Design and Implementation of a Cryogenic Loading Capability on the Spectrometer for Materials Research at Temperature and Stress - SMARTS

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4th International Workshop on Sample Environments at Neutron Scattering Facilities

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Outline

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- Current capabilities on SMARTS
- Motivation for a cryogenic capability
- Cryogenic system design, performance, and evolution
  - System for unloaded coupon testing
  - Initial cryogenic loading capability
  - Latest cryogenic loading capability
- Proposed design improvements
- Snapshot of results
- Summary

Spectrometer for MAterials Research at Temperature and Stress

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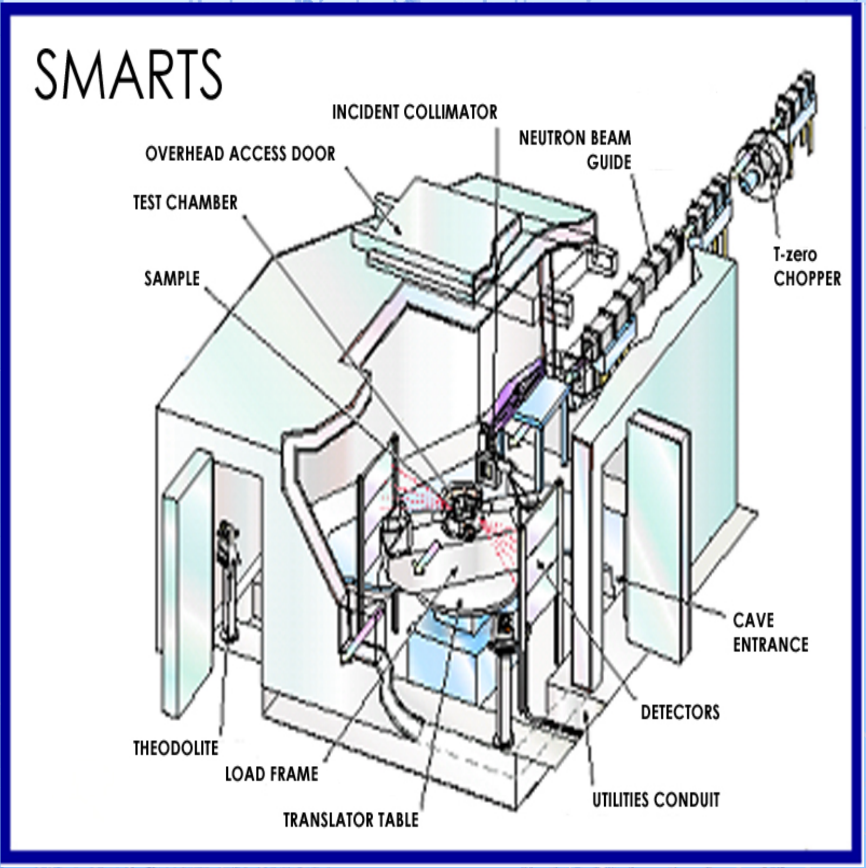

Current Capabilities

- Spatially resolved measurements
- *In situ* loading capability
  - Tensile and compressive loading
  - Applied forces up to 250 kN
- High temperature measurements
  - Test temperatures up to 1800°C
- No low temperature loading capability!

Motivation for a Cryogenic Capability

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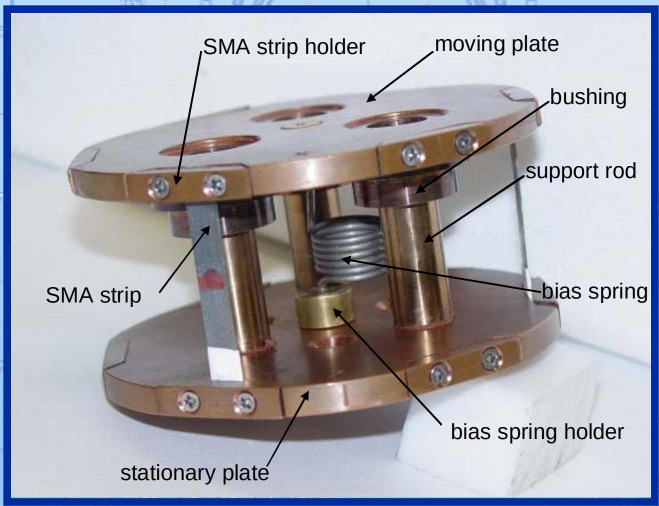

