Multi-Resolution Characterization of Hydrogen-Assisted Intergranular Fracture of Ni-201

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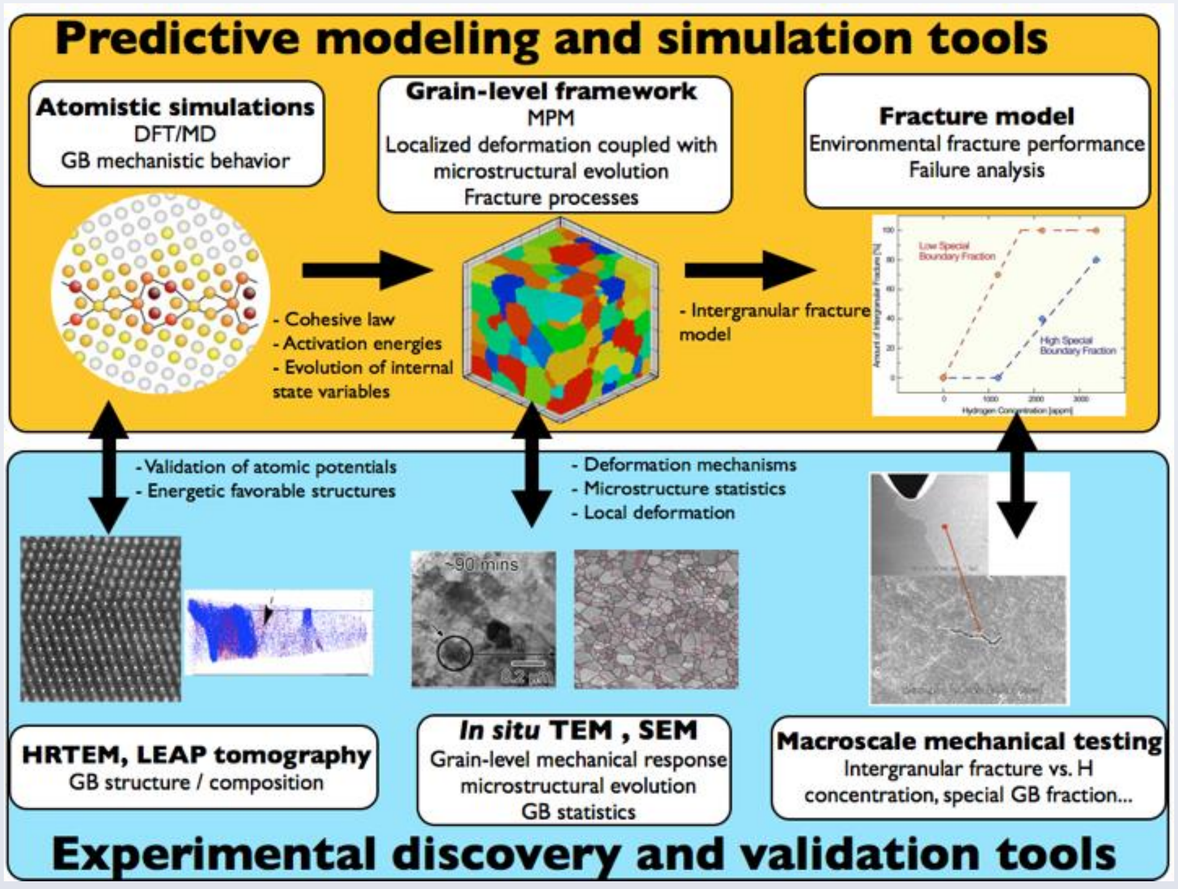
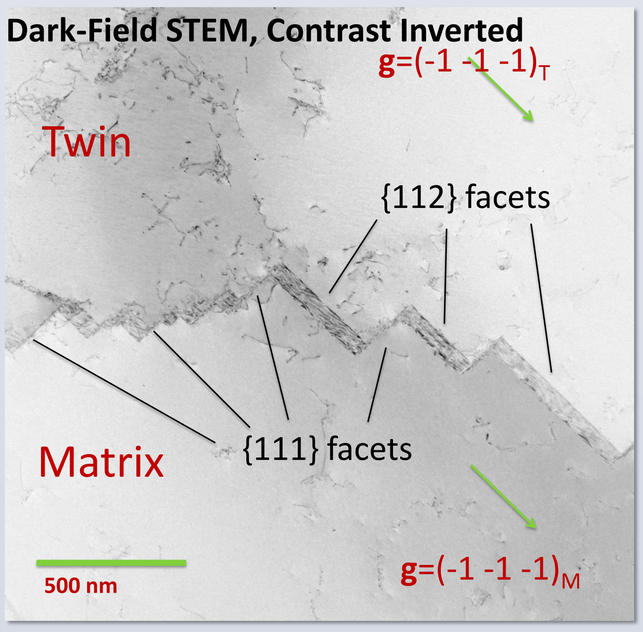
ABSTRACT

