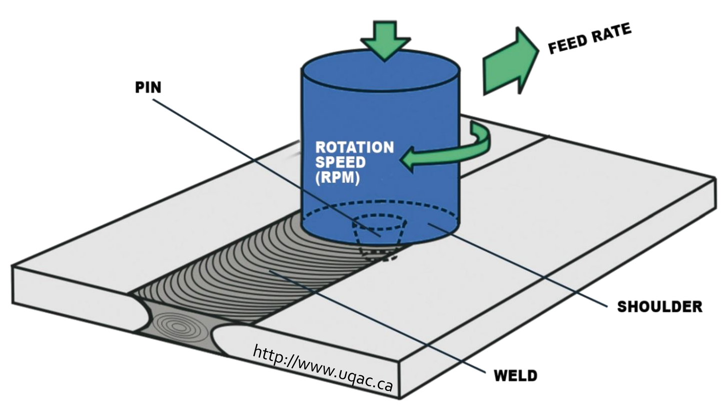
Microstructural Analysis of Friction Stir Welded Joints Between Ni-Based Alloy and Steel

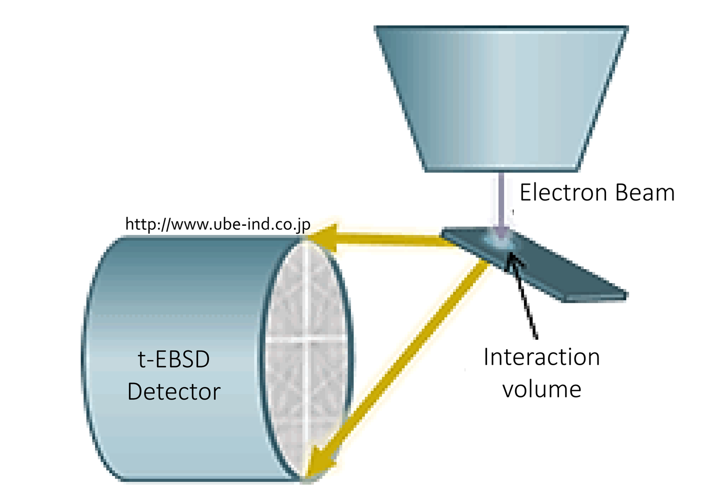
Genevieve Lee, Jonathan Orsborn, Antonio J. Ramirez
Welding Engineering Program, Department of Material Science and Engineering, The Ohio State University

Introduction & Background

- **Ni-based alloys** are used in conjunction with structural steels to maximize cost-efficiency where excellent corrosion resistance and high strength is required: pressure vessels, oil and gas pipes, power plants.
- They are often fusion welded together, causing weldability issues stemming from the disparity of material properties observed in melting, mixing, and solidification of the alloys.
- **Solid-state (SS) welds** can circumvent these issues by welding below melting temperatures, however other problems may be introduced to the material.
- Microstructural characterization has been conducted by J.Rodriguez, but not extensively at spatial resolutions finer than 20 µm.
- **Transmission Kikuchi Diffraction (TKD)** or transmission Electron Backscatter Diffraction (t-EBSD) is a Scanning Electron Microscopy (SEM) method that can provide crystallographic structure and orientation analysis. Paired with Energy Dispersive X-ray Spectroscopy (X-EDS) which provides chemical data, samples can be characterized at spatial resolutions up to 10 nm.



Friction Stir Welding (FSW) is a SS weld method that metallurgically bonds metals by plunging a rotating pin into a lap or butt joint and stirring or plasticizing the metal through heat generated by friction and deformation.