- NASA project at UCF to fabricate low temperature shape memory alloys for actuator applications
- Thermal conduction switch for Mars
- Seals, valves, fluid-line repair and self-healing gaskets
- Deformation involves stress induced transformation and twinning
- *In situ* studies during loading of stress induced transformation and twinning in low temperature shape memory alloys
- General – behavior at low temperatures
  - phase transformations, e.g., steel
  - twinning, e.g., Zr
  - Cu-Nb conductors

Investigation of Weld Repair on Space Shuttle Flowliners

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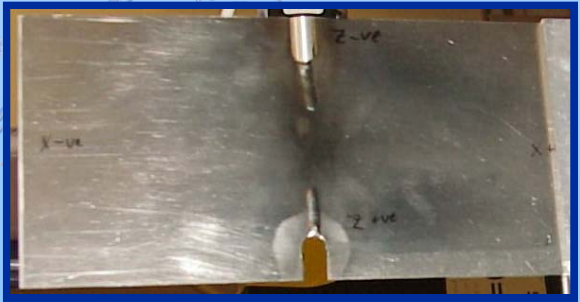
Hunt for cracks moves to shuttle Endeavour
BY WILLIAM HARWOOD
STORY WRITTEN FOR CBS NEWS "SPACE PLACE" & USED WITH PERMISSION
Posted: July 9, 2002

Discovery main propulsion unit
Flow liner crack ≈ 5mm length



Initial Cryogenic System – CAD and FE Design

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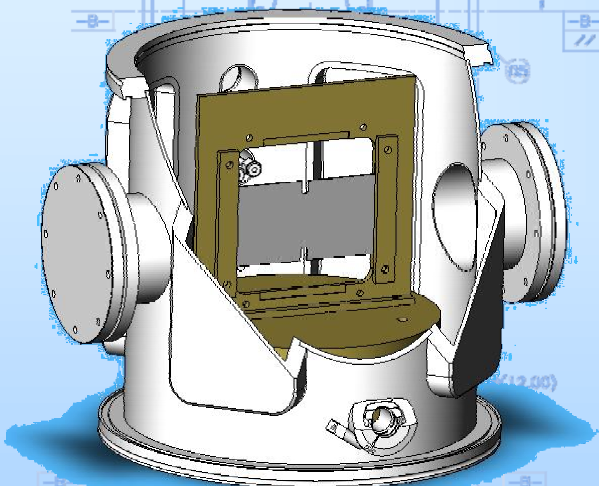
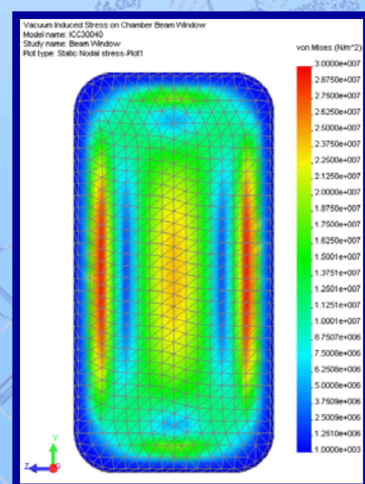
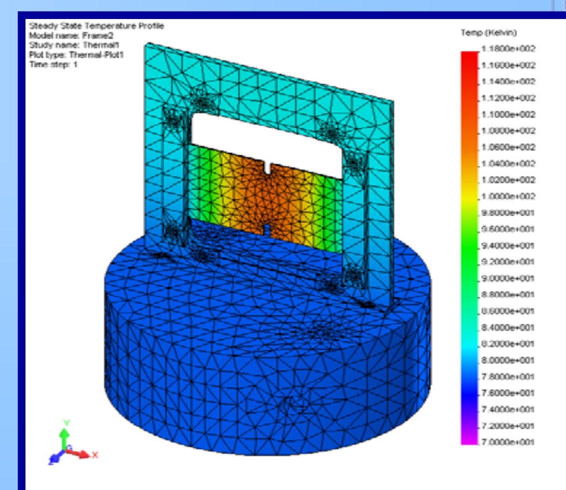


