Application of In-situ Diffraction Tools towards Fundamental Understanding of Material Behavior During Thermo-Mechanical Processing

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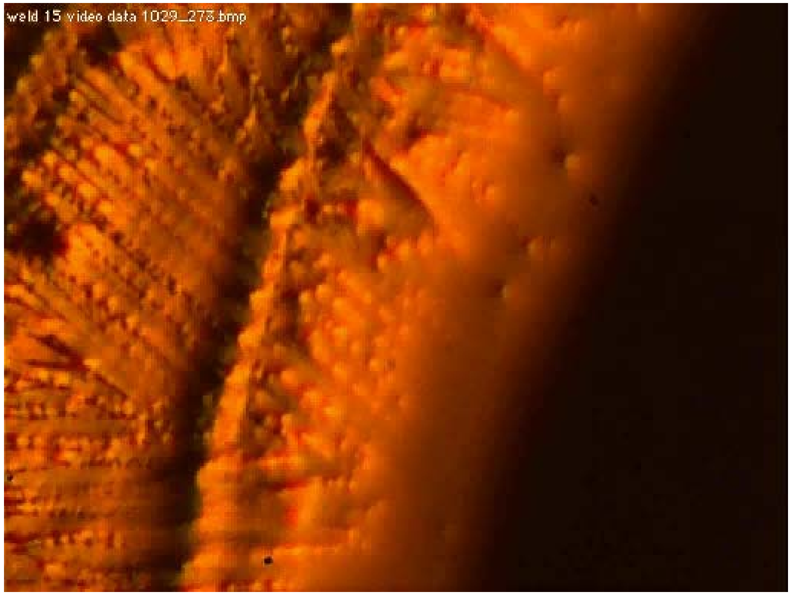
- David Q Wang (formerly at ORNL), P. G. Radaelli (ISIS), and A. C. Hannon (ISIS)

Outlines

- Background
  - Integrated Computational Weld Modeling
  - Thermo-mechanical-chemical-physical properties.
  - Approaches for In-situ Studies
- Experimental Case Studies
  - Nickel Base Superalloys – Phase Transformations
  - ISIS-GEM: Polycrystalline Alloys – Thermal
  - LANL-SMARTS: Single Crystal – Thermal-Mechanical
- Challenges: Scientific and Applied
- Future outlook
- Summary and Conclusions

Welding is one of the complex thermomechanical process that is dynamic in nature and often difficult to predict due to poor understanding of mechanisms.

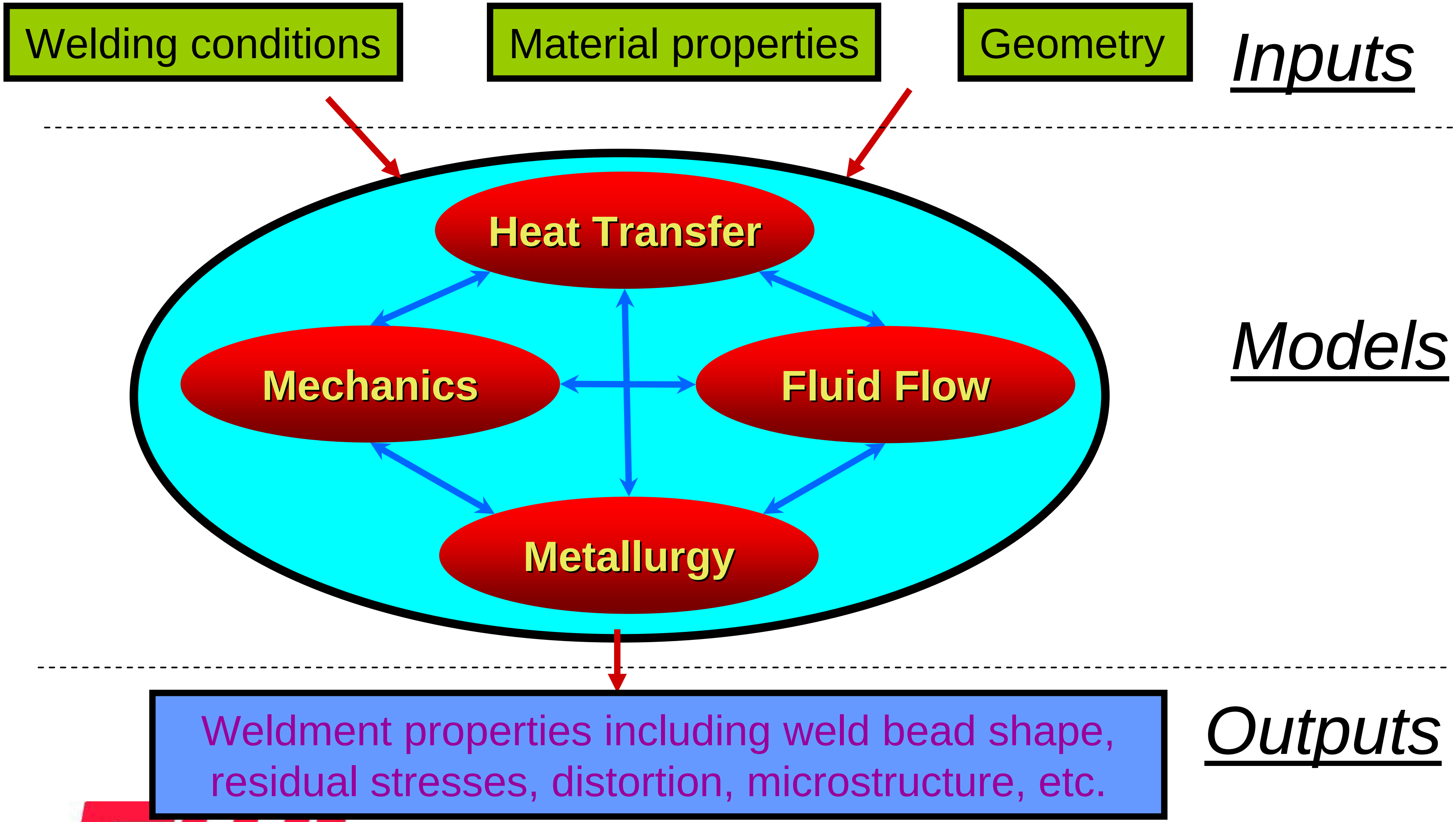


Liquid-solid interface during welding

Magnetic pulse welding of tubular

- The wide range of processes lead to interaction between different physical process. How can we predict these interactions?

Integrated modeling framework is based on considering the interactions between different physical processes.



