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Pseudoelastic Response of Ion-Implanted Nickel-Titanium Shape Memory Alloy: Combining Experimentation and Forward Modeling

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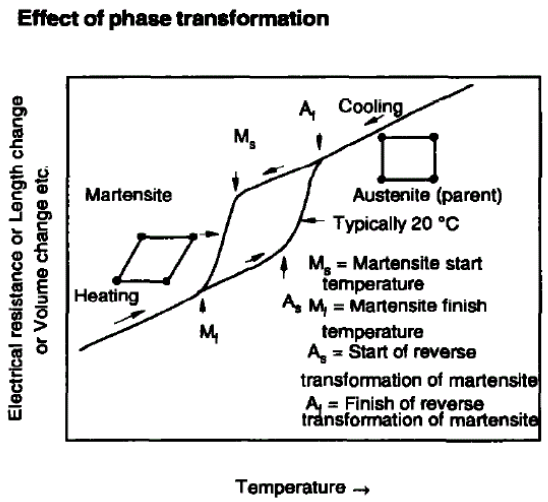
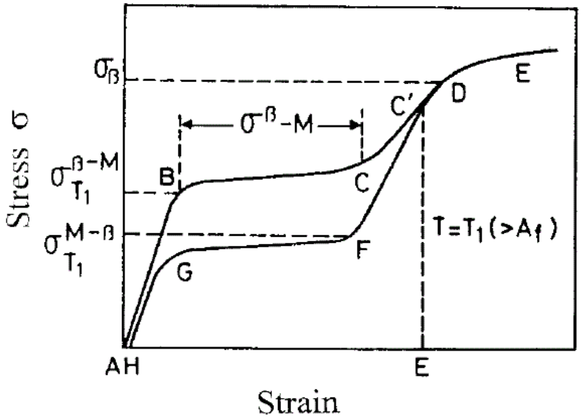
Shape Memory Effect

- **Martensitic Phase Transformation between low temperature martensite phase B19' and high temperature austenite phase B2 enables desirable adaptive material properties included in:**
  - Medical Devices
  - Solid state actuators
  - Smart alloys



Effects of phase transformation on various properties that exhibit a discontinuity at the transformation temperatures

Duerig, Pelton 1994

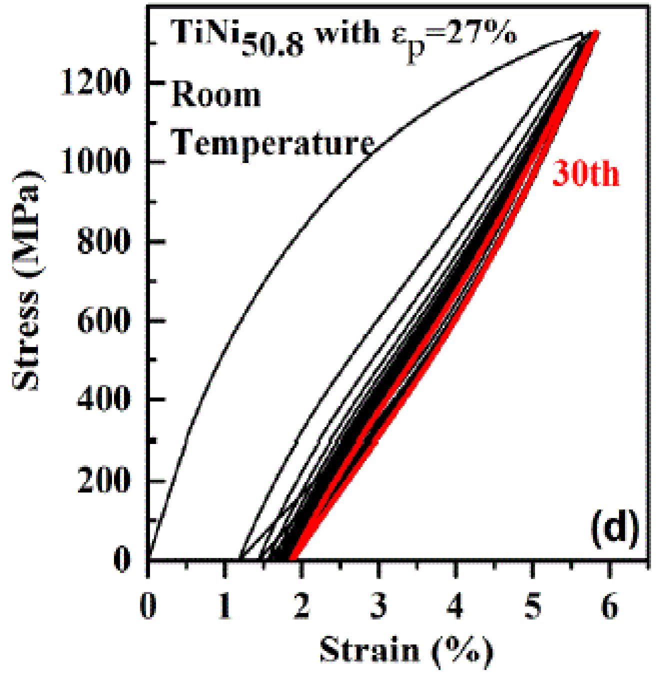


Characteristic Stress Strain Curve of NiTi:
A-B elastic deformation of the parent M phase
B-C Martensitic transformation beginning at $\sigma_T^{\beta\text{-}M}$
C-D elastic deformation of the martensitic phase
C' If stress is relieved at or before this point:
C'-F elastic unloading of martensite
F-G reverse transformation to austenite
G-h Elastic recovery of the parent phase

Gil, Plannell 1998

Defect Engineering of NiTi

- **NiTi SMAs can be engineered to control the hysteretic nature of the martensitic transformation and attain desirable material properties. Common methods include:**
  - Composition
  - Precipitation
  - Thermal Cycling
  - Mechanical Cycling
- **Our goal is to engineer a strain glass NiTi system that has predictable linearized forward and reverse transformation.**

