Neutron Diffraction Studies of Mesoscopic Deformation Behavior of Structural Alloys

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SNS-HFIR User Meeting, Oak Ridge, TN (Oct. 11 - 13, 2005)
Engineering Diffraction: Materials Behavior Studies with Neutrons

Acknowledgements

- Oak Ridge National Laboratory (HFIR, HTML, M&C)
- NSF International Materials Institutes (IMI) Program (DMR-0231320)
- LANSCE and ISIS

Outline of the talk

- Intergranular strain evolution during high-temperature tensile deformation

1. "Neutron diffraction measurement" of internal strains during deformation

- Other examples of current research projects in collaboration with ORNL (esp. NRSF2 instrument at HFIR through HTML user program)

2. Fundamental fatigue and fracture mechanics studies
3. Residual strains and texture in a weld joint
4. Intergranular strain evolution during plastic deformation
5. In-situ phase transformation studies

Example #1

Intergranular Strain Evolution during High-Temperature Tensile Deformation

H. Choo (UT)
D. W. Brown (LANSCE)

Outline

1. Objectives

2. Experimental Details

   - Materials
   - Neutron diffraction measurements

3. Results and Discussion

   - Lattice strain evolution during Quasi-static tensile deformation

4. Summary

Objectives

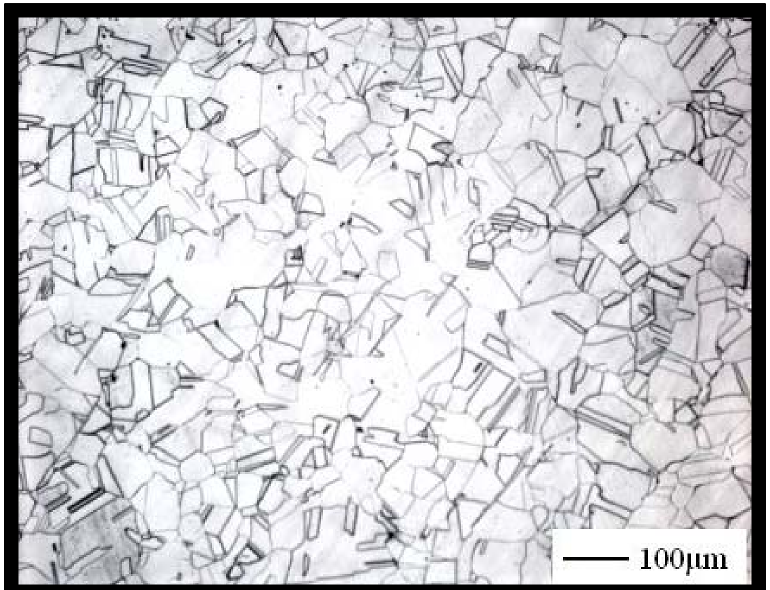
- To experimentally study the intergranular strain (or lattice strain) evolution during deformation of a single-phase fcc alloy at elevated temperatures using neutron diffraction.
- To Provide experimental database for polycrystal deformation modeling dealing with high temperature deformation behavior.

Material

- **Material**
  - 316FR stainless steel
  - Overall composition (17 Cr – 11 NI – 2 Mo wt%) is similar to 316L SS
    - low-carbon grade SS
    - more closely specified nitrogen content
    - chemistry optimized to enhance elevated-temperature performance.
- **Application**
  - 316FR SS is a candidate material for the Japanese Demonstration Fast Breeder Reactor Plant
  - Nippon Steel Corporation

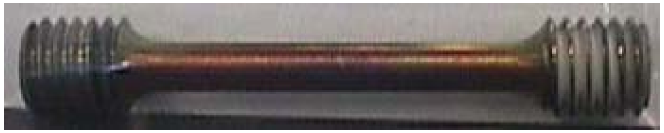
Material

- Microstructure
  - Annealed at 1050°C for 0.5 h followed by water cooling
  - Single phase, fcc
  - "Clean" - free of any precipitates
  - Grain size: approx. 50 μm
  - Random texture



Typical optical micrograph of 316FR

- Neutron Sample geometry
  - Cylindrical tensile specimen
  - Diameter: 6.5 mm
  - Gauge length: 45 mm
  - Overall length: 75 mm



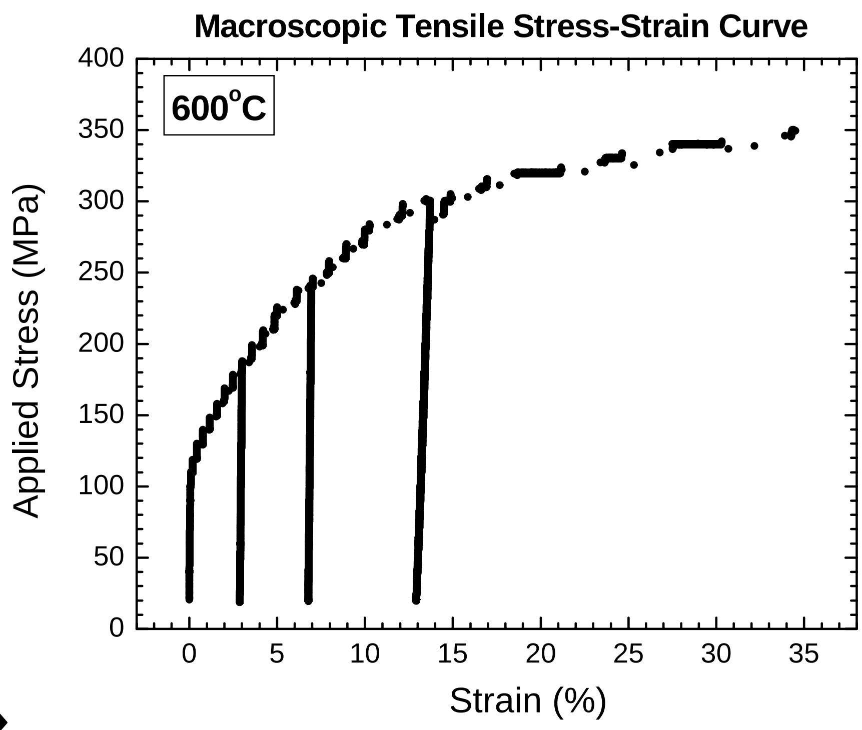
Tensile Behavior

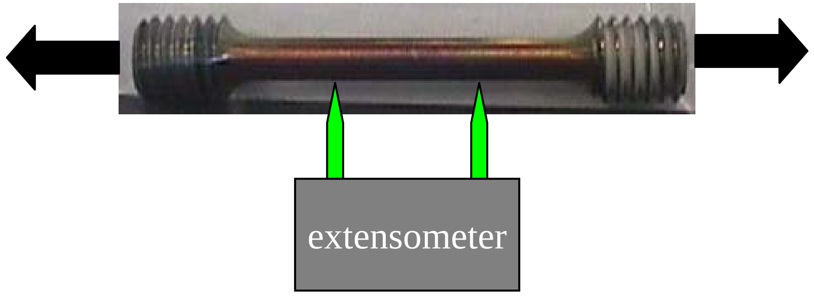
Micro-mechanical understanding of the
in-situ high-temperature deformation behavior
during quasi-static tensile deformation

Tensile Deformation at 600°C

Tensile Stress-strain curve:

- Tensile testing was conducted at 600°C (873K, $0.48T_m$).
- The specimen was incrementally loaded up to 340 MPa under the load control mode.
- Macro-strains were measured using an extensometer.



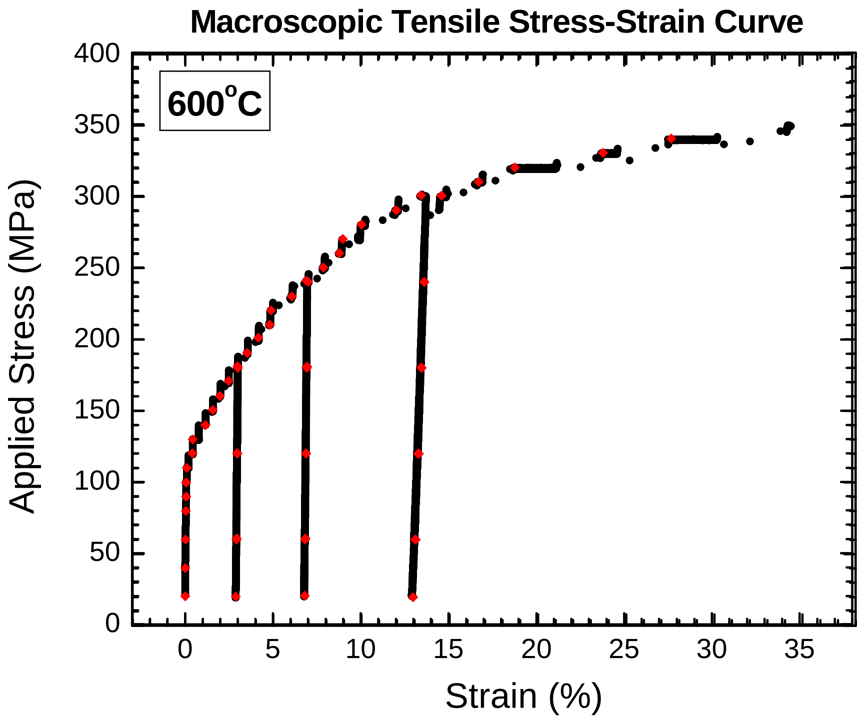


Measures bulk-averaged macroscopic strains $f(\sigma)$

E= 165 GPa
ν= 0.32
G= 62 GPa
σ_{ys}= 120 MPa

Tensile Deformation at 600°C

- **In situ neutron diffraction during tension test:**
  - Diffraction patterns were measured at each stress level (red points).



