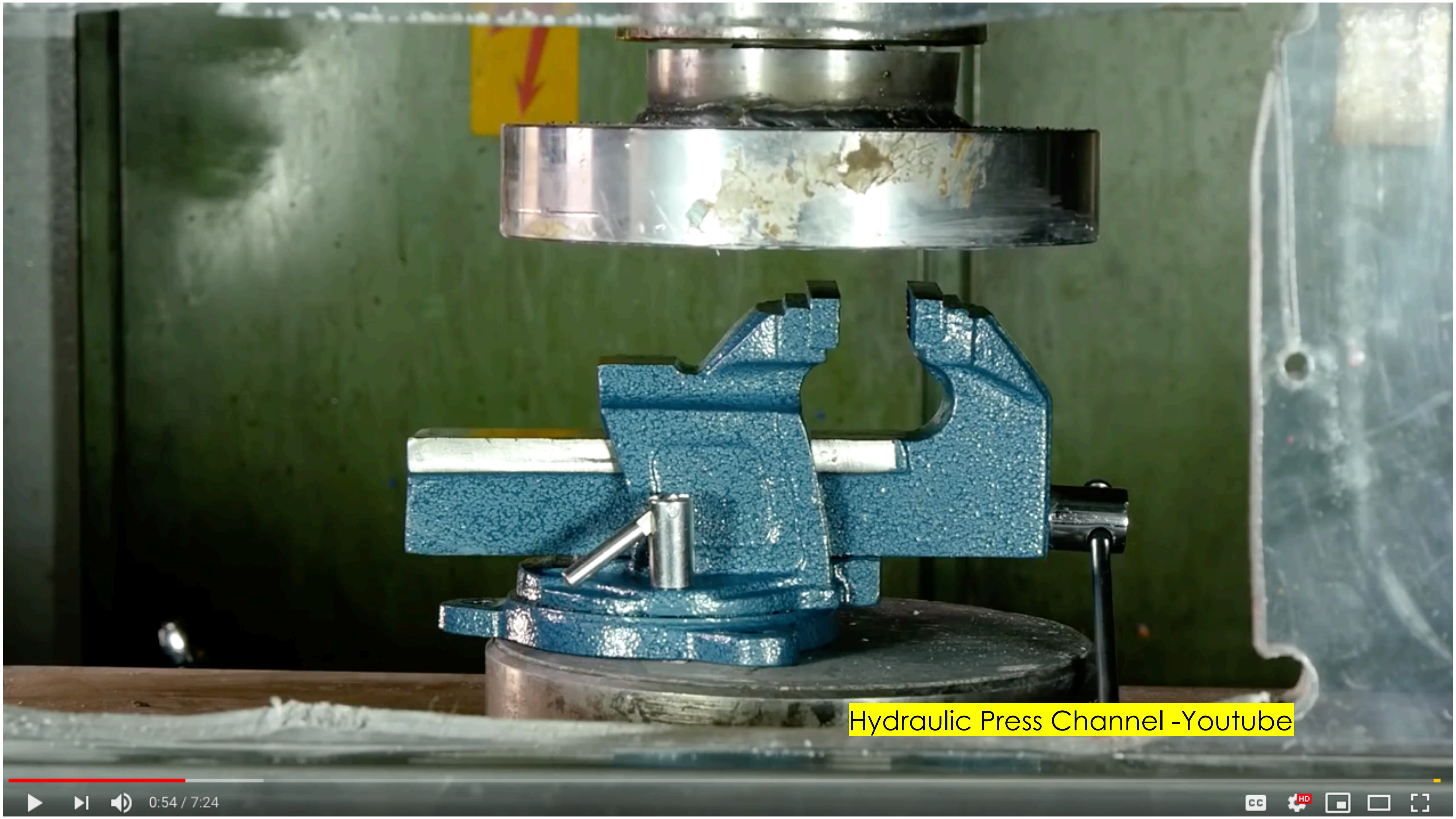
Materials under stress, what we learn by using neutrons?

Ke An

Spallation Neutron Source

***Acknowledgement**: The neutron work was done at the Spallation Neutron Source, a user facility sponsored by Office of Basic Energy Science, DoE. VULCAN TEAM, Students, Users*


Hydraulic Press Channel -Youtube
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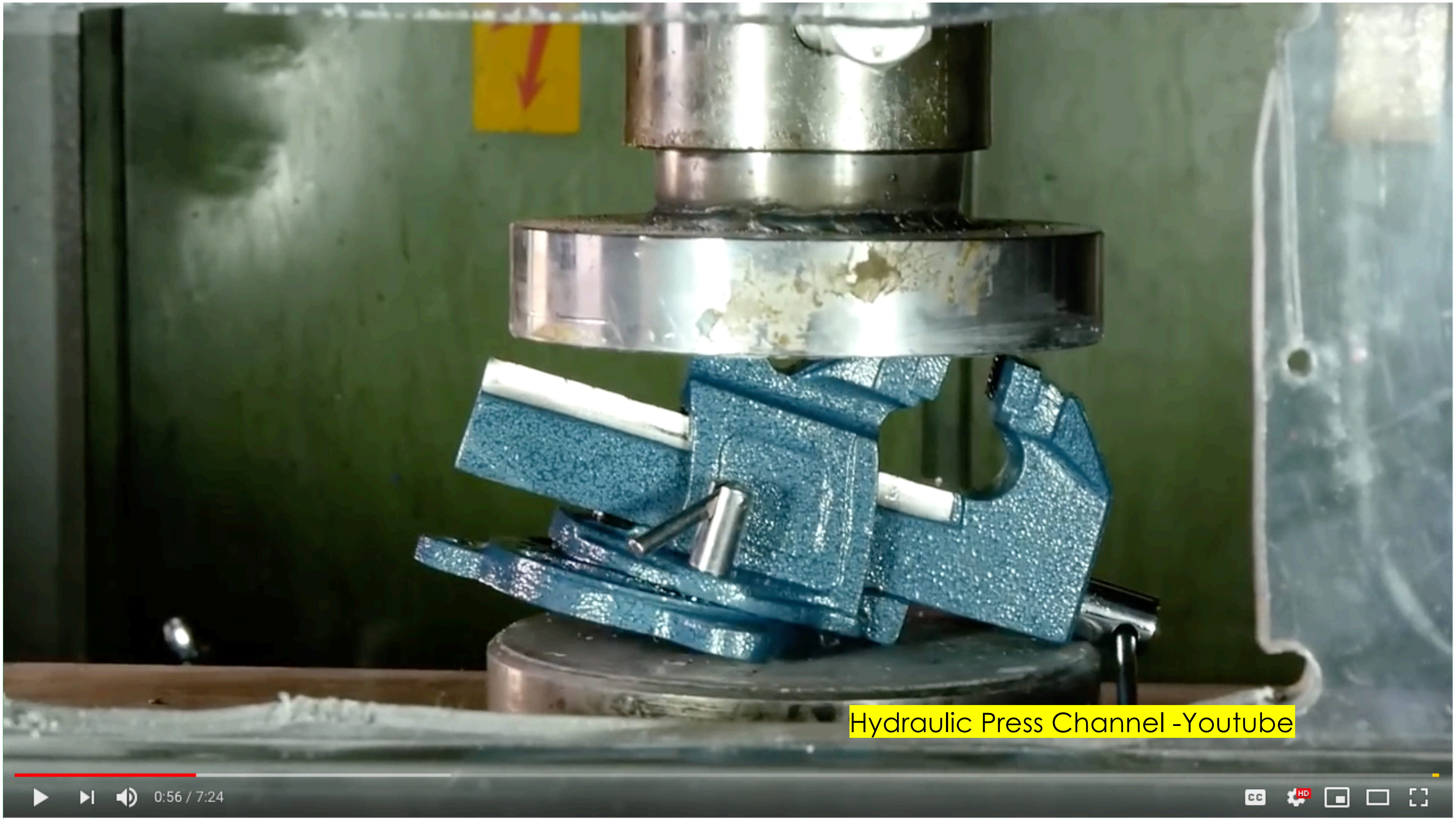
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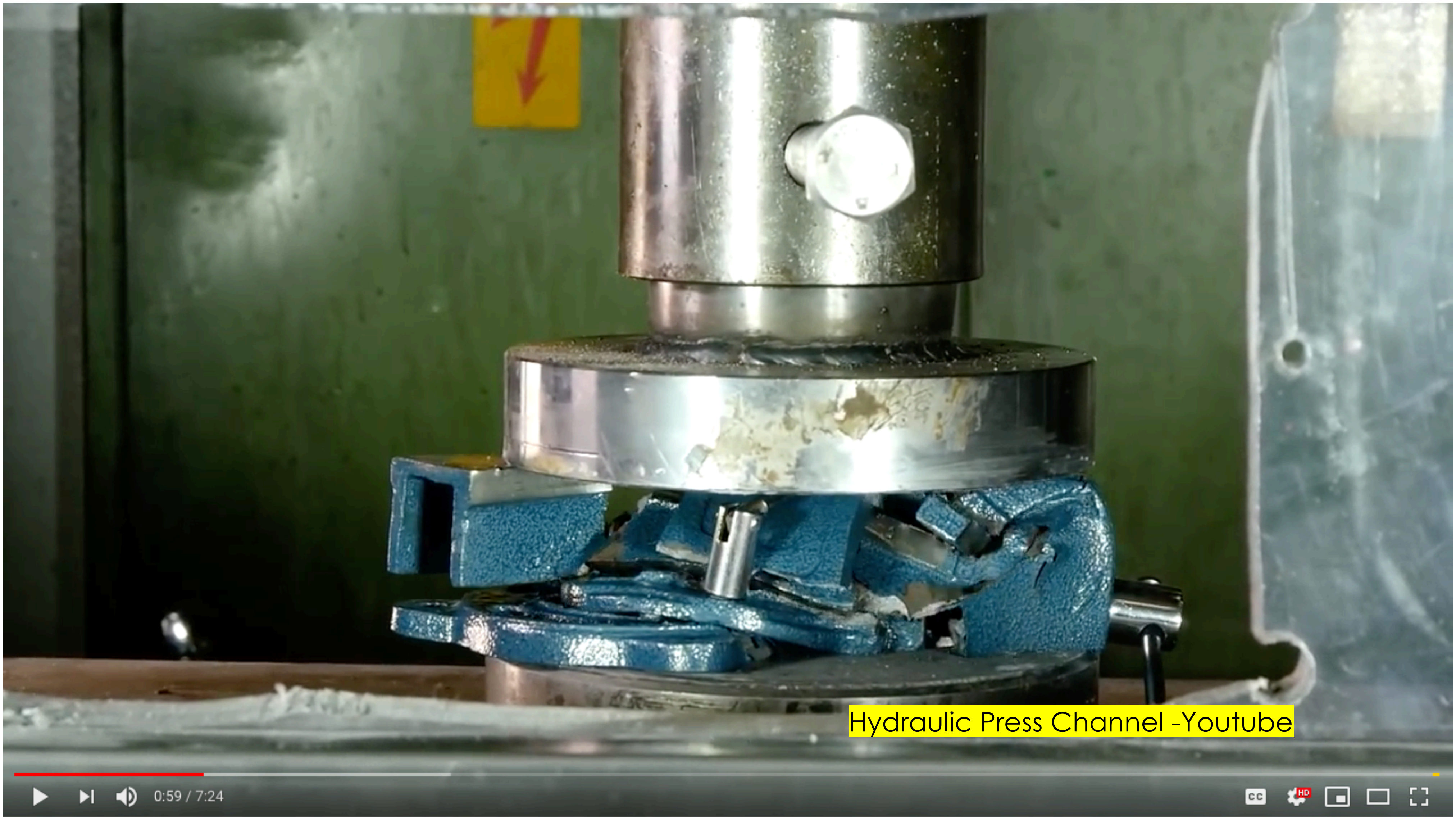
Hydraulic Press Channel -Youtube
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Deform to Comply