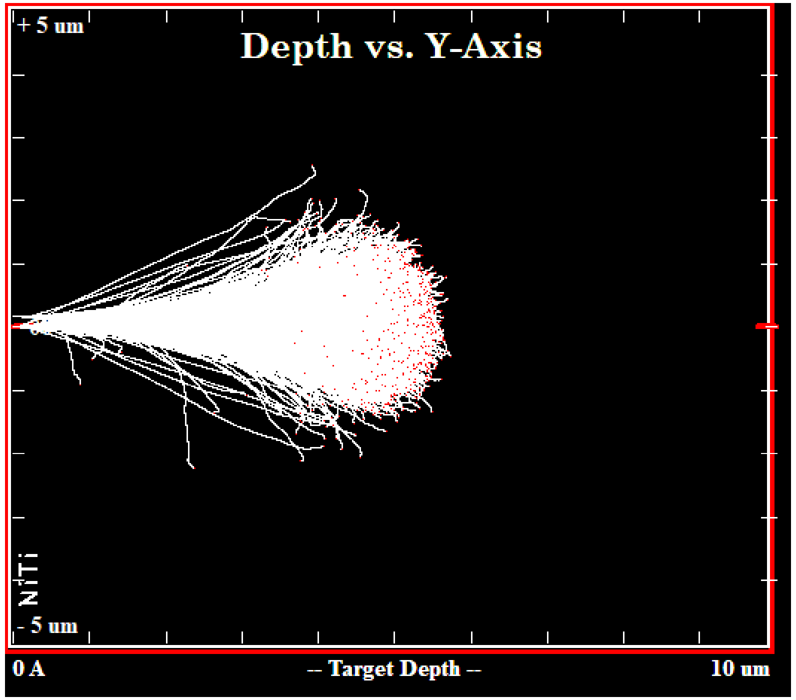


Hysteresis of the stress strain curve in 27% cold rolled $Ni_{50.8}Ti_{49.2}$ over 30 cycles of tensile testing at room temperature

Lang, Wang et al. 2017

Ion Implantation

- **Irradiation can create a range of defect damage from point defects and dislocation loops to amorphization and voids.**
  - Two primary types of ion damage:
  - Electronic Stopping – inelastic collisions between the electron clouds of the ion transferring energy
  - Nuclear Stopping – direct elastic collisions between an ion and lattice atoms, creates a chain reaction of lattice displacements or a collision cascade
  - Irradiation damage is quantified by Displacements per Atom (DPA)

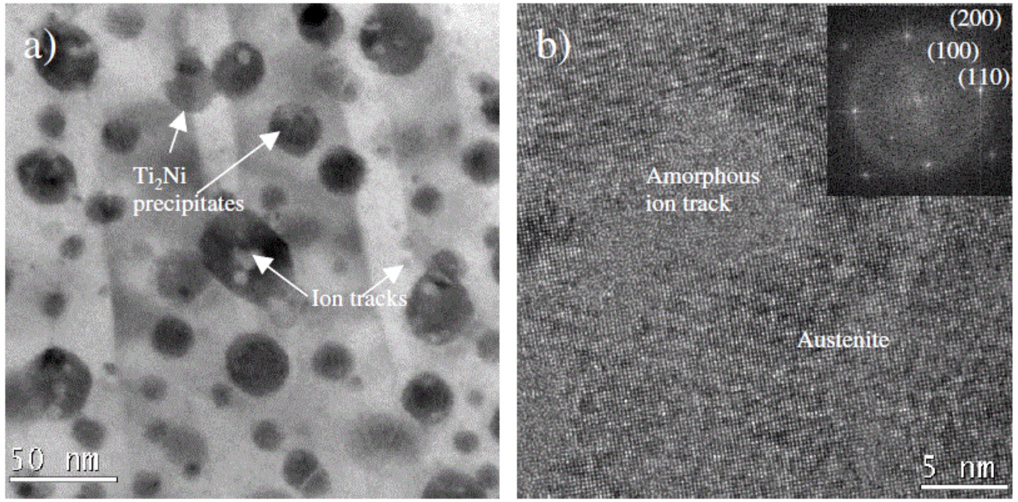


Plot of ion/recoil distributions using the Stopping Range of Ions in Matter software package

Ziegler 1981

Ion Implantation of Nickel-Titanium

- **Prior irradiation and ion implantation have induced amorphization of the NiTi Crystal structure in thin films shutting down the martensitic transformation**
  - Brimhall, Kissinger et al. 1985 (2.5 MeV Ni+)
  - Moine, Riveiere et al. 1985 (390 keV Ni+)
  - Lagrange, Schäubin, et al. 2006 (350 MeV Au+)
- NiTi fully amorphizes at 0.1 DPA and above, shutting down the martensitic phase transformation
- Deposited energy density determines degree of amorphization, fluence influences amount of amorphous regions
- Irradiation can induce residual amorphized, R, Ti_2Ni, and austenite phase regions

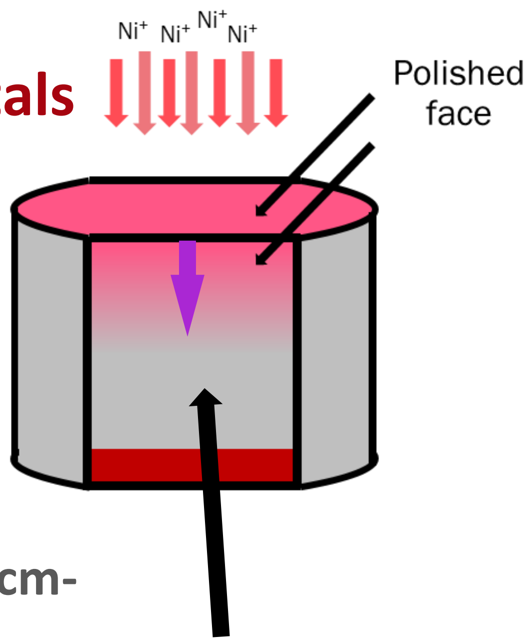


Brightfield imaging of ion tracks left by Au ion bombardment showing formation amorphous region surrounding the track, Ti_2Ni precipitates, and residual austenite in parent martensite material

