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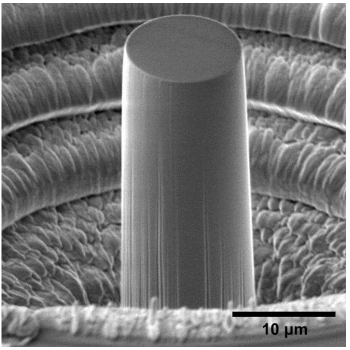

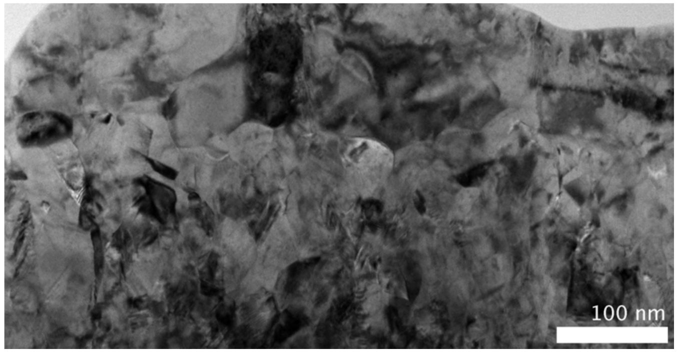

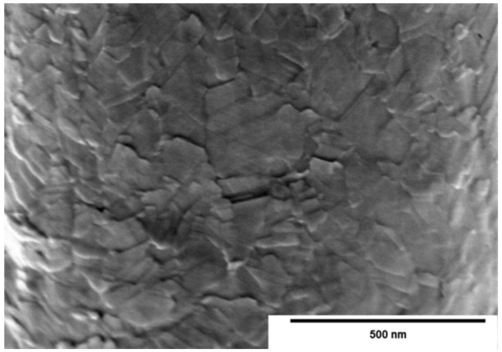

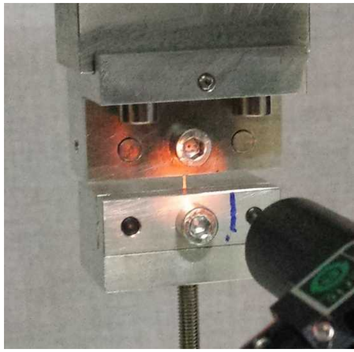

Room temperature time-dependent plasticity of nanocrystalline nickel thin films

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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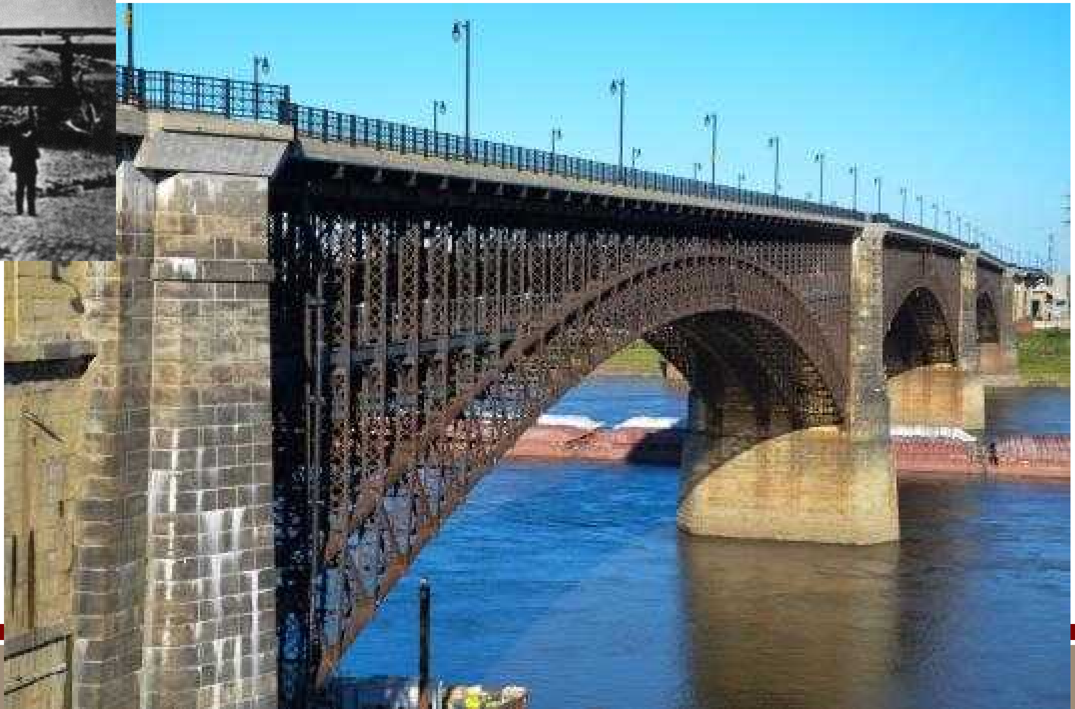
Why do we need to worry about it at room temperature?

