International Workshop on Mechanistic Behaviour of HCP Alloys

30TH, 31ST MARCH AND 1ST APRIL 2016

PEMBROKE COLLEGE, UNIVERSITY OF OXFORD, UK

Final Programme

Organising Committee:

Ben Britton, Fionn Dunne and Saira Naeem (Imperial College London)

Welcome & workshop overview

We would like to welcome you to our workshop that focusses on the behaviour and performance of hexagonal metals. These materials are an important and interesting class of materials used in many industrial applications and their behaviour is rather interesting and unknown. The aim of this workshop is to draw together a collection of leading academic and industrial experts to discuss state-of-the-art research and to scope out important challenges in this exciting area of research. We expect that discussion will be wide ranging and cover issues such as: experiments & simulations of deformation in HCP alloys across time and length scales; non-destructive testing & innovative characterisation strategies; high rate deformation & foreign object damage; and engineering with HCP alloys.

This workshop has been put together with a view to encourage discussion amongst all participants. We have four inspiring keynote lectures (Mills, Curtin, Grey and Dawson) that will explore the behaviour of materials across time and length scales using sophisticated experimental and modelling techniques. These are supported by two industrially led discussion sessions (Romero, Darby, Rugg and Fox) that will provide a forum to outline needs, progress, achievements and future directions to promote synergies between R&D and ultimately enhance impact.

The majority of the workshop is dedicated towards a wide range of exciting and interesting contributed talks and posters. The thirty-six contributed talks are loosely grouped into themes of three or four talks, with only 'burning questions' allowed within a session. After each block of talks we will invite all speakers together from the session, and with active audience participation, we will encourage the group to query, argue and develop ideas within the dedicated discussion sessions. Finally, we are looking forward to our fourteen posters which provide an opportunity for further discussions over drinks & nibbles.

We are fortunate to be able to host our workshop in the lovely surroundings of Pembroke College Oxford. The College provides an intimate venue with excellent conference facilities on site, with closely linked breakout spaces and a well-equipped and modern lecture theatre. Registration for the workshop includes tea & coffee in the breaks, lunch and food as indicated within the programme. The banquet dinner is to be hosted in the College dining hall and we're looking forward to some extra tasty food and light entertainment, which will provide a casual backdrop to further discussions and networking. If you have chosen to have accommodation with your registration, this is provided for the Wednesday and Thursday nights only (extra nights are available within the college, but these must be organised in advance).

We would like to thank EPSRC for providing strategic funding to support this workshop and the HexMat Programme Grant for supporting our research. We are also tremendously thankful to our invited speakers from academia and industry for agreeing to participate in the workshop.

Please find our preliminary programme attached and we very much hope that you enjoy the workshop!

Regards,

Dr Ben Britton, Professor Fionn Dunne and Dr Saira Naaem

Programme Overview - Wednesday:

09:00	Registration, coffee & biscuits	
09:40	Welcome & workshop outline	
	Session 1- Chair: Tony Paxton	
10:00	Z.X. Wu and W. A. Curtin	Keynote: Dislocation Mechanics of Plasticity in Mg with Implications for Other HCP Metals
10:40	Emmanuel Clouet, Nermine Chaari, David Rodney and Daniel Caillard	First-Principles Modelling of Screw Dislocation Mobility in Zr and Ti
11:00	J. D. Robson and P. Hidalgo-Manrique	The Effect of Precipitation on Mechanical Anisotropy in Magnesium Alloys
11:20	P. Hidalgo-Manrique, J.D. Robson and T. Pérez-Prado	Hardening effect of precipitates against slip and twinning in magnesium-rare earth alloys
11:40	Haidong Fan, Sylvie Aubry, Athanasios Arsenlis, and Jaafar A. El-Awady	Grain Size Effects on Dislocation and Twinning Mediated Plasticity in Magnesium – A Discrete Dislocation Dynamics Study
12:00	Discussion	
12:20	Lunch	
	Session 2 – Chair: Ben Britton	
13:20	Industrial Discussion – Nuclear & Zr	Ted Darby (Rolls-Royce) and Javier Romero (Westinghouse)
14:00	Howard Chan, Steve Roberts, and Jicheng Gong	Micromechanical testing of zirconium hydride
14:20	Jing Hu, Alistair Garner, Sergio Lozano-Perez, Michael Preuss, and Chris Grovenor	High-resolution characterization of corrosion and hydrogen pickup of nuclear fuel cladding alloys
14:40	Michael Martin and Salah Muflahi	The Application of Coupled Structural and Diffusion Analysis to the Prediction of Delayed Hydride Cracking
15:00	Vivian Tong, and Ben Britton	Grain growth in Zircaloy-4
15:20	Discussion	
15:40	Coffee	
	Session 3 – Chair: Ian Jones	
16:00	D. Caillard, M. Rautenberg, X. Feaugas, F. Onimus, and E. Clouet	In situ straining experiments in Zr and Zr alloys at high temperature
16:20	Elisabeth M. Francis, Prasath Revathy, Allan Harte, Philipp Frankel, and Michael Preuss	Microstructural evolution in proton- and neutron irradiated Low Tin Zirlo® studied by Scanning transmission electron microscopy and atom probe tomography
16:40	Mitesh Patel, Daniel Balint, Mark Wenman and Adrian Sutton	Multiscale Modelling of Delayed Hydride Cracking
17:00	F. Fournier-dit-Chabert, M. Perrut, S. Naka and E.P. Busso	Effect of heat treatment on the microstructure and mechanical behavior of the Ti5553 alloy
17:20	Discussion	
17:40	Drinks + Posters	
19:30	Banquet Dinner & Award of Poster Prize	

Programme Overview - Thursday:

	Session 4 – Chair: Angus Wilkinson	
09:00	H. Li, I. Dastidar, V. Khademi, P.Eisenlohr, D.E. Mason, T.R. Bieler, M.A. Crimp, and C.J. Boehlert	Determination of critical resolved shear stress ratios for hexagonal deformation systems from surface slip trace analysis
09:20	Matthew Kasemer, Romain Quey, Donald Boyce, and Paul Dawson	Investigating the Influence of Microstructural Features on the Yield Strength and Ductility of Ti-6Al-4V
09:40	D. Lunt, T. Busolo, J. Quinta da Fonseca, and M. Preuss	Strain localisation behaviour in Ti-6Al-4V with a bi-modal microstructure
10:00	Soran Birosca, Peter Davies, Paul Garratt, and Roger Thomas	The Hierarchy of Microstructure Parameters Affecting Tensile Ductility in Cast and Forged Ti-834 Alloy during High Temperature Exposure
10:20	Discussion	
10:40	Coffee	
	Session 5 – Chair: Euan Wielewski	
11:00	George T. (Rusty) Gray III	Keynote: The Response of HCP-Alloys to Extreme Loading Environments
11:40	Jack Patten, Thomas White, Daniel Eakins, and David Chapman	High Resolution Strain and Temperature Imaging of Adiabatic Shear Bands at High Rates
12:00	Joao Quinta da Fonseca, Feng Li, and Alberto Orozco-Caballero	The effect of temperature on the formability of CP-Ti
12:20	T. Richeton, K.E.K. Amouzou, S. Berbenni, and M.A. Lebyodkin	Elasto-viscoplastic self-consistent modeling of hardening mechanisms in commercially pure alpha-titanium in tensile condition
12:40	A. Marchenko, M. Mazière, S. Forest, and J.L. Strudel	Polycrystalline modeling of dynamic and static strain aging phenomena in commercially pure alpha titanium.
13:00	Discussion	
13:20	Lunch	
	Session 6 – Chair: Michael Preuss	
14:20	Paul Dawson	Leverhulme Keynote: Developing Principle-Based Approaches to Quantify Mechanical Property Distributions of Titanium Alloys
15:00	Euan Wielewski, Donald Boyce, Jun-Sang Park, Matthew Miller and Paul Dawson	Determining Ti-6Al-4V material parameters using a discrete spherical harmonic analysis of lattice strain pole figures
15:20	Bo Lan, Michael J.S. Lowe and Fionn P.E. Dunne	Texture determination from ultrasonic wave speeds for HCP and cubic materials
15:40	Wenqi Li, Paul Marrow, Steven J Lainé, Matt Clark, and Steve D Sharples	Getting a grip on hexagons - surface acoustic wave interactions with hcp Ti
16:00	Discussion	
16:20	Coffee	
	Session 7 – Chair: Mark Rainforth	
16:40	V. Doquet and B. Barkia	Combined AFM, SEM and crystal plasticity analysis of grain boundary sliding in titanium at room temperature
17:00	Rajesh Korla, Jicheng Gong, Hamidreza Abdolvand, and Angus J Wilkinson	Investigation of Bauschinger effect at small length scales using new micro mechanical test geometry
17:20	Yi Guo, Ben Britton, and Angus Wilkinson	Slip-band grain-boundary interactions in titanium
17:40	Discussion	
18:00	Drinks + Posters	
19:30	Dinner	

Programme Overview - Friday:

	Session 8 – Chair: Bill Curtin	
09:00	Jicheng Gong and Angus J Wilkinson	Ultra Small Scale High Cycle Fatigue Testing by Micro-cantilevers
09:20	Tea-Sung Jun, Zhen Zhang, David Armstrong, Fionn Dunne, and Ben Britton	Strain rate sensitivity of Ti-6Al-2Sn-4Zr-xMo –micromechanical comparison between slip systems and alloys: Part 1 - Experiments
09:40	Zhen Zhang, Tea-Sung Jun, Ben Britton, and Fionn P.E. Dunne	Strain rate sensitivity of Ti-6Al-2Sn-4Zr-xMo –micromechanical comparison between slip systems and alloys: Part 2 - Simulations
10:00	Mitchell A. Cuddihy, and Fionn P. E. Dunne	Investigations into the effects of multiaxial stress states in cold dwell fatigue
10:20	Discussion	
10:40	Coffee	
	Session 9 – Chair: Minh Son-Pham	
11:00	Matt C. Brandes, Adam Pilchak, Stan Rokhlin, Somnath Ghosh, James C. Williams, and Michael J. Mills	Keynote: Titanium in aerospace applications - characterization driving model development
11:40	Olivier MacKain, Maeva Cottura, David Rodney and Emmanuel Clouet	Atomic scale study of twinning in zirconium
12:00	Zebang Zheng, Daniel S. Balint, and Fionn P.E. Dunne	Discrete dislocation and crystal plasticity analyses of load shedding in polycrystalline titanium alloys
12:20	Gi-Dong Sim, Steven Lavenstein, and Jaafar A. El-Awady	Crystal Size, Temperature, and Strain Rate Effects on the Competition between Slip and Twinning in Magnesium Microcrystals
12:40	Hamidreza Abdolvand, and Angus Wilkinson	Assessment of stress fields at deformation twin tips and the surrounding environments
13:00	Discussion	
13:20	Lunch	
	Session 10 – Chair: Fionn Dunne	
14:20	Industrial Discussion Session – Ti	Stephen Fox (Timet) and David Rugg (Rolls-Royce)
15:00	C.T. Gillen, A. Garner, P. Wiringgalih, A. Plowman, M. Preuss, C. P. Race, and P. Frankel	Development of a Novel Experimental Rig for Testing of Iodine-induced Stress Corrosion Cracking of Zirconium Alloys
15:20	S.B. Setiadinata, M.P. Moody, P.A.J Bagot, T.L. Martin, and C.R.M. Grovenor	Understanding the iron impurity redistribution during the oxidation of zirconium-niobium alloys cladding
15:40	Sudha Joseph, Trevor C. Lindley, and David Dye	Investigation of hot salt stress corrosion in Ti-6Al-2Sn-4Zr-6Mo
16:00	Discussion	
16:20	Conference Wrap-Up	
16:30	Finish	

