provides insights into the deformability of the material before fracture. At the smallest investigated length scale (contact radius ~20 nm), elastic limit is followed by plastic deformation. At the largest investigated length scale (contact size ~1 mm), only elastic then micro-crack induced response was observed. A map of elastic/inelastic regions of enamel from millimetre to nanometre length scale is presented. Possible underlying mechanisms are also discussed.

Tuesday 30th March, 09.00

Spherical nanoindentation as a complementary technique to micropillar compression for characterizing size and dislocation density effects

Easo George

Oak Ridge

In this talk I will discuss how results from spherical nanoindentation can be used to complement those from micropillar compression to enhance our understanding of size effects in mechanical behavior. There are two fundamental length scales in the case of nanoindentation: a material length scale determined by the dislocation spacing, which can be systematically controlled by varying the pre-strain, and a geometric length scale that varies with the indenter radius. Analogous length scales exist in the micropillar problem except that its geometric length scale varies as the pillar size rather than indenter size. Dislocation densities and strain gradients were measured before and after deformation with 3D X-ray microscopy. For small spheres and low dislocation densities, the shear stresses at pop-in are close to the calculated stress for homogeneous nucleation of a full dislocation loop. However, as the indenter size and/or the pre-strain increase, the pop-in stresses decrease significantly because the probability of finding mobile dislocations in the highly stressed zone underneath the indenter increases. Pop-ins then are caused by the activation of pre-existing dislocations rather than the nucleation of new dislocations. An analogous transition from ideal to bulk strength is observed in pillars as a function of increasing size and/or prestrain. A simple statistical model consisting of a distribution of dislocations with different pinning strengths interacting with the applied stress is found to reproduce these experimental trends. This research was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy.

Tuesday 30th March, 09.20

Size effects in the mechanical response of thin coatings on glass

Steve Bull

University of Newcastle

Although optical coatings are generally designed for their functional requirements it is often the mechanical properties of the system which limits performance. For instance, the major in-service failure mechanism of modern solar control coatings for architectural glass can be scratch damage. Many of these coatings are multilayer structures made from individual layers of less than 100 nm thickness and different coating architectures are possible (i.e. different layer materials, thicknesses and stacking order). To assess their mechanical response, coated samples may be subjected to indentation and scratch tests. This presentation will focus on the problems of measuring the plasticity and fracture properties of very thin coatings.

Tuesday 30th March, 09.40

Indentation size effects: is smaller harder or just more difficult?

Andy Bushby
QMUL

Indentation size effects are important for two reasons. Firstly, in materials behaviour to develop understanding of size dependent plasticity. Such understanding could enable length scale engineering in nanotechnology to exploit the increased strength. Secondly, in metrology to develop non-destructive methods for critical health monitoring of structures through reverse engineering of the stress-strain behaviour. The hardness size effect, for pointed indenters, is proportional to the inverse square root of the contact depth. Such effects are usually explained by strain gradient plasticity theory, with smaller indentations naturally creating high strain gradients. However, size effects with spherical indenters are proportional to the inverse cube root of indenter radius and do not fit well to strain gradient theory. Furthermore, metals and ceramics appear to show a dramatic difference in the magnitude of the size effect. Recently, all of these size effects have been shown to be proportional to the inverse square root of the contact size. Combining the indentation size effect and the Hall-Petch effect, by varying the grain size from much less than the contact size to much greater, the interaction gives a clue to the origin of both effects. Using a dislocation mean free path model, modified to incorporate an "effective length", all of these indentation size effects could be predicted. Furthermore, the materials parameters of yield strain and Burgers vector were found to drive the magnitude of the size effect and explain the different response of metals and ceramics. In recent experiments using asymmetric indenter shapes, it has been shown that the critical parameter is not, in fact, the contact size but the restriction on the initial plastic volume beneath the indentation site. Using this approach indentation size effects can be predicted for both metrology and length scale engineering.

Tuesday 30th March, 10.00

Size effects in microcompression of hard materials

Sandra Korte & Bill Clegg
University of Cambridge

Size effects are commonly observed in mechanical tests at the micrometre scale. Extensive experimental work has been reported in the literature describing size effects in soft metals where the yield or flow stress at the micron scale is related to the specimen dimensions. Experiments on harder materials such as bcc metals or semiconductors have shown that the size effect is less pronounced in these materials, but in contrast to fcc metals little data are available in the literature. This paper presents experimental data obtained on a wide range of materials in order to identify the relationship between bulk yield stress and magnitude of the size effect. It is found that the simple relationships used to describe the size effect in fcc metals are not sufficient and different mechanisms need to be considered if the origin of the effect of size is to be studied for materials other than soft metals.

Tuesday 30th March, 10.20

Size effects in diverse structural states of a Zr-based bulk metallic glass

Rejin Raghavan, A. Dubach, K. Boopathy, R. Ghisleni, M. A. Pouchon, J. Löffler, U. Ramamurty & J. Michler
EMPA

Bulk metallic glasses (BMGs) exhibit size-independent strength, as compared to their crystalline counterparts conforming the ‘smaller is stronger’ prediction. But, whether this behavior is consistent for all structural states of a given BMG is unknown. Hence, in-situ SEM micro-pillar compression and instrumented indentation techniques were employed to compare size effects in the mechanical response of various structural states of a Zr-based BMG. The as-cast state was altered by the defect concentration of the alloy known as the free volume content (vf), by structural relaxation (decreasing vf), shot peening (increasing vf) and ion implantation (increases vf). The yield strength in compression was found to be independent of the specimen size and also of the structural state of the material, except for the ion implanted (severely irradiated) alloy. The hardness, in contrast, was found to be sensitive to the structural state. In particular, states with higher free-volume content such as the shot-peened and ion-implanted states showed a decrease in hardness. In-situ SEM testing shows that though the formation and stable propagation of shear bands is the mechanism of deformation and failure in all cases, subtle but important distinctions can be identified in the states with higher vf. The advantages and limitations of in-situ SEM testing, which aid in enhancing the current understanding of deformation and fracture in amorphous alloys, are discussed in light of these observations.

Tuesday 30th March, 11.20

Deformation and fracture studies of thin films using miniaturized mechanical tests

Gerhard Dehm
University of Leoben

The continuous trend in miniaturization of materials requires novel strategies to probe the mechanical properties at the micron- and submicron level. Thin film structures like in microelectronic devices, sensors or cutting tools are examples were small scale mechanical testing methods are most promising to determine strength and failure mechanisms to guide the development of robust products. Focussed ion beam microscopy and lithography routes are employed to create specimens for quantitative deformation and fracture experiments at small length scales. Nanoindentation systems are used for loading and electron microscopy studies provide insight in the underlying deformation mechanisms. Results will be presented for metallic and dielectric materials. Some of the challenges encountered in small scale mechanical testing are discussed.