TKD patterns are created by transmitting and diffracting electrons in the SEM through a thin foil less than 1 µm in volume

Method

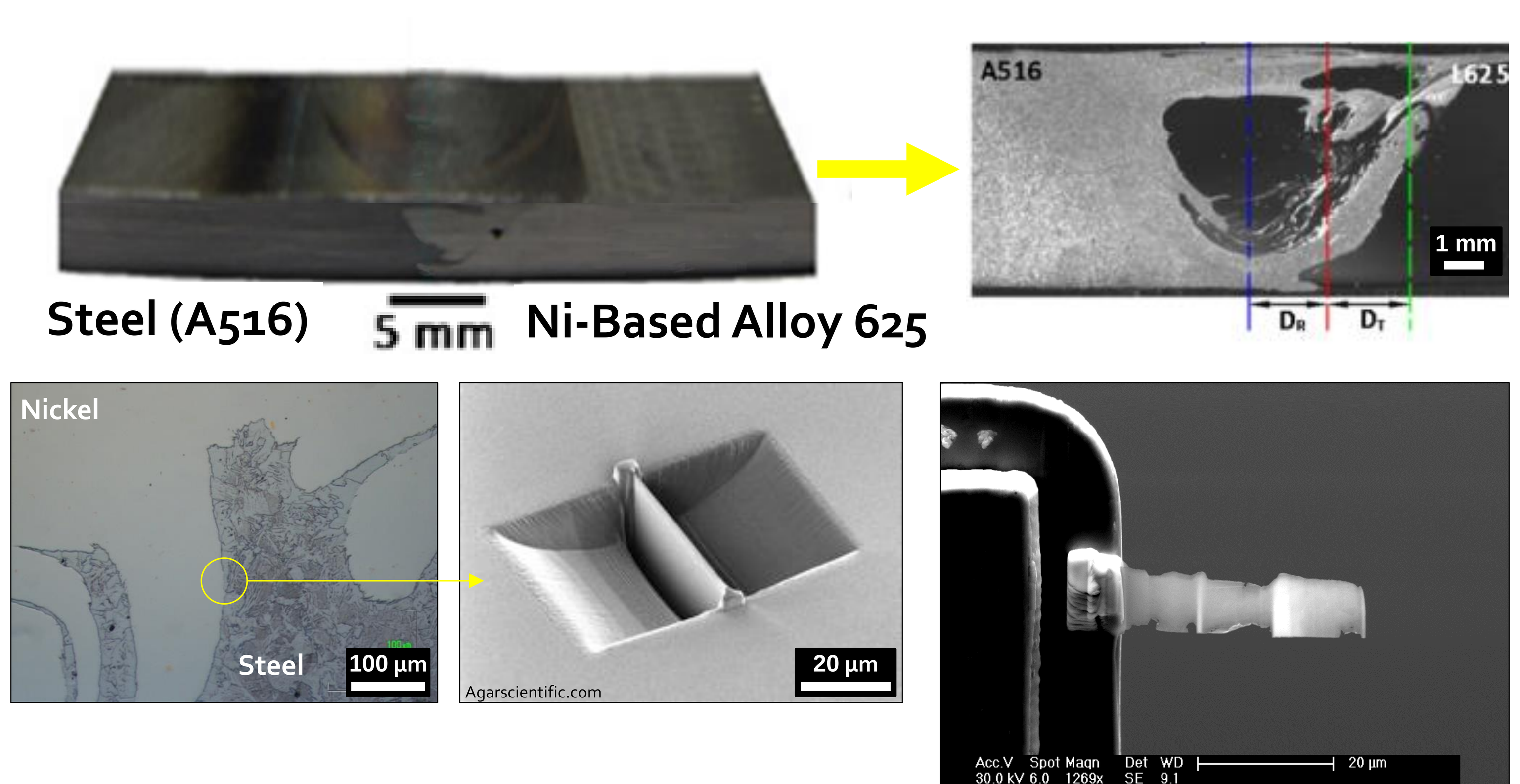


Fig 1. Weld Sample Preparation:
A cross section of the FSW between Ni-based alloy 625 and ASTM A516 Steel was polished and etched to reveal microstructure. At the Ni-Steel weld interface, thin foils 30x5x0.5 µm were created and mounted for TKD and X-EDS analysis.