Welding conditions

Material properties

Geometry

Inputs

Heat Transfer

Mechanics

Fluid Flow

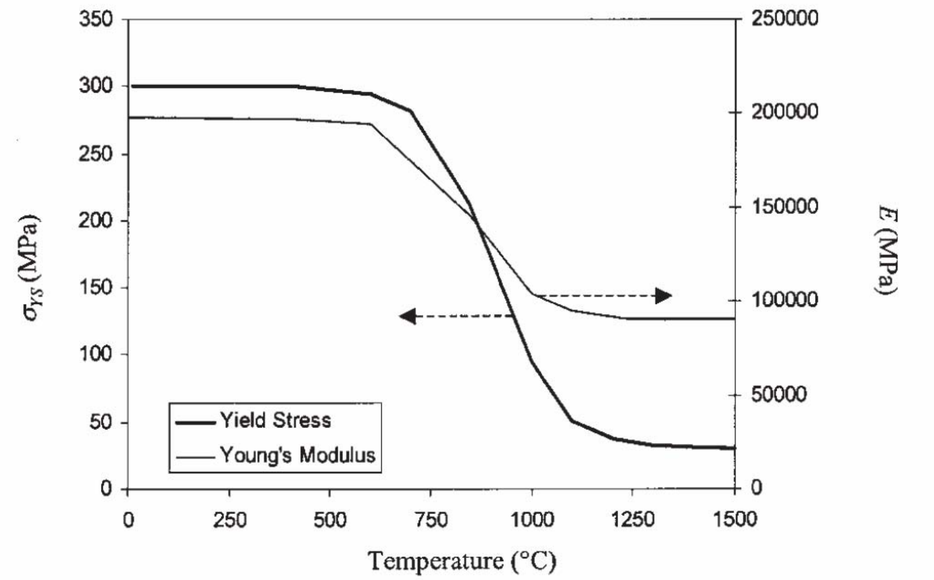
Metallurgy

Models

Weldment properties including weld bead shape, residual stresses, distortion, microstructure, etc.

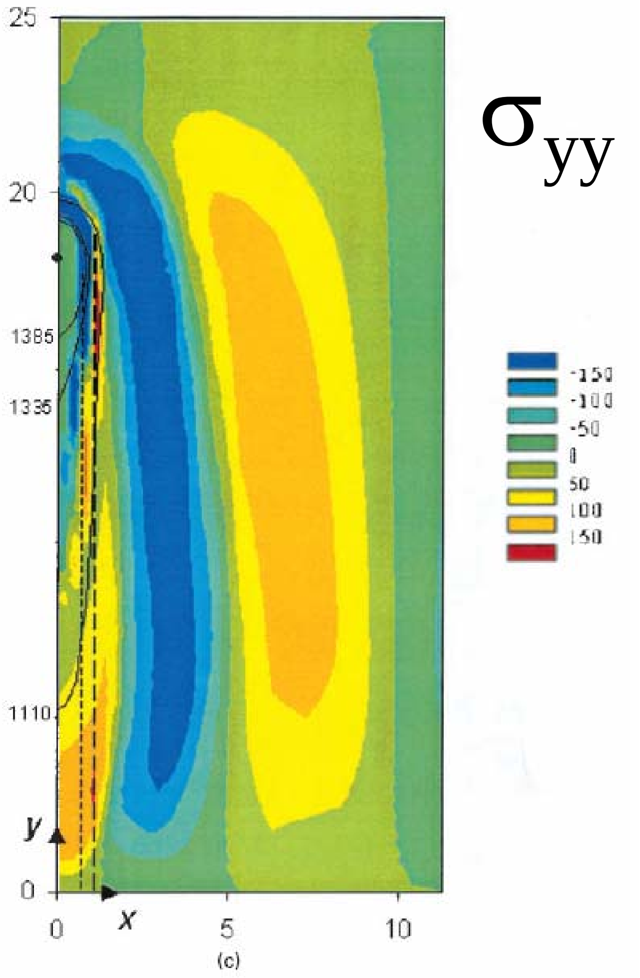
Outputs

The modeling results are very sensitive to thermo-physical-chemical-mechanical properties.



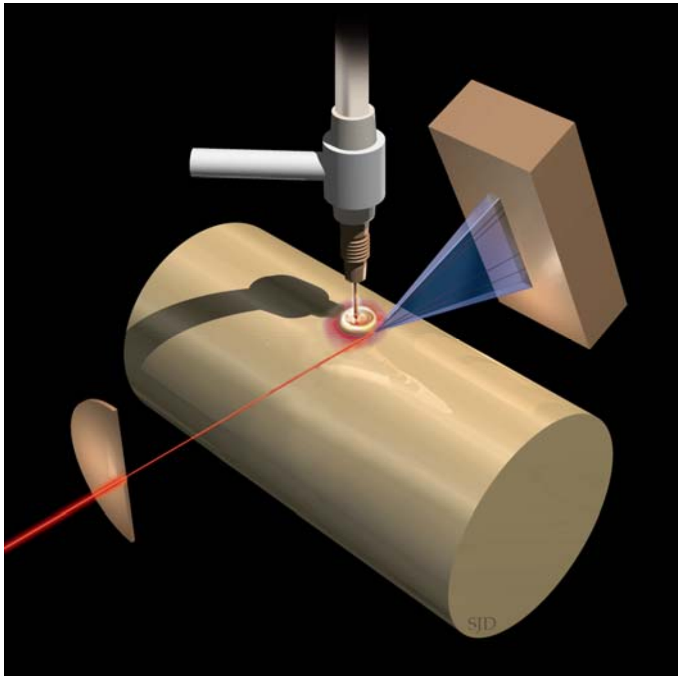
13 **Yield stress (σ_{YS}) and Young's modulus (*E*) as a function of temperature**

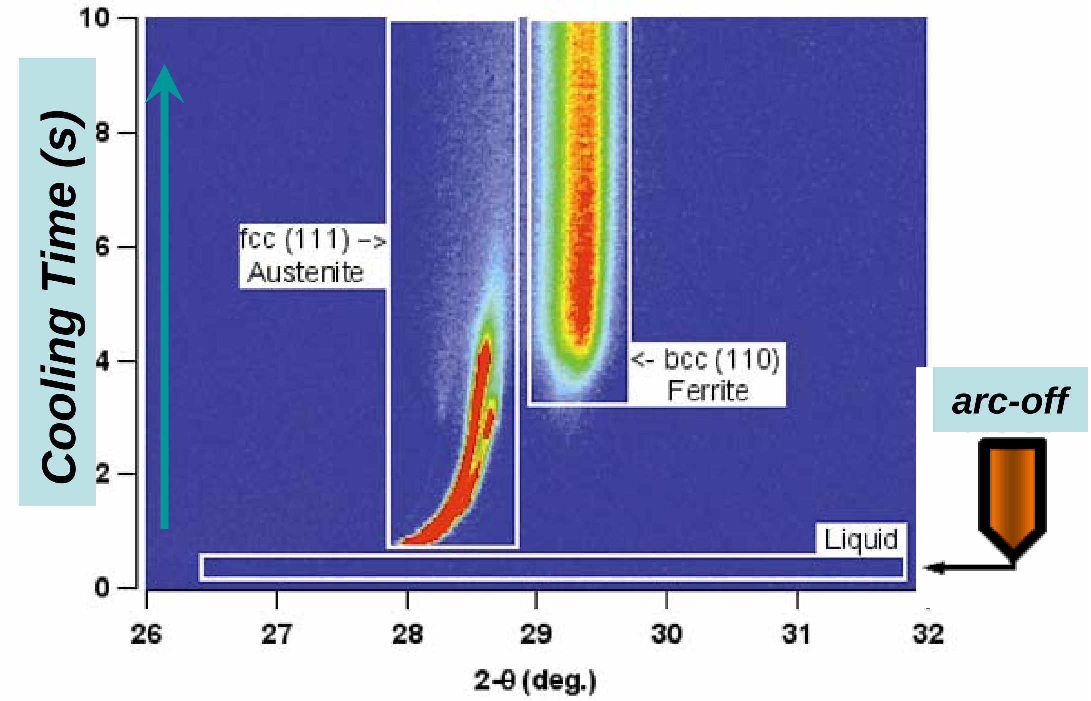
- Most of these properties are measured under equilibrium conditions.



