ChI-Scan for phase differentiation using combined simultaneously collected EDS and EBSD data

Materials Challenge

Electron backscatter diffraction (EBSD) pattern analysis can be used to identify both crystallographic structure and orientation; however, when the crystallographic structures present are the same or similar it can be difficult to uniquely identify the correct structure with confidence. By coupling the EBSD measurements with simultaneously collected Energy Dispersive Spectroscopy (EDS) chemical compositional information, the crystallographic structures can be more easily differentiated and a more complete phase description can be determined using both compositional and structural information. It is important to correctly identify constituent phases to enable accurate microstructural analysis of all phases present both collectively and individually.

Comparison with Existing Solutions

EBSD can often be used to identify and differentiate crystallographic structures in multiphase materials. Using only EBSD for differentiation can have the following shortcomings:

- Constituent phases with the same cubic crystal structure and diffracting planes produce identical inter-planer angles. This makes differentiation impossible when considering only these angles while indexing.

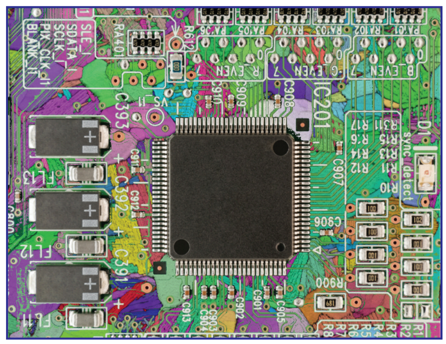
- Including diffraction line band-width analysis while indexing can help improve differentiation of phases with the same crystal structure. This approach, however, requires accurate determination of the band edges, which, in turn, requires higher resolution EBSD patterns. High resolution patterns take longer to collect and are more sensitive to changes in pattern quality due to intrinsic and extrinsic factors such as residual plastic deformation and sample preparation artifacts.

- Constituent phases with similar crystal structures can produce similar inter-planar angles, and the precision with which the bands are detected limits the ability to differentiate these structures. EBSD pattern resolution and quality will again directly impact the performance of phase differentiation.

- Indexing each point with each candidate crystallographic structure and determining the best-fit solution can become time consuming as the number of potential structures increases.

Chemical Indexing Scan or ChI-Scan, provides a solution for these shortcomings by combining the structural information measured with EBSD with the complete spectrum chemical information measured with EDS. Using simultaneously collected EDS data, the list of candidate crystallographic structures at each measurement position is reduced to only those matching the measured chemistry. This approach offers the following advantages:

- Constituent phases with the same or similar crystallography can be easily differentiated chemically using the simultaneously collected EDS information.



- High resolution EBSD patterns and diffraction line band-width detection are not required for structural differentiation. Data collection using faster lower resolution EBSD patterns yield superior results in shorter times and the differentiation is not as sensitive to sample deformation or preparation quality.

- The total data collection time is reduced as the number of structures to be analyzed at each point is minimized by the chemical filtering. This becomes more important as the number of structures present increases. Microstructures with as many as 12 phases have been analyzed.

- Analysis of the full chemical spectrum of EDS data provides automatic determination of compositional phases. Because the full EDS spectrum is saved at each analysis point, selected EDS elemental maps can be rebuilt at any time, and no prior knowledge of the composition is necessary for accurate analysis.

Microanalysis Results

Figure 1 shows EDS elemental maps of copper and iron and a ChI-Scan generated phase map with a copper phase and iron-nickel phase from a polished printed circuit board. In this sample, metallic interconnects are deposited using electrochemical deposition. Using the EDS spectral information, the phases present are identified as copper and kovar, an iron-nickel-cobalt alloy. Both of these phases are Face Centered Cubic with identical diffracting planes and similar lattice constants, making traditional differentiation with EBSD very difficult. The EDS elemental maps show the spatial distribution of the phases. ChI-Scan uses this information to select the appropriate candidate structure at each analysis point. With the correct phase and orientation determined, further microstructural analysis is now possible.

