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

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Cryoindenter prototype development

A device was developed that allows performing micromechanical tests at subambient temperatures down to 125 K 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 the 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 125 K and hardness was mapped as a function of temperature.

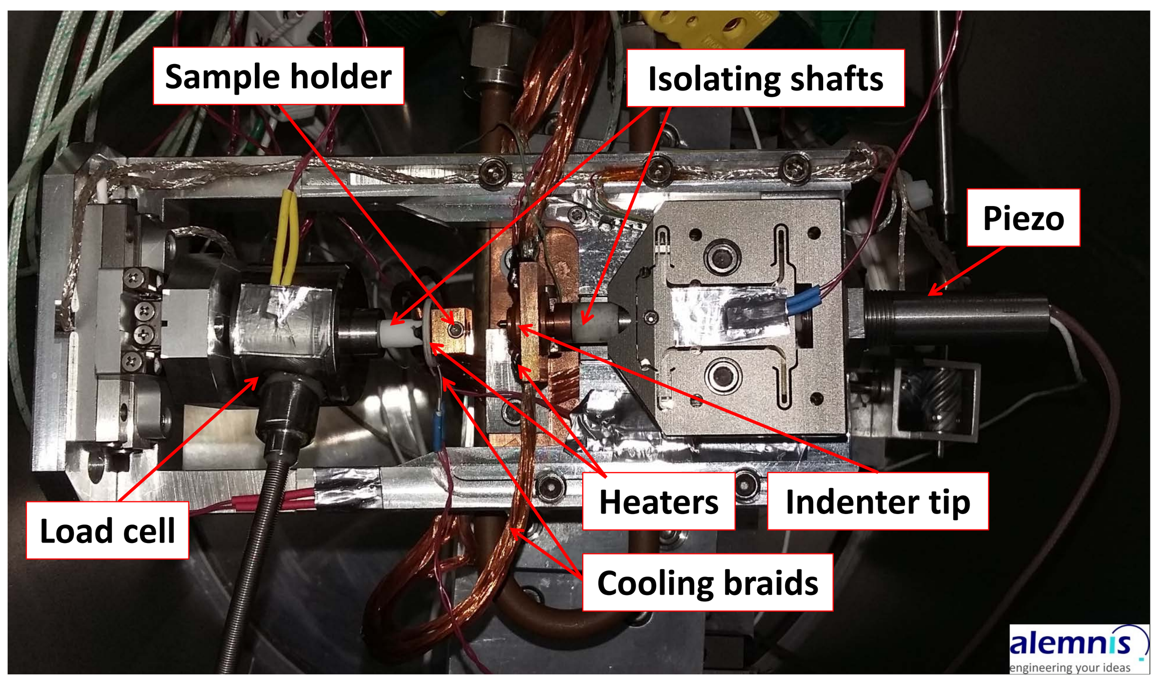

Fig. 1: Prototype of the *in situ* cryoindenter

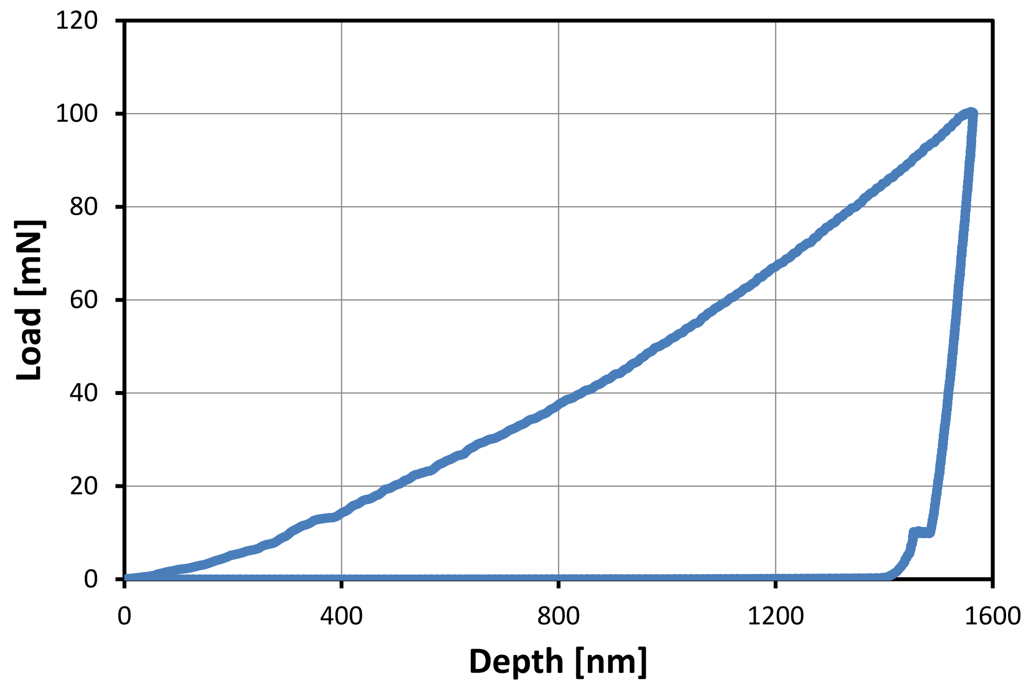

Fig. 2: Exemplary indentation in OFHC Cu at 200K

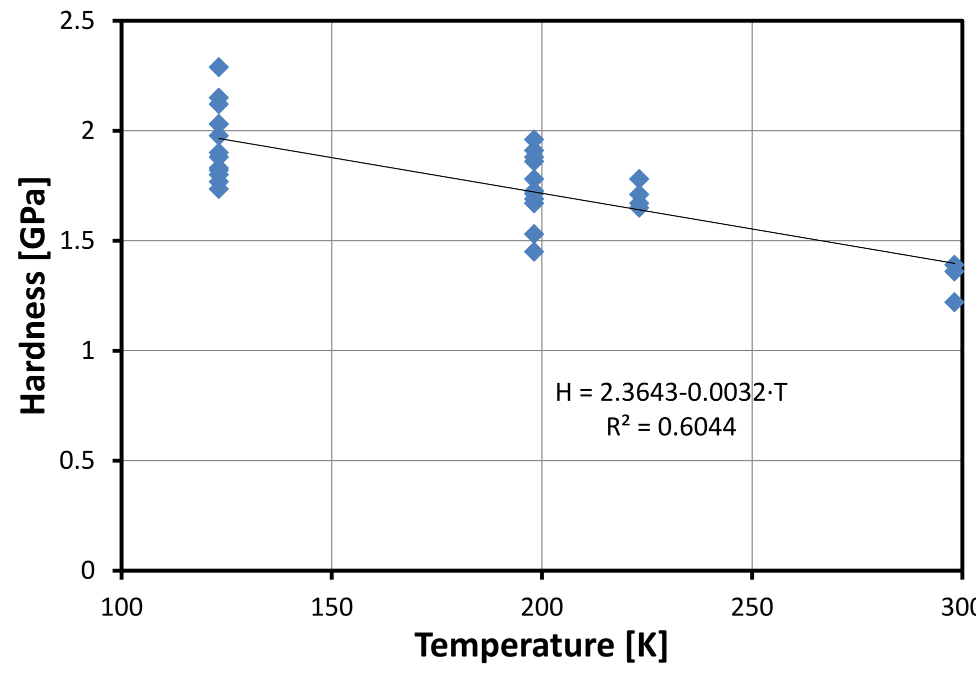

Fig. 3: Hardness vs. temperature for OFHC Cu

Experimental study: Activation parameters of nanocrystalline PdAu

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. Nanocrystalline metals yield at much higher stress levels due to the small grain size. Different deformation mechanisms such as grain boundary sliding, grain rotation, stress induced grain growth, etc., are active and the plastic deformation is strongly rate-dependent.

In the case of nanocrystalline metals, testing at elevated temperatures is not possible due to heat induced grain growth and thus changes in the microstructure during testing. Therefore, it is an attractive alternative to perform experiments at subambient temperatures.

Micromechanical tests were conducted in nanocrystalline PdAu produced by inert gas condensation with a grain size of approx. 12 nm. Micropillars were prepared by Ga focused ion beam (FIB) milling using a 3-step protocol.