Programme Overview - Posters:

P1	Maria S. Yankova, Cristopher P. Race	The effect of strain and interfaces on the relative stability of the tetragonal and monoclinic structures of Zirconium oxide – an atomistic modelling study
P2	Abigail K Ackerman, David Dye	Secondary alpha in Ti6246s
P3	Jack Patten, Thomas White, Jasmina Music, David Chapman, Daniel Eakins	High Speed Active Surface Temperature Imaging System, and its Integration with a Stereoscopic Digital Imaging Correlation Apparatus
P4	Rhys Thomas, David Lunt, Philipp Frankel, Michael Preuss	High resolution strain mapping of hydrides in zirconium alloy
P5	Christopher Daniel, Peter Honniball, Luke Bradley, Michael Preuss, João Quinta da Fonseca	Texture evolution in Zr-Nb alloys
P6	A.J. Knowles, N.G. Jones, C.N. Jones, H.J. Stone and D. Dye	Design, characterisation and properties of Ti-Fe-Mo alloys strengthened by ordered intermetallic precipitates
P7	Wiringgalih, P., Gillen, G., Garner, A., Frankel, P. and Preuss, M.	Comparison between cold-work hardening and irradiation hardening in zirconium alloys: preliminary study on iodine-induced stress corrosion cracking
P8	P. Kwasniak, H. Garbacz, K.J. Kurzydlowski	Solid solution strengthening of hexagonal titanium alloys: restoring forces and stacking faults calculated from first principles
P9	Yi Guo and Xavier Maeder	In-situ HR-EBSD characterization of twin nucleation and growth by micro-pillar compression
P10	M. R. Chini, L. Germain, N. Gey, S. Andrieu, T. Duval.	Advanced microtexture analysis of a Ti 10-2-3 product for better understanding of local variations in mechanical behavior
P11	Steven. J. Lainé, Kevin. M. Knowles and D. Rugg	Microstructural interactions of deformation twins in CP titanium and Ti–6Al–4V after room temperature ballistic impact testing
P12	M.A. Steiner, J.J. Bhattacharyya, and S.R. Agnew	The Origin and Enhancement of $\{0001\}\langle 11\bar{2}0\rangle$ Texture during Heat Treatment of Rolled AZ31B Magnesium Alloys
P13	Fulin Wang, Sean R. Agnew, Christopher D. Barrett and Haitham El Kadiri	Transmission electron microscope and molecular dynamics investigation of dislocation-twin interactions in magnesium
P14	Tea-Sung Jun, Xavier Maeder, Ben Britton	In-situ micropillar compression and HR-EBSD studies of morphological effects in a dual-phase Ti alloy

Session 1:

Keynote: Dislocation Mechanics of Plasticity in Mg with Implications for Other HCP Metals

Z.X. Wu[1,2], W. A. Curtin[2]

1. Institute for High-Performance Computing, A*star, Singapore
2. Ecole Polytechnique Federale de Lausanne, CH-1015 Lausanne, Switzerland

Plastic deformation is controlled by the motion of dislocations, and dislocation behavior is controlled by the nanoscale dislocation core structure. Atomistic simulations can reveal this behavior, but have proven difficult in Mg and other HCP metals due to the absence of accurate interatomic potentials. Here, we describe a suite of studies on Mg to reveal important features of deformation in Mg. First, we present a new interatomic potential for Mg that accurately predicts the dislocation core structures for basal, tensile twin, and Pyr. II <c+a> disocations in agreement with first-principles calculations. We then show that the easy-glide Pyr. II <c+a> edge dislocation undergoes a transition to a thermodynamically lower-energy state in which the dislocation is dissociated on the basal plane rather than the Pyr. II plane, and becomes immobile. Dissociation into <c> and <a> dislocations, with the <c> dislocation oriented along the basal plane, is also shown. These transitions are intrinsic to Mg and rationalize decades of experimental observations, and are the mechanistic origins for low ductility and anomalous strengthening in Mg. We then show that the same transition occurs for the Pyr. I mixed <c+a> dislocation in Mg, and relate this result to observations in Ti and Zr. Turning to the <c+a> screw dislocations, we present the mechanism of cross-slip between Pyr. I and Pyr. II dislocations in Mg, demonstrating that the cross-slip barrier depends on non-Schmid stresses, and that the relative stability of Pyr. I and Pyr. II screws can be changed by stress. This result is generalized to create a model for cross-slip in HCP metals. To modify dislocation behavior, and mitigate low ductility, we expect that solute additions can change the relative stabilities and energy barriers for the important transitions studied above. We thus review a quantitative model for solute strengthening, and demonstrate its application to basal <a> strengthening and twin strengthening. Overall, this fundamental base of mechanistic understanding of deformation in Mg is quantitative and so it can aid in the design of new Mg alloys.

First-Principles Modelling of Screw Dislocation Mobility in Zr and Ti

Emmanuel Clouet[1], Nermine Chaari[1], David Rodney[2] and Daniel Caillard[3]

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2. Institut Lumière Matière, Université Lyon 1, CNRS, UMR 5306, F-69622 Villeurbanne, France
3. CEMES-CNRS, 29 rue Jeanne Marvig, BP 94347, F-31055 Toulouse cedex, France

Titanium and zirconium have a close plastic behaviour arising from their hexagonal close-packed crystallography and from their similar electronic structure. In particular, plasticity in these two transition metals is controlled by screw dislocations gliding in the prism planes, with cross-slip in the first-order pyramidal planes or in the basal planes activated at high enough temperature and a strong hardening associated with O addition. We use ab initio calculations and NEB method to study core properties of the screw dislocations and their mobility in both metals. These calculations show that screw dislocations may adopt different cores that are dissociated either in a prism or in a pyramidal plane, in agreement with the existence of stable stacking faults in these two planes [1]. The prismatic core easily glides in its habit plane, whereas the pyramidal core needs to overcome an important energy barrier to glide. The prismatic glissile core is the most stable in Zr, but the dislocation ground state in Ti corresponds to the pyramidal core. As a consequence, dislocation glide is easy and confined in the prismatic planes at low temperature in pure Zr, whereas a locking-unlocking mechanism operates in Ti where the locked periods correspond to a slow and limited glide in pyramidal planes and the unlocked periods to a rapid and extended glide in prismatic planes, in agreement with in situ TEM straining experiments [2]. Calculations in Zr also reveal that basal glide of the screw dislocation share the same thermally activated process as pyramidal glide. Finally, we study the interaction of an oxygen atom with these different configurations of the screw dislocation. Ab initio calculations evidence a strong repulsion with the oxygen repelling the stacking fault ribbon, thus inducing dislocation cross-slip.

[1] N. Chaari, E. Clouet and D. Rodney, Phys. Rev. Lett. 112, 075504 (2014).
[2] E. Clouet, D. Caillard, N. Chaari, F. Onimus and D. Rodney, Nature Materials 14, 931 (2015).

The Effect of Precipitation on Mechanical Anisotropy in Magnesium Alloys

J. D. Robson and P. Hidalgo-Manrique

University of Manchester

The plastic deformation behaviour of magnesium crystals is highly anisotropic. Combined with the strong crystallographic textures, this leads to undesirable plastic asymmetry and anisotropy in polycrystalline wrought magnesium products. Many magnesium alloys exploit precipitation to obtain strengthening, and precipitation in magnesium itself provides highly anisotropic strengthening against both slip and twinning. This effect can either mitigate or enhance the inherent plastic anisotropy. In this presentation, the interaction of precipitates formed in magnesium with the various slip and twinning systems will be discussed. It is shown that simple Orowan based strengthening models can help explain the effect of precipitates on anisotropy in the case of slip mediated deformation. In the case of twinning, such models do not work. Instead, it is shown that the back-stress caused by precipitates embedded in twins often provides a dominant strengthening effect. A detailed exploration of the effect of precipitates on twinning in magnesium is presented. It is shown that the critical stress at which twins are nucleated is often largely unaffected by the presence of precipitates, but twin growth can be strongly affected. A simple back-stress based model is used to explain this effect and make predictions of how twin size and number density are affected by the presence of precipitates that compare well with observations.

It is shown that by careful selection of precipitation system, the desirable property goals of effective strengthening and reduced anisotropy may be obtained through preferential strengthening of particular deformation modes.

Hardening effect of precipitates against slip and twinning in magnesium-rare earth alloys

P. Hidalgo-Manrique[1], J.D. Robson[1] and T. Pérez-Prado[2]

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Magnesium alloys are promising structural materials due to their weight-saving possibilities. However, due to the low symmetry of the hcp lattice and the polarity of twinning, Mg wrought products, typically characterised by strong textures, are affected by distinct room temperature yield stress anisotropy and asymmetry, which restricts many of their potential applications. Additions of rare-earth (RE) elements is a very effective method of reducing the problem. This has been usually attributed to the development of significantly weaker deformation and/or recrystallization textures. However, some studies suggest that the mechanical behaviour of the RE-containing Mg alloys, containing prismatic precipitate plates, is actually determined by microstructure, able to alter the deformation system activity. In this work, the hardening effect of precipitates against slip and twinning is investigated. For this purpose, an extruded MN11 alloy (Mg-1 wt.% Mn-1 wt.% Nd) was thermally treated in order to obtain different amounts and sizes of precipitates. Afterwards, the particle-containing bars were tested under tension and compression at room temperature. A quantitative analysis of precipitation as well as examinations on the interaction between particles and dislocations were performed by transmission electron microscopy. Adequate expressions of the Orowan equation were developed for the predominant deformation systems and the effect of back-stress on such systems were estimated, the agreement between the predictions and the experimental data being checked and discussed. In addition, the effect of the relative critical resolved shear stress for the different deformation systems induced by precipitates on the so-called reversed yield stress asymmetry phenomenon, occasionally observed in Mg-RE wrought products, including the present MN11 extruded bar, was also examined.

Grain Size Effects on Dislocation and Twinning Mediated Plasticity in Magnesium – A Discrete Dislocation Dynamics Study

Haidong Fan[1], Sylvie Aubry[2], Athanasios Arsenlis[2], and Jaafar A. El-Awady[1]

1. Department of Mechanical Engineering, Johns Hopkins University, Baltimore, MD 21218, USA
2. Materials Science Division, Lawrence Livermore National Laboratory, Livermore, CA, USA

Understanding the competition between twinning and dislocation slip is of significant importance for the fundamental understanding of plasticity in hexagonal-close-packed (HCP) materials. Towards developing such an understanding, a unified discrete dislocation dynamics (DDD) framework for modeling dislocations, twinning, and their interactions in magnesium (Mg) polycrystals has been developed. A systematic interaction model between dislocations and {10-12} tension twin boundaries (TBs) was introduced into the DDD framework, and a nominal grain boundary (GB) model based on experimental results was also introduced to mimic the GB's barrier effect. This new framework is then utilized to study grain size effects on the competition between dislocation slip and {10-12} twinning in the deformation of micro-twinned polycrystalline Mg. It is shown that twinning deformation exhibits a strong grain size effect; while dislocation mediated slip in untwinned polycrystals displays a weak one. This leads to a critical grain size at 2.7 microns, above which twinning dominates, and below which dislocation slip dominates. Furthermore, the predicted evolution of the twin boundary, the twin boundary velocity, and the morphology of the twin boundary are all compared to experimental results and predictions from molecular dynamics simulations.

