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Metals Characterization by Principal Component EDS Analysis and EBSD

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INTRODUCTION

- Most metallurgical applications require knowledge of the distribution of both the crystallographic phases in a sample and the elemental composition. These two datasets are also linked since alloying elements act as stabilizers for the crystal phases of the balance element, e.g., nickel is an FCC γ-Fe stabilizer and molybdenum is a BCC α-Fe stabilizer. (Metallic nickel is FCC and metallic molybdenum is BCC.)
- Concurrent EDS-EBSD is a well-established tool for gaining full understanding of the microstructure crystallography and texture, as well as the chemical composition.
- The mechanical properties (local and bulk) can have a complicated relationship with the elemental and crystallographic compositions, so both must be known. To predict properties:
  1. Crystal phase (e.g., FCC or BCC) mostly identifies physical properties by determining slip planes; therefore, yielding stress and ductility
  2. Elemental composition determines thermodynamic stability of FCC vs. BCC phases, indirectly affecting mechanical properties
  3. Elemental composition also weakly but directly affects mechanical behavior through solution strengthening mechanisms
  4. Microstructural distribution of the above three effects, determines bulk properties

METHODS

- Sample
  - Cr-Ni-Mo duplex stainless steel with Nickel-rich weld filler material
  - Mechanically polished
- Analytical conditions for W-SEM
  - 20 kV accelerating voltage, ~10 nA probe current, dynamic focus, 70° tilt
- EDS analytical conditions
  - X-rays detected with single UltraDry EDS detector (60 mm² active area)
  - Each EDS-EBSD map area was acquired with 1024x884 pixels at 100x. Large area montage was done manually and single map section of interest was chosen for analysis
  - Component maps were extracted from spectral images using the COMPASS algorithm for multivariate statistical analysis [2,3]
  - Mean (3x3 kernel) and “Highpass 1” (3x3 kernel) filters used to show fine detail in component maps
- EBSD analytical conditions
  - Quasor II EBSD detector used to collect Electron Backscatter Patterns (EBSPs)
  - 15 ms exposure time with 2x2 binning
  - Map enhancement feature used on “High” to interpolate missing pixels and remove outliers
  - Grain ID feature used for large montage image

RESULTS

- EDS
  - **Weld:** Nickel-rich compared to base material with fine-scale Cr and Mo segregation at dendrite solidification front and long-scale Ni variation due to weld process instability
  - **Base:** Fine-scale planar duplex structure with Ni-rich phase in FCC and Mo/Cr-rich phase in BCC
  - **HAZ:** More chemically homogeneous than base material, Ni-rich phase found primarily along BCC grain boundaries, some located inside grains but are smaller and randomly oriented compared to planar duplex structure
- EBSD
  - **Weld:** Entirely face-centered cubic (FCC)
  - **Base:** Fine-scale planar FCC-BCC duplex structure. FCC correlates with Ni-rich phase
  - **HAZ:** FCC phase located primarily at grain boundaries of BCC phase. Significant grain growth of BCC phase at expense of FCC phase. FCC phase areas correlate with Ni-rich component “C3”

CONCLUSIONS

Concurrent EDS-EBSD gives a complete method for understanding the microstructure of the weld, base material, and heat-affected zone in a complex duplex stainless steel. However, standard EDS analysis did not extract the fine detail present in the heat-affected zone. This detail was instead extracted through primary component analysis (PCA) using the COMPASS algorithm and showed that the crystallographic phase distribution in this region was in fact due to chemical variation. This variation was not visible in the elemental distribution map without PCA.

REFERENCES

1. Keenan et al. Pat. 6,675,106 B1. 06Jan.2004.
2. Keenan et al. Pat. 6,675,106 B1. 24Jun.2003.

OVERVIEW

Motivating Questions: How has welding the duplex steel base material affected the starting base material microstructure near the weld? What is the distribution of alloying elements and how does this relate to the microstructure? Is the crystal phase distribution in the heat-affected zone (HAZ) related to variations in elemental chemistry?

Methods: Concurrent EBSD-EDS was executed using a Thermo Scientific™ Quasor II™ EBSD detector, a Thermo Scientific™ UltraDry™ EDS detector (60 mm² active area), and a Thermo Scientific™ Pathfinder™ microanalysis system. Data also attainable with Thermo Scientific™ Lumis™ EBSD detector. Primary component analysis (PCA) was performed using the Thermo Scientific™ COMPASS™ algorithm [1,2]. The elemental map of PCA component 3 was overlaid on top of the FCC EBSD phase map to highlight the correlation between the elemental distribution and crystal structure.

