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Variable temperature ultra-nanoindentation system: Elevated and cryogenic temperature measurements

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[1] J. M. Wheeler, D. E. J. Armstrong, W. Heinz, R. Schwaiger, Current Opinion in Solid State and Materials Science, In Press, DOI:10.1016/j.cossms.2015.02.002 [2] J. Nohava, N. X. Randall and N. Conte, Novel ultra nanoindentation method with extremely low thermal drift: principle and experimental results, J. Mater. Res. , Vol. 24, No. 3 (March 2009) 873-882 [3] Xu Huang, J. Nohava, Bin Zhang, A. G. Ramirez, International Journal of Smart and Nano Materials, 2:1, 39-49, 2011

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VARIABLE TEMPERATURE ULTRA-NANOINDENTATION SYSTEM: ELEVATED AND CRYOGENIC TEMPERATURE MEASUREMENTS

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One of the primary motivations for development of instrumented indentation was to measure the mechanical properties of thin films. Characterization of thin film mechanical properties as a function of temperature is of immense industrial and scientific interest. The major bottlenecks in variable temperature measurements have been thermal drift, signal stability (noise) and oxidation of/condensation on the surface. Thermal drift is a measurement artifact that arises due to thermal expansion/contraction of indenter tip and loading column. This gets superimposed on the mechanical behavior data precluding accurate extraction of mechanical properties of the sample at elevated/cryogenic temperatures [1]. Reliable load-displacement measurements up to 700 °C have additional technical requirements including a differential displacement measurement system, independent tip and sample heating and active thermal management of the system as well as answers to scientific questions like the temperature in the contact area or the tip wear. It is then mandatory to have a suitable device for exploring such scientific limits to technical goals and understanding nanoscale high temperature deformation and fracture. Such a device must be able to maintain the thermal drift below 0.1 nm/sec, and should be implemented in a robust system which minimizes noise (electrical, vibrational, thermal, etc..), with a continuous correction based on active top- referencing.

A novel vacuum nanoindentation system that can perform reliable load-displacement measurements over a wide temperature range (-150 to 700 °C) will be presented. This system is based on the Ultra Nanoindentation Tester (UNHT [2], [3]) that utilizes an active surface referencing technique comprising of two independent axes, one for surface referencing and another for indentation. This results in negligible compliance of the system and very low thermal drift rates. Vacuum is essential to prevent sample/tip oxidation at elevated temperatures and condensation at cryogenic temperatures. The sample, indenter and reference tip are heated separately and the surface temperatures matched establishing an Infrared bath to obtain drift rates as low as 5nm/min at 700 °C. Instrumentation development, system characterization, experimental protocol, operational refinements and thermal drift characteristics at various temperatures will be presented. The system was validated by performing extensive testing on calibration materials like fused silica and single crystal aluminum. Case studies on elevated temperature properties of P91 and 316L steels and low temperature properties of nanocrystalline nickel and copper will be presented. Finally, the current status and future roadmap for variable temperature nanoindentation testing will be discussed.

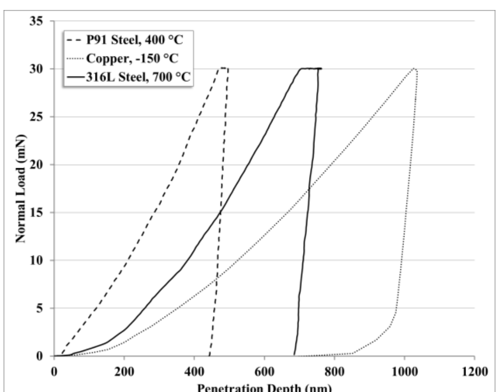

Figure 1 – Cryo to High Temperature tests

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