Session 2:

Micromechanical testing of zirconium hydride

Howard Chan, Steve Roberts, Jicheng Gong

Department of Materials, University of Oxford

Hydrogen diffusion in hexagonal zirconium based nuclear claddings can lead to embrittlement. The brittle fracture of the microscale Zr hydride is significantly involved in delayed hydride cracking (DHC). Pre-notched microcantilevers were prepared using focused ion beam (FIB) in the hydride phase or in the Zr metal phase individually to compare their micromechanical properties. The pre-notch was also milled along the phase boundary in order to test the fracture properties of the interface. The microcantilevers were tested in bending using a nanoindenter at room temperature for the load-displacement measurement. The analysis showed that three types of specimens have distinct deformation properties. The fracture toughness and the yield strength of the microscale Zr hydride were determined and compared with macroscopic testing results. Cleavage was observed on the fracture surface of the hydride while interfacial voids were seen along the Zr-hydride phase boundary after testing.

High-resolution characterization of corrosion and hydrogen pickup of nuclear fuel cladding alloys

Jing Hu[1], Alistair Garner[2], Sergio Lozano-Perez[1], Michael Preuss[2], Chris Grovenor[1]

1. Department of Materials, University of Oxford, Parks Road, Oxford, UK
2. Materials Performance Centre, School of Materials, University of Manchester, UK

As part of the MUZIC2 project, several high resolution analysis techniques have been used to study the microstructure of a range of commercial and developmental Zr alloys under autoclave simulated PWR conditions1. Samples were chosen to be representative of the early, pre-transition, transition point and post-transition stages of the oxidation process. We have studied the development of the equiaxed-columnar-equiaxed grain structure using TEM and TKD (Transmission Kikuchi Diffraction), and observe that the columnar grains are both longer and show a stronger preferred texture in more corrosion-resistant alloys2. Fresnel imaging revealed the existence of both parallel interconnected pores and some vertically interconnected pores along the columnar oxide grain boundaries, which become more disconnected near the metal-oxide interface. We have studied the distribution and composition of second phase particles by TEM, SEM and glancing angle XRD analysis, and EDX mapping revealed the preferential dissolution of Fe-containing particles in the regions of columnar oxide grains. These particles also have a lower number density at the metal-oxide interface in the post-transition samples than pre-transition samples. Electron Energy Loss Spectroscopy analysis and automated crystal orientation mapping with transmission electron microscopy have also revealed Widmanstatten-type suboxide layers in some samples with compositions in different regions of either the hexagonal ZrO structure predicted with ab initio modelling3 or Zr3O2. Some of these layers are much thicker than observed previously in other oxidised zirconium alloys4. Preliminary data on neutron irradiated Zr alloys shows preferential dissolution of Fe and Cr inside SPPs, also shorter columnar oxide grains. These observations will be discussed in the context of current models for oxidation and hydrogen pickup in zirconium alloys.

The Application of Coupled Structural and Diffusion Analysis to the Prediction of Delayed Hydride Cracking

Michael Martin and Salah Muflahi

Rolls Royce plc

Zirconium alloys, as used in water-cooled nuclear reactors, are susceptible to a time-dependent failure mechanism known as Delayed Hydride Cracking, or DHC. Corrosion of zirconium alloys in the presence of water generates hydrogen that subsequently diffuses through the metallic structure in response to concentration, temperature and hydrostatic stress gradients. As such, regions of increased hydrogen concentration develop at stress concentrating features, leading to zirconium hydride precipitation. Regions containing zirconium hydride are brittle and prone to failure if plant loads are sufficient.

This presentation will describe the DHC mechanism before leading to a discussion of the application of coupled structural and diffusion analysis to the prediction of DHC. In particular, the use of sequentially coupled finite-element analysis will be discussed in which structural analyses and hydrogen analyses are undertaken sequentially with coupling between the two provided by the hydrostatic stress field. At a particular point in a thermo-mechanical load cycle, the method proceeds by running a hydrogen diffusion analysis with the resulting field of hydride volume fraction collapsed to a single region to provide an equivalent hydride length. Simple process-zone representation of this single equivalent hydride within the structural analysis is achieved using standard cohesive elements. To match the equivalent length obtained from the hydrogen diffusion analysis, the cohesive strength of the process-zone is adjusted and the structural analysis re-run. The resulting hydrostatic stress field is passed back to the hydrogen diffusion analysis and the process repeated. In this way, the change in stress field at a stress concentrating feature that results from precipitation-derived transformation strain can be accounted for and the ensuing development of hydride regions with thermo-mechanical cycling understood. The requirements for the supporting experimental programmes necessary to provide input data and validation are also addressed.

Grain growth in Zircaloy-4

Vivian Tong, Ben Britton

Department of Materials, Imperial College London, United Kingdom

Zircaloy-4 is in nuclear reactors in the form of thin-walled fuel rod cladding tubes. Maintaining a fine grain size is important to withstand the large thermal, mechanical and irradiation stresses in operation. Under certain conditions, abnormally large grains, or blocky alpha, grow within the small grained matrix, which is undesirable for structural integrity of the fuel rod cladding. Understanding the mechanism by which blocky alpha nucleates and grows is essential for both optimising manufacturing processes and understanding in-service performance. In this work, a strain-anneal method for consistently growing blocky alpha grains has been developed. It was found that grain size after annealing is dependent on the applied strain, and the critical strain required for grain growth is temperature dependent. The grain growth kinetics has also been studied, and a mechanism for blocky alpha formation is proposed based on these results. Understanding blocky alpha formation in zirconium alloys will enable manufacturers to avoid certain strain paths and temperatures in order to avoid blocky alpha e.g. during the pilgering process in tube manufacture, and also allow the long term microstructural stability of Zr in nuclear reactors to be better understood and potentially optimised.

Session 3

In situ straining experiments in Zr and Zr alloys at high temperature

D. Caillard[1], M. Rautenberg[2], X. Feaugas[3], F. Onimus[4], E. Clouet[4]

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The dislocation mechanisms responsible for the high-temperature mechanical properties of Zr and Zr alloys are still poorly understood. Indeed, whereas the role of dynamic strain ageing close to 350°C seems well established, various mechanisms have been proposed to account for a more or less constant stress exponent n ~ 6 above this temperature.

In situ straining experiments have been carried out in pure Zr and in a M5 alloy between 20°C and 450°C. Above 20°C, dislocations start to cross slip in the basal plane where screw segments are subjected to a high Peierls friction force. No indication of cross slip in pyramidal planes has been obtained at any temperature. At 350°C, the kinetics of mobile dislocations becomes very jerky and inhomogeneous, in agreement with a dynamic strain ageing mechanism.

Above 350°C, the motion is again steady and homogeneous in the M5 alloy. Extensive cross-slip forms super-jogs which are efficient pinning points against the glide motion. These super-jogs can however be eliminated by glide along the Burgers vector direction, never by climb. The glide velocity between super-jogs is linear as a function of the total driving stress (applied stress minus line-tension stress due to dislocation curvature), in agreement with the solute dragging mechanism. The origin of the stress-strain rate dependence with an exponent larger than unity is then discussed.

Microstructural evolution in proton- and neutron irradiated Low Tin Zirlo® studied by Scanning transmission electron microscopy and atom probe tomography

Elisabeth M. Francis, Prasath Revathy, Allan Harte, Philipp Frankel, and Michael Preuss

University of Manchester

Under certain operating conditions, zirconium alloys used as cladding material in nuclear reactors show accelerated growth behaviour induced by neutron irradiation. The occurrence of accelerated growth has been linked to the formation of c-component dislocation loops and an increase in solute concentration around second phase particles, but the mechanisms are still not fully understood. Improvements in analytical electron microscopes mean that smaller variations in the local composition can be determined and mapped with greater spatial resolution than previously possible. A (scanning) transmission electron microscope capable of such high resolution EDX spectral imaging (FEI Titan G2 S/TEM “ChemiSTEM”) was employed here to record the distribution of alloying elements in non-irradiated as well as proton- and neutron irradiated zirconium alloys in order to study the mechanisms leading to irradiation growth. The results of the EDX spectral imaging and transmission electron microscopy will be compared to atom probe tomography. The work presented here focuses on a Zr-Nb-Fe-Sn alloy. The evolution of the dislocation structure and its relation to the alloying elements will be detailed. Nb and Fe are found to form clusters during irradiation. Nb clusters are associated with dislocation loops after proton irradiation to 2.3 dpa according to the APT and EDS spectral imaging.

Multiscale Modelling of Delayed Hydride Cracking

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Delayed hydride cracking (DHC) is a failure mechanism affecting zirconium alloy components in nuclear reactors. In the reactor environment, an aqueous corrosion process allows hydrogen to enter the bulk zirconium matrix. Through diffusion under the influence of gradients of stress, chemical potential and temperature, the H atoms form elevated concentration profiles ahead of stress-raisers such as loaded cracks and notches. Once the solvus is exceeded, zirconium hydride platelets precipitate in the vicinity of the flaw tip. The hydride phases are significantly more brittle than the parent metal and hence have a detrimental effect on the mechanical properties of the component. As such, these hydrides are more prone to fracture, which enables the flaw to propagate. The interplay and repetition of diffusion, precipitation and fracture can ultimately lead to structural failure of the component. The overarching aim of DHC research is to quantify this complexity and develop a rigorous failure criterion.

We present a multiscale mathematical model of DHC sub-phenomena in which analytical calculations play an important role. In particular, the stress state of the system is determined using theoretical continuum mechanics: the planar elasticity methods of Green tensors, complex potentials and conformal mappings. Subsequently, linear irreversible thermodynamics is used to study stress-driven diffusion and obtain equilibrium hydrogen profiles. As hydrogen atoms preferentially occupy tetrahedral sites in hexagonal close-packed zirconium, the anisotropy exhibited at the atomic scale is described using the elastic dipole model of point defects. A unique simplistic treatment of hydride needles as Somigliana-Volterra dislocation dipoles is discussed. Additionally, various techniques concerning the computational geometry of polygons are employed to study the effects of heterogeneity and incorporate a set of constitutive rules for the morphological evolution of hydrides. The advantage of this framework is that the calculations are less computationally intensive, supporting a statistical treatment of the DHC problem.

Effect of heat treatment on the microstructure and mechanical behavior of the Ti5553 alloy

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The Ti-based alloy Ti5553 is a beta-metastable industrial material with potential applications in aircraft landing gears. In this work, the influence on the microstructure and the mechanical behavior of a post-forging heat treatment has been studied. Possible variations in the microstructure in terms of the primary (nodules size and volume fraction) and secondary (needles size and fraction) alpha phases are identified. The main relationship between these microstructural features and the alloy's mechanical properties obey a Hall-Petch type relation expressed in terms of the mean spacing between the secondary alpha needles. Finally, a suitable constitutive model is proposed to describe the cyclic behavior of this alloy.

