Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes
Room Town & Country A - Session H3-1-TuA

Characterization of Coatings and Small Volumes in Extreme and Cyclic Conditions I

Moderators: Prof. Dr. Peter Hosemann, University of California, Berkeley, USA, **Prof. Dr. Barbara Putz**, Montanuniversität Leoben, Austria

1:40pm **H3-1-TuA-1 Local Deformation Mechanisms under Ambient and Non-Ambient Conditions Tested via Advanced Nanoindentation***, **Verena Maier-Kiener**,* Montanuniversität Leoben, Leoben, Austria **INVITED**

Nanoindentation over the recent years established itself as a versatile tool for probing local mechanical properties beyond hardness and modulus. By adapting and improving standard nanoindentation testing methods, reliable protocols capable of probing thermally activated deformation processes can be accomplished. Abrupt strain-rate changes within individual indentations allow determining the strain-rate dependency of hardness at various indentation depths. For probing lower strain-rates and excluding thermal drift influences, long-term creep experiments can be performed by using the dynamic contact stiffness for determining the true contact area. From both procedures hardness and strain-rate, and consequently quantities such as strain-rate sensitivity, activation volume and activation energy can be reliably deducted within individual indentation tests, permitting information on the locally operating thermally activated deformation mechanism.

This presentation will first discuss various testing protocols including possible challenges and improvements, with particular emphasis towards testing at higher temperatures and under hydrogen atmosphere. Second, it will showcase different examples highlighting the direct influence exerted by microstructure, phase transformations and environmental conditions on the underlying deformation behavior in pure and highly alloyed material systems.

2:20pm **H3-1-TuA-3 Extracting High-Temperature Stress-Strain Curves and Assessing Transformation Pressures: The Spherical Indentation of Silicon***, **Gerald Schaffar**,* Montanuniversität Leoben, Austria*; D. Tscharnuter,* KAI Kompetenzzentrum Automobil- und Industrieelektronik GmbH, Austria*; V. Maier-Kiener,* Montanuniversität Leoben, Austria

In the past nanoindentation with spherical tips has already been extensively employed to characterize the mechanical properties of silicon. This work aims to combine advances in spherical indentation testing [1], [2] with the idea of using continuous stiffness measurement (CSM) during the unloading of silicon [3]. Combining both advanced methods allows directly the calculation of the compression flow behavior, including the acquirement of the pressure-induced phase transformations in silicon. Therefore, the use of CSM during the indentation unloading process permits the measurement of the phase transformations during unloading. These transformations can, at room temperature, be seen as "elbows", "pop-ins" or mixes thereof in the load-displacement curve [4]. In the current work, this combined approach is applied to both room-temperature and high-temperature indentations up to 950°C. Subsequently, confocal Raman spectroscopy is used to identify the phase transformations occurring at lower testing temperatures. Further, confocal laser scanning microscopy is used to check the remaining indentations for irregularities, such as pronounced pile-up behavior. Indentation tests using self-similar Berkovich tips, performed beforehand across this entire temperature range, revealed that the plastic deformation behavior is controlled by phase transformations up to ~ 400 °C. At even higher temperatures dislocation plasticity dominates the plastic deformation underneath the indenter. This transition between phase transformations and dislocations as the main mechanism of plasticity when indenting silicon is in good agreement with previous observations from high-temperature indentation testing [5].

[1]S. Pathak and S. R. Kalidindi, *Mater. Sci. Eng. R Reports*, vol. 91, pp. 1–36, 2015.

[2]A. Leitner, V. Maier-Kiener, and D. Kiener, *Mater. Des.*, vol. 146, pp. 69–80, 2018.

[3]G. J. K. Schaffar, J. Kappacher, D. Tscharnuter, and V. Maier-Kiener, *Jom*, vol. 74, no. 6, pp. 2220–2230, Jun. 2022.

[4]V. Domnich, Y. Gogotsi, and S. Dub, *Appl. Phys. Lett.*, vol. 76, no. 16, pp. 2214–2216, 2000.

[5]V. Domnich, Y. Aratyn, W. M. Kriven, and Y. Gogotsi, *Rev. Adv. Mater. Sci.*, vol. 17, no. 1–2, pp. 33–41, 2008.

2:40pm **H3-1-TuA-4 Micro-Impact Tests of Novel Thermal Barrier Coating Systems and >1000C Nanoindentation on Ni-Base Superalloy***, **Ben Beake**,* Micro Materials Ltd, UK*; C. Chalk,* Cranfield University, UK*; S. Goodes, A. Harris,* Micro Materials Ltd, UK*; L. Isern, J. Nicholls,* Cranfield University, UK

Thermal barrier coatings (TBCs) with improved erosion resistance are needed to increase the efficiency of gas turbines in aero-engines by enabling them to operate at higher temperatures. Rare earth zirconate (REZ) TBCs have potential as low-conductivity TBCs permitting higher temperature operation by more effectively thermally shielding the Ni-base superalloy turbine blade material. Although they have much lower thermal conductivity than current yttria-stabilised zirconia (YSZ) TBCs they have lower toughness making them susceptible to erosion. Nanomechanical tests are used to streamline the development of advanced multilayered TBC systems that can combine optimum thermal and mechanical properties.

