Combining SEM, EDS & EBSD: Challenges and considerations in the micro-analysis of rock thin sections

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Background Traditional analytical techniques answer the question of what minerals are present in the samples, but not *where* in the samples. That is, because for most of them the sample has to be powdered, or in aqueous solution. However, due to the advent of computing capacity and detector technology, it is feasible to map large areas and record information in situ. Thus, analysis information from an interaction volume is tagged to a location in the sample surface.

Currently, for geological samples, automated mineralogy is performed either on a particulated sample (e.g. feed material in epoxy), or a thin section of a rock. Geological samples are usually complex, polycrystalline materials. For automated mineralogy applications, the grains are detected by setting grey level thresholds (Fig. 1), and to speed up automated mineralogy acquisition, one (EDS) spectrum is recorded per grain. Fig. 2 shows whole thin section automated mineralogy mapping.

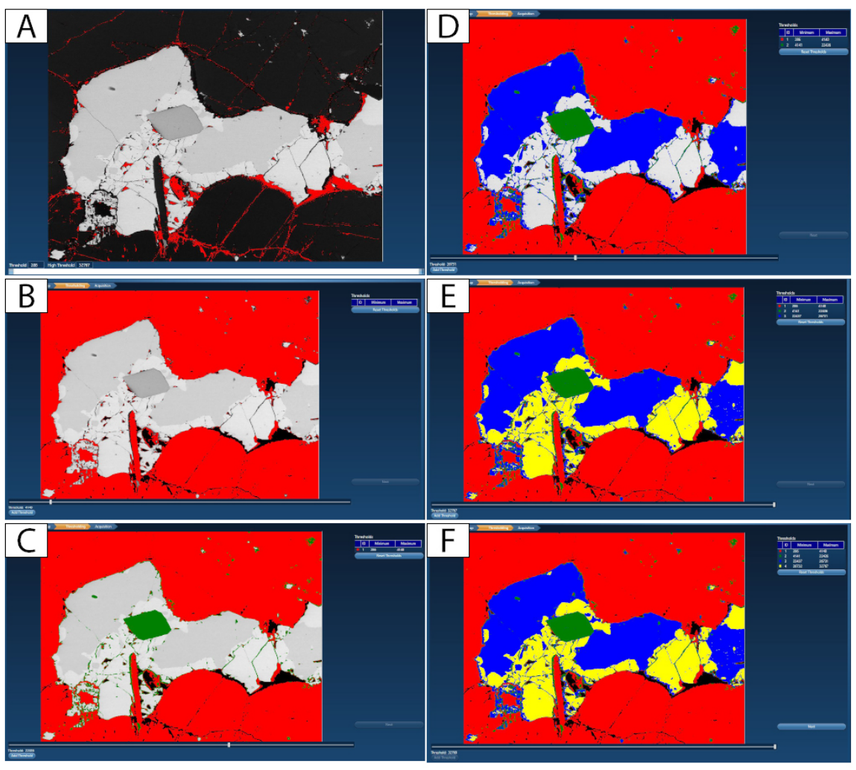

Fig. 1 Grey level thresholding process. (A) Initially the porosity (cracks, holes) are recognised. (B) The surrounding phase is orthopyroxene (red). (C-F) The sulphide mineral assemblage is comprised of pyrite (dark green), pyrrhotite (blue), pentlandite and chalcopyrite (yellow), which cannot be distinguished due to the very close atomic number values of nickel and copper.

