IN SITU MICROMECHANICAL TESTING INSIDE THE SCANNING ELECTRON MICROSCOPE AT SUBAMBIENT TEMPERATURES

Jakob Schwiedrzik, Empa Swiss Federal Laboratories for Materials Science and Technology
jakob.schwiedrzik@empa.ch
Juri Wehrs, Empa Swiss Federal Laboratories for Materials Science and Technology
Michael Deckarm, Universität des Saarlandes
Rainer Birringer, Universität des Saarlandes
Johann Michler, Empa Swiss Federal Laboratories for Materials Science and Technology

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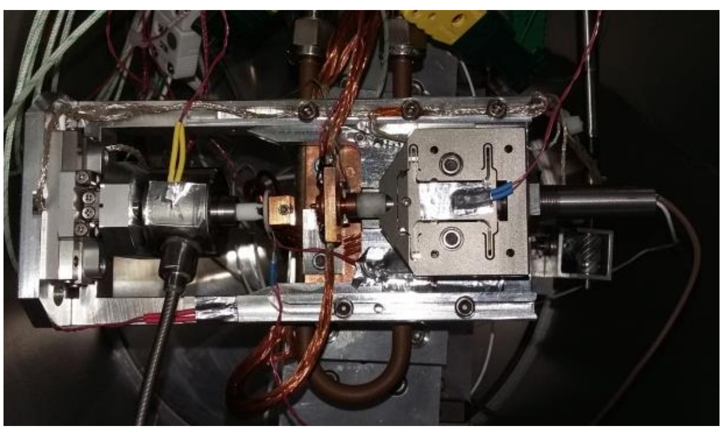
In material science, the measurement of mechanical material properties as a function of temperature is of great interest, as it allows determining the activation parameters of the underlying deformation mechanism. In the case of nanostructured materials or MEMS devices, it is interesting to probe local properties by means of micromechanical experiments. However, in the case of nanograined metals, testing at elevated temperatures is not possible due to heat induced grain growth and thus changes in the microstructure during testing.

We developed a device that allows performing micromechanical tests at low temperatures down to -150°C inside a scanning electron microscope. A cold finger connected to the sample and tip holders by copper braids is cooled by circulating nitrogen. Independent thermal management of the indenter and the sample allows minimizing temperature differences and thereby drift. Local cooling, thermal isolation of the cold regions, and a closed loop frame temperature control reduce frame drift, noise, and the need for a temperature-dependent calibration. A symmetric design of the cooling bodies was chosen in order to minimize bending moments on the indenter and the sample, which increases the accuracy of the measurements. The high vacuum environment minimizes condensation of water vapor and hydrocarbons on the indenter and sample. Positioning as well as *in situ* observation is made possible by the use inside a scanning electron microscope. For validation of the system, indentations in Cu were performed down to -150°C.

As a model system, micromechanical tests were conducted in nanocrystalline PdAu. Micropillars were prepared by focused ion beam (FIB) milling in a sample produced by inert gas condensation. Strain rate jump micropillar compression experiments were performed at strain rates between 10^{-4} to $10^{-2}s^{-1}$ between 25°C and -55°C. Activation energy and volume were determined from the observed changes in apparent yield stress as a function of temperature and strain rate. The results compare favorably with trends observed during an earlier study using the same technique at elevated temperatures up to 125°C.



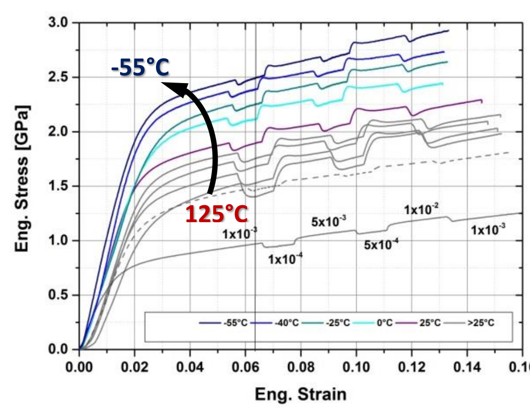


Figure 1: Left: Prototype of the cryoindenter. Right: Stress-strain curves of nanocrystalline PdAu micropillars