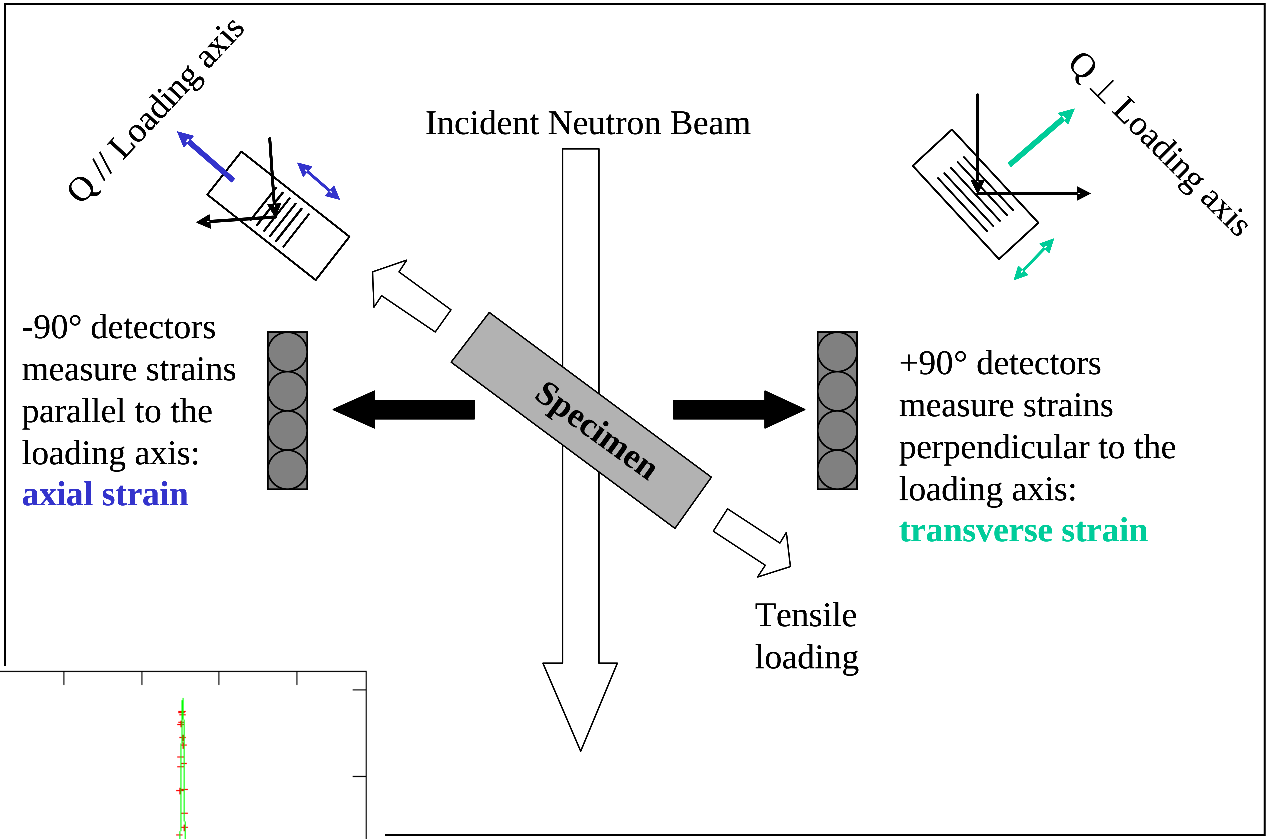
- *How do we obtain intergranular strains from the measured diffraction patterns?*

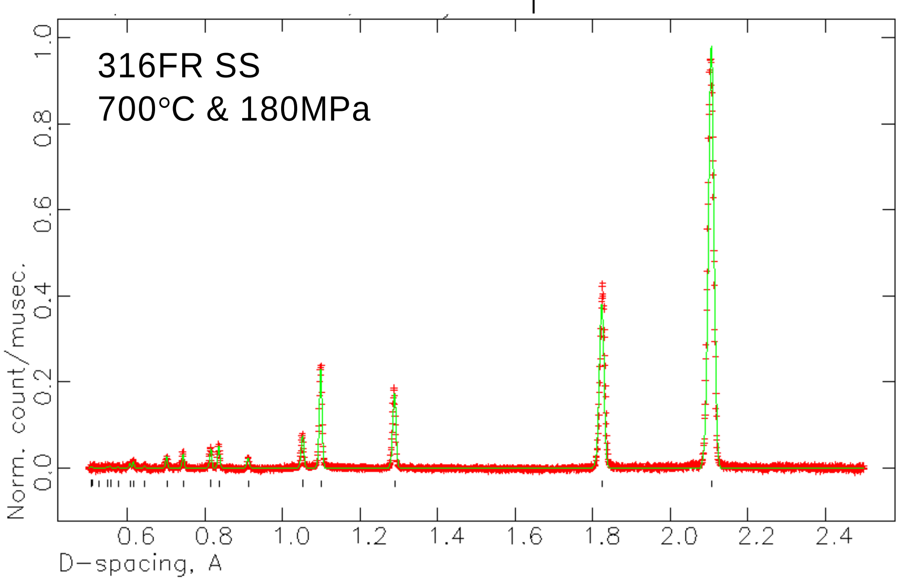
Neutron Diffraction Measurements

- In-situ HT loading measurements:

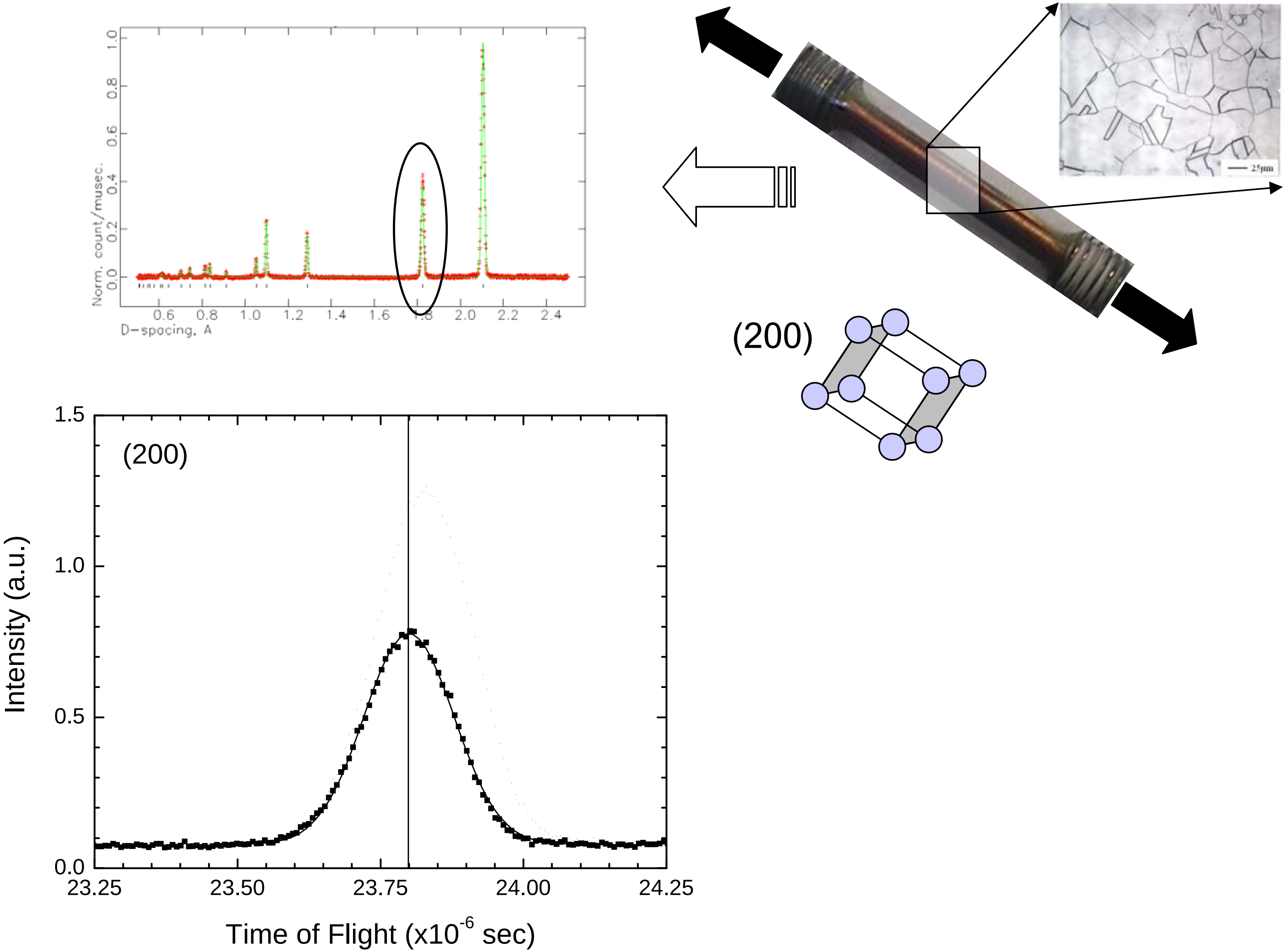
- SMARTS instrument (an engineering neutron diffractometer)

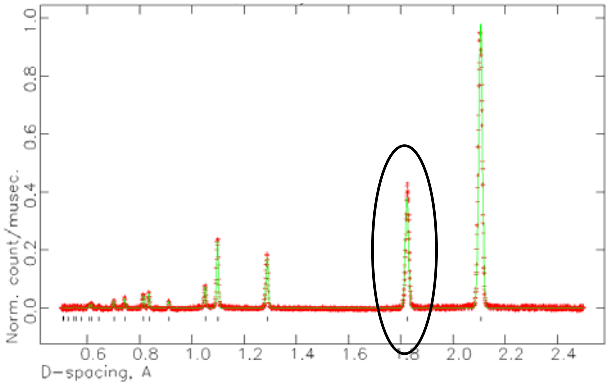
- LANSCE (pulsed neutron source)

