CAMTEC IV

Symposium on Fine-Scale Mechanical Characterisation and Behaviour

9th-10th April, 2018

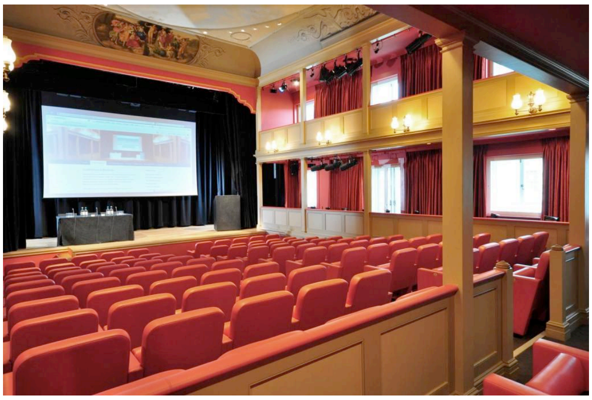
The Gordon Laboratory

The CAMTEC series

The first 3 Symposia in the CAMTEC series were held in April 2006, March 2010 and April 2014. They have all been in Cambridge, the first two in Downing College and the third in the Materials Science Department. CAMTEC IV will take place in April 2018, reverting to Downing as the venue. The main focus is on various types of indentation process, although other types of (fine scale) mechanical testing procedures and results are also covered.

CAMTEC IV

This Symposium will take place on 9th - 10th April, 2018. It will be chaired by Bill Clyne, James Dean and Bill Clegg (all of the Materials Science Department in Cambridge). The main venue will be the Howard Lecture Theatre, where there is 160 m^2 of exhibition space on the ground floor, with adjacent catering facilities, and a 100-seat auditorium (right).



All presenters have been personally invited. There will be 63 delegates, including 15 representatives of organisations participating in the Industrial Exhibition. There will be 28 talks (all 20 minutes, including a few minutes for questions), divided into 5 themed sessions. In addition, there will be 18 posters, constituting a prominent element of the meeting, on display throughout, in the area where breaks and lunches will be taken and the Industrial Exhibition held (upper right). The meeting will start with lunch on the Monday (9th) and finish on the Tuesday afternoon, with the Symposium dinner being held on Monday evening. Overnight accommodation (lower right) is available in the College. There will be a session of short talks by poster authors. Poster prizes will be awarded, on the basis of delegate voting. These prizes will be presented at the end of the Symposium.

The Oral Programme

Monday 9th April

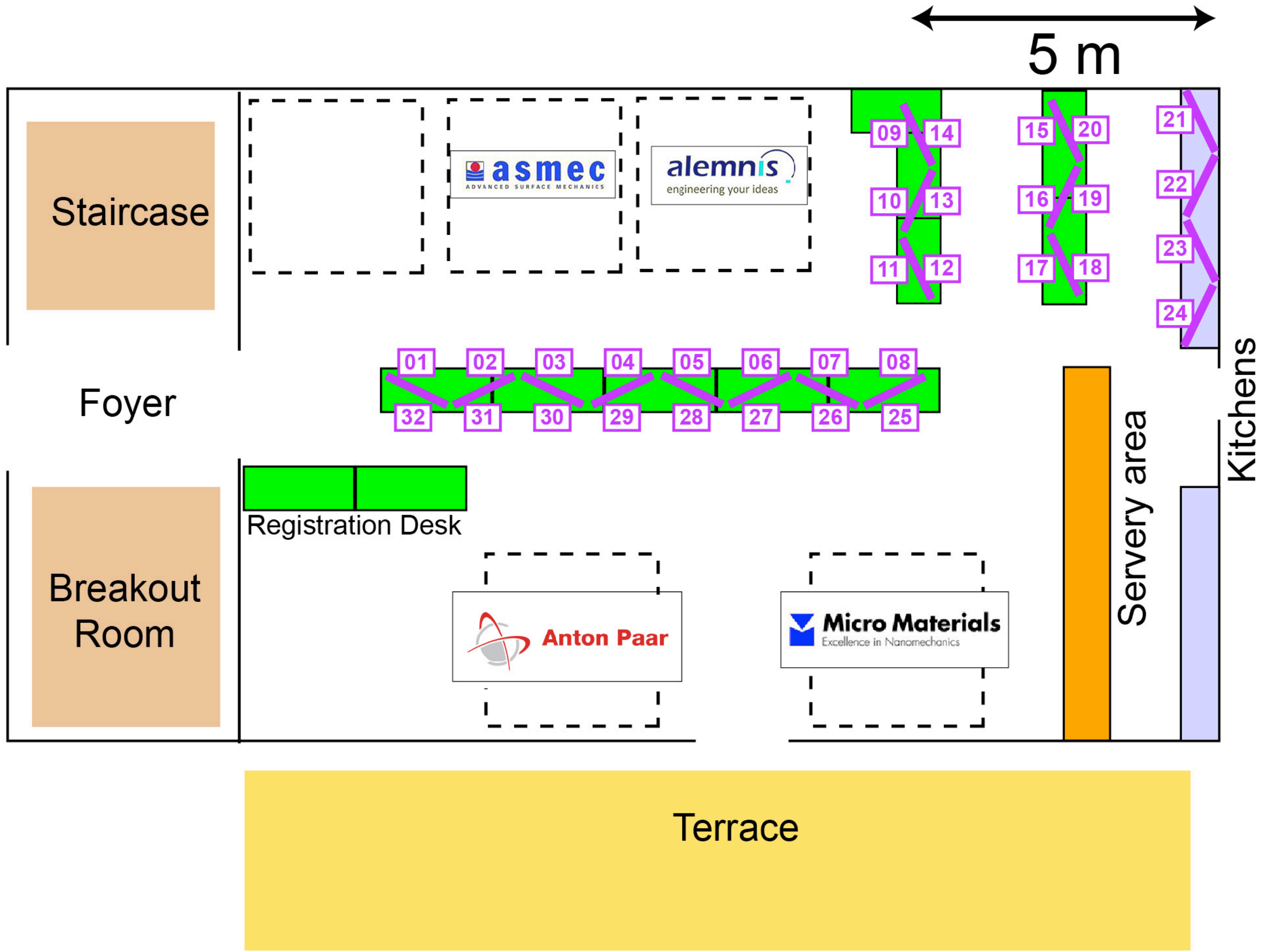
Time	No.	Presenter	Affiliation	Title
09.00-12.30	**Registration / Setting up of Exhibition and Posters**			
12.30-13.30	**Lunch**			
13.30-15.30	Session 1: **Novel Test Configurations and Conditions** (Chair: James Dean)			
13.30-13.50	**1**	Sandra Korte-Kerzel	RWTH Aachen	*Small-scale Deformation at High Rates - Non-Newtonian flow to the Theoretical Strength at Room Temperature from Metallic to Silica Glasses by Impact Nanoindentation*
13.50-14.10	**2**	Nick Randall	Anton Paar, Switzerland	*High Temperature Nanoindentation up to 800˚C for Characterizing High Temperature Properties of Materials*
14.10-14.30	**3**	Johann Michler	EMPA Thun	*Recent Advances in In-situ SEM Nanomechanical Testing: Cryogenic Temperature, High Strain Rates, In-situ EBSD Experiments*
14.30-14.50	**4**	Ben Beake	MicroMaterials UK	*Exploring the Temperature Dependence of Indentation Size Effects, Strain Rate Sensitivity and Pile-up in Polycrystalline Tungsten to 950˚C*
14.50-15.10	**5**	Ruth Schwaiger	Karlsruhe Inst. Technology	*High-temperature Nanoindentation of bcc Metals and Alloys*
15.10-15.30	**6**	Russell Goodall	Sheffield U.	*Shear Banding Behaviour of Bulk Metallic Glasses through Dynamic Electrical Measurements*
15.30-16.00	**Tea / Exhibition / Posters**			
16.00-17.40	Session 2: **Extraction of Bulk Properties from Fine Scale Testing** (Chair:Johann Michler)			
16.00-16.20	**7**	Gunther Eggeler	Bochum U.	*In-situ SEM Micro Shear Testing of Engineering Materials*
16.20-16.40	**8**	Gaurav Mohanty	EMPA Thun / Alemnis	*In-situ Microcompression Transient Plasticity Tests for Reliable Extraction of Deformation Activation Parameters: Creep, Stress Relaxation and Strain Rate Jump Tests*
16.40-17.00	**9**	Verena Maier-Kiener	Leoben U.	*Essential Refinements of Spherical Nanoindentation Protocols and a Multi-sharp Tip Approach for the Reliable Determination of Mechanical Flow Curves*
17.00-17.20	**10**	James Dean	Cambridge U.	*The SEMPID (Software for the Extraction of Material Properties from Indentation Data) Package for Plasticity*
17.20-17.40	**11**	Jeff Wheeler	ETH Zurich	*Combinatorial In Situ Micromechanics of the Al-Cu System at Low Temperatures*
17.40-18.00	**12**	Guénhaël Le Quilliec	Tours U.	*Identification of Material Properties by Instrumented Indentation using Manifold Approach*
18.00-18.40	**Oral Presentation of Posters** - 2 minutes per poster (Chair:Bill Clegg)			
18.40-19.00	**Free**			
19.00-19.30	**Reception in Senior Combination Room**			
19.30-22.00	**Symposium Dinner in Hall**			