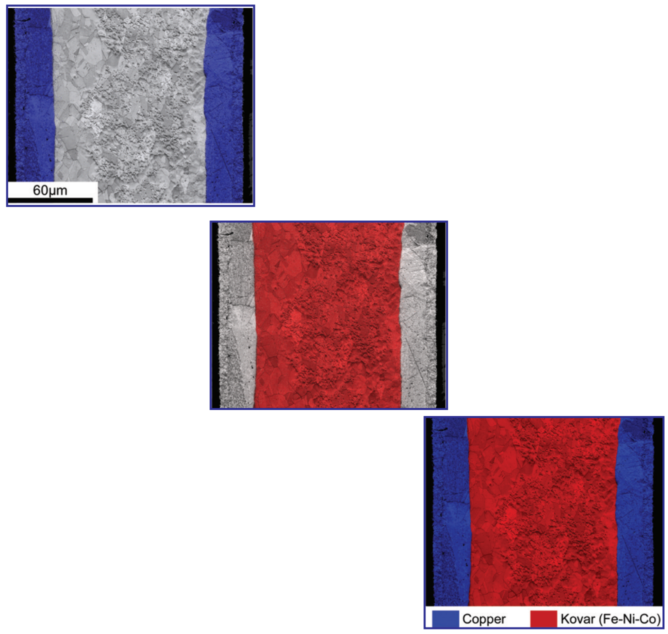


Figure 1. EDS elemental maps for copper (top) and iron (center) and ChI-Scan phase map (bottom) for printed circuit board metals.

Figure 2 shows grain maps from the copper phase (left) and the kovar phase (right), where grains are randomly colored to show size and morphology. The copper phase has a bimodal grain size distribution with larger grains adjacent to the kovar interface and smaller grains away from it. This suggests two different deposition and grain growth mechanisms were active during the deposition process. The kovar phase has a more homogeneous grain distribution. The grain size distributions from both phases are shown in Figure 3. Analysis of the grain misorientations indicates that the kovar phase has significant twinning (approximately 50% of the grain boundaries within the phase) while the copper phase has far fewer twin boundaries (approximately 7%). This type of detailed analysis would not be possible without the accurate phase differentiation provided by ChI-Scan.

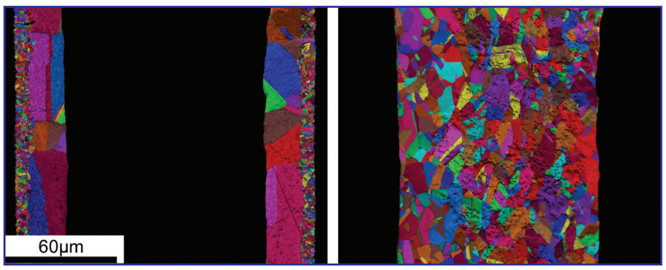


Figure 2. EBSD grain maps for copper phase (left) and kovar phase (right) showing a bimodal grain structure for the copper phase.

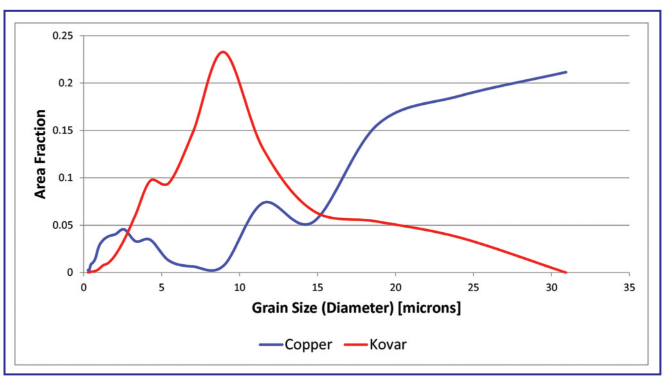


Figure 3. Grain size distributions for copper and kovar phases.

Recommended EDAX Solution

The ChI-Scan feature in TEAM™ Pegasus Analysis systems enables accurate phase differentiation and phase mapping for scientists and engineers characterizing multi-phase materials. ChI-Scan provides automatic chemical phase determination using full EDS spectral data to eliminate structural based ambiguities during EBSD pattern analysis. This technology improves the final data quality and decreases the data collection time to provide users with fast and accurate microstructural analysis. ChI-Scan can be used on metallic, ceramic, semiconductor, and geological multi-phase samples. Successful applications of ChI-Scan include carbide analysis in steels, oxide phase identification in rare earth magnets, inclusion analysis in aerospace alloys, and mineral analysis in copper ore bearing rocks. ChI-Scan requires simultaneously collected EDS and EBSD data which can be obtained using the Octane series of EDS detectors and the Hikari XP or DigiView IV EBSD detectors.