Ex-situ and In-situ Characterizations of Phase Transformations in Multi-Length Scales

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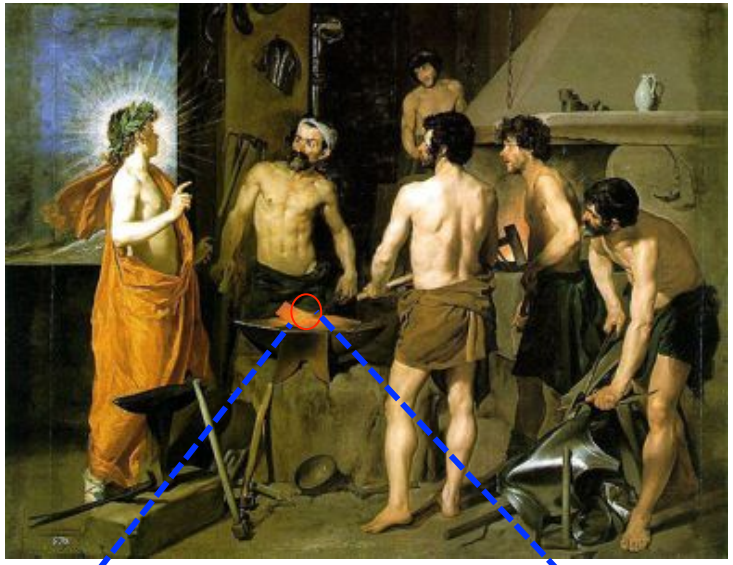
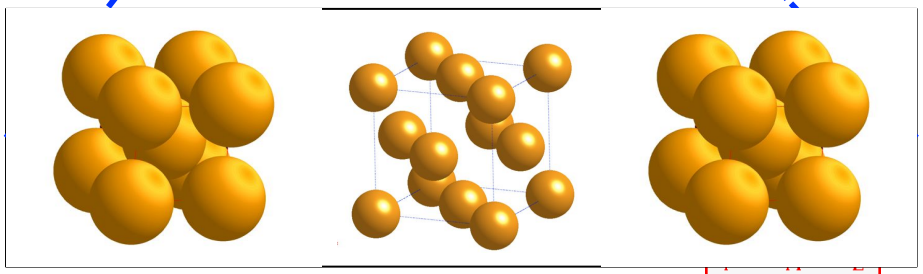


Ref: The **Forge of Vulcan** – Diego Velasquez (1630); Courtesy Wikipedia



Acknowledgements

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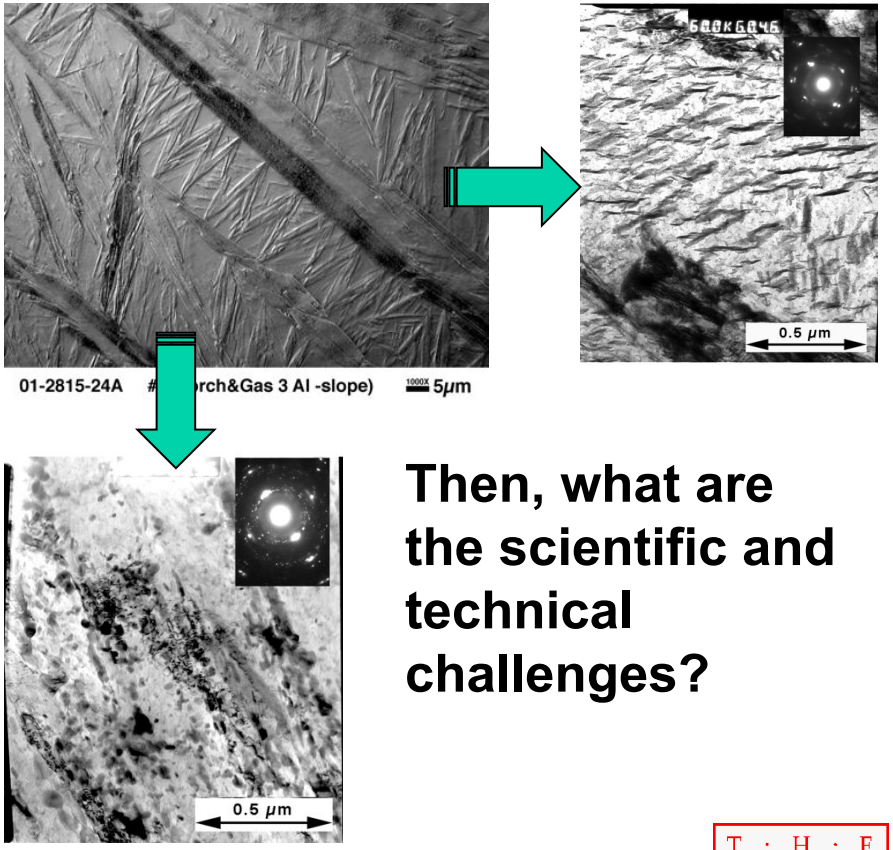
- John Elmer (LLNL), D. Brown (LANL), Colleagues at ORNL & BES who inspired to me get into the field of ex-situ and in-situ characterizations
- Thanks to Dr. X. Wang for inviting me to this workshop

Outline

- What are transformations in different length scales?
- Heat affected zone of BlastAlloy-160 ®
  - Recent Ex-situ Characterization
- Relevance to SNS-Vulcan Instrument
  - Proposed Experiments
  - Impact of the proposed Experiment
  - Future Needs
- Summary and Conclusions

Metallurgists developed structural materials by manipulating phase transformations in different length scales through controlled heat treatments

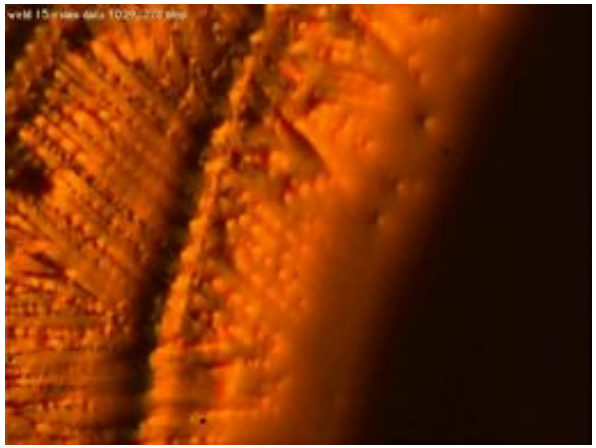
- How to leverage fine grain size effect & precipitation?
- <u>Quench (fcc->bct)</u> : Form fine martensite sub-structure; but brittle structure!
- <u>Temper</u>: to reduce the carbon and precipitate fine carbides (bcc + M_xC_y)

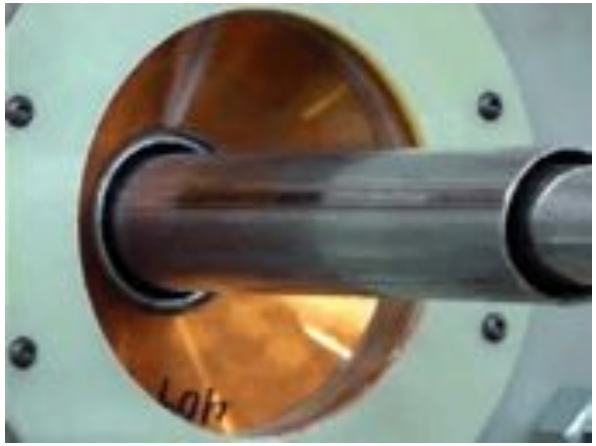


Then, what are the scientific and technical challenges?