Results: An overlay between the PCA results of elemental distribution and the EBSD phase map showed that there is elemental segregation in the heat-affected zone (HAZ) with areas of increased nickel and decreased chromium located primarily along the BCC grain boundaries. These areas correspond with areas of FCC phase, indicating that the variations in crystal phase (BCC vs. FCC) do correspond with subtle elemental variation. This variation was not clearly apparent in the EDS elemental phase map which appeared to be homogeneous in the HAZ.

Fig. 1a: Large area EDS map of a welded Cr-Ni-Mo stainless steel with Ni-rich weld filler. Ni, Cr, and Mo atomic concentration maps were used as three channels for a RGB map as shown in the color legend. An area showing the weld, base material, and heat affected zone (HAZ) was chosen as a region of interest for further primary component analysis (PCA).

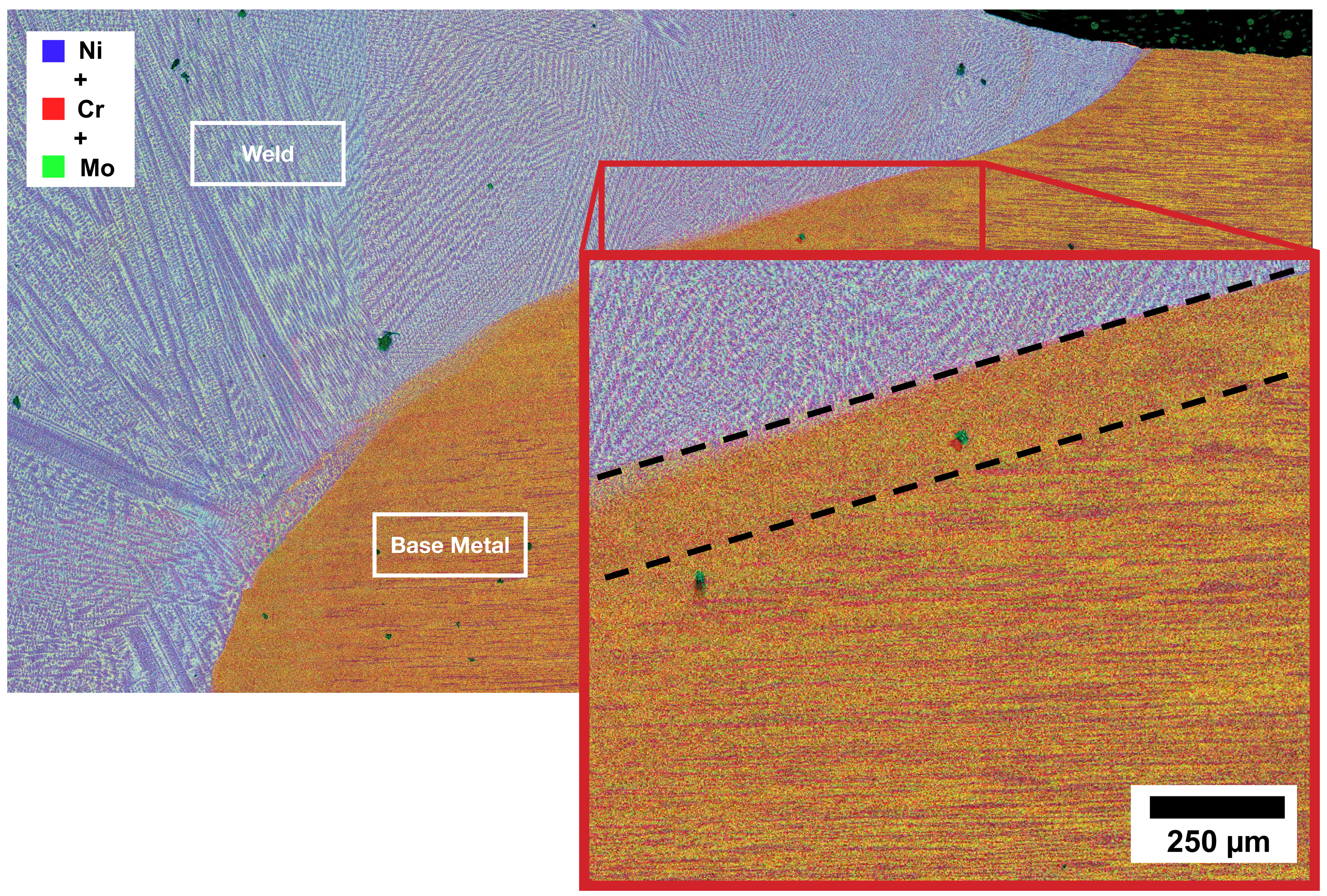


Fig. 1b: Large area EBSD map collected concurrently with Fig. 1a. The grain ID tool was used to colorize all grains across the montage.

