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# Comparison of in situ micromechanical time dependent plasticity techniques: micropillar compression, nanoindentation and micro-tensile tests Materials Science & Technology



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# **Objectives**

- Comparison of strain rate jump tests performed in: uniaxial tension, uniaxial compression and triaxial nanoindentation
- Comparison of strain rate sensitivity and activation parameters, obtained from experiments that cover the range from bulk to nanoscale

# Introduction

Electrodeposited nanostructured metals are attractive for producing micro components due to fabrication (design flexibility and dimensional accuracy) and mechanical property (high strength, ductility) considerations [1-4]. However, their mechanical characterization, especially for thin films is not straight forward. This study aims at the development and validation of suitable uniaxial testing methods for mechanical characterization of these microcomponents.

# Strain rate jump (SRJ) tests

Recently, there have been attempts at extracting the strain rate sensitivity exponent from micromechanical tests: nanoindentation [6] and micropillar compression [7]. This study aims at an overall comparison of results from room temperature SRJ tests on the same sample (nanocrystalline Ni) using uniaxial tension, micropillar compression and nanoindentation:

- Nanoindentation and microcompression SRJ tests performed on the failed sample from uniaxial tensile SRJ test.
- Similar strain rates used for all three tests
- Strain rate varied over three orders of magnitude
- 3.5 -----

## Material

- Electrodeposited nanocrystalline nickel (sulfamate bath), grain size ~30nm
- Deposition of micro parts in LiGA-molds (~250 µm thickness)
- Microstructural characterization: XRD, ion channeling contrast imaging, TEM





Ni micro components, deposited into LIGA-mold

Ga-FIB CS of Ni deposit (Ion channeling, 30kV, 80pA)

TEM CS of Ni deposit (STEM, 200kV, 0.7nm)

# Micro bulk tensile testing of miniaturized samples

- Bulk tensile tests are uniaxial, straight forward to perform and well understood
- Modification of a commercial Kammrath & Weiss tensile stage for testing LIGA samples
- Dogbone shaped micro-tensile specimen: gauge 0.15µm x 0.25µm Strain measured using digital image correlation (DIC)



Stress-strain curve and a polynomial fit obtained from uniaxial microtensile SRJ tests. Jumps have been performed over one order of magnitude. Flow stress stabilizes immediately after each jump. The numbers represent applied strain rates  $(S^{-1}).$ 



Stress-Strain curve of micropillar SRJ test on nanocrystalline nickel (red) and single crystal nickel (black). The blue curve was obtained from a CSR compression test on the same nanocrystalline nickel.









Above: LiGA micro specimen dimensions *Left: In-Situ tensile stage (Kammrath &* Weiss) with modified displacement sensors, load cell and gripping system



- Stress-strain curves from miniaturized tensile test
- Maraging steel is the current industrial standard for fabrication of microcomponents
- Test of standard metrials yield textbook results  $\rightarrow$  validation

Tensile stress-strain curves of nc Ni and maraging steel



Load-displacement curve obtained from nanoindentation strain rate jump test. Strain rate varied by chaning P/P ratio over four orders of magnitude.

m=0.001 Micropillar SRJ (sx Ni) 0.2 1E-4 0.01 1E-3 Strain Rate [s<sup>-1</sup>]

Log-log plot of stress vs. strain rate comparing the results of SRJ tests in uniaxial tension (micro-bulk), micropillar compression and nanoindentation. Hardness values from nanoindentation have been converted to flow stress by dividing with a factor 2.8 [6]..

### **Results and conclusions**

- Three different micromechanical measurement techniques for assessing SRS were applied on the same nanocrystalline nickel specimen to study the comparability and consistency of the test results
- Consistent strain rate sensitivity exponent *m* values were obtained from all three tests. Excellent agreement with literature values [6,7].
- The obtained apparent activation volume 10b<sup>3</sup> for nanocrystalline nickel is consistent with grain boundary diffusion processes and dislocation glide based plasticity
- Due to the typical grain size variation in electrodeposited metals it is likely that both mechanisms are active at the same time
- This study suggests that the techniques compared herein can be reliably and



Postcompression high-resolution scanning electron micrographs: (a) nanocrystalline nickel showing displaced grains distributed throughout the pillar and (b) single-crystal nickel showing deformation confined to discrete slip bands traversing the pillar..

# **Micropillar preparation with Ga-FIB**



- Micropillars machined in the undeformed gripping section of tensile specimen
- Allows for multiple tests on one single sample
- Better comparability of test results

inter changeably used to measure SRS accurately in a wide variety of materials

Please see also: J. Wehrs et al: "Comparison of In Situ Micromechanical Strain-Rate Sensitivity Measurement Techniques", JOM, August 2015, Volume 67, Issue 8, pp 1684-1693

### **References:**

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# Work in progress

- > Investigation of time dependent plasticity phenomena in nanocrystalline thin films: relaxation, creep and strain rate jump tests at elevated temperatures
- Investigation of mechanical properties of nanocrystalline nickel-tungsten alloys for improved thermal stability

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