Session 4:

Determination of critical resolved shear stress ratios for hexagonal deformation systems from surface slip trace analysis

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Since the direct measurement of individual critical resolved shear stress (CRSS) values in hexagonal crystals faces experimental challenges, an alternative methodology using the statistical relation between (global) Schmid factor and the frequency of surface slip trace occurrence for a population of grains is proposed. The determination of Schmid factors and slip trace occurrence is based on grain orientation information from electron backscatter diffraction and scanning electron microscopy images, respectively. Implications of the size of the statistical population on the confidence of CRSS ratios is explored. The methodology is applied to a number of Ti alloys and reveals trends of slip system resistance with temperature and alloy compositions.

Investigating the Influence of Microstructural Features on the Yield Strength and Ductility of Ti-6Al-4V

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Ti-6Al-4V is a dual phase alloy that tends to have a large variation in the geometric features of its microstructure due to the thermomechanical processes used during manufacture. The mill-annealed microstructure is characterized by relatively small (~10um) equiaxed grains of HCP (alpha) phase and laths of BCC (beta) phase between grains. The complex beta-annealed microstructure contains alpha transformation structures (colonies) and remnant beta phase (lamella) that form within large (~500um) prior beta grains that were present above the transis temperature. The macroscopic response of these two microstructures is markedly different. When pulled in tension along the rolling direction, the mill-annealed microstructure tends to have a higher yield strength and ductility than the beta-annealed microstructure, as well as a smaller degree of variation in these measures. By simulating the response of virtual specimens to the point of deformation localization, and altering the geometric parameters of the virtual specimens, the influence of the microstructural features on the yield strength and ductility may be deduced. High fidelity virtual polycrystals are instantiated to include fine details, especially those of the beta-annealed microstructure. A method known as multilevel tessellation is utilized to mimic the prior beta grains, alpha colonies, and remnant beta lamella. Crystal plasticity finite element simulations are used to solve for the stress strain histories of virtual specimens. Mill-annealed specimens are instantiated with 4000 grains, (~750k elements), whereas the beta-annealed specimens contain 40 prior beta grains and multiple colonies per grain, yet require ~1.5M elements due to the presence of lamella, thus necessitating the use of a highly parallelized computational framework. Presented are the results of these simulations, detailing the difference of onset of necking between the two microstructures, as well as the influence of the finer geometric features of the beta-annealed microstructure.

Strain localisation behaviour in Ti-6Al-4V with a bi-modal microstructure

D. Lunt, T. Busolo, J. Quinta da Fonseca and M. Preuss

University of Manchester

High Resolution Digital Image Correlation (HR-DIC) has been used to study the effect of Ti3Al precipitates in Ti-6Al-4V on strain localisation in primary and secondary alpha grains of a bi-modal microstructure. The nanoscale Ti3Al precipitates are known to provide additional strengthening but also promote slip planarity. Due to the way a bi-modal microstructure is obtained Ti3Al is expected to predominantly form in the primary alpha phase. The bi-modal microstructure was created by heat-treating the as-received material at 950°C followed by water quenching. Subsequently, some of the material was aged at 550°C to promote Ti3Al precipitation while a second batch was annealed above the Ti3Al and water quenched. Full field surface strain maps were produced by correlating images at progressive applied strains with the image from the previous deformation stage. The maximum shear strain frequency distributions in the primary and secondary alpha phases have been compared to study the effect of the Ti3Al precipitates. The theoretical slip trace angles for all possible slip systems were calculated using Electron Back Scattered Diffraction (EBSD) orientation data and cross-correlated with experimental slip trace angles measured from nanoscale shear strain maps to predict the active slip domain for the primary alpha phase. The shear strain was then correlated with the slip activity by comparing the maximum shear strain frequency distributions for each slip system.

The Hierarchy of Microstructure Parameters Affecting Tensile Ductility in Cast and Forged Ti-834 Alloy during High Temperature Exposure

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Ductility regression is the main concern in using Ti-834 alloy at temperatures above 500°C for aerospace applications. Here the effect of prolonged exposure at 500°C on the tensile ductility of cast and forged Ti-834 alloys was investigated. The effect of microstructure, microtexture, α-case, α2 and silicide precipitate coarsening during high temperature exposure was studied. The effects of individual microstructural parameters on the ductility regression in Ti-834 alloy were quantified. The results showed that 7.1% strain differences between the cast and forged alloy are related to microstructural variations including; morphology, lath widths, grain size and shape, grain orientations and microtexture. A total of 9.6 % strain loss was observed in cast Ti-834 after aging at 500°C/500h and quantified as follow; 3.6% due to α-case formation during high temperature exposure, 0.2% due to α2-precipitates coarsening, 4.4% due to further silicide formation and coarsening, 1.4% due to further microstructure changes during high temperature exposure. Furthermore, silicide coarsening on α/β phase boundaries caused large void formation around the precipitates. A considerable element partitioning effect occurred in Ti-834 during exposure at 500°C/500h. Al and Ti depletion in the vicinity of the β phase in the lamellae, i.e., αs area, was responsible for reducing the strength of the alloy and facilitating the formation of TiZrSi precipitates. The Al depletion and nano-scale partitioning observed at the αs/β boundaries resulted in easy crack initiation and promoted propagation in the centrifugally cast colony microstructure and reduced the basal slip τcrss. Furthermore, silicides were not formed in αp (high Al, Ti and low Zr areas) in the forged duplex microstructure that promoted superior mechanical performance and ductility over the cast alloy, i.e. colony microstructure.

Session 5:

Keynote: The Response of HCP-Alloys to Extreme Loading Environments

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The high-strain-rate stress-strain, impact, and shock-loading response of hexagonal-closed-packed(HCP) alloys is receiving renewed interest related to systems calculations for crash-worthiness, foreign-object damage, high-rate machining, and ballistic applications. Interest in building physically-based predictive constitutive models to describe these processes requires a knowledge of the coincident influence of microstructure, temperature, strain rate, and texture on mechanical response and damage evolution, particularly under impact conditions. In this talk, a survey of the response of HCP-alloys to extremes in mechanical loading will be presented. An overview of the constitutive response of Ti-alloys, such as Ti-6Al-4V, Ti-54M, and Ti-5553, Ti-based intermetallics, as well as Mg-alloys, Zr, and Hf is reviewed and examples of how their mechanical behavior under dynamic loading can be modeled and then validated using Taylor impact testing is illustrated. The defect generation, rate sensitivity, and dynamic damage evolution response of HCP-alloys are discussed as a function of strain rate, temperature, and texture and contrasted to that in FCC and BCC alloys.

High Resolution Strain and Temperature Imaging of Adiabatic Shear Bands at High Rates

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Adiabatic Shear Band (ASB) formation is a route of catastrophic failure for materials in demanding, high-performance environments. Shear bands form on the scale of 10-100 µm, typically under high strain-rate (above 10^2 s-1) loading conditions. Such conditions are experienced in high-speed machining, jet engine turbines under rapid loading from a foreign object or ballistic penetration. Previously conducted experiments often aim to correlate material texture to shear band susceptibility by interrupting the experiment during loading. This can lead to results which are often difficult to interpret due to longer time scale effects and sample release. As such, results from these experiments are not necessarily representative of material structure under conditions of ASB formation.

We are developing combined 3D imaging of a surface as it is loaded to give high resolution in both temperature and strain, while maintaining high temporal resolution. This is the first time these capabilities will have been fielded simultaneously. Presented here is the approach for conducting experiments utilising a custom three-dimensional digital image correlation (DIC) system combined with reflectance thermometry specifically for high-rate experiments. Used in conjunction with a miniature Kolsky bar apparatus, and utilising a sample morphology designed to promote shear band formation in a specific region of known texture, strain concentrations and temperature fluctuations are being imaged in high resolution in situ. This direct imaging goes beyond the far-field methods of adiabatic shear band measurements such as recovery and strain gauges, leading to a closer comparison to microstructure-aware crystal plasticity models. This talk presents the overall approach of the combined system, while also highlighting some of the key challenges, such as surface texture, characterisation and lighting. Preliminary results of initial dynamic tests will also be shown as a guide for the capabilities of this combined system.

The effect of temperature on the formability of CP-Ti

Joao Quinta da Fonseca, Feng Li and Alberto Orozco-Caballero

The University of Manchester

It often reported that the ductility of HCP metals is limited by the the lack of independent slip systems, a situation which is only improved marginally by twinning. However, experiments show that, in uniaxial tension, elongation to failure of CP-Ti is about 30% at room temperature, increasing to over 80% at 500C. This increase in ductility with temperature cannot be explained by twinning since the amount of twinning is expected to decrease with increasing temperature. Instead, this increase in ductility with temperature is usually attributed to an increase in strain rate sensitivity with temperature. In this paper, we present the results of a study on the ductility and formability of Grade 2 CP-Ti with the aim of determining the mechanisms controlling ductility and formability. CP-Ti sheet was tested in uniaxial and biaxial tension between RT and 500°C. Work hardening rates, elongations to failure, R-values, strain rate sensitivities and biaxial stretching limits were determined at RT, 150°C and 500°C. We found that, whereas the ductility in uniaxial deformation increases between RT and 500C, the biaxial ductility decreases precipitously with increasing temperature, which cannot be explained by changes in twinning or strain rate sensitivity. Analysis of the deformation data and EBSD characterisation of the deformed microstructure suggests that the increase in uniaxial ductility is caused by an increase in the work hardening rate of the material with temperature. We propose that this is a direct consequence of an increase of the plastic anisotropy with temperature, which can also explain the dramatic decrease in biaxial ductility. CPFEM simulations lend support to this hypothesis.

Elasto-viscoplastic self-consistent modeling of hardening mechanisms in commercially pure alpha-titanium in tensile condition

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Tensile tests on commercially pure alpha-titanium show a three-stage behavior giving rise to a well on the strain dependence of the work hardening. An opposite strain rate effect on the well depth is found whether specimens are elongated along the rolling or the transverse direction. Slip lines analysis reveals an initial predominance of prismatic slip, particularly pronounced in specimens strained along the rolling direction. The relative activity of prismatic slip is then observed to decrease with the samples deformation. These results provide grounds for elaboration of an elasto-viscoplastic self-consistent model based on the translated field method and an affine linearization of the viscoplastic flow rule, and capable of explaining such peculiar work hardening behavior. The model considers crystal plasticity and deals separately with mobile dislocation density and dislocation velocity. It assumes lower strain rate sensitivity as well as higher dislocation multiplication rate for prismatic systems. Based on these assumptions, the model reproduces correctly the stress-strain curves and gives sound estimates of Lankford coefficients, prismatic slip activity and textures evolution. The effect of elastic anisotropy on these results, with the use of directional shear moduli in the Taylor hardening equation, is underlined. Most importantly, the opposite effect of strain rate on the well depth with regard to the orientation of the tensile axis is qualitatively retrieved, which allows putting forward an explanation of the observed phenomena.

Polycrystalline modeling of dynamic and static strain aging phenomena in commercially pure alpha titanium.