σ_{yy}

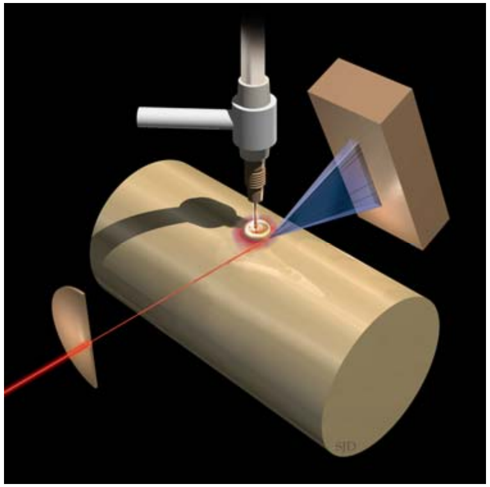
Using in-situ diffraction tools we can address this important need.

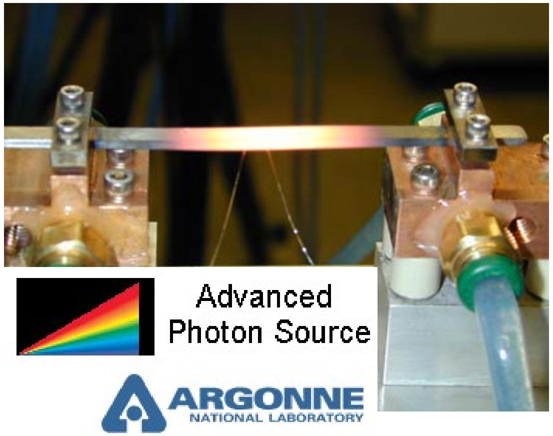




- For example, using in-situ synchrotron X-ray diffraction technique phase selection during weld solidification was monitored at a time resolution of 0.05 s!

In the last decade extensive work has been performed using in-situ synchrotron- and neutron scattering tools.





Advanced Photon Source

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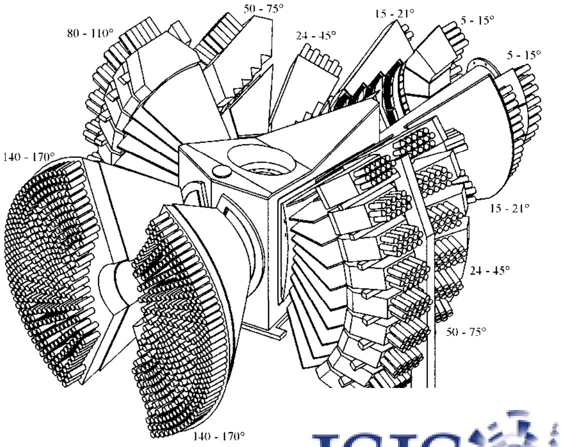

ISIS

STANFORD SYNCHROTRON RADIATION LABORATORY

SSRL

GEM
General Materials Diffractometer

- There is a need to continue these studies under controlled conditions; e.g. thermal, stress, magnetic field and environment conditions.

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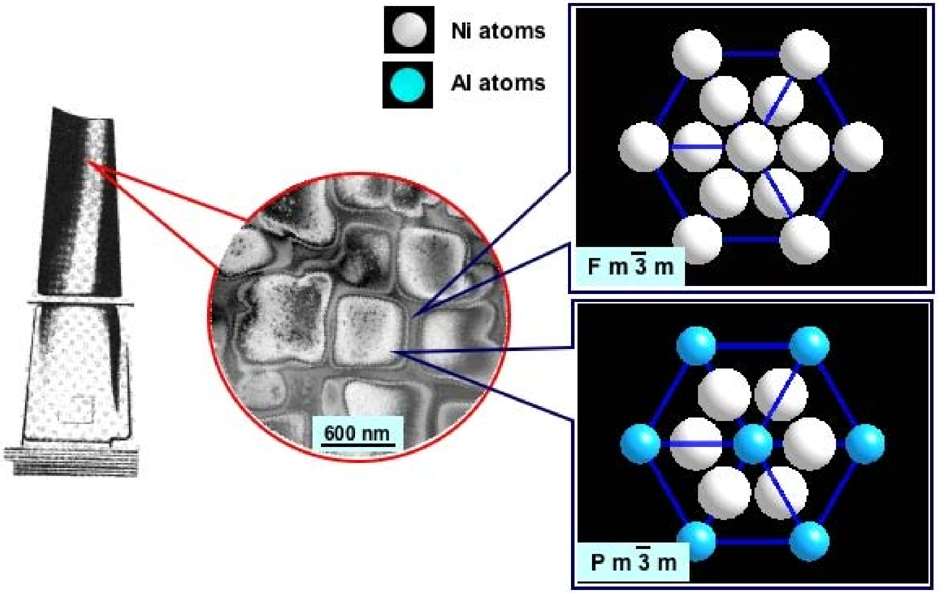
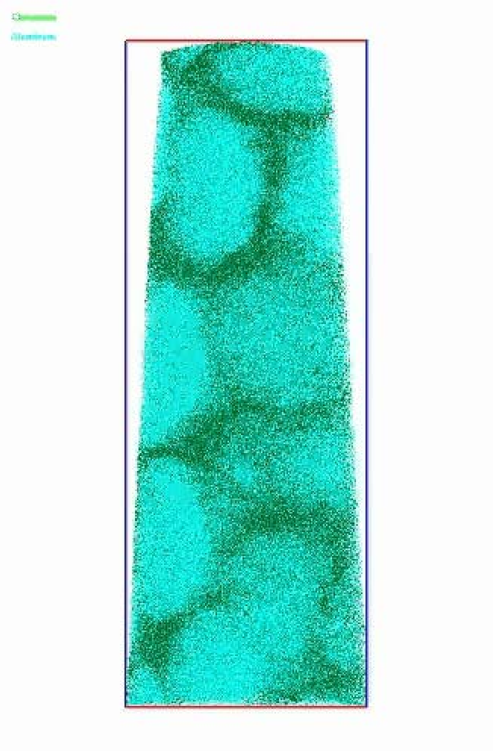
Performance of nickel base superalloys is related to the presence of γ’ precipitates in γ matrix.





- Composition, morphology and lattice misfit of these precipitates are crucial parameters.
- Question: How does different thermo-mechanical processing affect these parameters?

