Microsc. Microanal. 8 (Suppl. 2), 2002
DOI 10.1017.S1431927602107525

Automated X-ray Spectral Image Analysis of a Large Area of a Geological Material

P.G. Kotula, P.F. Hlava, and M.R. Keenan

Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185-0886

Comprehensive energy-dispersive x-ray microanalysis of a large area of a mineralogical specimen by conventional means is a significant challenge due to the extremely large number of point analyses required. X-ray mapping can readily survey large areas if the elements to map, as well as possible peak interferences, are known beforehand. Spectral imaging, where a complete x-ray spectrum is acquired from each point in a two-dimensional array, can solve the data acquisition problem (with proper sampling) but the data analysis is still a challenge. In this work, the automated analysis [1] of a 512x512 pixel by 1024 energy-channel x-ray spectral image, covering $1mm^2$ of area is described.

The analysis was performed on vein material from the Hilltop Mine in Organ, NM. This material is a primary ore for Tellurium in the form of PbTe (Altaite). Approximately 100 years ago, E. E. Fairbanks described the mineral Dunhamite (a Pb-Te-oxide) growing on the Altaite, which was later discredited due to an incomplete description [2]. An oxide of Altaite with the composition $PbTe_2O_4$ has been observed (by EPMA) in this work and further analysis on it is ongoing. In addition to Altaite and one of it's oxides, native Te, sulfides, silicates, oxides, etc. are expected in the vein material. The vein material was polished and carbon coated prior to x-ray microanalysis in a JEOL 5900LV with W-filament operated at 20kV. Spectral images were acquired at 512 by 512 pixel resolution with a 1mm field of view (~2μm/pixel), 9msec total dwell time per pixel, 40% dead time, resulting in an average of 100 counts total per pixel (i.e., spectrum). The software used in large part for the analysis has been described previously [1] but was modified significantly to perform such a large calculation on a standard personal computer (PC). A 512 by 512 pixel by 1024 energy-channel spectral image is over 2Gbytes in size and as such cannot fit in the RAM of a standard PC which can be up to 2Gbytes at present. As such it is impractical from a time standpoint to perform multivariate statistical analysis on this size data set at present. The modified method is based on spectral unmixing of pixel neighborhoods and retains full spectral and spatial resolution in the final pure component spectral and component map estimates. Therefore, the original data set was analyzed as 64 by 64 arrays of 8 by 8 pixel neighborhoods. This smaller data set was then automatically analyzed as before [1] resulting in 15 pure components-so called because they have been deconvolved on a sub-pixel scale. Figure 1 is an RGB composite of three of the components. The cyan region is Altaite (PbTe), the green region is Pb-S, the blue region is Pb-Te-O (possibly Dunhamite) and the red region is matrix material containing Mg, Al, Si, O, etc. Other components from this analysis are described elsewhere [3]. Figure 2 is the Te pure component image with enlarged region (indicated by the white square) and pure component spectrum. Te is separated out as a 'pure' component by the analysis software because it exists in three different phases: native Te (as clearly seen in FIG. 2B); Altaite; and a Pb-Te-O phase.

References

[1] P.G. Kotula et al., *Microsc. Microanal.* In press (2002).
[2] S. Northrop, *Minerals of New Mexico*, University of NM Press, Albuquerque, Rev. 1959.
[3] P.G. Kotula and M.R. Keenan, *These proceedings*, (2002)
[4] Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin

Company, for the United Stated Department of Energy (DOE) under contract DE-AC04-94AL85000.