Tuesday 10th April

Time	No.	Presenter	Affiliation	Title
08.00-09.00	**Breakfast**			
09.00-11.00	Session 3: **Interfacial Adhesion, Fracture & Toughness** (Chair: Gunther Eggeler)			
09.00-09.20	**13**	Megan Cordill	Leoben U.	*Stressed Overlayers to Assess the Adhesion of Brittle Film Systems via Nanoindentation*
09.20-09.40	**14**	Bill Clegg	Cambridge U.	*Toughening Non-metallic Crystals by Inhomogeneous Elasticity*
09.40-10.00	**15**	Ivo Utke	EMPA Thun	*Adhesion Investigations of ALD Films on Metal Substrates*
10.00-10.20	**16**	Dave Armstrong	Oxford U.	*Micro-mechanical Measurements of Interface Failure in Ceramic-ceramic Composites*
10.20-10.40	**17**	Finn Giuliani	Imperial Coll.	*In Situ Stable Fracture of Ceramic and Metal Ceramic Interfaces on the Micron Scale*
10.40-11.10	**Coffee / Exhibition / Voting for Poster Prizes**			
11.10-13.10	Session 4: **Microstructural and Mechanistic Effects** (Chair: Megan Cordill)			
11.10-11.30	**18**	Helena Van Swygenhoven	PSI Villigin	*Phase Transformations during Biaxial and Strain Path Changing Loadings : In-situ HR-DIC (SEM) and In-situ Synchrotron X-ray Diffraction*
11.30-11.50	**19**	Jon Molina	IMDEA Madrid	*Dislocation Propagation across Lamellar Boundaries in Fully Lamellar TiAl Alloys: Insight from Micro-tensile Experiments*
11.50-12.10	**20**	Jin-Chong Tan	Oxford U.	*AFM Nanoindentation to Quantify the Mechanical Behaviour of Nano- and Micron-Sized Hybrid Crystals*
12.10-12.30	**21**	Christophe Tromas	Poitiers U.	*Portevin–Le Chatelier Effect in Aluminium Alloys: A Nanoindentation Study at Room and Elevated Temperature*
12.30-12.50	**22**	Christian Greiner	Karlsruhe Inst. Technology	*The Origin of Microstructural Discontinuities underneath a Tribologically Loaded Surface*
12.50-13.10	**23**	Steve Bull	Newcastle U.	*Chemo-Mechanical Effects in Thin Film and Bulk Oxides*
13.10-14.00	**Lunch**			
14.00-15.40	Session 5: **Indentation of Soft / Compliant (Bio)Materials** (Chair: Bill Clyne)			
14.00-14.20	**24**	Amaia Cipitria	MPI Colloids & Interfaces, Berlin	*Dual Crosslinked Alginate Hydrogels with Patterned Mechanical Properties and Biomolecule Presentation*
14.20-14.40	**25**	Dave MacManus	Univ. College Dublin	*High Rate Indentation of Brain Tissue*
14.40-15.00	**26**	Andy Bushby	QMUL	*Determining the Mechanical Behaviour of Individual Hydrogel Microspheres with a Nanoindenter*
15.00-15.20	**27**	Florian Lacroix	Tours U.	*Influence of Vulcanisation and Aging on Local Properties of Polychloroprene Rubber*
15.20-15.40	**28**	Shahrouz Amini	MPI, Berlin	*Quasi-Plasticity and Shear-Induced Cracking in Contact-Damage Resistant Biological Models*
15.40-16.30	**Tea / Exhibition / Presentation of Poster Prizes / Group Photograph**			

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.



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.

The Poster Programme

No	Presenter	Affiliation	Title
Novel Test Configurations and Conditions			
1	Ping Xiao	Manchester U.	*Evaluation of Silicon Carbide Coatings using In-situ Nanoindentation up to 500°C*
2	Gaurav Mohanty	EMPA Thun / Alemnis	*Microcompression High Cycle Fatigue Tests up to 10 Million Cycles*
3	Nigel Jennett	Coventry U.	*Scratching the Surface of Lateral Size Effects (LSE): Failure of Indentation to Predict Scratch Hardness*
Extraction of Bulk Properties from Fine Scale Testing			
4	Max Burley	Cambridge U.	*Johnson-Cook Parameter Evaluation from Ballistic Indentation Data via Iterative FEM Modelling*
5	Fernando Roberto-Pereira	Cambridge U.	*Inferring Superelasticity Parameters of NiTi SMA from Instrumented Indentation Data*
6	Tobias Kalfhaus	FZ Jülich	*Development and Mechanical Evaluation of Repair Methods for Nickel Based Super Alloys*
7	Rob Thompson	Cambridge U.	*Optimisation Algorithms for Extracting Material Properties from Indentation Data*
8	Jimmy Campbell	Cambridge U.	*Issues for SEMPID Usage: Compliance Calibration, Indenter Radius and Penetration, Constitutive Law Selection, Friction and Material Anisotropy*
Interfacial Adhesion, Fracture & Toughness			
9	Jiawei Jiang	Oxford U.	*Reliable Measurement of Fracture Toughness of Ceramics at the Microstructural Scale*
10	Luc Vandeperre	Imperial Coll.	*Deriving Hardness from Load-displacement Data in the Presence of Extensive Cracking*
Microstructural and Mechanistic Effects			
11	Guillaume Kermouche	St. Etienne	*About the Use of Nanoindentation Testing to understand Microstructural Evolution Induced by Surface Thermo-mechanical Treatments*
12	Mathias Göken	Erlangen U.	*The Deformation Behaviour of Superplastic Metals as Investigated by Nanoindentations and Micropillar Tests*
13	Gerold Schneider	Hamburg U.	*Organically Linked Iron Oxide Nanoparticle Supercrystals with Exceptional Isotropic Mechanical Properties*
14	Erica Lilleodden	Hamburg U. / Geesthac	*Understanding Size Effects in the Mechanical Behavior of Nanoporous Gold through MicroLaue Diffraction*
15	Andy Bushby	QMUL	*New Methods for Determining the Influence of Size Effects in Nanoindentation Testing*
16	Christoph Kirchlechner	MPI Düsseldorf	*Beyond Hall-Petch: Mechanism-based Description of Dislocation-Grain Boundary Interactions*
Indentation of Soft / Compliant (Bio)Materials			
17	Riaz Aktar	Liverpool U.	*Dynamic Nanondentation of Highly Compliant Hydrogels: A Critical Comparative Analysis with Rheometry*
18	Eneko Axpe	Cambridge U.	*A Unified Model for the Solute Diffusion in Hydrogels*

Attending Delegates

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Abstracts – Oral Presentations

Session 1: Novel Test Configurations and Conditions

Talk 1: Monday 9th April, 13.30-13.50

Small-scale Deformation at High Rates - Non-Newtonian flow to the Theoretical Strength at Room Temperature from Metallic to Silica Glasses by Impact Nanoindentation

S Korte-Kerzel

Department of Physical Metallurgy and Metal Physics, RWTH, Aachen, Germany

In many daily applications, glasses are indispensable, and novel applications demanding improved strength and crack resistance are appearing continuously. Up to now, the fundamental mechanical processes in glasses subjected to high strain rates at room temperature are largely unknown and thus guidelines for one of the major failure conditions of glass components are non-existent. Here, we elucidate this important regime for the first time using glasses ranging from a dense metallic glass to open fused silica by impact as well as quasi-static nano-indentation. We show that towards high strain rates, shear deformation becomes the dominant mechanism in all glasses accompanied by Non-Newtonian behaviour evident in a drop of viscosity with increasing rate covering eight orders of magnitude. All glasses converge to the same limit stress determined by the theoretical hardness, thus giving the first experimental and quantitative evidence that Non-Newtonian shear flow occurs at the theoretical strength at room temperature.