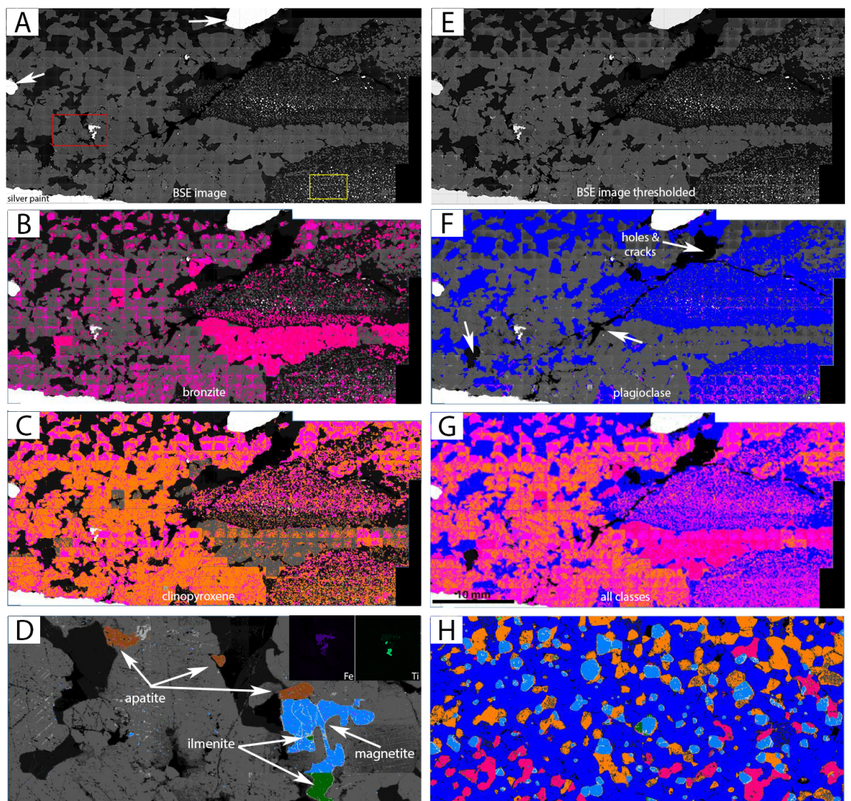

Fig. 2 Automated mineralofy whole thin section mapping results. Fig. 2A is a BSE image of the whole thin section before thresholding and Fig. 2E is the processed BSE image. Fig. 2B, C, and F show the major phases (orthopyroxene in vibrant pink, clinopyroxene in orange, and plagioclase in blue) distribution in the section. Fig. 2G shows all phases, even though a pink colour is assigned by default to areas with more than one phases present (not be confused with orthopyroxene in Fig. 2B). The white patches on top of the section is silver paint, used to enhance sample conductivity. Fig. 2D and H show close-ups of the red and yellow rectangles, as those are marked in Fig. 2A. In Fig. 2D Apatite is shown in brown colour, magnetite in light blue, and ilmenite in dark green. In Fig. 2H, orthopyroxene in vibrant pink, clinopyroxene is shown in orange, plagioclase in blue, magnetite in light blue.

EBSD is another in situ bulk analysis technique, which exploits the strong backscatter electron yield to measure grain orientations. Grain detection is intuitive to the human eye – for instance, in an optical image. BSE imaging and EDS mapping do not cope so efficiently with grain detection, and require image processing as shown in Fig. 1. On the contrary, EBSD is uniquely capable of detecting grains based on their orientations. Fig. 3A shows an example of an entire thin section EBSD mapping. The map is coloured in inverse pole figure (IPF) scheme, which colours grains with similar orientation in similar colour following the colour key provided (top right). Fig. 3A shows all major phases in the thin section, whereas Fig.3 B-D are filtered accordingly to show orthopyroxene, clinopyroxene, and plagioclase with the corresponding inset pole figures.

Comparing Fig. 2 and Fig. 3 maps, it is obvious that the EDS and the EBSD dataset match perfectly even though they were acquired independently, and show different information (the former chemical, the latter crystallographic). It is, therefore, concluded that the combination of EDS - EBSD datasets is a powerful bulk rock characterisation tool, because chemical qand structural information can be correlated in-situ, for example, to detect zones with higher breakage potential within a rock, which can facilitate rock breakage. Predicting rock breakage is challenging and to-date there is no universal model.