The micro-scale impact test capability in the NanoTest has been modified to closely simulate real erosion conditions. The resistance of single-layered YSZ coatings and bilayer TBC coatings comprising a top layer of the REZ $Gd_2Zr_2O_7$ and YSZ sub-layer of similar total thickness to repetitive high strain rate contacts has been studied in detail. YSZ showed almost identical impact behaviour in 75 and 90 degree impact tests but in contrast the REZ coating was more resistant in angled impact than normal impact. The performance of the coatings in the novel micro-impact tests correlated with that in conventional erosion tests.

The nanomechanical behaviour of the Ni-base superalloy substrate (Nimonic 75) has been assessed at temperatures above 1000 °C for the first time. The critical role of the nanoindentation load-time profile to mitigate the enhanced creep at these temperatures and obtain more accurate measurements of hardness and elastic modulus is investigated.

Funding support from Innovate UK is acknowledged ("High temperature tools for designing sustainable erosion resistant coatings" Project#10020751).

3:00pm **H3-1-TuA-5 Influence of Si on the Mechanical Properties and High-temperature Fracture Toughness of Cr-Si-$B_{2\pm z}$ Coatings***, L. Zauner, **Rainer Hahn**,* CDL-SEC at TU Wien, Austria*; O. Hunold, J. Ramm,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein*; S. Kolozsvari, P. Polcik,* Plansee Composite Materials GmbH, Germany*; H. Riedl,* CDL-SEC at TU Wien, Austria

Si-alloyed diboride systems show outstanding oxidation resistance compared to their unalloyed binary or ternary counterparts. This allows operating temperatures up to 1200 °C, further extending the possible applications of such systems, such as protective coatings in turbines. In addition to the necessary oxidation resistance, adequate mechanical protection is also of great interest. This work investigates the impact of Si segregates on the structural and mechanical properties of Cr-Si-$B_{2\pm z}$ thin films from ambient to elevated temperatures. Overstoichiometric, AlB_2-structured Cr-Si-$B_{2\pm z}$ thin films with Si-content up to 15 at.% were synthesized on Ti-6Al-4V substrate by magnetron sputtering using a substrate bias of -120 V.

Enhanced surface diffusion of film constituents promotes the growth of mechanically superior, (001)-oriented coatings with a hardness of H~30 GPa up to a Si content of 3 at.%. Higher Si concentrations result in a significant hardness loss to H~20 GPa related to a bias-independent solubility limit in the CrB_2 structure, causing the formation of mechanically weak Si grain-boundary segregations. The as-deposited hardness of all Cr-Si-$B_{2\pm z}$ compositions is maintained after annealing up to 800 °C despite the initiation of material recovery. In addition, minimum interdiffusion and excellent adhesion are observed on the Ti-6Al-4V-alloy.

In line with the room temperature hardness, an increasing Si content is accompanied by a decreasing fracture toughness, reducing from K_{IC}~2.9 ($Cr_{0.28}B_{0.72}$) to ~1.7 MPa√m ($Cr_{0.24}Si_{0.10}B_{0.66}$), respectively. High-temperature cantilever bending up to 800 °C revealed a brittle-to-ductile-like transition for $Cr_{0.28}B_{0.72}$, resulting in a fracture toughness increase to K_{IC}~3.3 MPa√m. Similar behavior is observed for Si-alloyed coatings up to 400 °C, whereas beyond this temperature, Si-segregates enable high-temperature plasticity and, thus, a significantly increased damage tolerance.

4:00pm **H3-1-TuA-8 Nanoindentation Measurements at Combined High Sustained Strain Rates and Elevated Temperatures***, Benoit Merle,*
University of Kassel, Germany **INVITED**

Constant strain rate nanoindentation is a versatile method well suited for measuring the microscopic mechanical properties of materials within a wide range of temperatures. Recent developments have focused on increasing the permissible strain rates beside the typical ~0.1/s threshold present in most commercial systems. The current limitation primarily derives from the plasticity error of the continuous stiffness measurements (CSM) and has recently been overcome with a custom evaluation method avoiding the need for a measurement of the contact stiffness. With this improvement, the experimental upper strain rate limit is only limited by the hardware time constants of the system.

Based on a study of an intermetallic alloy, the presentation will demonstrate that nanoindentation is a potent technique for systematic investigations of changes in mechanical properties as a function of the testing temperature and deformation rate.

Refs:

1. B. Merle, V. Maier-Kiener, G. M. Pharr. Influence of modulus-to-hardness ratio and harmonic parameters on continuous stiffness measurement during nanoindentation (2017) Acta Materialia, 134, pp.167-176.
2. B. Merle, W.H. Higgins, G.M. Pharr. Critical issues in conducting constant strain rate nanoindentation tests at higher strain rates (2019) Journal of Materials Research, 34(20), pp. 3495-3503.
3. B. Merle, W.H. Higgins, G.M. Pharr. Extending the range of constant strain rate nanoindentation testing (2020) Journal of Materials Research, 35(4), pp. 343-352.

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