Results & Discussion

TKD – Crystallographic Mapping

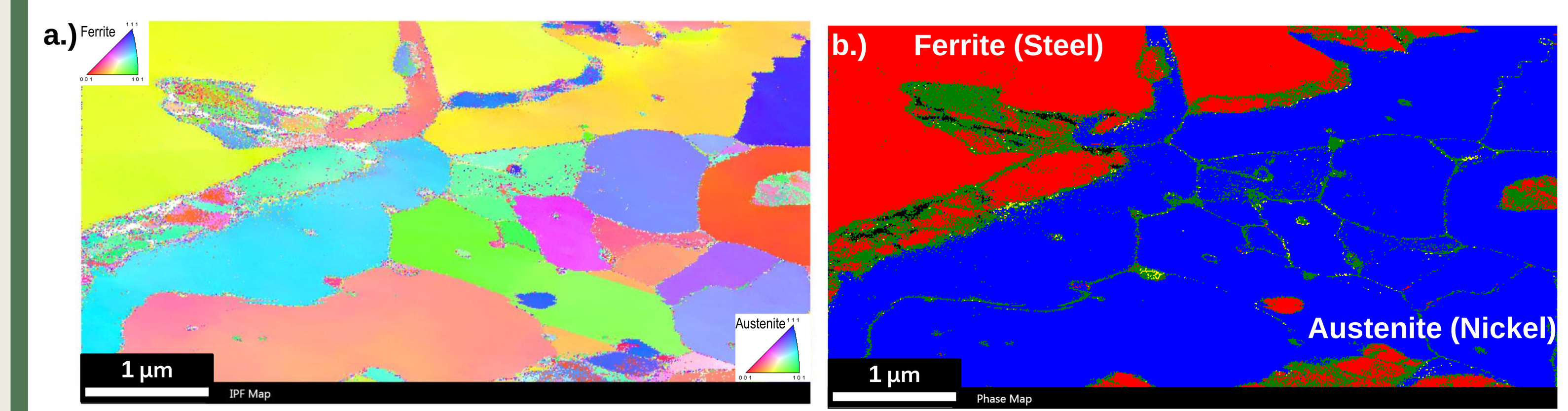


Fig 2. IPF Map and Phase Map: The figure on the left presents crystal orientations or Inverse Pole Figures (IPF) of grains at the region of interest. On the right, crystal structures are depicted in red for Body Centered Cubic (BCC) structured Steel Ferrite; blue is Face Centered Cubic (FCC) structured Nickel Austenite; green is FCC Cr_6C.

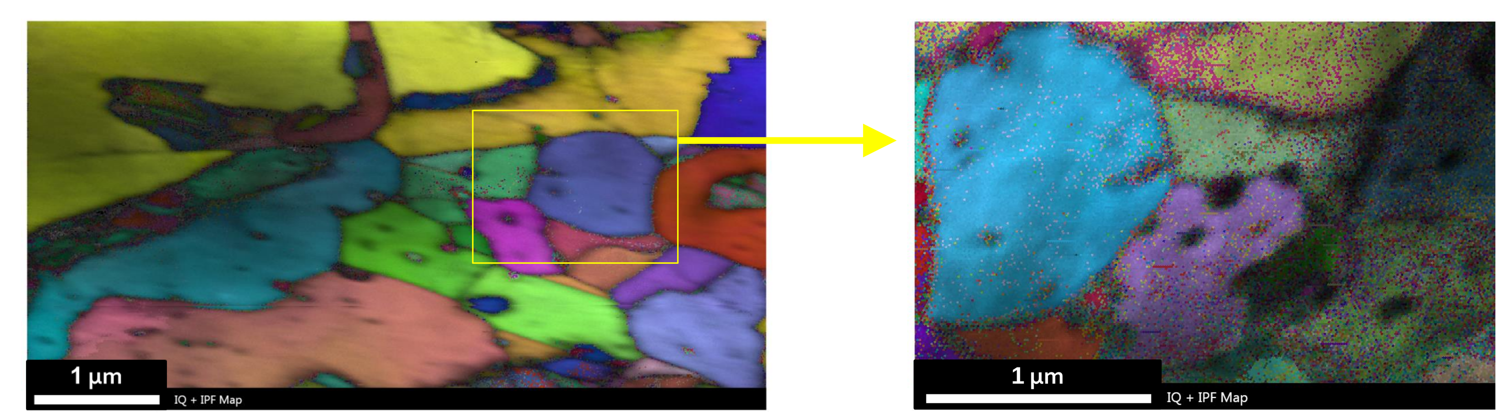


Fig 3. IPF and IQ Maps: The figures show IPF + Image Quality (IQ) measurements on the same scans, confirming locations of grains and precipitations within the sample. IQ is also affected by strain and dislocation and may indicate the presence of Martensite in steel.