Cryogenic Cold Mass

- Large thermal mass incorporated to absorb energy during LN2 Dewar changes
- Sample frame designed to cool flowliner coupons via conduction while minimizing thermal gradients
- Designed to maximize conductive heat transfer without preventing thermal contraction





Custom Vacuum Chamber

- Four aluminum windows for neutron beam
- One clear glass view port for video sample monitoring
- Two platen feed through flanges for future loading capability

Initial Cryogenic System - Performance

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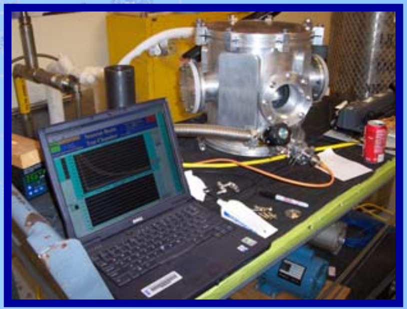
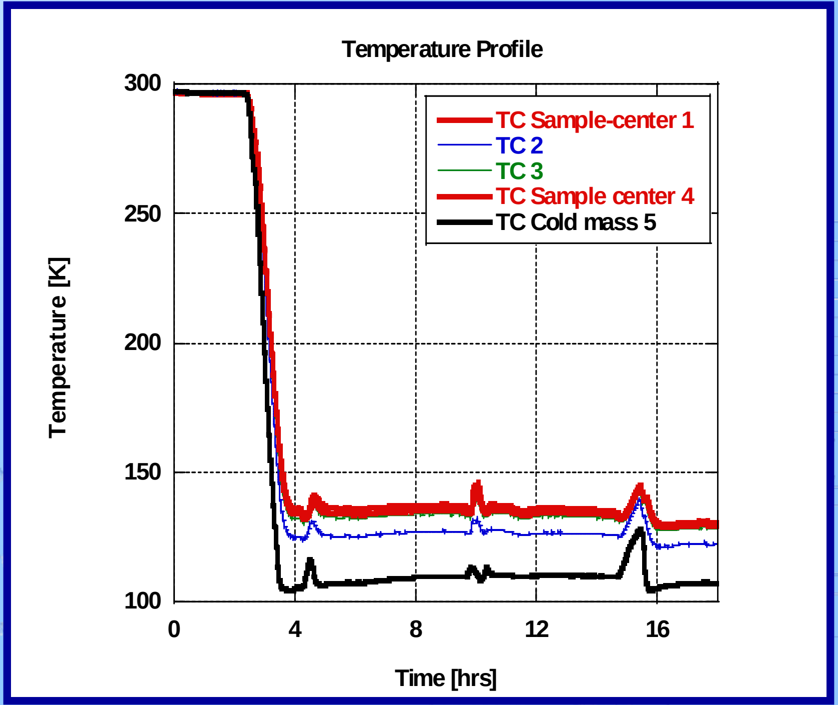


- Required an average LN2 flow rate of 27 L/hr
- Required 3.5 hours to reach a steady state sample temperature
- Obtained a steady state sample temperature of 135 K
- Temperature gradient across sample at steady state was 8 K