Talk 2: Monday 9th April, 13.50-14.10

High Temperature Nanoindentation up to 800˚C for Characterizing High Temperature Properties of Materials

NX Randall[†], M Conte[†], G Mohanty[§], J Schwiedrzik[§] & J Michler[§]

[†]Anton Paar TriTec SA, Rue de la Gare 4, 2034 Peseux, Switzerland

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One of the primary motivations for development of instrumented indentation was to measure the mechanical properties of thin films. Characterization of thin film mechanical properties as a function of temperature is of immense industrial and scientific interest. The major bottlenecks in variable temperature measurements have been thermal drift, signal stability (noise) and oxidation of/condensation on the surfaces. Thermal drift is a measurement artifact that arises due to thermal expansion/contraction of indenter tip and loading column. This gets superimposed on the mechanical behavior data precluding accurate extraction of mechanical properties of the sample at elevated temperatures. Vacuum is essential to prevent sample/tip oxidation at elevated temperatures. In this talk, the design and development of a novel nanoindentation system that can perform reliable load-displacement measurements over a wide temperature ranges (from -150 to 800˚C) will be presented emphasizing the procedures and techniques for carrying out accurate nanomechanical measurements. This system is based on the Ultra Nanoindentation Tester (UNHT) that utilizes an active surface referencing technique comprising of two independent axes, one for surface referencing and another for indentation. The differential depth measurement technology results in negligible compliance of the system and very low thermal drift rates at high temperatures. The sample, indenter and reference tip are heated/cooled separately and the surface temperatures matched to obtain drift rates as low as 1nm/min at 800˚C without correction. Instrumentation development, system characterization, experimental protocol, operational refinements and thermal drift characteristics over the temperature range will be presented, together with a range of results on different materials.

Talk 3: Monday 9th April, 14.10-14.30

Recent Advances in In-situ SEM Nanomechanical Testing: Cryogenic Temperature, High Strain Rates, In-situ EBSD Experiments

J Michler

EMPA, Thun, Switzerland

Talk 4: Monday 9th April, 14.30-14.50

Exploring the Temperature Dependence of Indentation Size Effects, Strain Rate Sensitivity and Pile-up in Polycrystalline Tungsten to 950˚C

BD Beake†, AJ Harris†, J Moghal§ & DEJ Armstrong§

† Micro Materials Ltd, Wrexham, UK

§ Department of Materials, University of Oxford, UK

Elevated temperature nanoindentation measurements were performed on polycrystalline tungsten to 950˚C. Tests were carried out under high vacuum conditions as tungsten oxidizes in air at >500˚C. The temperature dependence of the hardness, elastic modulus, strain rate sensitivity, activation volume and the indentation size effect in hardness were investigated at 25, 750, 800, 850, 900 and 950˚C. Thermal drift assessed from the last 60% of a hold period at 90% unloading was typically ~0.05 nm/s and it did not vary significantly with load or temperature [1]. The hardness measurements were in good agreement with previous determinations by non-depth sensing hot microhardness. Above 800˚C the hardness of tungsten changes relatively little but more pronounced time-dependent deformation was observed from the temperature, 850˚C, around where Milman et al reported changes to dislocation cell structure occur [2]. The strain rate sensitivity determined by analysis of indentation creep data increased with temperature. Activation volume reached a peak of ~50 b^3 at 750-800˚C. Decreasing activation volume above 800˚C was due to the increase in strain rate sensitivity. Although there have been reports of reduced indentation size effects at elevated temperatures, for a bcc metal such as W, lattice resistance depends on T/T_c (where Tc, the critical temperature, at which flow stress becomes insensitive to temperature = 527˚C for W) and so size effects would be expected scale with this relative temperature. Pillar compression tests on W(100) to 400˚C (T/T_c = 0.84) have determined much stronger size effects in strength at higher temperatures [3]. In this current study to T/T_c = 1.56 significantly stronger indentation size effects in hardness were also found at elevated temperatures. Elastic modulus measurements determined from standard elastic analysis of the unloading curves were higher than literature values, by ~13% at 750-800˚C and 30-40% at 850-950˚C. The greater discrepancy starts from the point (850 ºC) at which more pronounced time-dependent deformation was observed. Time-dependency was therefore accounted for by a viscoelastic compliance correction [4]. After correction values of the elastic modulus agree to within ~1% at 750-800˚C, and to 6% of literature values at 950˚C. The small remaining discrepancy is consistent with the influence of pile-up since AFM measurements show that pile-up is significant in these high temperature indentations and the pile-up height increases slightly over the range 750-950˚C.

[1] AJ Harris, BD Beake, DEJ Armstrong and MI Davies, Exp Mech (2016) DOI 10.1007/s11340-016-0209-3

[2] YV Milman et al, Acta Metal Mater 41 (1993) 2523-2532

[3] O Torrents Abad et al, Acta Mater 103 (2016) 483.

[4] G Feng, A.H.W. Ngan, J Mater Res 17 (2002) 660-668

Talk 5: Monday 9th April, 14.50-15.10

High-temperature Nanoindentation of bcc Metals and Alloys

R Schwaiger

Karlsruhe Inst. Techn.

Talk 6: Monday 9th April, 15.10-15.30

Shear Banding Behaviour of Bulk Metallic Glasses through Dynamic Electrical Measurements

R Goodall

Department of Materials Science & Engineering, Sheffield University, UK

Bulk metallic glasses (BMGs) have attracted attention owing to their unique and favourable material properties, such as high yield strength, wear resistance, formability, corrosion resistance and damping characteristics. These properties directly descend from the amorphous atomic structure, which unlike crystalline materials, cannot support features such as dislocations. The fundamental understanding of yield behaviour in BMGs is still relatively poor. Here a new detection methodology, observing indirectly-related changes in the resistivity of the material with time, is applied to the challenge of probing the yield behaviour of BMGs. Both weak and strong signals are observed; while the weak signals yield too little data for quantitative analysis, the strong signals permit the conclusion that most of the response arises from structural change, with a smaller thermal component at longer times. With further theoretical and model development such an approach gives a route to assess shear banding events in materials where they occur.

Session 2: Extraction of Bulk Properties from Fine Scale Testing

Talk 7: Monday 9th April, 16.00-16.20

In-situ SEM Micro Shear Testing of Engineering Materials

G Eggeler

Department of Materials Science & Engineering, Bochum University, Germany

Double shear micro shear testing is introduced as a new fine scale mechanical testing method. The strengths and the drawbacks of the method are discussed. The results obtained for solution annealed gold and on strain hardened Cu are presented, which show how reproducible the method is and how it reacts to increasing levels of dislocaton densities. Finally, the method is applied to single crystal Ni-base super alloys to document how different microscopic crystallographic shear systems react to shear loading and to show that the method can differentiate between prior dendritic and interdendritic regions. The potential of the method to assess high temperature mechanical properties is finally discussed.

Talk 8: Monday 9th April, 16.20-16.40

In-situ Microcompression Transient Plasticity Tests for Reliable Extraction of Deformation Activation Parameters: Creep, Stress Relaxation and Strain Rate Jump Tests

G Mohanty

EMPA, Thun, Switzerland

In this talk, micro-compression creep, stress relaxation [1] and strain rate sensitivity tests [2, 3] performed in-situ, in a scanning electron microscope, on nanocrystalline Ni at elevated temperatures (25-125 °C) will be presented. Nanocrystalline Ni was chosen not only due to its enhanced time dependent plasticity properties but also because testing large micropillars (~ 10 microns in diameter, 25-30 microns in height) mimics bulk scale tests interrogating millions of grains. Corresponding micro-tensile tests were performed to compare and validate these recently developed micropillar compression tests. All micropillar tests were performed on the same sample to remove sample-to-sample variation and allow direct comparison to understand the correlation between these three transient tests. The observed stress relaxation and creep were found to be significant at stresses even below the 0.2% offset yield strength demonstrating the enhanced time dependent behavior of nanocrystalline materials. The extracted exponents and activation parameters (activation volume and activation energies) provided an initial estimate of the footprint of the rate controlling deformation mechanism(s). Based on these results, the role of dislocation plasticity and grain boundary mediated processes will be discussed. Overall, this study aims to develop and validate each of these three time-dependent tests at the micron scale, bridge the gap between the three tests and provide useful insights into developing similar indentation based tests, for creep and stress relaxation measurements in particular. Finally, future research directions in this sub-field of micromechanics will be discussed.