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Materials under stress

Use neutrons to learn how materials

Deform to Comply

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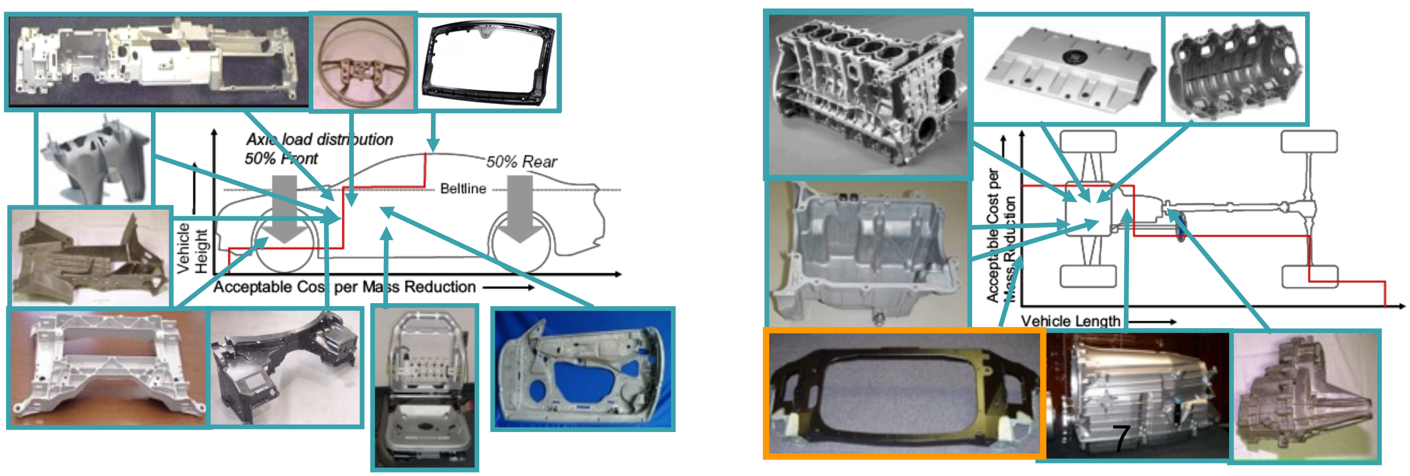
Why do we care?

- Understanding the deformation mechanism to make stronger or more flexible materials for structural, functional, and medical applications.



Magnesium (Mg) Alloy in automobile

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Materials under stress

Store elastic energy -> Dissipate energy -> Fracture

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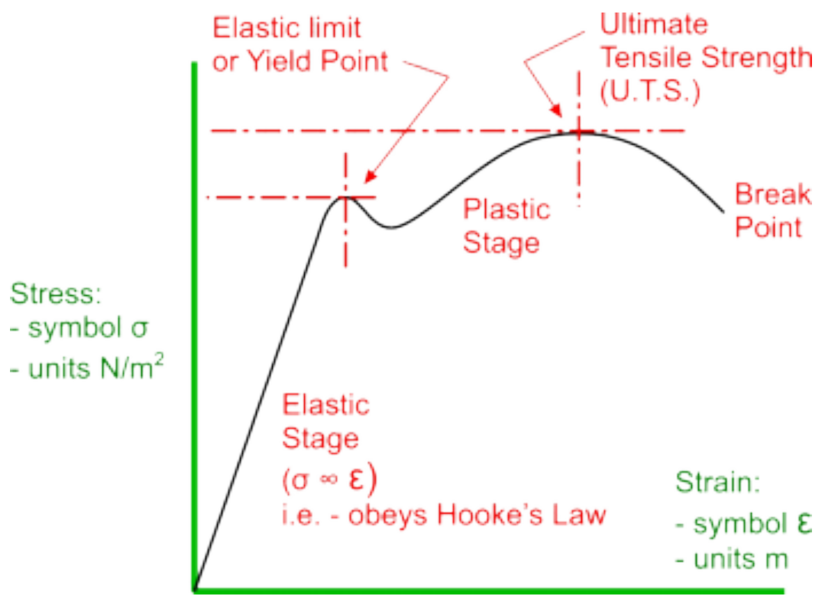
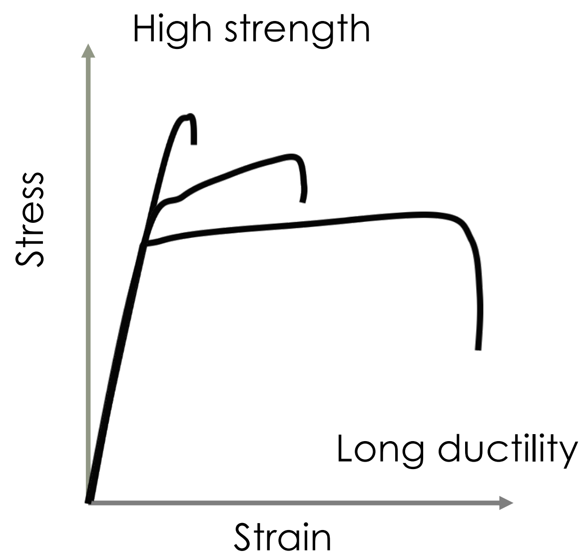
Materials under stress

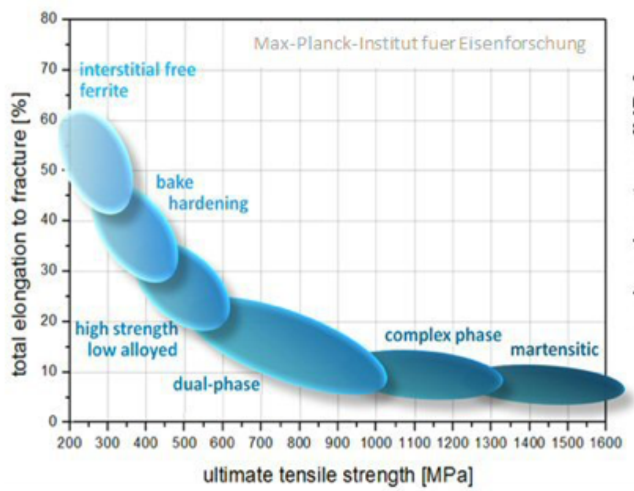
- Deform to comply by reversible elasticity, or irreversible plasticity before fracture.

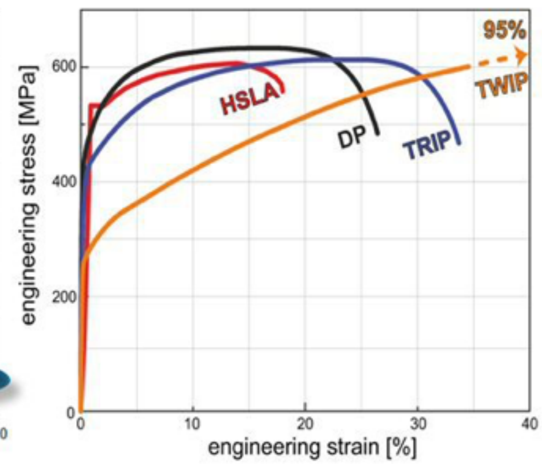


http://wiki.dtonline.org/index.php/Elasticity

Strength and ductility are commonly exclusive







http://www.dierk-raabe.com/steels-science/

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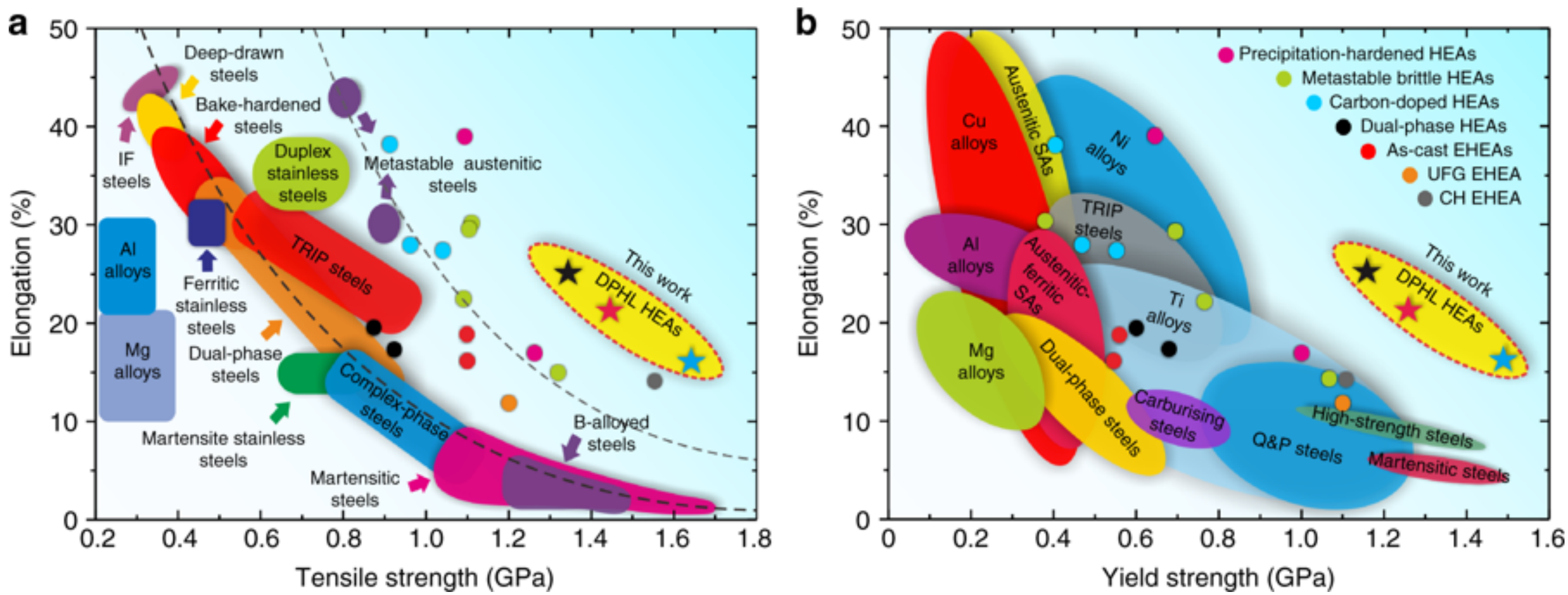
New alloys design pushes the envelope



Shi P. et al, Nature Communications **volume 10**, Article number: 489 (2019)

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Solid mechanics: physical, phenomenological and empirical approaches

- Experimental methods: Uniaxial, bi-axial, multiaxial loading; Proportional, nonproportional loading; Tension/compression/torsion/bending ;Monotonic, cyclic, random loading
- Parameters: Normal stress, shear stress, deviatoric stress(strain), hydrostatic stress(strain), principle stress (strain), equivalent stress(strain), hardening rate.
- Properties: Stiffness, yield strength, ductility, ultimate strength, hardness, fracture toughness, fatigue resistance, creep resistance
- Mechanics modeling: continuum mechanics, finite element analysis, life prediction

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Complexity of structural or functional materials responses under straining

- Hardening in structural materials.
- Shape memory effects, super elasticity.
- Piezoelectric effect, mechanical to electrical energy.
- Mechanocaloric effect, mechanical to thermal energy.
- Mechanically induced light emission.
- Stress induced magnetization, thermal and conductivity change materials.

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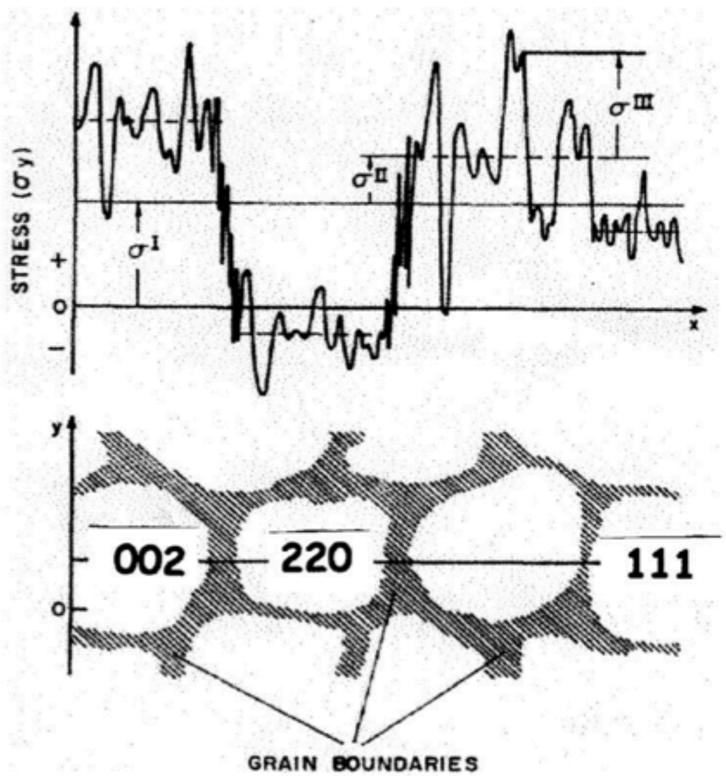
Different length scales, stress as an example



The macroscopic or type-1 stress is the average stress in a small region and has the same value in every grain.