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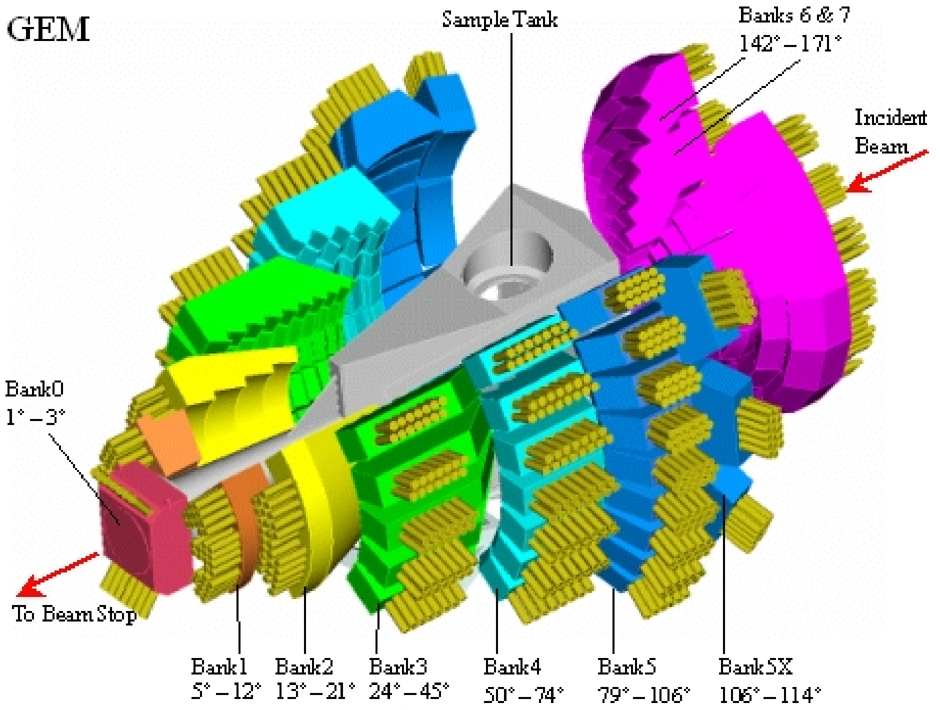
Case Study 1: How does initial microstructure affect the lattice misfit between the γ and γ' phases during heat treatment?

- Polycrystalline CM247CC alloy:
  - Ni- 8Cr - 9Co - 5.5Al - 0.8Ti - 0.1Nb - 0.6Mo - 3.2Ta - 9.5W - 0.08C wt. %
- Lattice misfit was studied as a function of isothermal holding time at 1000^{o}C.
- Condition 1: Solutionized at 1290^{o}C for 5 minutes and water quenched & heated to 1000°C and held isothermally (Nonequilibrium microstructure).
- Condition 2: Heated to 1190°C, cooled to 1000°C at the rate of 0.018°Cs^{-1}. (Equilibrium microstructure).

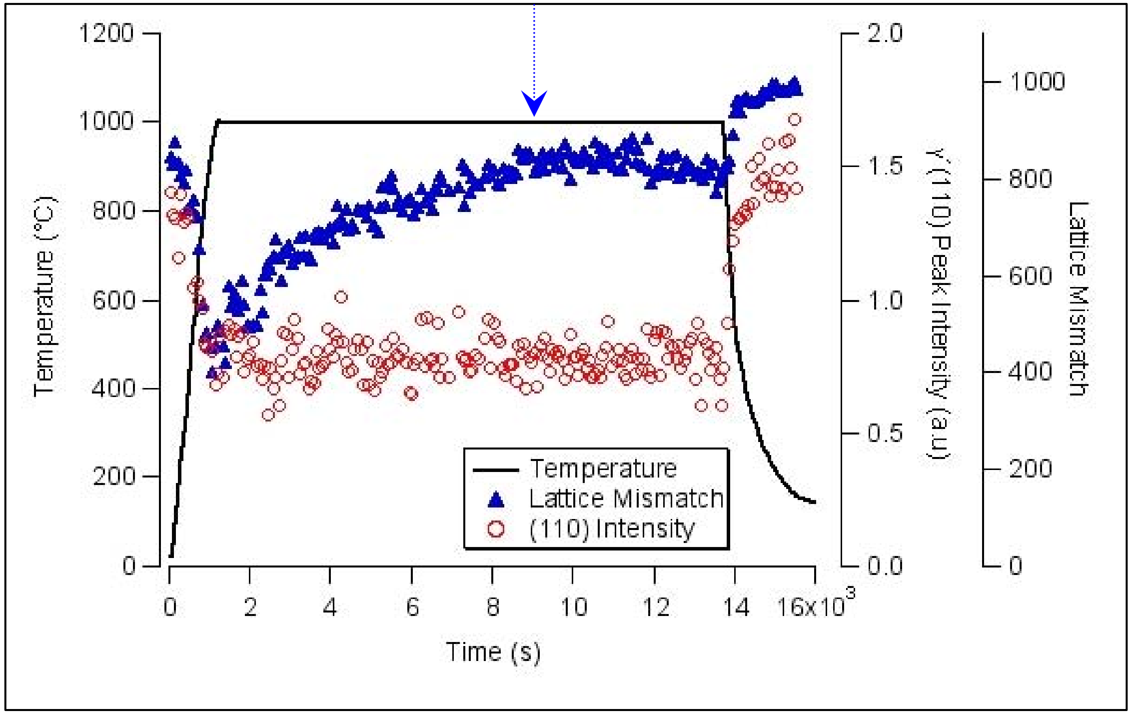
Heat treatments were conduced in-situ in GEM (ISIS) instrument while measuring diffraction data.

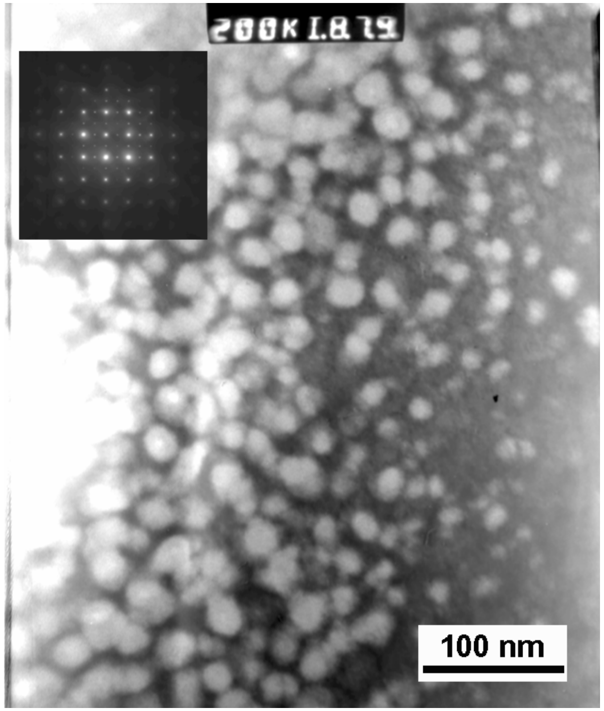
- Diffraction spectra were collected at 1-minute intervals using three banks of detectors placed nominally at 63.6°, 91.3° and 154.4° in 2θ.
- Quality of these diffraction spectra allowed for whole pattern analyses using the GSAS program.