[1] Mohanty G, Wehrs J, Boyce BL, Taylor A, Hasegawa M, Philippe L, et al. Room temperature stress relaxation in nanocrystalline Ni measured by micropillar compression and miniature tension. Journal of Materials Research 2016;31:1085-95

[2] Mohanty G, Wheeler JM, Raghavan R, Wehrs J, Hasegawa M, Mischler S, et al. Elevated temperature, strain rate jump microcompression of nanocrystalline nickel. Philosophical Magazine 2015;95:1878-95

[3] Wehrs J, Mohanty G, Guillonneau G, Taylor AA, Maeder X, Frey D, et al. Comparison of In Situ Micromechanical Strain-Rate Sensitivity Measurement Techniques. Jom 2015;67:1684-93

Talk 9: Monday 9th April, 16.40-17.00

Essential Refinements of Spherical Nanoindentation Protocols and a Multi-sharp Tip Approach for the Reliable Determination of Mechanical Flow Curves

A Leitner[§], D Kiener[§] & V Maier-Kiener[†]

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An appealing idea to material scientists is to characterize the mechanical behavior of materials with minimal experimental effort while guaranteeing highly reliable results. Nanoindentation is a candidate technique to reach this objective. Though it is already a standard method to extract hardness and Young's modulus, the technique is not yet fully exploited regarding the localize flow curves. Therefore, understanding and linking mechanical properties obtained by spherical indentation experiments to uniaxial data is extremely challenging. Since the first attempts in the early 20th century numerous advances gradually allowed to expand the output of indentation tests. Still, the extraction of flow curves from spherical nanoindentation has not yet been fully established, as tip shape problems and size effects impede a straight-forward implementation. Within this study, we show new calibration procedures originating from fundamental geometrical considerations to account for tip shape imperfections. This sets the base for strain-rate controlled tests, which in turn enables us to measure rate-dependent material properties either with constant strain-rate or by strain-rate jump tests. Finally, experimental evaluation of the constraint factor in consideration of the mechanical properties and induced strain enables the extraction of flow curves. Testing materials with refined microstructures ensures the absence of possible size effects. By using four different pyramidal tips with varying apex angle, it can be further demonstrated on materials with microstructures ranging from single crystalline to nanocrystalline dimensions, how both flow curves and Hall-Petch parameters can be extracted from nanoindentation testing. Applying appropriate definitions of indentation stress and strain and considering the indentation size effect, the obtained values coincidence well with literature values determined by uniaxial tests. This study contributes to a significant improvement of current experimental protocols and allows the obtaining of flow curves from single spherical imprints to be moved one step closer to implementation as a standard characterization technique for modern materials.

Talk 10: Monday 9th April, 16.00-16.20

The SEMPID (Software for the Extraction of Material Properties from Indentation Data) Package for Plasticity

J Dean & TW Clyne

Department of Materials Science, Cambridge University, UK

Hardness and stiffness can be measured routinely from indentation tests. However, development of indentation methodologies for extracting more complex (and potentially much more useful) material characteristics has the potential for expanding enormously the usefulness of the technique. Plasticity is a key area, with the objective being to evaluate the yield stress and the parameters quantifying the work hardening characteristics. Analytical methods cannot take proper account of the complexity of the evolving stress and strain fields. This can be done via iterative FE modeling, but the challenge is to incorporate these procedures into a user-friendly package, with clear indications provided about the probable reliability of inferred characteristics. A set of such packages, one of which is focused on quasi-static plasticity, is currently being developed and tested, under the SEMPID banner. A brief outline will be given regarding usage of the SEMPID plasticity package, covering the recommended experimental procedures, the screening and convergence algorithms implemented within the package (some of which are described in a recent paper [1]) and interpretation of the outputs. In addition to providing best-fit parameter values, the package can be used to explore the stress and strain fields generated during indentation.

[1] J Dean & TW Clyne. *Extraction of Plasticity Parameters from a Single Test using a Spherical Indenter and FEM Modelling*. Mechanics of Materials **105** (2017) p.112-122.

Talk 11: Monday 9th April, 17.20-17.40

Combinatorial In Situ Micromechanics of the Al-Cu System at Low Temperatures

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Diffusion couples have long been a fundamental technique in materials science, allowing the exploration of phase diagrams and diffusion constants. With the advent of the Materials Genome Initiative, diffusion couples and multiples are now being used as a high throughput means to investigate a wide range of materials properties. However, so far only the most basic nanoindentation techniques have been used to interrogate mechanical properties of diffusion couples. Here, the potential of applying advanced micromechanical techniques to diffusion couples will be demonstrated in the case of the Al-Cu binary system. Strain rate jump microcompression is employed in situ in the SEM to probe the strain rate sensitivity over a range of compositions from ambient to cryogenic temperatures. This provides insight into the Portevin-LeChatelier effect within a consistent single-crystalline microstructure with varying strain rates, temperatures, solute concentrations and precipitate density.

Talk 12: Monday 9th April, 17.40-18.00

Identification of Material Properties by Instrumented Indentation using Manifold Approach

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Any shape can be described in an Eulerian parametrization (e.g. pixel, voxel or levelset functions). By interpolation between a set of Eulerian representations of neighboring shapes, it is then possible to construct a shape manifold. Proper Orthogonal Decomposition is one of the popular methods allowing to reduce the dimensionality of the corresponding shape space. Any point in the reduced shape space belonging to the manifold corresponds to an admissible shape. Finally, the difference between two shapes can easily be quantified by calculating the distance between them in the shape space. From these properties, a nonlinear shape-manifold learning approach was proposed by the authors [1]. It was successfully applied to solve a variety of problems in mechanics including model reduction [2], optimization [3] and identification [4]. In this presentation, we focus on the application of the concept of shape manifold to identify material parameters from instrumented indentations. Two different sources of information were compared: load-displacement curves and residual imprints. From this approach, the maximum number of independent material properties was estimated and compared for both cases. Finally, the approach was applied by combining the two sources of information together, in order to improve the identifiability of the desired parameters.

[1] L. Meng, P. Breitkopf, G. Le Quilliec, B. Raghavan, P. Villon. Arch Computat Methods Eng. (2016) DOI 10.1007/s11831-016-9189-9

[2] B. Raghavan, G. Le Quilliec, P. Breitkopf, A. Rassineux, J.-M. Roelandt, P. Villon. Int J Mater Form (2014) DOI 10.1007/s12289-013-1145-8

[3] G. Le Quilliec, B. Raghavan, P. Breitkopf. Comput. Methods Appl. Mech. Engrg. (2015) DOI 10.1016/j.cma.2014.11.029

[4] L. Meng, P. Breitkopf, G. Le Quilliec. Int J Solids Struct. (2016) DOI 10.1016/j.ijsolstr.2016.12.002

Session 3: Interfacial Adhesion, Fracture and Toughness

Talk 13: Tuesday 10th April, 09.00-09.20

Stressed Overlayers to Assess the Adhesion of Brittle Film Systems via Nanoindentation

M Cordill

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Silicon nitride (Si_3N_4) is a frequently used passivation layer as well as an ion barrier in microelectronic devices. Its most outstanding properties are high fracture toughness and thermal stability, but in order to have a reliable device the adhesion to other thin films is of great importance. Qualitative tests like tape tests have shown that the adhesion of the brittle interface of Si_3N_4 and silicon glass (BPSG) is rather poor. In order to determine a quantitative measure of the adhesion of Si_3N_4 to BPSG other techniques such as nanoindentation and scratch testing are necessary. Indentation and scratching induce well-defined areas of delamination at the Si_3N_4-BPSG interface only when used in conjunction with a stressed overlayer of WTi. In this film system, the stressed overlayer helps the underlying Si_3N_4 film to buckle and supports the delaminated, brittle Si_3N_4 film from spalling from the substrate. Utilizing the delaminated areas and well established methods the adhesion of brittle thin films can be determined, which could not be measured before.

Talk 14: Tuesday 10th April, 09.20-09.40

Toughening Non-metallic Crystals by Inhomogeneous Elasticity

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High temperature structural materials must be resistant to cracking and oxidation. However, most oxidation resistant materials are brittle and a significant reduction in their yield stress is required if they are to be resistant to cracking. It is shown, using density functional theory, that if a crystal's unit cell elastically deforms in an inhomogeneous manner, the yield stress is greatly reduced, consistent with observations in layered compounds, such as Ti_3SiC_2, Nb_2Co_7, W_2B_5, Ta_2C and Ta_4C_3. The mechanism by which elastic inhomogeneity reduces the yield stress is explained and the effect demonstrated in a complex metallic alloy, even though the electronegativity differences within the unit cell are less than in the layered compounds. Substantial changes appear possible, suggesting this is a first step in developing a simple way of controlling plastic flow in non-metallic crystals, enabling materials with a greater oxidation resistance and hence a higher temperature capability to be used.

Talk 15: Tuesday 10th April, 09.40-10.00

Adhesion Investigations of ALD Films on Metal Substrates

I Utke

EMPA, Thun, Switzerland

Talk 16: Tuesday 10th April, 10.00-10.20

Understanding the Failure of Silicon Carbide Composites through Micro-scale Mechanical Tests

DEJ Armstrong

Department of Materials, University of Oxford, UK

Silicon carbide ceramics are a candidate material for the use in nuclear power generation and are suggested to be used in novel accident tolerant fuel (ATF) cladding designs due to its favorable properties, in particular reduced (compared to Zircaloy) oxidation under accident conditions, good neutronic performance, high temperature strength and stability under irradiation. Due to its inherent brittleness, it is suggested to be used in the form of SiC-fiber reinforced SiC-matrix composite. In order to reliably model behavior of highly non-uniform and anisotropic composite materials the knowledge of the individual properties of fiber and matrix, and, crucially, the fiber-matrix interfaces, is required. In addition, nuclear fuel cladding materials are exposed to elevated temperatures during their operation, and therefore the understanding of the temperature dependences of the relevant properties is essential. Micromechanical testing techniques, such as nanoindentation and microcantilever beam fracture, allow determination of such localized properties, and can be implemented in the wide range of temperatures.