The intergranular, grain-to-grain or type-2 stress is the deviation from the average stress in each grain. It varies between different grain groups due to anisotropic slip and elastic responses.

Type 3 stress varies inside grains around defects and near boundaries

Curtesy: T. Holden

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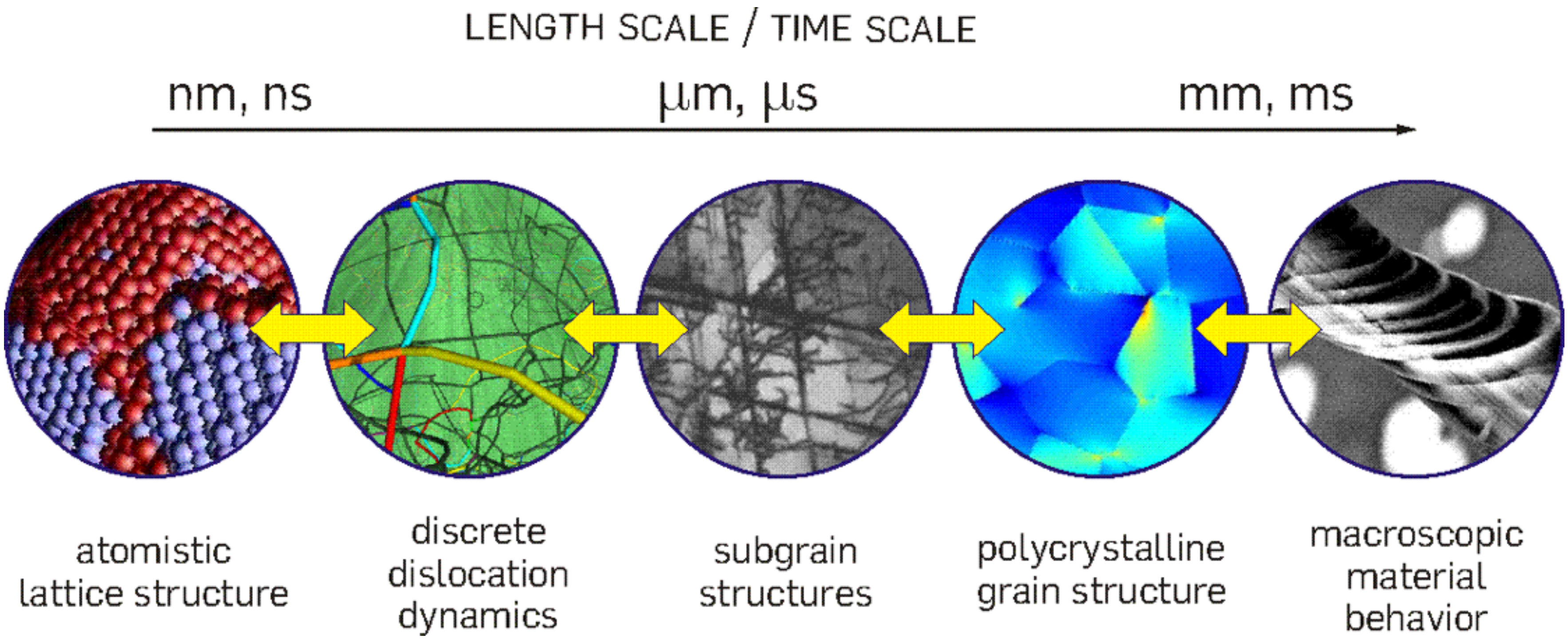
Elastic or plastic changes in different length and time scales



https://www.mm.ethz.ch/teaching.html

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Crystallographic understanding of materials under stress

Need tools

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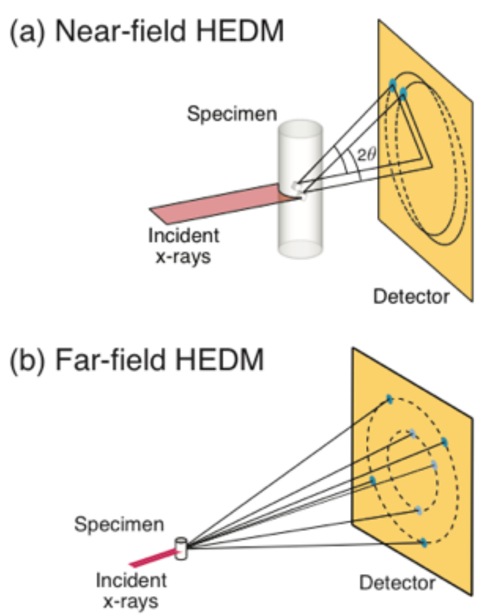
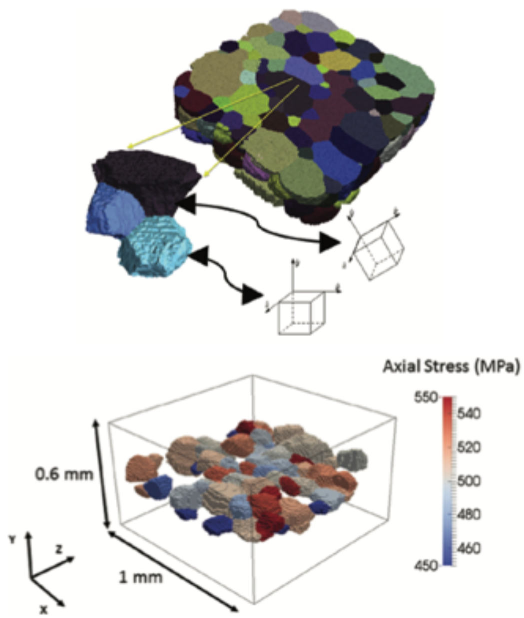
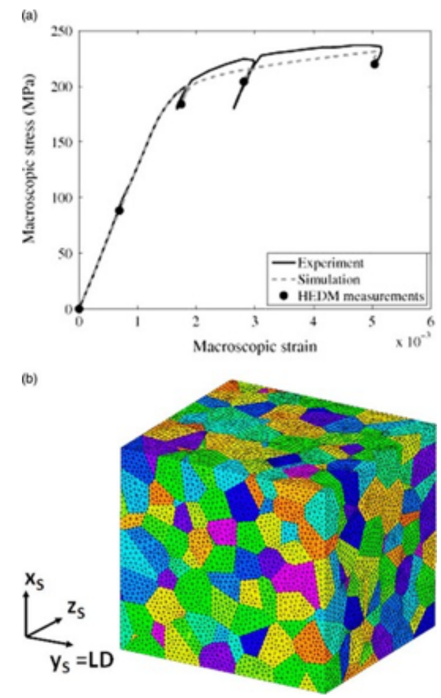
Tools for grain to meso scale





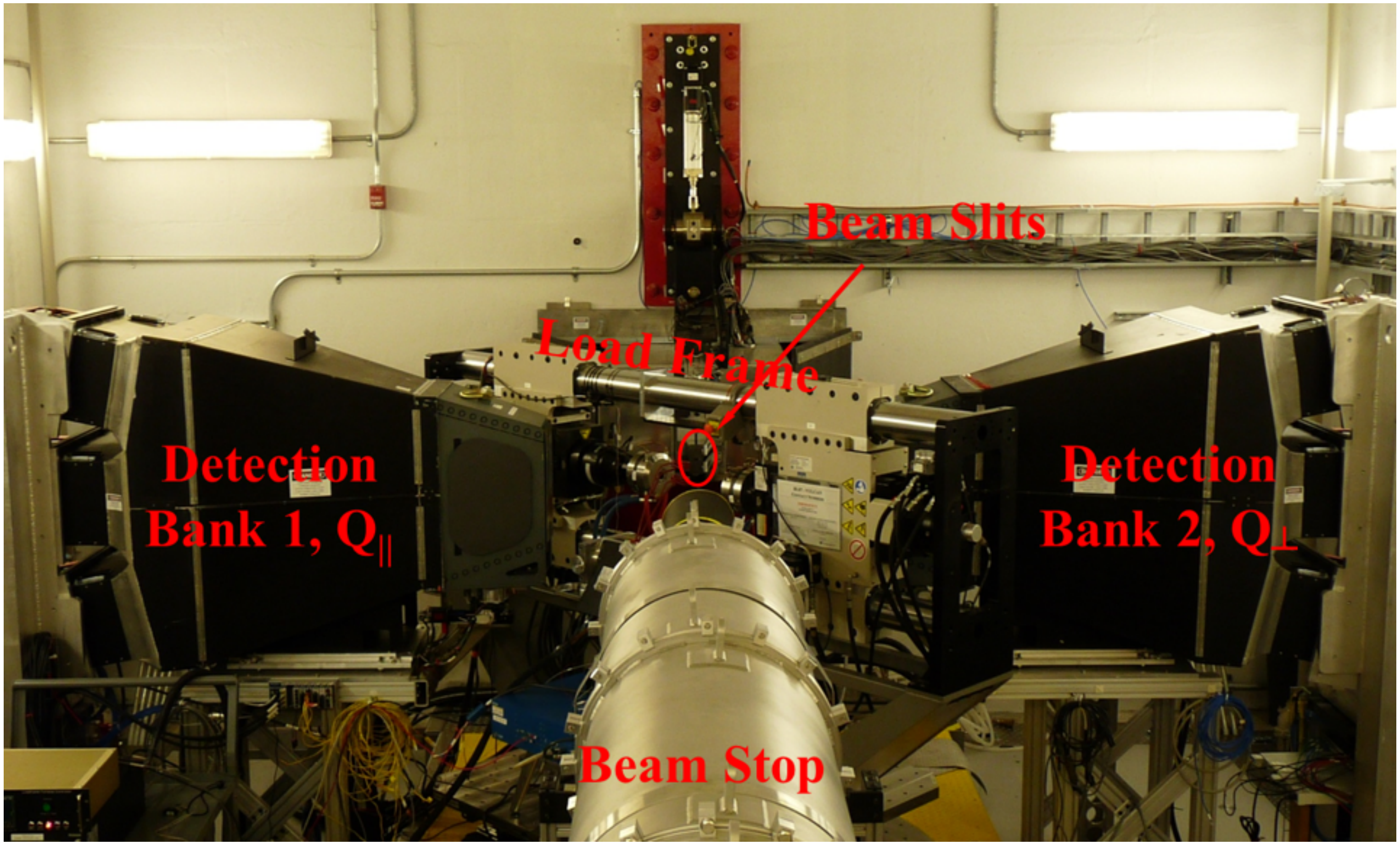


Synchrotron, far and near field

J.C. Schuren et al., Curr Opin Solid State Mater. Sci. 19, 235 (2015) and T.J. Turner et al., Integr. Mater. Manuf. Innov. 5, 235 (2016)