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The phenomenon of strain aging in titanium has been the object of multiple researches over the past few decades. Various physical mechanisms have been proposed in the literature to explain the occurrence of the strain aging in hexagonal close-packed metals in a large range of strain rates and temperatures. To date the issue of strain aging in Ti and its alloys stays debatable and needs a further investigation.

The experimental studies on commercially pure alpha titanium revealed a yield stress anomaly corresponding to static aging peak when the material is loaded in transverse direction. The presence of the stress peak can be attributed to the interaction of the activated <c+a> slip systems with the atoms of interstitial oxygen resulting in the dislocation pinning-unpinning process. On a pair with static strain aging, small serrations on the stress-stain curves typical for the Portevin-Le Chatelier effect have been observed at the lower strain rates. These serrations can be due to the non-planar core of screw-type dislocations that normally govern the room-temperature deformation of alpha titanium. The core structure of the screw dislocations can take alternatively two states: a metastable state, sessile spread in the prismatic plane and a stable state, glissile in the basal plane. Once it happens, dislocation motion become jerky with a series of sudden jumps between locking positions.

The proposed mechanisms of SSA and DSA were adopted in the present study and a phenomenological strain aging model was formulated. The modeling approach for strain aging suggested by McCormick is based on the internal variable called the aging time ta. This variable introduces a stress over-hardening corresponding to the pinning of dislocations due to strain aging phenomenon. Finite element simulations are then performed on the polycrystalline aggregates for different number of grains taking into consideration the effect of anisotropy of titanium.

Session 6:

Leverhulme Keynote: Developing Principle-Based Approaches to Quantify Mechanical Property Distributions of Titanium Alloys

Paul Dawson

Sibley School of Mechanical and Aerospace Engineering, Cornell University

Thermomechanical processing alters the microstructural state of engineering alloys and derivative mechanical properties. Historically, the processing routes that deliver superior properties have been determined by trial-and-error experimentation and codified as empiricisms between processing conditions and resulting properties. Quantitative links between the microstructural state and particular mechanical properties largely have been missing, but are needed to allow more direct prediction of the mechanical performance.

As a consequence, a long-standing goal of the materials science and mechanics communities is develop a principle-base approach to estimate an alloy's aggregate properties from knowledge of its microstructural state, the local properties of its constituent phases, and the type of loading being applied.

A combination of experimental and modeling capabilities have emerged over the past decade that provide practical tools to reach this goal. From the experimental side, these include electron and x-ray diffraction methods for quantifying microstructures of polyphase, polycrystalline alloys and for measuring mechanical behaviors at the scale of individual grains. From the simulation side, these include tools to instantiate virtual samples that adequately replicate measured microstructures and parallel finite element formulations capable of dealing with complex behaviors.

In this presentation, I discuss a multi-investigator project with the goal of coordinating these capabilities to estimate distributions of strength and ductility in a two-phase titanium alloy (Ti-6Al-4V) that has been processed to achieve distinctly different microstructures. Emphasis is placed on new analytical tools that have been developed to more effectively integrate together the data from the different sources and to reduce uncertainties in critical input parameters used in simulating the mechanical behavior. In closing, I'll discuss a few challenges that persist and offer interesting opportunities for continued research.

Determining Ti-6Al-4V material parameters using a discrete spherical harmonic analysis of lattice strain pole figures

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Determining reliable single crystal material parameters for complex polycrystalline materials is a significant challenge for the materials community. In this work, a novel methodology for determining those parameters is outlined and successfully applied to the titanium alloy, Ti-6Al-4V. Utilizing the results from lattice strain pole figure experiments conducted at the Cornell High Energy Synchrotron Source, an iterative approach is used to optimize the single crystal elastic and plastic material parameters used in crystal-scale finite simulations by comparing experimental and simulated lattice strain pole figures at discrete load steps during a uniaxial tensile test. Due to the large number of unique measurements taken during the experiments, comparisons were made using the discrete spherical harmonic modes of both the experimental and simulated lattice strain pole figures, allowing the complete pole figures to be used to determine the single crystal elastic and plastic material parameters.

Texture determination from ultrasonic wave speeds for HCP and cubic materials

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Crystallographic texture in polycrystalline HCP and cubic materials, often developed during thermomechanical deformations, has profound effects on properties at the macroscopic or component level. In this talk, a novel convolution theorem is presented, which couples the single crystal wave speed (the kernel function) with polycrystal orientation distribution function to give the resultant polycrystal wave speed function. The theorem expresses the three functions as harmonic expansions, thus enabling the calculation of any one of them when the other two are known. Hence, it presents a solution to the long-standing inverse problem for HCP and cubic materials of recovering crystallographic textures solely from polycrystal wave velocities. Preliminary finite element simulations established proof of principle for the technique, with the recovered textures from simulated wave speeds showing good agreements with the original ones.

Further experimental validations of the proposed theoretical model are then conducted, with a series of samples made of typical HCP and cubic materials, including commercially pure (CP) Ti, copper and Ti-6Al-4V, examined by carefully designed experimental setup for the measurement of angular variations of ultrasonic wave phase velocities. Wave speed surfaces of the polycrystal aggregates are then constructed from these measurements, and texture information extracted out from the speed surfaces using the theoretical model. Finally, this information is compared and calibrated against that measured independently by the well-established neutron diffraction technique on the same sample for a complete verification of the accuracy and sensitivity of the ultrasound technique.

Getting a grip on hexagons - surface acoustic wave interactions with hcp Ti

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We have been using spatially resolved acoustic spectroscopy (SRAS) for a number of years to image material microstructure. As well as giving beautiful texture information, full quantitative orientation information of materials with cubic microstructure - such as nickel superalloys, Inconel and stainless steel - have enabled other engineering and science research efforts in aerospace and power generation sectors.

Our focus has shifted to hexagonal close packed (hcp) materials, in particular titanium. Here, the physics lets us down, slightly; although electron backscatter or x-ray diffraction methods are capable of determining all three Euler angles, the second order elastic constants are such that the basal plane of hcp materials is isotropic. Acoustic waves, via the SAW velocity, are therefore only sensitive to the first two Euler angles. So it was initially a bit of a mystery when we found we were able to see evidence of sensitivity to the third Euler angle, under certain special conditions.

As well as discussing this interesting observation, we report progress on the optimisation of the scanning technique in terms of time/effort versus accuracy of information, and how this may be tuned to get more accuracy in the characteristics that interest us the most.

This is work in progress, and I hope the presented findings will spark interesting discussions with colleagues.

Session 7:

Combined AFM, SEM and crystal plasticity analysis of grain boundary sliding in titanium at room temperature

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Grain boundary sliding (GBS) is generally expected at relatively high temperature in metals. It was however reported at room temperature in several HCP polycrystals: zinc [1] a magnesium alloy [2] and commercial purity titanium [3-4]. Even though these materials differ in terms of preferred deformation modes, they share a significant plastic anisotropy of their crystals and thus grain-to-grain compatibility issues, as well as a limited number of independent slip or twinning systems to accommodate these compatibility stresses.

To investigate this phenomenon, incremental tensile tests were run on T40 and T60 commercial purity titanium specimens covered with microgrids, after a preliminary mapping of crystals orientations. After each strain increment, high resolution SEM images were captured to compute the strain field by DIC and to detect GBS, by a relative shift of these grids on both sides of a grain boundary. GBS was often related to a significant difference in the value and principal directions of the local strain between neighbouring grains. AFM images of the 500 x 500µm areas covered with microgrids were also repeatedly captured to measure the out-of-plane component of the sliding displacement jump across the grain boundaries, and to evaluate the contribution of GBS to the overall deformation using the method proposed by Langdon. Crystal plasticity finite element simulations based on real microstructure were run to analyze the stress field near the grain boundaries. The shear stress along the boundaries that slide was found to be 28 to 68% higher than the mean shear stress over those that do not slide. However, a high shear stress does not seem to be a sufficient condition to trigger GBS.

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Investigation of Bauschinger effect at small length scales using new micro mechanical test geometry

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It has been observed in many metallic systems that the flow behaviour in reverse loading is different from that when deforming the sample in the loading phase. This Bauschinger effect plays an important rôle in metals and alloys that are processed and used in service with the complex loading history. There is still a lack of studies at the micro-scale, showing the response of individual grains and grain boundaries to reverse loading, because of the difficulty in the available micro-mechanical test geometry.

The present work focuses on the development of new 'micro-seesaw' test geometry and its application to a Ti-6Al HCP alloy. This geometry not only facilitates changing loading direction in a test using only the compression force of a nano-indenter but also excludes the effect of asymmetry in the tension-compression flow behaviour from the Bauschiger effect in the final load displacement curve. The 'seesaw' samples consisted of a 4 x 4 x 5 µm stem with a cross-beam 6 µm deep and 50 µm on top and centred on the stem. These were milled in selected grains of an as cast and annealed Ti-6Al alloy using FIB. Bending and reversed bending in the stem is achieved by loading in the nano-indenter at either end of the cross-beam. This new sample geometry was initially validated using finite element modelling with isotropic elastic approximation. Flow behaviour was studied under different loading patterns using nano indentations which shows a clear evidence of Bauschinger effect at small length scales. Microstructure evolution after each load reversal was studied using High Resolution EBSD and cross correlation algorithm. Crystal plastic FEA simulations were performed and the strain patterns and lattice orientation variation evolution will be compared with experimental findings. Using the load displacement data, dislocation back stress was calculated by comparison with FEA results.

Slip-band grain-boundary interactions in titanium

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Grain boundaries are well known to strengthen materials and this is often due to the inhibition of slip propagation through grain boundaries. In titanium, dislocations may pile-up at grain boundaries leading to local stress concentrations and blocked slip bands, which were first predicted by Eshelby, Frank and Nabarro in 1951 [1]. The measurement of stress fields and pile-ups has now confirmed these predictions in 2D with high (angular) resolution electron backscatter diffraction (HR-EBSD) [2,3] and in 3D with differential aperture X-ray microscopy (DAXM) [4]. In this presentation I will outline these experimental approaches to characterise dislocation grain boundary interactions in commercially pure titanium and the potential to this new insight to mechanistically inform the impact of grain boundaries on strength in materials.

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Session 8:

Ultra Small Scale High Cycle Fatigue Testing by Micro-cantilevers

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A new method has been developed for testing high cycle and very high cycle fatigue properties of materials at the micro-scale based on micro-cantilevers. Focused ion beam was employed to cut micro-cantilevers on the surface of a selected grain in a bulk polycrystalline commercial pure titanium. The bulk specimen was pre-examined by EBSD, so the crystal orientation of all micro-cantilevers were known. The bulk sample with prepared micro-cantilevers was then attached to a high power ultrasonic generator, which can generate mechanical vibration at the frequency 20KHz. The bulk specimen moves with the ultrasonic generator, but the micro-cantilever lags somewhat behind. The resulting deflections generate cyclic stress in the micro-cantilevers.

The high vibration frequency means it can easily test into the high cycle and very high cycle regime. The design challenge is to generate enough stress to cause fatigue in these ultra-small specimens because the stress amplitude achieved in vibration is inverse to the cantilever size. Previous finite element model and experiments had shown that the classic micro-cantilever with uniform cross-section [1,2] can only generate stress a few MPa, even with the acceleration was tuned up to 106m/s^2. Instead, we designed a new 'hammer' shape micro-cantilever to increase the inertia. This design now generates sufficient stress and enables fatigue testing even in titanium, which is a challenging material due to the high strength to weight ratio (both high strength and low density require higher acceleration).