$$\delta = \frac{(a_{\gamma'} - a_{\gamma}) \times 10^{6}}{a_{\gamma}}$$

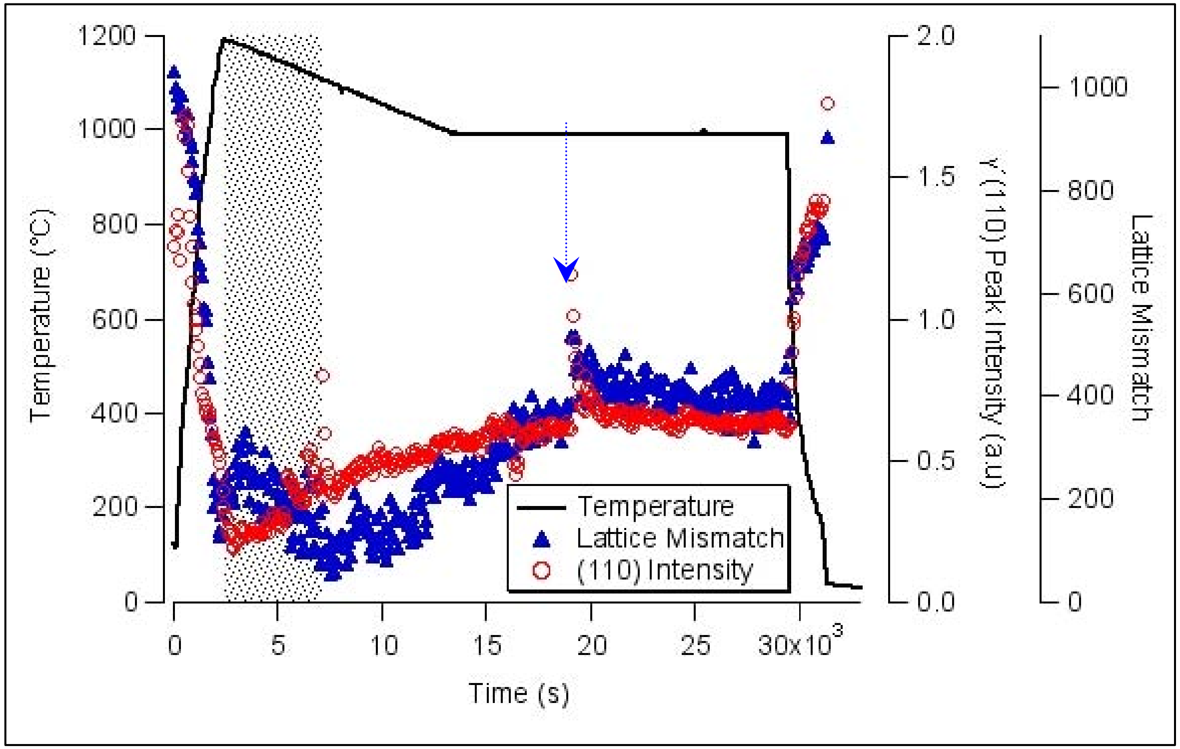
Nonequilibrium Microstructure: Lattice misfit continues to change for extended period of time after reaching the isothermal transformation temperature.

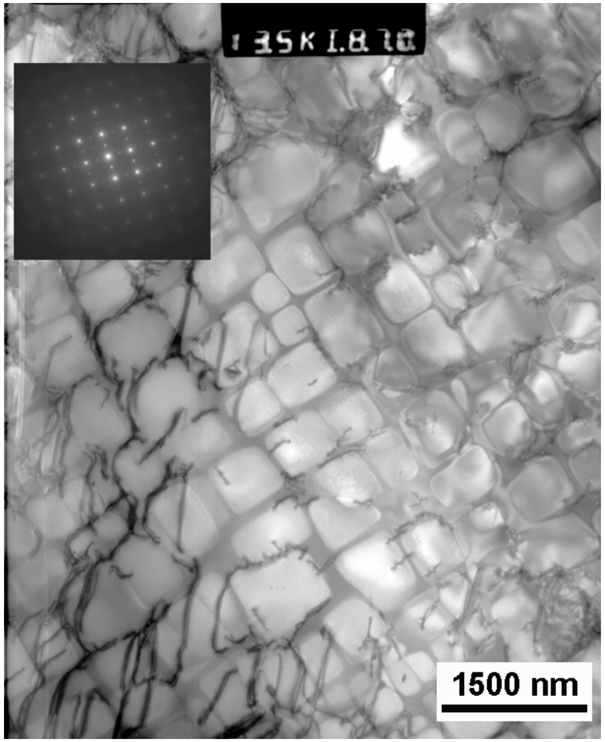




- How about equilibrium microstructure?

Equilibrium Microstructure: Lattice misfit changes stabilizes quickly after reaching isothermal transformation temperature.





- Implication: Lattice mismatch or the properties of these alloys are path dependent.

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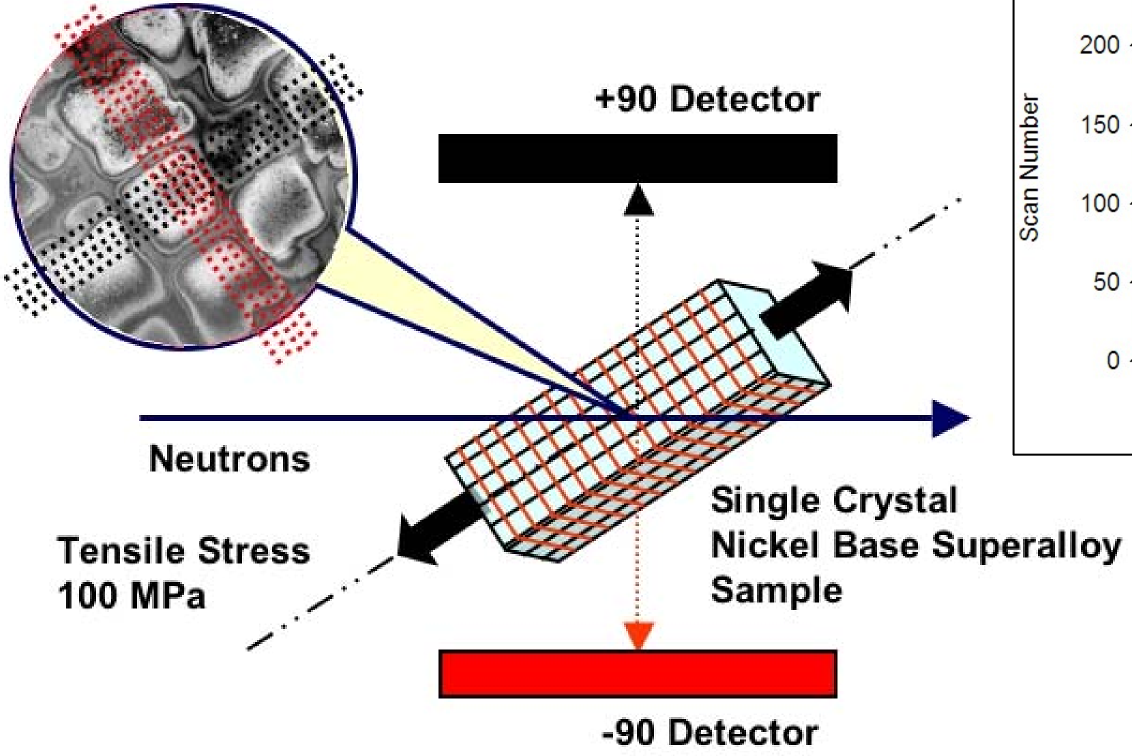
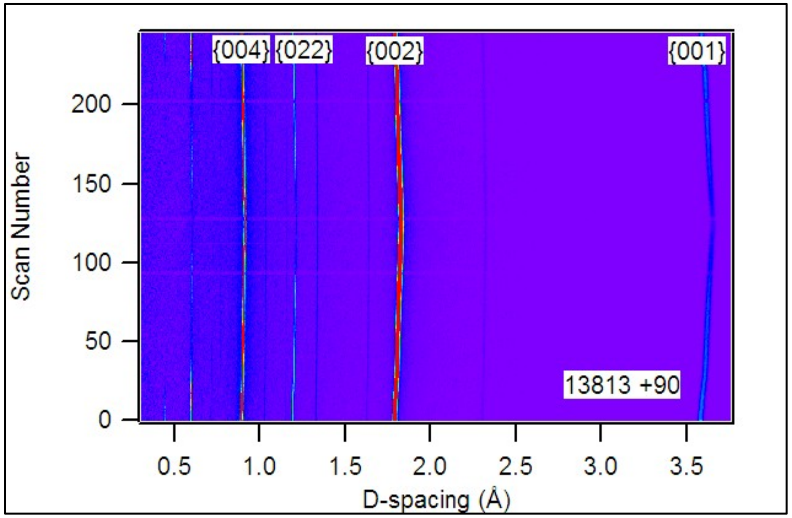
Case Study 2: How does stress affect the phase transformation and lattice-strains in the γ’ precipitate?