Microscopy Today, Volume 25, Issue 5 September 2017 , pp. 36-45

Neutrons



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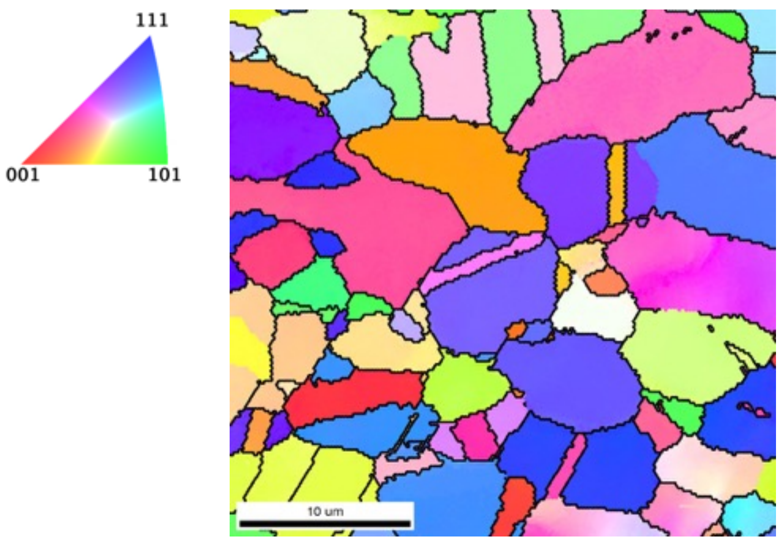
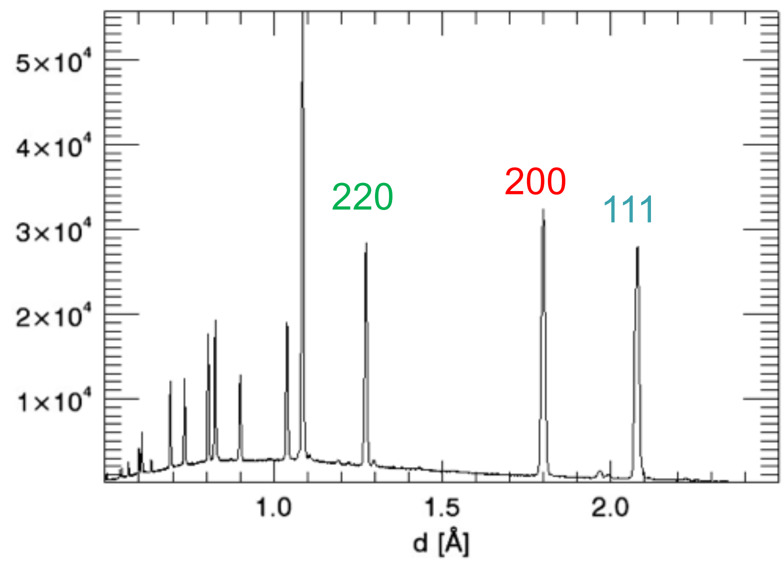
Diffraction approach to index grain level behaviors





- Diffraction lines measure the lattice of grains whose normals parallel to diffraction vector.
- hkl specific diffraction peaks thus reveal the corresponding lattice information including lattice strain, crystallographic orientations and defects.

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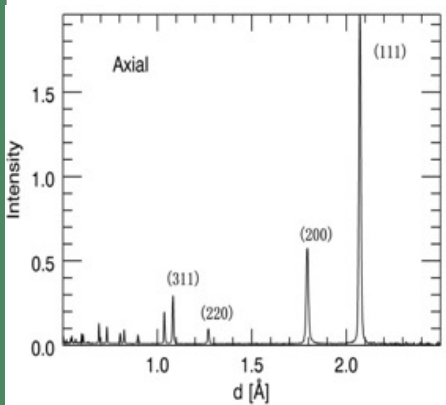
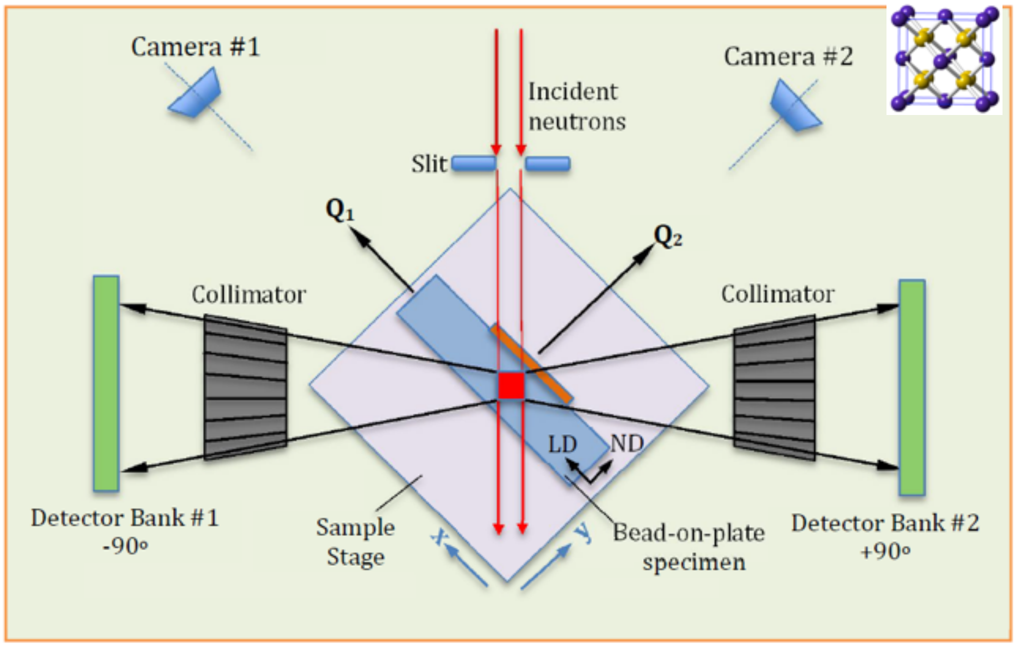
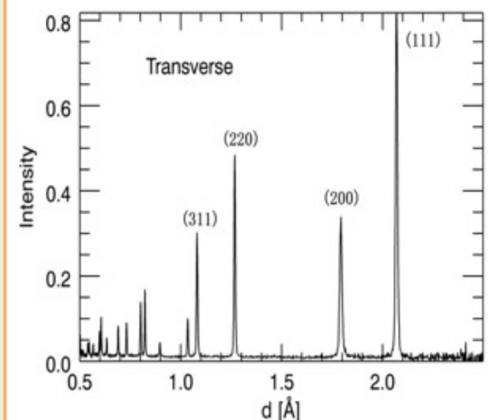
Engineering Neutron Diffraction Technologies







Schematic illustration of the bead-on-plate experimental set-up on engineering diffractometer like VULCAN at SNS (top view, not to scale). The -90° and +90° detector banks record diffraction peaks of the (h k l) lattice planes whose normals are parallel to Q_1 and Q_2, respectively. Strain components along these two directions are measured simultaneously. The bead-on-plate specimen is positioned on top of the sample stage and aligned at 45° from the incident beam.

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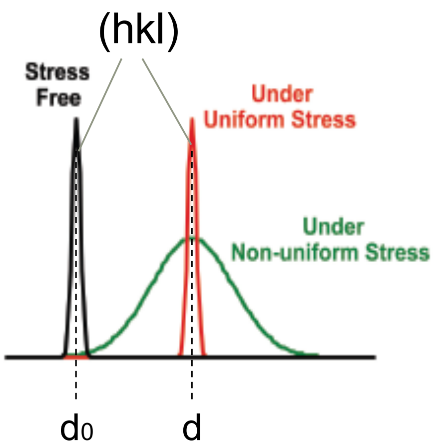
Lattice and stress measurement by diffraction

- Lattice strain measurement

$$\varepsilon^{hkl} = \frac{d_{hkl} - d^0_{hkl}}{d^0_{hkl}}$$

where d^0_{hkl} is the lattice spacing at zero applied stress.



- Stress tensor calculation

$$\sigma_{ij} = \frac{E^{hkl}}{(1+\nu^{hkl})}\left\{\varepsilon^{hkl}_{ij} + \frac{\nu^{hkl}}{1-2\nu^{hkl}}\left(\varepsilon^{hkl}_{11} + \varepsilon^{hkl}_{22} + \varepsilon^{hkl}_{33}\right)\right\}$$

where *i,j*=1,2,3 indicate the components relative to **three dimensional orthogonal axes**.
E^{hkl} and ν^{hkl} are [hkl] specific “diffraction elastic constants”.

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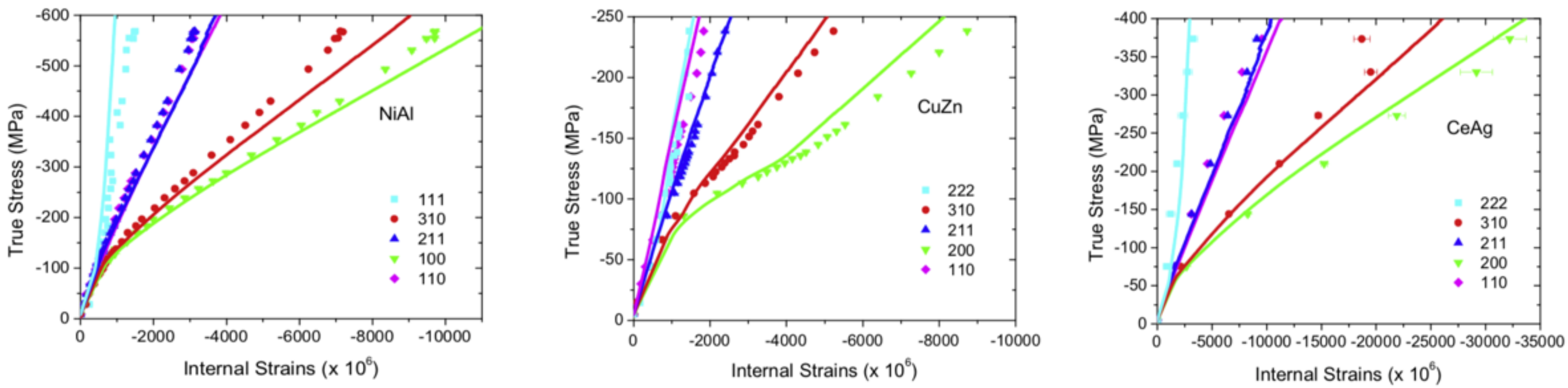
Lattice strain has a sensitivity of plastic deformation



Single crystal elastic constants and anisotropy factors of three B2 alloys

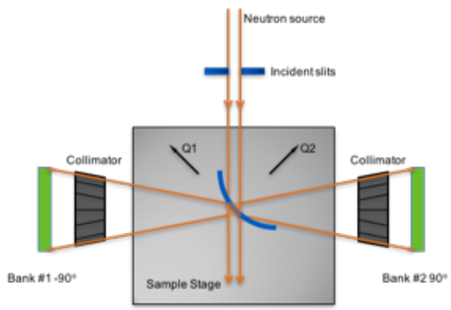
	C_{11} (GPa)	C_{12} (GPa)	C_{44} (GPa)	A
NiAl [1]	211.5	143.2	112.1	3.28
CuZn [32]	118.98	102.32	74.4	8.93
CeAg [36]	59.6	44.6	21.5	2.87

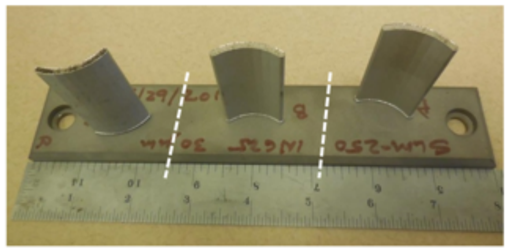
	Slip mode
NiAl	<100>{011}
CuZn	<111>{1$\bar{1}$0}
CeAg	<100>{011}

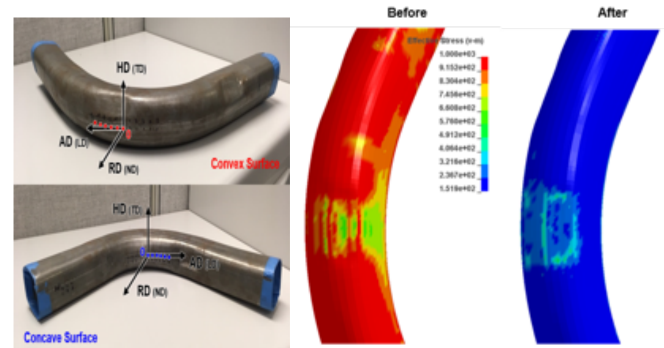
J.A. Wollmershauser et al. / Acta Materialia 57 (2009) 213–223