These materials need to be joined for deployment in service:

- Joining processes involves wide range of thermo-mechanical-metallurgical transients:
  - dT/dt: >10^3 °Cs^{-1}
  - dT/dx: >10^5 Km^{-1}
  - dε/dt: > 10^5 s^{-1}
- Traditional thermodynamic and kinetic models based on "close to equilibrium conditions" do not work very well.
- How can we address these challenges?





Approaches

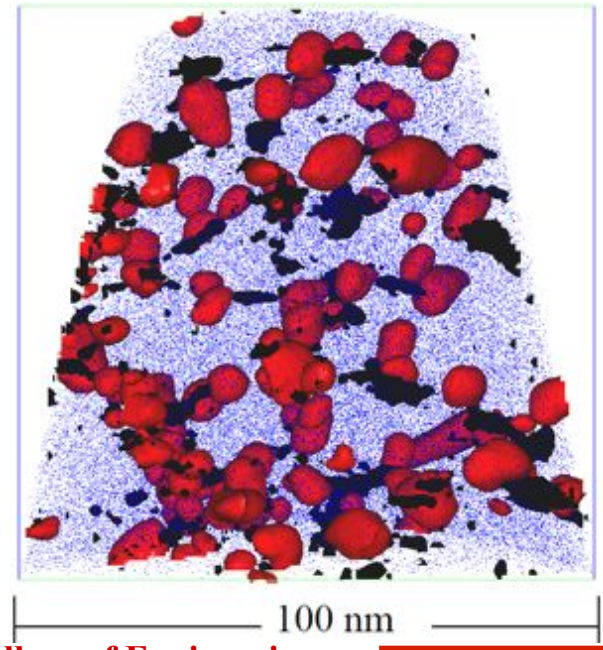
- Trial and Error Experimentation
  - Non-optimal solutions, Expensive and time-consuming, Deployment of new materials delayed at least by 20 years
  - This is a proven route!
- Integrated Computational Modeling
  - Assumptions (e.g. local equilibrium at interface) are made for non-equilibrium conditions
  - Fine tune the assumptions using ex-situ and in-situ characterization tools
  - Still expensive and time-consuming
- Let us demonstrate this with an example from BlastAlloy-160® development.

Blastalloy160™ Steel

- High-strength steel designed for naval surface ship decks
- Composition (Wt.%) : 0.059C, 3.39Cu, 6.8Ni, 1.90Cr,0.61Mo
- Martensitic/bainitic matrix
- Strengthened by M_2C carbides and BCC Cu-rich precipitates (~3-5nm)
- Transformation toughening provided by dispersed Ni-stabilized austenite precipitates (0.1 phase fraction)

•Heat treatment

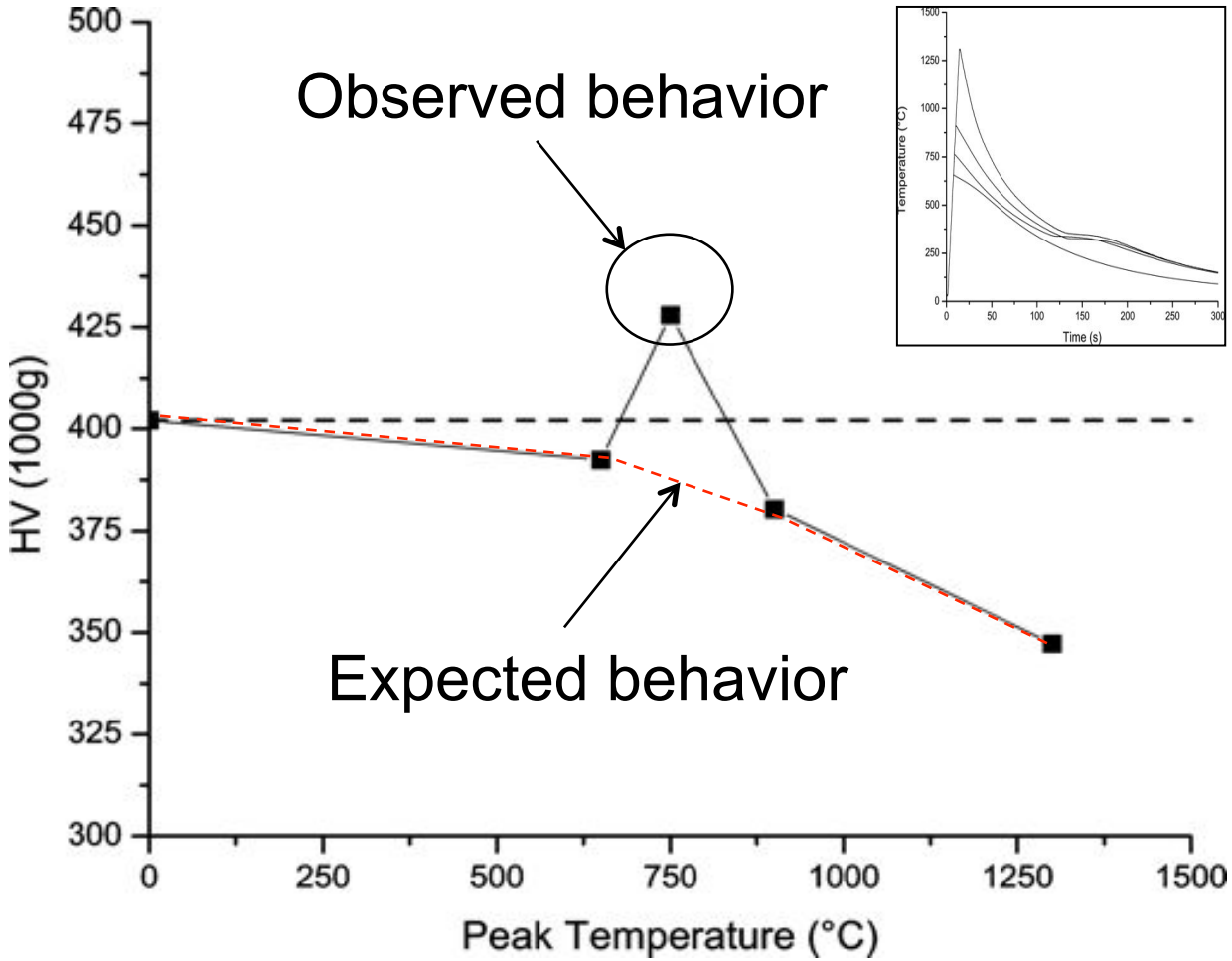
- •Austenitize 900°C/1hr, WQ
- •Liquid nitrogen hold 30min
- •Air warm to room temp
- •Temper 550°C/30min, WQ
- •Temper 450°C/5hr, AC



8%Cu
1%C
Isoconcentration surface
23M atoms

Courtesy M.D.Mulholland and D.N.Seidman

When subjected to a weld thermal cycles, anomalous hardening was observed.