SN curves in the testing range from 10^5 to 10^8 cycles have been obtained for these micro- single crystal Ti samples using a step test protocol. The stress to failure decreases systematically as the number of cycles to failure increases. However, there is strong dependence on the crystal orientation with the fatigue strength at 107 cycles for test pieces cut along the <c> direction being approximately twice that of those cut in the <a> direction. The fatigue strength of micro-fatigue test is significantly lower than the static strength measured on micro-cantilevers of identical size using a nanoindenter. Due to the small specimen size, it is suggested that the results reflect the behavior of fatigue nucleation rather than propagation.

Progressive tests and interactive observations were conducted in these micro-fatigue cantilever tests, showing the evolution of high cycle fatigue crack initiation in the single slip condition.

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Strain rate sensitivity of Ti-6Al-2Sn-4Zr-xMo –micromechanical comparison between slip systems and alloys: Part 1 - Experiments

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Dwell fatigue has been a critical issue in aeroengine industry as it can significantly reduce fatigue lifetime. This is clearly a time sensitive deformation mode, where simple evaluation of the critical resolved shear stress for individual slip systems is insufficient and therefore rate sensitive material properties should be considered. The motivation of this study is to experimentally characterise fundamental rate sensitivity mechanisms within dwell sensitive Ti-6Al-2Sn-4Zr-2Mo and dwell insensitive Ti-6Al-2Sn-4Zr-6Mo. We employ micropillar compression technique to activate target slip systems of alpha phase and explore rate dependent deformation and load-relaxation of individual slip systems. We further use nanoindentation to examine rate sensitivity of averaged alpha and beta phases within comparative grain orientations from both alloys. We will present an overview of strain rate sensitivity in dual-phase Ti alloys determined by our novel experiments and discuss how this can link to the dwell fatigue problem. These experimental results support a wider campaign to understand dwell sensitivity and complement state-of-the-art simulation studies.

Strain rate sensitivity of Ti-6Al-2Sn-4Zr-xMo –micromechanical comparison between slip systems and alloys: Part 2 - Simulations

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Integrated micro-pillar compression testing and crystal plasticity finite element modelling of single crystal alpha Ti-6242 have been used to investigate the strain rate sensitivities of the independent basal and prismatic slip systems at 20°C. Fully representative micro-pillar compression test models have been utilised to establish the contributions to pillar force-displacement behaviour resulting from the micro-pillar response itself together with that from the sample substrate and the loading machine stiffness. Appropriate determination of the contributory stiffnesses is shown to be essential in ensuring correct extraction of micro-pillar material properties, particularly where rate sensitivity is important. The crystal modelling has been utilised to extract the basal and prismatic slip system strengths and strain rate sensitivities in Ti-6242 at 20°C. The strain rate sensitivities of the independent basal and prismatic slip systems have been shown to be significantly different, with the former showing a much stronger dependence than the latter. The crystal model provides excellent prediction of micro-pillar stress relaxation and rate-sensitive response. Slip development and localisation occurring within the experimental micro-pillar tests is also well reproduced by the crystal model.

Investigations into the effects of multiaxial stress states in cold dwell fatigue

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Investigations into the critical failure mode of near alpha titanium alloys known as cold dwell fatigue is now a well-established field [1]. Considerable research has been conducted in laboratories using small scale uniaxial load to failure tests. These laboratory tests have been hugely informative thus far and have elucidated many important findings which further understanding. However, these tests may not be truly representative of the in-service loading environment of key rotating components, which is likely to be highly multiaxial in nature. Thus the motivation of this paper is to investigate the combination of multiaxial loadings on the stress state likely undergone in-service.

A representative polycrystal of pseudo-randomly orientated grains is simulated in a directionally solidified, 3D crystal plasticity finite element model [2]. This model has the rogue pairing of hard and soft grains, crucial for cold dwell, deeply embedded in the centre of the model, so as to minimise boundary effects. The local effects of multiaxial loading on the stress redistribution at the hard-grain/soft-grain interface is examined as it is understood to be a key driver of facet nucleation.

The introduction of a second principal stress (s2) is shown to lead to a significant variation in the stress redistribution on the hard grain from, very high, for a compressive s2, to negligibly small load shedding for full equibiaxial stress loading (i.e. s1=s2). This result appears to coincide with the experimental findings indicated by Doquet et al [3]. Sample testing in torsion is also investigated through shear loading and the formation of highly localised permanent slip bands (PSB) is noted. This may be representative of the results demonstrated by Helbert [4] and Lefranc et al [5] who show that these PSBs lead to failure through the coalescences of voids to form cracks, which is more significant in dwell as opposed to regular low cycle fatigue.

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Session 9:

Keynote: Titanium in aerospace applications - characterization driving model development

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Titanium alloys have complex microstructures and exhibit unusual behaviors such as a tendency for creep and sensitivity to dwell fatigue conditions at ambient temperature. Several important factors that influence the sensitivity to dwell conditions include the presence of microtexture regions, slip planarity, dwell time, and testing temperature. A multi-disciplinary effort at OSU examined the behavior of the commercial alloy Ti-6424 as well as a single α phase Ti-wt%Al alloy subjected to creep, cyclic fatigue and dwell fatigue. Recognizing that time-dependent load-shedding from soft-to-hard microstructure features is key to this behavior, small-scale mechanical testing was performed to elucidate the anisotropy of plastic deformation. The mechanisms of creep and fatigue in these alloys has also relied upon application of electron optical and X-ray techniques to help inform microstructure-sensitive deformation modeling of the time-dependent creep and fatigue behavior of polycrystals. These results will be presented and possible directions for future investigation will also be discussed.

Atomic scale study of twinning in zirconium

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Plasticity in zirconium, as well as in many other hexagonal close-packed metals, is controlled by the glide of dislocations with <a> Burgers vectors. Those dislocations can however not explain any deformation along the <c> axis and at low temperatures twinning is activated to accommodate such a strain. In this work, we focus on the mechanisms controlling twin growth using atomistic simulations relying either on an empirical interatomic potential of the EAM type or on ab initio calculations. The four different twinning systems, which can be activated in zirconium depending on the temperature or the applied strain, are modeled. We first study the perfect twin boundaries showing the ability of the EAM potential to predict their structures and relative energies taking the ab initio calculations as a reference. We then focus on the disconnections, i.e. the twinning dislocations which are responsible of the twin growth. For a given twinning system, several disconnections of different Burgers vectors, different heights and different core structures exist. Considering all these disconnections, we calculate their formation and migration energies using the NEB method. The elastic interactions between the defects and their periodic images are computed using linear inhomogeneous anisotropic elasticity, allowing to extract disconnection core energies that are intrinsic properties of the disconnections, independent of the cell dimensions. We use this information to develop a kinetic model of twin growth and study the competition between the different growth modes under various stress states.

Discrete dislocation and crystal plasticity analyses of load shedding in polycrystalline titanium alloys

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Discrete dislocation plasticity (DDP) models were performed to analysis the fatigue response of Ti6242 and Ti6246 alloys in order to understand the mechanistic basis of dwell sensitivity. The material properties were obtained by calibration with experiment cyclic fatigue loading tests using crystal plasticity modelling. The two dimensional DDP model which incorporating thermal activation process using the obtained activation energy was first validated by comparing with nanoindetation tests on soft and hard grains in Ti6242 and Ti6246 alloys. Then stress-controlled loading which contains stress dwell period was imposed on polycrystal DDP model. It was found that the dislocations in Ti6242 are easier to pass obstacles and subsequently piling up around grain boundaries due to the low activation energy. Ti6246 is quicker to reach equilibrium states and there are less dislocation activation events during stress dwell loading. The simulations showed significant load shedding phenomenon for Ti6242 and no dwell sensitivity for Ti6246. The total strain after the loading was assessed and the corresponding dislocation density was presented. The rate sensitivity under displacement-controlled loading and plastic response under stress-controlled loading of both alloys was compared and well explained.

Crystal Size, Temperature, and Strain Rate Effects on the Competition between Slip and Twinning in Magnesium Microcrystals

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The wide use of Mg alloys as a structural material is still challenging due to their poor room temperature formability. Owing to its hexagonal closed packed (HCP) structure and low crystal symmetry, complex deformation mechanisms, including dislocation-slip and twinning are typically reported. As a result, the mechanical behavior of Mg metals displays strong anisotropy and strong orientation dependence. Here we report on the plastic response of a-axis oriented magnesium microcrystals having sizes 2-25 microns tested in situ SEM at various temperatures (25-500 oC) and strain rates(10^-4 – 10^-1 s^-1). An anomalous strain hardening response is observed and correlated to a size dependent transition from plasticity dominated by single twin propagation followed by massive dislocation slip, to twin-twin interactions dominated response, and finally the recovery of bulk like response. Results are compared to bulk Mg single crystals to further understand the hardening response of Magnesium.

Assessment of stress fields at deformation twin tips and the surrounding environments

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Stress fields close to twin tips and the associated local neighbourhoods of a hexagonal close-packed (HCP) polycrystal were studied in detail. For this purpose, a coarse grain textured Zircaloy-2 sample was firstly strained uniaxially in a macroscopic direction that favours tensile twin formation. The sample was then unloaded and residual elastic strains and lattice rotations measured using the high-resolution electron backscatter diffraction (HR-EBSD) technique. Measured elastic strain maps of various clusters of grains including parent and twin pairs were then analysed. Stress, dislocation density, and their associated concentrations close to twin tips, within twins, in the immediate neighbouring grain, at the intersection of two twins, and within parent grains were investigated. Measured grain shapes and orientations are also imported into the Abaqus finite element solver and are modeled by a crystal plasticity finite element code. The code is revised to account for local twin shear transfer and study its effects on the induced stress and rotation in the neighbouring grains. It is shown that the stress field at the twin tips varies as a function of local neighbourhood. High stress, lattice rotation, and dislocation density concentrations were generally observed close to twin tips both within twins and within the immediate neighbouring grains. It is shown dislocation density concentration is maximum at the intersection of two twins which can potentially provide susceptible site for crack nucleation.

Session 10:

Development of a Novel Experimental Rig for Testing of Iodine-induced Stress Corrosion Cracking of Zirconium Alloys

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The corrosive effects of iodine and other volatile fission products, coupled with stresses due to the thermal expansion of the fuel pellet during power ramps can lead to stress corrosion cracking (SCC) of zirconium alloy fuel cladding tubes. This phenomenon, also known as pellet cladding interaction (PCI), is currently guarded against with a combination of empirically driven innovations, such as the introduction of liner material in BWR fuel assemblies, and costly conservatism in power manoeuvring. Here we present the experimental results from a rig designed and built at the University of Manchester to simulate the conditions required for iodine-induced SCC. This work was performed as part of the PACE (Pellet Assisted Cladding Degradation) programme.

The experimental set-up developed at the University of Manchester, allows different aspects of the SCC process to be examined, allowing independent control of temperature, applied stress and iodine concentration. The rig has been used to test both commercial alloys and liner material, currently used in BWR fuel assemblies to reduce susceptibility to SCC. Iodine has been observed to cause grain boundary pitting on the cladding surface, with coalescence of these pits leading to microcracks and eventual cracking of the sample. The liner material shows increased resistance to SCC, which is commonly attributed to the increased ductility in the low-alloyed liner material. This work is complemented by ab initio density functional theory (DFT) calculations, also being performed at the University of Manchester, which are used to investigate how iodine interacts with zirconium grain boundaries and bulk zirconium.

