# X-ray Mapping of Terrestrial and Extraterrestrial Materials Using the Electron Microprobe: A Progress Report

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Lunar samples returned from the Apollo program motivated development of the Bence-Albee algorithm for the rapid and accurate analysis of lunar materials, and established interlaboratory comparability through its common use. In the analysis of mineral and rock fragments it became necessary to combine micro- and macroscopic analysis by coupling electron-probe microanalysis (EPMA) with automated stage point counting. A coarse grid that included several thousand points was used, and initially wavelength-dispersive (WDS) and later energy-dispersive (EDS) data were acquired at discrete stage points using ~5 sec count times [1]. A ~50 μm beam diameter was used for WDS and up to 500 μm beam diameter for EDS analysis. Average analyses of discretely sampled phases were coupled with the point count data to calculate the bulk composition using matrix algebra. Use of a defocused beam resulted in a contribution from multiple phases to each analytical point, and the analytical data were deconvolved relative to end-member phase chemistry on the fly. Impressive agreement was obtained between WDS and EDS measurements as well as comparison with bulk chemistry obtained by other methods. In the 30 years since these methods were developed, significant improvements in EPMA automation and computer processing have taken place. Digital beam control allows routine collection of x-ray maps by EDS, and stage mapping for WDS is conducted continuously at slew speed and incrementally by sampling at discrete points. Digital pulse processing in EDS systems has significantly increased the throughput for EDS mapping, and the ongoing development of Si-drift detector systems promises mapping capabilities rivaling WDS systems. Spectrum imaging allows a data cube of EDS spectra to be acquired and sophisticated processing of the original data is possible using matrix algebra techniques.

The study of lunar and meteoritic materials includes the need to conveniently: (1) Characterize the sample at microscopic and macroscopic scales with relatively high sensitivity, (2) Determine the modal abundance of minerals, and (3) Identify and relocate discrete features of interest in terms of size and chemistry. The coupled substitution of cations in minerals can result in significant variation in mineral chemistry, but at similar average Z, leading to poor backscattered-electron (BSE) contrast discrimination of mineralogy. It is necessary to discriminate phase chemistry at both the trace element level and the major element level. To date, the WDS of microprobe systems is preferred for mapping due to high throughput and the ability to obtain the necessary intensity to discriminate phases at both trace and major element concentrations. It is desirable to produce fully quantitative compositional maps of geological materials, which requires the acquisition of k-ratio maps that are background and dead-time corrected, and which have been corrected by $\phi(\rho z)$ or an equivalent algorithm at each pixel. To date, turnkey systems do not allow the acquisition of k-ratio maps and the rigorous correction in this manner.

X-ray maps of a chondrule from the Ourique meteorite, and a comb-layered xenolith from the San Francisco volcanic field, have been analyzed and processed to extract phase information. The Ourique meteorite presents a challenge due to relatively low BSE contrast, and has been studied using spectrum imaging [2]. X-ray maps for Si, Mg, and Fe Kα were used to produce RGB images. The xenolith sample contains sector-zoned augite, olivine, plagioclase, and basaltic glass. X-ray maps were processed using Lispix and ImageJ software to produce mineral phase maps [3,4]. The x-ray maps for Mg, Ca, and Ti were used with traceback to generate binary images that were converted to RGB images. These approaches are successful in discriminating phases, but it is desirable to achieve the methods that were used on lunar samples 30 years ago on current microprobe systems. Current research includes x-ray mapping analysis of the Dalgety Downs chondrite by micro x-ray fluorescence and spectrum imaging, in collaboration with Kenny Witherspoon of IXRF Systems and Dale Newbury of NIST.

References

[1] Albee, A.L. et al., 8th Int. Congr. X-ray Opt., (1977), 526.

[2] Kotula, P.G. et al., Microsc Microanal 10 (Supl 2, 2004), 892.

