Application Note

Mechanical testing at the micro- and nano-scale

Designing micropillar strain rate jump tests to study time dependent plasticity

Introduction

Nanocrystalline and ultra-fine grained materials show enhanced strain rate sensitivity (SRS) in comparison to their coarse grained counterparts. Conventionally, SRS of these materials has been measured on bulk specimens, either in compression or in tension. With the emergence of electrodeposition and magnetron sputtering as promising methods for deposition of controlled microstructures of nanocrystalline and twinned materials, it has become necessary to adapt bulk mechanical tests to smaller samples. Due to inherent difficulty in micro-sample preparation and testing, majority of the micro and nano-mechanical efforts to measure strain rate sensitivity involve approaches based on nanoindentation [1-3]. The current study aims to extend the repertoire of nanomechanical testing approaches for strain rate sensitivity measurements by designing micropillar strain rate jump tests on nanocrystalline nickel. The results from these tests will be compared directly with more established methods like tensile and nanoindentation strain rate jump tests. For maximum comparability, all tests will be performed on the same sample.

Experimental

Nominally pure, nanocrystalline nickel tensile specimens were produced by means of a proprietary electrodeposition process on a silicon wafer.

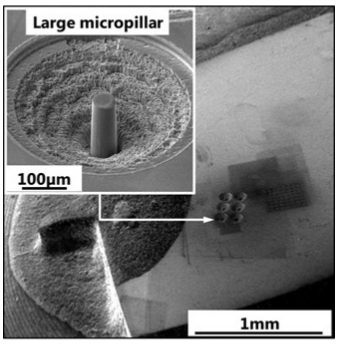

Figure 2. *Nanoindentation and micropillar compression tests were performed on the undeformed gripping section of the tested tensile bars after tensile tests.*

The samples had an average grain size of ~30 nm, determined from TEM analysis. Micropillars with nominal diameter of 3.5 µm and aspect ratio of 2.5 were machined on the undeformed gripping section of failed microtensile samples (fig. 1) using a TESCAN Lyra gallium focused ion beam (FIB) system. Micropillar compression and nanoindentation tests were performed using an Alemnis nanoindenter (Alemnis AG) inside a Zeiss DSM962 SEM. For verification and control, micropillars were also fabricated on a ⟨123⟩-oriented single crystal of pure nickel (Goodfellow Ltd., Cambridge, UK) which does not exhibit strain rate sensitivity.

Results and discussion

Since the nanoindenter uses a highly responsive piezoelectric actuator and is intrinsically displacement-controlled, strain rates can be varied back and forth over four orders of magnitude in a single experiment with high degree of accuracy.

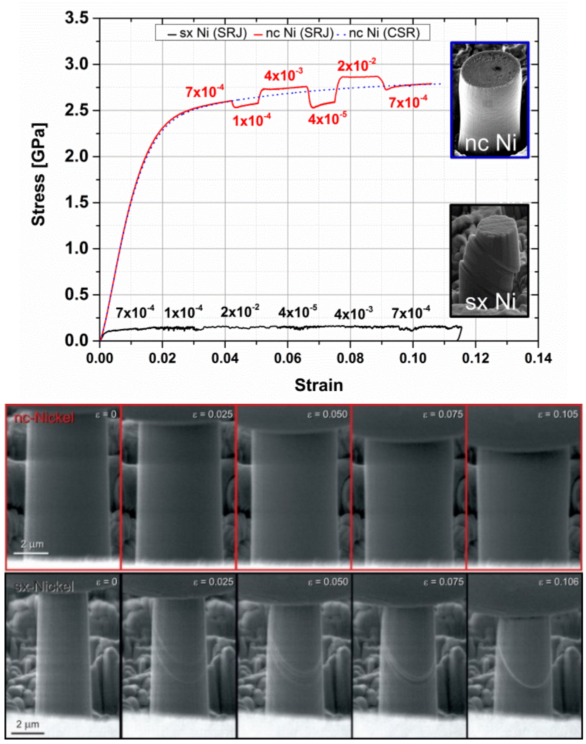

Figure 2. Micropillar stress-strain curves for nanocrystalline and ⟨123⟩ oriented single crystal nickel. In-situ SEM observation of the deformation behavior of nanocrystalline (nc) nickel (red) and single crystal (sx) nickel (black) micropillars during compression test at selected strains.