Understanding the iron impurity redistribution during the oxidation of zirconium-niobium alloys cladding

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Zr alloys are one of the most common materials for nuclear fuel cladding due to their low neutron absorption cross section and resistance to aqueous corrosion. However, the operating lifetimes of commercial alloys in service are still limited by oxide growth at 300-400ºC and hydrogen pickup (HPU) that can lead to a loss of mechanical properties. Given that hydrogen solubility in Zr is extremely dependent on temperature, hydride precipitation during maintenance operations is expected as a result of HPU and this can lead to delayed hydride cracking as the zirconium hydrides are very brittle.

The existence of Fe impurity in Zr alloys is well-known, but little is understood about its role in controlling both corrosion and HPU. As the Fe-containing features may be very small and contain low concentration of Fe, a high-resolution characterisation technique is required. In this study, atom probe tomography (APT) has been carried out to examine the Fe distribution in the metal, oxide, and near the oxide/metal interface of Zr-Nb alloys. These samples have been corroded in an autoclave at 340-360ºC in different environments, including light and heavy water. This presentation will focus on the redistribution process over the course of oxidation as well as its correlation with both oxidation and HPU behaviour of the materials.

Investigation of hot salt stress corrosion in Ti-6Al-2Sn-4Zr-6Mo

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The temperature and stress dependence of hot salt stress corrosion cracking in Ti 6246 alloy has been investigated in order to elucidate the chemical mechanisms that occur. NaCl salt-deposited samples were loaded to a stress of 520MPa in a two-point bend fixture. A sample tested at 350°C did not fail even after 350 h, with very limited cracking observed. However, a sample tested at 450°C fractured after 60 hours of exposure. Cracking appeared to initiate beneath the salt particle in the presence of oxidation and crack growth occurred in a direction normal to the applied tensile stress. The fracture surface appeared brittle in appearance. The observed transgranular fracture may be due to hydrogen embrittlement; X-ray diffraction analysis confirms the formation of hydrides. SEM EDX analysis shows oxygen enhancement on the fracture surface, with the presence of chlorine and sodium. Aluminium also appears to be involved in the oxidation reactions. The role of intermediate corrosion products such as Na2TiO3, Al2O3 and TiCl2 will be discussed.

Poster 1: The effect of strain and interfaces on the relative stability of the tetragonal and monoclinic structures of Zirconium oxide – an atomistic modelling study

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Oxidation of the zirconium alloy cladding tubes that contain the nuclear fuel in light water reactors is one of the key degradation mechanisms, limiting the amount of fuel burned. The tetragonal-monoclinic phase transition has an important role in this process but the mechanistic understanding of how it affects the oxidation kinetics is still incomplete. We are exploiting the techniques of atomistic materials simulation to study important microstructural features that are thought to control this phase transition such as the effect of crystal anisotropy, boundaries and interfaces, and the affinity of defects to these boundaries. We have performed density functional theory (DFT) calculations to explore the effect of biaxial strain on the relative stability of the two phases and benchmark DFT calculations to determine the properties of representative grain boundaries in the oxide microstructure. We have then used the results of these calculations to test a range of empirical potentials for the Zr-O system and to establish their suitability for use in the computational modelling of the Zr-O system at microstructural length-scales.

Poster 2: Secondary alpha in Ti6246s

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Ti-6Al-2Sn-4Zr-6Mo-0.1Si (Ti6246) is widely used in high temperature applications, such as high pressure compressor discs in gas turbine engines, due to its high specific strength, good corrosion resistance and low density. Its Widmanstätten microstructure is thought to be key in the resulting properties of the alloy. Secondary alpha growing within the surrounding beta matrix is of particular interest, as it is thought that the presence of this particular phase gives Ti6246 its high strength and good fatigue properties. The growth of secondary alpha is predominantly controlled by stages of thermomechanical processing. A cooling rate of 10°C min-1 was applied from above the beta transus, with microstructures being fixed at a variety of temperatures via water quench. The resulting microstructures were then investigated using optical microscopy, scanning electron microscopy and electron backscattered diffraction, with a range of volume fractions of primary and secondary alpha being presented. Samples quenched at higher temperatures showed predominantly grain boundary alpha with little to no secondary alpha growth. Samples quenched at lower temperatures presented the final Widmansätten microstructure. It is thought that this is due to the amount of supersaturation that occurs during the processing, as presented by Semiatin et al. [1].

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Poster 3: High Speed Active Surface Temperature Imaging System, and its Integration with a Stereoscopic Digital Imaging Correlation Apparatus

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During dynamic deformation the formation of Adiabatic Shear Bands (ASB) can lead to catastrophic failure in demanding high performance environments. Pivotal to understand the evolution from formation to failure, is probing the underlying spatial distribution of temperature through the lifetime of an ASB. Previous attempts to spatially resolve the temperature distribution during ASB formation have relied upon infra-red imaging systems. However, due to the low intensity of infra-red light emitted, these systems cannot combine high spatial resolution with high temporal resolution, and are severely limited in their ability to interrogate the formation of ASB.

We propose an active temperature diagnostic based on thermoreflectance measurements, which is better suited to dynamic temperature measurement on the micron length scales appropriate to ASB formation. The thermoreflectance technique relies upon the known spectrally dependent change in reflectivity of gold as a function of temperature. By coating a sample with a calibrated gold film before dynamic deformation, the surface temperature of the sample during loading can be inferred by imaging the change in reflectivity. This poster describes the development of a multi-axis imaging system which combines the thermoreflectance measurement in one plane and a more traditional stereoscopic digital image correlation (DIC) in another. Thus allowing the combined thermomechanical measurement of ASB evolution for the first time.

Poster 4: High resolution strain mapping of hydrides in zirconium alloy

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During service, zirconium alloys (used as fuel cladding in nuclear reactors) pick up hydrogen as a result of aqueous corrosion. Once precipitated, hydride phases in the component lead to degradation in mechanical properties and enable the delayed hydride cracking failure mechanism. The high resolution digital image correlation (HR-DIC) technique allows in-plane strain fields to be quantified at sub-grain scale. By combining the technique with conventional SEM imaging and EBSD, the effect of hydrides on strain localisation can be examined. The ability to generate a strain map using the technique relies on being able to image a pattern with suitable feature size and spacing for the target resolution. A random, nanoscale (~40 nm) speckle pattern was applied to the surface of ZIRLO (Zr1.0Sn1.0Nb0.1Fe) using the styrene-vapour gold remodeling process. Tensile tests on dog-bone samples with varying hydrogen concentrations were performed to progressive macroscopic strain levels before imaging the resultant gold speckle pattern with high resolution FEG-SEM. Using DaVis image correlation software, strain maps were generated which give an insight into strain evolution surrounding hydrides in ZIRLO.

Poster 5: Texture evolution in Zr-Nb alloys

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α + β dual phase ZrNb alloys have higher strength and fracture toughness than single phase α alloys. These alloys develop different textures during thermomechanical processing, which can affect the hydride orientation in nuclear reactor components in service. Single phase α Zr alloys form a split basal ND texture, where (0002) basal poles are orientated ~30° from the normal toward the transverse direction. α + β dual phase ZrNb alloys develop a strong transverse basal (TD) texture, at 90° to normal and rolling directions. The origin of this texture component is not fully understood and cannot be predicted by current texture evolution models. The work presented here is an investigation of the texture evolution during high temperature rolling (800°C) of two model alloys: Zircaloy-4 +2.5wt.% Nb and Zircaloy-4 +7wt.% Nb, produced to contain different volume fractions of the constituent phases. The aim was to determine the relative roles of plastic strain partitioning between phases, the activity of the different deformation modes and phase transformation on the final texture. EBSD analysis of the hot rolled materials show that the strength of the transverse basal texture component increases with rolling reduction. Software reconstruction analysis of EBSD maps suggests that the TD texture evolves from deformed β grain regions, followed by preferential selection of the α phase under the Burgers relationship upon cooling. Results are interpreted with the aid of in-situ neutron diffraction measurements of internal phase strain evolution during compression.

Poster 6: Design, characterisation and properties of Ti-Fe-Mo alloys strengthened by ordered intermetallic precipitates

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Ordered intemetallic reinforcement has been demonstrated as a potent strategy for the development of strength alongside damage tolerance in structural applications. This has been extensively investigated and employed in A1 (fcc) nickel based superalloys. There has been recent interest in high strength maraging steels, where ferrite is strengthened by L21 Heusler or B2 ordered intermetallic precipitates. However, similar strengthening strategies have not been extensively explored and employed in titanium alloys.

Ti-Fe based eutectic alloys consisting of A2 Ti and B2 TiFe phases, have been shown to produce high strengths, > 2.5 GPa, and elongations to failures, ~15%. These impressive properties are thought to be a result of the fine microstructural lengthscale and a high lattice misfit between the phases. However, even greater microstructural refinement might be possible if the B2 reinforcing phase were formed through solid state precipitation, rather than eutectic solidification. Exploring this possibility, alloys within the Ti-Fe-Mo system have been investigated. Here, we report on the elevated temperature phase equilibria of this ternary system, which was used to design precipitate reinforced alloys. Microstructural characterisation found that within these alloys ultra-fine microstructures can be produced. Initial assessment of their mechanical properties is promising and this will be discussed in terms of the microstructure.

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Poster 7: Comparison between cold-work hardening and irradiation hardening in zirconium alloys: preliminary study on iodine-induced stress corrosion cracking

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To reduce the susceptibility of zirconium alloys to iodine-induced stress corrosion cracking (SCC) in pellet-cladding interaction (PCI), fuel assemblies in boiling water reactors (BWRs) are lined with low-alloyed zirconium. Currently, as-forged zirconium liner materials (Zr-0.25Sn) with varying iron concentrations (550 and 1000 ppm) have been supplied by Westinghouse. Samples from each processing step were archived from the normal direction for subsequent characterisation using optical and scanning electron microscopy, micro-hardness tester and x-ray diffraction. Argon-purged heat treatment of the processed materials was undertaken to establish the recrystallisation temperature. The recrystallised materials are then subjected to proton irradiation to a range of dose, from 0.25 to 1 dpa, in order to provide the required hardening effect. After irradiation, the samples underwent heat treatment to study the hardness evolution. TEM analysis was undertaken to verify the changes in the irradiated microstructure and determine its hardening effects. These results were then compared with the hardness evolution in cold- worked materials. The investigation consequently establishes the effect of annealing on the hardness of materials (both cold-worked and irradiated) in the SCC rig currently being developed at the University of Manchester. This investigation is crucial in determining the stability of damage structures during the proposed exposures in an iodine atmosphere at up to 300°C. Complementary modelling study is being undertaken at the University of Manchester to support this investigation.

Poster 8: Solid solution strengthening of hexagonal titanium alloys: restoring forces and stacking faults calculated from first principles

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Ti and Mg are immensely valuable for all industrial sectors in which reduction of weight and energy consumption have the major role. One of the most important method for improving mechanical properties of these metals is solution strengthening. Enormous ductility enhancement in Mg-Y alloys [1] and high strength with balanced malleability in Ti-O systems [2,3] both occurring in single phase alloys, motivate further investigation of solution effects.