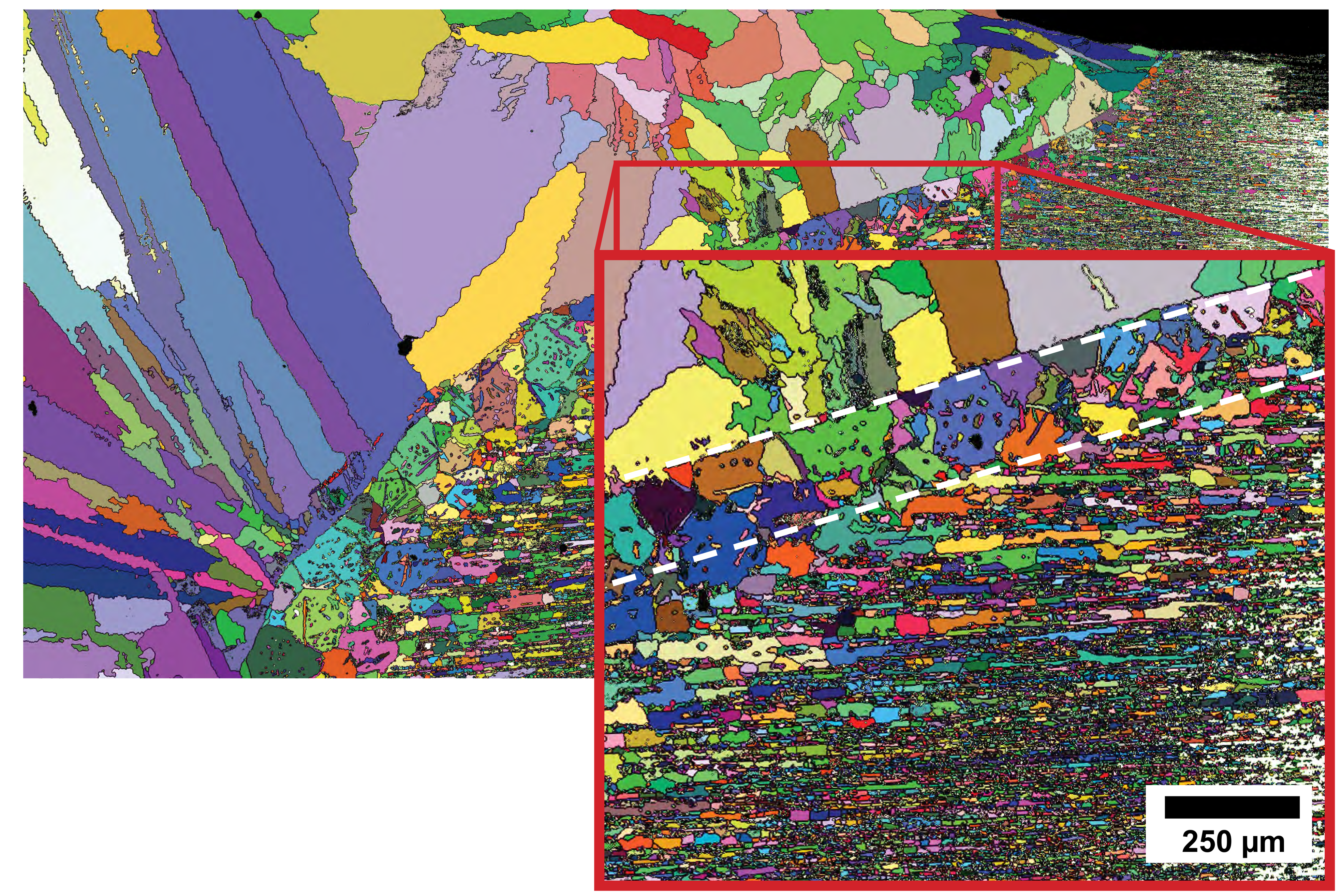


Fig. 2: Results of primary component analysis (PCA) using COMPASS algorithm: The overlaid EDS spectra indicate the components (C1, C2, and C3) identified by the COMPASS algorithm. The spectral maps show the relative intensity and distribution of these components across the analyzed area. The color of the spectra corresponds with the border color of the spectral images.

