TruPhase: solving phases with similar crystal structures but different chemistry

Application Note

Introduction

EBSD identifies crystalline phases on the basis of their particular crystallographic characteristics and allows the orientation of the mineral grains within a sample to be determined. However, it has traditionally been a challenge to differentiate between phases with very similar crystal structures. TruPhase, a feature in Oxford Instruments' **AZtec®** software, enables phases with very similar crystal structures to be differentiated by simultaneously collecting EBSD and Energy Dispersive X-ray Spectrometry (EDS) data. EDS data are used to refine the EBSD solution at each point. EBSD has a wide range of applications, including materials science, metallurgy and geology. Here we present two examples, a geological application and a metallurgical application, demonstrating how TruPhase can be used to discriminate between phases with similar crystal structures, but different chemistry.

Example 1: Geological application

In this application, EBSD is used to gain microstructural information about a sample of mylonitic metapelite (MSEF1) from the Cardeto Metamorphic Complex (Calabria, Southern Italy), in order to investigate wider deformation processes. Alongside quartz, albite, almandine and apatite, this sample contains both muscovite and biotite mica. These minerals have similar crystal structures; both are monoclinic, belong to the space group C2/m, and have similar unit-cell dimensions in all but the c-direction (Table 1.) Differentiating these two phases on the basis of EBSD analysis alone is difficult, even when the analysis is conducted under optimum analytical conditions (Fig. 2C). However, these mineral phases have different chemistry; most notably biotite mica contains higher concentrations of Fe but lower concentrations of Al than muscovite (Fig. 2).

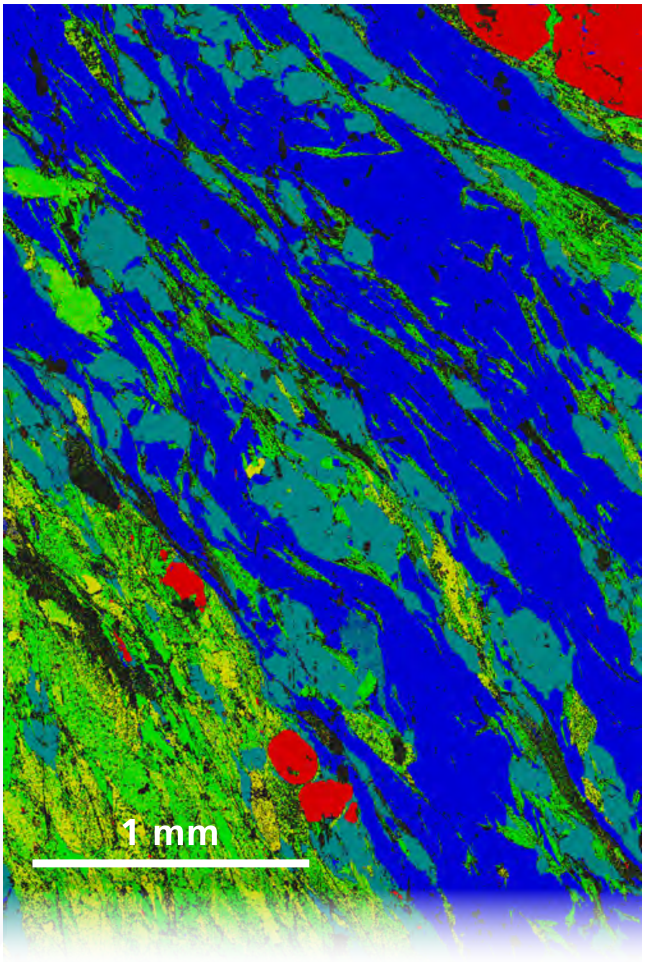

Fig. 1. A large area EBSD map of a mylonitic metapelite.