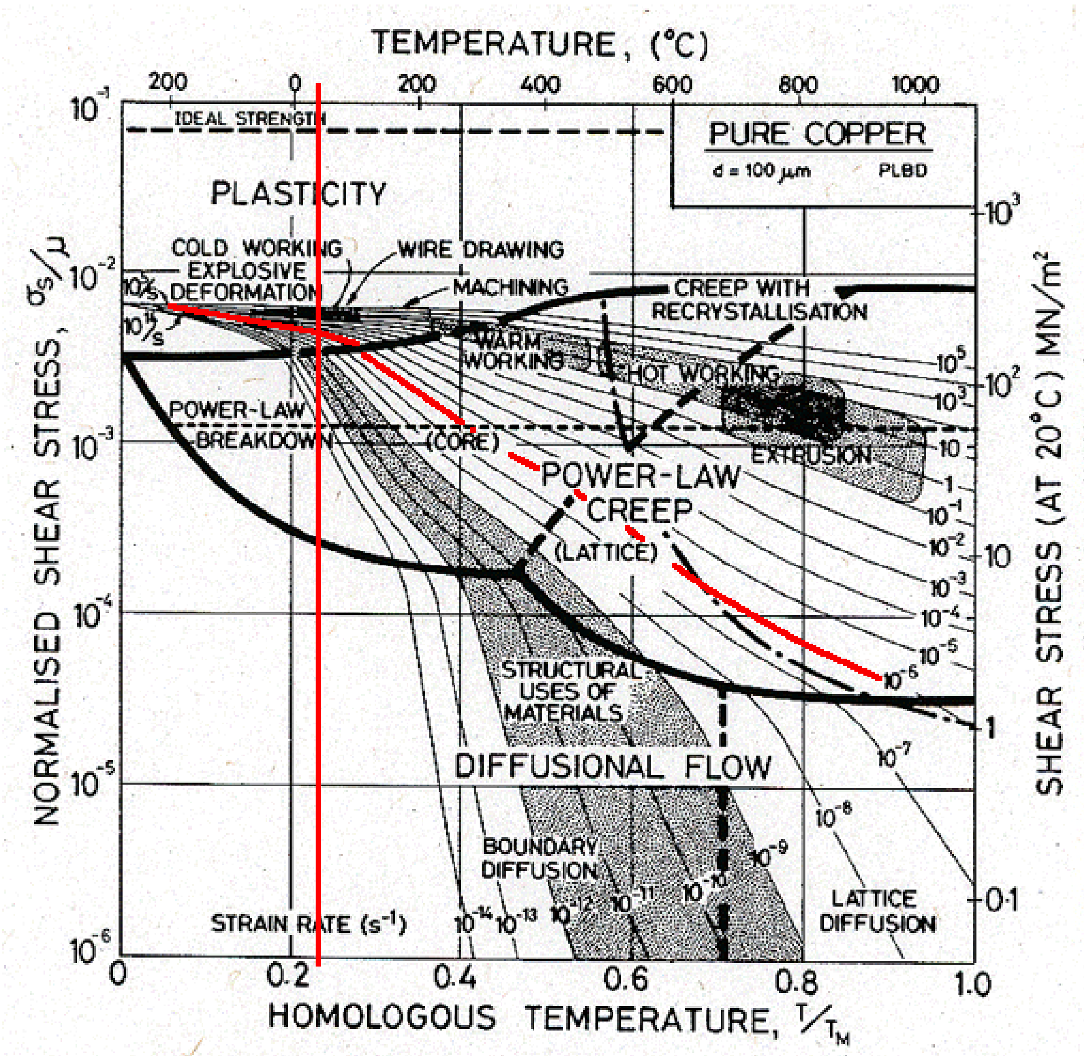
Eads bridge, St. Louis ca. 1874

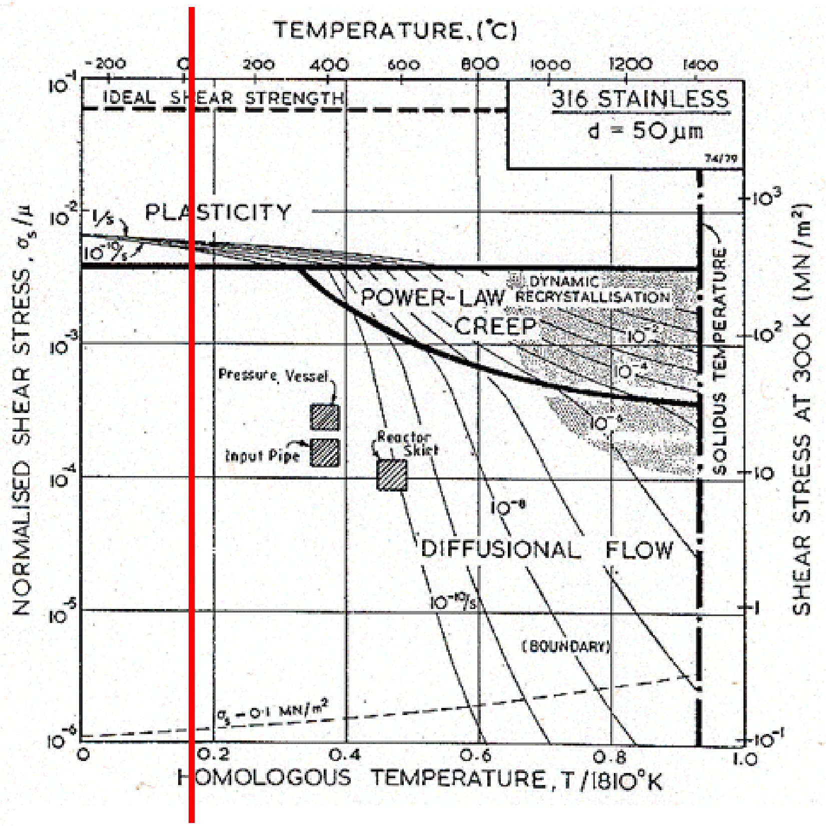
It's been 140 years, creep of chrome steel to failure at ambient St. Louis temperature has not been an issue.

Eads bridge, St. Louis 2012



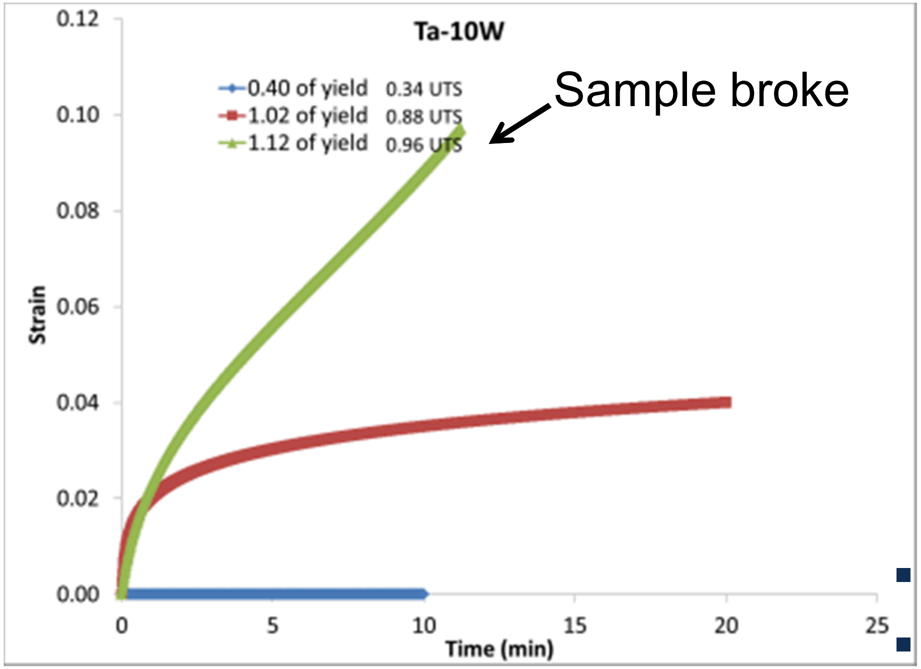
How do we think about creep?





All metals creep at room temperature

Conventional wisdom is that at room temperature, metals only age by fatigue or corrosion



298K/3308K = 0.09*T_m
1381°C = 0.5*T_m

- Springs
- Glass to metal seals in electrical connectors.
- Electrical connections
- Pressure vessels
- Anywhere there's a designed residual stress