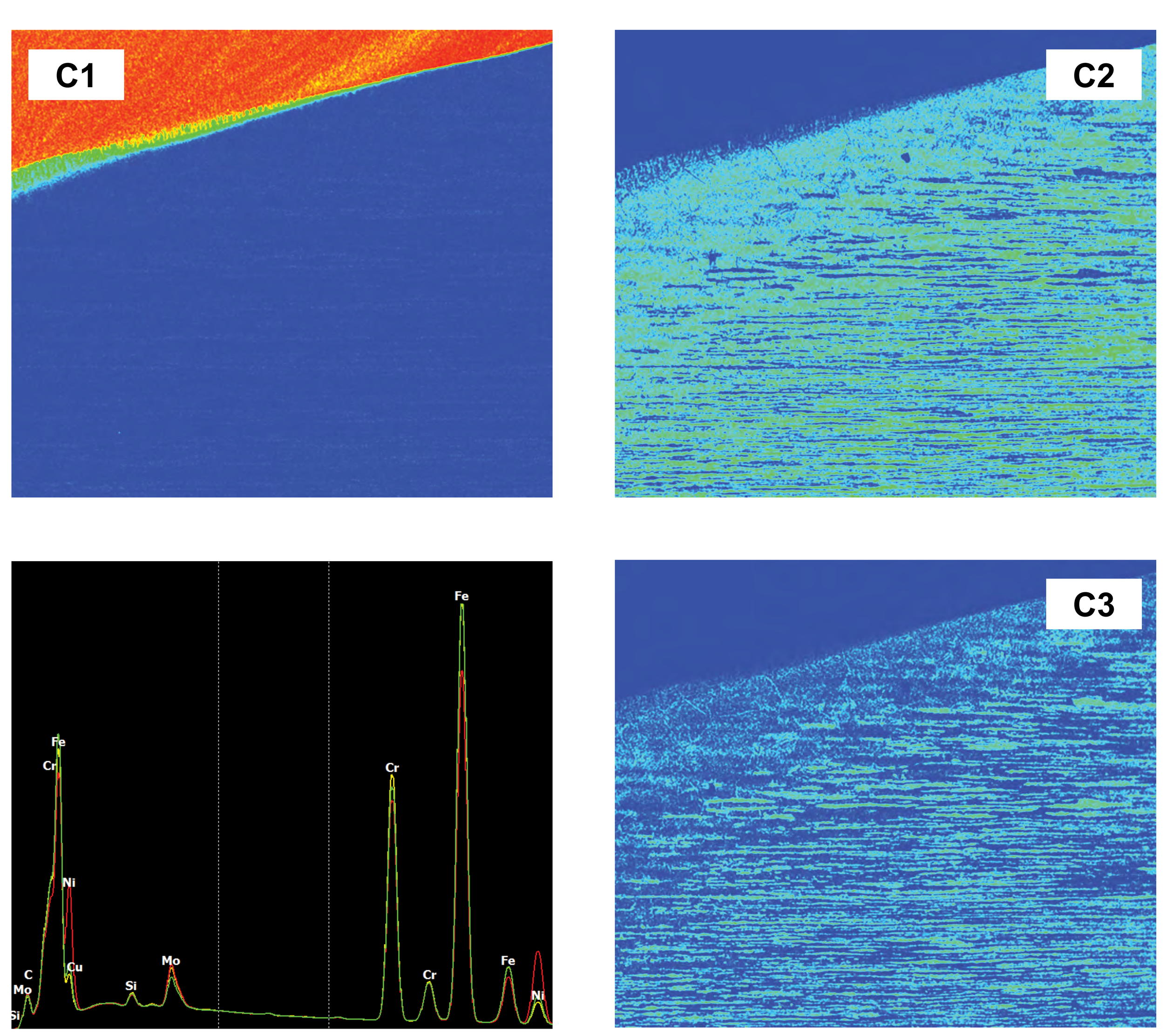


Fig. 4: Overlaid composite image of the “C3” spectral component (identified by the COMPASS algorithm on top of the FCC phase map) showing strong correlation between the two.