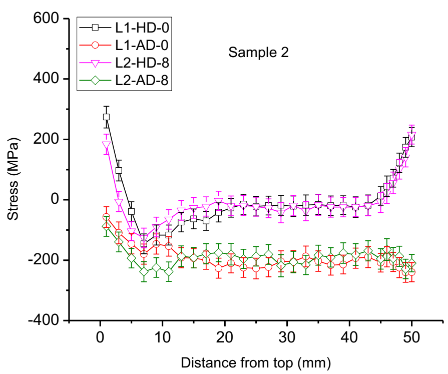
Stress causes cracks

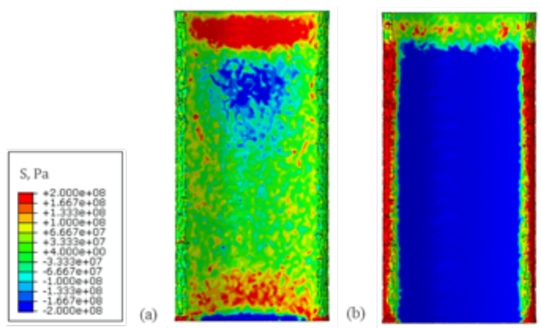
Residual stress in manufactured structures

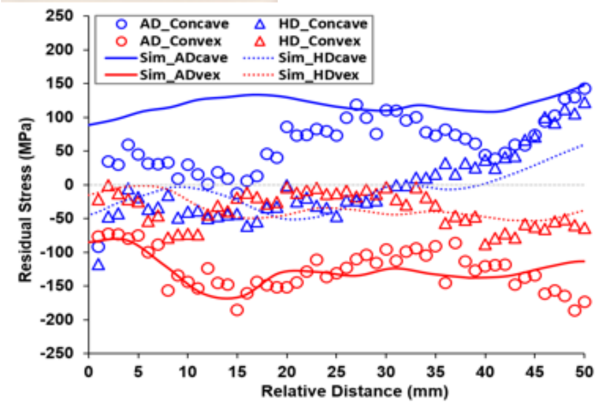








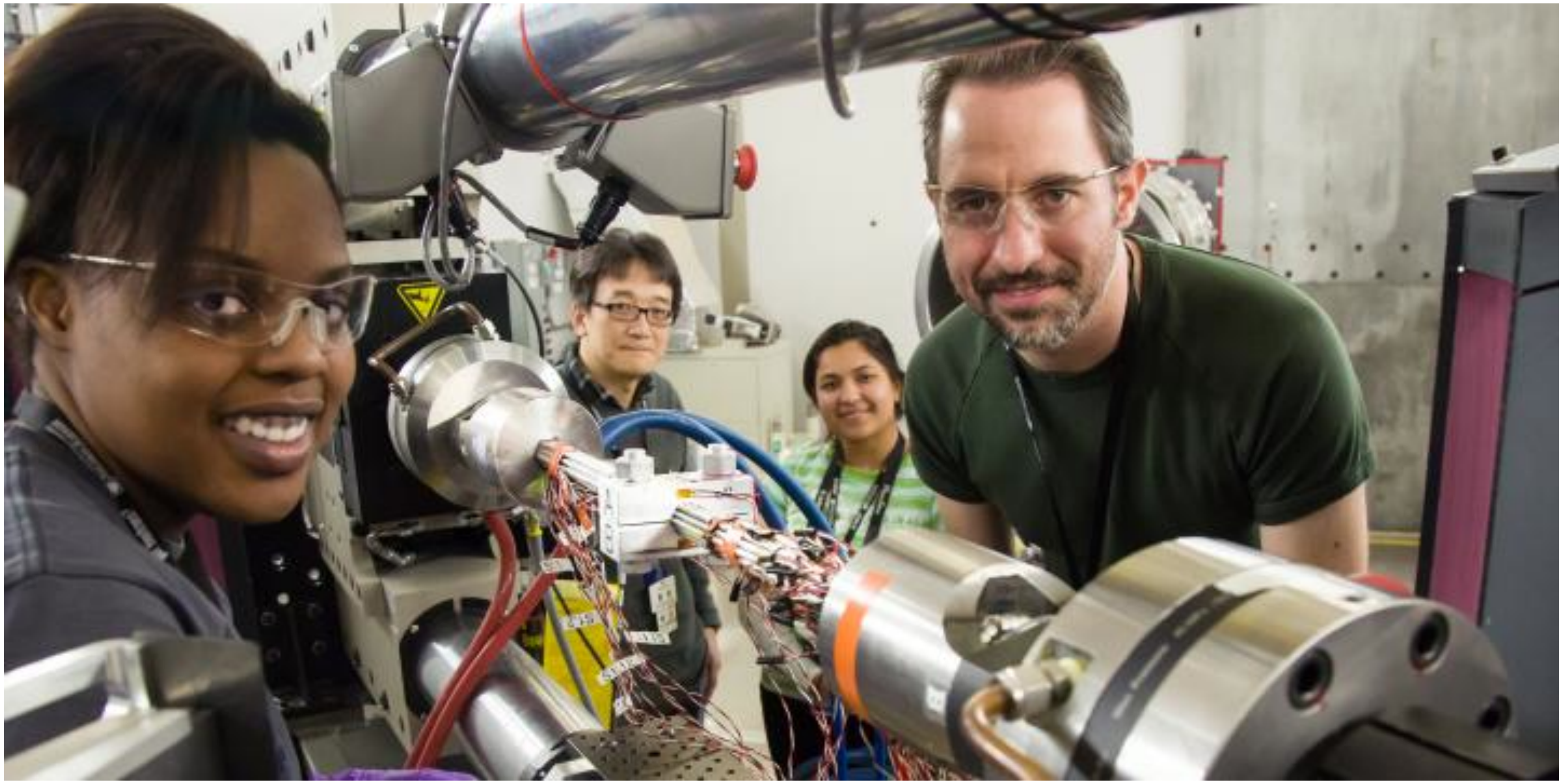




Ke An, et al. Materials & Design, 2018

L. Huang, Y. Chen, M. Chen, D. Yu and K. An, WCX™ 18: SAE World Congress Experience, SAE International, 2018

Suspension Bridge Cable Design



Columbia University Study Suspension Bridge Cable Design, 2015-. (For the story go to neutrons.ornl.gov)

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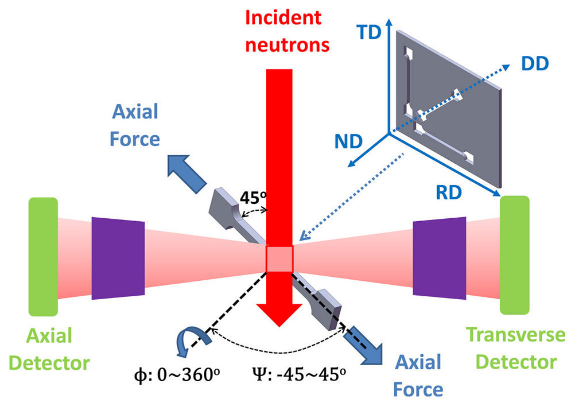
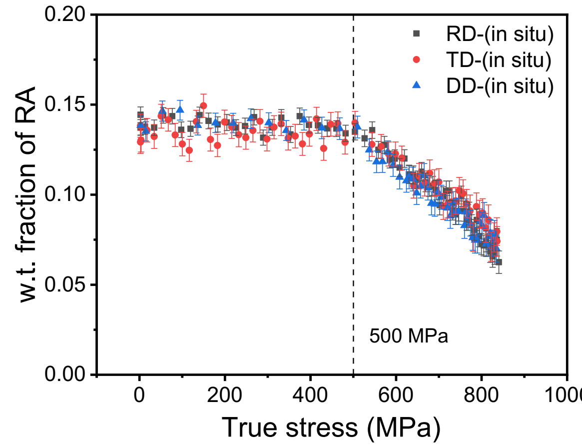
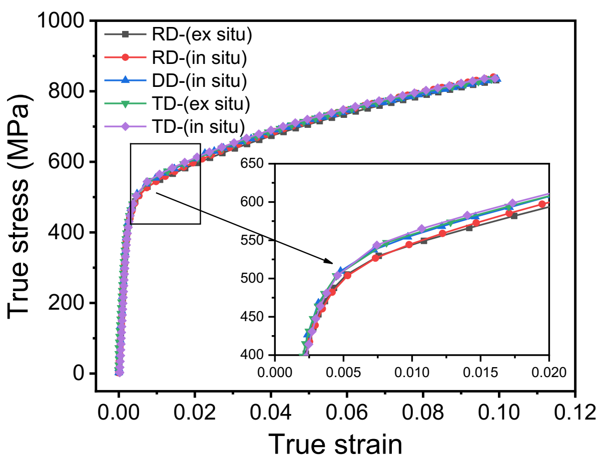
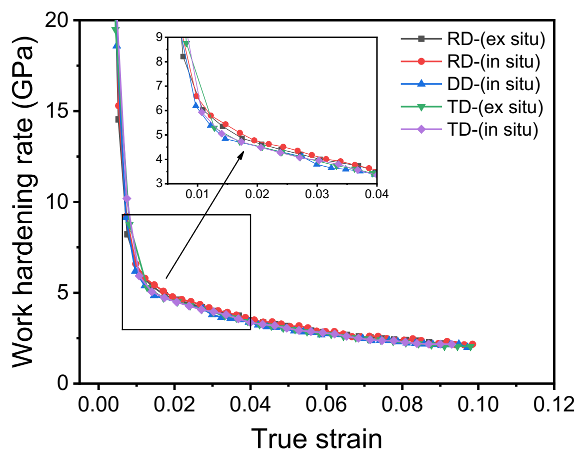
Advanced high strength steels

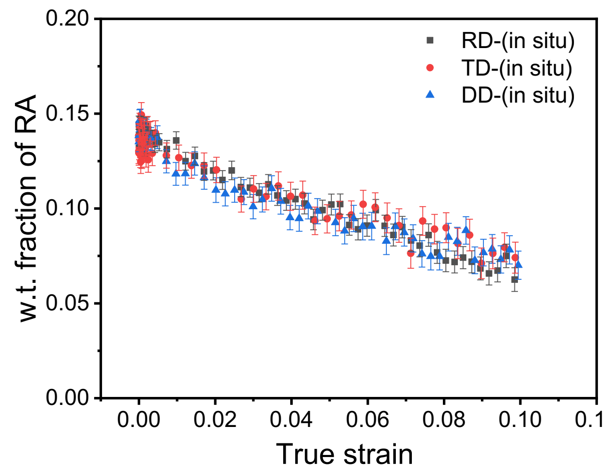
- In situ tension in RD, TD and DD
  - RD shows relatively lower yield stress and higher strain hardening
  - Phase transformation starts at ~500 MPa in all directions
  - No significant dependence on loading direction







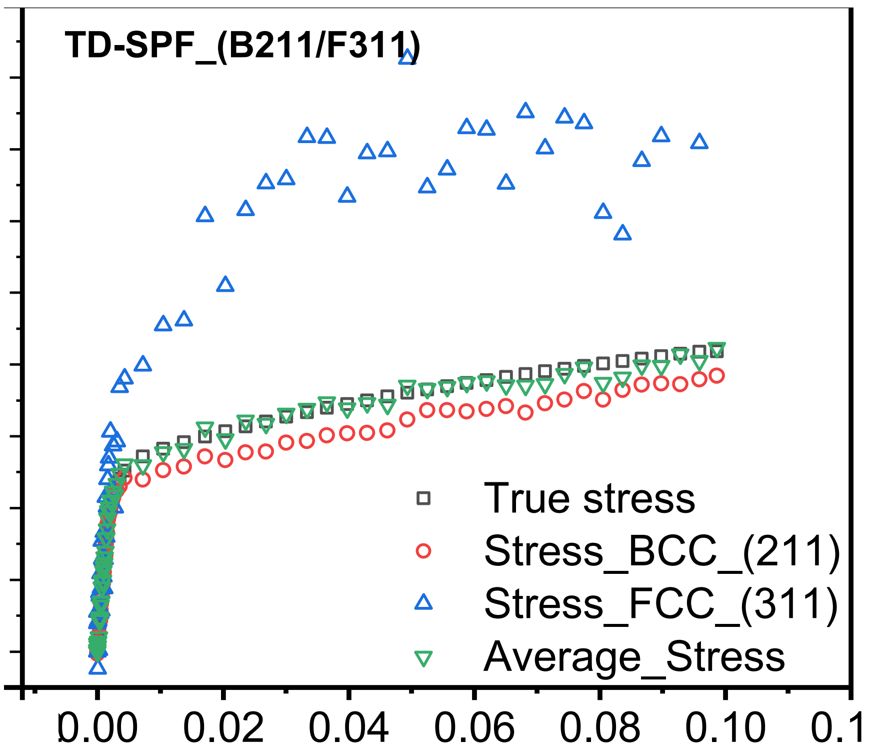




Yu D. et al JOM, 2018

Phase specific stress during deformation in AHSS

- **SPF indicated a good match of calculated averaged stress and macroscopic true stress.**