- PWA1480 Single Crystal Superalloy
  - Ni-11.0Al-11.5Cr-1.9Ti-5.1Co-4.0Ta-1.3W (at.%)
- Heated 1473 K at a rate of 2.7 Ks^{-1} and cooled at the same rate to room temperature.
- One of the samples was loaded to ~ 100 MPa of tensile stress before cooling to room at high temperature.
- The TOF positions and intensity of diffraction peaks of both γ' [{001}, {002}, and {004}] and γ [{002} and {004}] phases were measured *in situ* at a time resolution of 3 minutes.

Thermomechanical simulations were performed in-situ with SMARTS instrument while measuring diffraction at two different detectors.

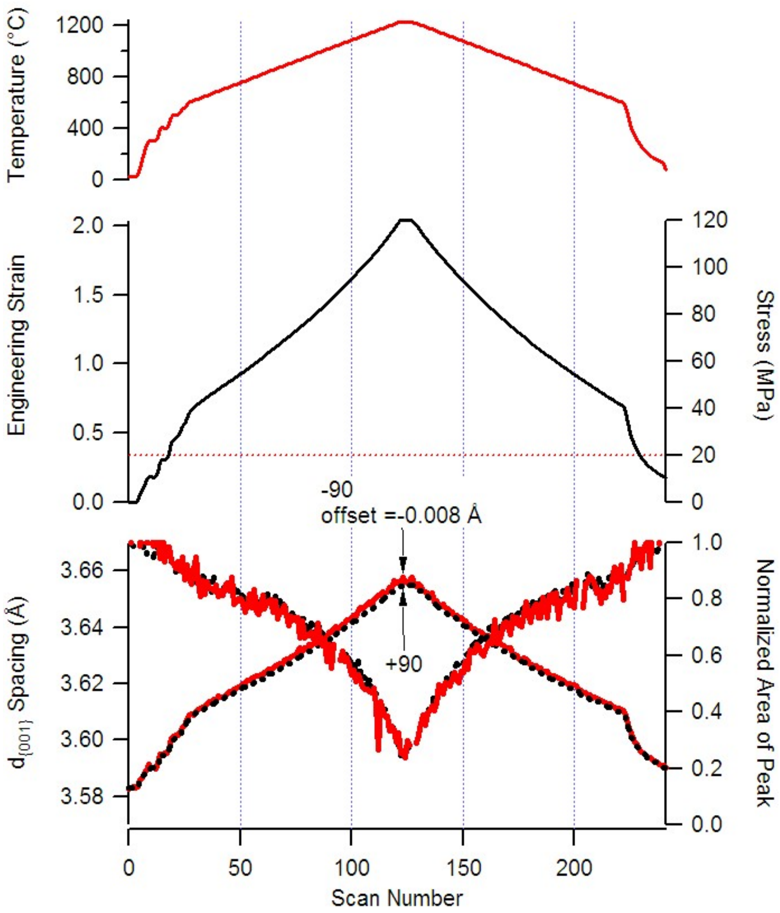


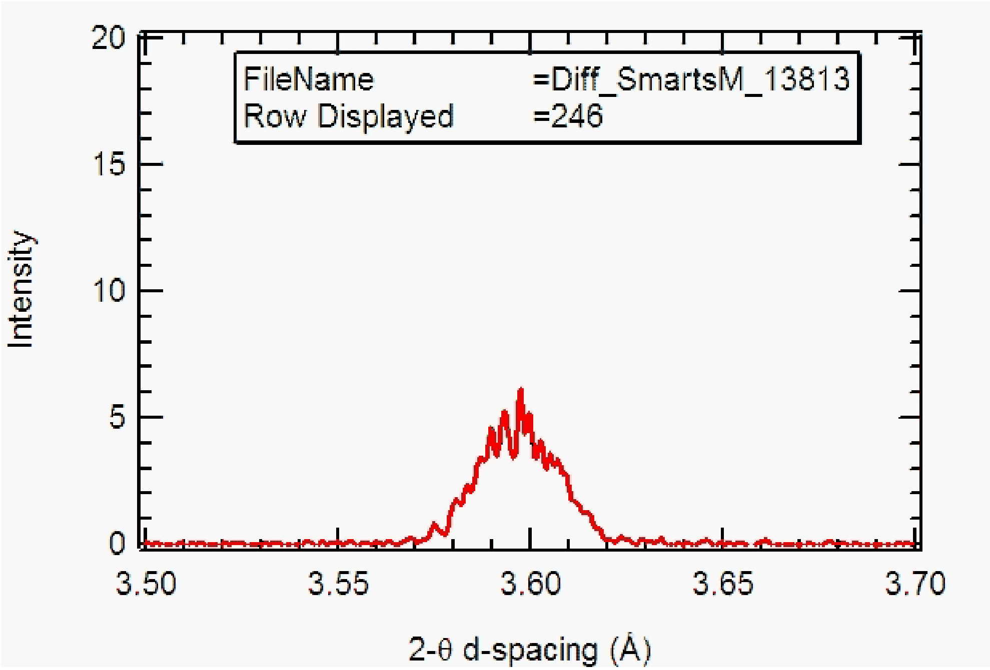


- Pre-alignment of samples were done by traditional X-ray diffraction at ORNL.

