Rickover Fellowship Program In Nuclear Engineering

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Dislocation Impact(s) on Hydrogen Embrittlement

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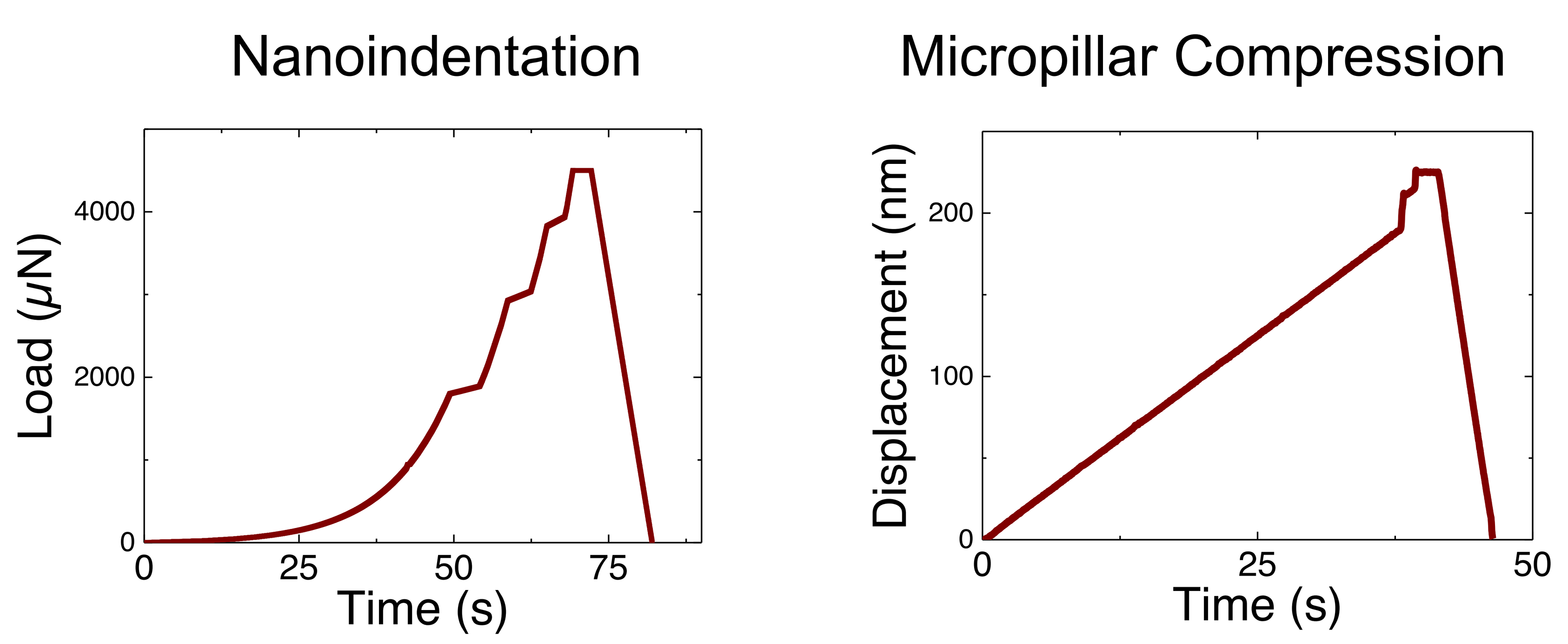
Motivation and Background

- The mechanical energy necessary for dislocation motion can be described by the apparent activation volume (V*) multiplied by a critical stress
- V* and Q (activation energy) measurements for silicon require testing at small scales
- Nanoindentation and micropillar compression have very different stress states so V* and Q values should be compared
- Here, strain rate jump (SRJ) tests are used to determine activation volumes in nanoindentation and micropillar compression
- Changes in these parameters can give insight to how hydrogen impacts dislocation motion

