Improvements in the Characterization of Multiphase Materials by Automated Electron Backscatter Diffraction

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Automated electron backscatter diffraction (EBSD) or Orientation Imaging Microscopy (OIM) has become a well-accepted technique for characterizing local orientations in polycrystalline materials. As spatially specific orientations can be measured, it is possible to characterize orientation relationships existing between different phases in a polycrystal. One of the key aspects of characterizing multiphase structures is the ability to reliably differentiate the constituent phases during the scanning process. When the crystal structures are similar the phase differentiation capabilities of EBSD are limited. For example, consider a sample with two face centered cubic phases such as copper and nickel. The primary difference in the EBSD patterns from these two materials is the width of the diffraction bands. However, the difference in *d*-spacing of these two materials is only 2.5%. This slight difference results is reflected as a slight difference in width of the diffraction bands. This slight difference is not easily discerned in high-resolution resolution EBSD patterns, much less in the rapidly collected, low-resolution patterns typically used during scanning. However, these two phases are easily differentiated by composition. The chemical composition can be used in the phase differentiation process by simultaneously collecting elemental information via X-Ray Energy Dispersive Spectroscopy (XEDS) and crystallographic information via EBSD [1]. Counts for the constituent elements or full spectra can be recorded at each point in an OIM scan along with the diffraction patterns or, more efficiently, the locations of the detected bands in the patterns. With the elemental information provided by XEDS and the structural information derived from the EBSD patterns, it is possible, off-line, to greatly improve the reliability of the phase differentiation process.

The combined data can be used in several ways. One approach is to create minima and maxima filters for each of the chemical elements collected. A set of filters would be created for each constituent phase. In the nickel and copper example there would be two filters. For the nickel phase the nickel filter would be set up to accept points with high nickel counts and low copper counts. The opposite would be true for the copper phase. A second approach is based on the ideas of principal components analysis [2]. This is particularly useful when full spectra are collected. In the implementation presented here, a cluster analysis approach was used. In this approach the principal chemical components are determined by comparing spectra. If the spectra of two points in the scan grid are similar they are assumed to belong to the same component. By adjusting the tolerances defining the allowed deviation in spectra the components derived from the cluster analysis can be correlated to the constituent phases. Each scan point is assigned to one of the components and the associated EBSD pattern can be indexed using the structure parameters of the phase corresponding to the assigned component.

There may be cases where the chemical composition is the same but the crystal structure is different. A simple example would be ferrite (body-centered-cubic) and austenite (face-centered cubic). These two phases have the same composition but the structures are different enough to distinguish by EBSD alone. However, at some specific orientations if an inadequate number of bands are detected, it may be difficult to accurately differentiate between the bcc and fcc phases. A good example is the alpha (hexagonal) and beta (tetragonal) phases of titanium nitride. If only 6 bands are detected there are specific orientations where it is not possible to unambiguously identify the phase from a pattern from this corresponding orientation. Using simulated patterns from these two phases it is possible to predict suspect orientations [3]. This is shown in figure 1. Figure 2 shows the indexing solutions for both the alpha and beta phases for a simulated alpha phase pattern. This predictive tool helps optimize the indexing parameters for the phase differentiation process.

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[3] M. M. Nowell and S. I. Wright, Microsc. And Microanalysis, submitted (2004).

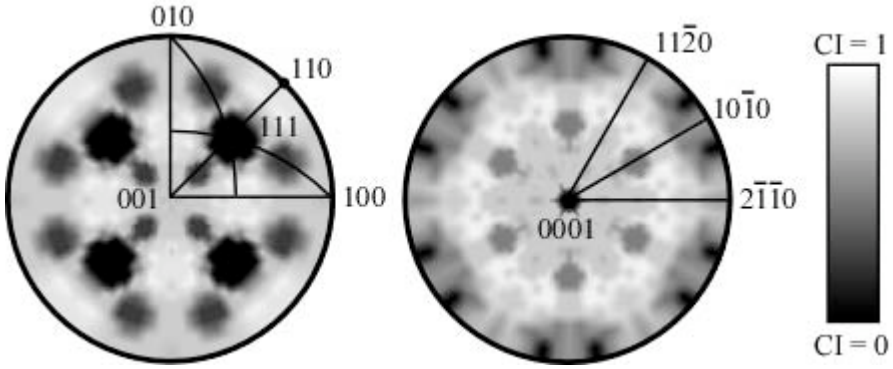

Figure 1. Inverse pole figures showing the confidence index (CI) as a function of orientation for simulated patterns of the tetragonal beta (left) and hexagonal alpha phases of titanium. A CI value of 0 indicates the pattern could not be unambiguously identified with either phase.