Yu D. et al JOM, 2018

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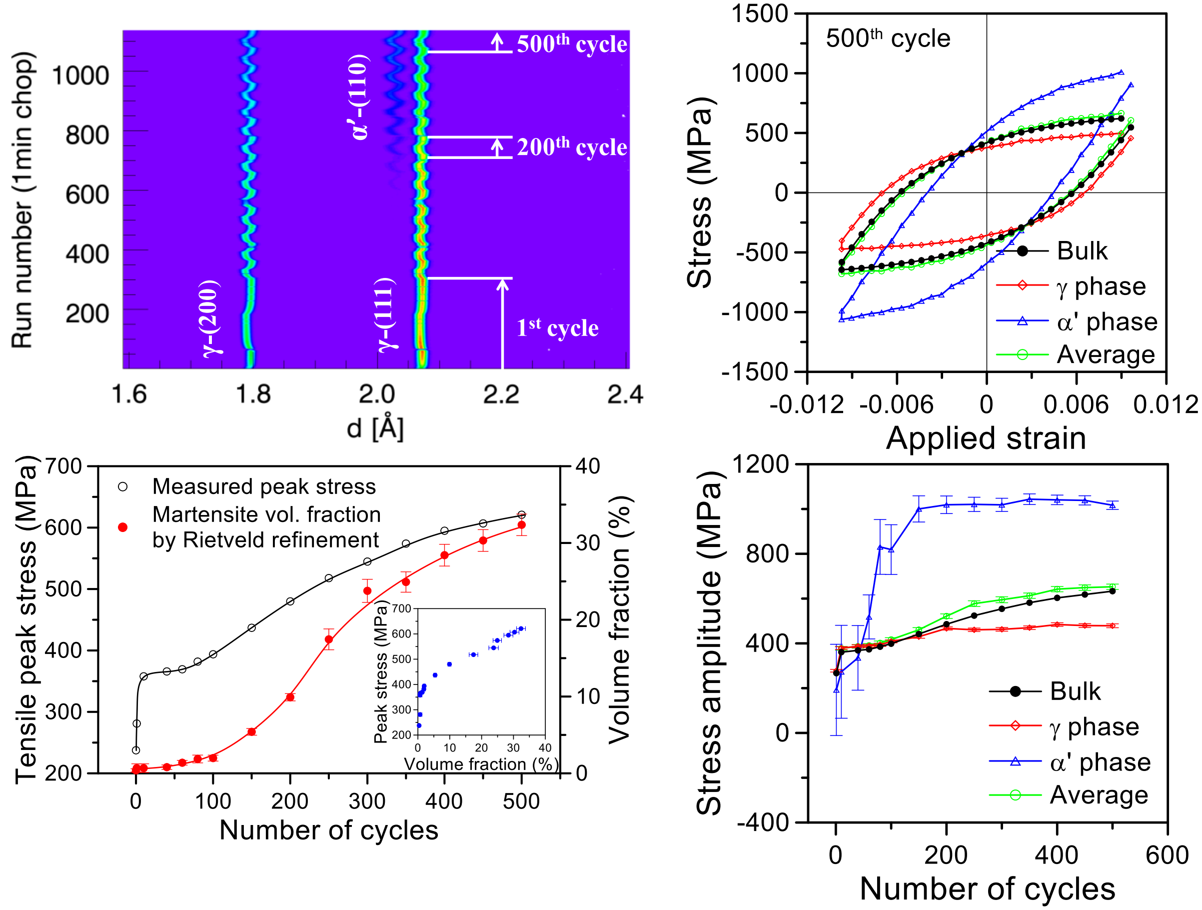
Cyclic hardening: neutron diffraction reveals cyclic hardening mechanisms for an austenitic stainless steel



D. Yu, K. An, Y. Chen, X. Chen, **Scripta Materialia, 2014**,

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Directional composite: Neutron diffraction reveals reinforcing mechanisms for a directionally solidified NiAl-Cr(Mo) eutectic composite

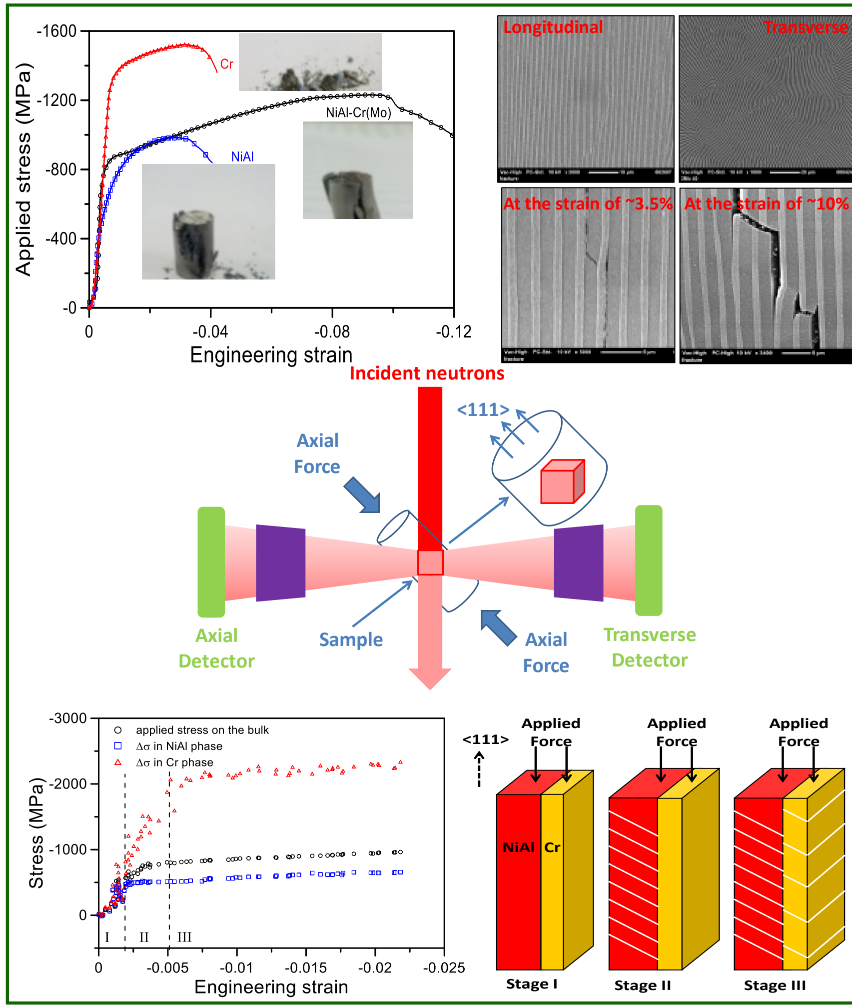
Scientific Achievement

The mechanism for the toughness enhancement in a directionally solidified NiAl-Cr(Mo) eutectic lamella composite is revealed by in-situ neutron diffraction. The Cr_{ss} layers with thickness of ~400 nm can bear very high stresses and deform plastically before fracture, unlike in bulk form, where it fractures due to little ductility.

Significance and Impact

The mechanical properties of the high temperature structural NiAl-Cr(Mo) can be increased with fine lamellae, thus allow the composite to possess higher toughness.

D. Yu, H. Bei, Y. Chen, E.P. George, K. An,, **Scripta Materialia, vol 84-85, pp 59-62, 2014**



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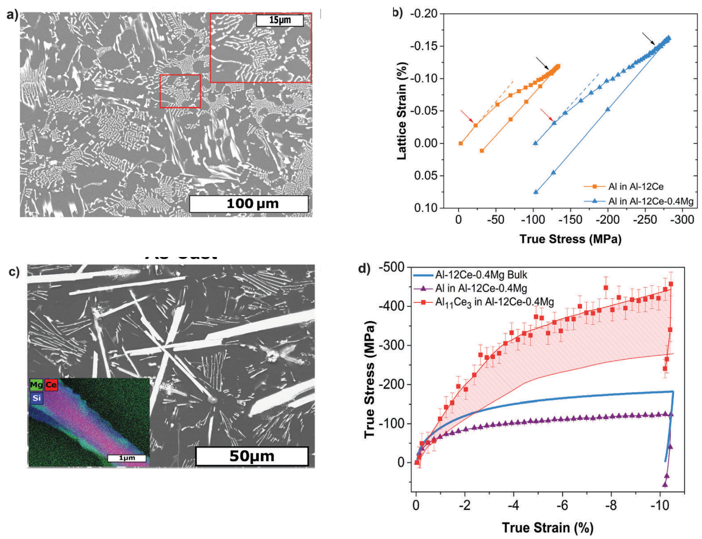
New alloy design: Neutron unravels deformation mechanism of high performance aluminum–cerium alloys for high-temperature applications



a) SEM of Al-Ce, b) The Al lattice strain behavior of Al-Ce, Al-Ce-Mg, c) SEM of Al-Cel-Mg, d) load sharing of Al and precipitates in the two alloys during different stages.

This research was sponsored by the Critical Materials Institute, an Energy Innovation Hub funded by the U.S. Department of Energy (DOE), Office of Energy Efficiency and Renewable Energy, Advanced Manufacturing Office and Eck Industries. Neutron experiments were performed at VULCAN, ORNL, which are sponsored by Scientific User Facilities Division, BES, DOE.

Scientific Achievement

Alloying aluminum with cerium creates a highly castable alloy, that exhibits dramatically improved high-temperature performance. Neutron diffraction under load provides insight into the unusual mechanisms driving the mechanical strength.

Significance and Impact

- Light-weight high-temperature alloys are important to the transportation industry where weight, cost, and operating temperature are major factors in the design of energy efficient vehicles. Aluminum Cerium alloys with high-temperature mechanical performance could fill this gap economically.

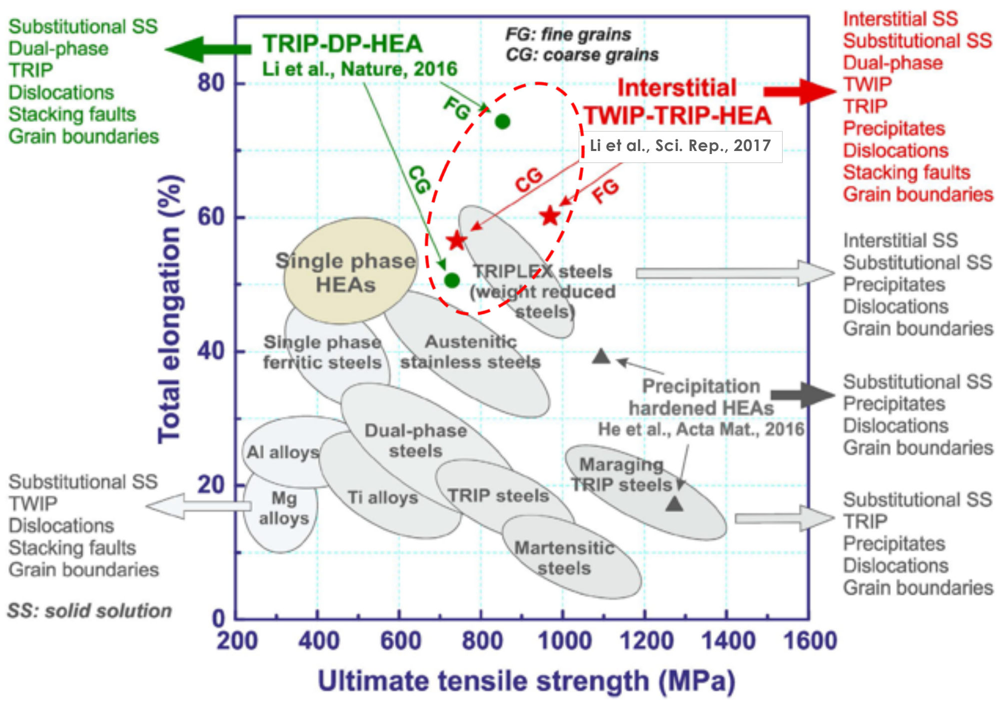
Research Details

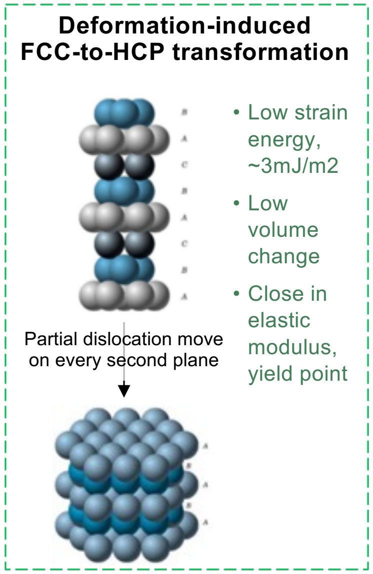
- AlCe and AlCeMg alloys are casted and these compositions display a room temperature ultimate tensile strength of 400 MPa and yield strength of 320 MPa, with 80% mechanical property retention at 240 8C.
- In-situ neutron diffraction unraveled the loadsharing of Al matrix and the hardening precipitates at different loading stages.

