

Validating a New Approach to the Mapping of Phases by EDS by Comparison with the Results of Simultaneous Data Collection by EBSD

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When the goal is to find regions of similar material composition (“phases”) clustering techniques can be more flexible and sensitive than approaches based on “MSA”, multivariate statistical analysis [1]. MSA can be used to identify the minimum number of significant “components” necessary to represent a dataset but the number of phases can easily exceed the number of components because a phase can be any mixture of components.

A new approach, AutoPhaseMap, uses a clustering approach applied to any number of images derived from X-ray spectral image data. These images are typically X-ray maps corresponding to bands of energy in the X-ray spectrum or can be “TruMaps” which plot the X-ray intensity for specific characteristic X-ray emissions, determined by processing individual pixel spectra to correct for background and peak overlap.

“Clusters” are groups of pixels showing a similar spectral profile as defined by the intensities in each of the maps. The algorithm splits all the pixels into groups on a statistical basis that takes into account the different statistical precision of each input map. For the resulting clusters, every pixel will be within a certain distance of the centroid of an n-dimensional hyperellipsoid that is spread according to the Gaussian statistics of the input data. Unlike algorithms such as “k-means”, there is no limit to the number of clusters that can be found and if there was systematic variation across regions in addition to statistical measurement scatter, the algorithm would pick up more phases than an operator would consider necessary. Therefore, an additional pass is applied that groups together clusters that have significant overlap. In the vicinity of boundaries between phases, there can be a large systematic variation, particularly when the information volume, due to electron scattering, extends over more than one pixel. The operator can reduce the tolerance on this systematic variation to reject “boundary” pixels that may be a mixture of real phases.

An example of the practical use of this method is shown in Fig. 1A, where a map acquisition from an oceanic gabbro sample [2] is used. AutoPhaseMap reveals the microstructure and modal abundance of the phases in the sample. To validate this method, the result has been compared to phase map results determined by EBSD on data collected simultaneously with the EDS acquisition.

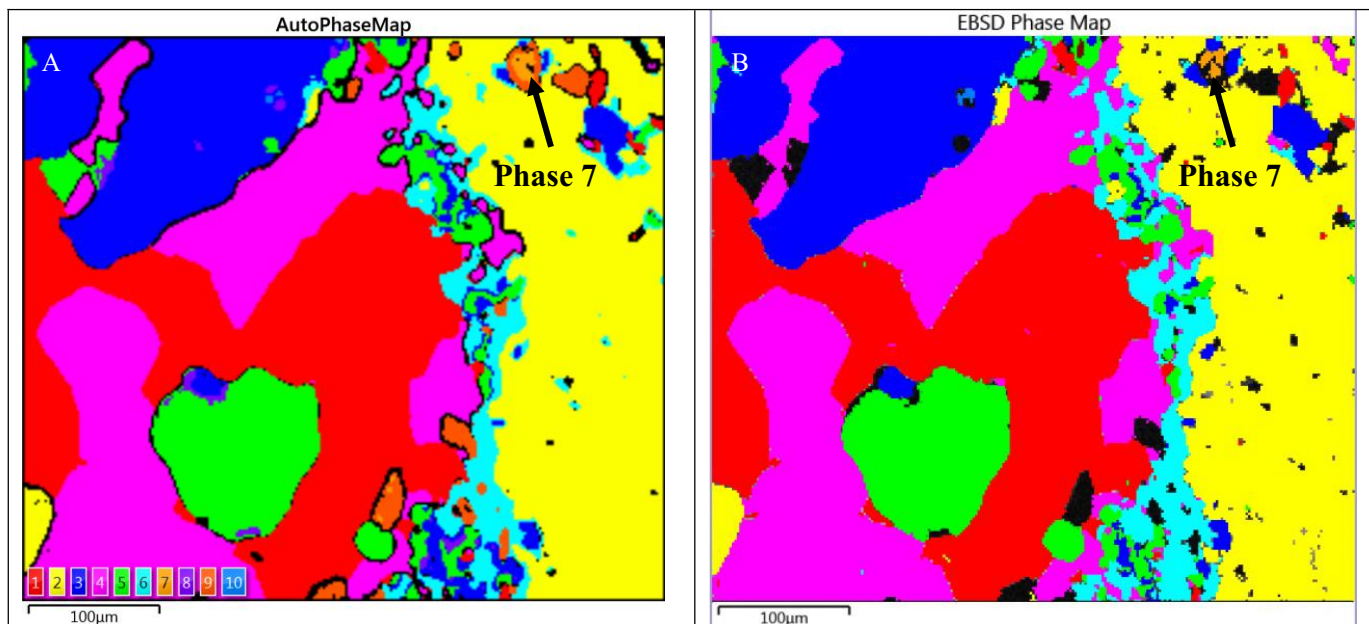
EBSD mapping is a more established method for phase analysis, analyzing the backscattered electron diffraction pattern produced at each pixel location to discriminate the correct phase among a set of possible crystal structures. In this way it identifies phases much more directly than the EDS-based characteristic chemistry AutoPhaseMap method. The phase map generated by EBSD is shown in Fig. 1B. EBSD phase colors were chosen to color phases similarly with their counterparts in the AutoPhaseMap for easier comparison. A comparison of area fractions calculated by the two methods is given in Table 1. On first sight the similarity of the results is encouraging and validates the AutoPhaseMap result obtained. On closer inspection differences reveal the different strengths of the two

methods. One strength of the AutoPhaseMap method is its relative insensitivity to surface preparation, unlike EBSD, for example phases/areas which do not give patterns still have characteristic chemistry. Thus black areas in the EBSD map yielded no patterns, and are shown to be mainly areas where altered orthopyroxene, olivine, and various states of alteration of iron sulfide have been found by EDS. EBSD is particularly useful in confirming more precisely phase identification. For example in the region identified by EDS as being most likely to be an iron sulfide (Phase 7 - light orange), the EBSD result not only confirms this but also that the mineral is pyrrhotite (Fe_{1-x}S) rather than pyrite (FeS_2). It can also be seen in other areas that the much higher spatial resolution of the EBSD technique at 20kV yields more precise results on finer scale structures and grains, for example in some of the areas where smaller grains of olivine (green) and clinopyroxene (blue) are found together. Collecting the data at higher resolution would probably improve the results of the EBSD and particularly the EDS in this respect.

References

[1] Stork and Keenan, *Microsc. Microanal.* 16, (2010), p810–820.

[2] Dr. P. Trimby of The University of Sydney provided the oceanic gabbro sample for this study



Phase No.	Phase	EDS Area %	EBSD Area %
1	Ilmenite	22.1	22.5
2	Plagioclase	23.6	24.3
3	Clinopyroxene	13.3	13.2
4	Magnetite	20.2	21.7
5	Olivine	8.5	7.9
6	Amphibole	4.9	5.3
7	Iron Sulfide	0.2	0.12
8	Orthopyroxene	0.12	0.03
9	Altered iron sulfide	1.3	0
10	Calcite	0.03	0.09

Fig. 1. A. EDS AutoPhaseMap result showing the distribution of phases in an oceanic gabbro sample. B. EBSD phase map calculated from data collected simultaneously with EDS.

Table 1. Phases identified by AutoPhaseMap and EBSD, with area % of each phase determined by the two methods. Phase numbers correspond to the key in Fig. 1A.