Experimental

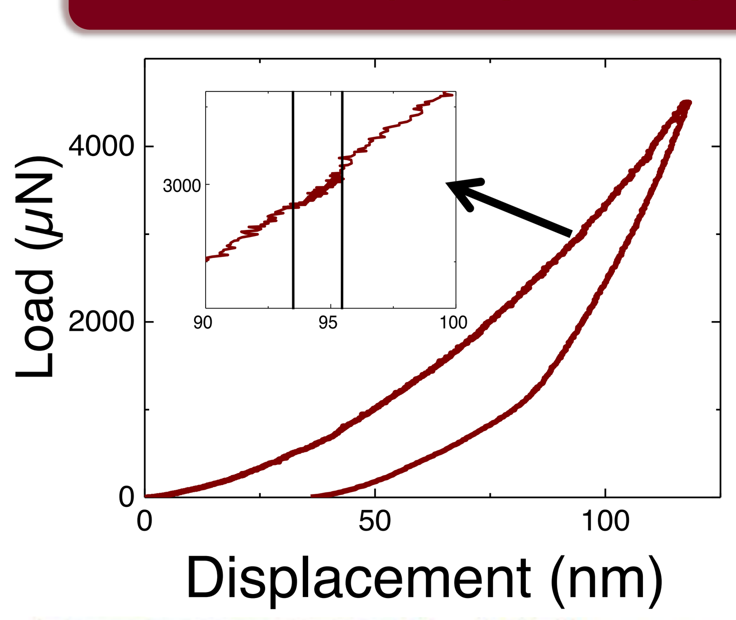
Material Preparation: Wafers of n-doped Silicon and arc melted Nickel were used. Micropillars were produced using a Focused Ion Beam (FEI Quanta 200 3D) using 30 kV and various beam currents. Pillar height to diameter ratio was 3:1 to 4:1. Si samples were cleaned with a HF and HCl solution.



Testing: All testing was completed on a Hysitron Triboindenter using a Berkovich tip for nanoindentation and a 5μm conical tip for micropillar compression. SRJ tests used multiple P/P's, loading rates and loads, for nanoindentation, and h's, displacement rates, for micropillars.

Hydrogen Charging: Samples were precharged for 10 days in high pressure hydrogen gas at 673 K.