Tuesday 30th March, 11.40

Deformation and failure mechanisms of fibre-reinforced composites studied by in-situ mechanical testing and computational micromechanics

Jon Molina Aldareguia, Luis Pablo Canal, Marcos Rodriguez, Carlos González & Javier Llorca
IMDEA

The prediction of the mechanical behavior of fiber-reinforced composites is very complex due to the intrinsic anisotropy and complex damage mechanisms encountered in these materials. Recent developments in physically-based computational micromechanics have demonstrated that this is possible by the numerical simulation of the composite microstructure, which explicitly takes into account the spatial distribution of the fibers, matrix and interfaces. However, in order to perform the experimental correlation of the deformation and failure mechanisms predicted with such models, novel methods of mechanical testing of composites are needed at the microscale. With that purpose, we have used in-situ mechanical testing of composites under different loading modes at the SEM, coupled with the Digital Image Correlation technique (DIC) to compute the strain fields developed at the microscopic level. In this work, some examples will be shown and compared with the outcome of the micromechanical simulations.

Tuesday 30th March, 12.00

In Situ Investigation of Plasticity and Fracture at Small Scales

Oliver Kraft
University of Karlsruhe

Size effects on strength and plasticity of metallic materials have been studied now for more than 50 years. Prominent effects that have been reported range from Hall-Petch behavior to the critical thickness theory for thin films and the indentation size effect. In the last few years, so-called micro-compression test on sub-micron single-crystalline metallic pillars, prepared by focused ion beam (FIB), have become quite popular. The unexpected finding of a strong size effect in a nominally uni-axial loading situation has led to a debate about the physical deformation mechanisms. In the talk, recent results of micro-compression tests, including a comparison between fcc and bcc metals, will be presented and discussed also with respect to preparation artifacts and testing geometry, which may contribute to some of the observations in an undesired manner. Furthermore, we have made an effort in our work to conduct in situ tensile experiments on metallic nanowires in order to circumvent some of the experimental difficulties in the micro-compression tests. The tensile tests are conducted in a dual-beam scanning electron microscope and focused ion beam (SEM/FIB), where specimen manipulation, transfer, and alignment are performed using a manipulator and the FIB. Results shown will include tests on single-crystalline Cu and Au nanowires having diameters between 50 and 500 nm. Typically, fracture of the nanowires occurs locally without much homogeneous ductile deformation. Preliminary results indicate that the measured strengths show just a weak size effect. This behavior will be discussed in the context of a statistical analysis with respect to the specimen size.

Tuesday 30th March, 12.20

Fracture of ceramics at the microscale

Richard Todd
University of Oxford

This research concerns strength and toughness testing of ceramics by bend testing of cantilevered beams of length ~10 microns. The specimens are made using focused ion beams and the mechanical force and displacement measurements are provided by a nanoindenter. Although MEMS provides a direct application of the research, the primary motivation is to develop a better understanding of fracture processes in macroscopic specimens. One such process is the initiation of fracture. Since brittle fracture takes place from a single microscopic origin, whose location is not predictable prior to fracture, the micromechanisms occurring immediately prior to fracture are difficult to investigate. Testing of a microscopic specimen gives insight into such processes by contraining the region of fracture to the assumed scale of such processes. A second area of investigation is the toughness of grain boundaries. Grain boundary toughness is known to be tremendously important to macroscopic fracture and wear behaviour and to be influenced by grain boundary structure and composition. Direct measurement is difficult, however, because of the many other factors involved in macroscopic fracture. We are measuring grain boundary toughness directly, by making specimens containing isolated grain boundaries.

Tuesday 30th March, 14.00

Using a SFM/ESEM Hybrid for the Analysis of Vibrating Surfaces

Heinz Sturm
BAM - Federal Institute for Materials Research and Testing, Berlin

As often discussed, complementary SEM (Scanning Electron Microscopy) and SFM (Scanning Force Microscopy) analysis have a number of advantages. Both microscope systems support a huge number of techniques, but have also certain limitations. An SEM is capable of scanning large areas at very high speed and samples properties can be analyzed at different depth. An SFM is limited by scan area and speed, but supports techniques to determine mechanical, electrical, thermal and optical properties with highest resolution. The 3D dimensional shape of the topography can be observed directly. One obvious advantage in a combined system is: different complementary analysis can be made at the same sample position. Time consuming, sometimes impossible position adjustments of a sample in two separate instruments are omitted. Beyond this, there is an additional important aspect of hybrid systems. Both probes, the electron beam and the tip of the SPM, can be used simultaneously and it is worth to look at this opportunity in detail. In a hybrid system these interactions of one probe can be analyzed simultaneously by the other probe. Beside a characterization of such interactions, well-known probe-sample interactions can be deliberately used, allowing analysis of local sample properties, which could not be determined in separate instruments. In this paper investigations on vibrating SFM cantilevers are used to explain a special hybrid technique able to detect vibrations enforced at selected frequencies. The SFM cantilever is excited at its fundamental resonance frequency or at higher normal or torsional modes by the dither piezo of the SFM working at a fixed frequency. The cantilever is imaged in the SEM by scanning the electron beam using a secondary electron (SE) detector, the dynamic response is analyzed using a lock-in amplifier. The aim of the first investigations presented here is to study the dynamic deformation of a polymer surface excited either in normal or lateral direction by a SFM tip in contact under a certain load. Normal vibrations of the contact are used in SFM to study the stiffness of surfaces (e.g. by Force Modulation Microscopy – FMM or Constant Dynamic Indentation Microscopy – CDIM) [1, 2]. Lateral vibrations lead to shear deformation of the surface and reflect the tribological properties (Modulated Lateral Force Microscopy – MLFM) [3–5]. Although the deformation response of the contact is in three dimensions, only the displacement in normal and lateral direction can be addressed using a SFM. In this talk, first results are presented demonstrating the deformation of a surface excited by a vibrating SFM tip.

1. M. Munz, H. Sturm, E. Schulz, G. Hinrichsen, Composites A 29A (1998) p1251.
2. J. Chung, M. Munz, H. Sturm, Surf. Interface Anal. 39 (2007) p624.
3. H. Sturm, Macromol. Symp. 147 (1999) p249.
4. H. Sturm, E. Schulz, M. Munz, Macromol. Symp. 147 (1999) p259.
5. H. Sturm, Proc. Viennano ´07, Vienna, March 2007.

