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Chemical and Structural Analysis in 3D: Combining µ-XRF, EDS, and EBSD Data Sets.

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Displaying internal structures of complex materials in 3D has become easier in recent times as technology has made significant advances, for example in X-ray computed tomography (CT). However, the chemical analysis of samples in 3D is far more complicated due to absorption effects. In order to obtain high-quality quantitative results, the sample has to be sectioned and thereby destroyed. Currently available destructive techniques include atom probe tomography (APT) and focussed ion beam (FIB) serial sectioning combined with EDS/EBSD. These techniques are optimized for the analysis of small internal structures (<100 nm for APT and <100 µm for FIB). On the other hand, if chemical analysis of samples with internal structures in the µm to mm range needs to be performed, there is currently no straightforward 3D solution.

In this study, we want to present a 3D data set combining micro-X-ray fluorescence (µ-XRF), energy dispersive spectrometry (EDS) and electron backscatter diffraction (EBSD) with serial sectioning (manual grinding/polishing). The object studied was a meteorite sample (Gujba), containing both silicate and metal phases. Figure 1 shows a mixed element map (Fe, Ca, Si and S) of a complete 2D section of the sample obtained with an M4 Tornado µ-XRF spectrometer. Analytical conditions were 50 kV accelerating voltage, 200 µA beam current, 15 µm spotsize, and with an input count rate of about 150 kcps. Using an image resolution of 1067x680 pixel and a 5 ms dwell time/pixel, resulted in a 77 minute total acquisition time. The green rectangle in Figure 1 indicates the EDS acquisition site shown in Figure 2. For this image, 9 sections were analyzed with a Quantax EDS system (using an XFlash® EDS detector with an input count rate of 300 kcps, 20 kV accelerating voltage, ~30 nA beam current, 60 min acquisition time) and reconstructed in 3D with Amira® software. The information from µ-XRF and EDS provides an ideal basis for examining selected regions of interest on an even smaller lateral scale with additional techniques, such as EBSD. The yellow rectangle on the EDS map indicates the EBSD acquisition site shown in Figure 3. The crystal orientation map (here, overlay with pattern quality map), obtained with an e$^-$Flash^{1000} EBSD detector, resolves twinning phenomena within the enstatite. Please note that no data cleaning or post-processing was applied.

In conclusion, µ-XRF, EDS and/or EBSD combined with serial sectioning is a promising technique for the 3D chemical analysis of samples on the micrometer and/or millimeter scale. The time required for the acquisition of µ-XRF and EDS element maps was minimized by using modern SDD technology with high input count rates. Furthermore, the integrated EBSD and EDS software allows the simultaneous acquisition of EDS and EBSD datasets. The total time spent, including sample preparation and instrument time, was within the order of time that would typically be spent producing a FIB serial section combined with EDS or EBSD (15-20 hours).

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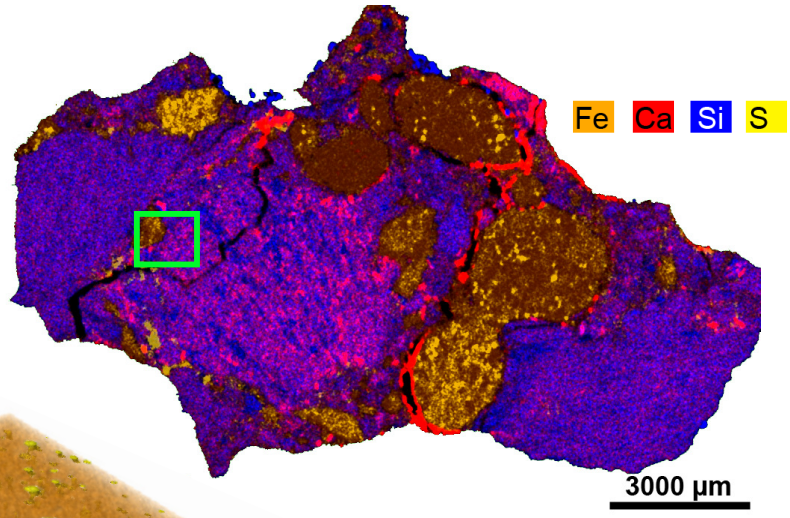


FIG. 1. Mapping of the Gujba meteorite sample with µ-XRF. The green rectangle indicates the EDS mapping site shown in Fig. 2.