In most metals, RT creep occurs by dislocation glide

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HELD AT 2% STRAIN FOR 1 DAY

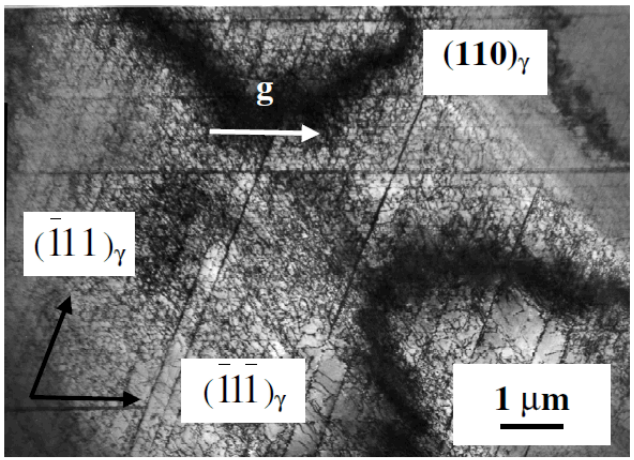


FIGURE 13 – AISI 304L before testing in A-TEM

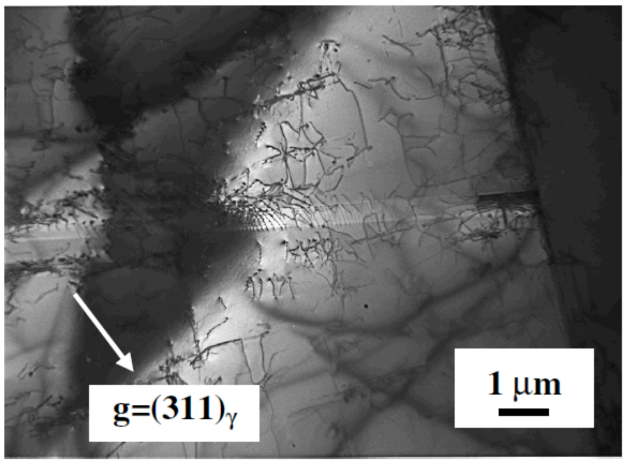


FIGURE 14 – AISI 304L after relaxation testing at Rp_2 in A-TEM

RESULTS FROM LOW TEMPERATURE CREEP AND RELAXATION EXPERIMENTS OF FOUR DIFFERENT STAINLESS STEELS

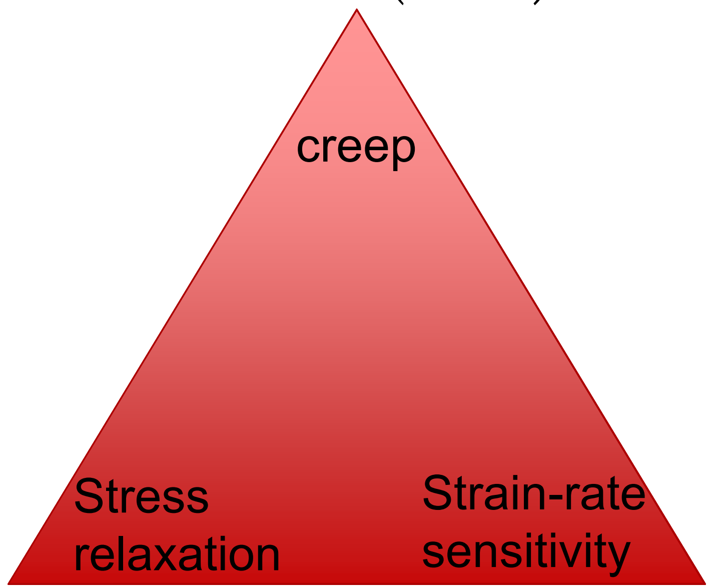
U.H. Kivisäkk and E. Wallin
AB Sandvik Materials Technology
R&D
SE-811 81 SANDVIKEN
SWEDEN

NACE INTERNATIONAL CORROSION 2008 CONFERENCE & EXPO

Creep and stress relaxation are interrelated

$$\dot{\gamma} = A\rho_m b(A'/l)\nu_{id} \exp(-\Delta G/kT)$$

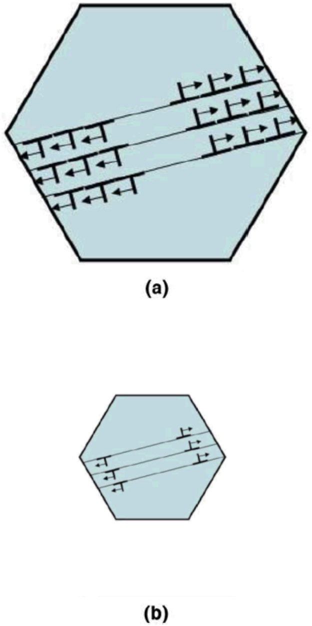
$$\Delta\tau = \frac{-kT}{V_r}\ln\left(1 + \frac{t}{c_r}\right)$$



$$\Delta\gamma_p = \frac{kT}{MV_c}\ln\left(1 + \frac{t}{c_c}\right)$$

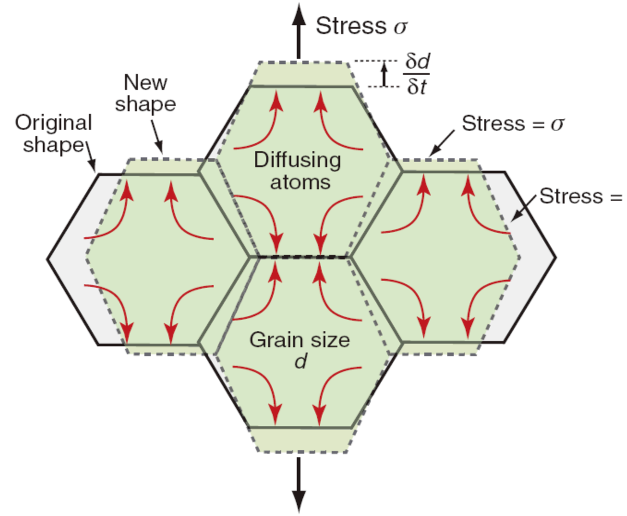
$$\Delta\tau = \frac{kT}{V_a}\ln\left(\frac{\dot{\gamma}_1}{\dot{\gamma}_2}\right)$$

How do nanocrystalline metals fare?