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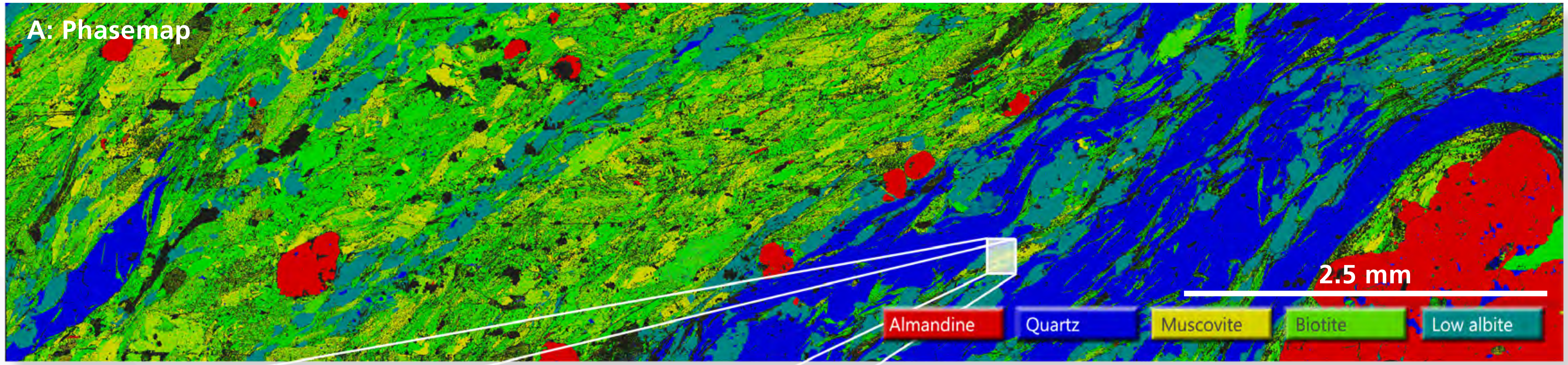
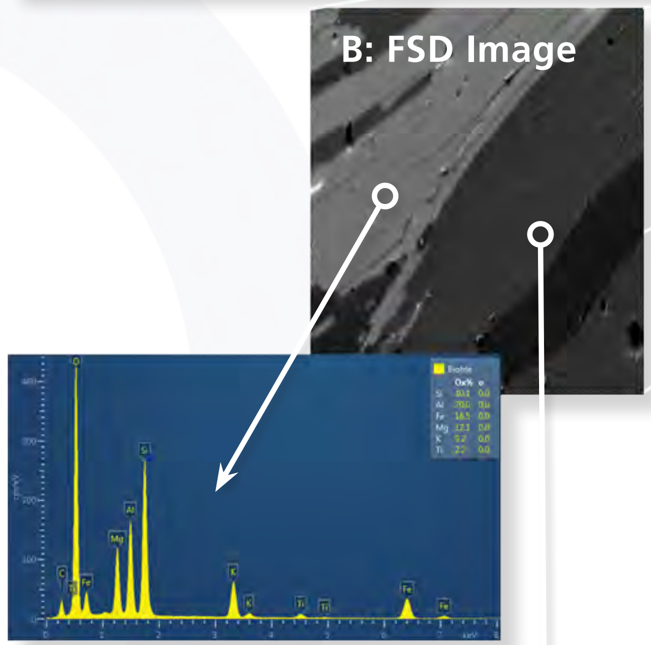
Methodology and Results

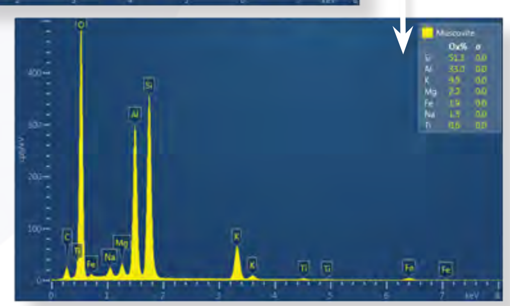
Following initial investigation of the sample, a reference EDS spectrum was collected for each of the mineral phases present in the sample (Fig. 2, A and B). The sample was then analysed using **AZtec** TruPhase - this simultaneously acquires both an electron backscatter pattern (EBSP) and an EDS spectrum from each of the analysed points. The obtained EDS spectrum is then used by **AZtec** to rank the possible EBSD solutions. As a result, TruPhase successfully differentiates between biotite and muscovite mica, as shown in Fig. 2D. This is an important result because the formation of different mineral phases in geological samples indicates different metamorphic conditions. In addition, by resolving the crystallographic relationships between muscovite and biotite mica using EBSD, we gain insights into the deformation behaviour of these minerals. EBSD analysis can be used to obtain mica crystallographic preferred orientations (CPOs), quantify crystal distortion, and to determine the dominant slip systems thus gathering information on the contribution of each mica phase to the overall schistosity of the sample.

	Biotite	Muscovite
Crystal structure	Monoclinic	Monoclinic
Space group	C2/m	C2/m
Unit cell parameters	a[Å]= 5.343	a[Å]= 5.189
	b[Å]= 9.258	b[Å]= 8.995
	c[Å]= 10.227	c[Å]= 20.097

Table 1. The crystal properties of biotite and muscovite mica are very similar and can be difficult to differentiate using traditional EBSD.