Talk 17: Tuesday 10th April, 10.20-10.40

In Situ Stable Fracture of Ceramic and Metal Ceramic Interfaces on the Micron Scale

F Giuliani

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The fracture toughness of ceramics is often dominated by the structure of their grain boundaries. Our capacity to improve the performance of ceramic components depends on our ability to investigate the properties of individual grain boundaries. This requires development of new fracture testing methods providing high accuracy and high spatial resolution. Recently, several techniques have been developed using small scaled mechanical testing, based within a nanoindenter, using a variety of tip and sample geometries, including: micropillar compression, microcantilever bending and double-cantilever compression. However, the majority of the published work relies on load-displacement curves for the identification of crack initiation and the geometries can result in a complex analysis of force distribution and stress intensity factor. Our approach uses a double cantilever geometry to obtain stable crack growth and we calculate the fracture energy under a constant wedging displacement. The tests are carried out within an SEM, this has two benefits: the sample is well aligned for a controlled test and images are recorded during the test for later analysis. Crucially this allows us to use beam deflection and crack length rather than critical load to measure fracture toughness. Our tests have proved it is possible to initiate and stably grow a crack in a controlled manner in ceramic materials for several microns. This approach has been validated on SiC where it gives a good approximation of the surface energy and then extended to SiC bi-crystals along with Ni-Al_2O_3 interfaces where crack blunting and bridging mechanism can be observed and measured.

Session 4: Microstructural and Mechanistic Effects

Talk 18: Tuesday 10th April, 11.10-11.30

Phase Transformations during Biaxial and Strain Path Changing Loadings : In-situ HR-DIC (SEM) and In-situ Synchrotron X-ray Diffraction

H Van Swygenhoven
PSI Villigin, Switzerland

Talk 19: Tuesday 10th April, 11.30-11.50

Dislocation Propagation across Lamellar Boundaries in Fully Lamellar TiAl Alloys: Insight from Micro-tensile Experiments

J Molina
IMDEA Madrid, Spain

Fully lamellar gamma titanium aluminides are very promising materials for aerospace applications, due to their increased thrust-to-weight ratios and improved efficiency under aggressive environments at temperatures up to 750˚C. For that reason, they are projected to replace the heavier Ni- base superalloys currently used for low pressure turbine (LPT) blades manufacturing. However, their ductility is limited due to their inherent anisotropy, associated to their lamellar microstructure. The objective of this work was to study the mechanical response of single colonies of polycrystalline γ-TiAl, as a function of layer thickness and layer orientation, and to relate this mechanical response with the operative deformation mechanisms. With this aim, micropillars with lamellae oriented at 0˚, 45˚ and 90˚ with respect to the loading direction were compressed at room and elevated temperature. A thorough study of pillar size effects revealed that the results were insensitive to pillar size for dimensions above 5 µm. The results can therefore be successfully applied for developing mesoscale plasticity models that capture the micromechanics of fully lamellar TiAl microstructures at larger length scales. The results revealed a large plastic anisotropy, that was rationalized, based on slip/twin trace analysis, according to the relative orientation of the main operative deformation modes with respect to the lamellar interfaces: longitudinal, mixed and transversal deformation modes. Additionally, microtensile specimens were also milled out of single colonies and in-situ tested in the SEM, to study the role of interlamellar interfaces on the plastic deformation and fracture under tension. EBSD was used before and after the test to study the role of different type of interfaces (true twin, pseudo twin and order variant) on slip/twin transfer. This study emphasizes the complexity of the micromechanics of fully lamellar TiAl alloys, where the activation of different deformation modes is strongly affected, not only by the lamellar orientation, but also by the character of the interfaces between the different lamellae.

Talk 20: Tuesday 10th April, 11.50-12.10

AFM Nanoindentation to Quantify the Mechanical Behaviour of Nano- and Micron-Sized Hybrid Crystals

J-C Tan

Department of Materials, University of Oxford, UK

Talk 21: Tuesday 10th April, 12.10-12.30

Portevin–Le Chatelier Effect in Aluminium Alloys: A Nanoindentation Study at Room and Elevated Temperature

C Tromas[†], G Mohanty[§], A Joseph[†], A Joulain[†], J Bonneville[†] V Gauthier-Brunet[†], S Dubois[†], D Eyidi[†], JJ Schwiedrzik[§], J Michler[§] JP Monchoux[‡] & M Conte[$]

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The Portevin-Le Chatelier (PLC) is a plastic instability observed in different alloys, and particularly in aluminum alloys, which is characterized by a serrated flow during plastic deformation. The PLC effect originates from the competition between gliding of mobile dislocations and pinning of these dislocations by diffusing solute atoms. This dynamic strain hardening leads to a negative strain rate sensitivity, which is often used to characterize or quantify the PLC effect. The PLC effect has been widely investigated in the case of stress-strain curves obtained in macroscopic uni-axial tests. However, in the case of the aluminum matrix composites Al/ω-AlCuFe, it has been observed that copper atoms diffuse during the material synthesis from the reinforcement particles to the aluminum matrix. The aluminum matrix thus presents a heterogeneous concentration of copper atoms leading to local PLC effect. In a first part, this presentation will report nanoindentation experiments at room temperature in the aluminum matrix of the Al/ω-AlCuFe composite. A detailed analysis of the individual nanoindentation curves, obtained in displacement controlled mode, shows serrated behavior characteristic of Portevin-Le Chatelier effect associated to dislocation pinning by solute atoms. The comparison between chemical (SEM – EDXS analysis) and hardness maps as well as the quantitative analysis of the deformation curves gives evidence of a strong correlation between the chemical heterogeneities and mechanical properties of the Aluminum matrix. In a second part, a study of the PLC effect during nanoindentation tests in the temperature range from 25°C to 300°C will be presented in the case of the Al3%Mg alloy, used as a model material for easy comparison with uniaxial tests from the literature. The experiments were performed in displacement controlled mode in a recently developed vacuum high temperature nanoindenter based on active surface referencing and non-contact tip and sample heating. In this configuration, the PLC effect appears as successive load drops on the loading curves. The load drop magnitude and its occurrence frequency were statistically analyzed for different temperatures of testing. The results will be discussed in terms of an expanding plastic volume beneath the indenter interacting with the solute atoms in the complex stress field of the indenter.

Talk 22: Tuesday 10th April, 12.30-12.50

The Origin of Microstructural Discontinuities underneath a Tribologically Loaded Surface

C Greiner

Karlsruhe Inst. Techn., Germany

Frictional loading of a metal surface induces microstructural changes underneath the surface. A typical tribo-induced microstructure displays distinct discontinuities parallel to the surface, which separate the near surface layer from the bulk. By systematically decreasing the number of passes of a sapphire sphere sliding over high-purity copper, we find that the origin of the microstructural discontinuity is already laid after the first sliding pass even for very mild sliding conditions. A distinct dislocation structure is formed 100-150 nm under the surface. This distinct dislocation self-organization is attributed to a sign change in the stress field underneath the sliding indenter. The dislocation structure evolves into the known microstructural features with increasing number of sliding passes. Consequently, the microstructure and mechanical properties of the surface layer are determined in the very first loading pass. Control of the initial tribological loading could therefore be exploited to precondition interfaces for superior tribological properties.

Talk 23: Tuesday 10th April, 12.50-13.10

Chemo-Mechanical Effects in Thin Film and Bulk Oxides

S Bull

School of Engineering, University of Newcastle, UK

The environmental sensitivity of the surface mechanical properties of ceramics has long been known and properties such as plasticity, creep, fracture and fatigue have been shown to be susceptible to changes in the environment. These effects are often called Rehbinder effects after his pioneering work in the 1930s. Most relevant to indentation testing is "anomalous indentation creep" which has been observed by many workers in ceramics using both conventional microhardness and nanoindentation testing. Whereas a constant, time-independent hardness was found in microhardness measurements by testing in dry toluene the hardness was found to drop as the indentation time increased when testing in moist air. In nanoindentation testing it is often observed that the surface hardness of a ceramic material is reduced after exposure to water or moist air and this can be reversed by solvent or vacuum treating the material. However, the mechanism for this change is not generally well understood. It is usually accepted that it is not due to the formation of a measureable reaction layer on the surface, rather mechanisms arising from the effect of adsorbates on friction, surface energy and dislocation core energy are often postulated. In this study the chemomechanical effects arising from water exposure on a range of oxide ceramics and ceramic coatings have been investigated by nanoindentation and the potential mechanisms which explain the observed effects have been assessed. It is concluded that adsorption results in the modification of the energy levels of a crystalline ceramic near the surface and this will affect the energy levels in the core of a dislocation. In this way changes in dislocation mobility can be induced. The effect of disrupting surface adsorbates and introducing radiation damage by titanium ion implantation on the chemomechanical behaviour of single crystal sapphire will be discussed.