Despite numerous studies devoted to plastic deformation of α-Ti alloys, selective impact of solutes on activity of individual deformation modes is still unexplained. Recently confirmed polymorphism of dislocation cores [4,5] indicate also that dislocation nucleation and motion may be related with the core reconstruction caused by the alloying elements.

We present here a systematic study of the GSFE calculated by the first principle approach for all active slip systems in a series of hexagonal Ti-X alloys. X = Al, Sn, V, Zr and O were selected to determine the effect of valence structure and lattice position of solutes on γ curves characteristic. Additionally, two concentration of alloying elements and their effective interaction range with the glade planes were analysed as well. Our study revealed a strong, nonlinear influence of X position on GSFE and migration of O atoms during the crystal slip. This new effect together with other results and available experimental data are discussed in terms of dislocations nucleation and possible dislocation core structure modification in studied alloys. Identified solution strengthening mechanisms [6] and the elaborated calculation methodology are highly valuable for designing new light and strong Ti-based alloys.

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Poster 9: In-situ HR-EBSD characterization of twin nucleation and growth by micro-pillar compression

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In-situ HR-EBSD characterization during micro pillar compression is used to better understand microstructure evolution during deformation of alpha titanium. Example will be given of a bi-crystal which was milled using focused ion beam in such a way that one of the grains has c axis near perpendicular to the pillar long axis (soft grain) and the other one has c axis near parallel to the pillar long axis (hard grain). Com-pression was performed inside SEM chamber with in-situ HR-EBSD scans at differ-ent levels of plastic strain. No visible slip band was observed at all strain levels, pos-sibly due to the particular geometry of the pillar. As deformation level increased, a compression twin nucleated at the edge of the pillar in the hard grain and propagat-ed towards the grain boundary. It is interesting to notice that this twin stopped prop-agating towards the grain boundary after a certain strain level, and started to thicken with subsequent straining. HR-EBSD stress mapping and serial sectioning were performed to study the possible reasons for this behavior. At higher strain level, the grain boundary started to buckle. Local stress concentration leads to the nucleation of a second twin from the grain boundary which then propagated across the whole grain. The local stress conditions before these nucleation and propagation events were well captured by HR-EBSD.

Poster 10: Advanced microtexture analysis of a Ti-10-2-3 product for better understanding of local variations in mechanical behaviour

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Ti 10-2-3 parts for high strength application are classically obtained by a complex transformation process including β forging and further α/β forging before aging. The expected microstructure is typically composed of primary α_p nodules embedded in a matrix of residual β_r and α_s platelets.

In this contribution, in depth microtexture analysis using EBSD and advanced data processing with Merengue 2 software have been performed at different scales ranging from millimeter down to sub-micron. Our goal was to reconstruct the prior high temperature α_p/β microtexture and to quantify remaining traces of orientation relationship between α_p and β phases as well as β to α_s variant selection.

New insights of the inherited microstructure are revealed and may explain why local configurations supposed to be similar from microstructural observations could exhibit different mechanical behavior.

Poster 11: Microstructural interactions of deformation twins in CP titanium and Ti–6Al–4V after room temperature ballistic impact testing

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Since the 1950s titanium has been used extensively and successfully in the aerospace industry. Nevertheless, there is a noticeable lack of literature and understanding regarding high strain rate deformation, deformation twinning modes and the microscale mechanics during the deformation of titanium alloys in ambient conditions. In this work, commercial purity titanium and Ti–6Al–4V have been tested at room temperature, at a strain rate of 103 s−1 using ballistic impact testing. The Ti–6Al–4V was heat-treated under various conditions to test a range of different microstructures. The results demonstrated that there was substantial deformation twinning at this strain rate. Twin interactions with various grain boundaries were also evident in both materials. These interactions were often found to span many neighbouring alpha-phase grains. Hence, the effective structural unit size with which the deformation twins interact could be significantly larger than the alpha-phase grain size in both materials. The implications of the interactions with grain boundaries and the presence of rare deformation twinning modes will be discussed with relation to aerospace titanium alloys for gas turbine engines, in particular Ti–6Al–4V alloys for fan blade and compressor blade applications that are subjected to high strain rate deformation in service.

Poster 12: The Origin and Enhancement of {0001}⟨11$\bar{2}$0⟩ Texture during Heat Treatment of Rolled AZ31B Magnesium Alloys

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The microstructure and texture evolution during annealing of rolled Mg alloy, AZ31B, at temperatures ranging from 260 to 450 °C is characterized and a grain growth exponent n = 5 is revealed, indicating inhibition of grain growth. Indeed, some Zener pinning was observed and the possibility of abnormal grain growth was explored. More interestingly, it is observed that the initially strong basal texture, which develops during rolling of the Mg alloy, greatly increases in strength during annealing at 400 and 450 °C. Indeed, a specific texture component {0001}⟨11$\bar{2}$0⟩ emerges as dominant. Electron backscatter diffraction is used as input into a Monte Carlo Potts grain growth model. The model reveals that the {0001}⟨11$\bar{2}$0⟩ texture component, is the result of a grain size advantage that results from recrystallization; where grains closer to the {0001}⟨11$\bar{2}$0⟩ orientation are larger on average than competing {0001}⟨10$\bar{1}$0⟩ oriented grains. As a consequence, there is no need to invoke anisotropy in grain boundary energy or mobility, which were originally hypothesized to be responsible for the effect. The observed textural evolution shows a surprising similarity to that of hexagonally close packed Zr alloys despite underlying differences in the deformation mechanisms between the two alloy systems, though crystal plasticity modeling has revealed that prismatic slip is far more active during deformation than original supposed.

Poster 13: Transmission electron microscope and molecular dynamics investigation of dislocation-twin interactions in magnesium

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Conventional transmission electron microscope (CTEM) diffraction contrast imaging of dislocations is used to test the hypothesis that the transmission of two basal <a> type dislocations through a twin boundary will yield a <c+a> dislocation. This hypothesis was originally proposed by Price (1961) and expanded upon by Yoo & Wei (1966). The first evidence for it was found in experiments performed on Zn crystals by Tomsett and Bevis (1969). Since that time, none of the focused studies (either experimental or modeling) have yielded support for the hypothesis. In the present study of Mg alloy AZ31, unequivocal experimental evidence for this so-called “transmutation” reaction is provided. Further, we show the same sort of reaction in a molecular dynamics simulation of pure Mg, by applying distinct initial and boundary conditions from previous modelers. We hypothesize that there are strong implications of these reactions on subsequent hardening behavior via two means. First, the reaction products could yield hardening via forest hardening of the twin material a la Basinski et al (1997). Second, debris left in the twin boundary after sweeping the surrounding dislocated matrix could render a decrease in the mobility of the twin boundary itself.

Poster 14: In-situ micropillar compression and HR-EBSD studies of morphological effects in a dual-phase Ti alloy

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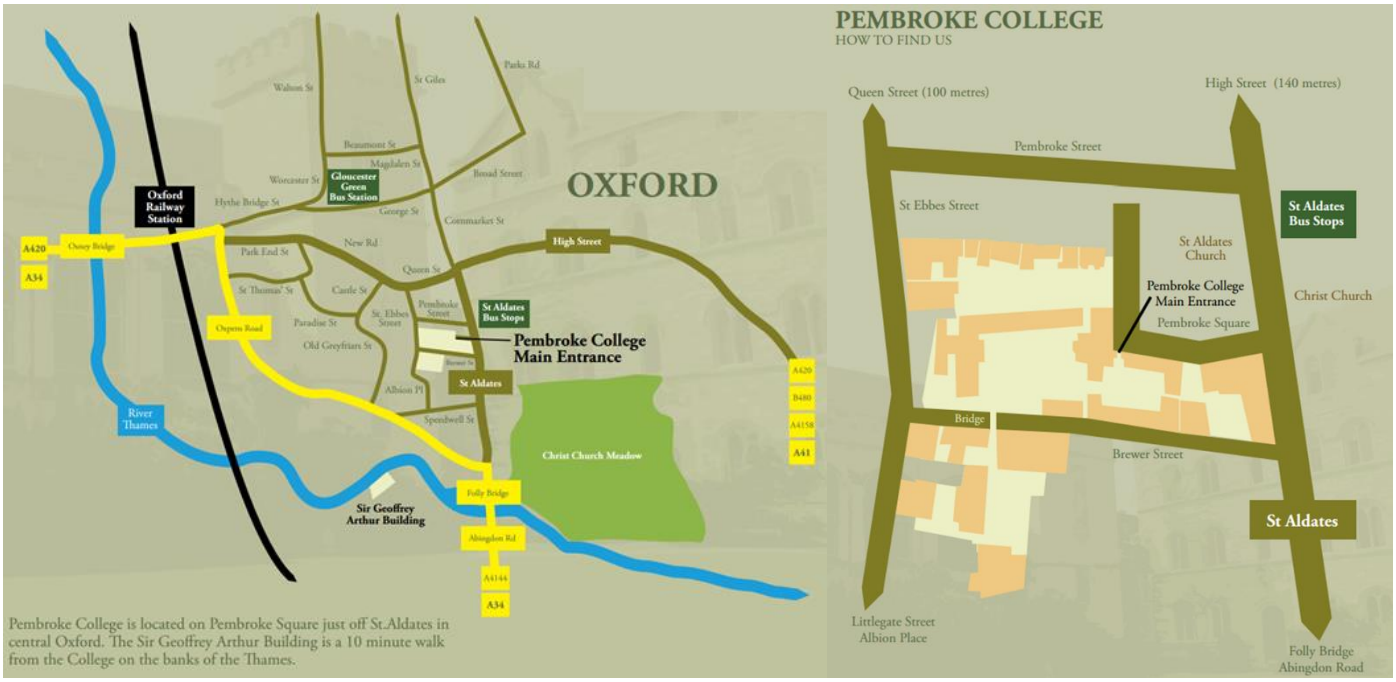
Understanding deformation mechanisms of α/β Ti alloy is rather challenging as complexities arise due to highly localised deformation and elastic/plastic anisotropy inherent to α-Ti, as well as interaction between both phases [1]. Small-scale experiments on a localised, confined area are therefore important and required to investigate the fundamental mechanisms on the level of the individual constituents. In the present study, we use combined micropillar compression and HR-EBSD techniques to examine how different morphologies of α/β colonies affect local deformation behaviour in Ti-6242 (Ti-6Al-2Sn-4Zr-2Mo). Cross section samples were made and pillars fabricated for in-situ HR-EBSD studies; mapping was undertaken before, during and after consecutive compressions. The in-situ compression tests were performed using a displacement-controlled Alemnis nanoindentation platform set inside a SEM and EBSD patterns were captured using a TSL-EDAX EBSD system. Analysis of each map was performed offline using CrossCourt from BLG productions (Bristol, UK). Field plots of elastic strain and GND density are compared to understand local deformation mechanisms and the influence of the β structure on pillar deformation mechanisms. The presence of beta and α/β interfaces within the pillars result in different load-displacement responses, as well as patterning of stress and GND density as measured with HR-EBSD.

Maps:



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More information on the programme of work can be found on our website: http://www.imperial.ac.uk/hexmat

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