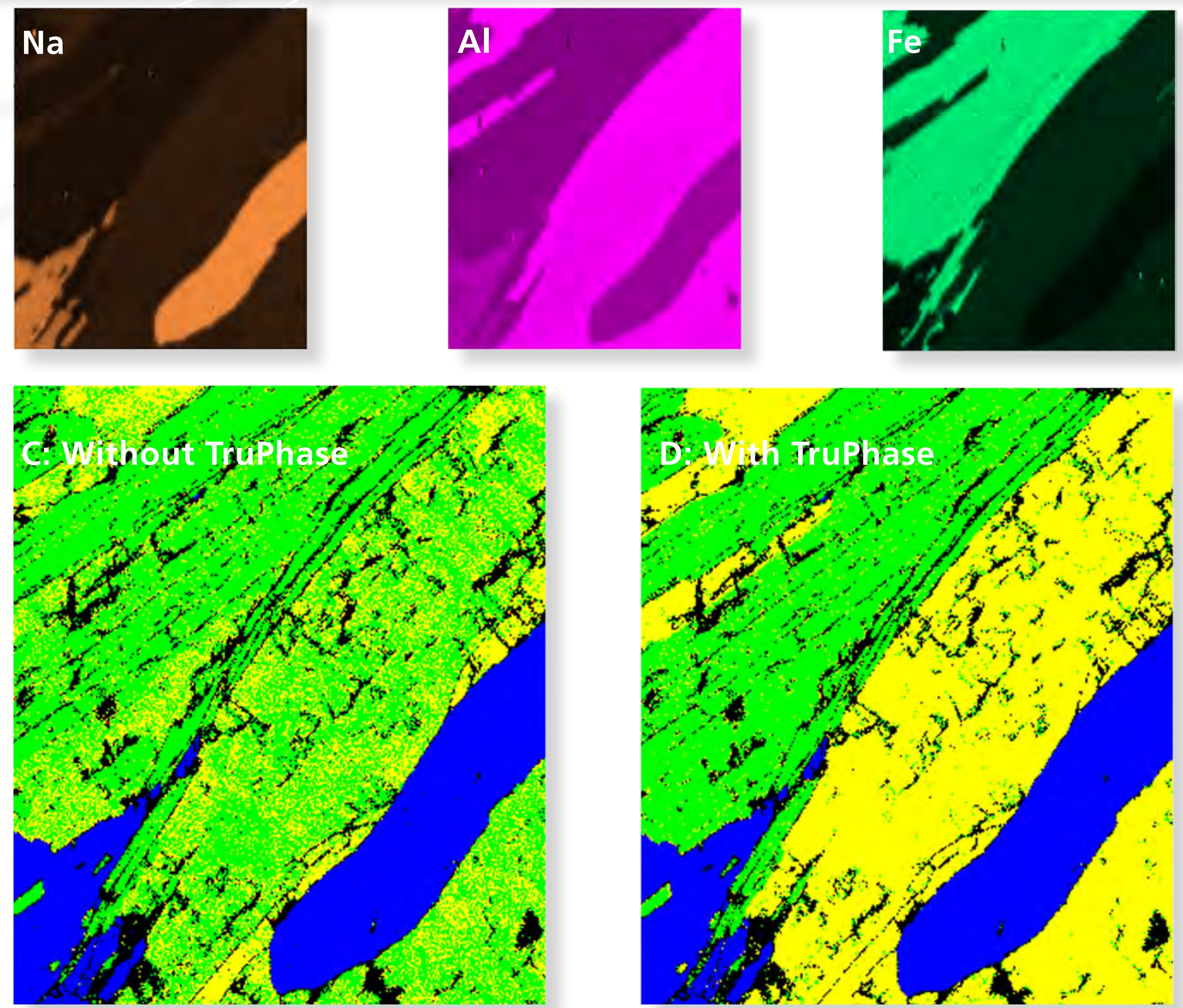


Fig. 2. (A) A large area map (LAM) acquired from 83 fields at 150X magnification for sample MSEF1 showing the distribution of the different mineral phases.

(B) A combined forescatter diode (FSD) image obtained for a small area of the sample and the reference EDS spectrum acquired for biotite and muscovite mica.

*(Opposite page) Element maps (Na, Al and Fe) acquired from a small area of the sample. High concentrations (i.e. brighter regions on the map) of Al indicate muscovite mica, higher concentrations of Fe indicate biotite mica, and higher concentrations of Na indicate of albite. A phase map acquired for the same area using traditional EBSD (C) demonstrating that it is difficult to distinguish biotite and muscovite mica. A phase map collected using **AZtec**'s TruPhase (D) showing the successful differentiation of biotite and muscovite mica.*

The geological sample used for this application note was kindly provided by Dr Elisabetta Mariani, University of Liverpool.

Example 2: Metallurgy Application

In this example a hole in a copper gasket has been filled with solder. Due to the heat applied to melt the solder a reaction has occurred between the solder and the surrounding copper. In order to investigate the phase transitions the sample was analysed using EBSD.