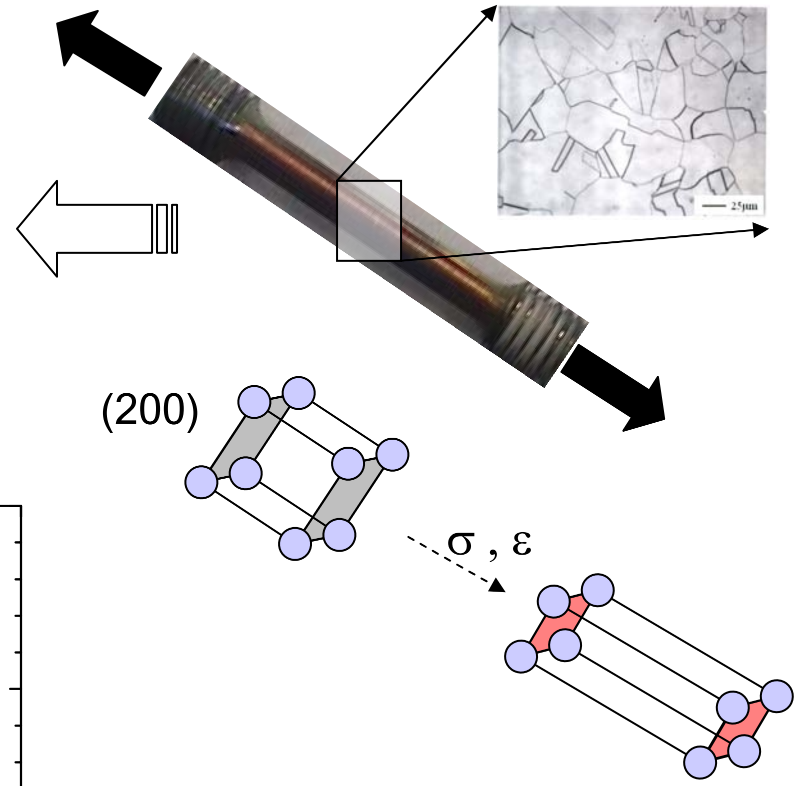


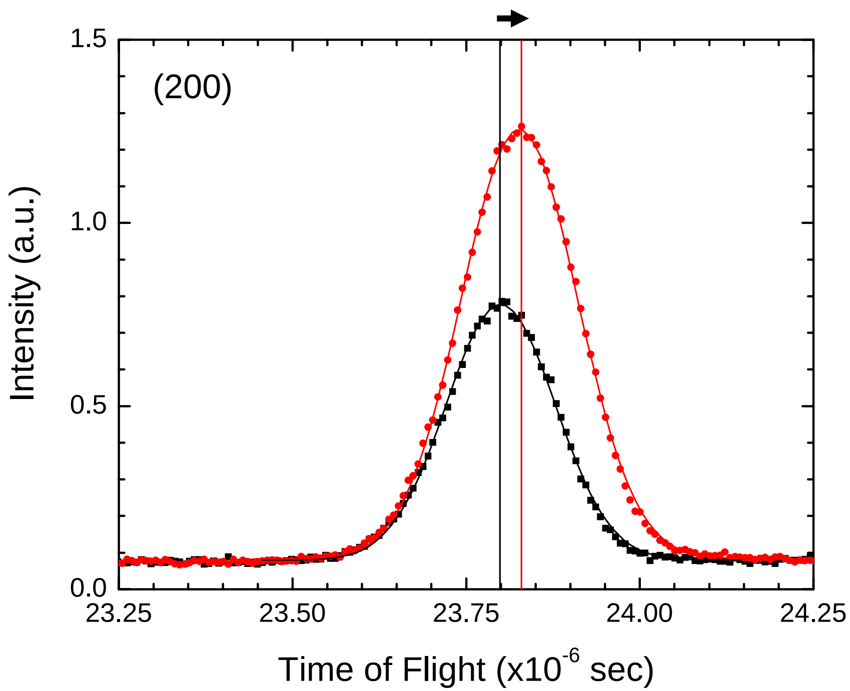


- Diffraction Diffraction gauge volume:
  - 315 mm^3
  - 2.5×10^9 grains sampled (at the center of the gauge length)
    - *Bulk-Averaged !*
    - *hkl specific !*


Norm. count/musec.
0.0 0.2 0.4 0.6 0.8 1.0
0.6 0.8 1.0 1.2 1.4 1.6 1.8 2.0 2.2 2.4
D-spacing, A
25μm
(200)
(200)
Intensity (a.u.)
0.0 0.5 1.0 1.5
23.25 23.50 23.75 24.00 24.25
Time of Flight (x10-6 sec)

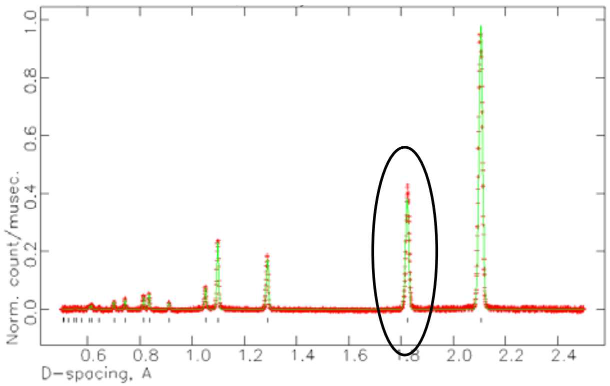


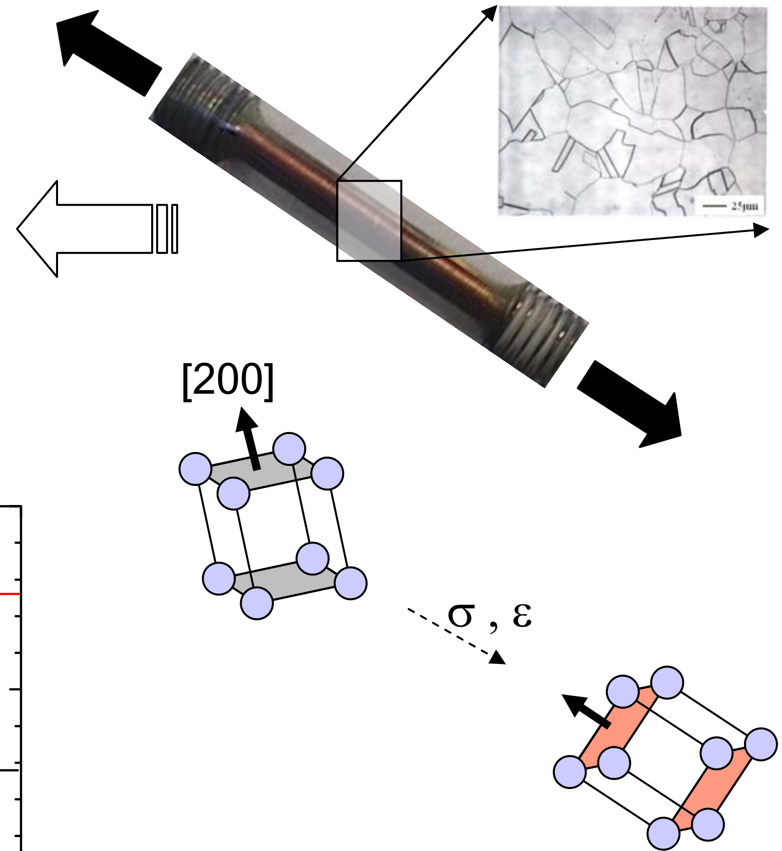


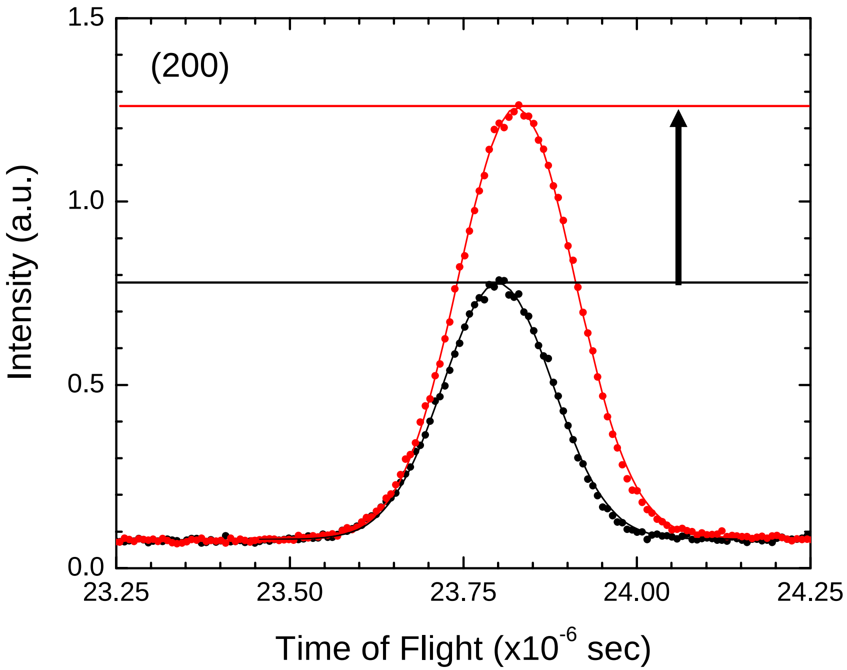


Changes in d-spacings:

- Development of intergranular strains

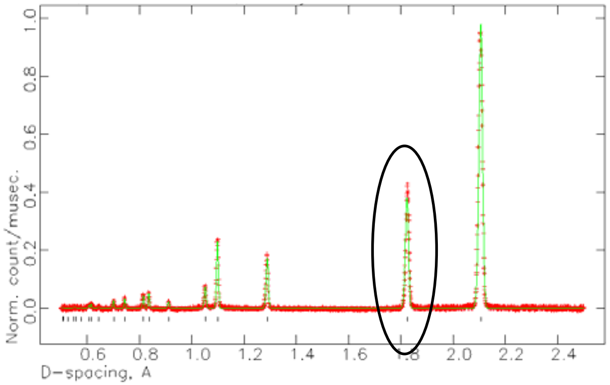


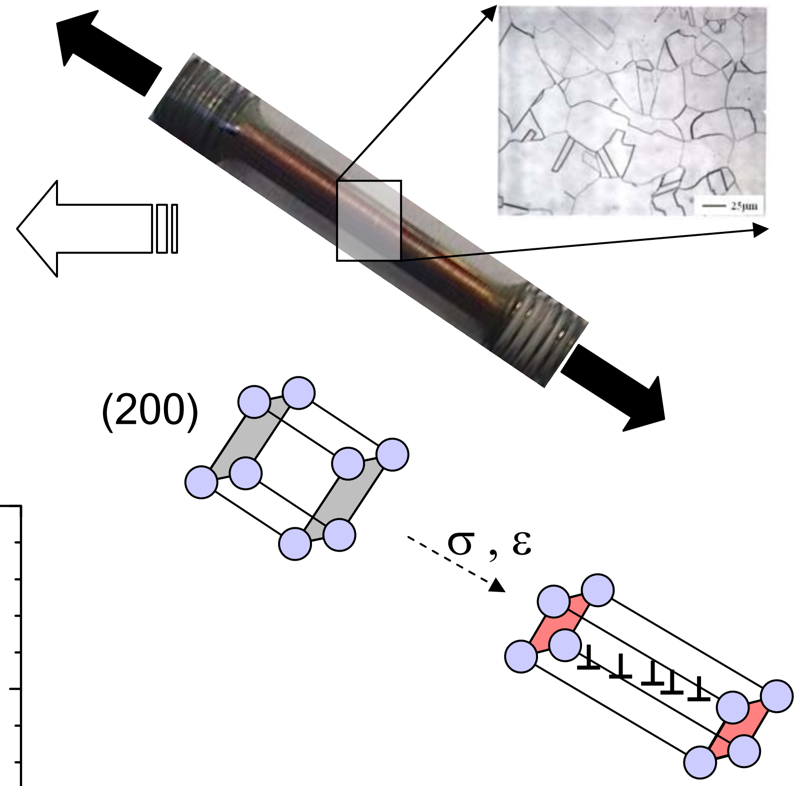


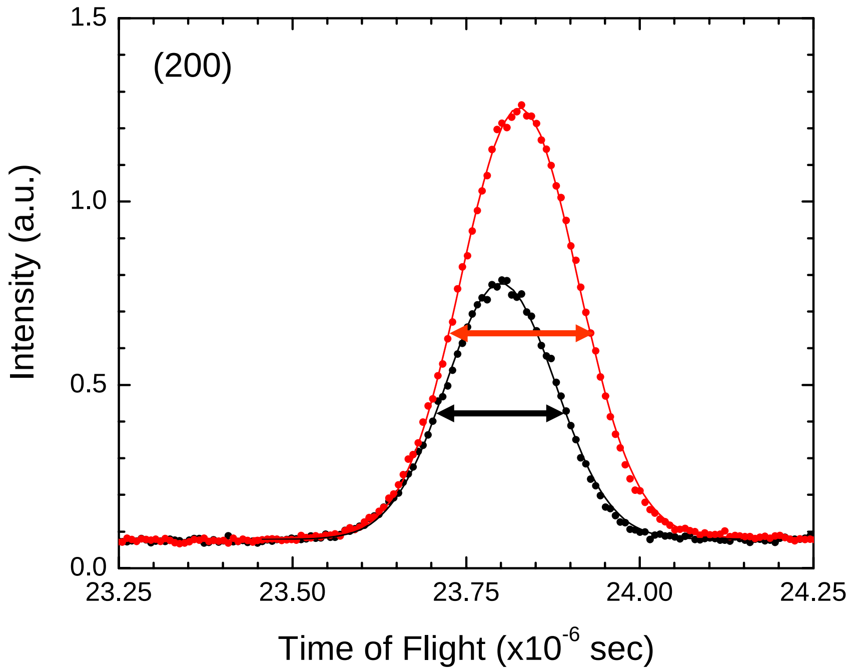


Changes in intensities:

- Grain rotation (i.e., development of texture)
- Phase transformation







Changes in peak width:

- Increase in dislocation density
- Increase in heterogeneous elastic strain distribution

Intergranular Strain (or Lattice Strain)

- d-spacings were obtained by fitting the individual diffraction peaks using *GSAS*.