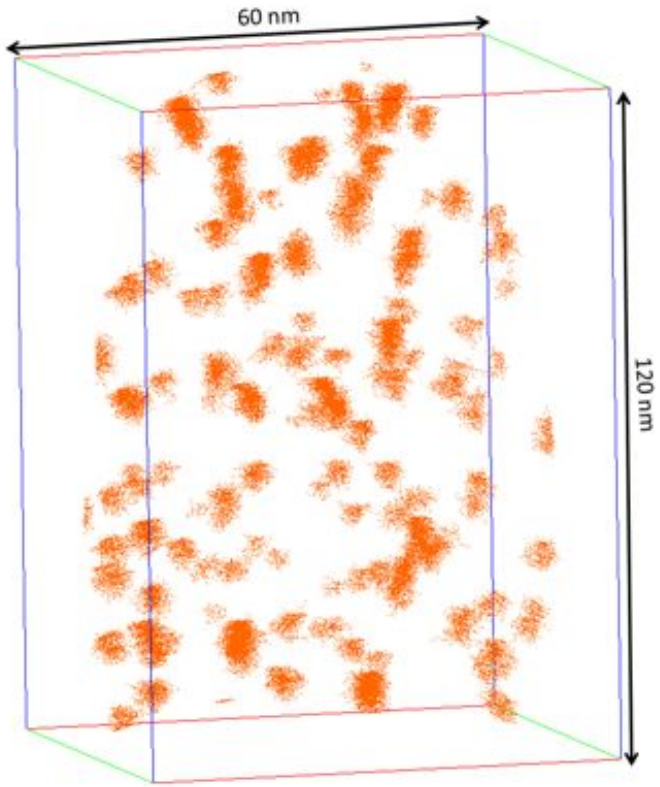


- **Why do we observe such hardening in and softening?**
- **Martensite matrix**
- **Precipitates (Cu and carbide)**

LEAP analyses confirmed the expected trend of copper precipitate dissolution!

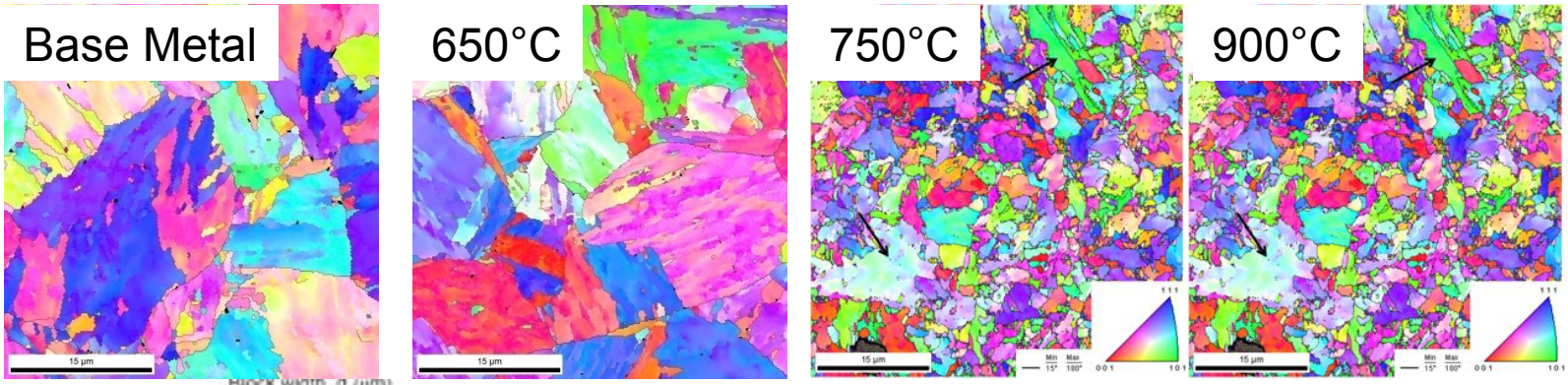
Base Metal Peak Aged Condition:
Radius: 2.4 nm
Number density: 4.2×10^{23} m^{-3}

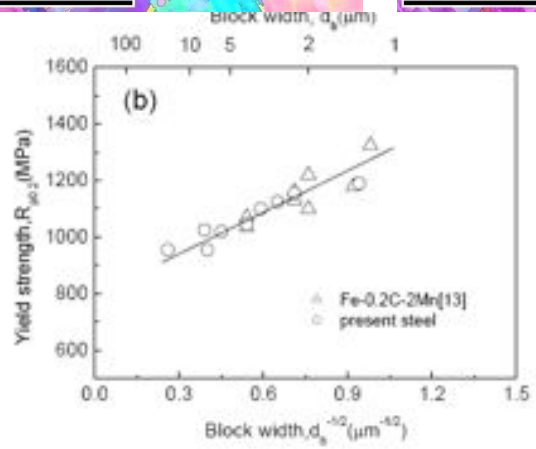
Heated to 750°C
Radius: 2.3 nm
Number density: 2.84×10^{23} m^{-3}

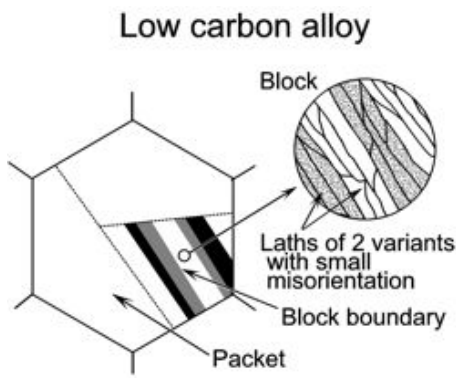


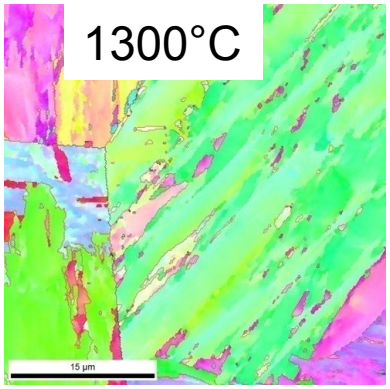
- **Still we cannot explain the strengthening.**

Electron back-scattered diffraction imaging shows drastic changes in martensite sub-structure (block size)