Strain rate jump tests were performed by varying the strain rate from $4 \times 10^{-5}\, s^{-1}$ to $2 \times 10^{-2}\, s^{-1}$ within each test (fig. 2). The jumps were observed to be sharp with rather quick stabilization of the flow stress and small transients for nanocrystalline nickel. The micropillar strain rate jump tests were found to be highly repeatable due to the uniform microstructure within the material. To illustrate the tremendous strengthening effect of the nanocrystalline microstructure, results for ⟨123⟩-oriented single crystal of nickel are also shown. The yield strength of the Ni single crystal at 1 % offset was observed to be only 0.13 GPa; nearly a factor of twenty less than its nanocrystalline counterpart. And as expected, it does not show any strain rate sensitivity.

While the nanocrystalline nickel micropillar accommodates most of the plastic deformation in the upper half, the single crystal pillar develops several large slip steps over the entire pillar diameter (Fig 2). This is an added advantage of in-situ testing in SEM that allows the observation of the specimen deformation throughout the test.

The strain rate sensitivity exponent, m, is a measure of the dependence of change in flow stress, σ, as a function of change in applied strain rate, $\dot{\varepsilon}$, at constant temperature, T:

$$m = \left[\frac{d(\ln \sigma)}{d(\ln \dot{\varepsilon})}\right]_T$$

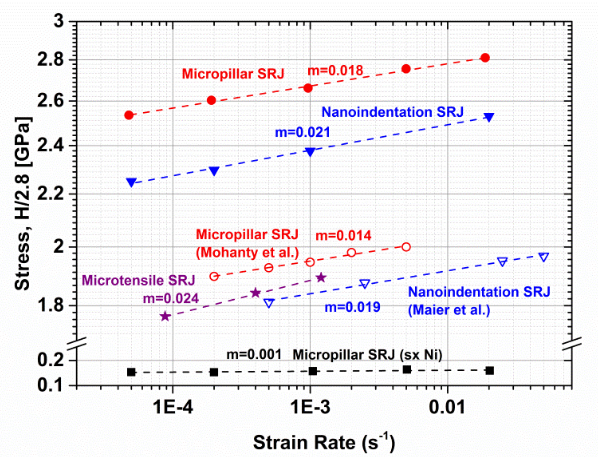

Figure 3. Flow stress as a function of strain rate for tension, microcompression and nanoindentation (H/2.8) tests from the current work and the literature [2, 4]

The log-log plot in figure 3 compares the resulting flow stress as a function of strain rate for the three applied test methods with literature results. The hardness values obtained from nanoindentation were converted into representative stress values by dividing them by a constraint factor of 2.8. Although figure 3 compares three different loading cases and three different nc-nickel samples with grain sizes of 30 nm (current study), 27 nm (Mohanty et al. [4]) and 80 nm (Maier et al. [2]), the results are all fairly consistent, especially when comparing the m values between measurements performed in this study. The variation in flow stresses between the small scale (nanoindentation, micro-compression) and larger scale (tension) measurements in this study can be attributed to crystallographic texture. XRD measurements revealed a ⟨111⟩-fiber texture in the loading direction of indentation/ microcompression experiments. The results from the single crystal nickel sample vary significantly from the nanocrystalline results in both absolute flow stress and strain rate sensitivity exponent values, as expected.

Conclusions

Micropillar strain rate jump tests were designed for studying time dependent plasticity of nanocrystalline Ni samples. The results were compared with two established micromechanical measurement techniques for assessing strain rate sensitivity on the same nanocrystalline nickel specimen in order to study the comparability and consistency of the test results. The extracted SRS exponents from these three methods were found to be in excellent agreement. This validates the developed micropillar strain rate jump tests and suggests that the techniques compared herein can be reliably and interchangeably used to accurately measure strain rate sensitivity in a wide variety of materials.

References

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For further information about the Alemnis range of mechanical testing instruments, please contact:

Dr. Nicholas Randall, nicholas.randall@alemnis.ch