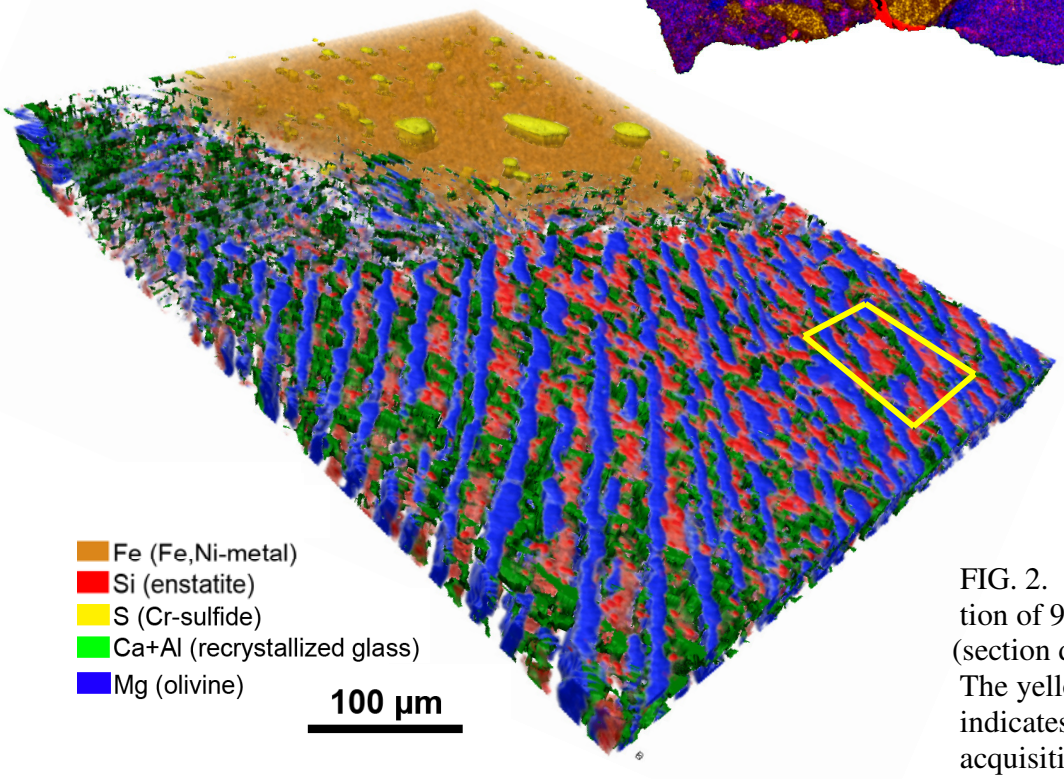
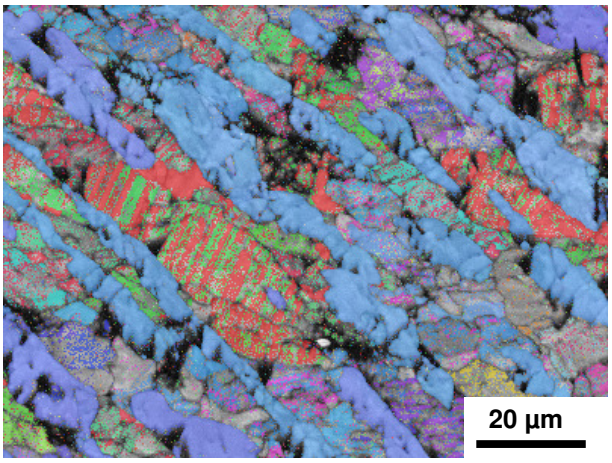


FIG. 2. 3D reconstruction of 9 EDS maps (section depth: 5±2µm). The yellow rectangle indicates the EBSD acquisition site shown in Fig. 3.



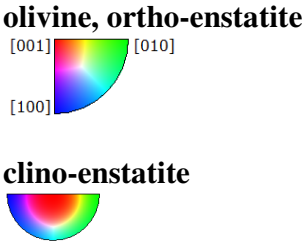


FIG. 3. Overlay of EBSD orientation (IPF-Y) and pattern quality map for the area indicated by the yellow rectangle in Fig. 2.

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