Tuesday 30th March, 14.20

Nanoindentation measurements of hydraulic permeability

Michelle Oyen
University of Cambridge

Characterization of the mechanical properties of hydrated tissues and gels is challenging, but of increasing interest in both commercial and medical applications. As such, there is a clear need for the development of characterization techniques that allow for the measurement of multiple facets of material behavior. Hydrated materials can be described as poroelastic: the mechanical response is described with a continuum two-phase model that incorporates fluid flow through a porous elastic solid. Contrasted with an empirical viscoelastic approach, poroelastic models contain information that is physically meaningful, such as a time constant that relates to an intrinsic length scale within the material. Although poroelasticity has been studied for many decades, the coupled nature of poroelastic constitutive equations results in a problem that is typically solvable only with numerical or computational models. Thus, poroelastic analyses have not been popular for routine data analysis. In the past decade, however, automated nanoindentation testing has allowed for the generation of large volumes of mechanical data, and the need for high- throughput data analysis for time-dependent materials has been established. In the current study, a new method is developed for high- throughput data analysis based on a master library of indentation creep curves for spherical indentation of a poroelastic material. The new scheme allows for rapid generation of constitutive parameters maps and has been shown to give quantitative measurements of permeability consistent with published values. Indentation tests are compared for hydrated equine bone samples and hydrogels tested at two length-scales, encompassing both nanoindentation and microindentation instruments. This study demonstrates the richness of information that arises from examining inelastic aspects of a tissue's nanoindentation response.

Tuesday 30th March, 14.40

Obtaining Viscoelastic Properties from Instrumented Indentation

Yang Tse Cheng
University of Kentucky

Instrumented indentation is widely used for the characterization of small-scale mechanical behavior of "soft" matters, such as polymers, composites, and biomaterials that exhibit viscoelastic behavior. Modeling instrumented indentation in linear viscoelastic solids has played an important role in developing methods for analyzing indentation measurements in these materials. In this presentation, we will discuss our recent results on modeling instrumented indentation in linear viscoelastic solids. We first examine the relationships between initial unloading slope, contact depth, and viscoelastic properties for various loading conditions, including load- or displacement-control. We then discuss several commonly used methods, such as the "hold-at-peak-load" and "hold-at-the-maximum-depth" techniques, as well as the constant indentation strain-rate method. We will also evaluate methods for obtaining storage and loss modulus using dynamic indentation using either spherical or pyramidal indenters. Finally, we will present a set of methods for measuring shear relaxation modulus and creep compliance using axisymmetric indenters of power-law profiles. These investigations may help improve instrumented indentation techniques for measuring viscoelastic properties of materials.

Tuesday 30th March, 15.00

Spherical nanoindentation to measure the elastic modulus and indentation yield point on biological materials

Ulrike Wegst[†], Amalie E. Oroho[†], Johann Michler[§], Siddartha Pathak and Surya R. Kalidindi[†]
[†]University of Drexel, [§]EMPA Thun

The success of biomimetic materials design depends on a precise knowledge of both the structure and the mechanical response at each structural level of the biological material hierarchy. To gain information on the mechanical properties at the nano- and microstructural level, a novel spherical nanoindentation technique can be applied that allows for the conversion of raw load-displacement data from depths as small as a few nanometers into much more meaningful indentation stress-strain curves. The method is based on continuous stiffness measurements (CSM) and relies on a new procedure for establishing both the effective zero-load and zero-displacement point in the raw dataset, and on a new definition of indentation strain. A comparison of the material properties measured with this technique on purely polymeric and mineralized biological materials with those obtained by in situ testing in FIB/SEM systems and macroscopically illustrates that spherical nanoindentation captures the material’s elastic modulus, indentation yield point, and post-yield characteristics.

Abstracts – Poster Presentations

Poster 1

Applications of Micro-Cantilever Testing

DEJ Armstrong, M Rogers, FW Herbert, TB Britton, AJ Wilkinson & SG Roberts
University of Oxford

We have developed test methods that use FIB-machined micro-cantilevers, which are then tested using a nanoindenter. These methods have been used to test elastic, plastic and fracture properties at the micron-scale: crystalline anisotropy of Young's modulus in copper, fracture toughness of selected grain boundaries in copper 0.02% bismuth, yield and flow behaviour as a function of size in single crystal copper, and in-situ testing of intergranular stress-corrosion cracking. The test methods (including possible pitfalls), results and their interpretation will be described.

Poster 2

An Investigation of the effect of moisture on the mechanical properties of polymers by depth sensing indentation

Ian Ashcroft, K. Altaf & R. Hague
University of Loughborough

SL (stereolithography) resins are highly hygroscopic and their mechanical properties are significantly affected by the level of moisture in the environment. In addition, the load response of these materials is highly time-dependant, hence, an appropriate viscoelastic or viscoplatic constitutive model is required to characterise their mechanical behaviour. In this work, the time dependent mechanical behaviour of an SL resin is investigated under varying humidity conditions using DSI (depth sensing indentation) tests. The potential advantages of this test method are that spatial variations in properties, which are often seen in SL parts, may be investigated and that by testing at different locations and depths the moisture distribution in the part may be determined. However, the drawback is in interpreting the data, which is complicated by the complex loading conditions, moisture and time dependent material response and moisture uptake behaviour. In the experimental study a Nano Test 600 indentation system fitted with a humidity control unit was used to explore the influence of moisture on the mechanical properties of the SL resin; Accura 60. Samples were tested with 45%, 65%, 75%, 85% and 95% relative humidity (RH) inside the chamber while the temperature was kept at 22.5˚C. It was seen that hardness and modulus decreased with increasing absorbed moisture in the resin whilst creep strain increased. After equilibrium conditions had been obtained the samples were tested in a drying environment to investigate recovery of the initial, dry properties. The parameters obtained through these tests were used to develop a coupled stress - diffusion finite element model incorporating a viscoelastic constitutive model. It is proposed that this model can be used in predicting the effect of the environment on the performance of SL manufactured components.

Poster 3

Elevated temperature (to 750˚C) and high strain rate nanomechanics for extreme applications

Ben Beake
MicroMaterials Ltd

During extreme machining applications such as high speed cutting significant heat is generated by friction in the cutting zone, and for interrupted contact (e.g. end and face milling) fatigue and fracture resistance is also important. Advances in the NanoTest enable nano-scale measurements to be made under conditions that closely mimic those in real contacts. Nanomechanical data are presented at elevated temperatures up to ~750˚C either quasi-statically (nanoindentation) or repetitively at high strain rate (nano-impact/impulse indentation). The nanomechanical test data show excellent correlation with field trials of coated cutting tools in high speed machining [1-8]. Advanced nanomechanical test methods will be more extensively used in the near future to speed up the pace of materials development for extreme applications.