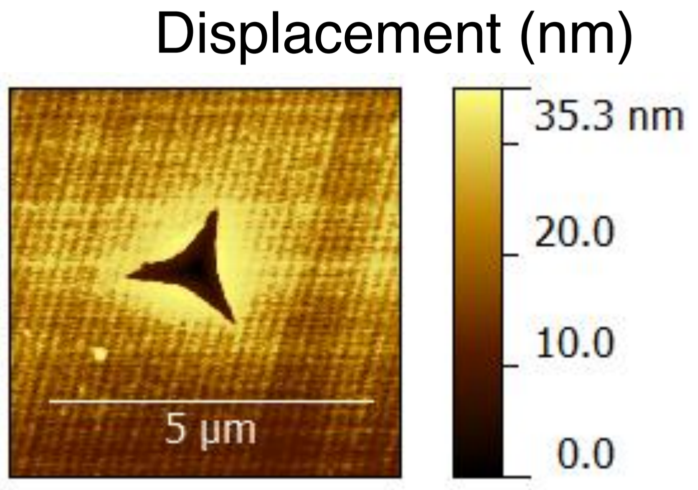
Nanoindentation - Silicon

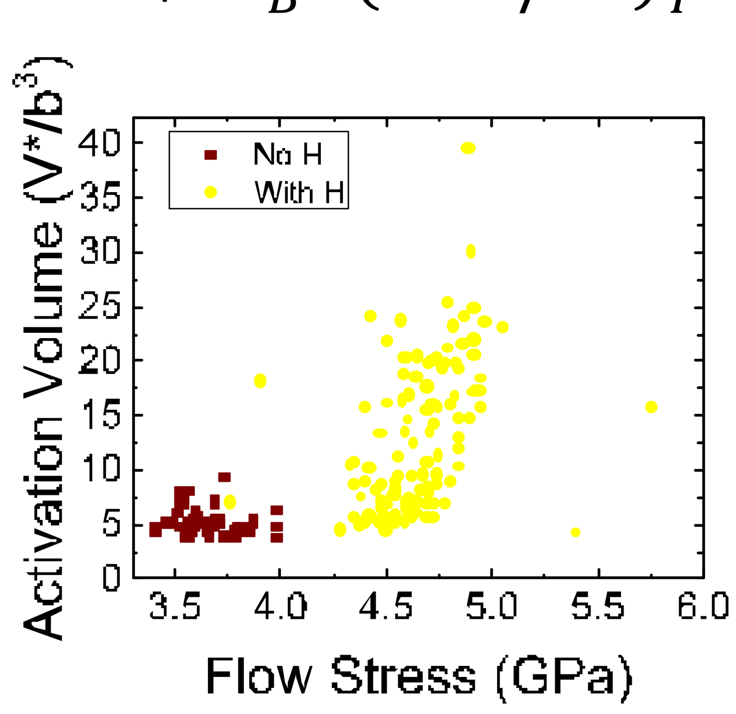


$$m_{indentation} = (\partial H/\partial ln\dot{\varepsilon})_T$$

$$H \cong 2.4 * \sigma_{flow}$$

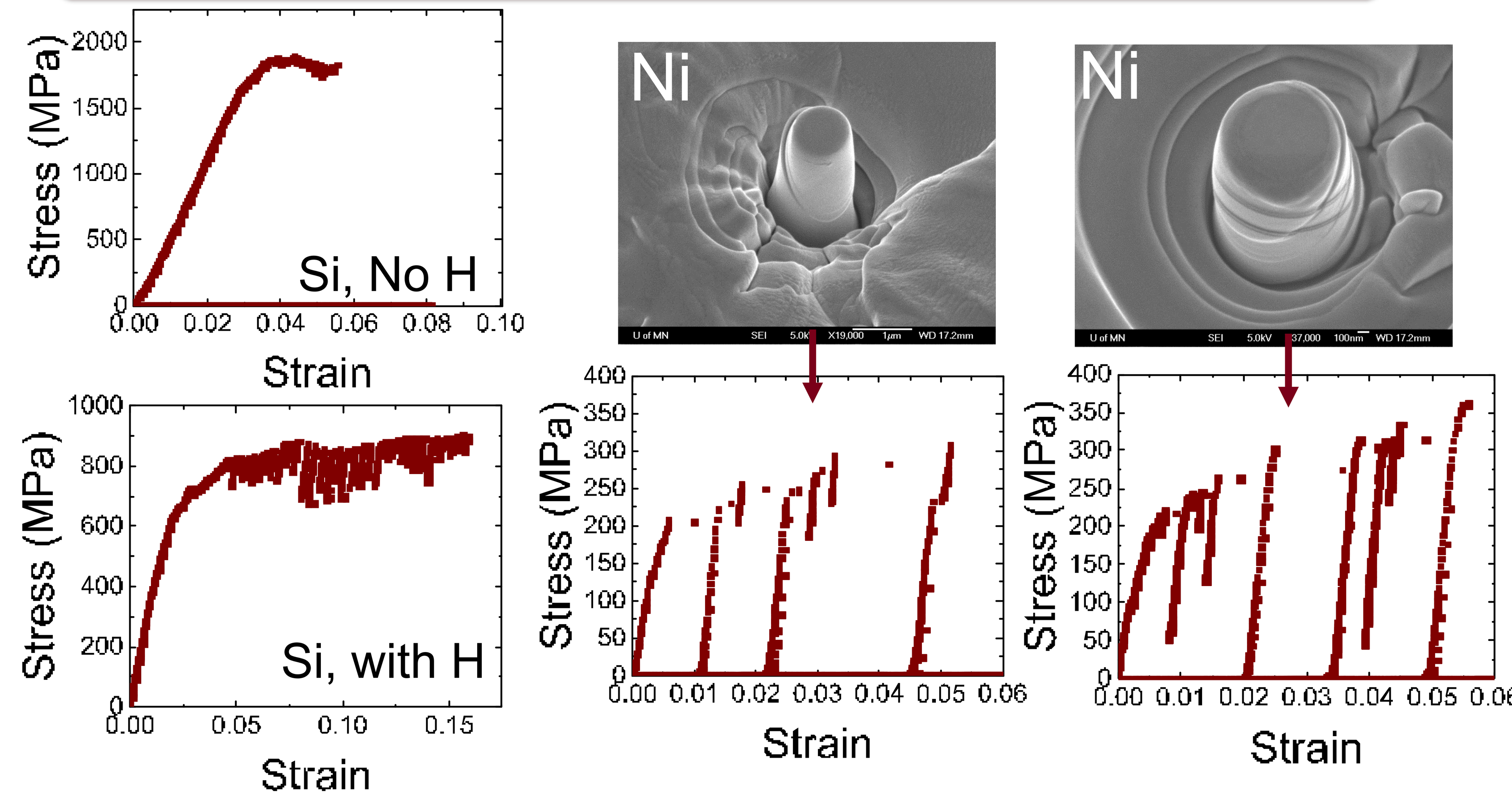
$$V^* = \sqrt{3}k_B T(\partial ln\dot{\varepsilon}/\partial\sigma)_T$$