Microcrystalline

Nanocrystalline



Diffusion mechanisms

Less dislocation mediated activity

Extensive grain boundary diffusion

Hardness

$$H \propto \frac{1}{\sqrt{d}}$$

Hall-Petch behavior

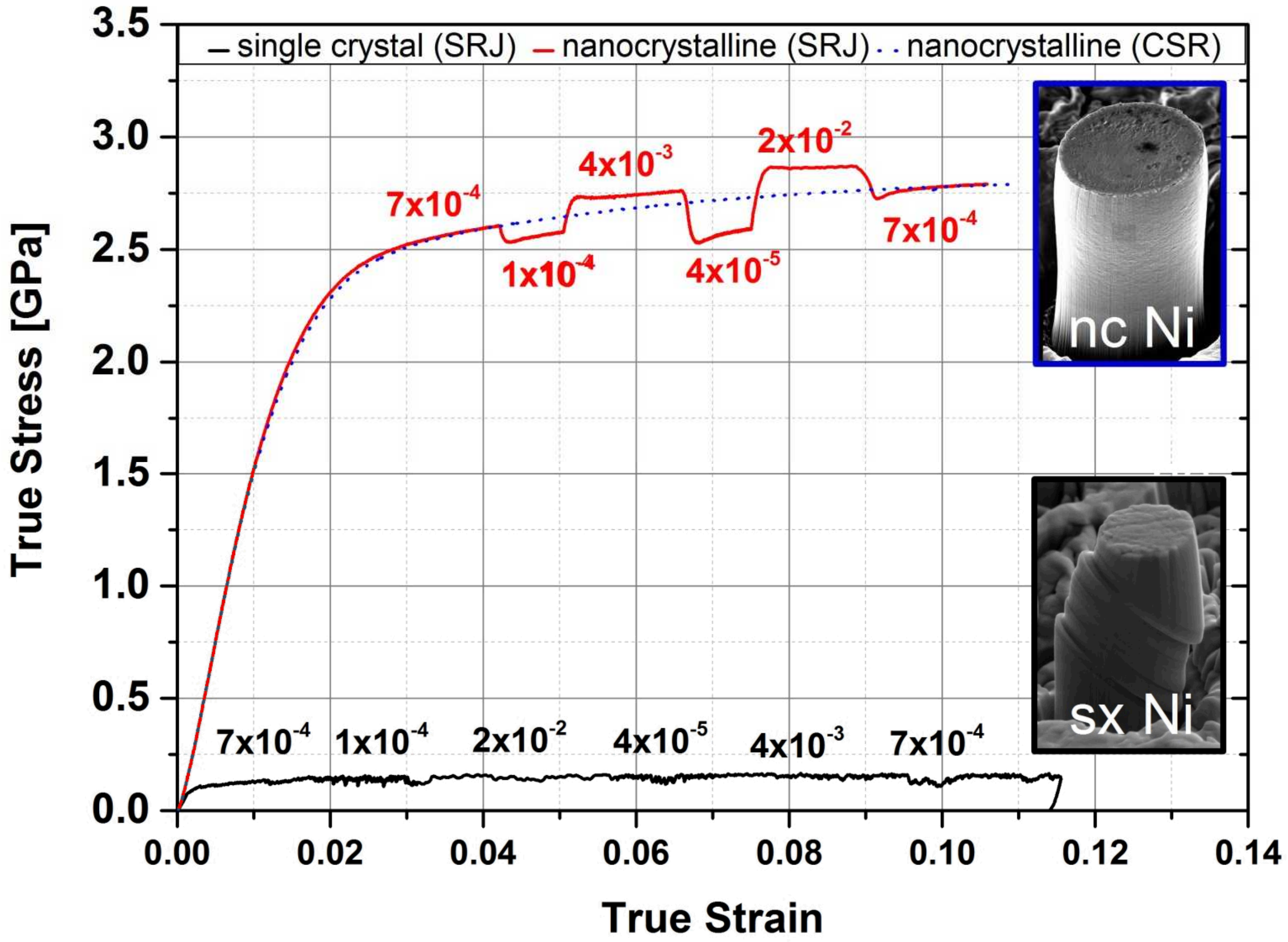
Decrease in grain size

Creep resistance

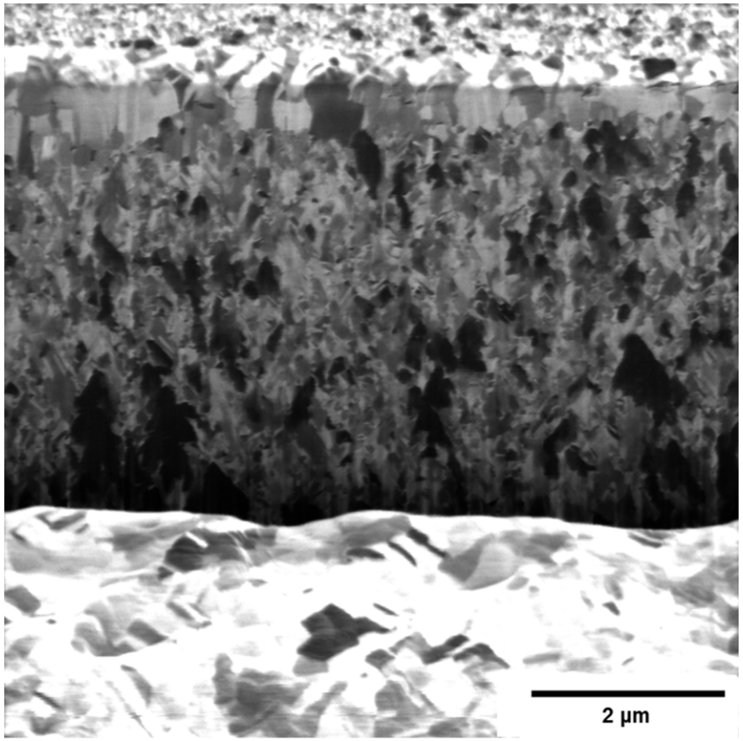
$$\frac{1}{\dot{\varepsilon}} \propto d^3$$