1. G.S. Fox-Rabinovich et al, Surf. Coat. Technol. 200 (2006) 5738.
2. B.D. Beake et al, Surf. Coat. Technol. 201 (2007) 4585.
3. G.S. Fox-Rabinovich et al, J. Appl. Phys. 103 (2008) 083510.
4. G.S. Fox-Rabinovich et al, Surf. Coat. Technol. 202 (2008) 2985.
5. G.S. Fox-Rabinovich et al, Surf. Coat. Technol. 204 (2009) 489.
6. A.I. Kovalev et al, Vacuum 84 (2009) 184.
7. B.D. Beake et al, Surf. Coat. Technol. 203 (2009) 1919.
8. B.D. Beake et al, Chapter 6, CRC Handbook of nanostructured thin films and coatings, Ed. S. Zhang (2010) in press.

Poster 4

ESP – Enhanced Stiffness Procedure applied on soft or hard µm-coatings

Gottfried Bosch, Tanja Haas and Hans-Peter Vollmar
Fischer

The ESP Measuring Mode, which is based on the Instrumented Indentation Test, is explained in detail in the following. With the ESP Measurement, loading and unloading occurs in increments. This allows for a faster load- and depth-dependent determination of characteristic quantities such as EIT, HIT or HV at the same sample location. Multiple partial unload processes are added to the load cycles. Thus, several partial unloading plots are obtained in one test cycle. The characteristic quantities can be determined for each of these curves. No hardware change is required. The ESP Measuring Mode is an add-on in the software. The capability of the ESP method can be recognized when determining the characteristic material quantities of coating systems. This will be shown by examples of hard coatings like titanium nitrite on steel with a coating thickness below 3 µm. Also the characteristic quantities of soft lacquer coatings can be examined with this method. An indenter area function resulting from EIT measurements based to the ESP Mode is shown. It will be compared to an indenter area function calculated from measurements of the Martens Hardness.

Poster 5

Nanohardness of high purity Cu (111) single crystals: the effect of indenter load and prior plastic sample strain

Munawar Chaudhri[†], S. N. Dub[§] & Y. Y. Lim[§]
[†]University of Cambridge, [§]National Academy of Sciences of Ukraine

Experimental investigations have been carried out in which nanohardness of single crystals of Cu (111) samples containing prior plastic strains of 0, 0.06, 0.24, and 0.61 has been measured using a Berkovich diamond indenter of tip radius of ~ 200 nm. The projected contact areas of nanoindentations were determined using a calibrated AFM and these were used for determining the nanohardness values. It has been found that for every sample the nanohardness was the highest for the lowest indenter load of 0.625 mN. At the lowest applied indenter load, the overall highest hardness was obtained in crystals with the lowest prior strain of 0 or 0.06. At higher indenter loads, in the range 40 - 125 mN, the hardness increased with increasing prior plastic strain in the sample. A three-stage qualitative model, previously proposed by us, has been used for explaining the hardness versus indentation load data. In the first stage nucleation of dislocations under the indenter occurs. If the sample is well annealed, the nucleation of dislocations is homogeneous, but for the Cu (111) samples containing prior strains of 0.06, 0.24 and 0.61 the nucleation is heterogeneous. The highest hardness was obtained for a well annealed sample of the Cu (111) using a Berkovich diamond of tip radius of (407 ± 32) nm. The mean normal pressure, p_m, values at the moment of homogeneous nucleation of dislocations when the indenter was loaded on the Cu (111) and Cu (100) surfaces were determined as 16.75 and 9.32 GPa, respectively. From these p_m values the critical resolved shear stress of the copper single crystal has been determined as 4.56 and 3.80 GPa, respectively. The mean of these two values is 4.18 GPa, which is about 58% higher than the theoretically calculated ideal shear strength of copper of value 2.65 GPa. Young's modulus values along the normals to the Cu (111) and Cu (100) surfaces of well annealed and high purity copper crystals have been determined using the "pop-in" loads and the Hertzian elasticity theory. The values thus determined were 170 and 52 GPa for the Cu (111) and Cu (100) surfaces, respectively. These values are close to the literature values of 190.3 and 66.7 GPa. Young's modulus values were also determined using the unloading curves and the ISO 14577 method; these determinations were 135 and 110 GPa for the Cu (111) and Cu (100) surfaces, respectively; and are significantly different from the literature values, which indicates the inherent erroneous nature of the ISO 14577 method of nanoindentation data analysis.

Poster 6

Micro wear investigations of DLC coatings with nanometer resolution in normal and lateral direction

Thomas Chudoba
ASMEC GmbH

In the last years several techniques have been developed to investigate the wear of surfaces with nanometer resolution. This includes mainly the Atomic Force Microscopy in the lowest force range. However, in the higher force range between about 1 mN and 1 N there exist scarcely measuring techniques with nanometer resolution in the displacement measurement. In the other hand it is just in this load range especially interesting to investigate single asperity contacts with contact radii between 0.5 µm and 50 µm to investigate and understand the dominating wear mechanisms. This is now possible with the Universal Nanomechanical Tester of ASMEC, which has both in normal and lateral direction nanometer and micro-newton resolution during oscillatory wear experiments. The instrument was used for wear measurements on diamond like carbon coatings (DLC) of different hardness, produced by CVD and PVD methods. Two conical diamond indenters with spherical ends of 12 µm and 140 µm diameter and a hard metal sphere with 200 µm diameter have been applied. The oscillation length was 80 µm. By variation of the normal force it was investigated at which force and contact pressure limit wear starts and how the wear rate per cycle develops. It was found that only about one atomic layer is removed per slide and that there is no correlation between friction and wear rate.

Poster 7

Influence of mechanical properties on deformation behaviour of TiFeN, TiN and TiFeMoN films on Si in nano-scratch tests

John Colligon†, Vladimir Vishnyakov† & Ben Beake§
†Manchester Metropolitan University, §Micro Materials Ltd

TiFeN, TiN and TiFeMoN nanocomposite films were formed on Si using a dual ion beam system to vary deposition parameters and produce coatings with a wide range of mechanical properties. The coating hardness (*H*) and Young's modulus (*E*) were determined by nanoindentation and their tribological behaviour assessed by nano-scratch testing. High resolution scanning electron microscopy (SEM) of the nano-scratch tracks was performed to study the deformation behaviour in detail. The SEM images were correlated with the probe depth and frictional data used in the scratch tests to determine failure behaviour and assess how this varied with the mechanical properties measured by nanoindentation. The relation between the ratio of hardness to modulus (*H*/*E*) of the film and failure behaviour of TiFeN, TiN and TiFeMoN films was studied. Dramatic film failure behind or to the side of the moving indenter barely registered in the friction signal whereas oscillations were seen for failures in front of the indenter. The data provide further evidence that the combined mechanical properties of the film and substrate, rather than the adhesion strength, dominate the deformation behaviour in nano-scratch testing. Results for these nanocomposite films show there exists an optimum combination of hardness and toughness for applications where they could be exposed to high shearing forces. Similar trends have been observed in nano-scratch and nano-wear testing of Ti-Si-N [1] and hard amorphous carbon films on Si [2], implying that the behaviour is generic for hard thin films deposited on brittle, lower modulus, substrates.

