doi:10.1017/S1431927610062501
Microsc. Microanal. 16 (Suppl 2), 2010

Combined STEM/STXM Elemental Quantification for Cometary Particles

Z. Gainsforth,* A. L. Butterworth,* R. C. Ogliore,* T. Tyliszczak,** and A. J. Westphal *

* Space Sciences Laboratory, University of California Berkeley, 7 Gauss Way, Berkeley, CA, 94720
** Advanced Light Source, Lawrence Berkeley Laboratory, MS 6-2100, Berkeley, CA, 94720, USA.

We present a case study of elemental quantification for a chromite grain retrieved from Comet 81P/Wild2 via NASA's Stardust mission[1]. The sample was especially challenging for a number of reasons: 1) The sample is a 1 x 0.5 x 1.5 μm unique, irreplacable grain with irregular morphology. 2) The sample is surrounded by a glass rim of variable thickness. 3) Trace element V should be known to about 1 part per thousand to answer pertinent scientific questions, but 4) The Ti Kβ peak interferes with V Kα in EDX.

The sample was the terminal particle in Stardust track C2052,2,74, and was prepared by embedding in epoxy, ultramicrotoming sections ~100 nm thick, and positioning on a Cu TEM grid with an amorphous carbon subtrate prepared by Ladd Research. It was then studied with a STEM/EDX, (Philips CM200/Oxford at the National Center for Electron Microscopy) and STXM (Beamline 11.0.2 at the Advanced Light Source[2]) in order to obtain exact elemental quantification and constrain geothermometric and geobarometric formation conditions. We focus on the elemental quantification issues here. The final composition of the chromite grain is shown in table 1 which combines STXM and TEM results.

Because minerals tend to shatter on microtoming, the sample is a fracture product and varies in thickness from ~0.5 micron to a sharp edge. This is ideal for combined analysis on TEM and STXM, where TEM favors samples < 100 nm thick but the STXM used in this work favors samples 100 nm - 1 micron thick depending on the element being investigated.

Since STEM/EDX work would preclude any hope of obtaining part per thousand concentrations of V, the STXM was used for quantification using the V $L_{2,3}$ absorption edges. The value V/Ti was determined from STXM and then combined with the Ti value obtained from EDX to obtatain a concentration of V of 0.23 +/- 0.05 At% (20% relative) providing microprobe accuracy. As such, the dominating uncertainty in the V concentration is the EDX uncertainty for Ti. In addition, STXM was able to map other major elements and provide a side check to ensure all matrix effects had been correctly accounted for in the EDX quantification including sample geometry, and the glass rim.

This case study demonstrates a synergy between STXM and STEM/EDX. Sample preparation is identical for both methods and a single sample can be probed in both instruments. Elemental interferences in EDX are often different than interferences in STXM, allowing quantification of seemingly intractable samples. For many elements, STXM also has a very high signal to background because it lacks brehmstrallung as in EDX[3]. Additionally, while matrix effects are significant on the TEM they are limited in STXM samples when the x-ray OD < 1. Even more important however, matrix effects on the STXM are predictable as they are only a function of elemental composition and thickness - general geometry does not play a significant role. Ab-initio detection limits and sensitivities are therefore easy to compute and provide accurate values. Both instruments are capable of preparing elemental maps that can be directly compared.

References

[1] D. Brownlee et al., *Science* (2006) 314, 1711.
[2] T. Warwick, et al., *Synch. Rad. News* (2003) 16, 22.
[3] D.B. Williams, C.B. Carter., *Transmission Electron Microscopy*, Springer, New York, 1996.
[4] The operations of the Advanced Light Source and National Center for Electron Microscopy at Lawrence Berkeley National Laboratory are supported by the Director, Office of Science, Office of Basic Energy Sciences, U.S. Department of Energy under contract number DE-AC02- 05CH11231.

FIG. 1. LAADF image of the chromite grain and some surrounding glasses and minerals. On the left is an overlay of a STXM Cr map.

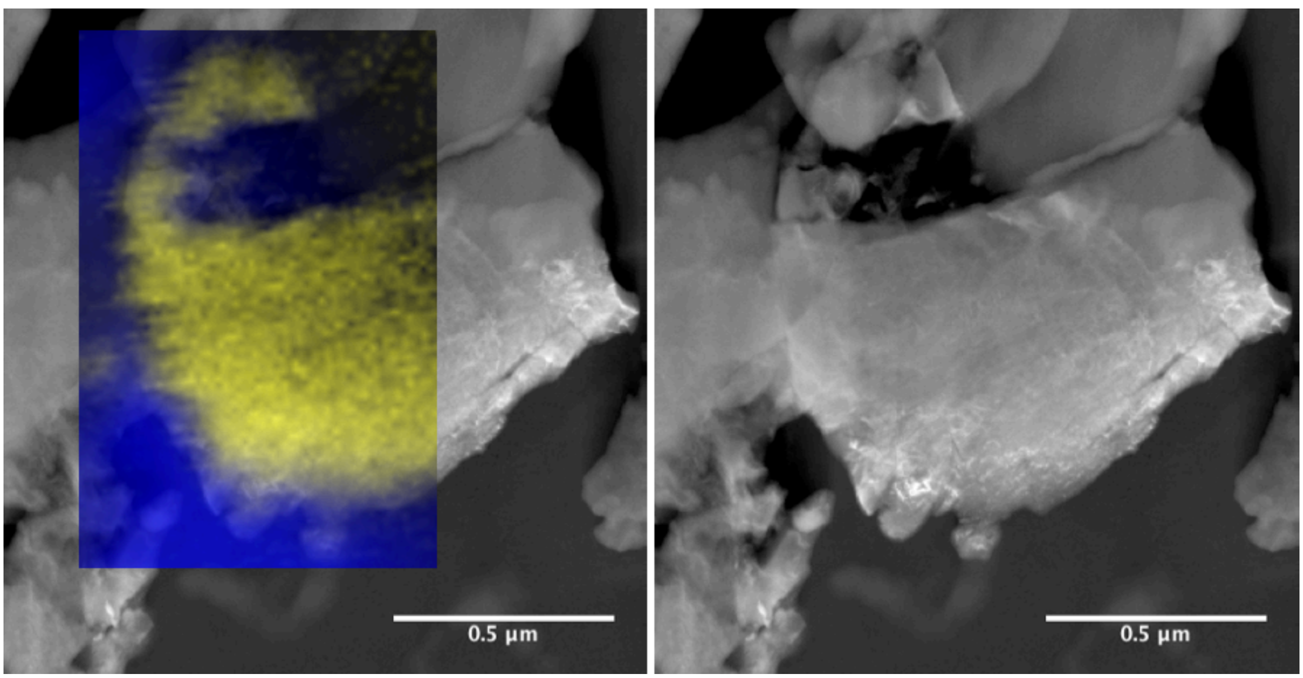

TABLE 1. Elemental quantification of the chromite grain in the terminal particle of C2052,2,74

Element	Atomic %	% error 1σ	Element	Atomic %	% error 1σ
O	56.79	1.3	V	0.23	0.05
Mg	4.27	0.3	Cr	17.49	1.1
Al	9.56	0.2	Fe	11.36	0.6
Ti	0.54	0.1			

https://doi.org/10.1017/S1431927610062501 Published online by Cambridge University Press