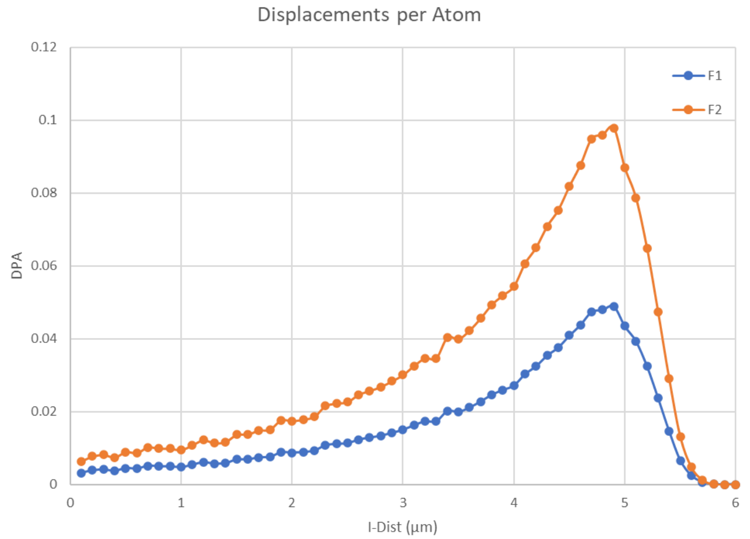
Lagrange, Schäubin, et al. 2006

Experiment: Ion Implantation

- **Using 30 MeV Ni+ ions, chose 2 fluences to probe the 0-0.01 DPA range of interest.**
- **Material supplied by Fort Wayne Metals**
  - 50.5at% Ni 49.5at% Ti
  - <111> Textured extruded wire
  - 8 µm grain size
- **Irradiation Dosage**
  - 30 MeV Ni+ ions
  - Fluences: F1 5x1013 cm-2 and F2 1x1014 cm-2
  - Performed by Khalid Hattar at CINT



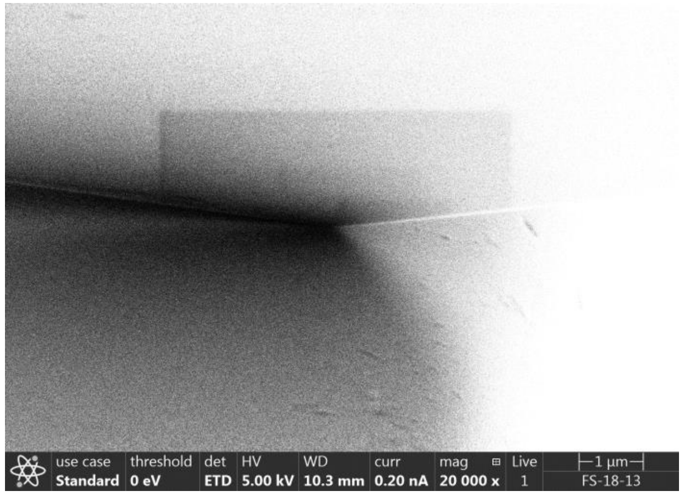
DPA damage gradient over a ~6µm depth

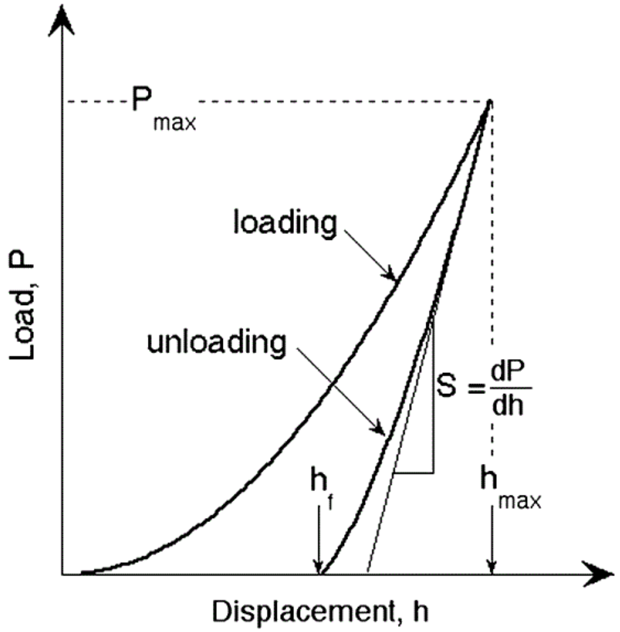


DPA prediction calculated from SRIM results

Nanoindentation

- **Nanoindentation can probe the mechanical properties of small volumes of ion implanted material.**
  - DSC unable to analyze small masses of homogenously implanted material (<3mg)
- **In Situ Nanoindentation**
  - 200 nm Indent
  - Berkovich Indenter tip
  - Performed by Nan Li at CINT