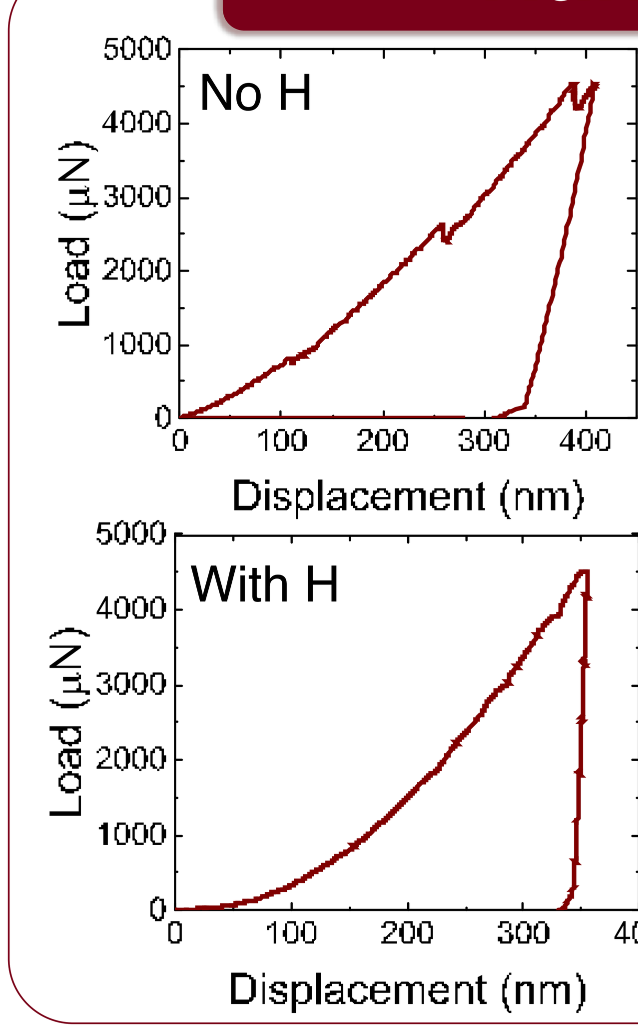
- Activation volume for [100] Si without hydrogen: V*= 5.3 ± 1.2 b^3 assuming full dislocations (b = 0.38 nm) over 50 tests
- With hydrogen additions, flow stress increases and activation volume also generally increases indicating an increase in dislocation velocity, material toughness, and crack tip shielding

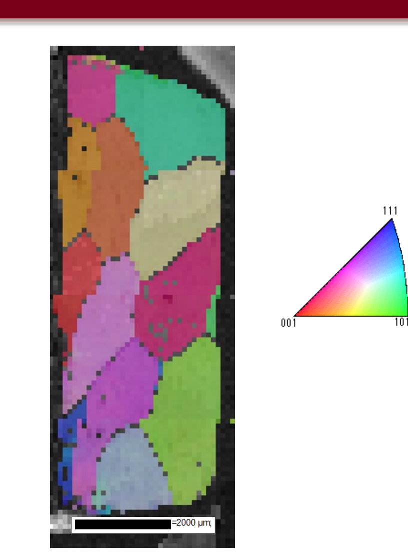
Micropillar Compression

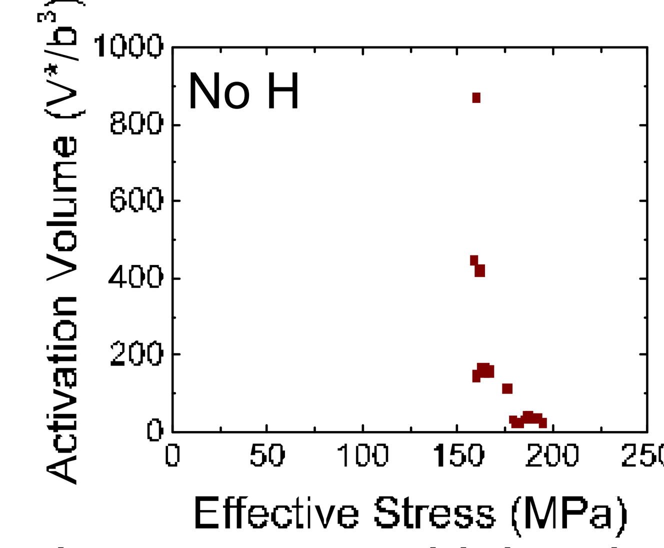


- Normal compression tests on Si showed an increase in plasticity with the addition of H. Stresses seem abnormally low and additional experiments are needed.
- Catastrophic failure occurred in many tests with/without H in Si.
- SRJ tests in Ni could not be resolved with the load drops due to dislocation bursts.
- Pillars were sent out for H-charging, but did not survive shipping

Nanoindentation - Nickel







- An obvious strain rate sensitivity is shown in non-H charged samples, but decreases to almost no effect with H.
- Similar grain orientations showed an apparent increase in elastic modulus with H charging.

Conclusions

- Activation volume for n-type Si generally increased for nanoindentation with the addition of H. Effective stresses also increased with H charging, indicating increased dislocation velocities and increased toughness in the material.
- Nickel nanoindentation shows little to no strain rate sensitivity with the addition of hydrogen even in pre-strained samples unlike uncharged counterparts.
- Nanopillar compression tests need further refinement but should be able to assess if dislocation interactions could increase the potential back stress.

Acknowledgements

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