- Then, hkl-specific lattice strains were calculated using:

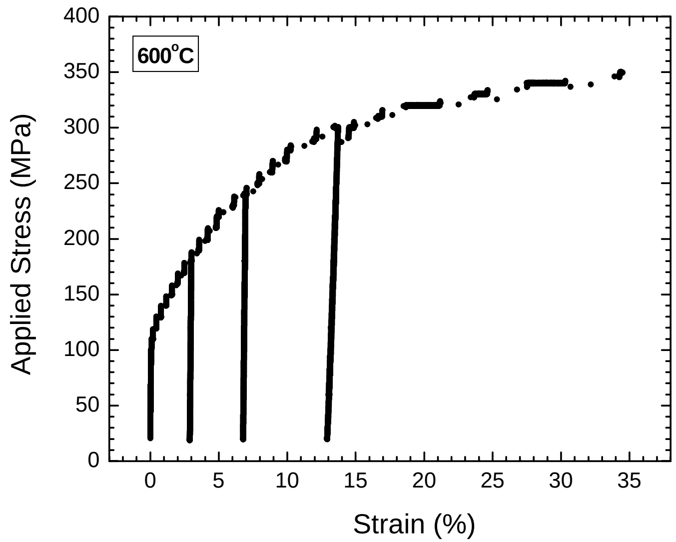
$$\varepsilon = \frac{d_i - d_0}{d_0}$$

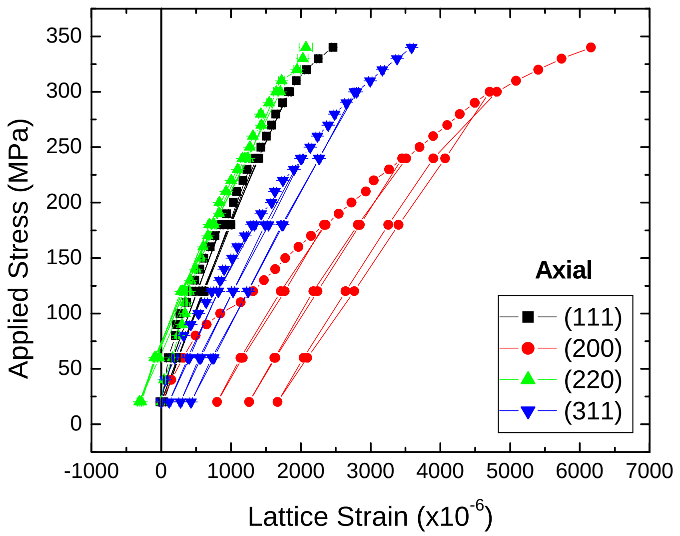
  - Where d_0 and d_i are the d-spacings of the γ-Fe when unloaded and loaded, respectively.

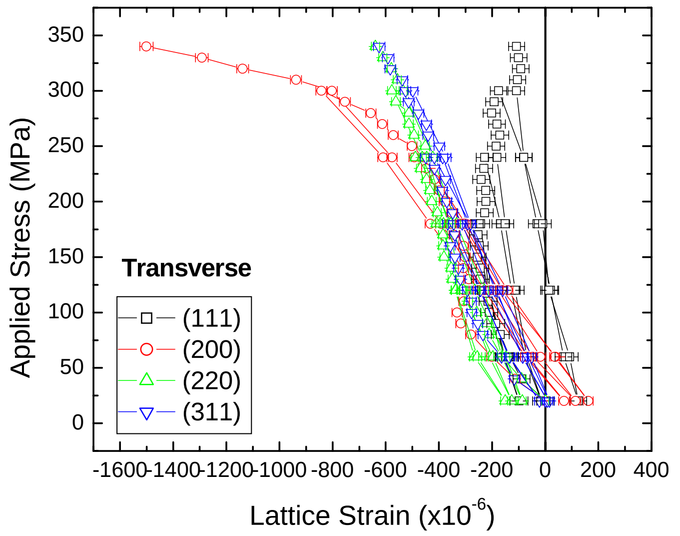
  - For quasi-static tension tests d_i varies with the changes in the applied load.

Tensile Deformation at 600°C

- Macro Stress-Strain Behavior vs. Lattice strain evolution

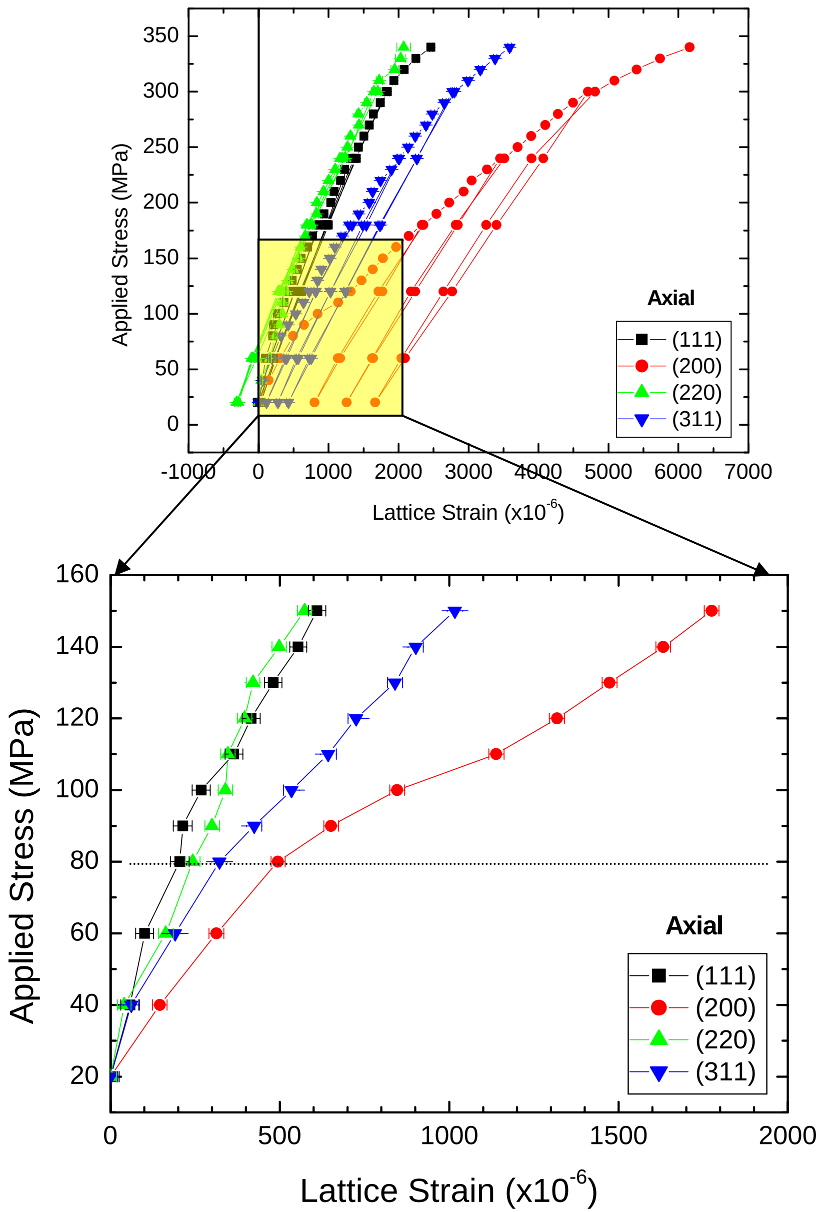






- Lattice strains measured for:
  - multiple *hkl*s
  - axial and transverse (Poisson) orientations
  - shows elastic/plastic anisotropy in intergranular strain evolution

Lattice Strain Evolution at 600°C - Axial Direction



- **Elastic anisotropy**
  - Strains linearly increase
  - (111) stiff
  - (200) compliant

- **Plastic anisotropy**
  - The rate of lattice strain accumulation decreases when **"slip"** occurs in the orientation
  - e.g., upward inflection in (220) strains
  - Such deviation from the initial linearity influences the evolution of other lattice strain components.

- The tensile behavior at 600°C is qualitatively similar to the RT behavior previously reported.

A Few Other Examples of Current Research Projects

2. Fatigue and Fracture Mechanics Studies

3. Residual Stress and Texture Studies

4. Intergranular Strain Evolution during Plastic Deformation

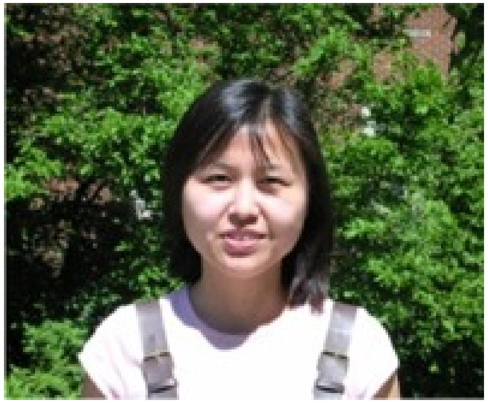
5. In Situ Phase Transformation Studies

Research Example #2

Effect of overloading during fatigue on the internal stress, plastic zone, and crack growth rate