Initial Cryogenic System - Results

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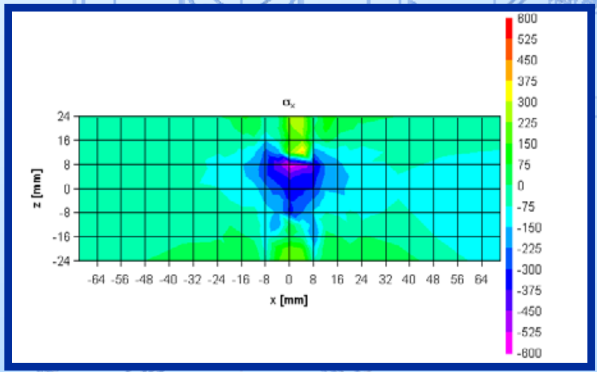
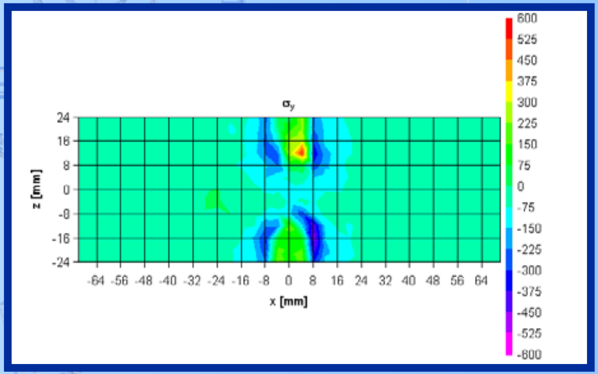


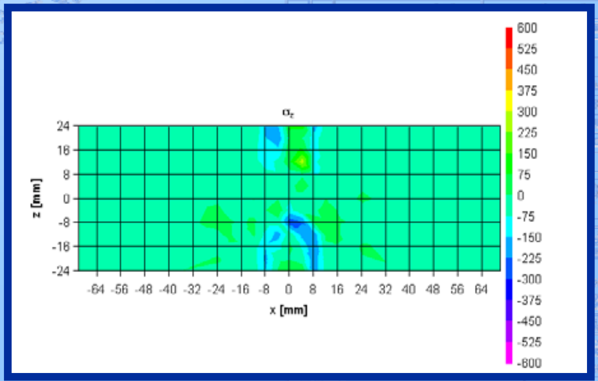
Residual Normal Stresses in x-direction



Residual Normal Stresses in y-direction

Residual Weld Stress Mapping

- Diffraction patterns allowed determination of d-spacing throughout flowliner weld coupons at low temperatures
- d-spacings were converted into strains and then to stresses



Residual Normal Stresses in z-direction

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Initial Cryogenic Loading Capability – CAD and FE Design

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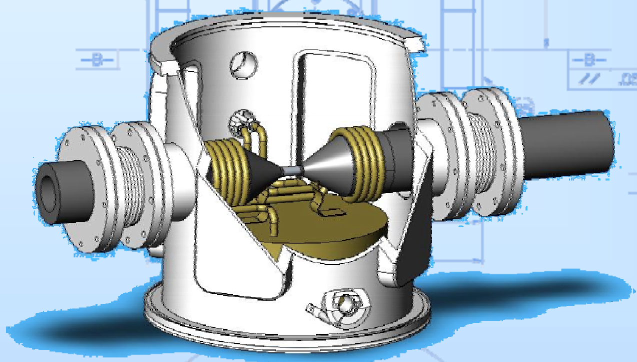