Progress in Materials Science 51 (2006) 427–556

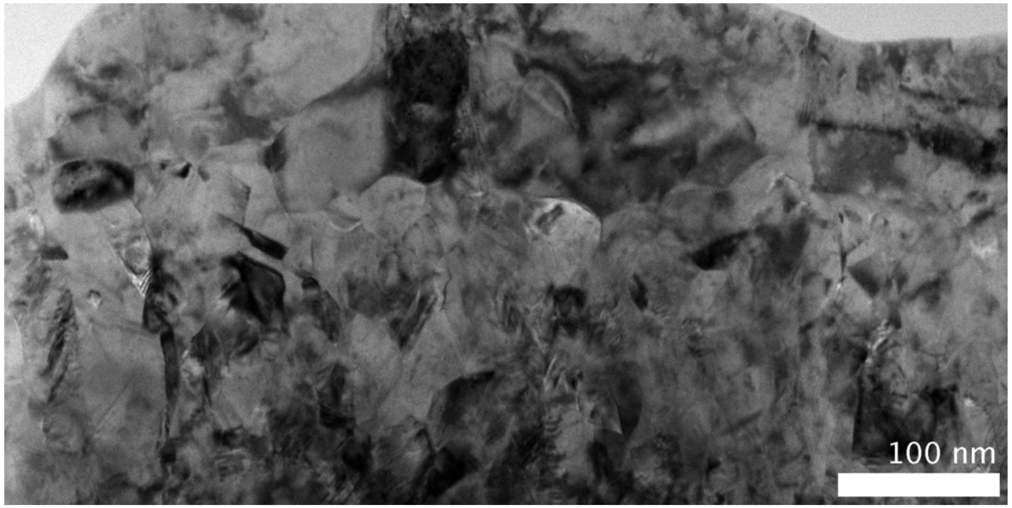
Microcompression strain-rate jump



Electroplated nanocrystalline Ni



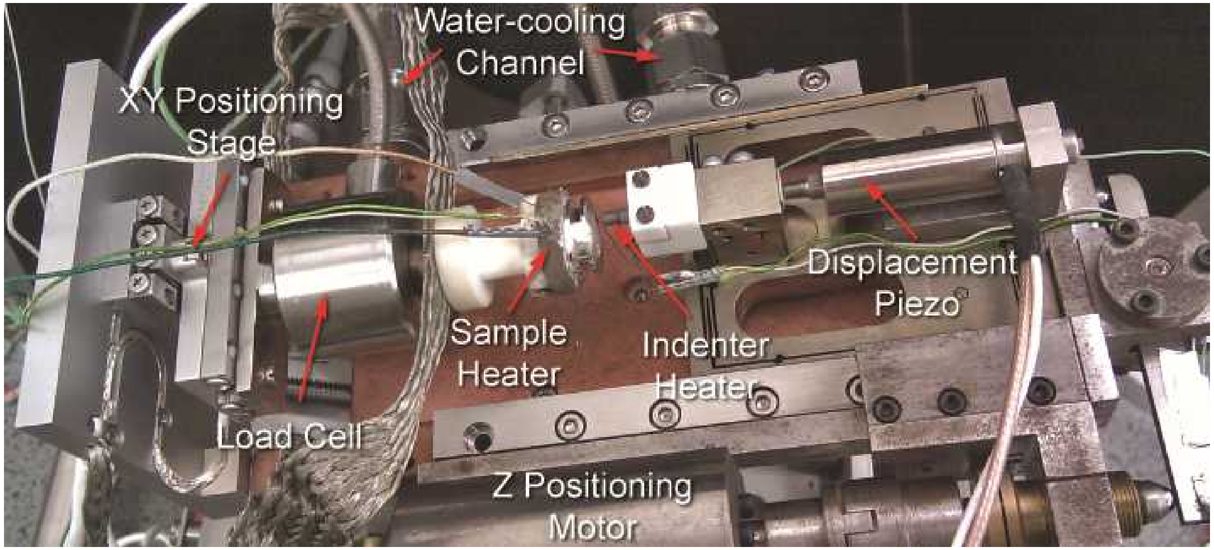
XRD crystallite size: 30nm



FIB channel contrast imaging of the microstructure

TEM of sample cross section

How to test creep in thin films…



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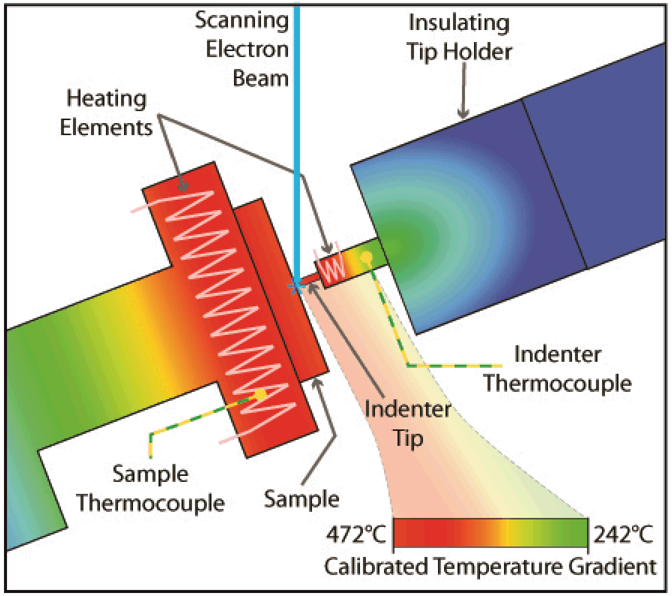
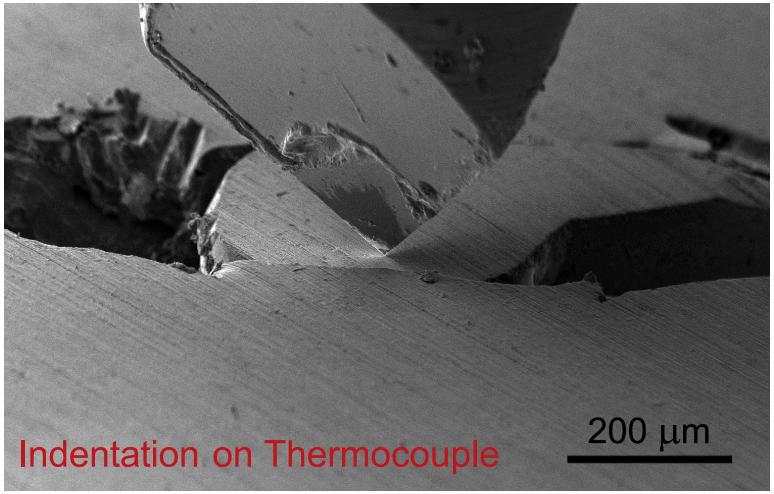
- System parameters
  - Displacement resolution: 0.1nm   Load noise floor: 15µN
  - Precise positioning (< 50nm) and direct observation of deformation inside the SEM
  - Intrinsically displacement controlled, but pseudo-load control possible with feedback loop
- Current max temperature: 500°C *(~800°C with improved sample heater)*
- Independent indenter and sample heating with feedback control thermocouples
  - Testing performed at thermal equilibrium with Contact Drift tunable to < 0.1 nm/s
  - Thermally calibrated indenters for referenced surface temperature matching

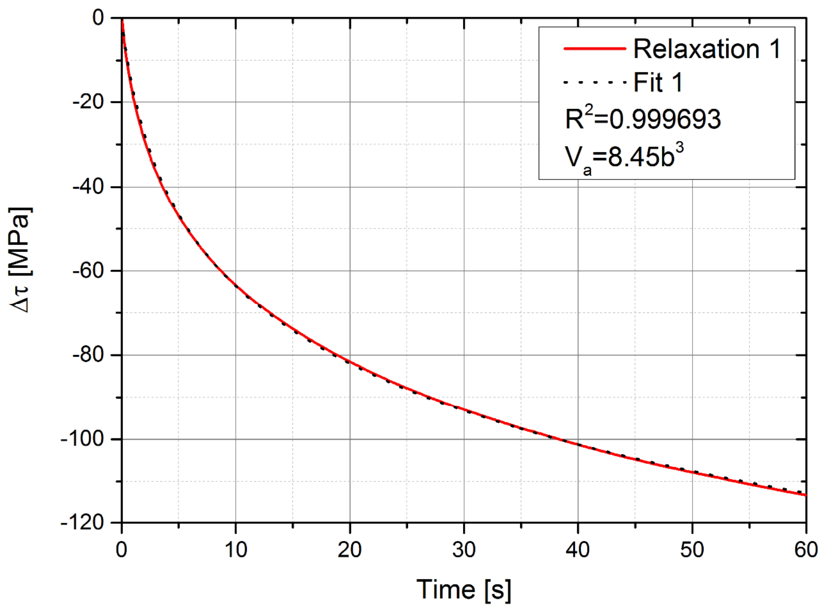
How to test creep in thin films…





- Local heating of nano-contact:
  - Miniaturised heaters for both the sample and indenter efficiently heat only regions of interest.
  - Watercooling of the frame and a high conductivity thermal escape path prevent the displacement and load sensors from significantly heating.
- Precisely calibrated temperatures at the nano-contact – minimize drift
  - Sample and indenter temperatures must be at equilibrium to prevent thermal drift/heat flow.
  - Control thermocouples are spatially separated from the nano-contact, resulting in thermal gradients obscuring the true sample/indenter temperatures.
  - Indenter tip temperatures calibrated via contact measurements on thermocouples and Raman spectroscopy.