Research Team

Y. Sun (UT)
C. Fan, Y. Lu, P. K. Liaw, & H. Choo (UT)
K. An, F. Tang, C. R. Hubbard (HFIR)
D. W. Brown (LANSCE)



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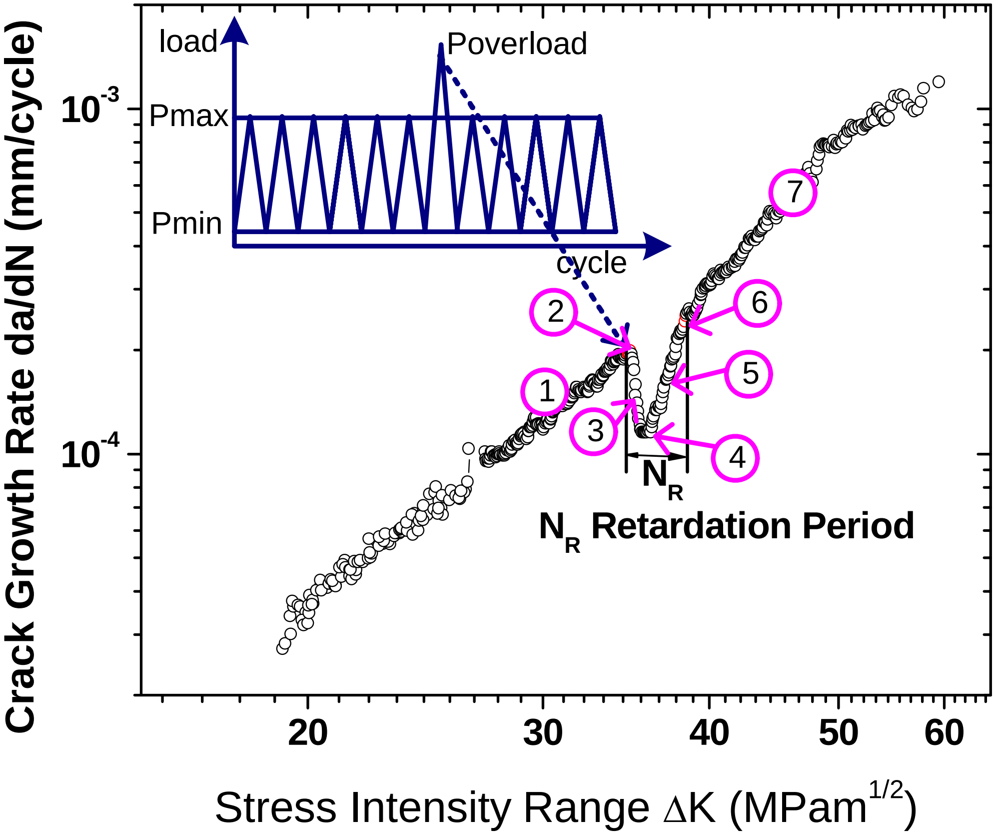
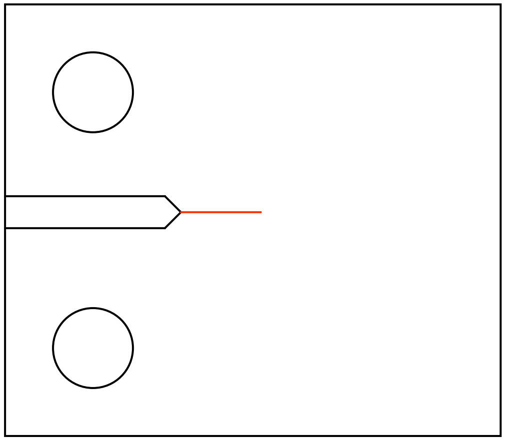
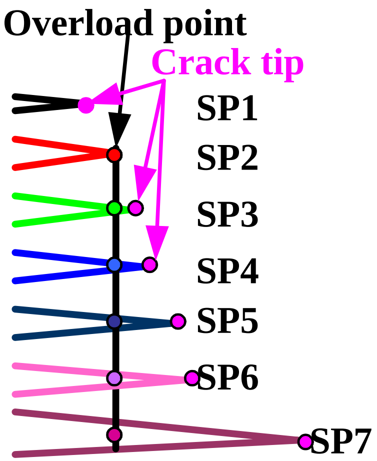
Effect of overloading during fatigue on the crack growth rate



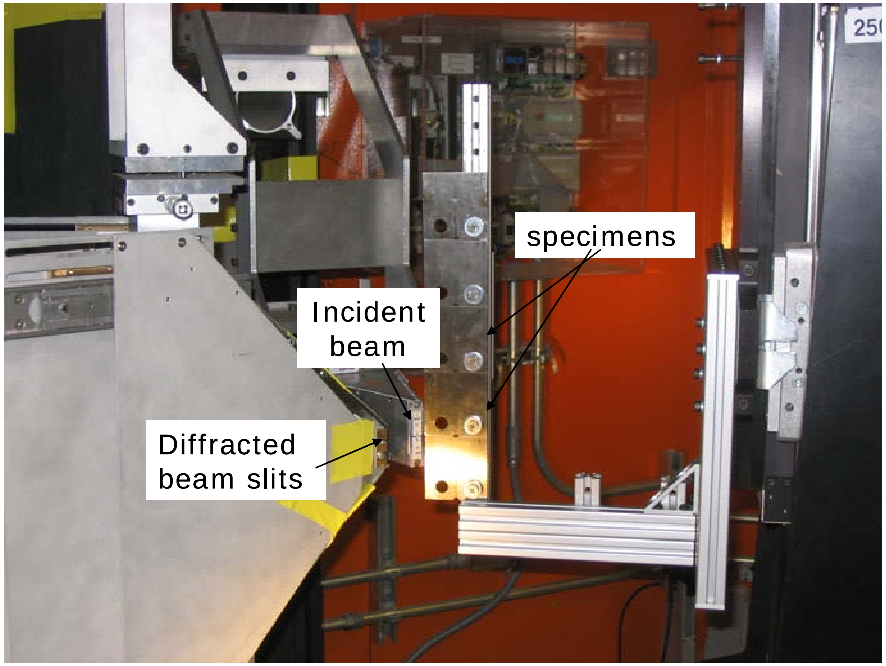




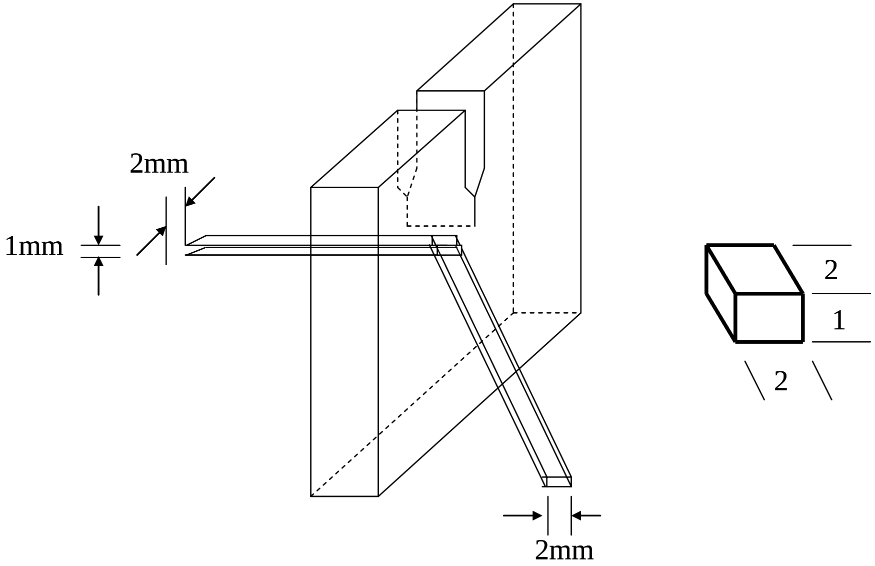
- After overload, there is a period of crack growth retardation that is related to the magnitude and number of overloads.

Measurement of the internal strain using neutron diffraction

- Experimental setup at the NRSF2 instrument at HFIR

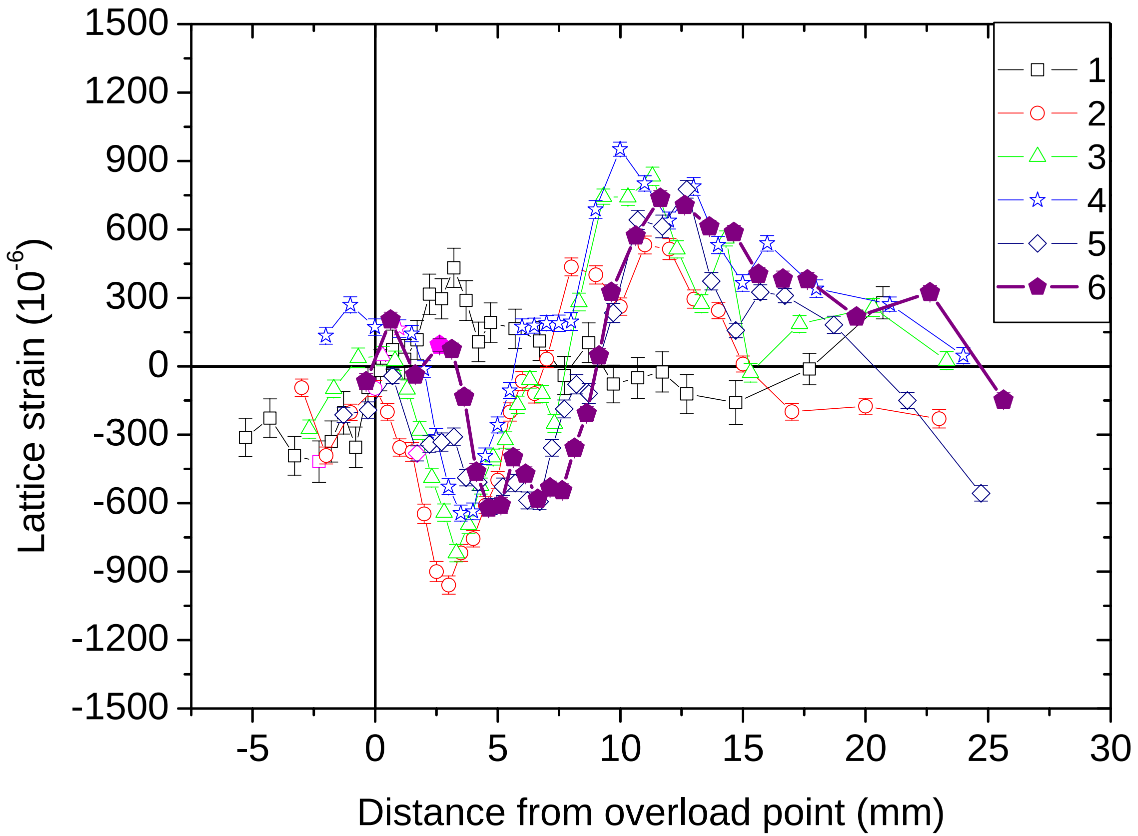


- A total of seven specimens were prepared:
  - to investigate the elastic lattice strain evolution at different stages during the retardation period

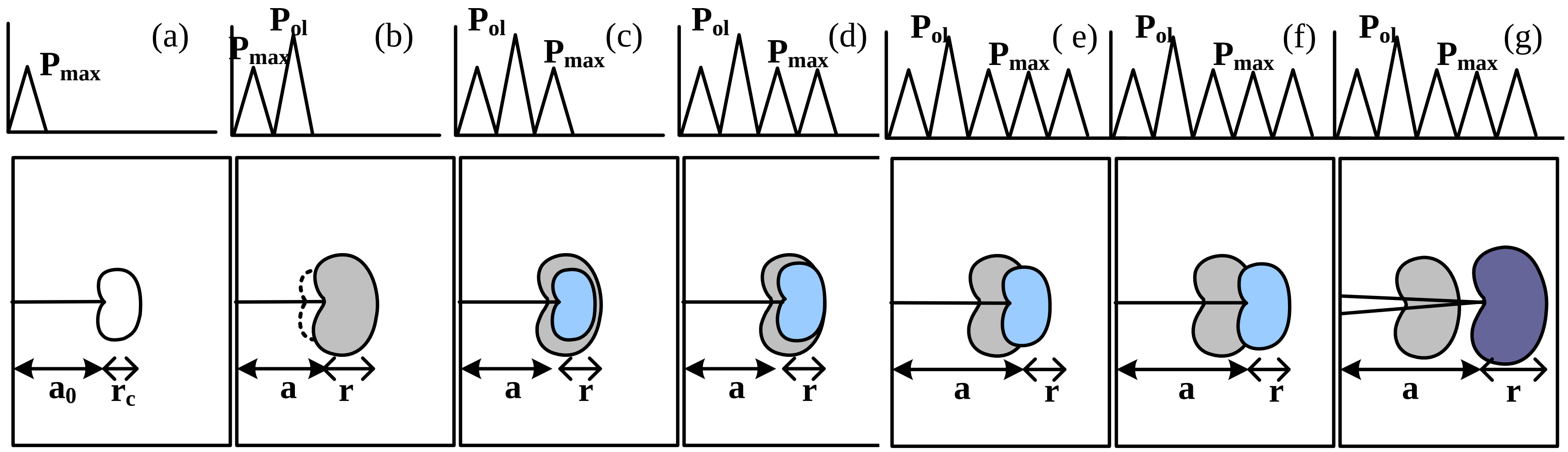


- Internal strain was measured:
  - from the (2x2x1 mm) volume
  - within the specimen
  - along the crack