- While doing this experiment, the engineering stress-strain is also measured.

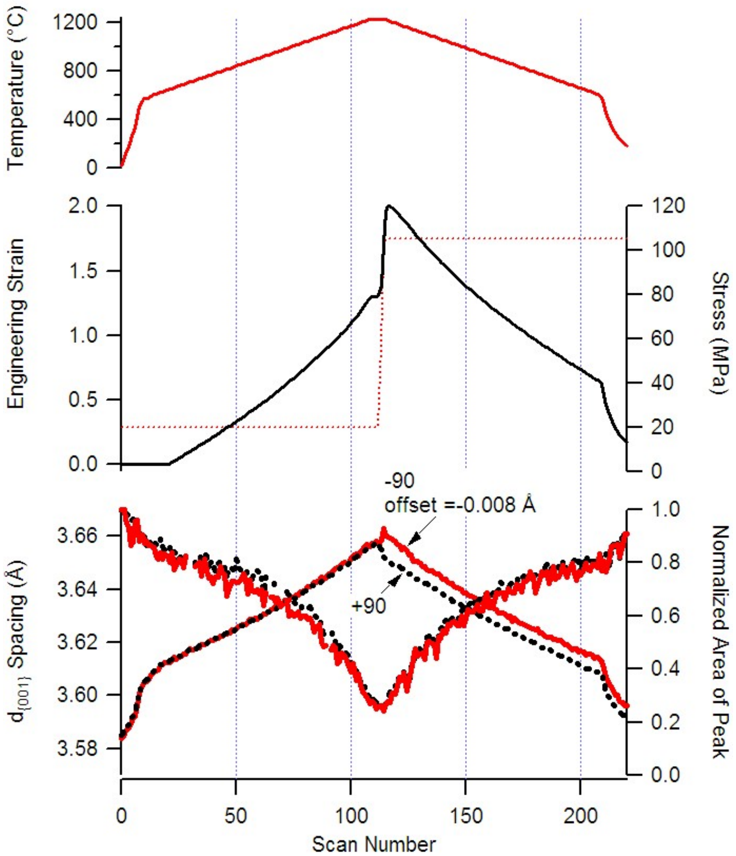
Diffraction data from γ' precipitate with small (20MPa) external tensile load shows only dissolution and growth of γ' during heating and cooling.

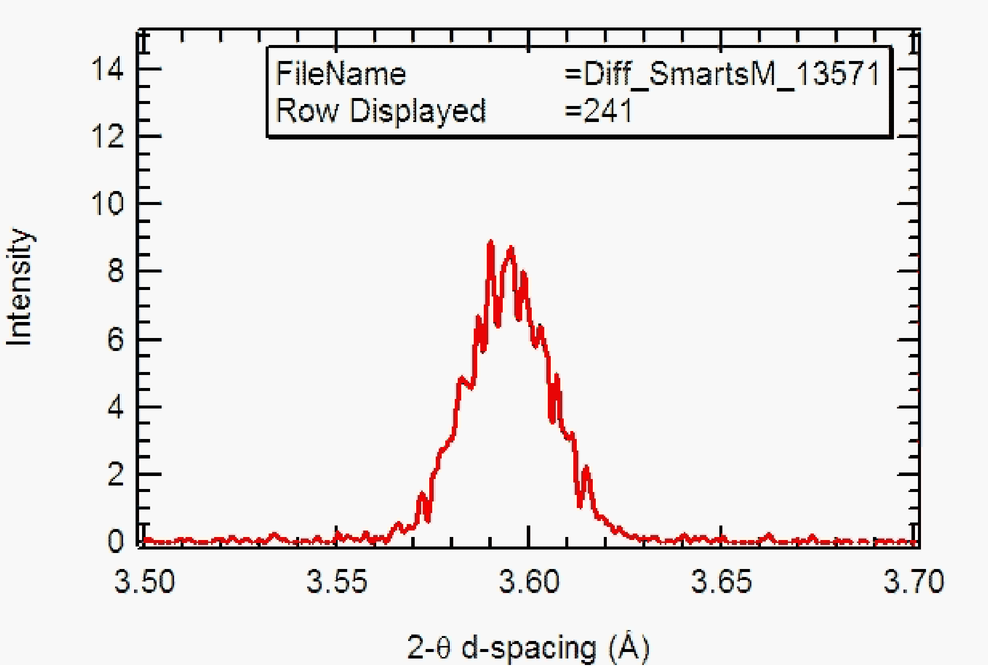




- The data from +90 and -90 detectors are almost identical!

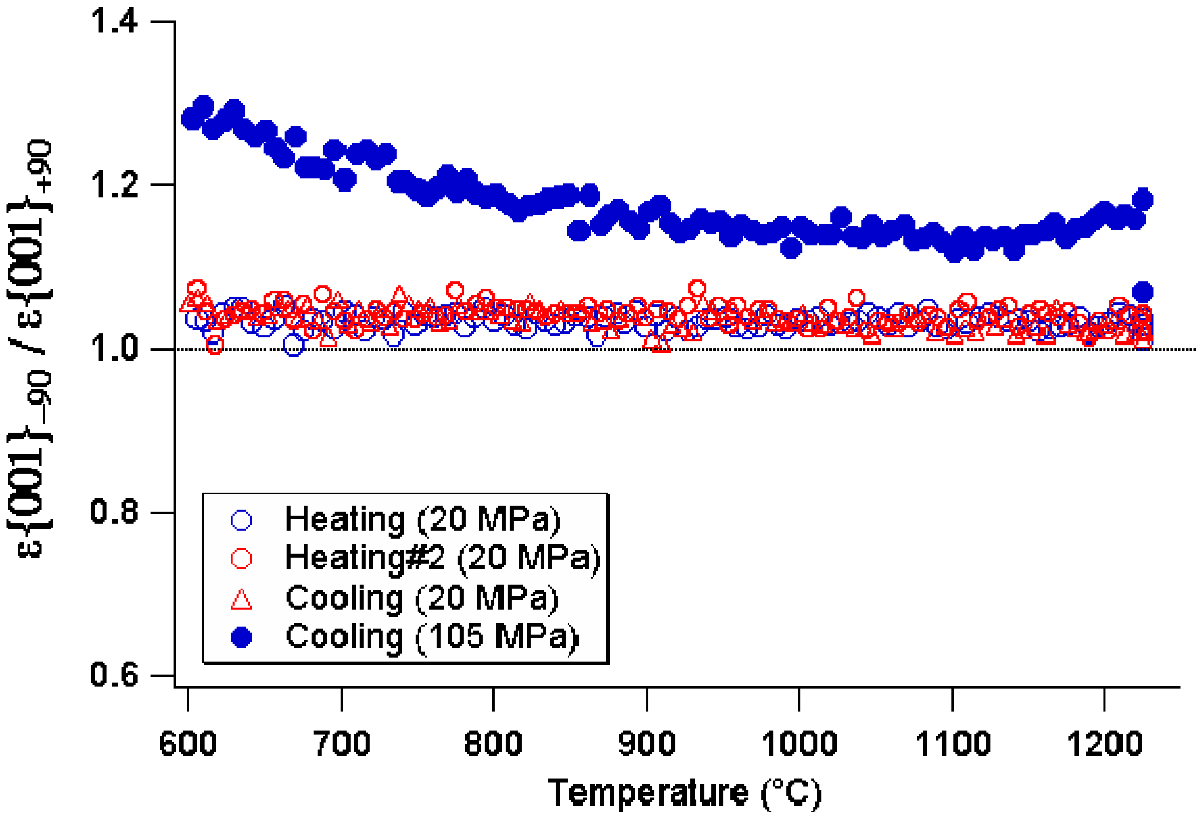
Experiment with 100 MPa tensile load shows dissolution and growth of γ’ during heating and cooling and differences in the +90 and -90 detector data.