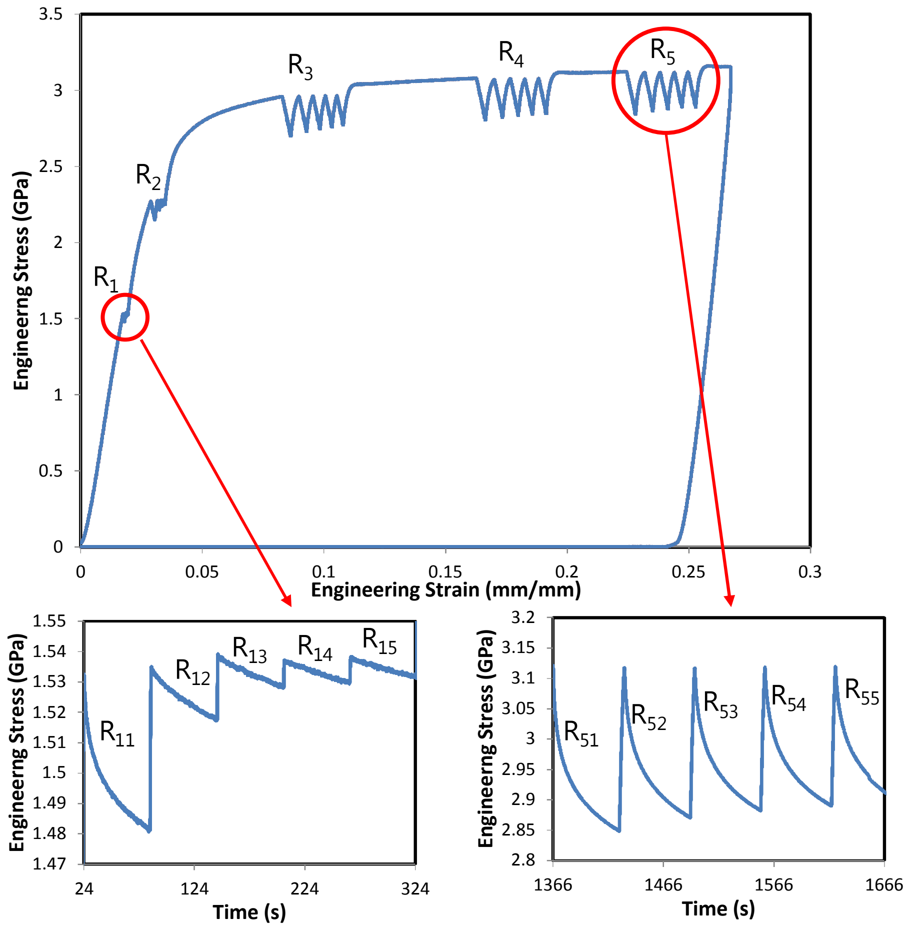
Conventional stress relaxation



$$\Delta\tau = -\frac{kT}{V_a}\ln\left(1+\frac{t}{c}\right)$$

- Stresses relax by 5-10% after only 1 minute!
- The ***apparent*** activation volume is convoluted by the evolving microstructure.

Repeated relaxation…

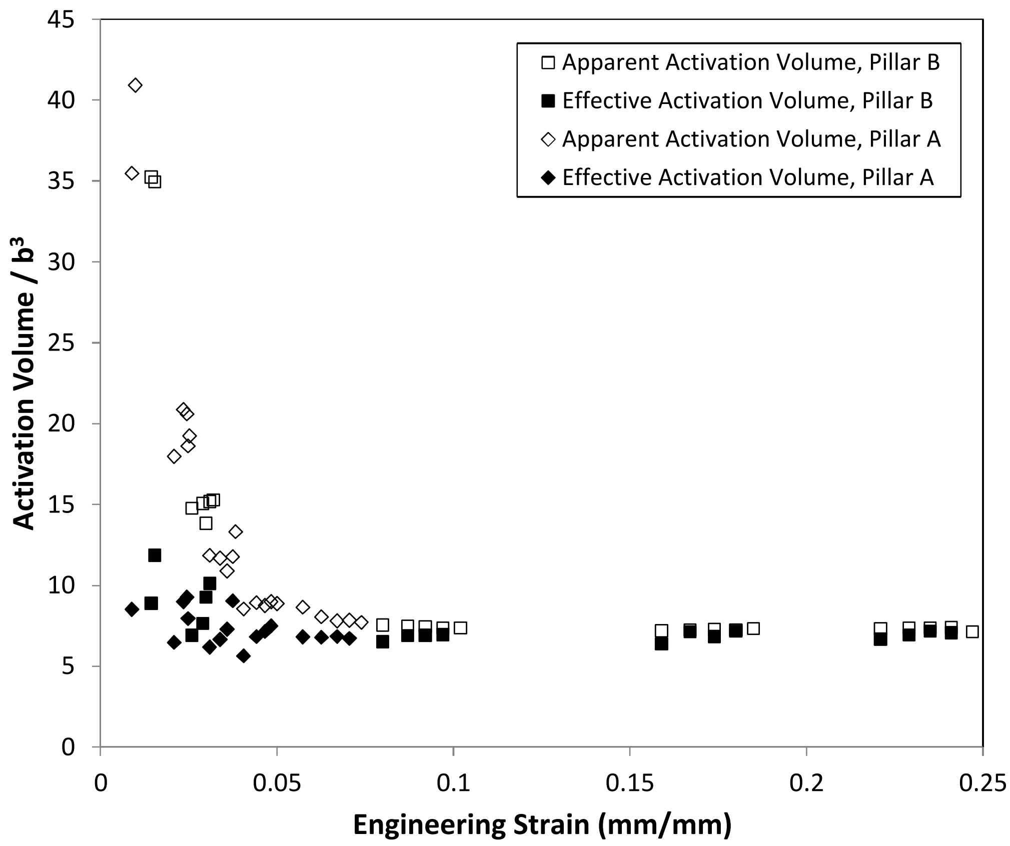


$$V_e = kT\frac{\ln(\dot{\gamma}_{i2}/\dot{\gamma}_{f1})}{\Delta\tau_{i2-f1}} = kT\frac{\ln(\dot{\tau}_{i2}/\dot{\tau}_{f1})}{\Delta\tau_{i2-f1}}$$

$$\dot{\tau}(t) = -\frac{kT}{V_a}\frac{1}{(c+t)}$$

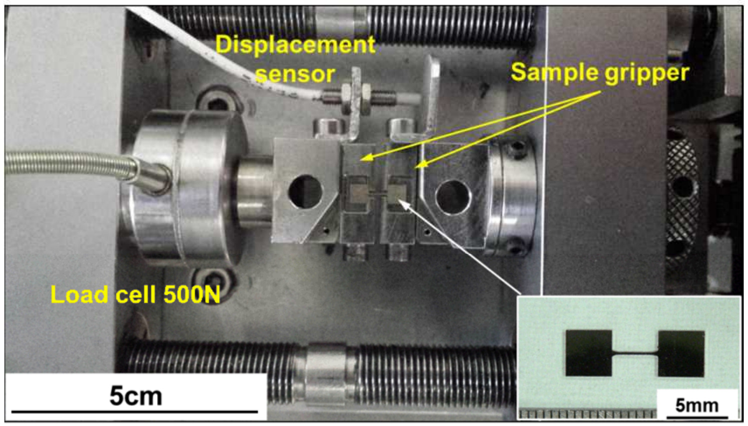
- The ***effective*** activation volume gives an instantaneous measure under constant microstructure