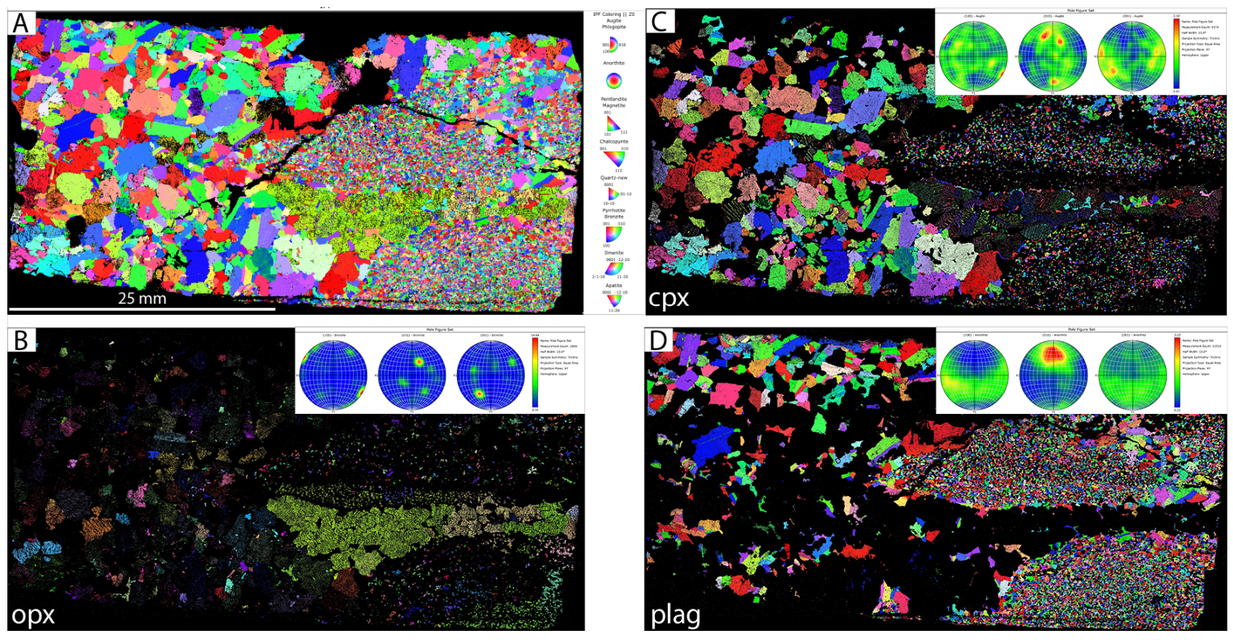

Fig. 3 IPF maps of the thin section from Fig. 2. (A) IPF colour map including all major mineral phases. (B) Filtered IPF colour map showing orthopyroxene occurrence throughout the thin section. Note the orthopyroxene lamellae. Inset: pole figure set displaying the dominant orthopyroxene orientations. (B) and (C) Similarly, the maps are filtered to show the clinopyroxene and plagioclase occurrence and in the inset, the preferred grains orientations, respectively.

Conclusions The combination of EDS and EBSD makes a powerful, non-destructive, bulk analysis technique. The advantages include:

1) No need to powder a sample, less time for sample preparation.

2) Throughput maximisation.

3) Improved sampling: large area mapping.

4) Chemical (EDS) and crystallographic (EBSD) data extracted with one measurement, in the same instrument, under the same conditions.

5) a. Enhancing mineral liberation analysis (MLA) by including EBSD. Reliable identification of chemically similar minerals with different structures.

b. Grain statistics (number of grains per phase, composition, grain size etc.) is still available, with the extra crystallographic information.

6) Advanced rock characterisation with multi-technique analysis in one instrument. Crystallographic data assisting in modelling rock breakage behaviour during comminution (=mechanical grinding etc. to reduce grain size).

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