X-EDS – Chemical Mapping

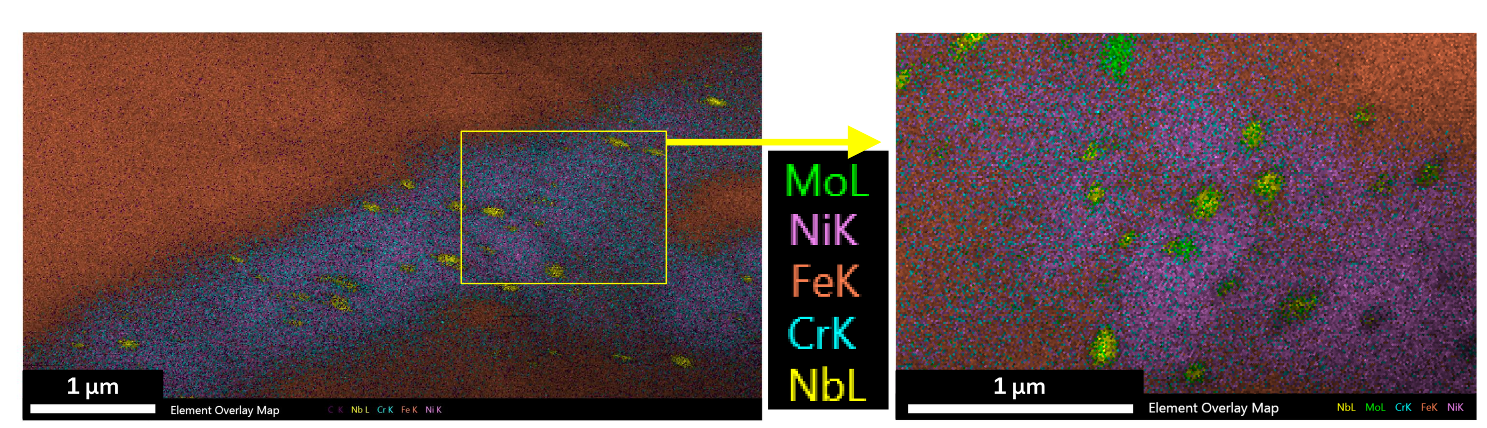


Fig 4. Element Overlay Maps: The figures show chemical composition measured by X-EDS run simultaneously with TKD, seen in Fig 2. Presence and composition of precipitates can be observed.

