Accurate EBSD phase discrimination using Tru-I™

Introduction

EBSD is often used to discriminate between different phases in a material on the micro or nano scale. However, when two phases with the same crystal structure co-exist these phases are typically discriminated using their chemical composition, rather than directly from EBSD patterns.

Particles of Ti and N, and Ti, Mo and C, in a Nickel alloy are analysed to show the discrimination of two phases with the same fcc crystal structure and different lattice parameters. This application highlights a new algorithm in Oxford Instruments **AZtec**HKL system that enables phases of the same crystal structure to be discriminated from their respective EBSD patterns by using the diffraction band width in the solving algorithm.

The simultaneous acquisition of X-ray maps provides the elemental composition of the particles analysed.

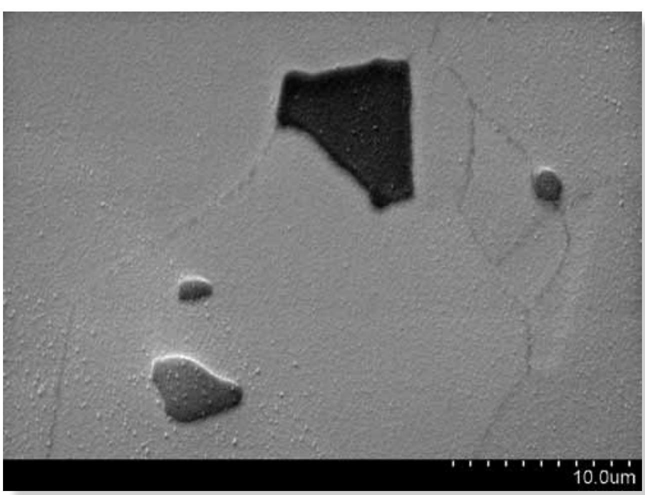

Figure 1 *SEM image showing an angular dark contrasting particle and several grey rounded particles.*

Experimental

Samples from a nickel alloy sheet were mounted in conductive bakelite and specimens prepared using standard mechanical polishing procedures for SEM-EBSD-EDS examination. The samples were examined in a FEG-SEM operating at acceleration voltage and probe currents of 20kV and 20nA respectively.

The Oxford Instruments **AZtec**® system coupled with HKL**Nordlys**Max and **X-Max**® detectors were used for EBSD and EDS acquisition.

Results and Discussion

An SEM image, Figure 1, shows several particles, including a dark contrasting angular particle and rounded particles with relief and grey contrast.

EBSD patterns from both the matrix and the particles are given in Figures 2, 3, and 4. The EBSD patterns from particles appear very similar, but are different in intensity and 'sharpness' to the pattern from the matrix. However, each pattern could be indexed using the nickel fcc phase, with acceptable mean angular deviations.