- Three strain components were measured:
  - In plane, Longitudinal, & Normal



Lattice strain (10^{-6})
Distance from overload point (mm)
1
2
3
4
5
6



(a)
(b)
(c)
(d)
(e)
(f)
(g)
P_{max}
P_{ol}
a_0
r_c
a
r

Research Example #3

Neutron Diffraction Studies of Friction Stir Processed Al and Mg Alloys

Research Team

W. Woo (UT)
P. K. Liaw, & H. Choo (UT)
Z. Feng, S. A. David, C. R. Hubbard (ORNL)
D. W. Brown (LANSCE)

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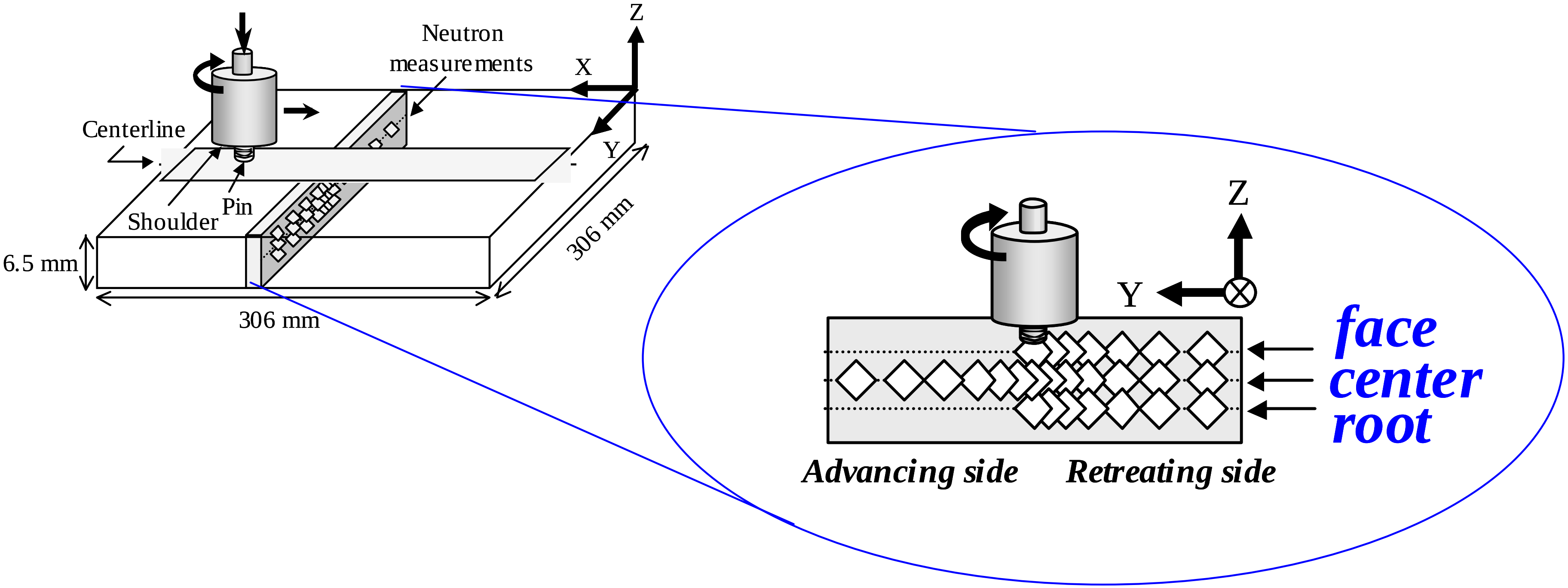
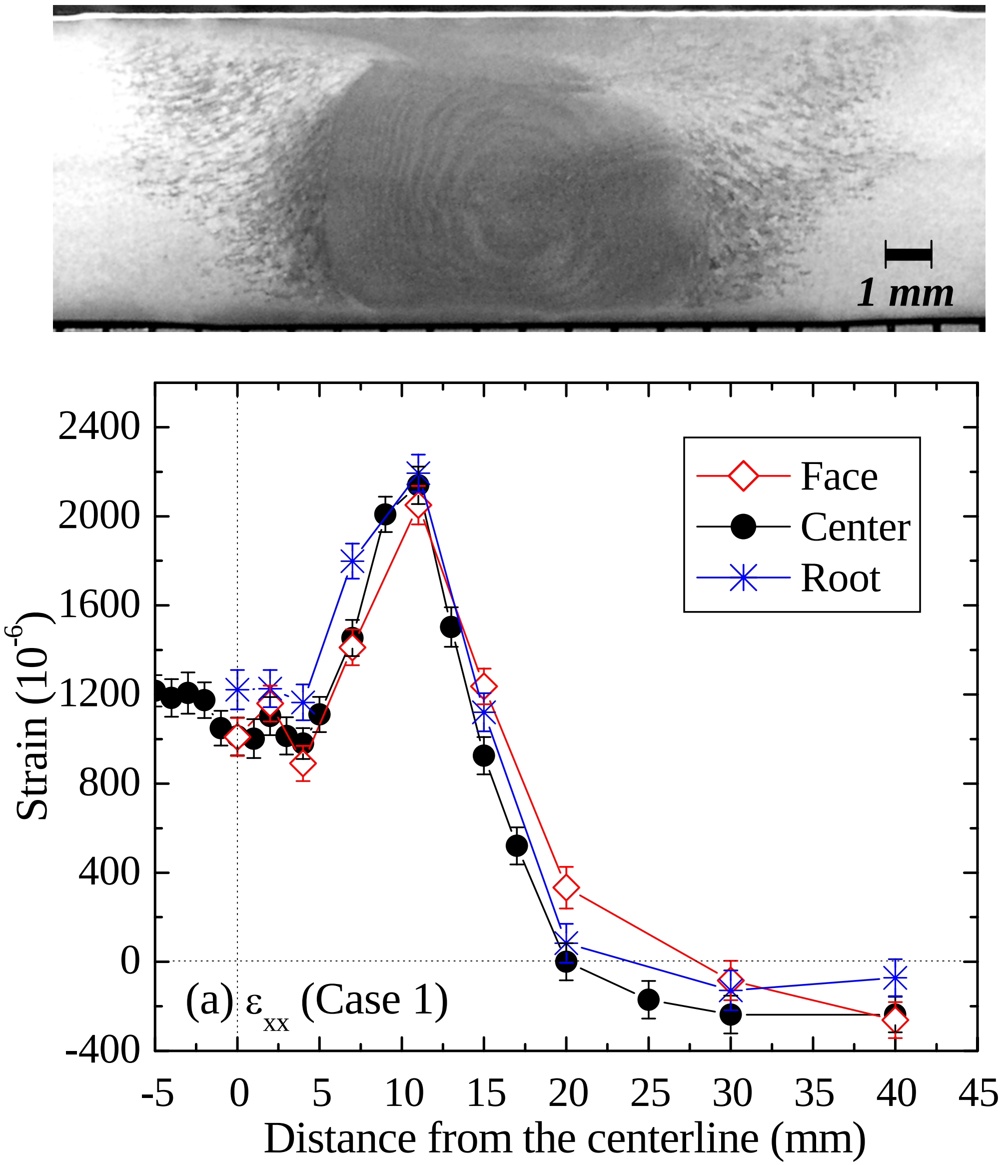
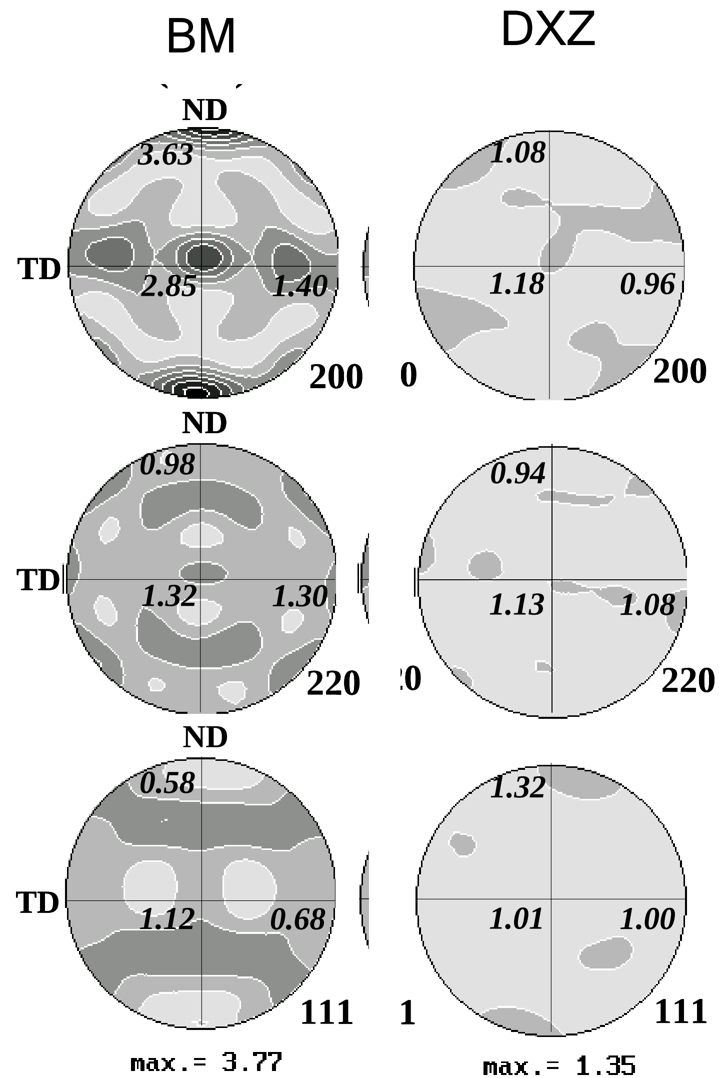
Friction Stir Processing