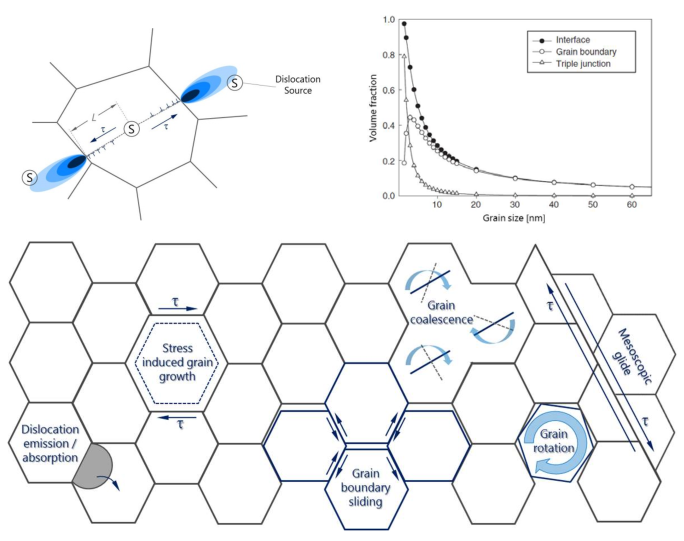

Fig. 4: Strengthening and deformation mechanisms in nanocrystalline metals

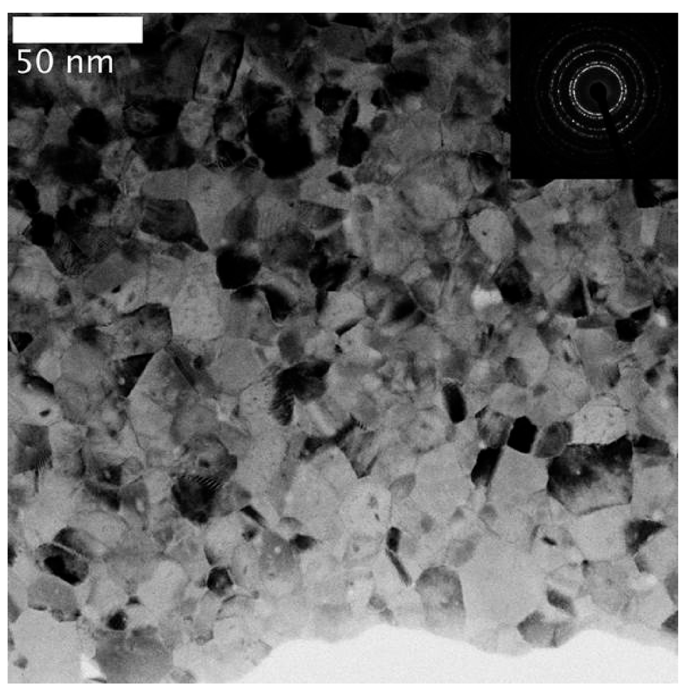

Fig. 5: TEM image of nanocrystalline PdAu

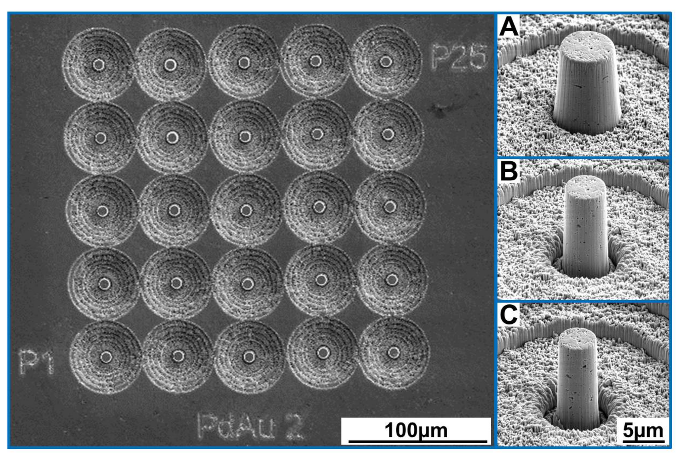

Fig. 6: Preparation of micropillars in nanocrystalline PdAu using Ga FIB

Strain rate jump micropillar compression experiments were performed at strain rates between 10^{-4} to $10^{-2}s^{-1}$ and temperatures between -55°C and 25°C (218K and 298K). Strain rate was changed instantaneously during the loading protocol and variations in yield stress were monitored.

The strain rate sensitivity *m* and yield stress $_y$ were determined for each temperature. Based on this information, the activation volume was computed:

$$_{,} = \frac{\sqrt{3}}{\ }$$

It was found that the activation volume remains approx. constant at 10 - 11 b^3 in the tested sub-ambient temperature range. In line with trends observed during an earlier study of strain rate jump micropillar compression experiments at elevated temperatures up to 125°C, the data suggests not a single dominant plasticity mechanism but rather a sophisticated interplay of coexisting mechanisms[1].