1. B.D. Beake, V.M. Vishnyakov, R. Valizadeh and J.S. Colligon, J. Phys. D: Appl. Phys 39 (2006) 1392.
2. B. Shi, J.L. Sullivan and B.D. Beake, J. Phys. D: Appl. Phys 41 (2008) 045303.

Poster 8

Indentation Testing of Porous Materials

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Random porous solids, such as cement-based materials, shales, and bone, exhibit a multiphase composite nature, with features that span several orders of magnitude in length-scales, from the nm to the µm-scale. The most prominent heterogeneity of these systems is the pore space (ϕ) that ultimately defines their macroscopic mechanical performance for several safety-critical applications, ranging from multi-span bridges to calcium-phosphate bone replacement cements. Thus, there is an increasing drive to characterize the nano-/micro-mechanical response of porous media and understand their poro-mechanical coupling. Indentation testing of porous materials provides a framework for fine-scale mechanical characterization and model parameters identification for mechanical upscaling. In this work, a framework of indentation on porous media is proposed. Scaling functions of indentation modulus (M) - packing density ($\eta=(1-\phi)$), $M=M(\eta)$, and hardness (H) - packing density, $H=H(\eta)$, are developed. The scaling functions are obtained by (a) analytical homogenization (for the elastic range, M) and (b) computational yield design methods (for the plastic range, H). Experimental data on various porous solids (shales, cement-based materials, natural and synthetic tissues) are presented.

Poster 9

Using Indentation to Measure Residual Stresses in Surface Layers

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Equal biaxial residual stresses were generated in thin copper foils bonded to massive titanium substrates, by differential thermal contraction. The copper foils were then indented and the peak indentation load at maximum indenter depth was recorded as a function of the pre-indentation residual stress. Hardness data, also as a function of pre-indentation residual stress, were also obtained. Detectable decreases in the peak indentation load with increasing (tensile) residual stresses were observed, even for small changes in the pre-indentation residual stress. In contrast, no discernable change in the measured hardness data was obtained. These results are consistent with FEM predictions. Using an FEM model, and knowing the constitutive relation governing plastic deformation of the material, such nanoindentation data can be used to evaluate residual stress levels, with good sensitivity.

Poster 10

Ultra-High Strength Nanoporous Silver Produced by Inkjet Printing

Brian Derby, Rui Dou & Bojun Xu
University of Manchester

Silver, either as a nanoparticle suspension or a soluble salt, is the material of choice for inkjet printing conductive tracks on a range of flexible substrates, because of its ability to be heat treated under ambient atmospheric conditions to produce a dense, high conductivity, metallic deposit. However, fully dense metal films fail at relatively low plastic strains and this may make them incompatible with compliant substrates as required for flexible electronic applications. Here we explore the mechanical properties, using nanoindentation, of low density part sintered nanoporous silver films deposited by inkjet printing and show that high strength and low elastic modulus tracks can be printed with reasonable electrical conductivity.

Poster 11

Investigation of creep and time dependent properties of elastomers and polymers

Quynh H. Duong, Jiri Nohava & Philippe Kempe
CSM Instruments

Characterization of soft materials such as elastomers and polymers by indentation has always been a difficult task as it requires instruments with perfect thermal stability. The most important source of error is a phenomenon called thermal drift i.e change in displacement signal while constant load is applied on thermally stable material. Indentation studies of viscoelastic properties of soft materials have therefore been performed using the Ultra Nanoindentation Tester (UNHT), an indentation system with extremely low thermal drift. The experiments were carried out in a force controlled mode with a Berkovich indenter. The materials were selected from hard polymers to elastomers in order to cover a large range of viscoelastic properties. Different parameters were investigated: maximum loads, loading times and loading rates. The results show that mechanical properties of soft materials can be characterized using the UNHT despite a long hold time and well estimated even by Berkovich indentation using Oliver-Pharr approach.

Poster 12

Isothermal contact nanoindentation in a controlled environment up to 750˚C

Nicola Everitt & M.I. Davies
University of Nottingham

High temperature nanoindentation is particularly useful since it may be possible to obtain hardness (plastic yield) and modulus (elastic deformation) values at in-service temperatures of small sample volumes. However care must be taken to obtain reliable and repeatable data. Isothermal contact nanoindentation has been performed up to 750˚C on a variety of bulk solid and coated materials in a chamber which can be Argon purged. Indentations on fused silica produced values of modulus which compared well with those produced by acoustic methods. Indentations on hard coatings showed a difference in performance ranking at high temperature compared to that at room temperature.

Poster 13

Micro- and nano-tribology experiments with a novel in-situ test system

Mark Gee, John Nunn & Andres Muniz Piniella
NPL

A new tribometer has been designed to carry out micro and nanoscale tribological experiments. The system has been designed for both use on the laboratory bench and in-situ in a scanning electron microscope. The main requirement that the system addresses is to evaluate the tribological response that occurs at the asperity level in macroscale tribological contacts so that a better understanding can be developed so that reliable prediction of performance for engineered sur-faces can be made.through improved modelling. A secondary aim is to evaluate the tribological response of system elements in MEMS and NEMS devices.The test system has been designed so that it can either be used as a bench top system or an SEM In-situ sys-tem. Bench top experiments enable tests in air or other controlled atmospheres. In-situ experiments enable high resolution near real time experiments to be carried out where the build up of damage in samples can be ex-plored with high resolution imaging and analysis car-ried out at frequent intervals during a multipass ex-periment without removal of the sample from the SEM. A range of reciprocating tests using 2 mm steel balls and 1 micrometre radius diamond indenters has been carried out on a range of coatings including hard ce-ramic wear resistant coatings and low friction carbon coatings.

Poster 14

Characterisation of Free Volume in Amorphous Metals by Nanoindentation

Russell Goodall, John Plummer & Iain Todd
University of Sheffield

Free volume, or 'empty space' is a characteristic structural feature of amorphous solids, frozen in by cooling the melt below the glass transition temperature. It is believed that this influences the propensity for plastic flow in amorphous metals, as it determines the extent and ease of local atomic rearrangements, enabling plastic flow. During the production of these materials, rapid cooling is required in order to bypass the favourable kinetics for crystallisation, potentially leading to a spatial distribution of free volume within the sample, as a result of differing cooling rates experienced by the centre and edge regions. In order to probe the influence of the structure on mechanical behaviour in samples where the structure differs over short distances, micromechanical testing is needed. Here it is investigated whether nanoindentation is a suitable technique to characterise free volume in as-cast cylindrical rods of various bulk glass forming alloys, and whether such a distribution does indeed exist.