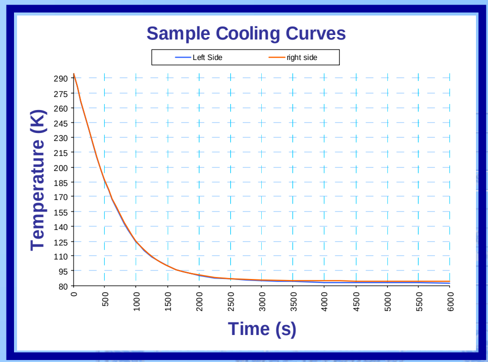
Finite Element Analysis

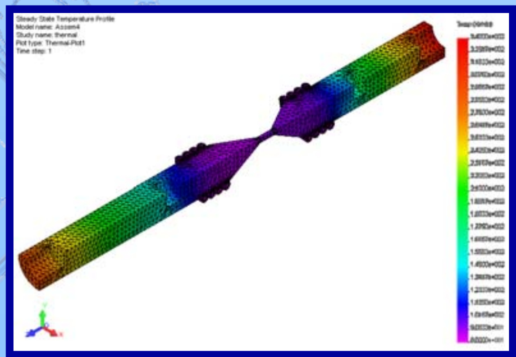
- Numerical codes used to predict and analyze the thermal response of various design
- Stress analysis codes used to analyze the thermo mechanical effects of transient and steady state thermal loads

CAD Modeling with SolidWorks

- Geometric models were constructed to check fits and clearances
- Model was designed to fit the existing load frame on the SMARTS spectrometer
- Convective cooling coils were developed to fit existing SMARTS compression platen

design





Initial Cryogenic Loading Capability - Implementation

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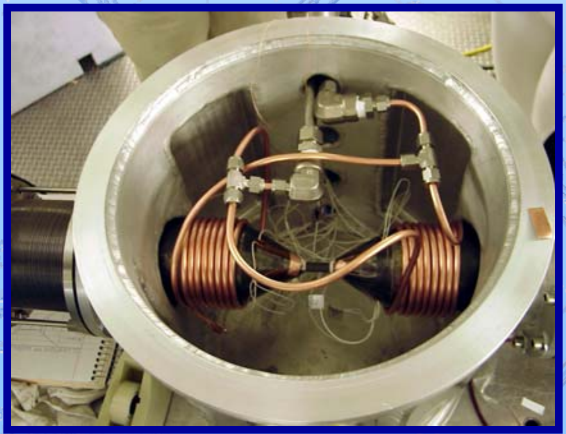
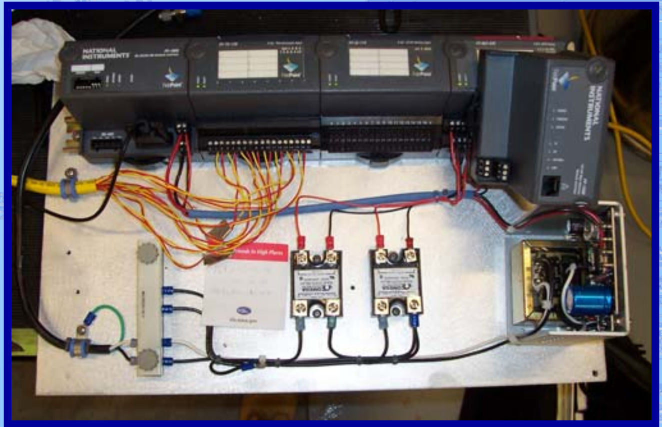
Data Acquisition and Control System

- Used to monitor thermocouple readings both inside of and outside of the vacuum chamber
- Used to control a series of sample heaters that maintain a specified sample temperature and minimized temperature gradient





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Initial Cryogenic Loading Capability - Issues

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- **Large volumetric flow rate of LN2 was required (27L/hr)**
- **High steady state temperature was obtained (216K)**
  - **Poor insulation from large heat leak generated by load frame hydraulic cylinder**
  - **Poor contact between convective cooling coils and compression platens**
  - **Convective heat leak was introduced by a poor vacuum level due to small nitrogen leaks at hard to seal plumbing connections**
- **Strains had to be estimated using crosshead displacement**
  - **Chamber design did not allow feed through of wiring for strain gages or extensometer**