Published in: Z. C. Sims, O. Rios, D. Weiss, P. E. A. Turchi, A. Perron, J. R. I. Lee, T. T. Li, J. A. Hammons, M. Bagge-Hansen, T. M. Willey, K. An, Y. Chen, A. H. King and S. K. McCall. Mater. Horiz., 2017, 4, 1070 , DOI: 10.1039/c7mh00391a

Next generation alloys: high entropy alloys

TRIP-HEAs provide a distinguished metastability engineering strategy to achieve an excellent strength-ductility combination for structural materials.





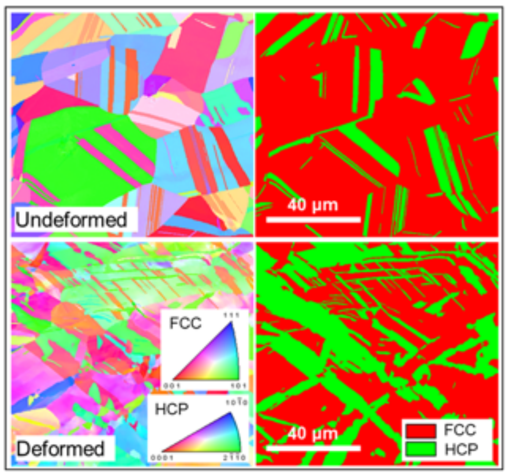
Figures cited from
https://www.slideshare.net/rpclemson/module2-71196024

Z. Li, C.C. Tasan, H. Springer, B. Gault, D. Raabe, Interstitial atoms enable joint twinning and transformation induced plasticity in strong and ductile high-entropy alloys, Scientific reports 7 (2017) 40704

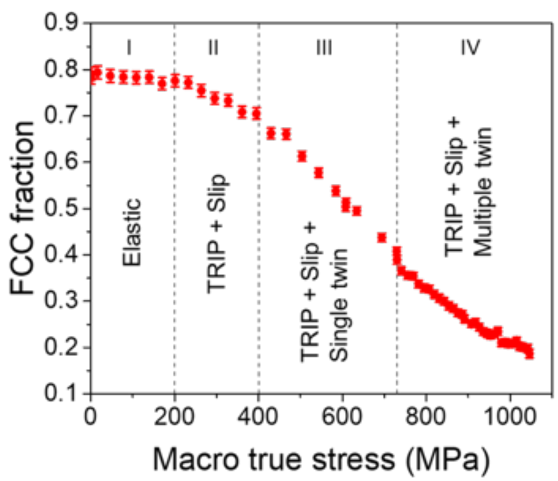
Deformation deformation characteristics revealed by ND

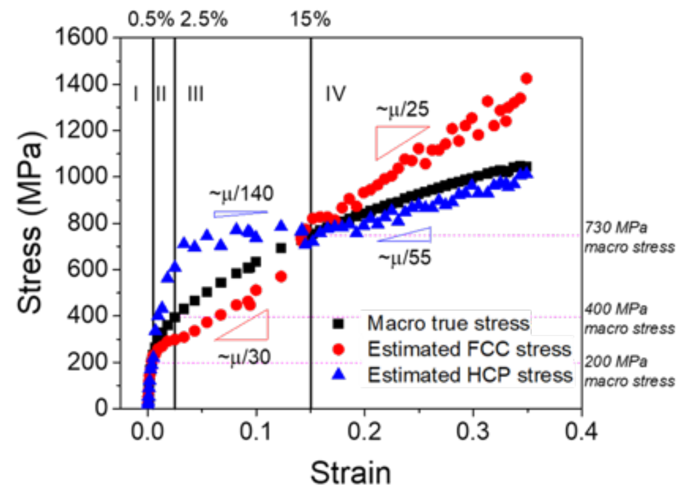
The **easily-triggered persisting TRIP** as well as the work-hardening potential of the HCP contribute together to the **persisting bulk work-hardening** of material.

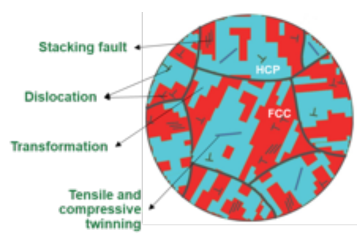
- Li et al. 2016 reduced atomic ratio of Mn to Fe in the FeMnCoCr quandary system and formed **dual-phase FCC-to-HCP TRIP-HEA**.



S. Fu, H. Bei, Y. Chen, T.K. Liu, D. Yu & K. An (2018) Materials Research Letters, 6:11, 620-626







Z. Li, K.G. Pradeep, Y. Deng, D. Raabe, C.C. Tasan, Nature 534(7606) (2016) 227-30.

- Enhanced strain hardening in FCC phase
- Persisting strain hardening in HCP phase

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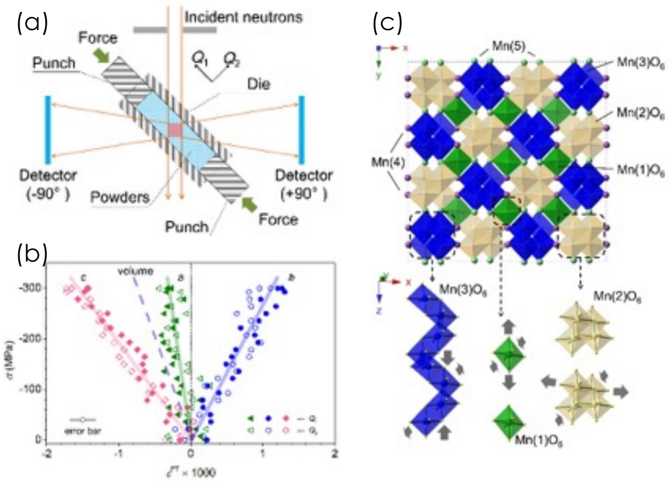
In Situ Neutron Diffraction Reveals a Stress-induced Charge-ordering Process in LiMn2O4



(a) A schematic illustration of the *in situ* loading setup. (b) Progressive lattice distortions of $LiMn_2O_4$ showing linear dependence upon stress. (c) Consistence of the lattice distortion with the Jahn-Teller effect at Mn(3) sites in the $LiMn_2O_4$ superlattice, showing the initial stage of the charge-ordering process.

This work was supported by the Division of Materials Science and Engineering, Office of Basic Energy Sciences (BES), U.S. Department of Energy (DOE). Experiments were performed at the ORNL Spallation Neutron Source's VULCAN instrument, which is sponsored by Scientific User Facilities Division, Office of BES, US DOE.

Scientific Achievement

In situ neutron diffraction captures the charge-ordering process through the progressive orthorhombic distortion in $LiMn_2O_4$ under loading with low stresses.

Significance and Impact

The results provide a new understanding of the charge-ordering process in spinel-type frustrated systems, and moreover, important considerations of physical compatibility for the material's applications in batteries.

Research Details

- A special die set was designed for compacting $LiMn_2O_4$ powders up to 300 MPa with *in situ* neutron diffraction.
- Rietveld refinement extracts the lattice parameters at longitudinal and transverse directions. By subtracting the elastic component, the pure lattice distortion exhibits linear dependence upon the applied stress, consistent in both directions but not a first behavior.
- The pure lattice distortion due to Jahn-Teller effects reveals an initial stage, where the stress continuously induces the localization of e_g electrons preferentially at the Mn(3) sites.

Y. Chen, D. Yu, and K. An, *Materials Research Letters*, **2016**, DOI: 10.1080/21663831.2016.1197858.

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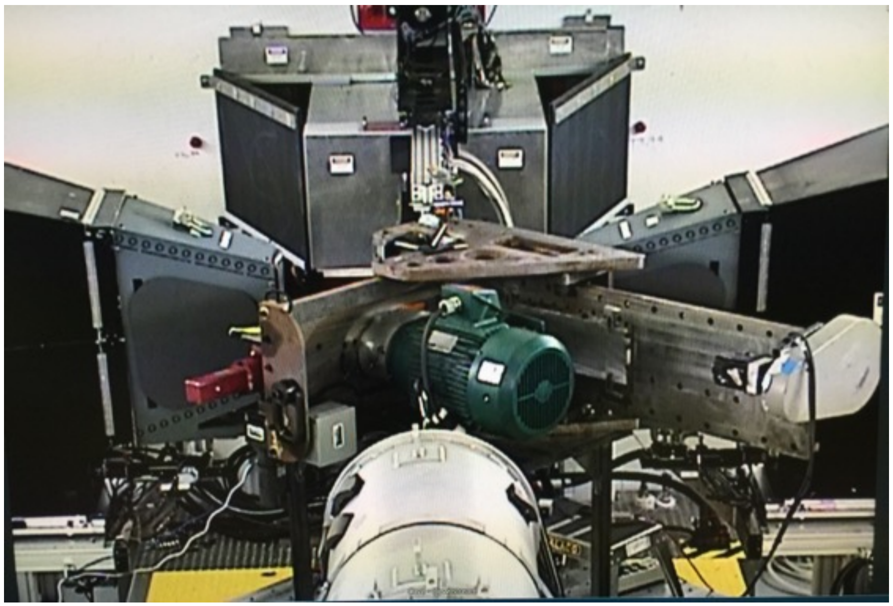
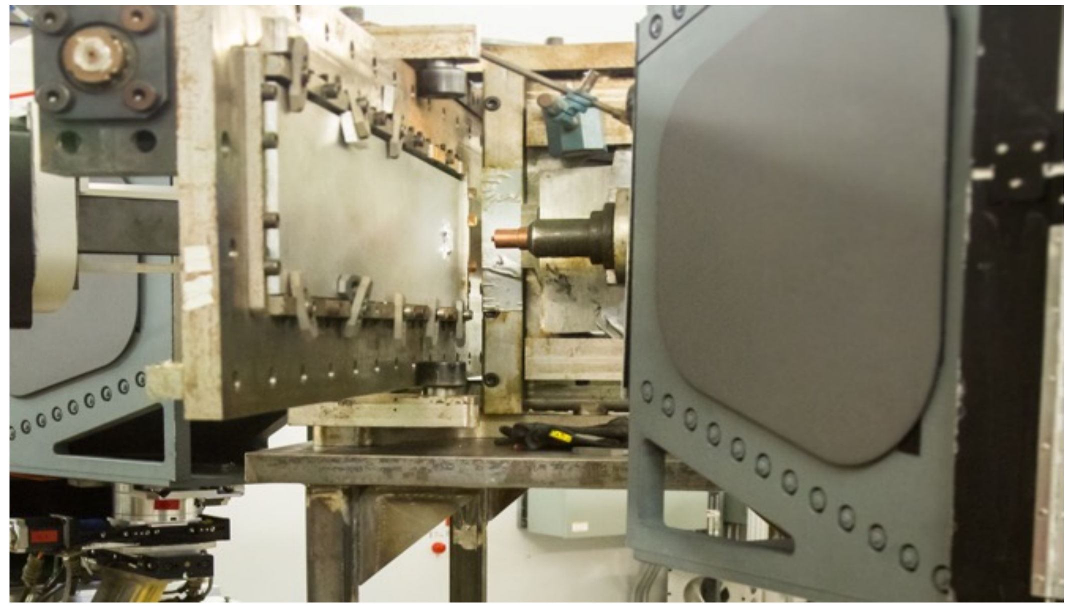
Extreme processing





