HIGH TEMPERATURE NANOINDENTATION UP TO 800°C FOR CHARACTERIZING HIGH TEMPERATURE PROPERTIES OF MATERIALS

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One of the primary motivations for development of instrumented indentation was to measure the mechanical properties of thin films. Characterization of thin film mechanical properties as a function of temperature is of immense industrial and scientific interest. The major bottlenecks in variable temperature measurements have been thermal drift, signal stability (noise) and oxidation of/condensation on the surfaces. Thermal drift is a measurement artifact that arises due to thermal expansion/contraction of indenter tip and loading column. This gets superimposed on the mechanical behavior data precluding accurate extraction of mechanical properties of the sample at elevated temperatures. Vacuum is essential to prevent sample/tip oxidation at elevated temperatures.

In this poster, the design and development of a novel nanoindentation system that can perform reliable load-displacement measurements over a wide temperature ranges (from -150 to 800 °C) will be presented emphasizing the procedures and techniques for carrying out accurate nanomechanical measurements. This system is based on the Ultra Nanoindentation Tester (UNHT) that utilizes an active surface referencing technique comprising of two independent axes, one for surface referencing and another for indentation. The differential depth measurement technology results in negligible compliance of the system and very low thermal drift rates at high temperatures. The sample, indenter and reference tip are heated/cooled separately and the surface temperatures matched to obtain drift rates as low as 1nm/min at 800 °C without correction. Instrumentation development, system characterization, experimental protocol, operational refinements and thermal drift characteristics over the temperature range will be presented, together with a range of results on different materials.

Fig. 1: Load-depth curves for pure molybdenum tested at 23°C and 810°C in vacuum with maximum load 100 mN, loading rate 200 mN/min. and hold time at maximum load of 300 s. The average drift rate over the entire hold time at 810°C was 1.2 nm/min from raw, uncorrected data.