Latest Cryogenic Loading Capability

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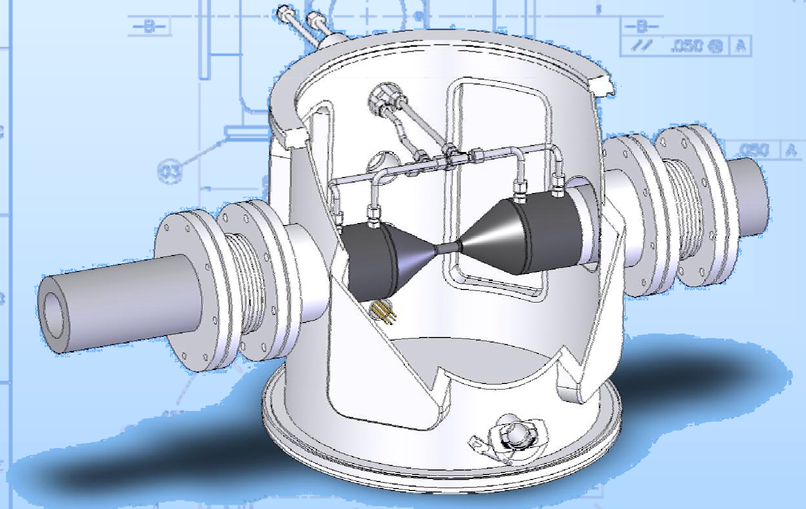

Cooling System Modifications

- New flow-through compression platens were designed to utilize internal convective cooling
- Vascomax push rods were replaced with 17-4PH stainless steel design because of the lower cost and relatively low thermal conductivity
- New platen attachment method was developed to insulate compression platens from the high temperature load frame using Macor© ceramic

Vacuum System Modifications

- Same vacuum chamber was utilized
- New vacuum feed throughs were designed to allow wiring of a strain gage

Latest Cryogenic Loading Capability – Platen Design

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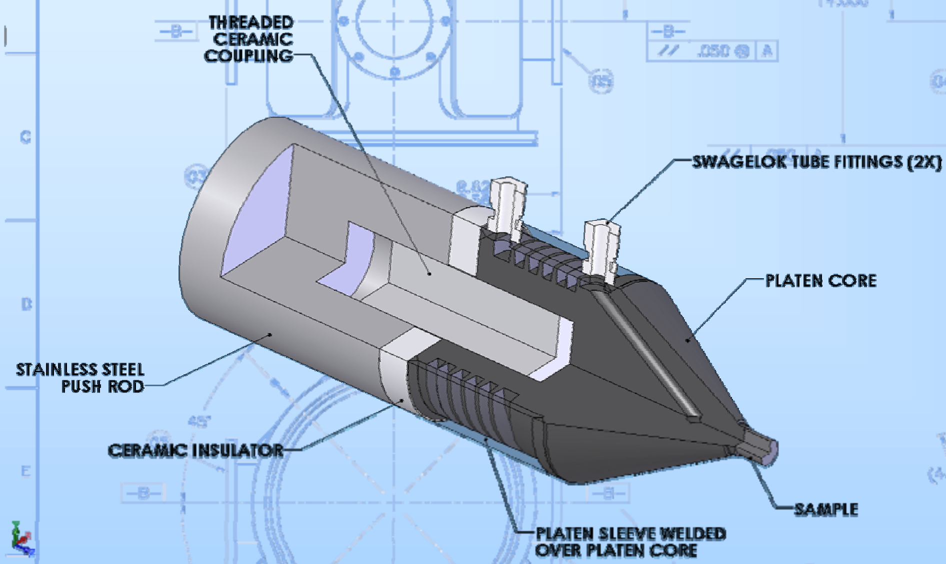
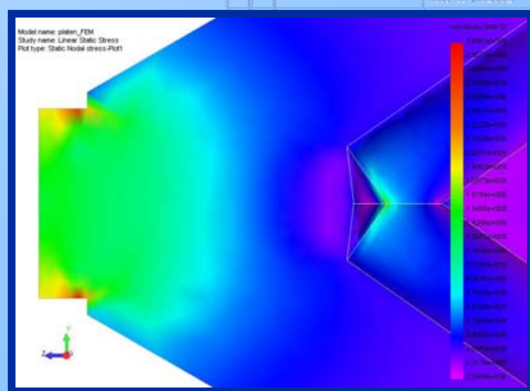