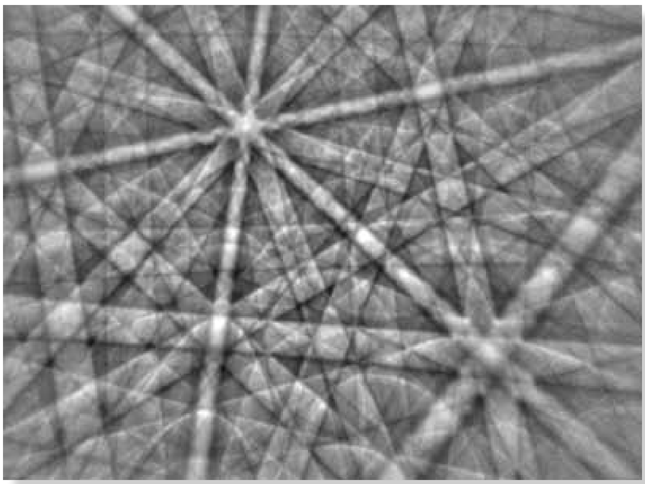

Figure 2 *EBSD pattern from nickel matrix.*

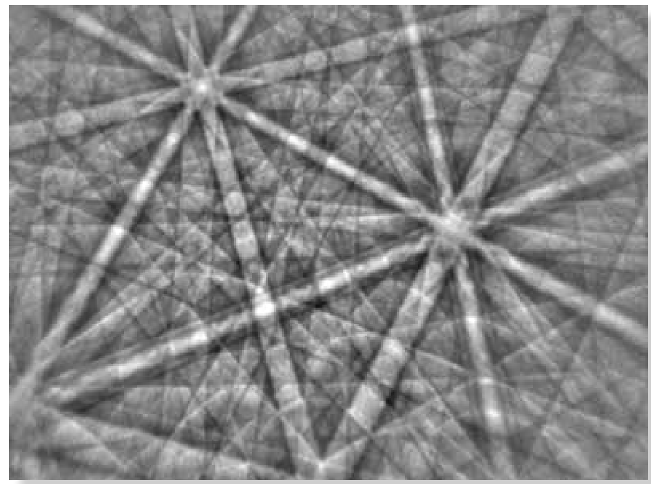

Figure 4 *EBSD pattern from the grey rounded particle.*

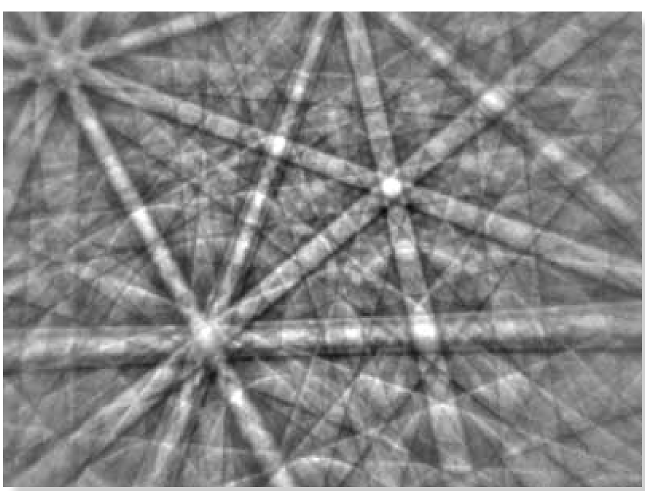

Figure 3 *EBSD pattern from the dark angular particle.*

The particles were investigated using EDS. The spectra in Figures 5 and 6 are from the angular and rounded particles respectively. The chemical data indicates that the former are rich in Ti and N while the latter in Mo, Ti and C. Both particle types could be indexed with acceptable MAD values using a TiN phase.

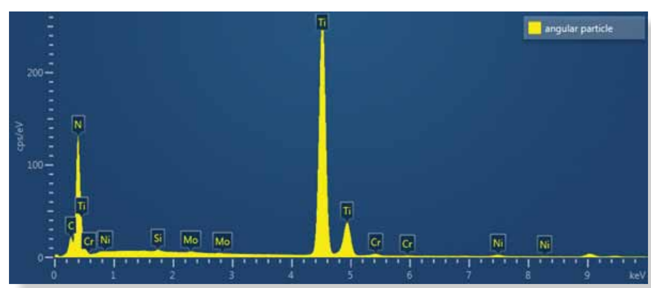

Figure 5 *EDS spectrum from the angular particle in Figure 1.*

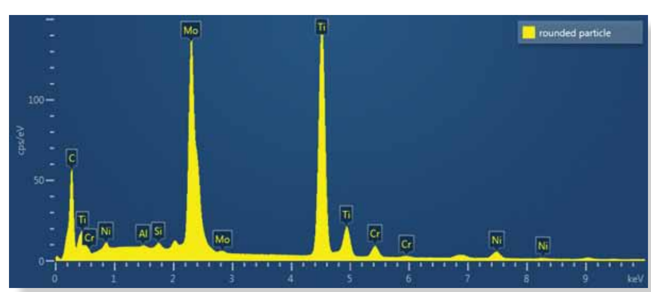

Figure 6 *EDS spectrum from a rounded particle in Figure 1.*

Despite these compositional differences the patterns from the two particle types appear very similar. In addition, the lattice parameter and crystal structures are likely to be similar for both phases, as Mo is substituted for Ti and C is substituted for N.