Session 5: Indentation of Soft / Compliant (Bio)Materials

Talk 24: Tuesday 10th April, 14.00-14.20

Dual Crosslinked Alginate Hydrogels with Patterned Mechanical Properties and Biomolecule Presentation

A Lückgen, DS Garske, RM Desai, P Fratzl, DJ Mooney, GN Duda & A Cipitria

MPI Colloids & Interfaces, Berlin, Germany

Biomaterials featuring patterns in mechanical properties and biomolecule presentation hold great promise for tissue engineering applications, as they could enable spatial control over cell behavior and tissue formation. We developed an alginate-based material system that permits the creation of these patterns on the µm-scale by combining two orthogonal covalent crosslinking chemistries: Diels-Alder and thiol-ene. As the Diels-Alder reaction occurs spontaneously, and the thiol-ene reaction requires initiation by UV, the precise location and timing of this second step can be controlled via photomasks to generate patterns. Initial experiments were performed on single-crosslinked, unpatterned materials. Diels-Alder crosslinked hydrogels were fabricated by combining alginate modified with norbornene or tetrazine functional groups. The degree of crosslinking and resulting mechanical properties were controlled by the amount of norbornene and the stoichiometric ratio of norbornene to tetrazine. Thiol-ene crosslinked hydrogels were prepared by exposing norbornene-coupled alginate and a dithiol crosslinker to UV, and mechanical properties were tuned by changing the amount of crosslinker. Both material sets were characterized for rheological and mechanical properties using uniaxial unconfined compression testing. Materials were rendered cell-compatible via conjugation of thiol-coupled RGD sequences linked to remaining available norbornene groups. This modification was confirmed by quantifying mesenchymal stromal cell (MSC) viability and proliferation in 2D and 3D over one week. While preparing dual-crosslinked, single-phase materials, it was noted that the timing of UV exposure (and therefore thiol-ene crosslinking) enables control over stiffness: instant UV exposure led to soft gels, while later UV exposure led to increased gel stiffness. Patterns in mechanical properties were confirmed by nanoindentation surface mapping, yielding comparable results to those obtained by uniaxial unconfined compression testing on single-crosslinked, unpatterned materials. In addition, patterns in RGD molecule were visualized by analyses of cell attachment and spreading. Ongoing work focuses on patterned, surface immobilization of bone morphogenetic protein-2 (BMP-2) peptide mimics and cell differentiation assays into the osteogenic lineage. This biomaterial-based strategy allowing biophysical and biochemical patterning could direct MSC behavior and support guided tissue regeneration in clinically-challenging situations.

Talk 25: Tuesday 10th April, 14.20-14.40

High Rate Indentation of Brain Tissue

D MacManus

Univ. College Dublin, Ireland

Traumatic brain injuries (TBI) are a major global socioeconomic problem that have potentially devastating consequences on patients who have suffered and are living with a TBI [1]. In order to elucidate the biomechanics of TBI and improve accuracy of current and next generation finite element (FE) models of the brain, we require local, high rate data for brain tissue [2]. Here, we present a custom-built micro-indentation apparatus that is capable of applying up to 35% strain at 100/s strain rates in two brain regions of a mouse model [3]. Inverse FE techniques are subsequently used to fit constitutive models to the raw force-displacement data. We show the region and rate dependent properties of brain tissue as well as a strain-softening phenomenon encountered at 100/s and 35% strain.

[1] Roozenbeek et al., Changing patterns in the epidemiology of traumatic brain injury, Nat. Rev. Neurol., 2013, 9, 231-236.

[2] MacManus et al., A viscoelastic analysis of the P56 mouse brain under large-deformation dynamic indentation, Acta Biomater., 2017, 48, 309-318.

[3] MacManus et al., Region and species dependent properties of adolescent and young adult brain tissue, Sci. Rep., 2017

Talk 26: Tuesday 10th April, 14.40-15.00

Determining the Mechanical Behaviour of Individual Hydrogel Microspheres with a Nanoindenter

A Bushby & K Shuturminska

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Hydrogel microspheres, with diameters in the range 50-500 microns, are used in oncology treatment for the embolization of blood vessels surrounding tumours. These gelsphers are delivered by catheter to the target site and distributed in a series of blood vessels of different diameter. The mechanical properties of the spheres are important to their delivery and performance. However, determining these properties from an individual gelsphere is challenging. Here we use a nanoindentation instrument to compress individual gelspheres between 2 flat surfaces under liquid conditions. Hertzian mechanics can be applied to determine the elastic properties during loading and to observe deviation from the Hertzian response for materials that display visco-elastic, poro-elastic or visco-plastic properties. Furthermore, by utilizing a ramp-and-hold test protocol and a creep compliance analysis method, the time dependent mechanical response can be determined from a single gelsphere.

Talk 27: Tuesday 10th April, 15.00-15.20

Influence of Vulcanisation and Aging on Local Properties of Polychloroprene Rubber

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Nanoindentation has been, and is still, widely studied and used in mechanics and materials' science. Small scale instrumented indentation is based on the application of a very small force on a sample and the simultaneous computing of the induced displacement. It has been decided here to apply this technique on elastomers. These materials possess a very specific mechanical behavior. They present a high compliance, a non-linear elasticity, a strain-rate dependence and a complete reversibility of their deformations. Regarding literature papers, nanoindentation works carried out on rubbers can be found [1-3]. Nevertheless, the characterization of such materials with this technique being relatively new, the consequences of the viscosity or the local structure for example may not be well understood yet or taken into considerations for the results. In this study we focused on the local response of a carbon black filled neoprene. In a first phase and based on previous results [4-5], local properties have been investigated to highlight mechanical trend for various curing times. These results have been compared with global characterisation. Then, for each curing state, the skin effect has been observed. The influence of aging have been also investigated in this study.

[1] F. Alisafaei, C. -S. Han and S. H. Reza Sanei, Polym. Test., 32, 1220 (2013)
[2] Z. Chen, S. Diebels, N. J. Peter and A. S. Schneider, Comput. Mat. Sci., 72, 127 (2013)
[3] J. Kohn and D. Ebenstein, J. Mech. Behav. Biomed. Mater., 20, 316 (2013)
[4] C. Fradet, F. Lacroix, G. Berton, S. Méo, E. Lebourhis, , 10th European Conference on Constitutive Models for Rubbers 2017, Munich, August 2017.
[5] C. Fradet, F. Lacroix, G. Berton, S. Méo, E. Le Bourhis", Mat. et Tech., 105, 109, (2017)

Talk 28: Tuesday 10th April, 15.20-15.40

Quasi-Plasticity and Shear-Induced Cracking in Contact-Damage Resistant Biological Models

S Amini

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Highly mineralized biological materials such as vertebrate teeth, crustacean appendages, and seashells have been studied extensively due to their remarkable mechanical properties and performances. Although these hard tissues are stiffened by biominerals such as calcium phosphate and calcium carbonate phases, they are complexified with their organic domains and fibrils in different levels of their hierarchy. These organic features not only play a crucial role in their formation but also regulate their property and performance. Since these biomineralized models have been evolved to be functionalized under contact stresses, classical contact mechanic theories, through different techniques, have been used to analyze and classify the mechanical response of these biological models. However, despite traditional engineering materials such as ceramics and metals, which have been the platform to develop these classical theories, the mechanical responses of biological samples have significantly affected by their organic matrix frameworks. In fact, by facilitating the sliding motions through organic phase and fibrils as solid lubricants, especially in hydrated conditions, the mechanical response of these biological models have been differentiated from the traditional engineering materials under the same mechanical stimuli. Here, we provide comprehensive depth-sensing indentation studies using both sharp and spherical contacts to evaluate the elastic-plastic response as well as the fracture behavior of three highly mineralized biological models; shark tooth, parrotfish tooth, and mantis shrimp dactyl club. A wide range of indentation studies, post-indentation field emission electron microscopy and in-situ high resolution Raman spectroscopy were used to observe the micro-mechanisms of contact deformation and map the residual post-indentation stresses. Our data reveal that shear-induced slidings promote damage localization and toughening mechanisms by shear-induced micro cracking and quasi-plasticity under sharp and spherical contact geometries, respectively. These findings suggest that when biological samples are supposed to be characterized, the classical contact damage and fracture theories, which have been mainly developed based on tension-induced damages, should be modified for shear-induced damages, the dominant deformation mechanism in biological models.