Cooling Features

- Three-piece welded design incorporates cooling channel into platen core to deliver LN2 closer to sample
- Internal cooling coils are incorporated to eliminate thermal contact resistance



FEA Design Validation

- Stress concentrations were analyzed around cooling passages to eliminate brittle fracture of platen core under load

Latest Cryogenic Loading Capability - Performance

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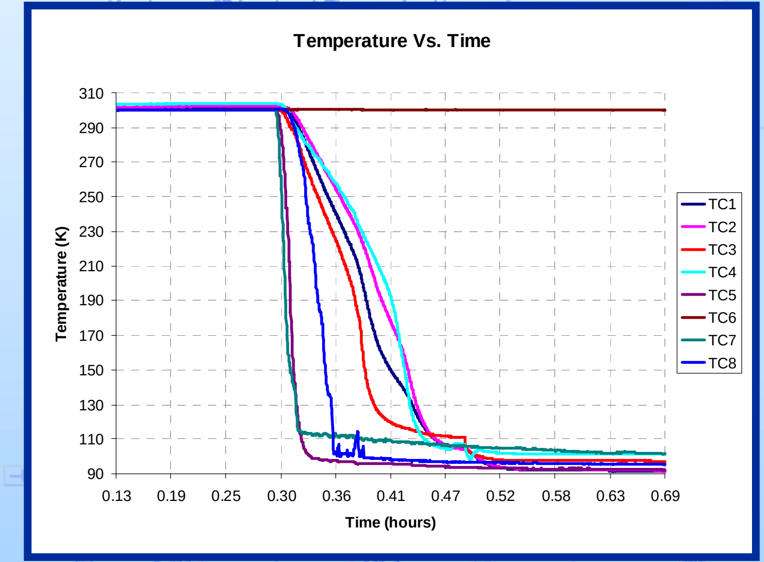

Cooling Curve for NiTiFe Sample

Problems w/ Latest System Design

- Large thermal offset in strain gage measurements
- Brittle ceramic couplings can fracture due to thermal contraction of mating components

LN2 Volumetric Flow Rate

- Reduced to an average of 16 L/hr

Steady State Temperature

- Reduced to 89 K

Required Cooling Time

- Reached steady state in 18 min

Proposed Design Improvements

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Vacuum System Improvements

- Modification of vacuum feed throughs to allow for wiring of additional strain gage or extensometer

Cooling System Improvements

- Utilization of composite polymer materials to create more resilient insulated couplings

Control System Improvements

- Implementation of an automated LN2 flow control and a Dewar manifold to maintain more constant system temperatures over longer periods of time
- Utilization of a PID temperature controller to speed temperature changes and eliminate overshoot
- Incorporation of higher wattage cartridge heaters into compression platens to speed temperature changes and reduce warm-up time during sample changes

Data Acquisition Improvements

- Addition of a second strain gage channel to reduce thermal offset and/or addition of a ceramic legged extensometer for low-temperature strain measurements