[3] Lispix Public Domain Program, by David Bright, http://www.nist.gov/lispix/

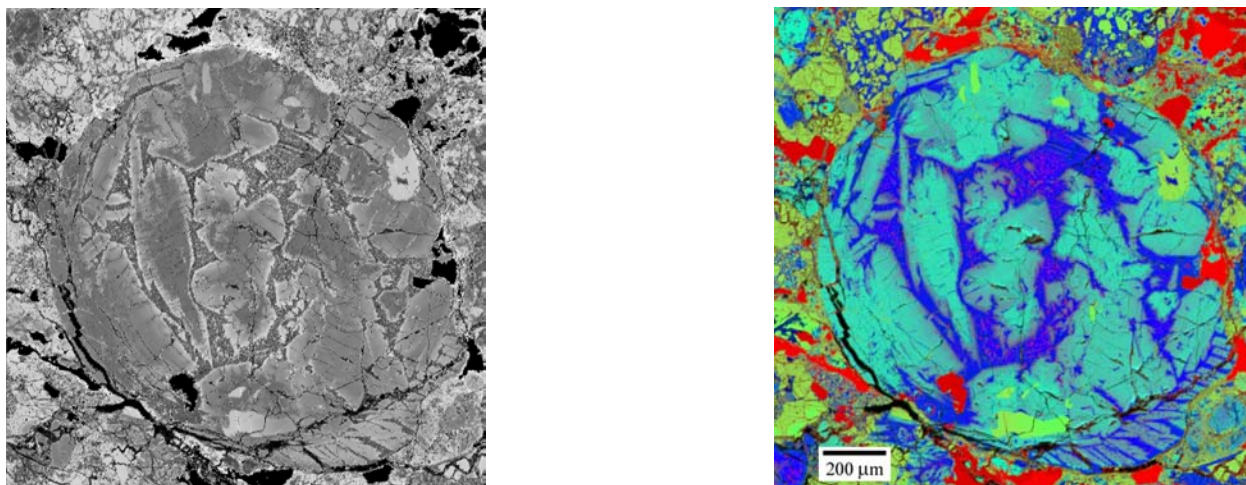[4] ImageJ Public Domain Program, by Wayne Rasband, http://rsb.info.nih.gov/ij/

Fig. 1A. (Left) BSE image of Ourique chondrule with contrast enhancement. Rim zoning and matrix phases have high BSE and phase identification is difficult.

Fig. 1B. (Right) RGB image of Ourique chondrule using merged x-ray maps for Fe K$\alpha$ (red), Mg K$\alpha$ (green), and Si K$\alpha$ (blue). FeNi metal and FeS are both red, hypersthene is yellow green, skeletal olivine is aqua, and interstitial glass is blue.
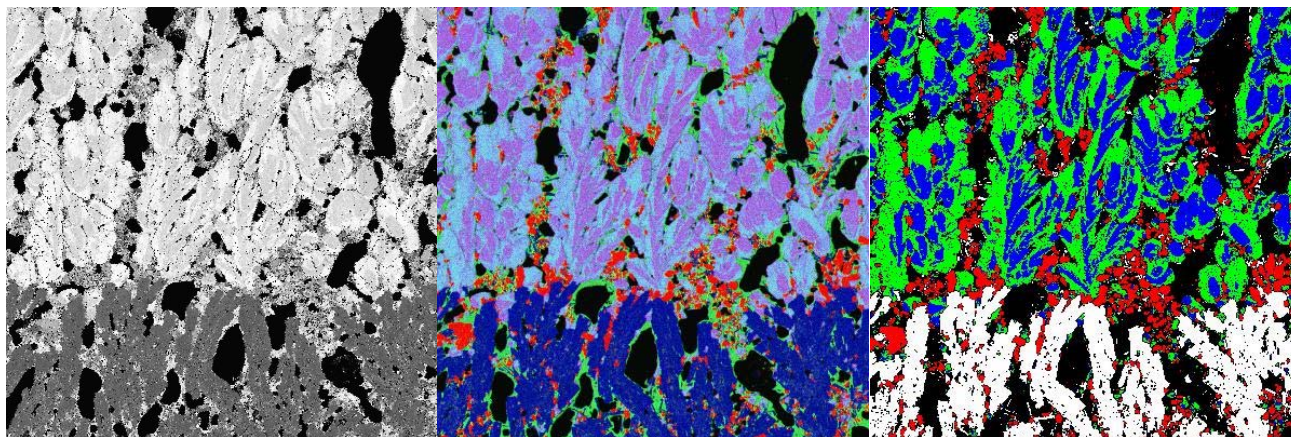


Fig. 2A. (Left) BSE image of comb layering. Lower layer is dendritic plagioclase and upper layer is dendritic sector-zoned augite. Augite core has elevated Mg and rim has elevated Ti and Cr. Discrimination of olivine is difficult, and identification of compositional changes requires x-ray mapping. Field width is approximately 1 mm.

Fig. 2B. (Center) RGB image using x-ray maps for Mg K$\alpha$ (red), Ti K$\alpha$ (green), and Ca K$\alpha$ (blue). Olivine is red, augite rim is light blue, augite core is purple, plagioclase is blue, and basalt glass is green. Success of RGB image here is due to good intensity saturation of WDS x-ray maps and cannot be made by conventional EDS.

Fig. 2C. (Right) Phase map using Lispix traceback and ImageJ. Augite core is blue, rim is green, olivine is red, and plagioclase is presented in grayscale.