Subtle changes to the local chemistry and grain boundary structure can have dramatic consequences on material performance [1-3]. Some lots of as-received commercially pure Ni-201 exhibit brittle intergranular fracture when charged with hydrogen. After grain-boundary engineering (thermo-mechanical treatments that increase the fraction of twin and other low-sigma "special" boundaries), a more ductile fracture is observed [2]. We have assessed the chemistry of different boundaries (in terms of segregated hydrogen and various impurity elements) using atom-probe tomography, the structure of the boundaries using scanning transmission electron microscopy (TEM), and the deformation behavior using both *in situ* TEM and engineering-scale mechanical testing. This experimental work is presented in the context of atomistic and mesoscale simulations of the structure [1-3,5,6,7], chemistry, and cohesive strength of the boundary and detail the building blocks that are needed for a multi-scale, multi-physics predictive model for environmentally-assisted fracture.

A PREDICTIVE MULTISCALE FRAMEWORK FOR FRATURE [4]



TRANSMISSION ELECTRON MICROSCOPY

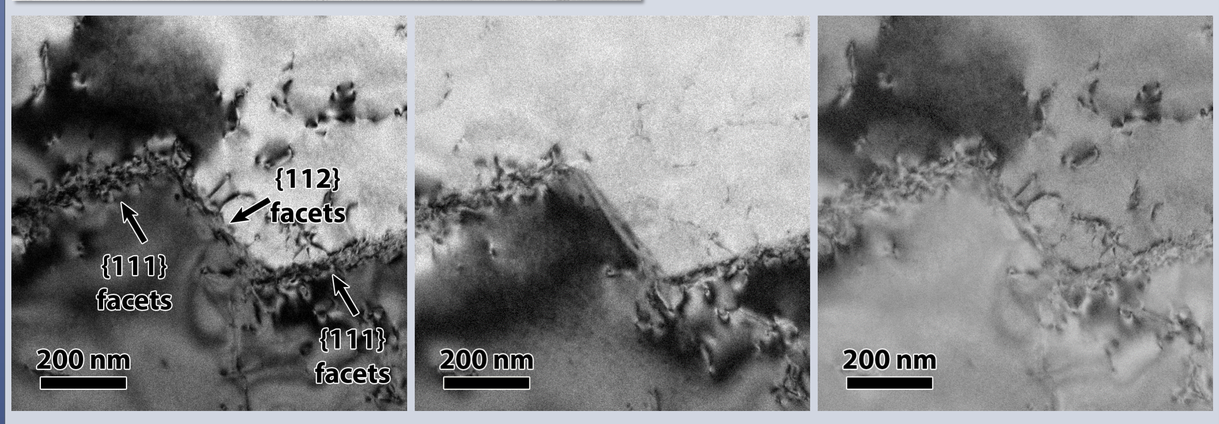


As-received Ni-201

- Hot rolled; 797 °C/7 min anneal
- Ca. 40% "special" grain boundaries
- Ca. 30 um grain size
- Charged to 4000 appm H (97 MPa/300 °C)
- Strained to 3.5%

Asymmetric inclination is accommodated by faceting on {111} coherent and near-{112} lateral twins. Misorientation is accommodated by interfacial dislocations.

Tilt-series DC-STEM and tomography allow these interfacial defects to be reconstructed in 3-D.



In situ H_2-charging of Ni-201 using 8 mbar H_2 at 500 °C .

The dislocation structure near the faceted boundary changes as H_2 is introduced.

