STUDYING DEFORMATION MECHANISMS OF NANOCRYSTALLINE NICKEL BY THERMAL ACTIVATION ANALYSIS AT SUBAMBIENT TEMPERATURES AND HIGH STRAIN RATES

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Electrodeposition and magnetron sputtering are promising methods for depositing thin films with nanocrystalline (nc) microstructures. Nc metals are attractive materials, as they show considerably higher mechanical strength compared to their poly- or monocrystalline counterparts. However, they also feature pronounced time- and rate-dependent inelastic behavior and their microstructure may change drastically when exposed to elevated temperatures or ion irradiation. Therefore, in order to assess the mechanical behavior and deformation mechanisms of these materials under controlled conditions and at a constant microstructure, it is desirable to perform thermal activation analysis at subambient temperatures and high strain rates on pristine samples.
Large arrays of micropillars were fabricated by electrodeposition of nc Ni into lithography molds by LIGA leading to non-tapered, damage-free microspecimens. X-ray diffraction (XRD) measurements and transmission electron microscopy (TEM) imaging revealed a grain size of approximately 28nm. EDX analysis showed a homogeneous elemental composition and no concentration of impurities at the grain boundaries. A micromechanical testing device was developed that allows performing nanomechanical experiments at sub-ambient temperatures down to 120K in a large range of strain rates between 10^{-4} and $10^{3}s^{-1}$.
Micropillar compression experiments were performed on the nc Ni micropillars in a scanning electron microscope (SEM) at temperatures ranging between 160K and 293K and strain rates from 10^{-3} to $5{\cdot}10^{2}s^{-1}$. Yield stress was extracted based on a 2% strain offset rule and found to vary strongly with temperature. Post-experimental high resolution images were taken in a SEM to reveal deformation patterns. Furthermore, TEM lamellae were prepared from micropillars tested at strain rates of $10^{-3}s^{-1}$, $25s^{-1}$, and $500s^{-1}$ and imaged to reveal deformation-induced changes in microstructure. Strain rate sensitivity exponent m was determined to be in the range of 0.003 to 0.008. Apparent activation volume was found to decrease with temperature from $10b^3$ at 293K to $2\text{-}4b^3$ at 160K. These values are consistent with dislocation nucleation, but also other deformation mechanisms like grain boundary or defect diffusion.
This study highlights a new approach for assessing the mechanical behaviour of nc materials by testing large numbers of pristine electrodeposited micropillars at various subambient temperatures and in a large strain rate range. This allows assessing the deformation mechanisms by thermal activation analysis while keeping artifacts from specimen preparation and microstructural changes throughout the experiments at a minimum.

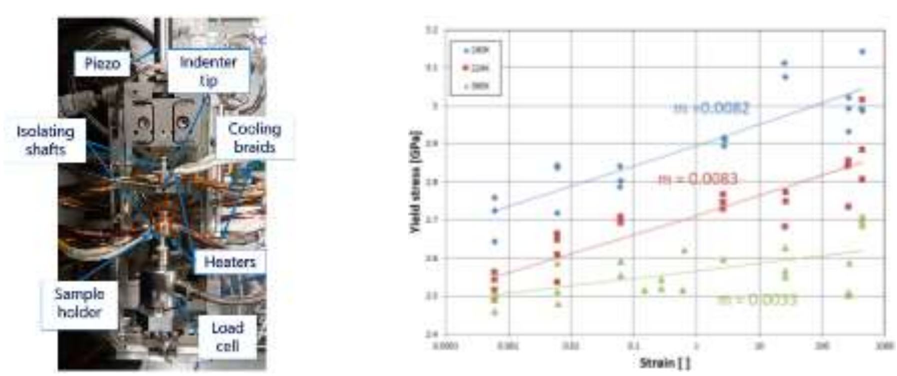

Figure 1 – Left: Cryogenic indenter setup. Right: Yield stress as a function of temperature and strain rate