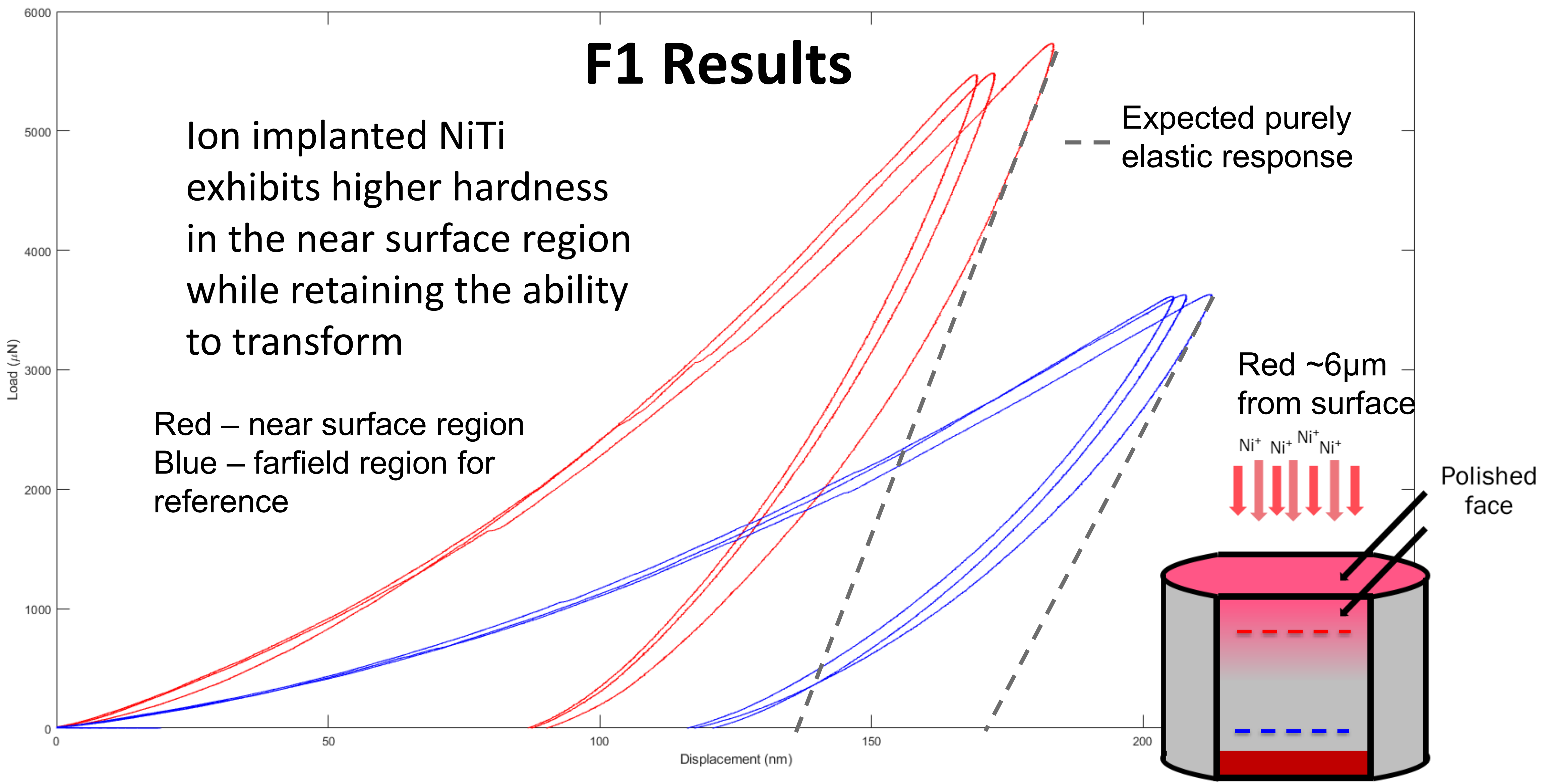
Characteristic load versus displacement curve. Extended initial unloading slope is typical of a purely elastic response

Oliver Pharr 2003

F1 Results

Ion implanted NiTi exhibits higher hardness in the near surface region while retaining the ability to transform