An EBSD map from the rounded particle is shown in Figure 7a, this map was solved using TiN and Ni, the blue pixels are Ni and the yellow is TiN. There is no discrimination between these two phases, on the map, however the particle is clearly visible in the band contrast image.

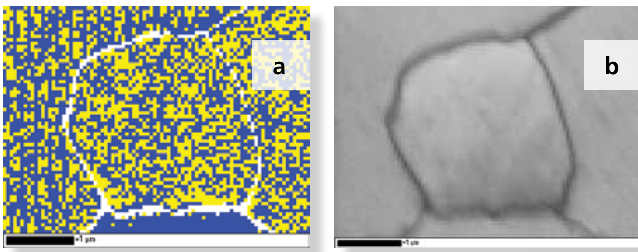

Figure 7 *a) Raw EBSD phase map blue=Ni and yellow=TiN phases, b) EBSD band contrast map clearly showing that a particle is present in the data in Figure 7a.*

There is a difference in lattice parameter between Ni = 0.345 nm and TiN = 0.424 nm, which is greater than 10% and will therefore affect the width of the diffraction bands. By including the band widths in the solver settings it is possible to discriminate these phases. The **AZtec**HKL software includes an algorithm to use 'band width' as criteria for sorting the solutions.

The experiment was repeated using the band width in the pattern solving. The results are shown in Figure 8a. The particle is well discriminated. A little amount of cleaning and extrapolating results in the maps shown in Figure 8b. The corresponding Ti and Mo X-ray maps are shown in Figure 9.

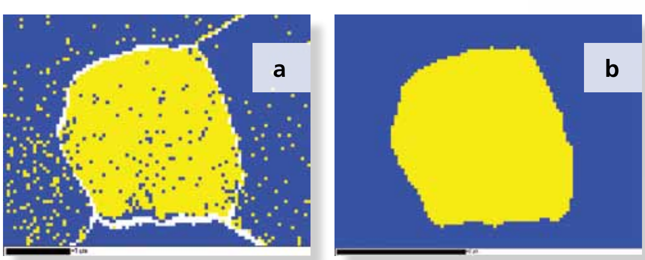

Figure 8 *a) Raw EBSD phase map using band width to sort the phases, Ni=blue, TiN=yellow. b) processed EBSD maps showing phase map.*

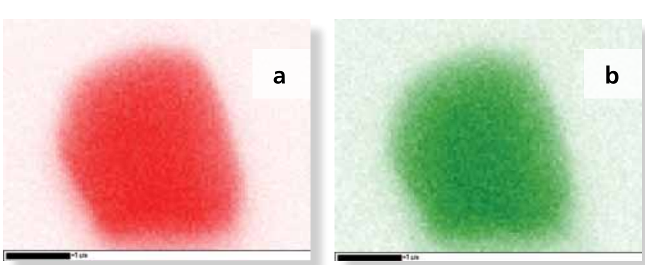

Figure 9 *a and b) EDS Ti, and Mo, maps respectively from particle in Figure 8.*

A larger map was collected, using the Ni and TiN phases, and solving using band width. The raw phase map, Figure 10, shows a clear discrimination of the particles present. The processed EBSD maps are shown in Figure 11 together with the X-ray maps.

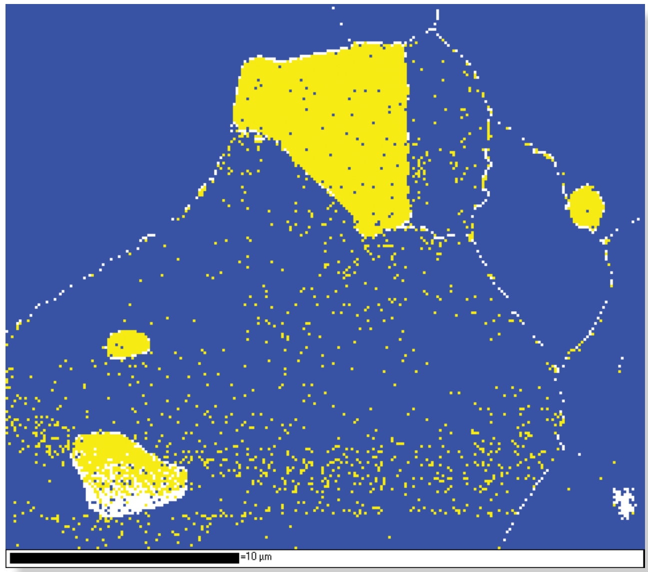

Figure 10 *Raw phase map from a region with several particles blue=Ni and yellow=TiN.*

Conclusion

This application note demonstrates the power in the Tru-I solver engine to discriminate between phases with very similar lattice parameters. Using the band width in the solving increases the capability of **AZtec**HKL to discriminate these phases, and on other systems this would only be possible by combining EBSD and EDS.

