Micro-XRF Spectroscopy

The M4 Tornado micro-XRF is an energy dispersive X-ray spectrometer designed specifically for the analysis of small sample areas. It has the same capabilities of a scanning electron microscope/energy-dispersive spectrometer (SEM/EDS), such as point analysis, line scan, and area analysis, but is a stand-alone, desktop instrument. The M4 Tornado uses a silicon-drift detector to identify elements with an atomic number ≥ 11 (Na and heavier), making it well-suited for geological materials.

Because micro-XRF spectroscopy is rapid, non-destructive, and adaptable to many different types of materials, it could prove to be a useful tool for characterizing and classifying meteorites. The M4 Tornado can analyze and map flat or rough surfaces up to 190 x 160 mm, eliminating the need for samples preparation (such as thin sectioning). The silicon drift detector technology allows for data acquisition times of < 1 ms per pixel. So, even with the lowest possible step size, a 1° round thin section can be mapped in as little as two hours. Though the 25 µm diameter X-ray spot size is larger than much of the fine-grained material in chondrites, the overall resolution can be improved using a small step size. Here we test the limits of the technique using L4 ordinary chondrite, Bald Mountain.

Methodology

Multiple 10x magnification images were combined to create a mosaic image of the Bald Mountain thin section (Fig. 2), which was then mapped using step size of 5 µm and a dwell time of 1.12 ms per pixel. Total mapping time was ~ 7 hours.

Elemental Distribution Maps

Quantitative mapping is performed by sequential, pixel by pixel, analysis of an image. Results are assembled as element maps where distribution can be presented as absolute or relative intensities. Absolute intensities are normalized to the highest intensity of each individual element, while relative intensities are normalized to the highest intensity of the whole map. Relative intensities are useful for comparing the distribution of all phases within a sample. Figure 3 shows absolute X-ray intensities of all elements in Bald Mountain.

Unlike a traditional imaging techniques, elemental distributions measured using the micro-XRF can be displayed using a wide variety of colors, and multiple maps can be combined into a single image. Color combinations can be selected specifically to highlight phases of interest. In Figure 4, the combination of red, blue, and yellow identifies taenite (purple), kamacite (pink), and trolite (orange).

Because any number of distribution maps can be combined into a single image, multiple mineral phases can be identified using a single map. Figure 5 shows relative X-ray intensities of Na (red), P (purple), S (yellow), Ca (light green), Ti (blue), Cr (pink), and Ni (neon green) in Bald Mountain. In ordinary chondrites these elements can be used to identify whitlockite (Ca & P), trolite (S), chromite (Cr), ilmenite (Ti), metal (Ni), and plagioclase (Na).

Phase Mapping

The phases function uses chemical information stored in every pixel to produce a map of similar elemental intensities. This function has incredible potential for determining modal abundances; however, it is limited by overall mineralogy and grain size of the samples. Phase mapping works best on coarse-grained samples with a few compositionally distinct phases. Here we test the effectiveness of the phase mapping tool on fine-grained, homogeneous samples.

Phase mapping can be carried out manually by selecting objects that represent each phase or by using an automatic, built-in principle components analysis (PCA). In both methods, elements must be selected by the user, and best results are achieved when only the elements that are most useful for distinguishing phases are selected. Elements present at low abundances and elements evenly distributed throughout the sample are ignored.

Figure 6 is a phase map of Bald Mountain created using the automatic PCA function. The automatic phase mapping tool was not able to distinguish between the primary silicate phases (olivine, opx, & cpx), although it could distinguish between kamacite and taenite.

Modal abundances of phases in Bald Mountain have been previously determined using an electron microprobe phase mapping technique [1] and position sensitive X-ray Diffraction [2]. XRF-calculated abundances do not correlate well with these previous techniques. Silicates are much higher, while metal and trolite are lower (Table 1).

Phase mapping in the ordinary chondrites works better on small areas of a sample where data can be collected at higher magnification, using longer dwell times. Mapping of a single porphyritic chondrule at 100x magnification using a step size 5 µm and a dwell time of 120 ms yields a phase map (Fig. 8) that is quite representative of the phases identified using elemental distribution maps (Fig. 7).

Conclusions

Although magnification of the micro-XRF cannot match that of an SEM, it has many useful applications in sample characterization. Whole sample elemental distribution maps can be collected quickly and easily combined to identify phases. In addition, the phase mapping function could prove valuable in determining modal abundances of chondrite components, such as chondrules or CAIs. 


Acknowledgements: Thanks to the Smithsonian Institution for the Bald Mountain thin section.