Abstracts – Poster Presentations

Area 1: Novel Test Configurations and Conditions

Poster 1

Evaluation of Silicon Carbide Coatings using In-situ Nanoindentation up to 500˚C

P Xiao

Materials Science Centre, University of Manchester, UK

Over the last decades nanoindentation has been established as a useful method to measure the mechanical properties of thin films and coatings. But conducting tests at elevated temperatures has been proven difficult due to experimental issues such as thermal drift in the electronics or oxidation of the indenter tip. Here, we have developed the in-situ nanoindentation technique to measure the mechanical properties of silicon carbide (SiC) coatings in spherical fuel particles up to 500˚C. Values varying from 340 to 400 GPa were determined for the elastic modulus of SiC coatings with differing microstructures, whereas the hardness was consistently around 35 GPa. With increasing experiment temperature the hardness was found to drop significantly, while the elastic modulus remained consistent in the different samples. Tests were also conducted on specimens that had been subject to neutron irradiation inside a nuclear reactor. The results illustrated the excellent radiation resistance of SiC. The determined elastic modulus was not impaired at all, whereas the hardness was marginally increased.

Poster 2

Microcompression High Cycle Fatigue Tests up to 10 Million Cycles

G Mohanty

EMPA, Thun, Switzerland

Nanomechanical tests are moving beyond hardness and modulus to encompass host of different mechanical properties like strain rate sensitivity, stress relaxation, creep, and fracture toughness by taking advantage of focused ion beam milled geometries. Adding high cycle fatigue to this list will be useful to extend the gamut of properties studied at the micro/nanoscale. However, this presents inherent challenges like low oscillation frequencies, long duration of tests and large thermal drift when attempted with standard indenters. This presentation will report the development of micropillar compression-compression high cycle fatigue tests going up to 10 million cycles. This has been made possible by the development of a novel piezo-based nanoindentation technique that allows accessing extremely high strain rates (>10^4 s^-1) and high oscillation frequencies (up to 10 kHz). The associated instrumentation and technique development, design of the fatigue tests at the micron scale, data analysis methodology, experimental protocol and challenges will be discussed. Validation data on single crystal silicon, a reference material, will be presented to demonstrate the reliability of the designed high cycle fatigue tests. Finally, case studies of compression-compression high cycle micropillar fatigue on nanostructured materials will be presented and their results will be discussed in light of existing literature data, particularly the operative deformation mechanism(s). It is hoped that this study will pave way for routine high cycle fatigue tests of metals at the micron scale and provide clues for designing a similar indentation fatigue test.

Poster 3

Scratching the Surface of Lateral Size Effects (LSE): Failure of Indentation to Predict Scratch Hardness

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All tribological interactions have (by definition) a shear component and, going back to the Mohs hardness scale, harder materials are more resistant to scratch deformation. Indentation is, however, more reproducible, has a smaller footprint than a scratch test, and has become the “go to” material parameter for predicting tribological performance. This implied relationship assumes that a material’s plastic properties depend only on the crystallographic direction of deformation and not on the test direction with respect to a free surface. Indentation size effects (ISE) indicate that material strength is genuinely length-scale dependent, suggesting anti-tribological wear applications. To determine whether further performance enhancement can be realised in practice, we have studied scratch deformation and lateral size effects (LSE) directly. We evaluate the relationship between indentation and scratch hardness and plasticity size effects - not as simple as it sounds. One issue is the presence of drag forces in a scratch test; absent from indentation. Clear evidence of LSE in single and polycrystal copper are presented and compared to ISE in the same samples [1,2]. Smaller scratches are harder. We scratched with two different Berkovich stylus orientations: Edge Forward (EF) and Face Forward (FF), with the same lateral projected area. The lateral force generated for EF and FF scratches are very different and correlate to the drag coefficients of the two geometries. We take a relationship that relates the indentation hardness to an effective length scale (a combination of spatial frequencies due to indentation size, grain size and dislocation-dislocation interaction distance) [3] and generalise it to include LSE. This analysis allows quantification of each length-scale in action and shows that the ISE and LSE responses have a striking difference: the LSE response to grain size is nearly four times stronger than observed for ISE in the same Cu samples. This is a clear health warning on an over-reliance on indentation hardness and Hall-Petch relationships in predicting scratch and tribological performance of materials.

[1] A. Kareer, et al. Philosophical Magazine 2016, 96:32-34, 3414-3429

[2] A. Kareer, et al. Philosophical Magazine 2016, 96:32-34, 3396-3413

[3] Xiaodong Hou and N. M. Jennett Acta Mater. 2012, 60(10) 4128-4135.

Area 2: Extraction of Bulk Properties from Fine Scale Testing

Poster 4

Johnson-Cook Parameter Evaluation from Ballistic Indentation Data via Iterative FEM Modelling

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A methodology is presented for evaluating the Johnson-Cook parameter, *C*, which is used to characterize the strain rate sensitivity of plastic deformation. This has been done using ballistic indentation experiments, using high-speed photography to monitor projectile motion and stylus profilometry to record residual indent shapes, coupled with iterative FE modeling of penetration and rebound. Cermet spheres of 5 mm diameter were projected with velocities in the range 50-250 m s^{-1} at copper samples, in both work-hardened and annealed states. Commercial ABAQUS software was used for the simulations. The level of agreement between predicted and measured outcomes was characterized using a goodness-of-fit parameter. Since the strain rate sensitivity is characterized by a single parameter value in the Johnson-Cook formulation, convergence on its optimum value is straightforward, although a parameter characterizing interfacial friction is also required. The best-fit values of *C* were ~0.016 and ~0.030 for work-hardened and annealed material. The strain rates operative during these experiments were $\sim 10^4 - 10^6$ s^{-1}. Customised software packages allowing automated extraction of such values from sets of experimental data are currently being developed.

Poster 5

Inferring Superelasticity Parameters of NiTi SMA from Instrumented Indentation Data

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A procedure is described for obtaining superelastic characteristics (critical stress and strain levels) from load-displacement data obtained during instrumented indentation of a NiTi alloy. These experiments were carried out using a spherical indenter that was sufficiently large (5 mm diameter) to ensure that representative volumes were being tested - the grain size was 30-50 μm. Penetration was carried out to a depth of about 50 μm (ie a depth/indenter radius ratio of about 2%), creating peak (von Mises) strains in the sample of around the maximum that can be achieved superelastically with this alloy (ie about 6%). The procedure involves iterative FE modeling of the process, using the superelastic simulation capability in ABAQUS commercial software. A goodness-of-fit parameter is evaluated for each set of parameter values and a screening operation is carried out, using a Nelder-Mead algorithm to converge on the best-fit set. The Young's moduli of the two phases were taken as pre-defined. This still left 5 parameters to evaluate, which would in general represent a very large parameter space to be explored. However, separate operations were carried out on loading and unloading parts of the load-displacement curve, so that these involved only 3- and 2-parameter fitting operations respectively. The coupling between the two sets of parameters is relatively weak, but nevertheless some switching was undertaken between the two convergence operations. Final outcomes were compared with the stress-strain curves obtained by conventional uniaxial testing (in compression) and a very good level of agreement was obtained. Since the NiTi samples being used were in the form of extruded rods, this testing was carried out in both axial and radial directions, to check on anisotropy effects (due to crystallographic texture). These were found to be relatively weak, but detectable. The indentation response is a multi-axial one, and was found to be very similar in different directions.

Poster 6

Assessment of the Mechanical Properties of Sprayed Layers on Superalloy Blades using Instrumented Indentation

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The hot section components in gas turbines are exposed to extreme environments, with high temperatures and chemically aggressive atmospheres, in combination with applied loads, often causing damage to turbine blades. Since the original production operation is expensive, repair of worn or damaged parts is highly desirable. The most promising procedure for such repair is Vacuum Plasma Spraying. In this study, single crystal CMSX-4 substrates have been sprayed with CMSX-4 powder (creating polycrystalline coatings of thickness ~500-1000 µm). This has been done under various conditions, including the use of a heated stage in the spray chamber (allowing the substrate temperature to be raised to 1000˚C), different powder sizes etc. Effects of post-deposition heat treatments were also examined and correlations were explored with microstructural information. Instrumented indentation was carried out on the coatings, in both in-plane and through-thickness directions, using relatively large (diameter 4 or 5 mm) spherical indenters to ensure that representative volumes were being tested. Iterative FEM simulation of the indentation process [1] was carried out, using SEMPID software, to evaluate the parameters characterizing the yielding and work-hardening behaviour. It is shown that a measure of control over these plasticity characteristics can be obtained by varying the spraying and heat treatment conditions, and that stress-strain properties fairly close to those of the substrate can be obtained.

[1] J Dean & TW Clyne. *Extraction of Plasticity Parameters from a Single Test using a Spherical Indenter and FEM Modelling*. Mechanics of Materials **105** (2017) p.112-122.

Poster 7

Optimisation Algorithms for Extracting Material Properties from Indentation Data

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Inverse FE is an established method, but the nature of parameter space and the "goodness of fit" vary by constitutive law, the nature of the experiment, and the choice of statistic to define best fit. These lead to sensitivity issues that can cause extremely inefficient optimization of the material properties or failure to optimize to a global minimum. Some characterization of the parameter space is explored, along with a comparison of various optimization algorithms and goodness of fit statistics.