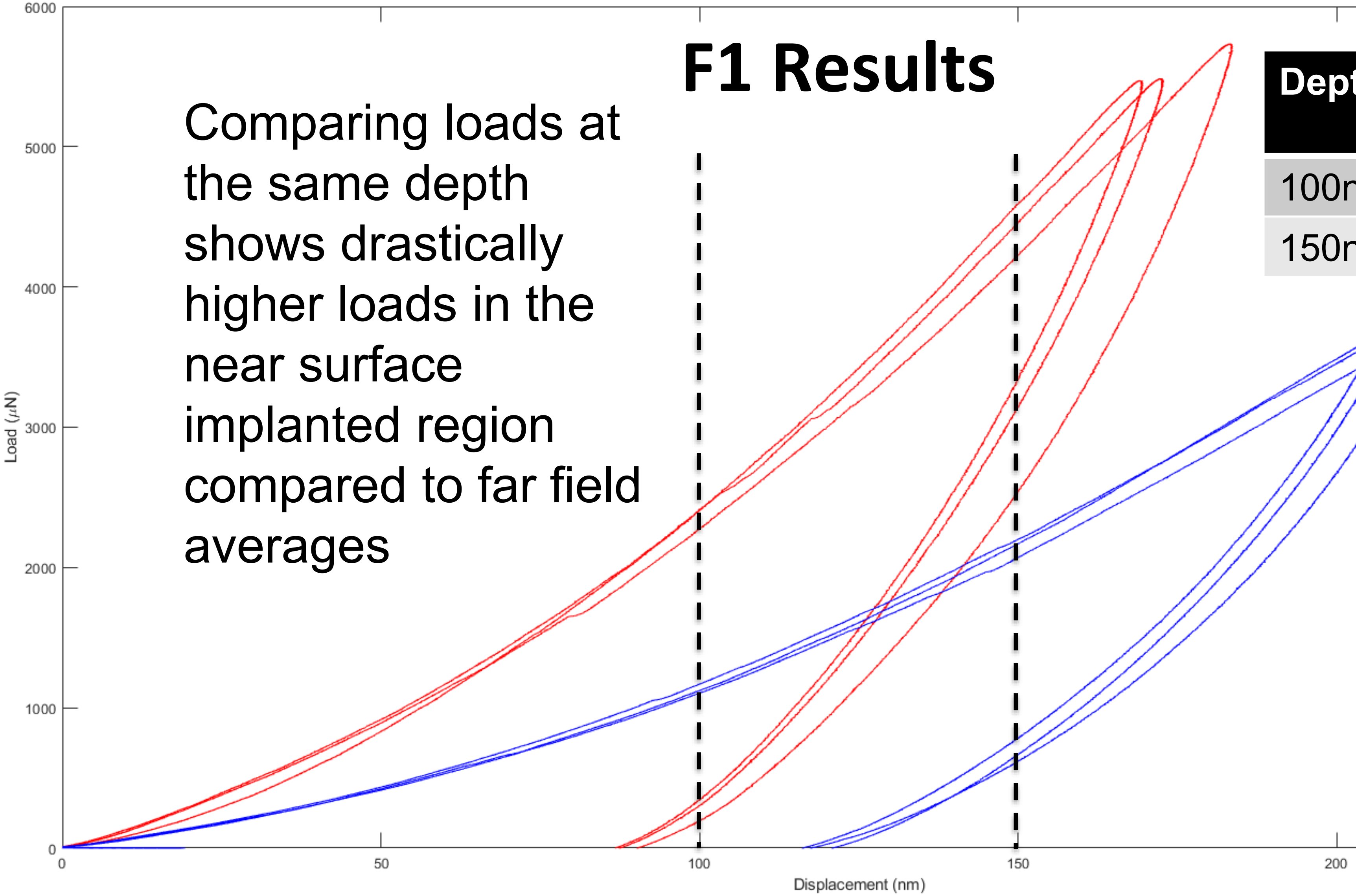
Red – near surface region
Blue – farfield region for reference



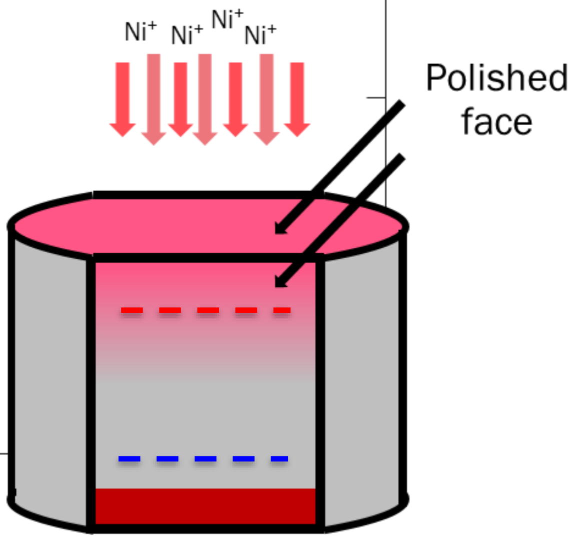
F1 Results

Comparing loads at the same depth shows drastically higher loads in the near surface implanted region compared to far field averages