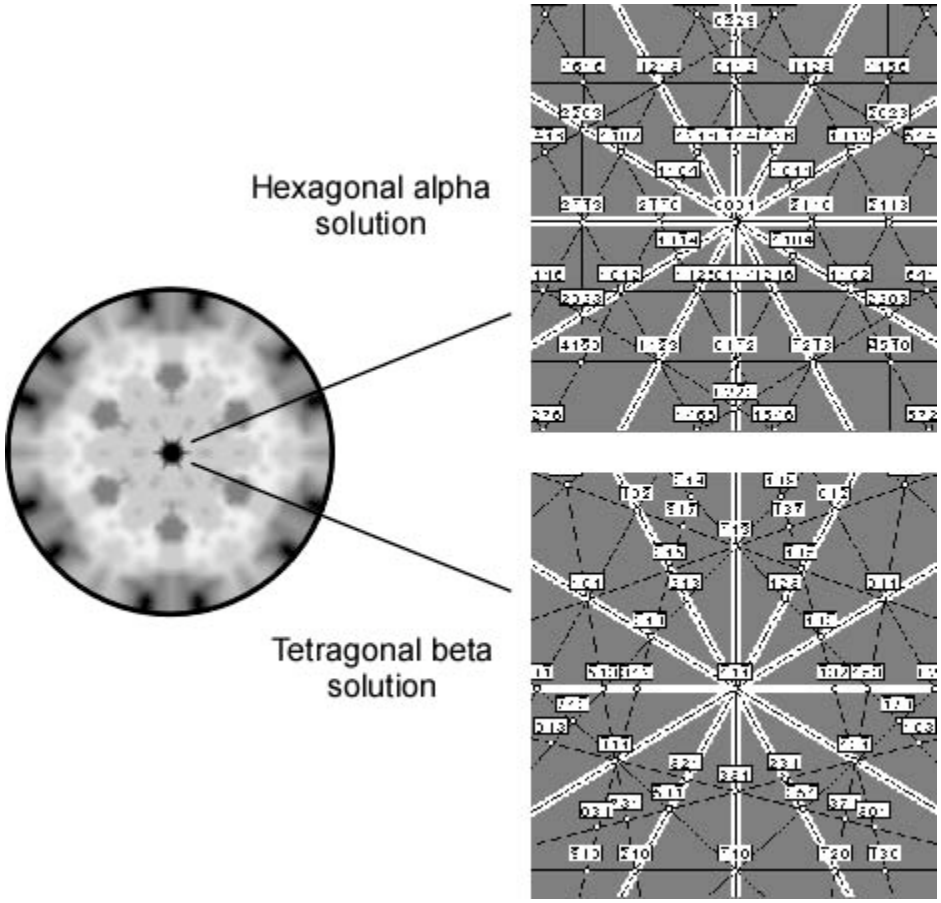

Figure 2. Simulated patterns (the white bands) for an alpha titanium crystal oriented with the basal plane normal to the EBSD phosphor screen overlaid with indexing solutions assuming a hexagonal alpha phase (top) versus a tetragonal beta phase (bottom).

1. Summary: EBSD is well suited for characterizing orientation relationships between phases in polycrystals. For accurate results, it is important to reliably differentiate between patterns from the constituent phases during the automated scanning process. When phases have similar crystal structures but dissimilar chemical composition, the reliability of the phase differentiation process can be dramatically improved by incorporating simultaneously collected chemical information via XEDS. In the converse situation, where the compositions are similar and the structures dissimilar there may be patterns from specific orientations that cannot be reliably identified. Using simulated patterns, suspect orientations can be identified and conditions optimized for improved reliability.

2. Keywords: EBSD, SEM, XEDS, phase differentiation