The sample contains copper, lead, tin and various combinations of copper and tin, where the copper and tin have mixed during the heating and melting of the solder. Copper and lead have very similar crystal structures; both phases are cubic, belong to the same space group (Fm-3m), and have similar unit cell dimensions, thus making differentiation on the basis of EBSD analysis alone difficult. However, these phases have very different chemistries and therefore can be differentiated using **AZtec** TruPhase.

Methodology and Results

The sample was initially analysed and mapped on the basis of EBSD analysis alone, and as shown in Fig. 3 (top) the copper and lead are not successfully differentiated on this basis; no copper appears in the sample. The sample was then investigated by collecting EDS maps to determine the distribution of the copper. After this characterisation a reference EDS spectrum was then collected for each of the phases present in the sample (Fig. 3 centre). The sample was then mapped using **AZtec** TruPhase and, as shown in Fig. 3 (bottom), the copper and lead phases have been successfully differentiated using this method. This demonstrates how simultaneous EBSD and EDS analyses in **AZtec** can be applied to identify and solve complex phase transformations.

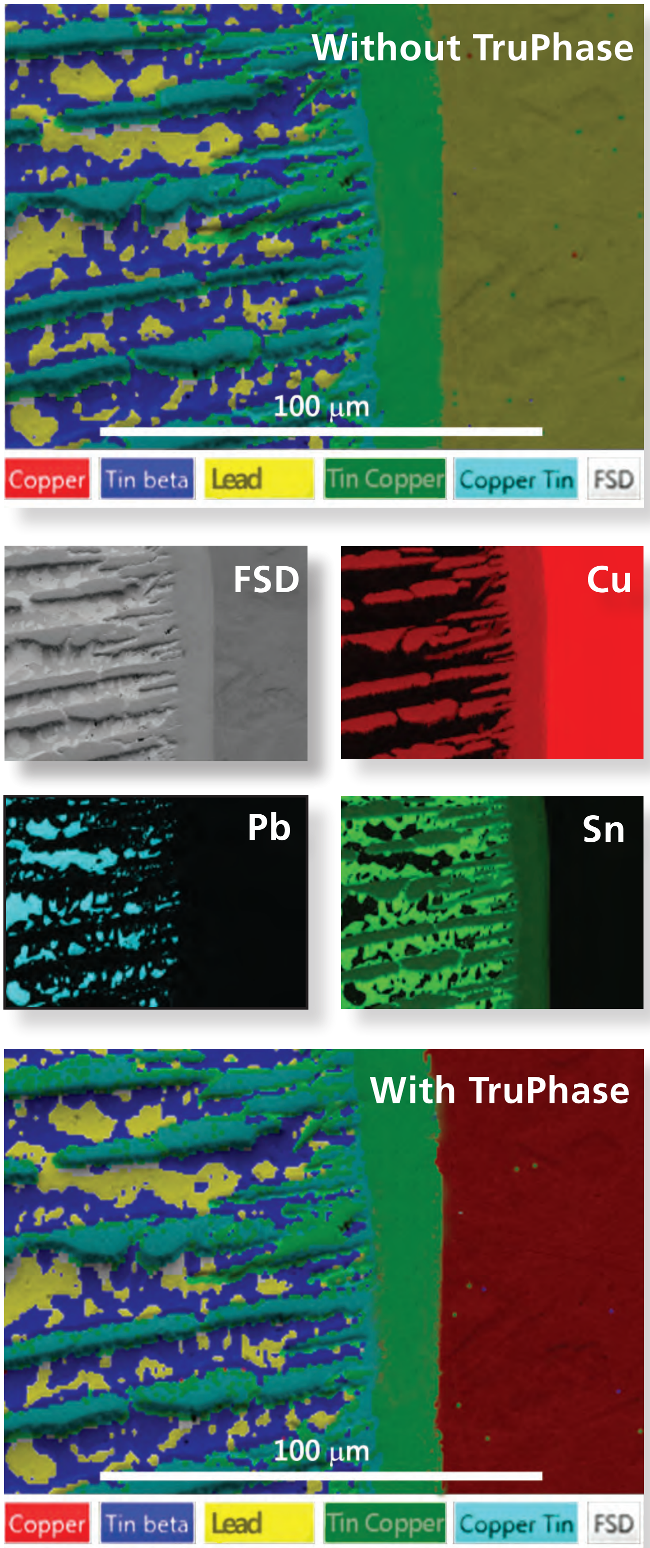


Fig. 3. (Top) A phase map collected using traditional EBSD which struggles to differentiate between the copper and lead present in the sample due to their similar crystal structures.

(Middle) FSD image and elements maps (Cu, Pb and Sn) acquired for the same area of the sample.

*(Bottom) A phase map collected using **AZtec** TruPhase showing the successful differentiation of the copper and lead within the sample.*

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