Depth of Load	Range of Load Increase
100nm	76-86%
150nm	79-95%



Red ~6μm from surface

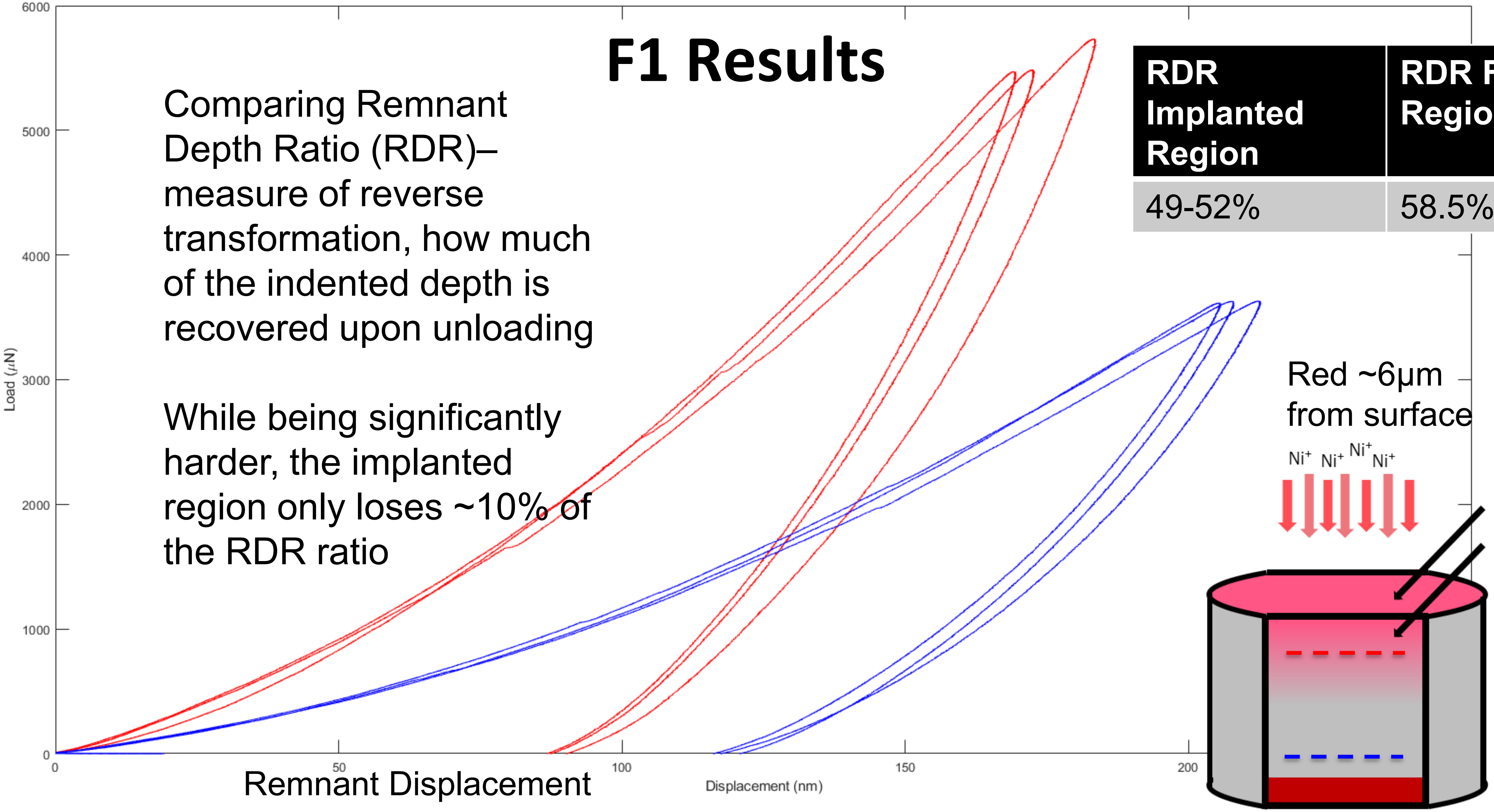


F1 Results

Comparing Remnant Depth Ratio (RDR)– measure of reverse transformation, how much of the indented depth is recovered upon unloading

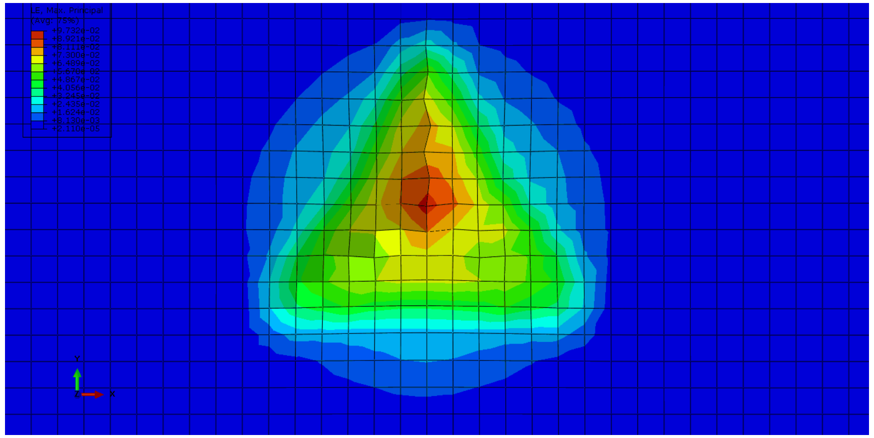
While being significantly harder, the implanted region only loses ~10% of the RDR ratio