- **So What?**

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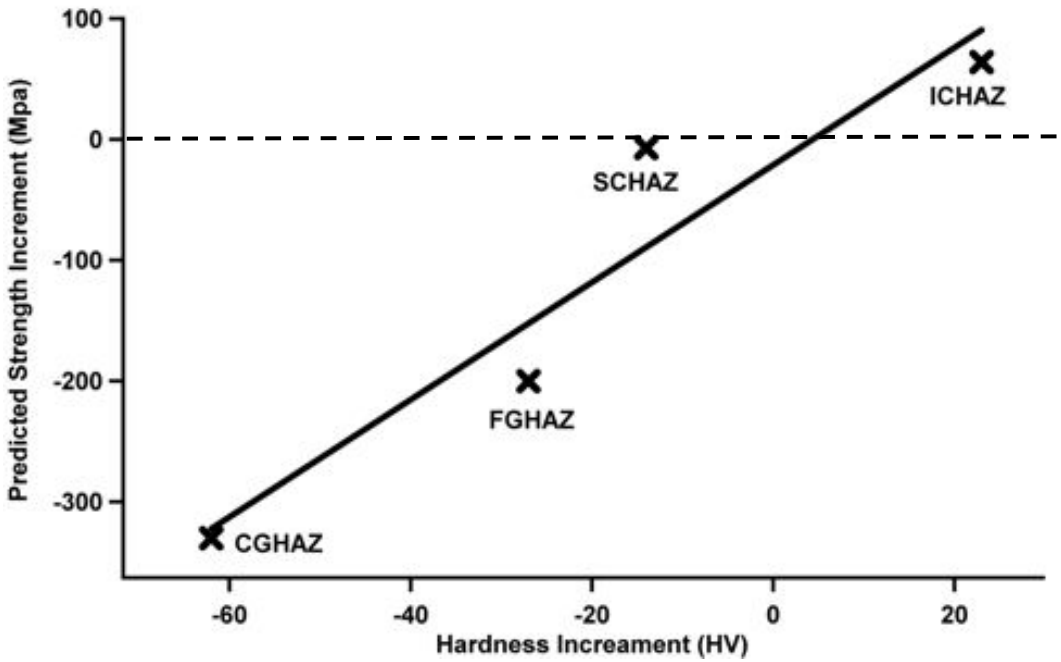
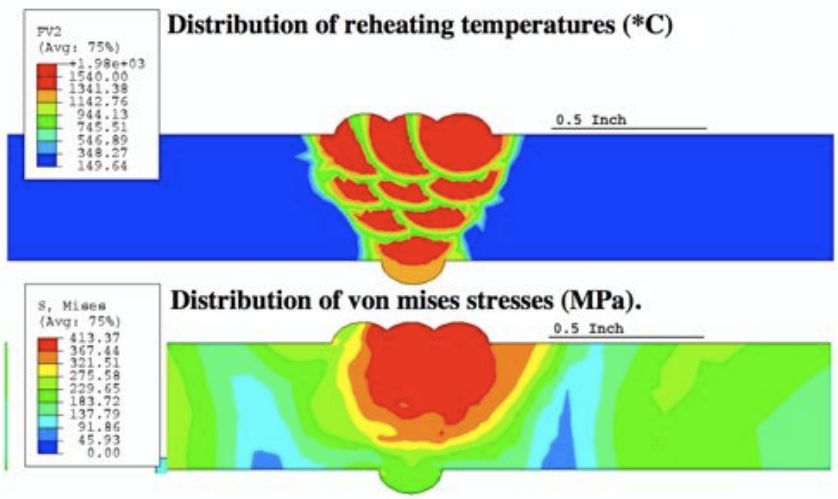
Comparison of strengthening and softening with reference to base metal



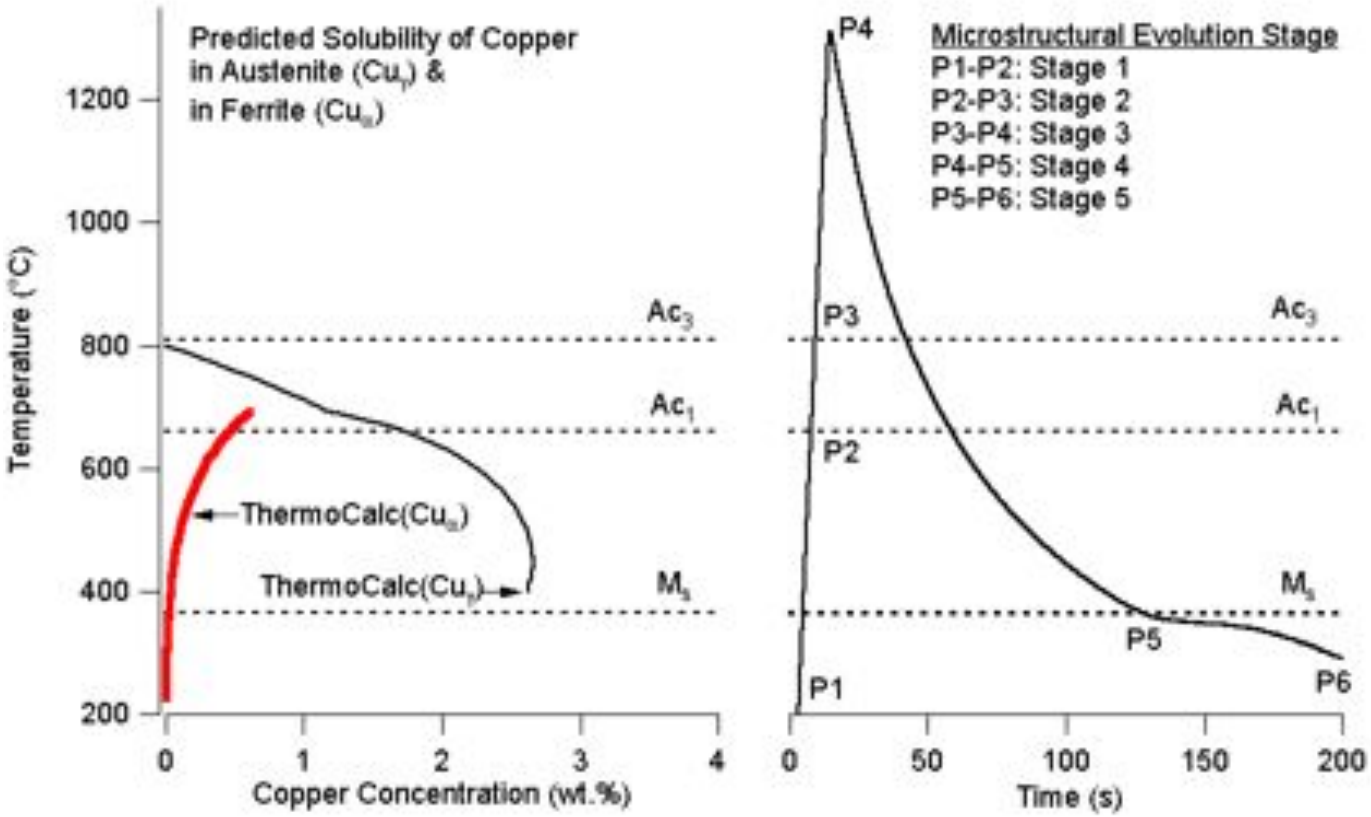
- **Research has explained the observed hardening and softening behavior in HAZ!**
- **So what are the scientific and technical challenges for deploying the BlastAlloy-160® to welded structures?**

Typical welded structure involves complex and many thermo-mechanical cycles

- Cannot generalize our results to all these regions.
- Cannot do exhaustive characterizations
- Existing computational thermodynamic and kinetic tools need calibrations
- So what is the proposed path?



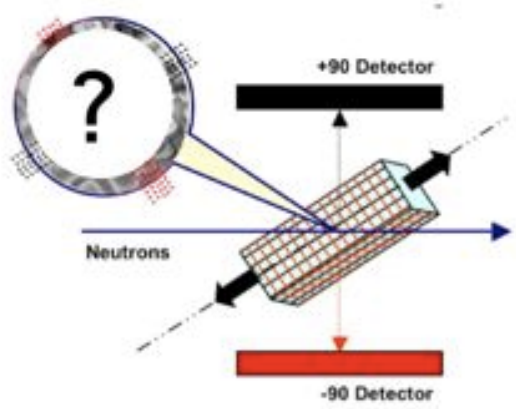
We need to track the microstructural transients under in-situ conditions