Poster 8

Issues for SEMPID Usage: Compliance Calibration, Indenter Radius and Penetration, Constitutive Law Selection, Friction and Material Anisotropy

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SEMPID (Software for the Extraction of Material Properties from Indentation Data) is a suite of user-friendly packages for inferring mechanical properties from indentation data (primarily load-displacement plots, although residual indent dimension data can also be used). The version for plasticity is now entering commercial usage. It involves iterative FE simulation of the penetration of a spherical indenter into a sample, with automated convergence on a best-fit set of parameter values that characterize the yielding and work hardening characteristics of the material (in a constitutive law). Issues involved in selection of the experimental conditions and of the model boundary conditions are investigated. These include the key dimensional scales of the indenter radius, R, and the depth of penetration, δ (and the ratio δ/R). A brief analysis is presented of the potentially conflicting requirements of deforming a volume large enough to represent bulk behavior and having a value of δ/R that creates plastic strains into a range that will adequately capture the work hardening response. The outcome is that a “mid-range” indentation facility is required, with a load capability of at least a kN, able to create δ/R values up to ~25%, with R ~0.5-2 mm. Other experimental issues covered include displacement measurement techniques and calibration of the machine compliance. Factors involved in choosing a constitutive plasticity law are briefly outlined. Also covered are the significance of interfacial friction and the possibility of material anisotropy (primarily as a consequence of crystallographic texture).

Area 3: Interfacial Adhesion, Fracture & Toughness

Poster 9

Reliable Measurement of Fracture Toughness of Ceramics at the Microstructural Scale

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Toughness measurements of individual microstructural components of ceramics are indispensable to a full understanding of their mechanical properties and impact behaviour. Such measurements can be made by testing microcantilever beams manufactured using focused ion beams (FIB) and including a notch at the chosen microstructural feature. However, residual stress caused by Ga-ion implantation at the notch root is thought to influence the results and therefore causes inaccurate toughness values. The present study attempts to solve this problem by two methods: (a) clarifying the extent of the effect of the implantation stress (IS), and (b) enabling stable crack growth prior to failure. The implications of the results for microstructural toughness measurements are described.

Poster 10

Deriving Hardness from Load-displacement Data in the Presence of Extensive Cracking

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When measuring the hardness of hard materials at typical loads for hardness measurements for armour materials, the number of indents whose size can actually be measured decreases rapidly with increasing load due to extensive cracking and the removal of material during unloading. Only measuring the indents, which have survived is not good laboratory practice as there could be a systematic reason why some indents survive and others not and hence the hardness obtained from a subset of indents might be biased. There is therefore a need for a method of determining the hardness of such indentations that does not rely on examination of the indentations after unloading. For small indentations, the use of load-displacement data recorded during loading and unloading is now well established for this purpose but when the loads are increased on a range of hard materials it was found that the derived values of the elastic modulus of the materials decreased at the loads where cracks started to be observed. Since the elastic modulus of the material is constant this indicates that some of the cracks form during loading and alter the interrelation between displacement and contact area by adding further compliance to the system. A study of the variation of compliance confirmed this. Therefore a new analysis was carried out and yielded a method, which can account for the cracking and give averaged hardness values from series of indentations at different loads.

Area 4: Microstructural and Mechanistic Effects

Poster 11

About the Use of Nanoindentation Testing to understand Microstructural Evolution Induced by Surface Thermomechanical Treatments

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Thermomechanical surface treatments - such as shot peening, burnishing, deep rolling, machining, … - are known for their efficiency to improve resistance to abrasive wear and local fatigue crack propagation. They are based on repeated contact loadings that create large plastic strains in the near-surface leading to compressive residual stress field and local grain refinement. A significant gradient of mechanical properties over 100 µm is usually observed. This paper aims to present a methodology based on nanomechanical testing –i.e. micropillar compression, nanoindentation - and EBSD measurements to explain microstructure changes induced by such treatments. This methodology is applied to various cases ranging from severe shot peening to sliding friction contacts. Such treatments combine effects of severe plastic deformation and high temperature rises, and thus enhances recrystallization phenomena. The resulting gradient of mechanical properties is shown to be significantly affected by this phenomenon. It led us to develop a new and original high-temperature nanoindentation procedure that addresses the issue of the measurement of recrystallization kinetics using nanomechanical testing.

Poster 12

About the Use of Nanoindentation Testing to understand Microstructural Evolution Induced by Surface Thermomechanical Treatments

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Poster 13

Organically Linked Iron Oxide Nanoparticle Supercrystals with Exceptional Isotropic Mechanical Properties

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It is commonly accepted that the combination of nanosize and elongated mineral constituents in biological materials is the main reason for their exceptional mechanical properties as compared to their rather weak mineral and organic constituents. Here we show that the self-assembly of spherical iron oxide nanoparticles in supercrystals linked together by a thermally induced coupling reaction of oleic acid or oleyl phosphate molecules leads to nanocomposites with exceptional microbar bending modulus and strength. Because the used nanoparticles are spherical, the shear load transfer mechanism for elongated particles is not responsible for the high strength. Instead, it is the covalent backbone of the linked organic molecules, which dominates the mechanical properties, leading to also a very high nanohardness. The self-assembly of the nanoparticles as well as mechanical properties of the supercrystals are discussed.

Poster 14

Understanding Size Effects in the Mechanical Behavior of Nanoporous Gold through MicroLaue Diffraction

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Poster 15

New Methods for Determining the Influence of Size Effects in Nanoindentation Testing

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Indentation size effects are well-known, in which materials appear to have higher hardness for smaller indentation sizes. This presents a problem whenever it is desirable to project to macroscopic mechanical property values from nanoindentation data, such as in the critical health monitoring of engineering structures. Here we report on test methods developed as part of the EU Strength-ABLE programme, based on Berkovich nanoindentations at a series of pre-determined indentation depths at the same location using a multi-cycle loading protocol. To investigate this method, a series of homogeneous metals are tested with varying internal length-scale through changes in grain size, work hardening and precipitate size and spacing, and hence varying the relationship between indentation size and material microstructure size. However, the measurement is complicated by the phenomenon of pile-up, which underestimates the true contact area of indentation, leading to overestimated values of hardness and modulus. We have developed an automated AFM method to determine the true contact area, the area in the plane of the original surface, and an estimate of the plastic zone size. Comparison of these measurements with the nanoindentation data provides a means to assess the length-scale dependence of the material. Determining the influence of size effects allows for new methods that can extract size-independent material properties. Furthermore, the method can be extended to extract estimates of internal length-scales within the material through consideration of the size-dependent response.

Poster 16

Beyond Hall-Petch: Mechanism-based Description of Dislocation-Grain Boundary Interactions

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The important role of grain boundaries as an obstacle for dislocation slip has long been recognized. However, until today, there are no quantitative, mechanism based models describing the interaction of a lattice dislocation with one particular grain boundary. The unique possibilities offered by micro pillar compression complemented by advanced in situ characterization (SEM, TEM, µLaue) is well able to quantitatively answer fundamental questions in dislocation grain boundary interaction: What is the impact of one grain boundary on (i) the observed yield stress and (ii) the measured apparent hardening? (iii) Can we define a meaningful “transmission stress”? (iv) Is the dislocation slip transfer process strain-rate dependent and (v) what is the strain rate dependence quantitatively? Within the contribution, these question will be answered for various grain boundaries in copper. Besides low angle grain boundaries also two different high angle grain boundaries – one permitting and one preventing dislocation slip transfer – and finally a coherent S3 twin boundary will be presented.

Area 5: Indentation of Soft / Compliant (Bio)Materials

Poster 17

Dynamic Nanondentation of Highly Compliant Hydrogels: A Critical Comparative Analysis with Rheometry

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Poster 18

A Unified Model for the Solute Diffusion in Hydrogels

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The usability of hydrogels on biomedicine depends on the control of an effective diffusion of solutes. Predicting this diffusion is challenging due to the peculiar structure of hydrogels. Currently, the diffusivity of solutes in hydrogels is modeled by at least one of three main theories –hydrodynamics, free volume and obstruction theory–, which assume different diffusion mechanisms. However, a standard, accurate predictive model is lacking, so that time –and capital– intensive trial-and-error procedures are used to test the viability of hydrogel applications. In this presentation we will present a model for the diffusivity of solutes in hydrogels that combines the three main theories. The model is verified by a combination of instrumented indentation, positron annihilation lifetime spectroscopy (PALS) and fluorescence recovery after photo-bleaching (FRAP) in polyethylene glycol (PEG) hydrogels with various mesh sizes. Our model outperforms traditional models in predicting solute diffusivity. This study may therefore improve the understanding of the physics behind diffusion in hydrogels. Our model may also be useful in predicting the transport properties of required molecule/hydrogel combinations in many biomedical applications.