RDR Implanted Region	RDR Farfield Region
49-52%	58.5%-61%

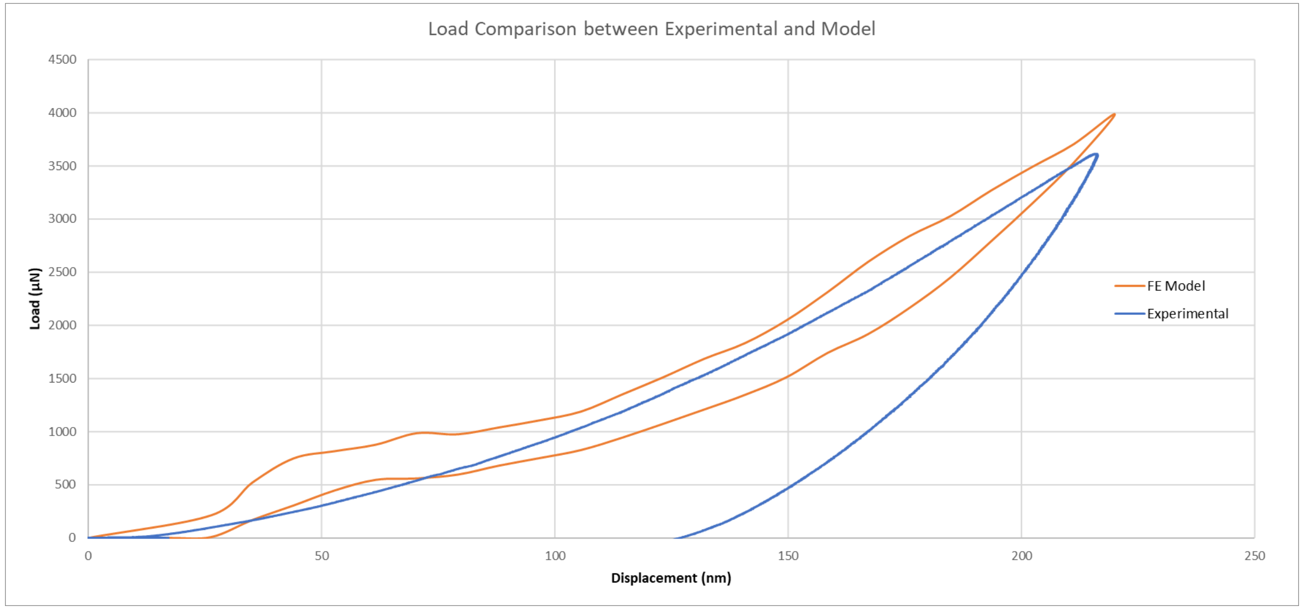


Preliminary Modeling

- **Correlate Experimentation and Modeling via Load Displacement Curves to determine material properties**
  - **Model: Auricchio, Taylor 1996**



Indent trace from the FE model indentation by a Berkovich tip



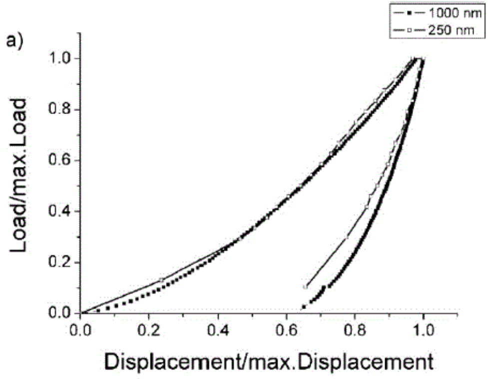
Conclusions

- **Ion implantation increases hardness in the near surface region without destroying the shape memory effect**
  - Likely caused by distributed nano scale defect structures and formation of non parent phase regions
- **Ion implantation at moderate doses creates a gradient of damage that, unlike prior literature, does not completely amorphize the material and destroy the shape memory effect**
- **Nanoindentation can be used in a variety of ways to evaluate small volumes of materials and correlate with FE modeling**

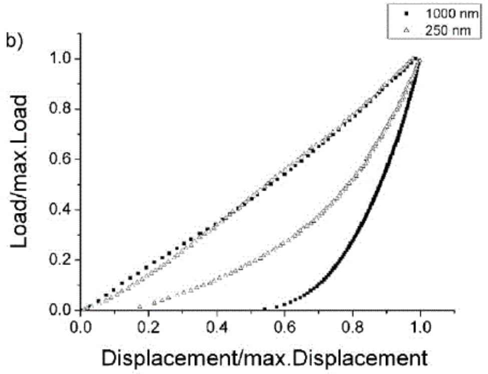
Future Work

- **Investigate F2 fluence and compare effects of implantation**
- **Alternate indenter tips**
- **Forward modeling of nanoindentation to determine material properties of ion implanted regions**
- **Collaborations for characterization and phase field modeling**

Berkovich Tip

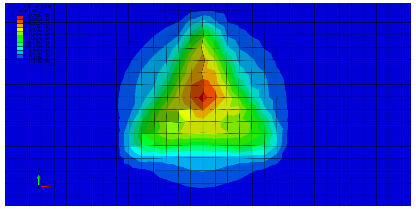


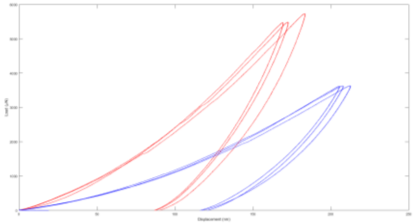
Hemispherical Tip



Pfetzing-Micklich,Wagner 2009

Project Collaborations



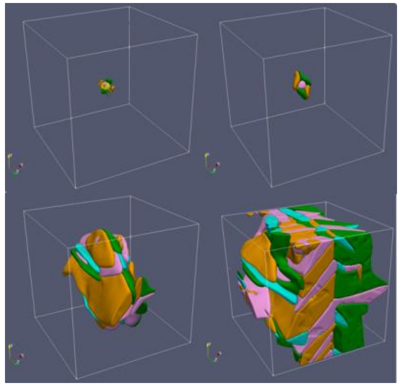


- FE modelling of Nanoindentation to determine stress fields
- Phase field modelling of defect structures and its interaction with NiTi

Nanoindentation and FE Modeling

- Nanoindentation to determine implanted regions with unique Mechanical Properties
- Microscopy to characterize region with altered response