- **Friction-stir welding (FSW):**
  - Solid-state joining process (Max. temp.= 0.8 Tm)
  - Frictional heating and severe plastic deformation provide strong bonding
- **Fundamental studies of the effect of processing parameters on:**
  - Internal stress evolution
  - Texture variations
  - Microstructural softening and aging effects

Internal Strain and Texture





Research Example #4

Intergranular Strain Evolution during the Deformation of Zircaloy-4

Research Team

E. Garlea (UT)
P. K. Liaw, & H. Choo (UT)
V. O. Garlea, C. R. Hubbard (ORNL)
E. C. Oliver (ISIS)

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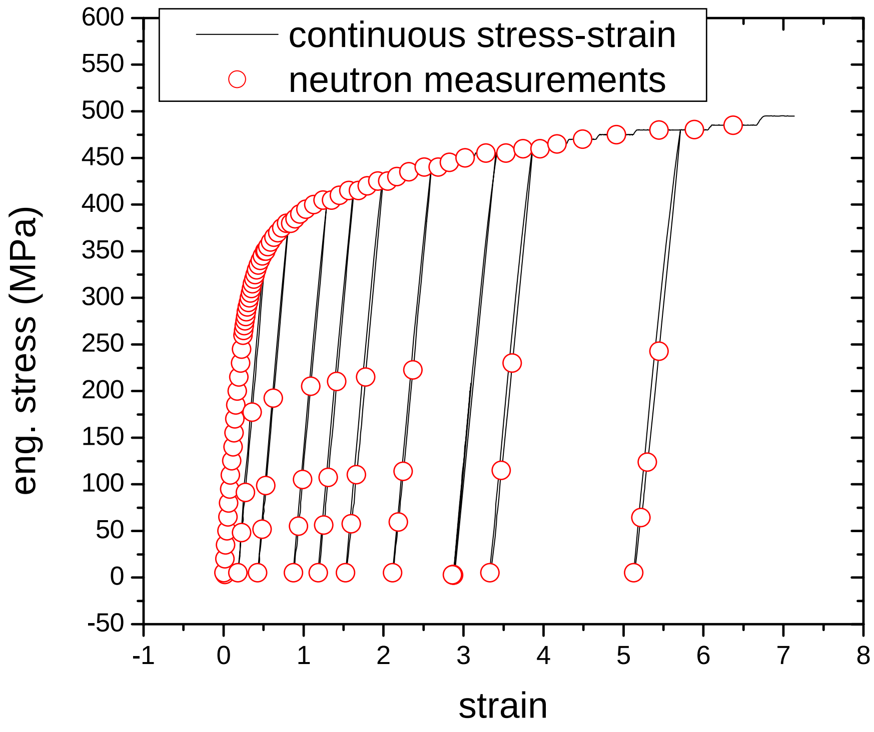
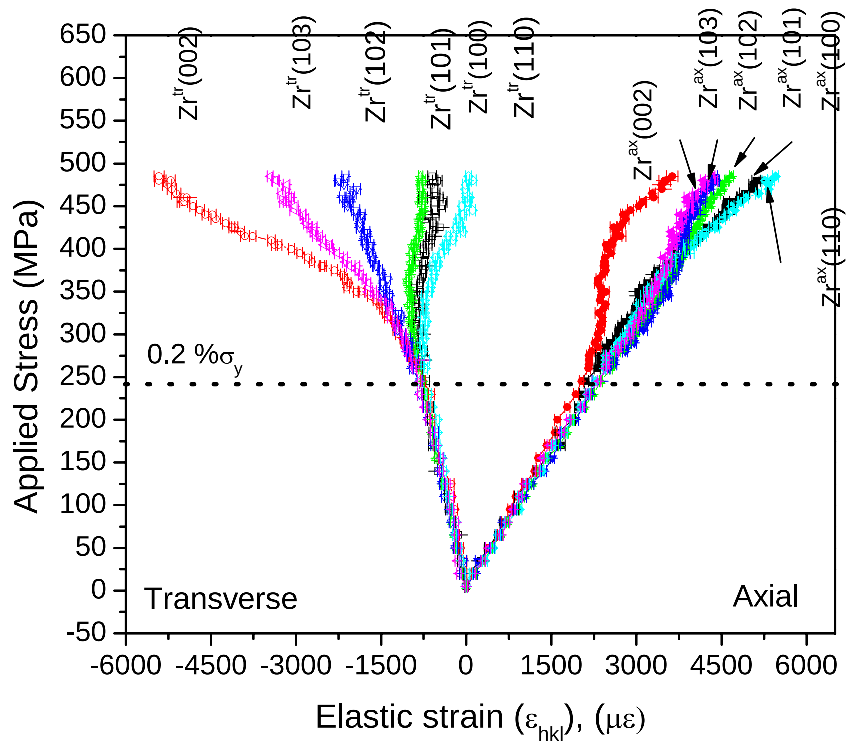
Intergranular strain evolution during tensile deformation at RT

- Uniaxial tensile stress-strain behavior of a Zircaloy-4 (Zr-1.4wt% Sn)

Macroscopic σ–ε behavior

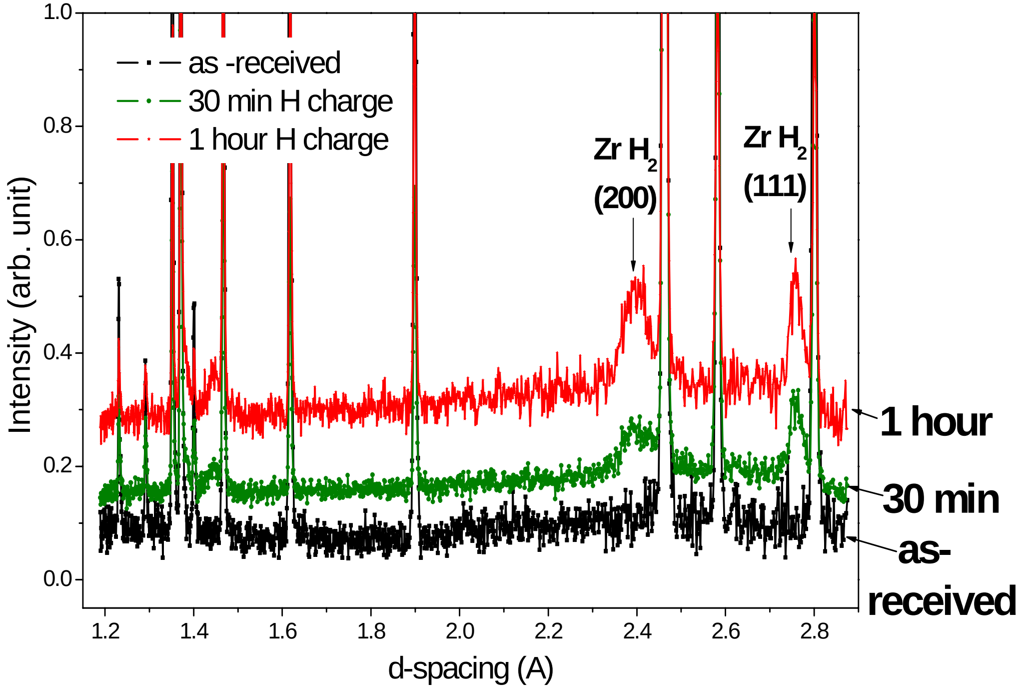


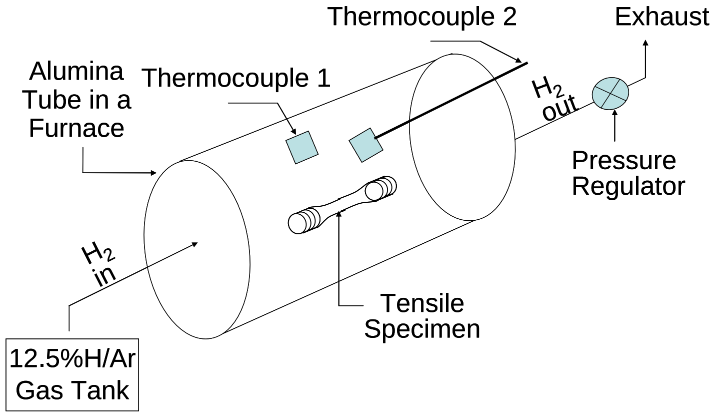
Intergranular strain evolution

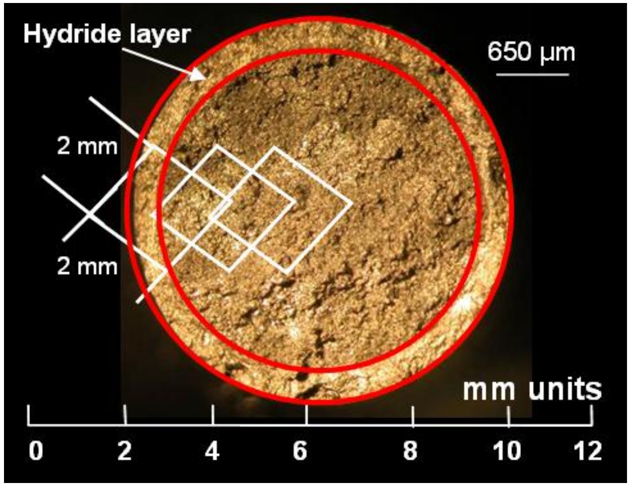


Hydrogen charging of a zirconium alloy

- Zircaloy-4 alloy is charged with hydrogen to study:
  - phase distributions
  - effects on the deformation micromechanics (future)







Research Example #5

Deformation Induced Phase Transformation in a Co-based Superalloy

Research Team

M. Benson (UT)
P. K. Liaw, R. A. Buchanan, & H. Choo (UT)
X.-L. Wang (ORNL)
D. W. Brown (LANSCE)

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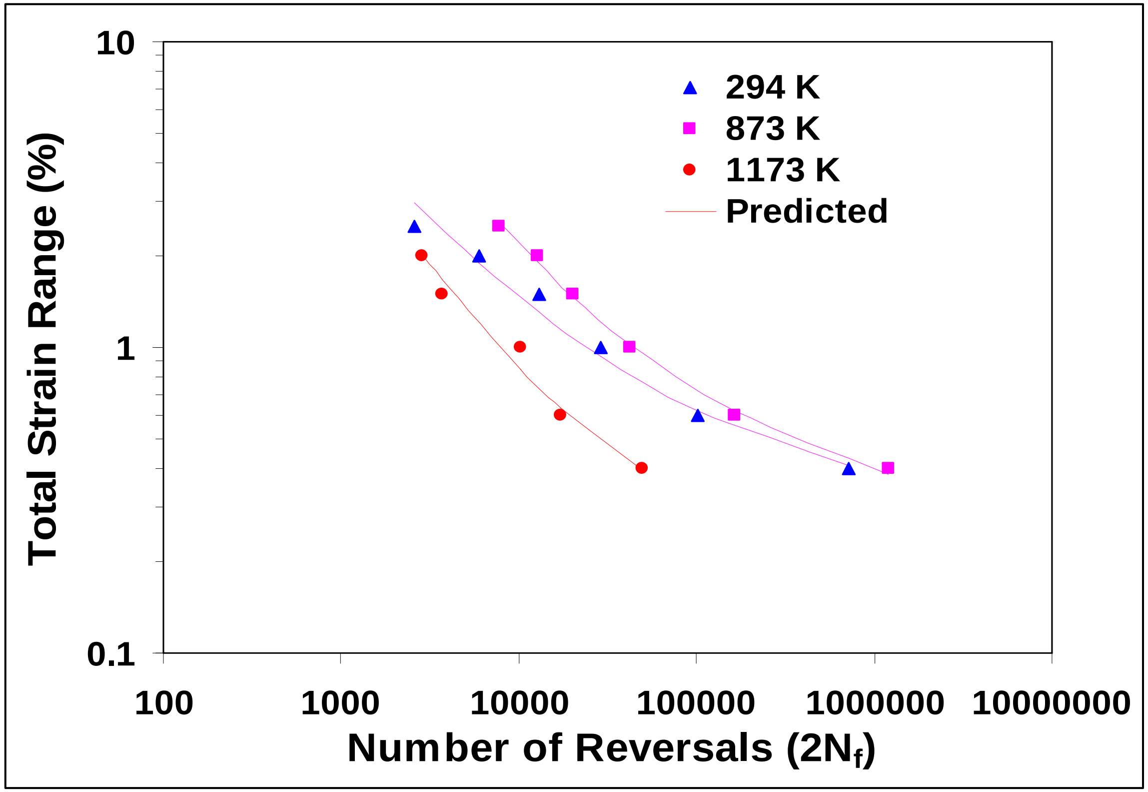
Fatigue lifetime of Co-based superalloy

- In general, the fatigue lifetime decreases with increasing temperature.