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Results from NiTiFe at 92 K

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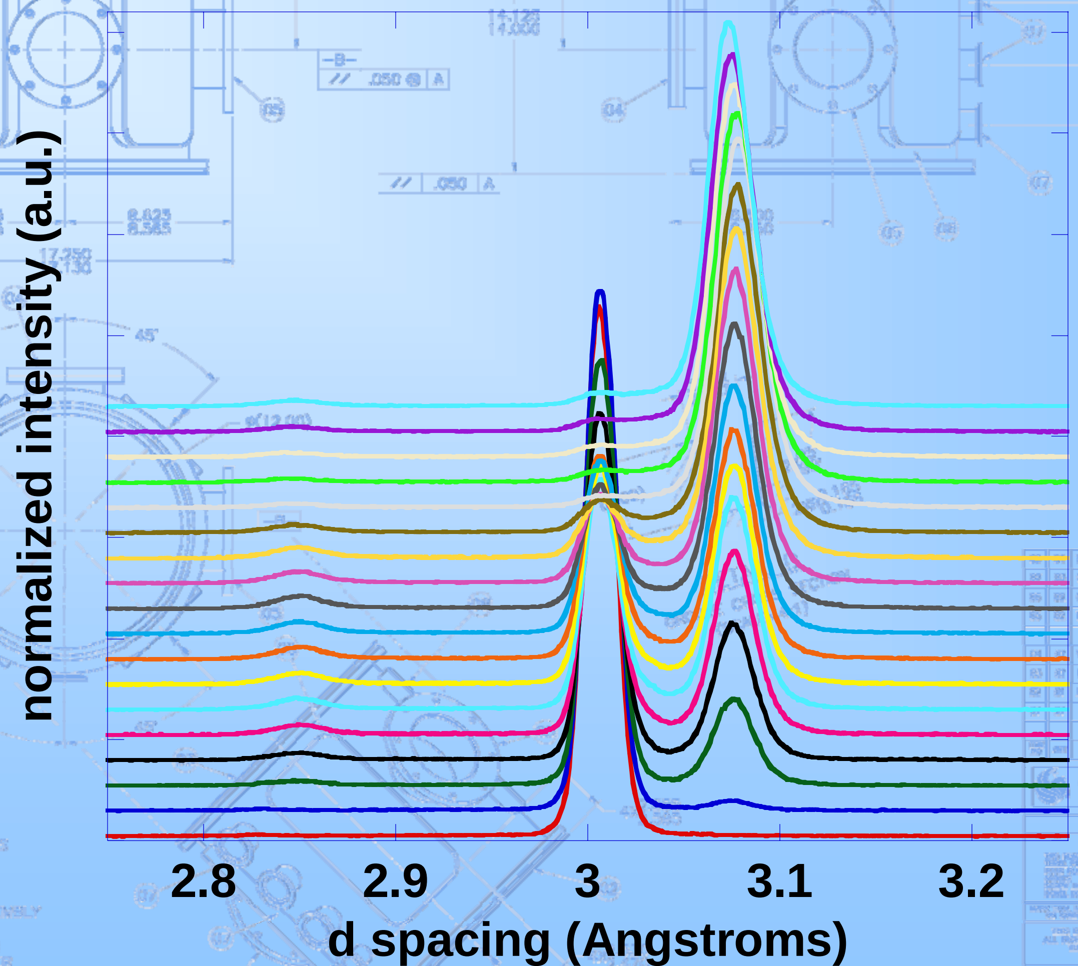

• 10mm Dia. X 24 mm Lg. cylindrical samples cooled to 92 K and deformed up to 425 MPa

Summary

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- **Cryogenic loading capability successfully implemented on SMARTS**
- **Three design iterations have been built and tested**
  - Unloaded design for testing a variety of static specimens at low temperatures
  - Two load-frame designs have been implemented resulting in test temperatures as low as 89 K
  - These systems have provided useful results for materials scientists at UCF and LANL, and NASA engineers
- **Cryogenic system improvements are planned**
  - Implementation of automated LN2 flow control and a Dewar manifold
  - Utilization of a PID temperature controller and higher wattage cartridge heaters
  - Addition of a second strain gage channel and/or a ceramic legged extensometer
- **Further cryogenic measurements are planned on shape memory alloys**

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Acknowledgements

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