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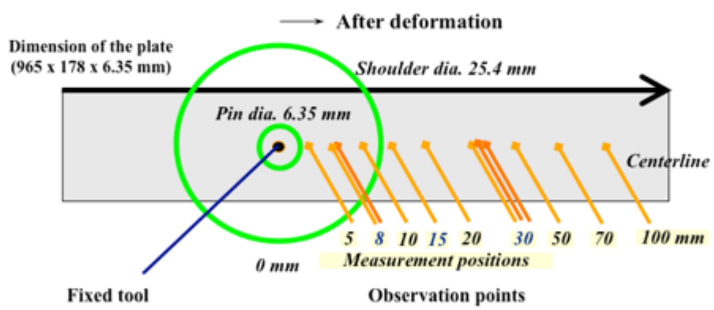
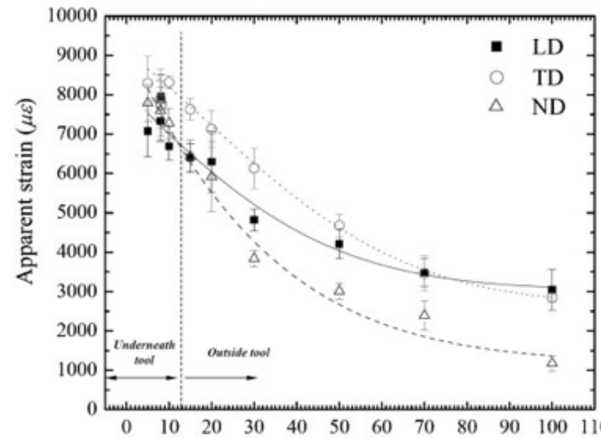
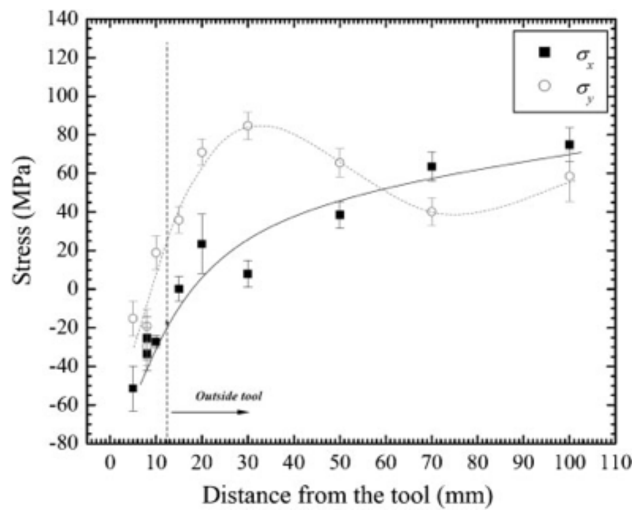
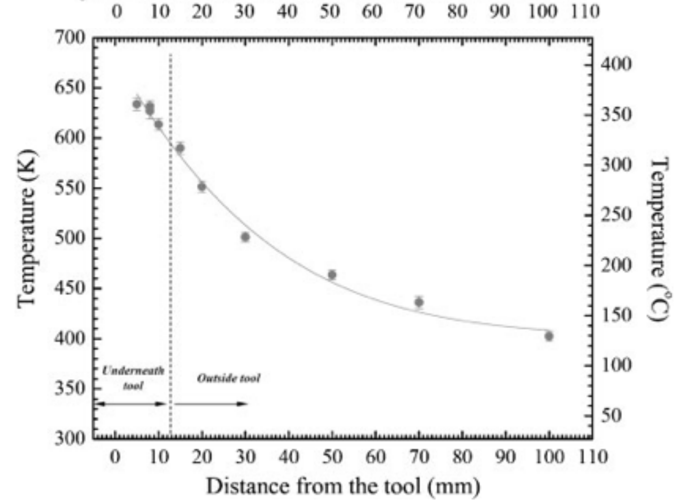
In-situ material behavior during FSW









Woo et al. Science and Technology of Welding & Joining, 12, 298-303, 2007

What about future?

To push the envelope further both in spatial and temporal under extremes.

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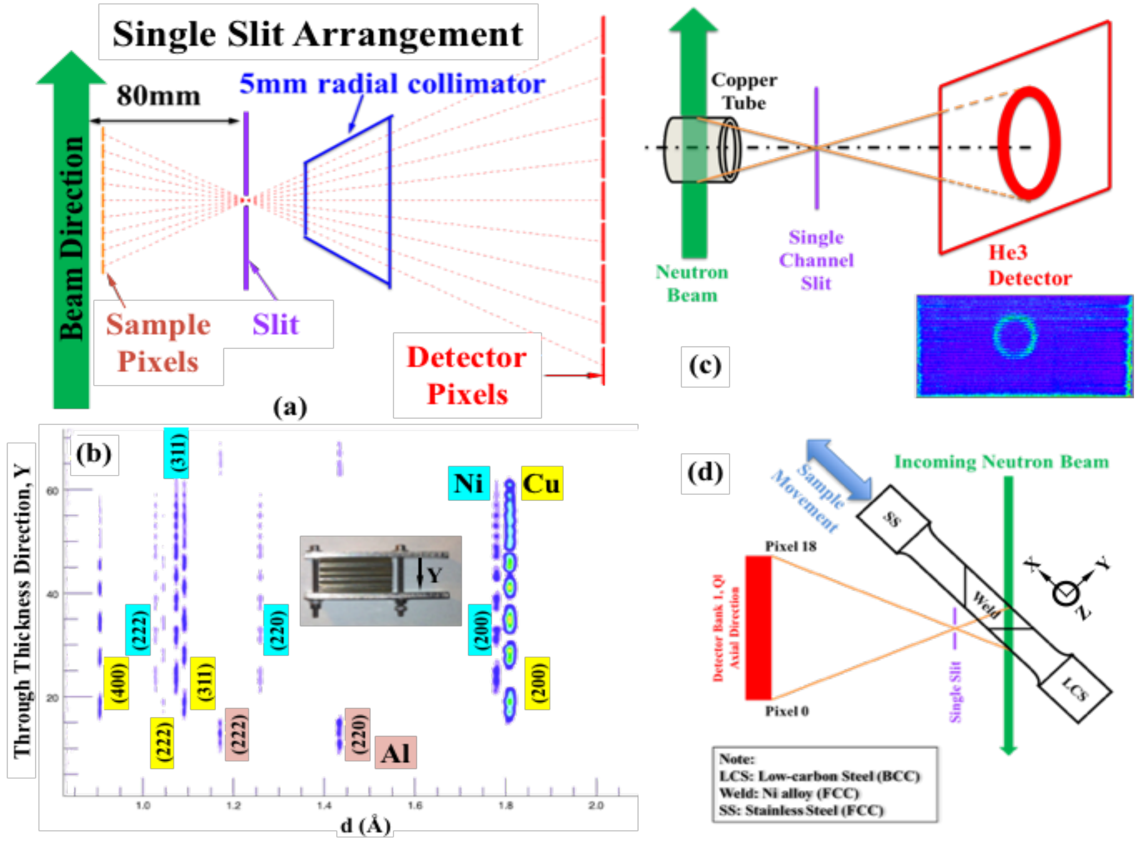
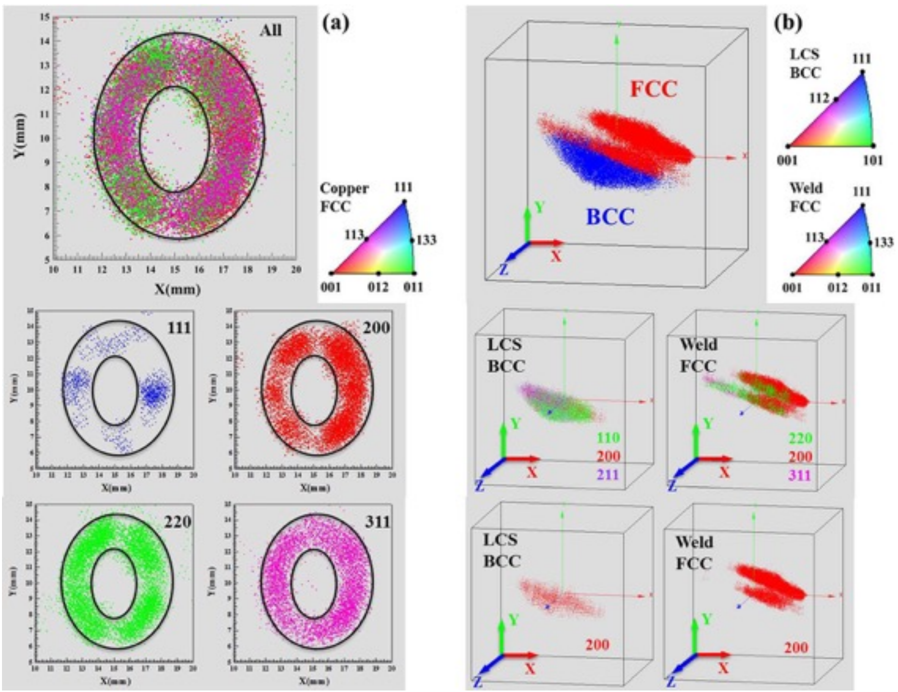
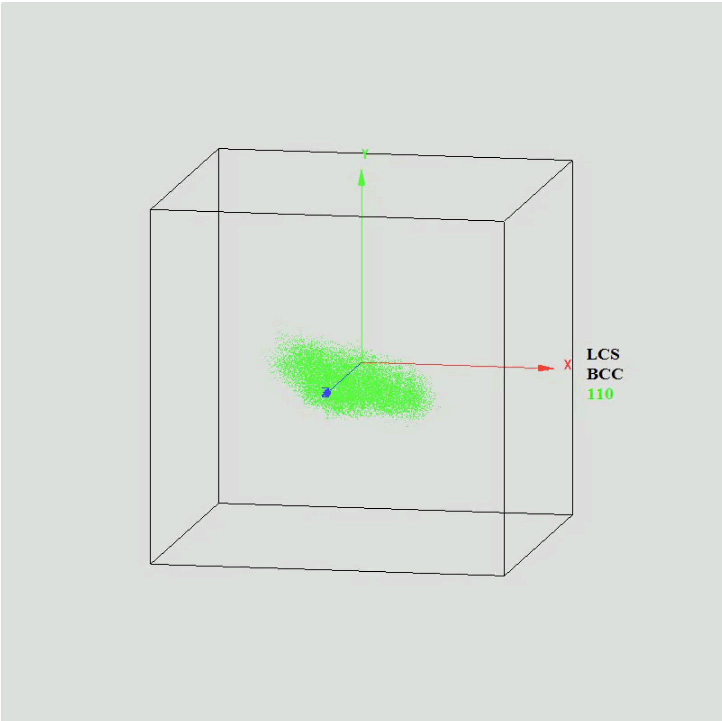
PIND: High spatial resolution by pinhole neutron diffraction



Wei Wu, Alexandru D. Stoica, Kevin D. Berry, Matthew J. Frost, Harley D. Skorpenske, and Ke An, "PIND: High spatial resolution by pinhole neutron diffraction". *APPLIED PHYSICS LETTERS 112, 253501 (2018)*

PIND: High spatial resolution by pinhole neutron diffraction





(a) The 2D distribution of different orientation grains inside the copper tube.
(b) The 3D distribution of various orientation grains in BCC and FCC phases inside the weld tensile sample.

Wei Wu, Alexandru D. Stoica, Kevin D. Berry, Matthew J. Frost, Harley D. Skorpenske, and Ke An, "PIND: High spatial resolution by pinhole neutron diffraction". *APPLIED PHYSICS LETTERS 112, 253501 (2018)*

Materials under stress, what we learn by using neutrons?

We learned a lot and will learn more in the future.

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