Additionally, there may be some rounding of the {111}/{112} facet junction.

This change has been predicted by hybrid Molecular Dynamic/Grand Canonical Monte Carlo modeling [5] at higher concentrations of H than are expected here.

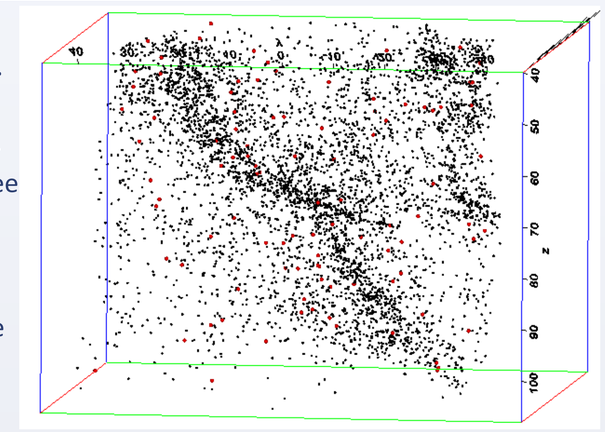
ATOM PROBE TOMOGRAPHY

Coherent twins show no discernible D segregation.

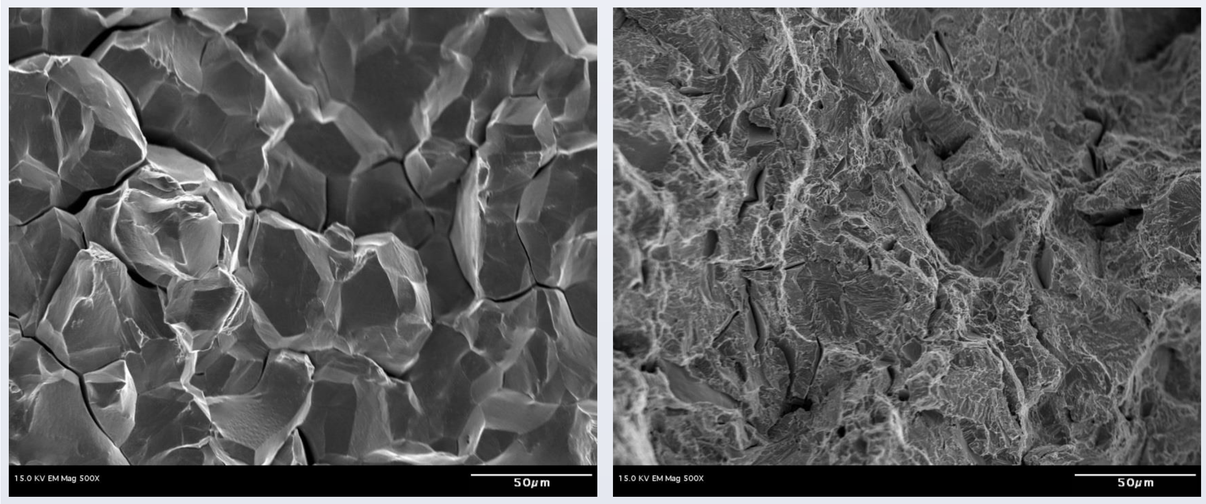
Higher volume twins do; here, 4 at.% D (an enrichment of over ten from the bulk) is observed.

Higher angle boundaries also show a greater degree of segregation of impurity elements (e.g. Pb, S) naturally present in Ni-201.

These impurities tend to lower grain boundary energy and may lower the cohesive strength of the boundaries.



MECHANICAL TESTING



The degree of environmentally-assisted intergranular fracture is reduced by grain-boundary engineering treatments that lead to a large fraction of low-sigma boundaries.

However, it also depends upon on trace quantities of impurity elements that tend to segregate to grain boundaries.

The two micrographs were from different heats of Ni, each charged to 5000 appm H.

They have a similar grain size (ca. 30 um) and fraction of "special" grain boundaries (ca. 40%).

However, a sample with 5.7 appm Pb (left, also highlighted in the TEM and APT micrographs) shows a greater degree of intergranular fracture than a sample with 0.05 appm Pb (right).

This is still well below what is permitted in the Ni-201 specification and is not great enough to lead to intergranular fracture of specimens tested in air.

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