Chemical Assisted Crystallographic Mapping

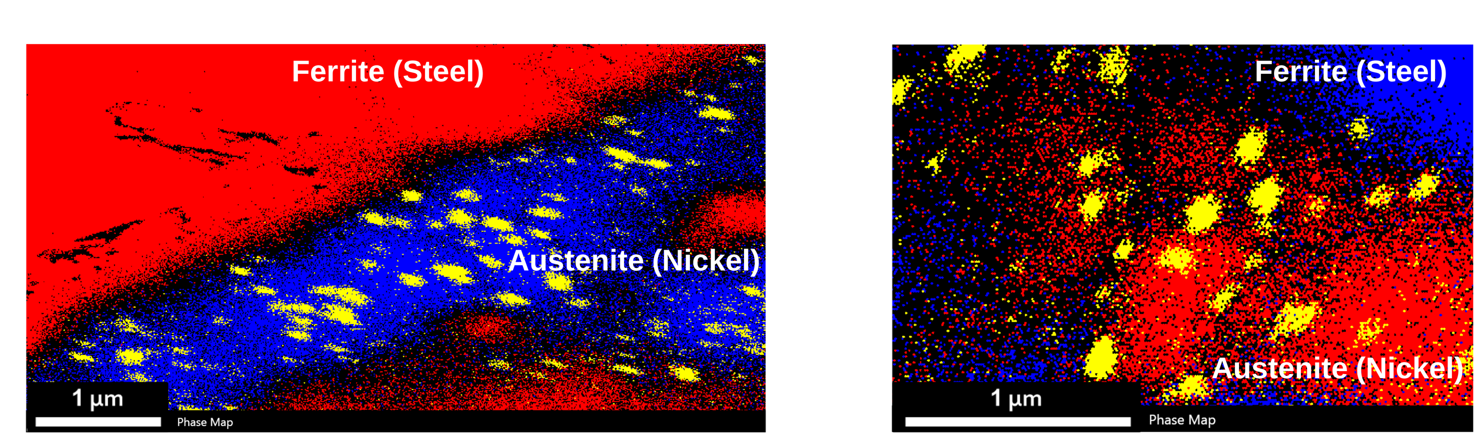


Fig 5. Rebuilt Phase Maps: The figures above show phases determined by TKD orientation, seen in Fig 2b, filtered by X-EDS data, seen in Fig 4. Precipitates of NbC are shown in yellow.

Future Work and Impact

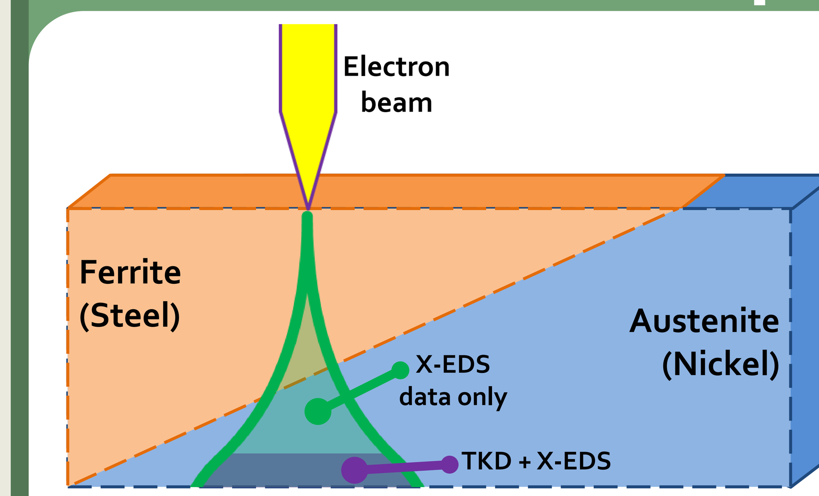


Fig 6. Electron Beam Interaction of TKD and X-EDS: TKD data examines only the bottom layer of the foil, X-EDS averages the entire thickness. Interface angles produce discrepancies between analyses. Here, TKD detects Nickel only, X-EDS recognizes both.

Work must be done on the interface region of Steel and Ni-alloy to accurately identify where mixing occurs with respect to crystal structure.