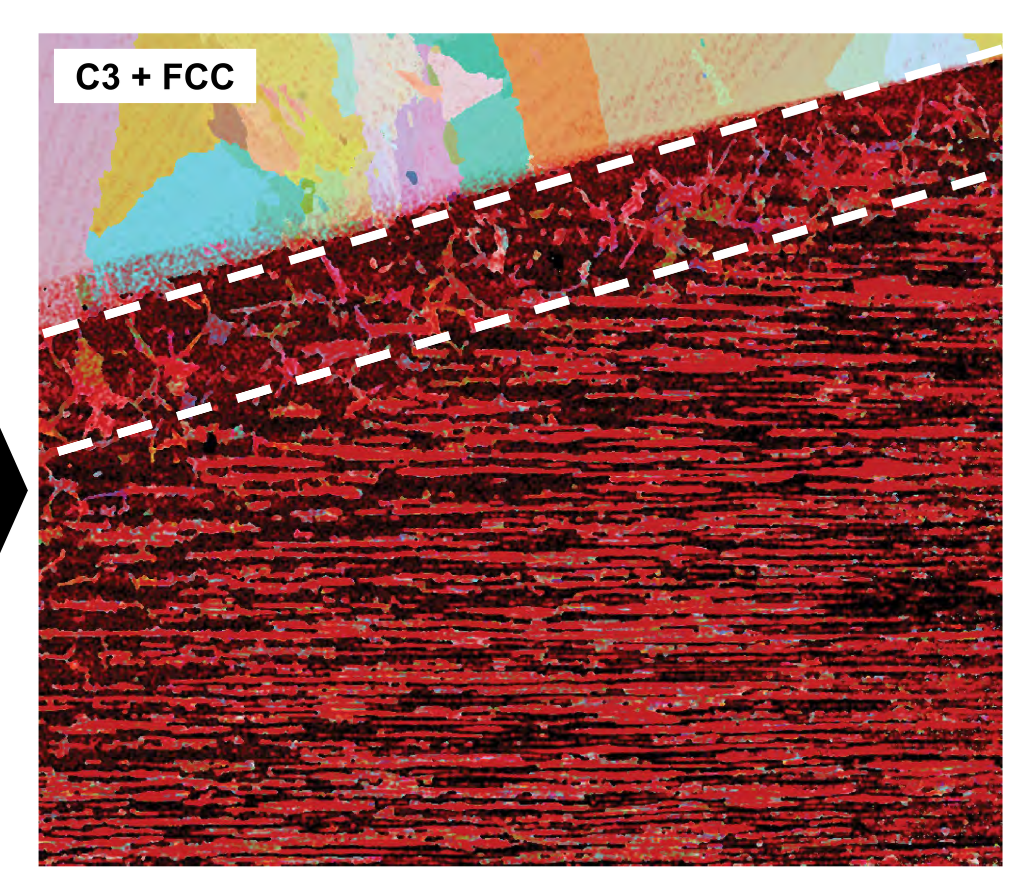
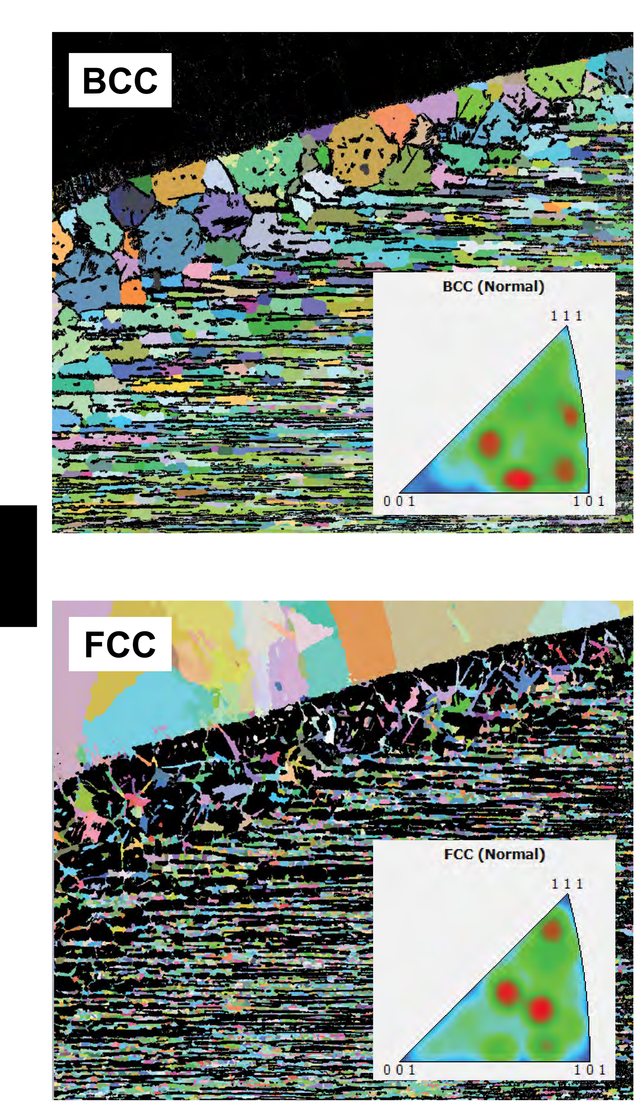


Fig. 3: EBSD phase maps for FCC and BCC showing the Euler orientations and inverse pole figure for each phase.



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