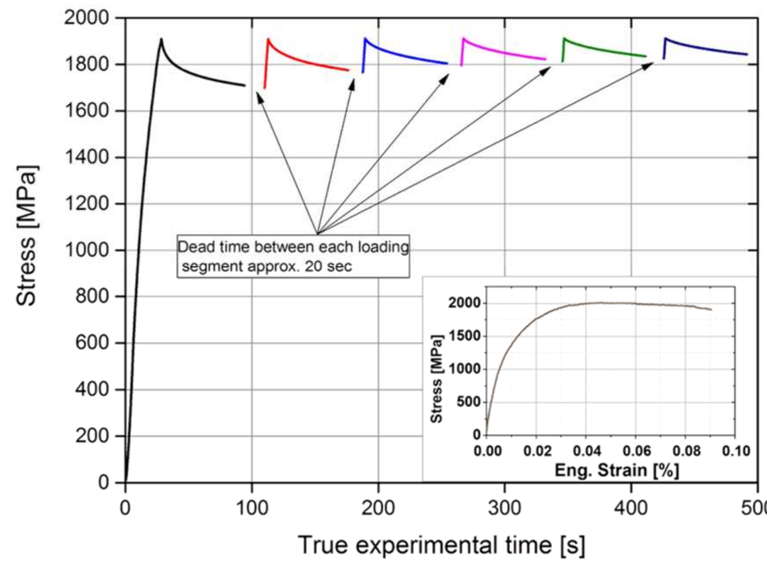
Activation Volume Determination

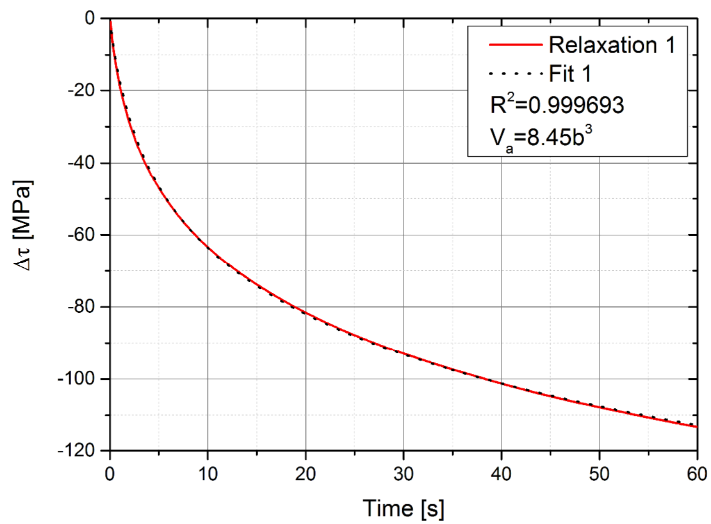


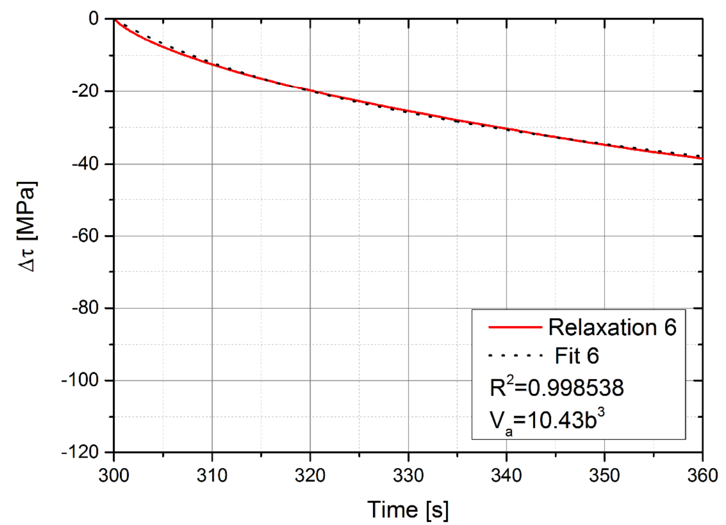
- The effective activation volume is more uniform throughout deformation at 6-10 b^3.

Confirming with miniature tension

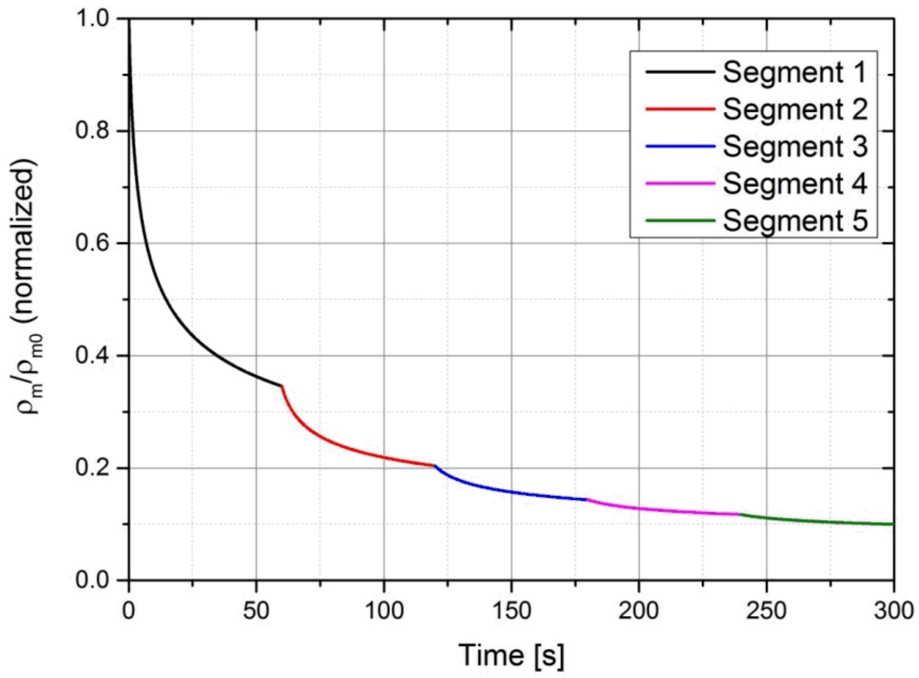








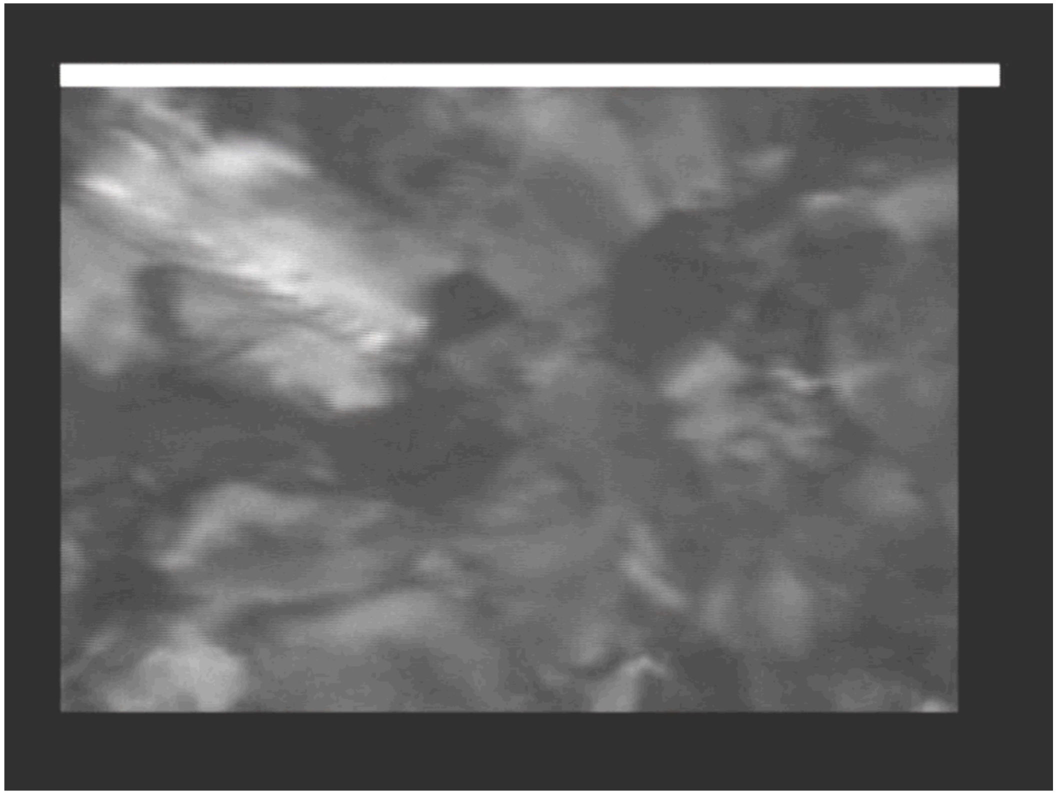
A dislocation exhaustion picture…



- The relaxation transient could be caused by a decay in the mobile dislocation density.