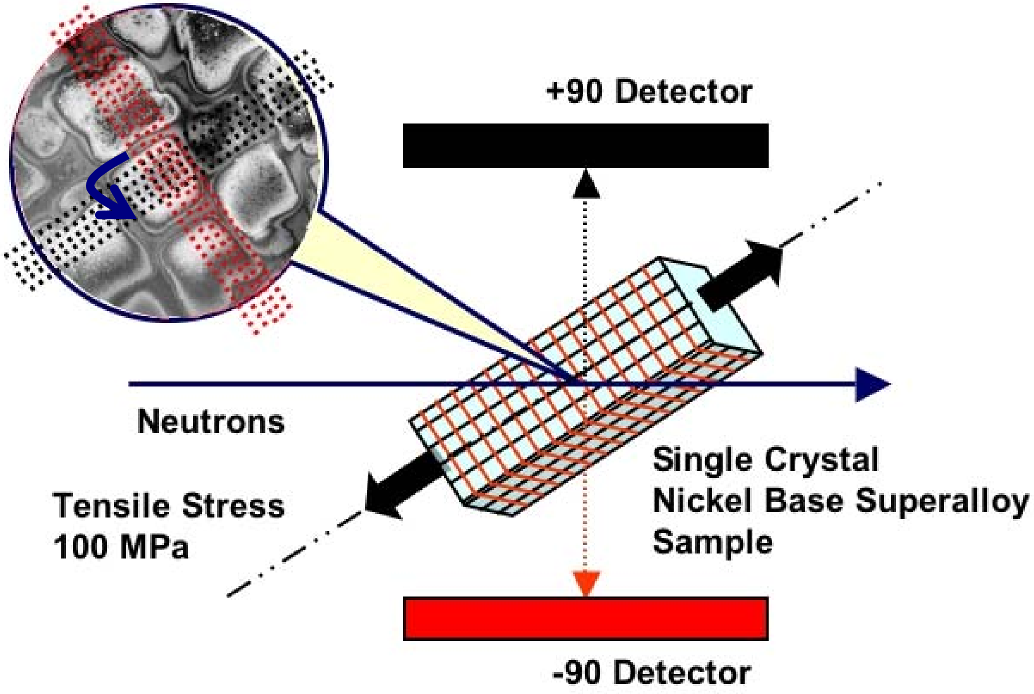
- Is this just a Poisson's ratio effect?

Ratio of lattice strains while cooling with 100 MPa load shows nonlinear behavior.



- Identical ratio while heating and cooling with no external load.

Implications: The lattice parameter of γ’ may change due to local changes in the γ channel compositions driven by applied load – Constraint effect.



- Further work is necessary to evaluate this effect.

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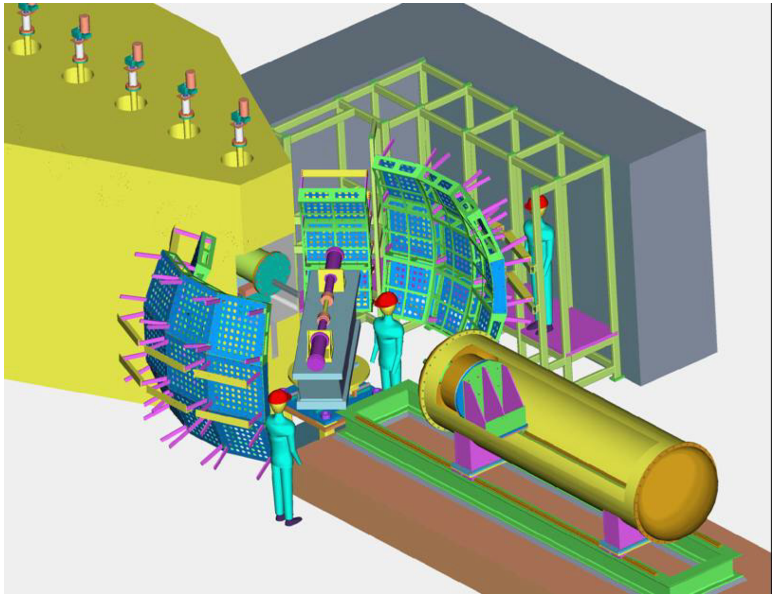
Challenges from a materials science point of view.

- Can we impose other effects during measurements?
  - Magnetic fields: Recent work shows acceleration of austenite to pearlite transformation.
- Can we track the microstructure? Similar to 4-D SRXRD microscopy?
- How do we decouple the effects of plastic strain and elastic strain and temperature effects?
- Can we do simultaneous small-angle scattering and diffraction studies?
- Can we do real-time dynamic stress mapping of during welding?
- How can we increase the temporal resolution to microseconds?
- How can we increase the spatial resolution simultaneously while increasing the temporal resolution?
- How can we increase the data analysis rates (full peak analysis)?

Future outlook: Engineering Diffractometer (SNS-VULCAN) and Newly updated HFIR may allow us to address these questions.



- http://www.sns.gov/users/instrument_systems/instruments/elastic/vulcan.shtml
- Operating: 30 Hz!
- 0.1 mm spatial resolution!
- Poor man’s SANS
  - 0.01 to 0.18 Å^{-1}
- Transmission Bragg Edges
  - Composition

A challenge for ourselves: Fundamental understanding of solid-state welding or joining of advanced materials.

- I hope by 2015 we will be able to analyze the plastic flow and stress build up in a dissimilar ODS steel to Al-MMC joint made by Friction Stir Weld or magnetic pulse welding by combination of Synchrotron and Neutron Scattering tools!

Summary and Conclusions

- Measurements of phase transformations as a function of thermo-mechanical processing are needed to develop better models to optimize the structural materials performance.
- In-situ neutron and synchrotron scattering tools allows us to understand the fundamental mechanisms.

- ISIS-GEM instrument allowed us to track lattice misfit in a polycrystalline nickel base superalloy as a function of temperature and initial microstructure.
- LANL-SMARTS instrument allowed us to track lattice strain in single crystal nickel base superalloy as a function of temperature and load.

- With future developments in SNS-VULCAN and HFIR we will be perform more detailed analysis of materials during and after welding/joining.