Poster 15

Do the individual pillars of a structured compliant surface communicate with each other?

Griselda Guidoni
INM Leibniz Institut

A common method to characterize adhesion is to determine the force required to pull-off a flat or spherical probe which is in adhesive contact with the surface. Here, we focus on the approach into contact instead on the pull-off response, which minimizes damping and viscoelastic effects. A structured PDMS fibrillar surface on a soft backing layer of the same material was studied. By means of a nanoindenter, we approach a spherical sapphire indenter, from a non-contact/non-interaction distance to the PDMS sample with constant speed. Load and displacement data were recorded both during approach and retraction. The first contact always results in a sudden tensile force, evidencing attractive forces between indenter and pillars. Contact between probe and each individual pillar is detected via sudden drops in the force vs. displacement reading. The experimental results were modeled considering the influence of the backing layer on the total deformation of the system. The backing layer was found to contribute 30% to the total deformation of the first pillar in contact. The combination of model and experiment allows us to obtain an in situ value of the elastic modulus of the fibril material.

Poster 16

Thin film interfacial adhesion studies by use of nanoindentation

Ude Hangen[†] & Florian Pape[§]
[†]Hysitron Inc., [§]University of Hannover

The interfacial adhesion of thin DLC films on steel substrates is difficult to characterize. For films with intrinsic stresses or films that are not well bonded the delamination occurs with a sudden event that results in an excursion on the loading curve. But delamination often occurs even though there is no clear signature on the loading curve. When imaging the indentation cup a pile-up type structure can be found around the indentation cup. This pile-up can either be caused by localized plastic deformation or by subsurface interfacial cracking. In order to gain more information nanoindentation needs to be combined with complimentary techniques. In this paper we show how the acoustic emission detection can be utilized to detect the interfacial cracking during an indentation experiment. The extent of delamination (area and spatial distribution) can then be characterized by modulus mapping. The derivation of interfacial adhesion properties from the measured values will be discussed.

Poster 17

Compression Splitting of Micropillars

Philip Howie & Bill Clegg
University of Cambridge

If micropillar compression is to be used as a technique for studying plastic flow in brittle materials, it is important that the conditions under which fracture occurs are understood. Using results from in-situ microcompression testing, it is shown that brittle failure of micropillars can occur by compression splitting. The mechanisms by which this happens are described using conventional fracture mechanics and it is shown that analyses for compression splitting in this way naturally yield a size-dependent fracture stress.

Poster 18

Enhanced Yield strength of materials - the "Thinness Effect"

Nigel Jennett[†], R. Ghisleni[§], X.D. Hou[†] & Johann Michler[§]
[†]NPL, [§]EMPA, Thun

In this presentation we show that yield strength is determined by 'thinness', i.e. by the smallest dimension of a structure. Thus, although 'smaller is stronger' thin metal structures exhibit the same enhanced yield strength regardless of total size (volume) or surface to volume ratio; this implies that nano-layers are as strong as nanoparticles as only the thinnest dimension in a material needs to be reduced to obtain improved properties.

Poster 19

Kleindiek nanotools for fine scale mechanical characterization

Stephan Kleindiek
Kleindiek Nanotechnik

Versatile Nanotools such as gripper, force and current sensor, and injector for easy characterization in the electron or ion microscope are presented. Applications range from nanomanipulation, nanoindentation, tensile testing and nanoforging to sheet resistance measurements.

Poster 20

In-situ electron backscatter diffraction (EBSD) during the compression of micropillars

Johann Michler, C. Niederberger, W. M. Mook & X. Maeder
EMPA, Thun

For the first time, in-situ electron backscatter diffraction (EBSD) measurements during compression experiments on micron sized pillars are demonstrated here. The experimental setup and the requirements concerning the compression sample are described in detail. EBSD mappings have been acquired before loading, under load and after unloading for consecutive compression cycles on a focused ion beam (FIB) milled GaAs micropillar. In-situ EBSD allows for the determination of crystallographic orientation at high spatial resolution. Thereby, it provides highly localized information pertaining to deformation phenomena such as elastic bending of the micropillar or the formation of deformation twins and orientation gradients due to geometrically necessary dislocations. The most striking features revealed by in-situ EBSD is the non-negligible amount of reversible (elastic) bending of the micropillar and the fact that deformation twinning and dislocation glide initiate where the bending is strongest. Due to this high spatial and orientation resolution, in-situ EBSD measurements during micromechanical testing are demonstrated to be a promising technique for the investigation of deformation phenomena on the sub-micron scale.

Poster 21

Erosion and the Role of Fluoride: A Nanomechanical Study

Charlie Parkinson
CSK

Nanoscope IV AFM, operating under standard conditions and in accordance with published operating procedures, were employed to explore the mechanical properties and topography of enamel as a function of treatment and subsequent acid challenge exposure time. Treatments consisted of (i) no treatment, (ii) human saliva (2 minute maturation time with dH_2O rinse) or (iii) 2 minute exposure to 1000 ppm fluoride (sodium fluoride) followed by saliva as per (ii). Following pre-treatment, the surfaces were exposed to 3x10-2M citric acid, pH3.8 for exposure times of 5 and 10 minutes, without agitation, at room temperature. Results: Fluoride and saliva are observed to inhibit near-surface (top 300 nm) softening of enamel. Complementary AFM images reveal fluoride and saliva inhibit enamel surface dissolution. Fluoride is observed to inhibit the dissolution of the c-axis of enamel rods. In the absence of either saliva or fluoride, enamel dissolution is initiated at the rod core/wall interface and preferentially at defects inherent within the enamel surface. Conclusion: Utilising a combination of AFM and nanoindentation, it has been shown that fluoride and saliva confer protection to the structural integrity of enamel against the deleterious effects of an aetiologically-relevant acid challenge.

Poster 22

Effect of pre-straining on the size effect in molybdenum pillars

Andreas Schneider[†], B.G. Clark[†], C.P. Frick[§], P.A. Gruber[*] & E. Arzt[†]
[†]INM Leibniz Inst. Saarland, [§]University of Wyoming, [*]University of Karlsruhe

The effect of prior deformation on mechanical behavior as a function of size is investigated for body-centered cubic (bcc) molybdenum (Mo) pillars. Experiments were performed using focus ion beam (FIB) manufactured [001] and [235] Mo micro/nanopillars, which were compressed, re-FIB machined, and compressed again. Unlike bulk materials, pre-straining has a negligible effect on stress-strain behavior of the pillars, suggesting that dislocation storage does not occur in small-scale bcc specimens. The prevailing mechanism behind the size effect is attributed to dislocation nucleation mechanisms.