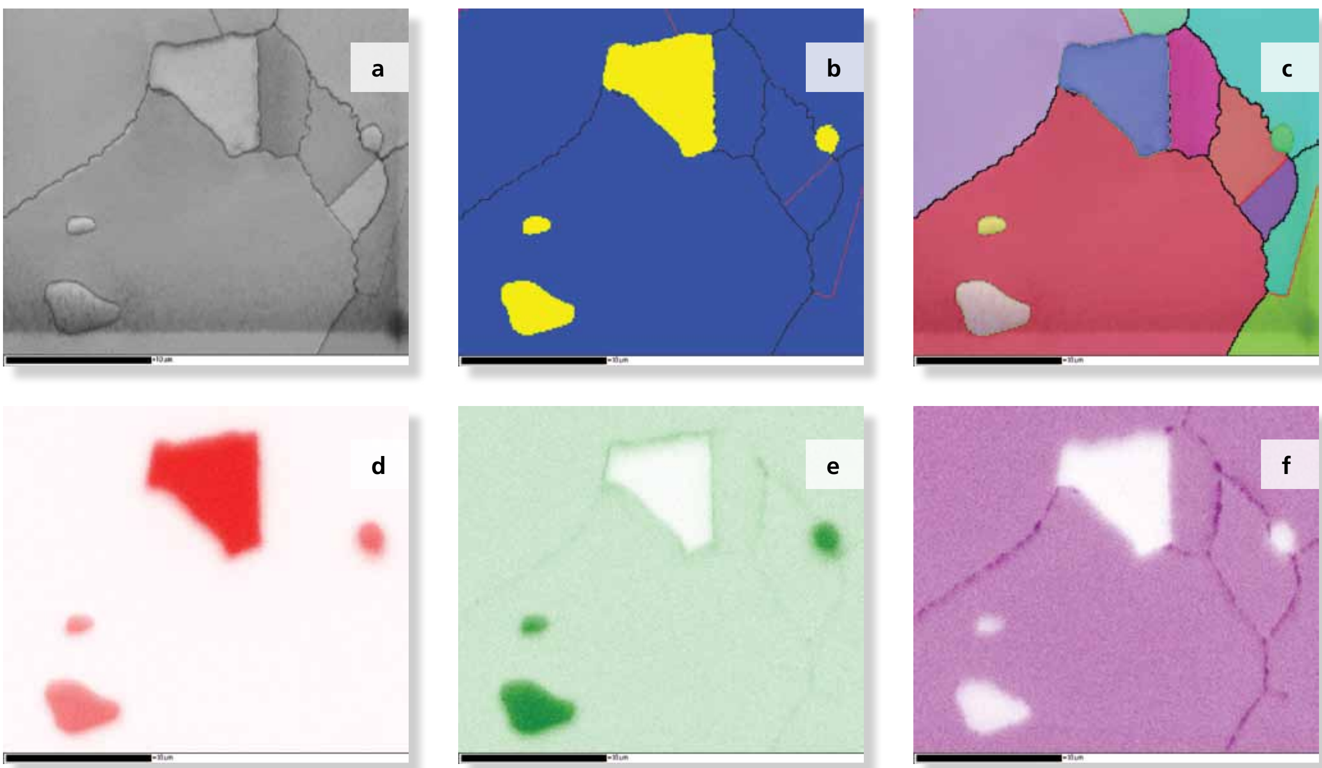

Figure 11 *a-c) Processed EBSD band contrast, phase and IPF maps respectively d-e) EDS maps for Ti, Mo and Cr respectively.*

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