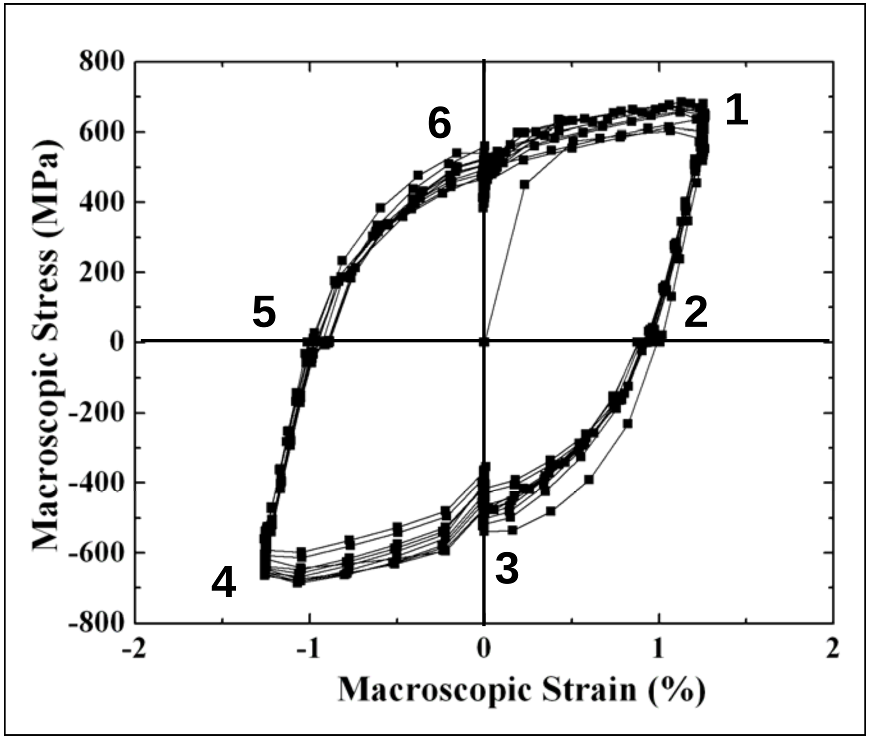


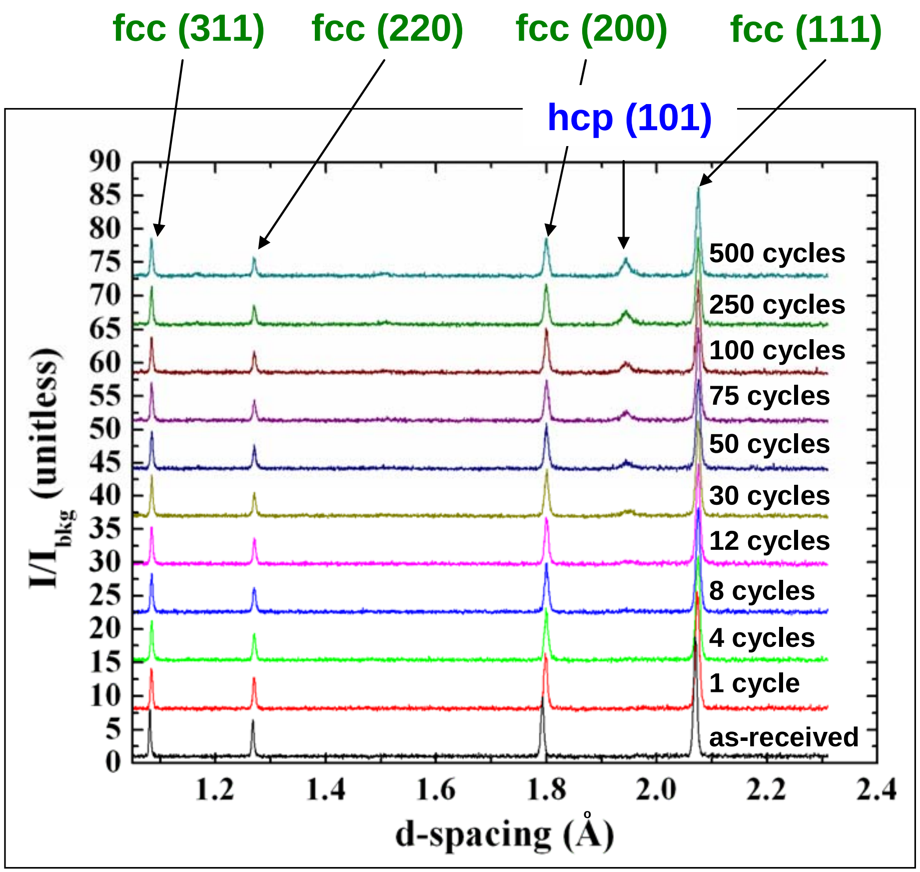
- However, the studies on ULTIMET alloy (Co-based superalloy) showed unusual behavior:
  - fatigue life increased from 294 to 873 K
  - then decreased from 873 to 1173

- The stress-induced phase transformation (below 873K) likely influences the fatigue life.

In-situ neutron diffraction studies of phase transformation at RT

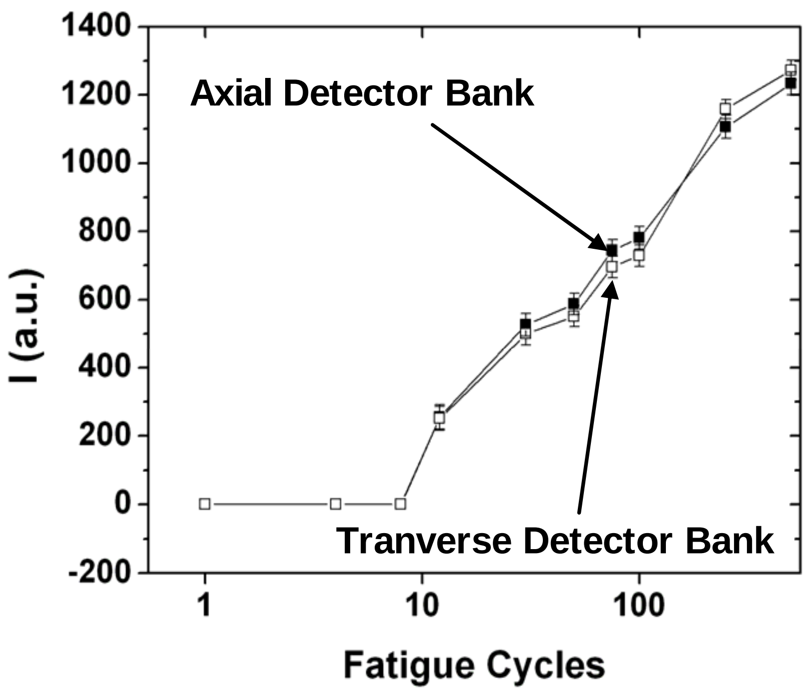
- Fatigue-induced phase transformation from the face-centered-cubic (fcc) to hexagonal-close-packed (hcp) phase was studies during strain-controlled cyclic loading.



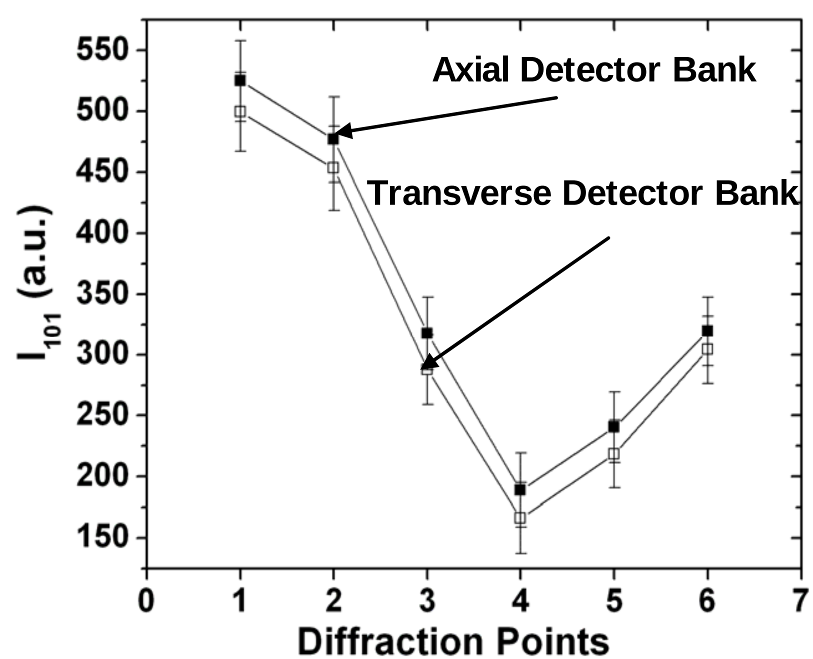


fcc → hcp transformation

Development of hcp phase as a function of the total number of fatigue cycles



Changes in the hcp peak intensity within a single fatigue cycle



Concluding Remarks

1. A few example research projects were presented:

   – In situ (or ex situ) neutron diffraction studies of deformation behavior of structural alloys

2. New capabilities of NRSF2 (HFIR) and VULCAN (SNS) enable:

   – Better measurements (accuracy, speed, exp setup/DAQ)
   – New types of studies:
     - Elastic / plastic deformation fundamentals
     - Fatigue and fracture mechanics (in situ mechanical loading + spatial mapping)
     - Creep studies (in situ loading + temperature + time resolved measurements)
     - Torsional (bi-axial) behavior
     - Phase transformation studies
     - Unique environments (e.g., hydrogen + mechanical loading)