Poster 23

Pharr's concept of the effectively shaped indenter extended to creep and viscous material behavior

Norbert Schwarzer
Saxonian Institute

There is a relatively great variety of nanoindentation results to be found in the literature, where there are illogic exponents from the exponential fit to the unloading curve. Apparently, it is not sufficiently known, that exponents bigger than 1.5 are questionable from the physical point of view. Especially in the case of materials showing significant creep, this sort of problematic results are very likely to appear. Why is this? The answer can easily be given by the means of Pharr's concept of the effectively shaped indenter. Interestingly, the author found many such strange analyzing results, where, by taking the time-dependency of the measurement into account, all such "funny" results disappeared. In the poster it will be demonstrated how the classical Oliver and Pharr method can be extended to "creepy" materials and how this method can be connected with the effective indenter concept mentioned above.

Poster 24

Small-scale Polymer Analysis using Sample Oscillation

J.F. Smith[†], S.R. Goodes[†], M. Tehrani[§] & M. Haik[§]
[†]Micro Materials Limited, [§]University of New Mexico

Instrumented indentation has been used for some time to probe the surface properties of viscoelastic materials. This generally involves superimposing a small sinusoidal oscillation on the background loading ramp during continuous linear loading. Loss and storage modulus values can be extracted by relating the variation in applied force to the consequential indenter motion. This report discusses an alternative approach in which the sample is oscillated in a similar manner to conventional DMA (Dynamic Mechanical Analysis). We discuss results obtained from different epoxy specimens in which loading is interrupted periodically to minimise creep during data acquisition. Two analytical procedures are compared: (1) a method in which the applied load amplitude is not required and average properties are obtained over the indented volume, and (2) a method which uses a calculated force amplitude to give variations in properties with depth.

Poster 25

Influence of crystal orientation on scratch testing with a spherical indenter

Greg Swadener[†], Heidi Bögershausen[§], Benedict Sander[§] & Dierk Raabe[§]
[†]Aston University, [§]MPI for Ferrous Research Düsseldorf

Spherical scratch tests were conducted in individual grains of a randomly oriented polycrystalline bcc Ti-Nb alloy. For each grain, scratch tests were conducted at four different levels of normal load, which resulted in varying amounts of plastic strain during indentation. The results show a dependence of the horizontal load component on the crystallographic orientation and on the amount of plastic strain. The component of the horizontal force that resulted from plastic deformation was found to correlate with the active slip systems for the particular grain orientation.

Poster 26

Relating Mechanical Properties and Chemical Bonding in Hybrid Framework Materials

Jin-Chong Tan
University of Cambridge

Crystalline hybrid framework materials that incorporate both inorganic and organic building blocks are attracting considerable attention, because their enormous chemical and structural diversity offers opportunities for creating many technologically relevant properties [1]. The 2 major categories of hybrid framework materials are: (a) nanoporous hybrids (pore size roperties are now available, hitherto reports relating to their mechanical characteristics are scarce. In view of the diversity of hybrids, which ranges from chains to sheets to 3-D frameworks and includes both organic and/or inorganic connectivities, their mechanical properties have the potential to exhibit a richness of behaviour that cannot be obtained in purely inorganic or organic systems alone. In addition, knowledge of their capacity to withstand elastic and plastic deformation is vital for all applications envisaged for this new class of material. We have synthesized single crystals of several prototypical inorganic-organic frameworks of different dimensionalities, and of dense and nanoporous architectures. The availability of high quality single crystals of sufficient size provided us with the interesting opportunity to perform detailed nanoindentation studies to unravel their mechanical behaviour [2-3]. Their elastic and plastic anisotropy, the onset of plasticity and the fracture toughness anisotropy have been characterised along the primary crystallographic orientations. In this talk, I will demonstrate how the anisotropy of their mechanical properties can be correlated with the intrinsic crystalline structures of different dimensionalities. Experimental studies are accompanied by atomistic modelling work (Density Functional Theory calculations) to predict the elastic constants of different architectures and to gain insights into the roles of the fundamental building blocks.

1. Cheetham, A.K. and C.N.R. Rao, *There's room in the middle*. Science, 2007. 318(5847): p. 58-59.
2. Tan, J.C., J.B. Orton, C.A. Merrill, and A.K. Cheetham, *Anisotropic Mechanical Properties of Polymorphic Hybrid Inorganic-Organic Framework Materials with Different Dimensionalities*. Acta Materialia, 2009. 57(12): p. 3481-3496.
3. Tan, J.C., J.D. Furman, and A.K. Cheetham, *Relating Mechanical Properties and Chemical Bonding in an Inorganic-Organic Framework Material: A Single-Crystal Nanoindentation Study*. Journal of the American Chemical Society, 2009. 131(40): p. 14252-14254.

Poster 27

Cantilever-based Nanoindentation for the Characterization of Thin Films and Soft Materials

Philipp Thurner
University of Southampton

The interest of the research community to characterize mechanical properties of samples with either low thickness or low elastic modulus is steadily increasing. Samples of interest comprise thin films providing wear or scratch resistance, anti-fouling properties etc. as well as soft biological tissues. Current state-of-the-art nanoindentation systems are often pushed to their limits, if either indents to few tens of nanometers or peak forces of a few micronewtons are required. An alternative is the use of cantilever-based nanoindentation using an atomic force microscope (AFM). In contrast to rather well-defined nanoindentation tips, AFM tips at the end of cantilevers are consumables and can possibly vary substantially. To overcome this issue we have developed a protocol to reconstruct the tip shape from imaging of a calibration sample consisting of sharp Si spikes (TGT-1, NT-MDT) and numerical deconvolution of the image using a custom algorithm. From this image a look up table for the projected area of the tip vs. indentation depth is generated. Indentation data is then analyzed using the Oliver-Pharr method together with this table. All imaging and force measurements were conducted using an MFP3D AFM (Asylum Research). Initial validation tests conducted on polymer samples of low-density polyethylene (LDPE), polytetrafluoroethylene or Teflon (PTFE) and polypropylene (PP) with peak forces of 2 µN and indentation depths around 100 nm yield results for elastic moduli very close to ones given by the manufacturer, i.e. ELDPE=0.42 GPa (EManufacturer=0.1-0.3 GPa), EPTFE=0.53 GPa (EManufacturer=0.3-0.8 GPa) EPP=1.08 GPa (EManufacturer=0.9-1.5 GPa), indicating that this approach is feasible. In an ongoing project we aim to apply this technique for the characterization of the mechanical properties airway vessel wall tissue. The rationale of this study follows a current hypothesis stating that persistent Asthma originates from airway walls that have become so stiff that they cannot be sufficiently dilated for normal breathing anymore.