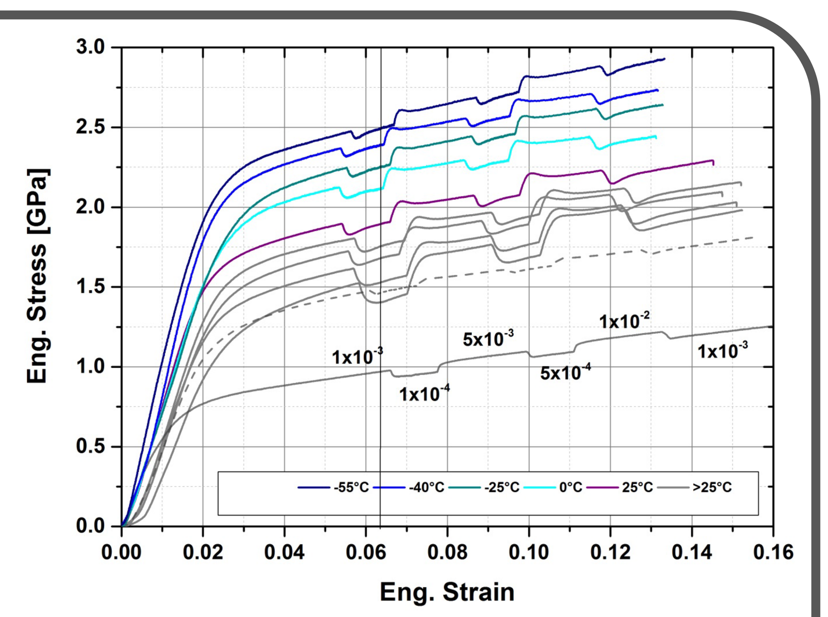

Fig. 7: Stress-strain curves of strain rate jump micropillar compression experiments at variable temperature

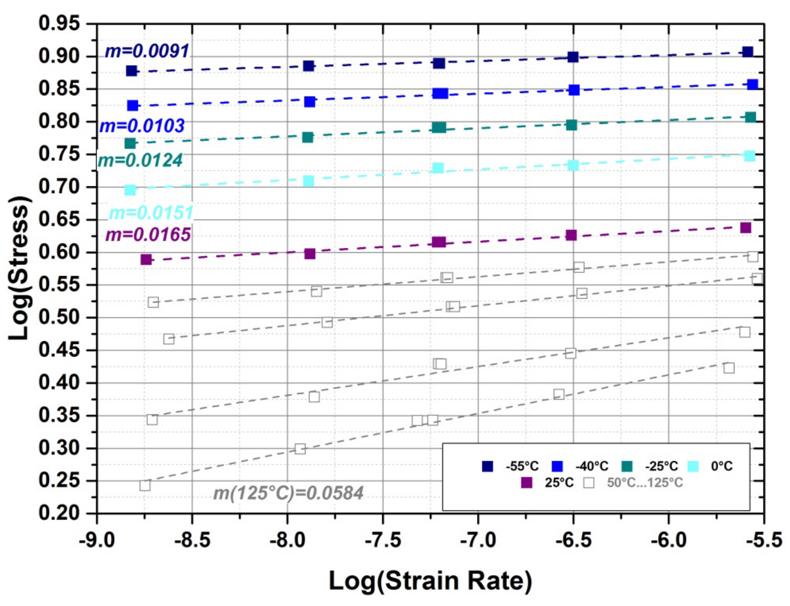

Fig. 8: Strain rate sensitivity exponent *m* as a function of temperature

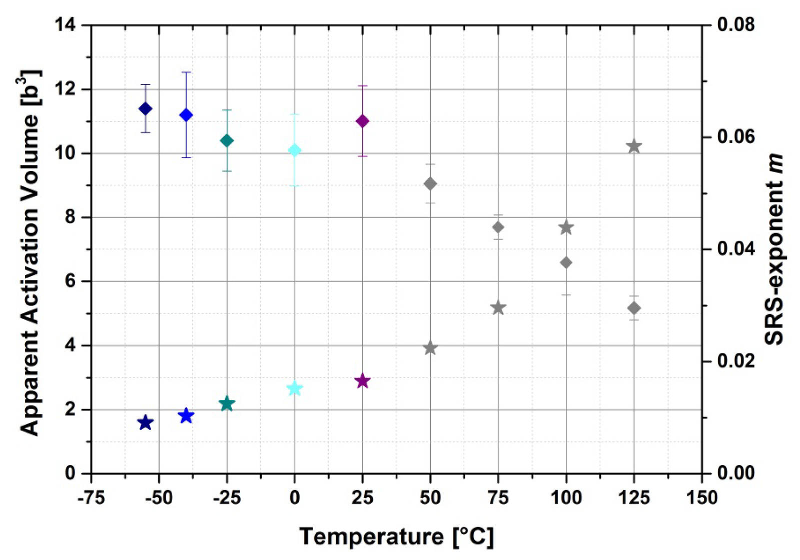

Fig. 6: Apparent activation volume and strain rate sensitivity exponent *m* vs. temperature

References

[1] Wehrs, J., Deckarm, M., Wheeler, J.M., Maeder, X., Birringer, R., Mischler, S., Michler, J. (2017). Elevated temperature, micro-compression transient plasticity tests on nanocrystalline Palladium-Gold: Probing activation parameters at thelower limit of crystallinity. *Acta Materialia* 129, pp. 124-137.

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