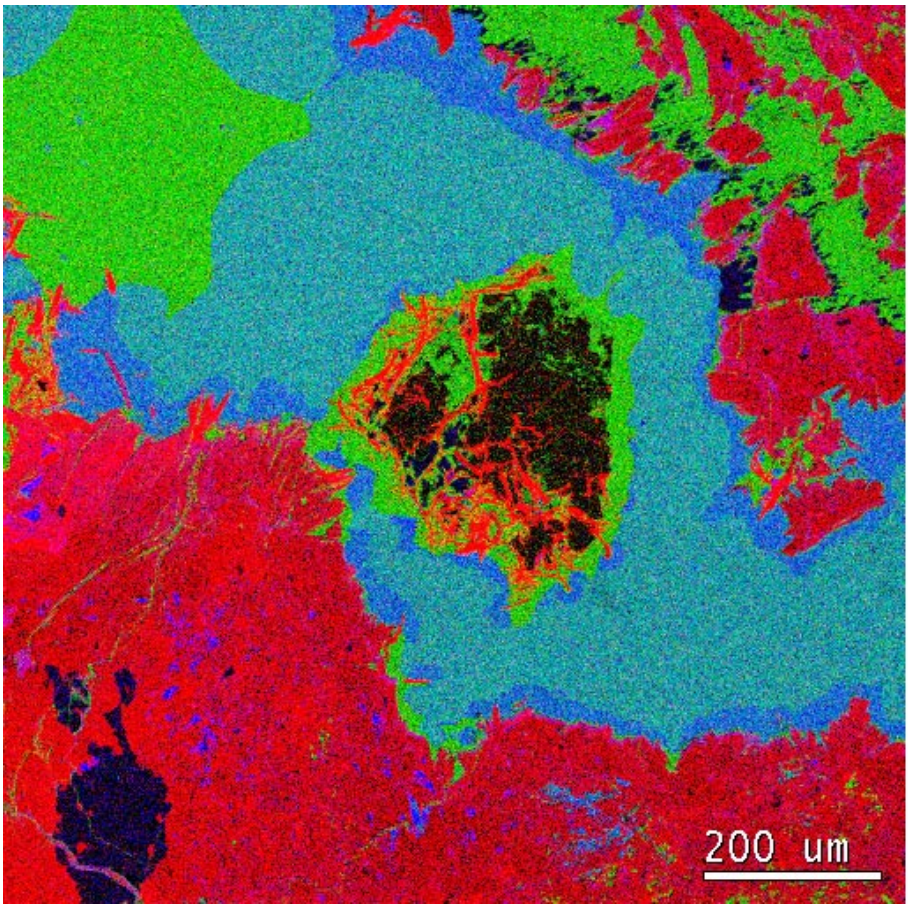

FIG. 1. RGB composite of several of the components from the automated analysis (see text for details).

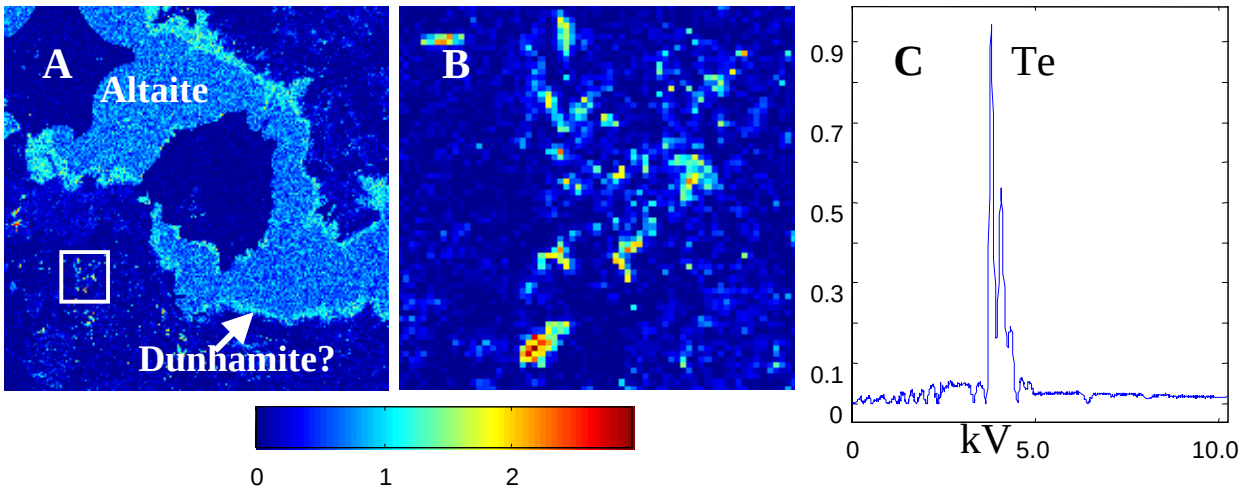

FIG. 2. Tellurium pure component image (A) with field of view of 1mm, enlargement of region in white box (64 by 64 pixels) (B) to field of view of 125μm, and pure component spectrum (C).

https://doi.org/10.1017/S1431927602107525 Published online by Cambridge University Press