Materials under stress, what we learn by using neutrons?

Ke An
Spallation Neutron Source

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Hydraulic Press Channel - Youtube
Materials under stress

Deform to Comply
Materials under stress

Use neutrons to learn how materials

Deform to Comply
Why do we care?

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

Magnessium (Mg) Alloy in automobile
Materials under stress

Store elastic energy -> Dissipate energy -> Fracture
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/
New alloys design pushes the envelope

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
Complexity of structural or functional materials responses under straining

- Hardening in structural materials.
- Shape memory effects, super elasticity.
- Piezoelectric effect, mechanical to electrical energy.
- Mechanoccaloric effect, mechanical to thermal energy.
- Mechanically induced light emission.
- Stress induced magnetization, thermal and conductivity change materials.
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
Elastic or plastic changes in different length and time scales

LENGTH SCALE / TIME SCALE

nm, ns  μm, μs  mm, ms

atomistic lattice structure  discrete dislocation dynamics  subgrain structures  polycrystalline grain structure  macroscopic material behavior

https://www.mm.ethz.ch/teaching.html
Crystallographic understanding of materials under stress

Need tools
Tools for grain to meso scale

(a) Near-field HEDM

(b) Far-field HEDM

Synchrotron, far and near field

Neutrons
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.
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 Q₁ and Q₂, 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.
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

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

where $i,j=1,2,3$ indicate the components relative to three dimensional orthogonal axes.

$E^{hkl}$ and $\nu^{hkl}$ are [hkl] specific “diffraction elastic constants”.
Lattice strain has a sensitivity of plastic deformation

<table>
<thead>
<tr>
<th></th>
<th>$C_{11}$ (GPa)</th>
<th>$C_{12}$ (GPa)</th>
<th>$C_{44}$ (GPa)</th>
<th>A</th>
</tr>
</thead>
<tbody>
<tr>
<td>NiAl [1]</td>
<td>211.5</td>
<td>143.2</td>
<td>112.1</td>
<td>3.28</td>
</tr>
<tr>
<td>CuZn [32]</td>
<td>118.98</td>
<td>102.32</td>
<td>74.4</td>
<td>8.93</td>
</tr>
<tr>
<td>CeAg [36]</td>
<td>59.6</td>
<td>44.6</td>
<td>21.5</td>
<td>2.87</td>
</tr>
</tbody>
</table>

*Slip mode*

- NiAl $<100>|{111}$
- CuZn $<111>|{110}$
- CeAg $<100>|{111}$

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)
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

**Results and Discussion**

![Graphs showing stress-strain and work hardening rate](image)

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

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.

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

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 °C.
- In-situ neutron diffraction unraveled the loadsharing of Al matrix and the hardening precipitates at different loading stages.

Next generation alloys: high entropy alloys

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

- Low strain energy, ~3mJ/m²
- Low volume change
- Close in elastic modulus, yield point

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

Z. Li, C.C. Tasan, N. 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.


- Enhanced strain hardening in FCC phase
- Persisting strain hardening in HCP phase
**In Situ Neutron Diffraction Reveals a Stress-induced Charge-ordering Process in LiMn2O4**

**Scientific Achievement**

*In situ* neutron diffraction captures the charge-ordering process through the progressive orthorhombic distortion in LiMn$_2$O$_4$ 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$_2$O$_4$ 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.

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Extreme processing

neutrons.orl.gov
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.
PIND: High spatial resolution by pinhole neutron diffraction

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.

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