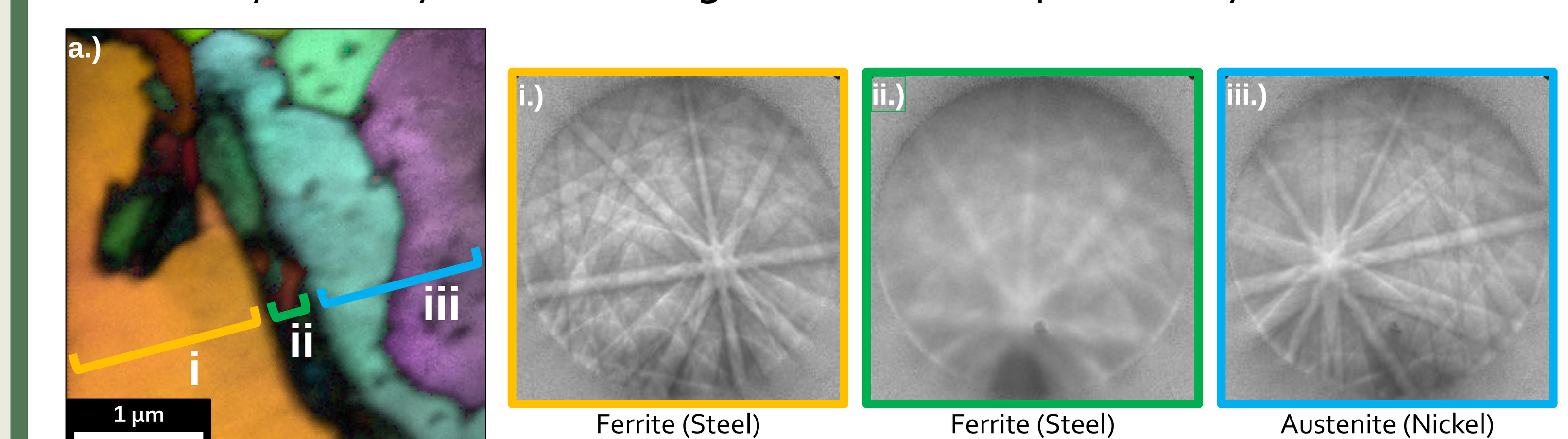


Fig 7. Transmission Kikuchi Patterns: Crystal diffraction patterns of the interface region can be seen above.

The blurry ferrite pattern from the region of fine grains at the interface may indicate the presence of martensite, a metastable, strained BCT structure similar to BCC ferrite. Future work with Nanoindentation will be done to ascertain the identity of this region.

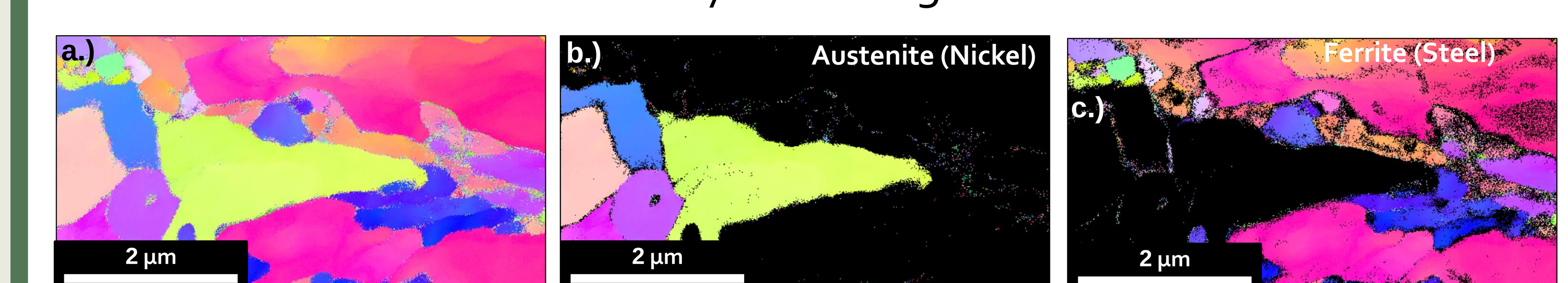


Fig 8. TKD Maps on Foil 2: TKD crystal orientation maps are shown above of a second foil taken from the same cross section sample. Figure 8b. shows only FCC Austenite grains, and c. presents only BCC Ferrite.

More analysis must be done throughout the cross section, as different metallurgical phenomena may occur at various regions of the FSW. More complex foils are seen at the shoulder and edges of the weld.

Impact

FSWs of Ni-based alloy and steel have not been studied extensively, especially at spatial resolutions of 10 nm. TKD and X-EDS allows rapid identification of samples with fine metallurgical features at high resolutions. Additionally, the development of TKD at OSU enables fast but high resolution characterization for specimens of any research study.

Acknowledgements & References

References

- J.R. Rodriguez and A.J. Ramirez, Mater. Charact. **110** (2015), 126-135.
- J.R. Fernandez, 'Microstructural characterization of ASTM A516 - Ni based alloy 625 friction stir welded joints' (Doctoral Dissertation). 2013.

Acknowledgements

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