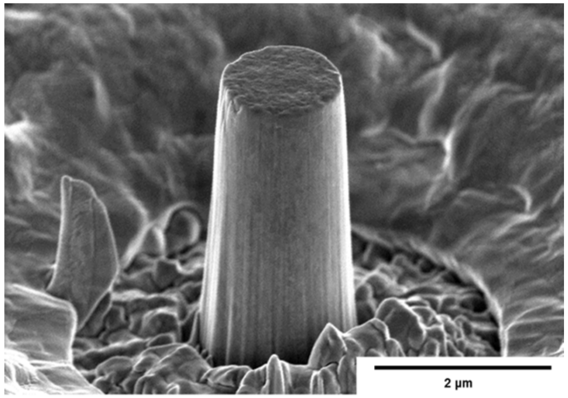
Dislocation exhaustion in action

In-situ TEM experiments: Marc Legros (CEMES-CNRS), Ehsan Hosseinian, Olivier Pierron (Georgia Tech)

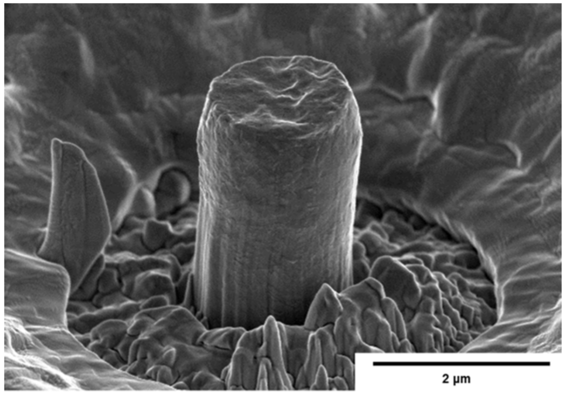


- In-situ TEM of larger grained Au shows dislocation exhaustion…

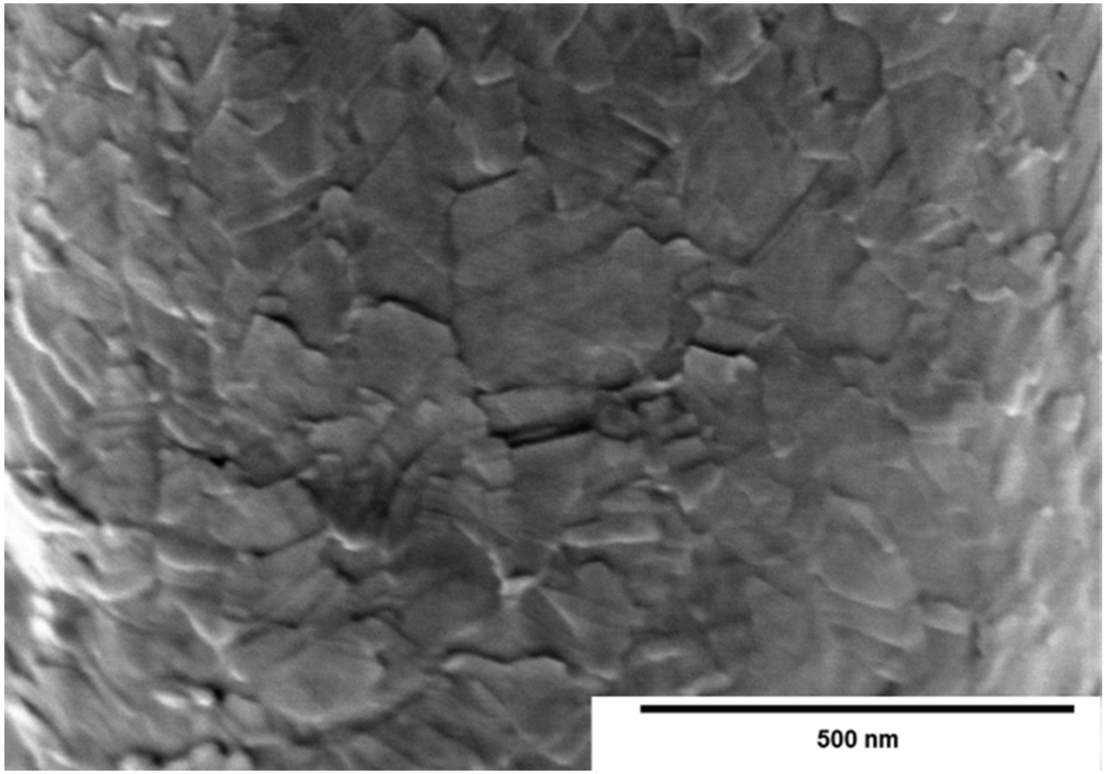
Grain boundary sliding also contributes



Before compression



After compression



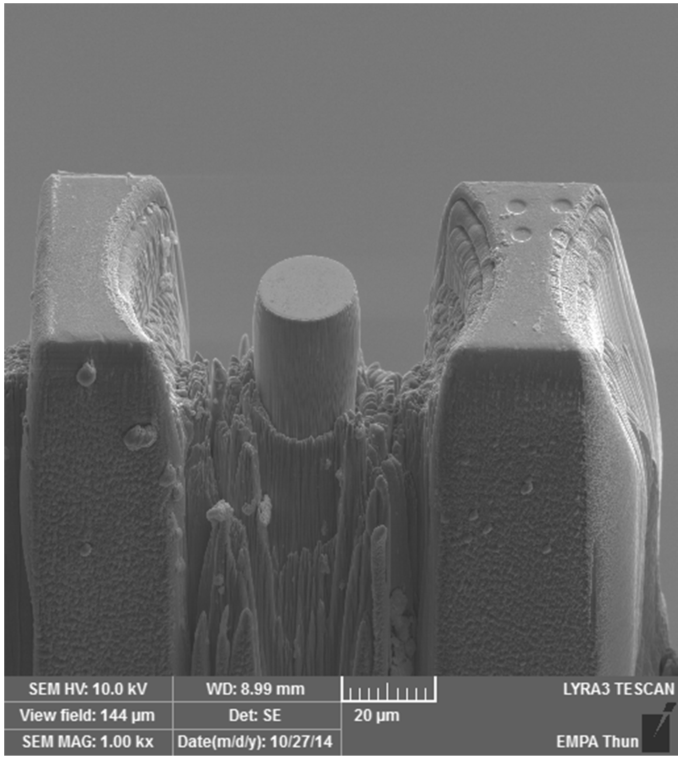
Extensive grain boundary sliding observed on pillar surface
Large grains exhibit slip traces

Summary

- Creep, stress-relaxation, and strain-rate sensitivity are interrelated forms of viscoplasticity
- While all metals creep at room temperature, nanocrystalline metals are particularly susceptible.
- We demonstrate the use of pillar compression to measure viscoplastic behavior
- Activation volumes of 6-10 b^3 are consistent with miniature tension, and published literature, consistent with GB-mediated mechanisms of either dislocation plasticity or sliding.

Backup Slides

Synchrotron tells us more…



Crystallite size determined from PDF

Ondrej Milkovic, Jana Michalikova, Karel Saskl