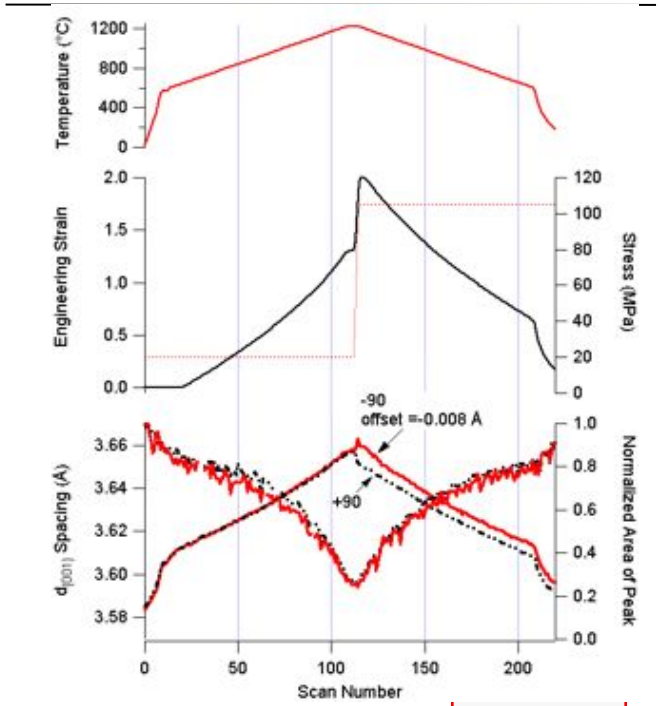


- **What will be the proposed experiment?**

Proposed experiment:

- Predict thermal and stress transients using computational weld mechanics models for critical locations
- Apply these (T & σ) variations on a well defined geometry
- Track lattice strains and phase transformations in different detectors at 0.01 to 0.1 second intervals.
  - On-heating transformations (BCC-Fe to FCC-Fe; BCC-Cu to FCC-Fe)
  - Austenite Recrystallization and grain growth (peak shapes & texture)
  - Austenite to Martensite Transformation (possibility of transformation induced plasticity)
- What is the difference between this and Synchrotron experiments?





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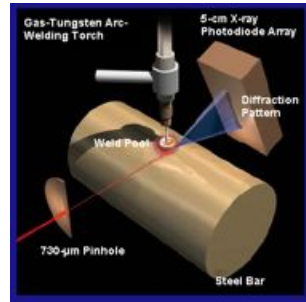
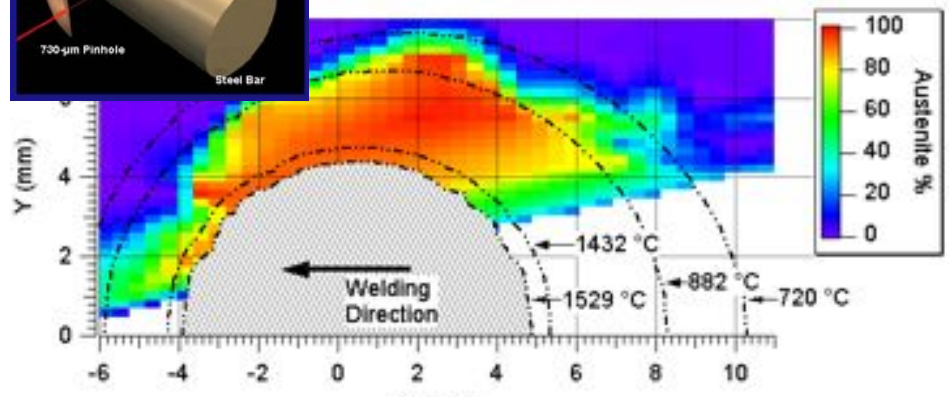
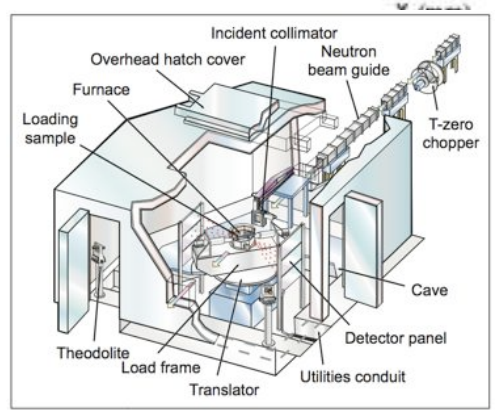
Why can't we do the same in Synchrotron? & Why not in SMARTS?

- **In-situ Synchrotron experiments:**
  - **Surface analyses only**
  - **Complex stress-states cannot be applied**
  - **Simultaneous measurements cannot be done**
- **Some of these experiments can be surely done in SMARTS**
- **SNS-Vulcan offers unique advantage of simultaneous measurements (e.g. dilatometry, future SANS)**



Ref: Elmer et al (1998-2008)

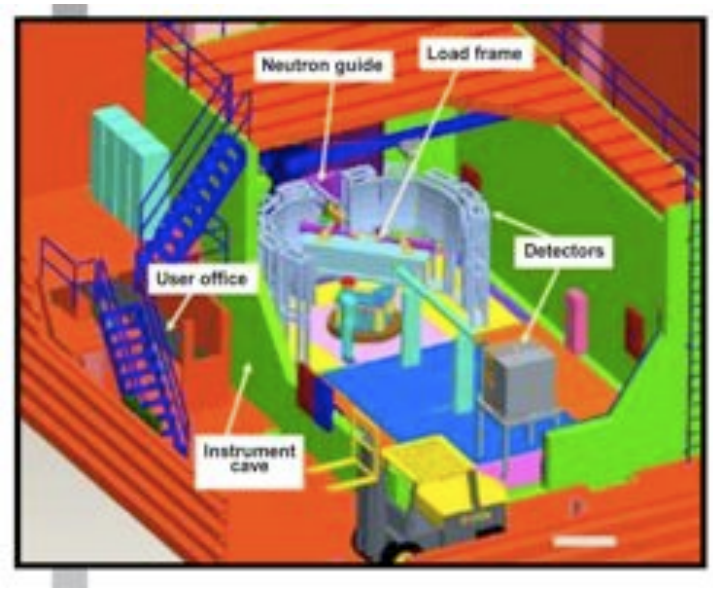




How about Copper precipitate evolution?

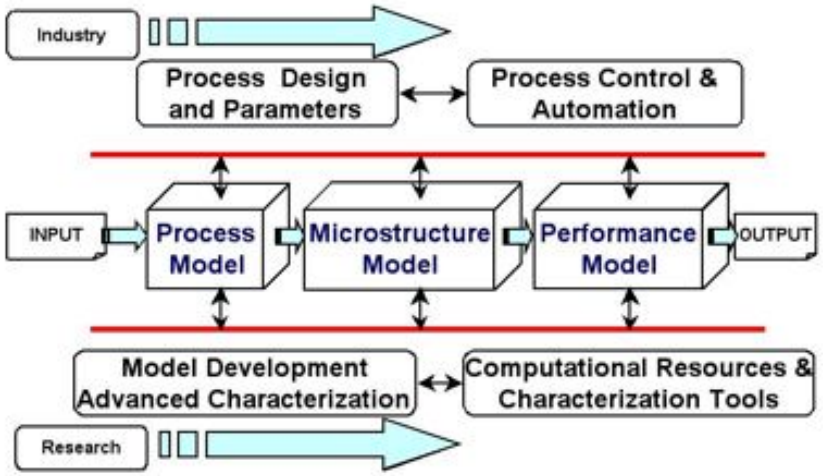
- There exists extensive research on Copper precipitate evolution using small-angle scattering in irradiated steels.
- Will we be able to do simultaneous high-angle diffraction and low-angle scattering studies? 0.1 s time resolution possible?
- Opportunity: The original specification for VULCAN had "poor-man's" 2-D detector for small-angle scattering studies

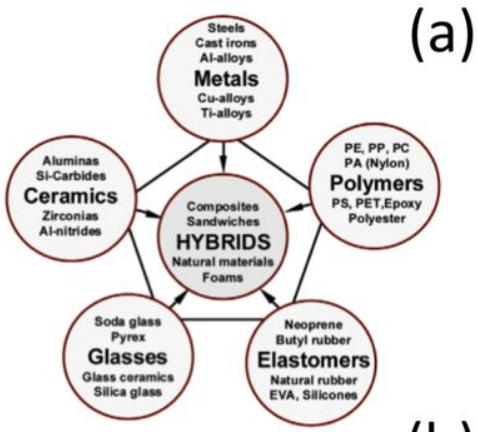
SNS-Vulcan



Summary and Future Outlook

- Through "targeted" in-situ and ex-situ data, we can indeed obtain high-impact scientific data.
- These data can be used calibrate integrated computational models to be accurate and robust!
- Within 20 years, we should be able to do "virtual joining experiments" with such integrated models.