Effect of single nano void on MT

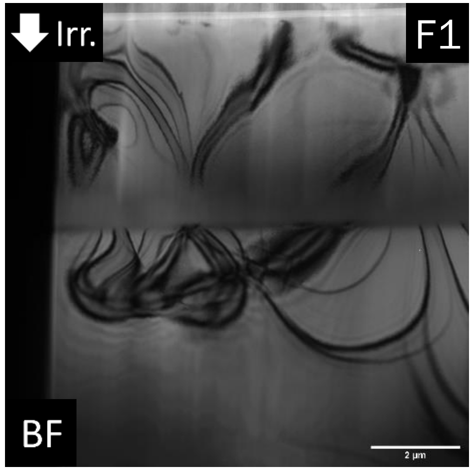


Phase Field Modeling

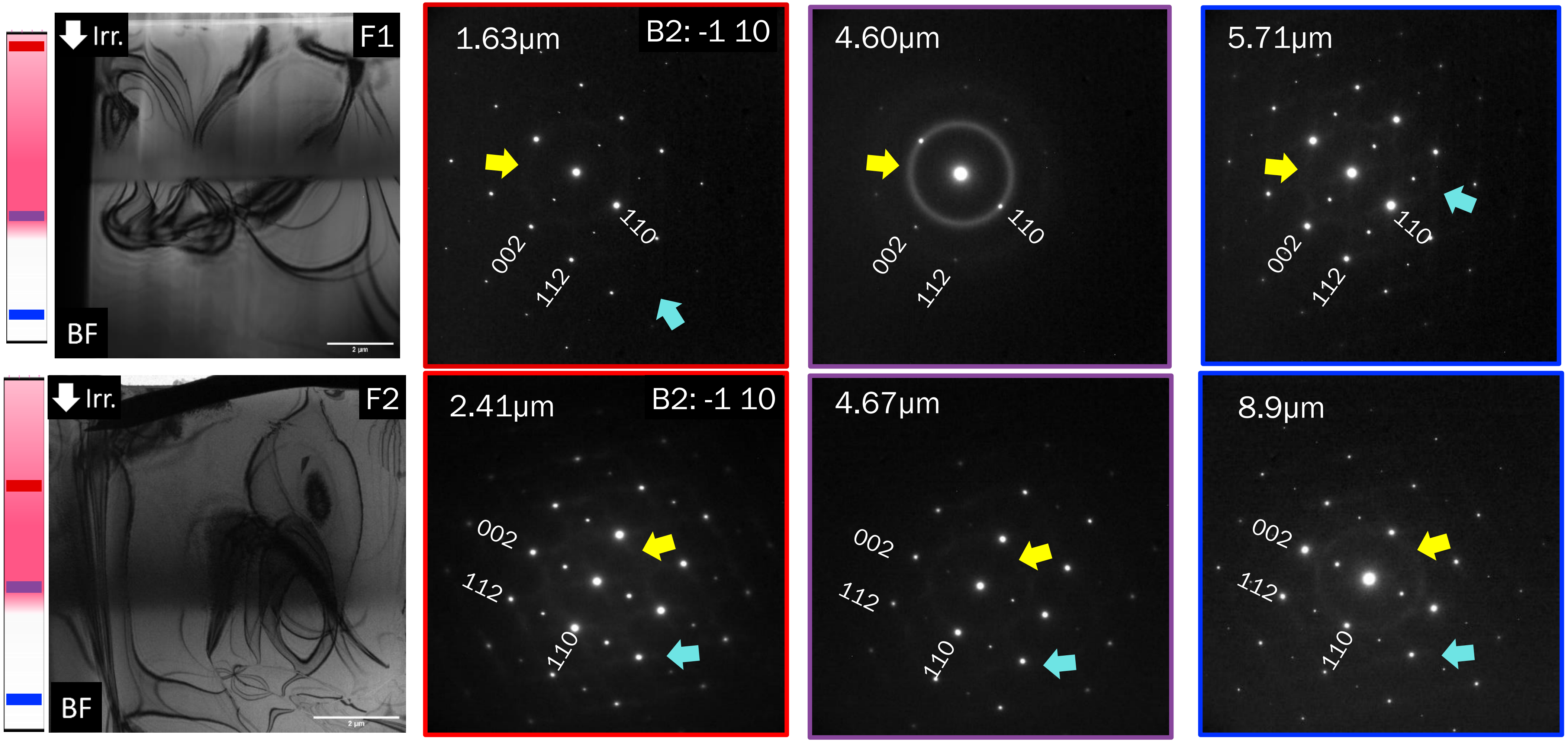
Microscopy

- Microscopy to identify defect microstructures
- Phase Field modeling to predict micrographs based on microstructures

Brightfield Image of Implanted NiTi



Microscopy Collaborations



- Diffuse amorphous ring around the (110) spacing.
- Streaking along the <112> in some areas.
- Strongest amorphous ring seen in the deepest portion of the foil, ~3.5μm below the implantation band.
- Yellow arrows point to the diffuse ring; Aqua arrows show streaking.

Acknowledgements

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  - Nan Li (CINT)
  - Jeremy Schaffer (Fort Wayne Metals)
  - Harshad Paranjape (Confluent Medical)
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  - Fort Wayne Metals

QUESTIONS?