Poster 28

Nanohardness cartography: influence of the dendritic/interdendritic chemical composition on the mechanical properties of a MCNG superalloy

Christophe Tromas[†], M. Arnoux[§], X. Milhet[§] & J. Mendez[§]
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Single crystal nickel-based superalloys are widely used as material for the hottest parts of aircraft engines. Their superior mechanical properties results mainly from their microstructure, consisting in coherent γ' precipitates embedded in a cubic face-centred γ matrix. In order to improve their high temperature capabilities, an increasing amount of refractory elements such as Rhenium (Re) and Ruthenium (Ru) were added the fourth generation of alloys such as MCNG. However, even after the standard heat treatments, these alloys exhibit chemical heterogeneities resulting from the solidification process by dendritic growth. In particular, Re is more concentrated in the dendritic regions. This may results in a difference in mechanical properties at a mesoscopic scale, likely to impact the global mechanical behavior of the material. In order to investigate the effect of this difference in chemical composition between the dendritic and interdendritic regions, large regular arrays of 1000 nanoindentations, with a 20 µm step size were performed on an undeformed sample. A 50 mN load was applied for each indent to obtain a large indentation size compared to the γ/γ' microstructure but small enough compared to the dendritic/interdendritic regions. The statistic analysis on the nanoindentation measurements reveals a bimodal hardness distribution, with respectively 5410 MPa and 5550 MPa mean hardness values. Since these two peaks are very close, a nanohardness cartography was established thanks to the regular nanoindentation array, and compared to the dendritic/interdendritic regions observed by scanning electron microscopy (SEM). The very good correlation between the SEM images and the nanohardness cartography allowed to unambiguously ascribe the bimodal hardness distribution to the dendritic and interdendritic regions. Even the secondary dendritic arms are revealed by the nanohardness cartography. The same analysis was performed after a 50 h isothermal creep at 1050°C / 140 MPa. The results are discussed in view of the microstructure in the dendritic and interdendritic regions observed by SEM and atomic force microscopy.

Poster 29

SEM integrated force and resonance measurements of focused-electron-beam-induced Cu/C nanowire deposits

Ivo Utke, Vinzenz Friedli & Johann Michler
EMPA, Thun

Bending and vibration tests performed inside a scanning electron microscope were used to mechanically characterize Cu/C high-aspect pillars grown by focused electron-beam-induced deposition. Supported by finite element (FE) analysis the Young's modulus was determined from load–deflection measurements using cantilever-based force sensing and the material density from additional resonance vibration analysis. The pillar material consisted of a carbonaceous matrix which embeds 5–10 at.% Cu deposited at 5 and 20 keV primary electron energy and 100 pA beam current, depending on primary electron energy. The Young's moduli of the FEB deposits increased from 17 ± 6 to 25 ± 8 GPa with increasing electron dose. The density of the carbonaceous matrix shows a dependence on the primary electron energy: 1.2 ± 0.3 g cm-3 (5 keV) and 2.2 ± 0.5 g cm^{-3} (20 keV). At a given primary energy a correlation with the irradiation dose is found. Quality factors determined from the phase relation at resonance of the fundamental pillar vibration mode were in the range of 150–600 and correlated to the deposited irradiation energy.

Poster 30

The relation between microstructural scale and the influence of porosity and second phases on hardness

Luc Vandeperre, Jiahye Wang & Naeem U-Rheman
Imperial College, London

Experimentally, hardness measurements are an extremely convenient tool for studying plasticity in ceramics. The interpretation of the hardness in terms of for example a critical resolved shear stress is more complicated than in uni-axial tests on single crystals. However, there is a wide body of evidence that sensible estimates for flow stresses can be made from hardness measurements. It is however recognised that in sintered ceramic microstructures other effects such as secondary phases and pores can influence the measured hardness strongly. Hence understanding the effect of microstructural scale on the measurement of properties of the constituent phases needs to be understood. The results of theoretical predications will be contrasted with experimental results for 2 case studies: (i) the influence of pore volume fraction and pore size on the measurement of the elastic and plastic properties of SiC, and (ii) the effect of grain size on the measurement of the elastic and plastic properties of the constituent phases of a zirconium diboride composite containing silicon carbide and boron carbide inclusions.

Poster 31

AFM Observation of Diamond Indenters after Oxidation at Elevated Temperatures

Jeff Wheeler†, Rachel Oliver§ & Bill Clyne§
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Use of diamond indenter tips at elevated temperatures can cause oxidation and thermomechanical damage, leading to changes in their topography. A Berkovich diamond indenter has been exposed to 450°C in air, followed by 750°C and 900°C in 1 bar of static, commercial purity argon (30-45 ppm O_2). The effects of oxidation on the geometry of the indenter were investigated using atomic force microscopy. A Berkovich and a 10 μm tip radius conospheroidal indenter were also examined, after being subjected to 5 years of intermittent use at elevated temperatures (≤ 400°C). Significant changes in tip topography were observed, suggesting that commercial purity argon may be an unsuitable atmosphere for high temperature indentation testing. Finally, a mechanism of oxidative etching, which may have potential as a method of sharpening indenters, is also reported.

Poster 32

Deformation and fracture behaviour of silicon carbide under indentation

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University of Manchester

Although the SiC produced by chemical vapour deposition has been extensively used as structural ceramics and abrasive materials, little is known about its microscopic deformation and fracture behaviour under indentation. In the first part of this work, we examine the deformation and cracking behaviour of 3C-SiC under indentation by using both indentation technique and transmission electron microscopy (TEM). Under indentation, SiC first deform elastically, then abrupt burst or pop-in of displacement was observed at indentation depth of 60 nm. After then, the SiC deformed plastically with further indentation. TEM examination of indented regions suggests that shear stresses caused both dislocation formation and crack propagation in the SiC. The reason for such deformation behaviour was explained with comparison of SiC bonding strength and stresses generated under indentation . In the second part of this work, we report the anisotropic fracture toughness of CVD SiC. A micro-beam of SiC has been produced with use of focused ion beam, then the beam was bended with the use of nano-indentation, which allow us to measure the fracture toughness of SiC at different orientation. The fracture toughness of SiC along the CVD growth direction was found much higher than that of SiC at orientation vertical to the growth direction, which is due to texture structure of the SiC produced with the CVD process.