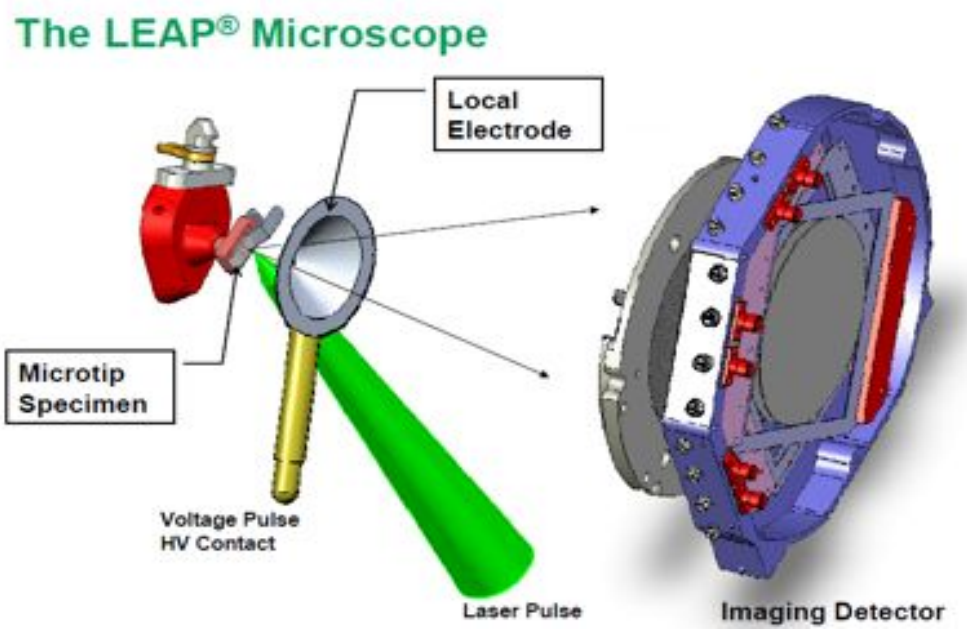


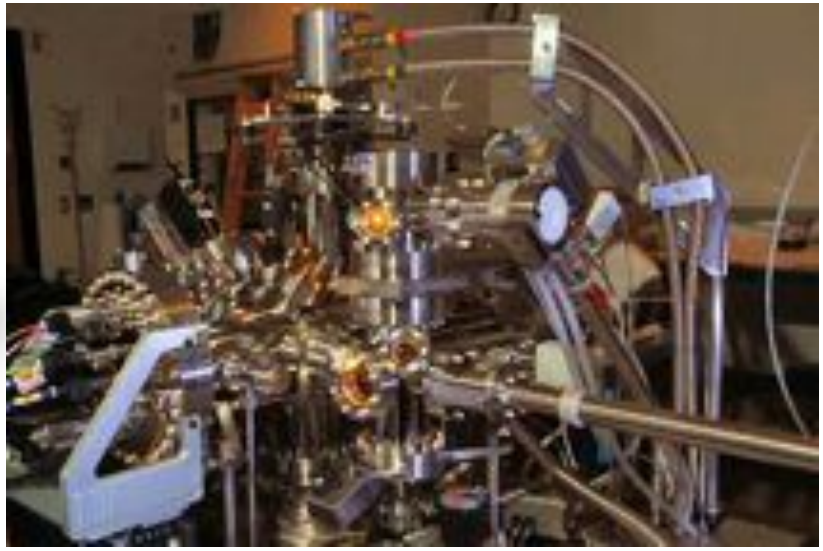


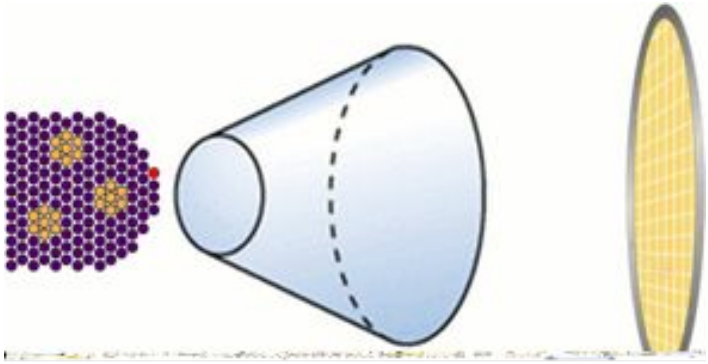
Questions

Additional Slides

LEAP® Tomography at NUCAPT





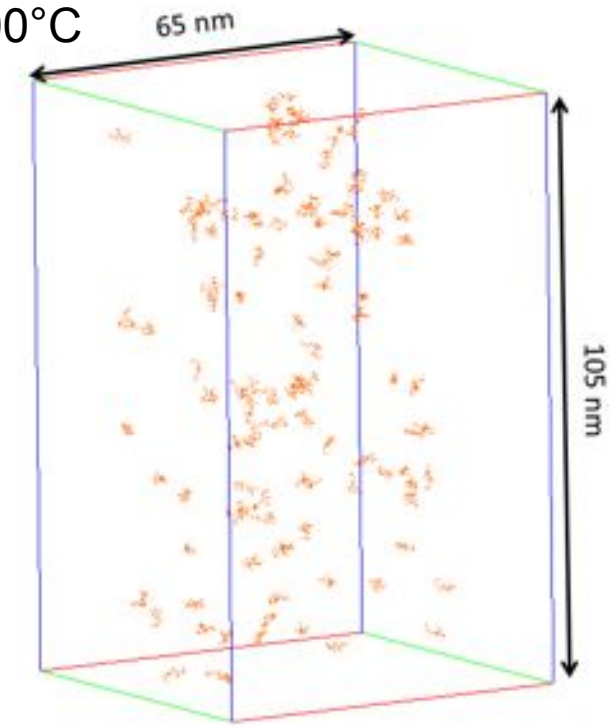


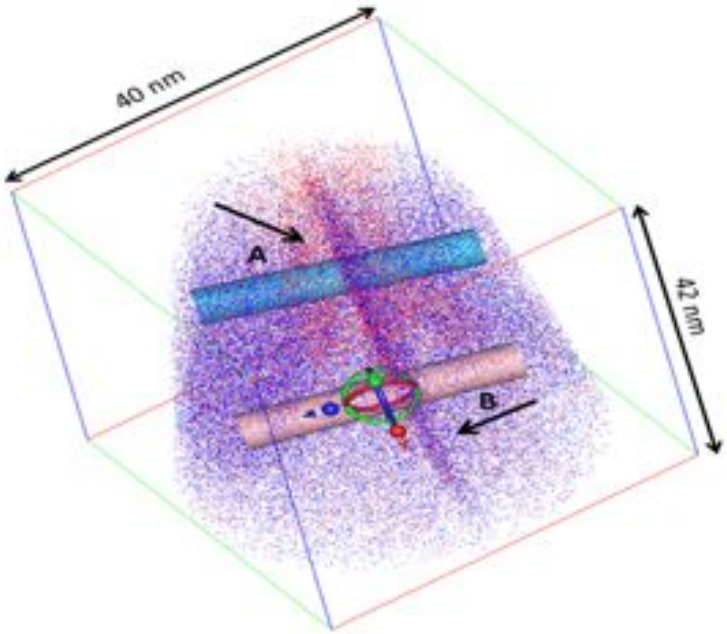
The laser was used due to the larger run sizes required. The conditions used were:

–Pulse Repetition Rate: 250-500 kHz
–Pulse Energy: 0.8-1 nJ
–Specimen Temperature: 75-85 K

Re-precipitation during cooling from higher temperatures, as well as, grain boundary segregation of Cu

T=1300°C





- **Why do we see strength anomaly?**

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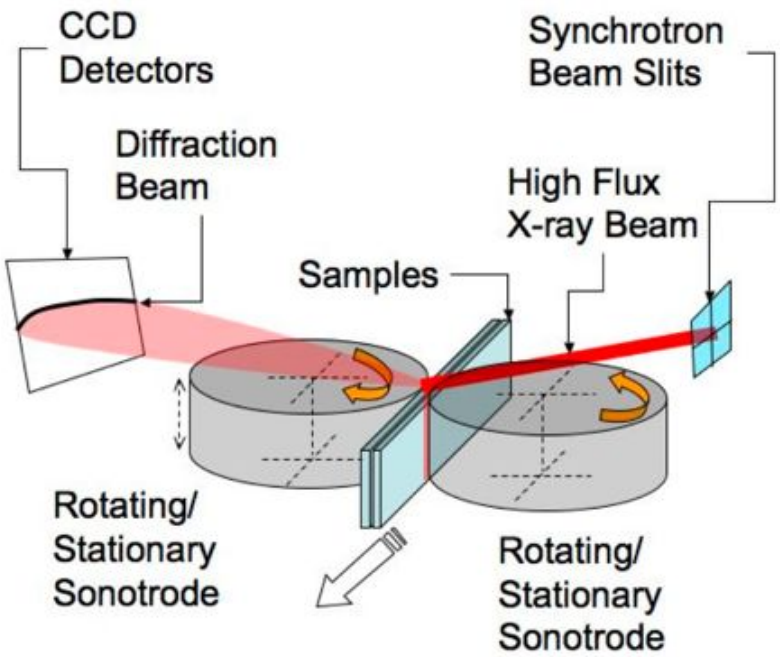
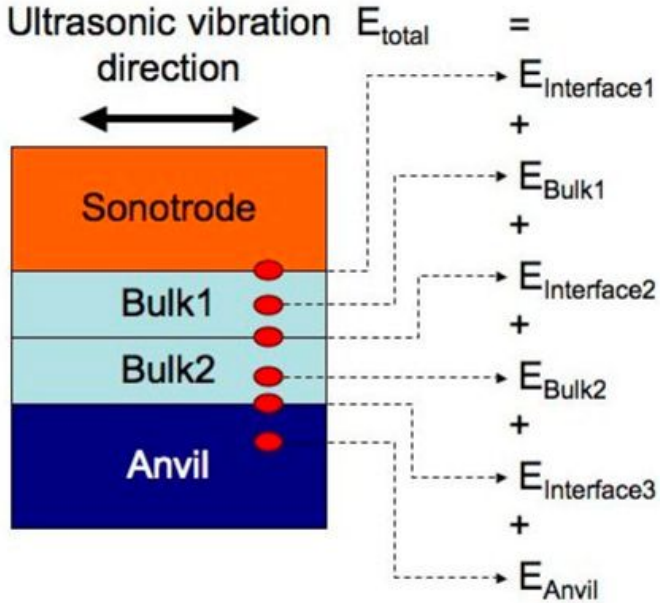
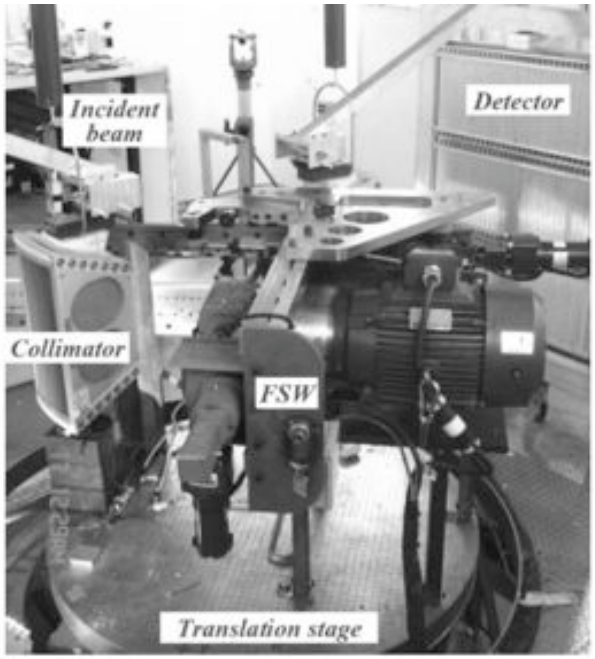
For UAM: Proposed In-situ Characterization



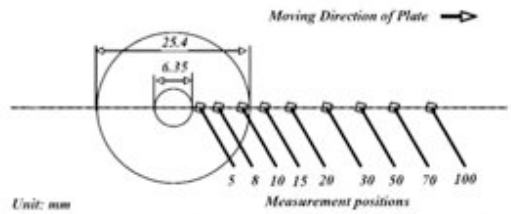


- **We need tools to evaluate the in-situ behavior at fine spatial and temporal resolutions**

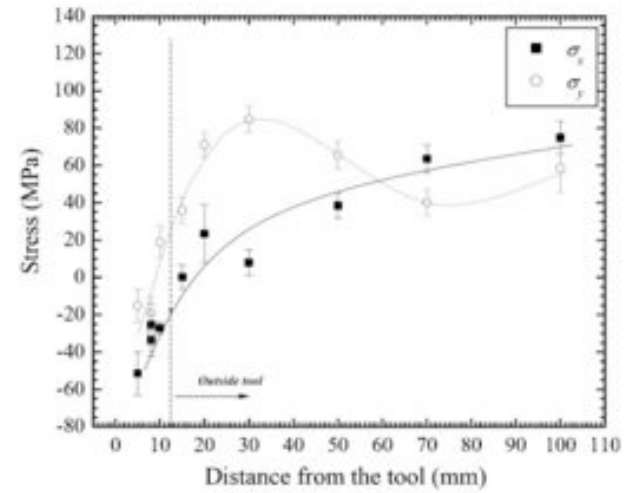
In-situ Scattering Research (Neutron) demonstration:



1 Experimental setup for *in situ* neutron diffraction measurement of FSW



2 Measurement positions: tool shoulder diameter is 25·4 mm; pin diameter is 6·35 mm

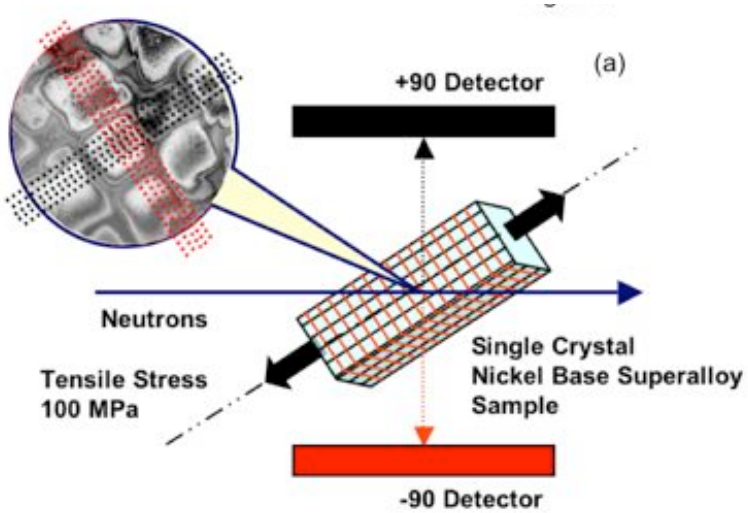


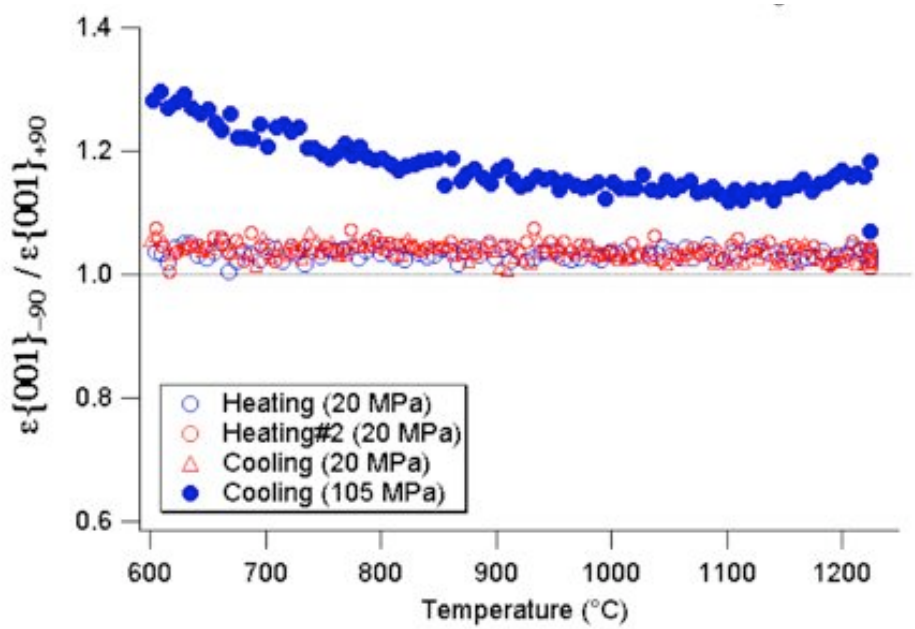
8 Thermal stress distribution along weld centreline behind tool as function of distance from tool centre: σ_x – longitudinal (LD) stress; σ_y – transverse (TD)

Ref: Woo et al (2007)
SMARTS-LANL

- **These tools have to be modified to consider severe gradients and rapid transients.**

Challenge 2: Characterization have to be mapped into material models

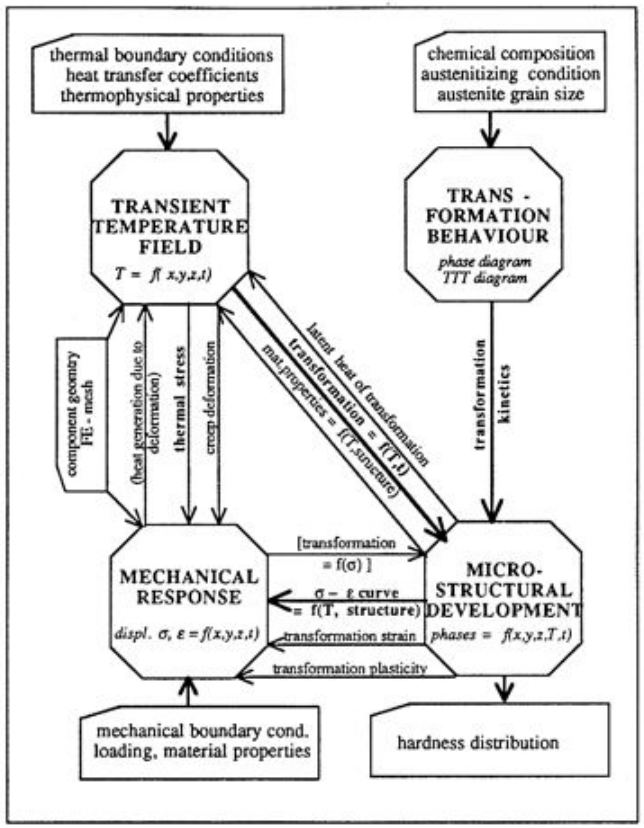


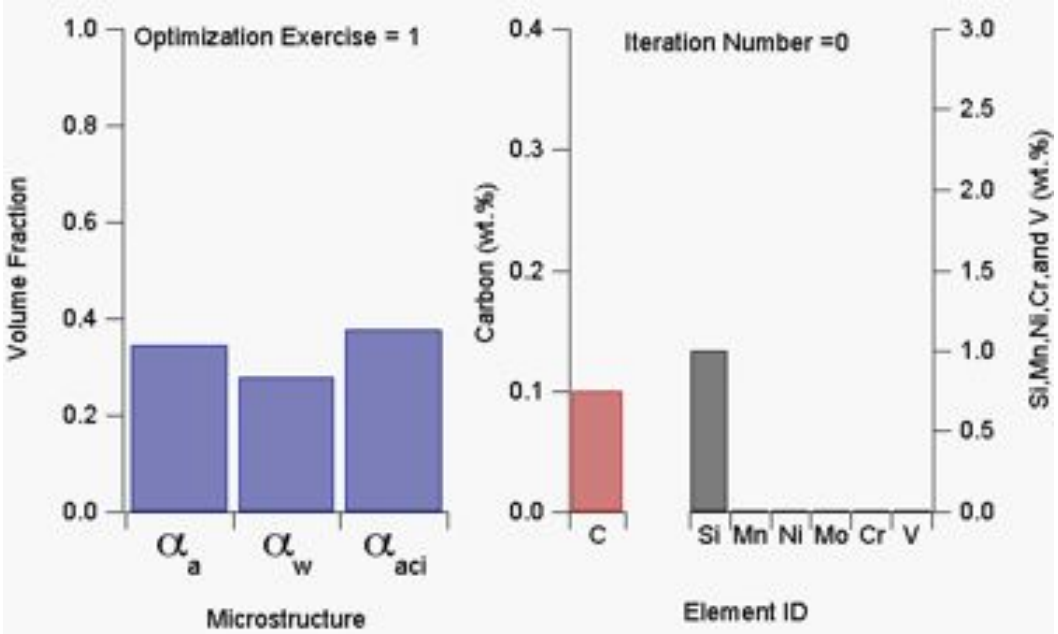


Ref: Babu et al (2005) Report at SMARTS-LANL

- **Verified and validated models will accelerate the deployment of process models**

Challenge 3: Optimization using integrated materials joining models





Ref: Murugananth et al (2007)

- **Above example is for fusion welding.**

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