

USING AUGER SPECTROSCOPY TO CHARACTERIZE SUB-MICROMETER PRESOLAR GRAINS IN SITU: AN OVERVIEW. F. J. Stadermann¹, C. Floss¹, and A. S. Lea², ¹Laboratory for Space Sciences and Department of Physics, Washington University, St. Louis, MO 63130, USA. ²EMSL, Pacific Northwest National Laboratory, Richland, WA 99352, USA.

Introduction: Presolar grains have isotopic compositions that clearly identify them as stellar condensates which survived prolonged residency in the interstellar medium before being incorporated into Solar System materials. Many such grains are first identified during *in situ* isotope imaging searches in various types of primitive extraterrestrial materials. To constrain a given particle's possible origins, it is beneficial to get compositional information beyond its isotopic makeup. Since most presolar grains are sub-micrometer in size, only a few analytical techniques are suitable for this purpose. We have previously shown that scanning Auger spectroscopy represents an excellent tool for the elemental characterization of presolar materials that have been identified by NanoSIMS isotopic imaging searches [1]. We have now used this combination of techniques for *in situ* isotopic and elemental characterizations of sub-micrometer presolar grains in primitive meteorites [2, 3, 4], Antarctic micrometeorites [5], and interplanetary dust particles (IDPs) [2, 6]. Here, we give an overview of these studies and discuss the qualitative and quantitative identification of presolar grain types.

Analytical Details: A scanning Auger spectrometer is an electron beam instrument similar to an electron microprobe, but it uses the characteristic energies of Auger electrons and not those of x-rays for the determination of a sample's composition. The elemental information obtained during scanning Auger imaging measurements has a spatial resolution of tens of nanometers which is comparable to what can be achieved in NanoSIMS isotope measurements (50 - 100 nm). Such a spatial resolution is significantly higher than what can be reached with energy dispersive x-ray- analysis and is more than sufficient for the characterization of typical (200 - 500 nm) presolar silicate and oxide grains. Another advantage of using Auger spectroscopy in connection with NanoSIMS measurements is that the analyses can be performed on the same, unmodified sample mount and that virtually any sample that is suitable for the NanoSIMS can also be analyzed in the Auger instrument.

To test spatial resolution, we prepared a mix of (terrestrial) sub-micrometer quartz and diamond particle standards and then used the Auger

spectrometer to image the elemental distributions of C, O and Si (Fig. 1). In this simplified case, the identification of the two grain types from the Auger data is straightforward and the spatial resolution of the elemental images is almost as high as that of the secondary electron image of the same sample. Although the creation of Auger electrons by backscattered electron ionization can lead to a decrease of the spatial resolution (especially at high primary beam energies), this does not interfere with the grain type identification at the given particle size range.

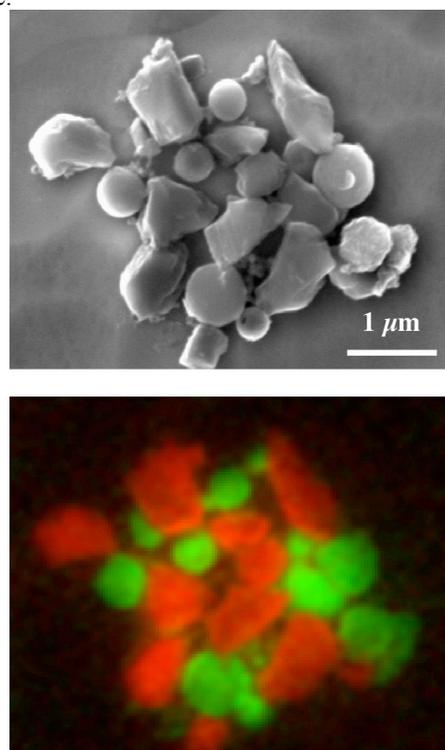


Figure 1. Test of sub-micrometer Auger elemental imaging of a dust standard consisting of a mix of quartz and diamond particles. The Auger secondary electron image (top) shows only the particles' morphology, but gives no information about their type. The colored image (bottom) shows the results of the Auger elemental imaging with C (i.e., diamond) as red and Si (i.e., quartz) as green.

The identification of actual presolar grains *in situ* is more complex, especially after the areas of interest have been sputtered during the preceding NanoSIMS measurements. The largest fundamental problem is the fact that SIMS measurements are inherently

destructive. After a grain has been consumed during the SIMS analysis, an identification by Auger spectroscopy is no longer possible. However, since NanoSIMS isotope imaging measurements are done in many successive scans across the sample, it is generally possible to monitor the signal from an isotopically anomalous grain in detail over time (*i.e.*, depth) and determine whether parts of the grain of interest are still present at the end of a NanoSIMS measurement.

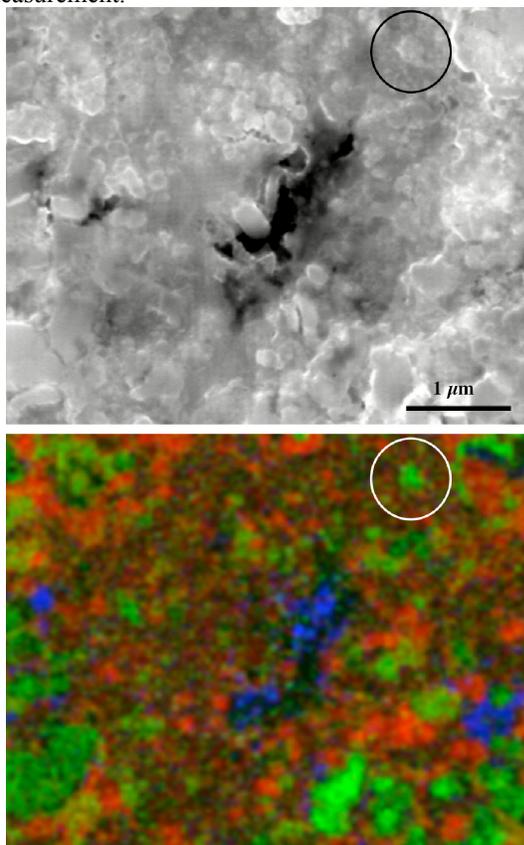


Figure 2. Scanning Auger microprobe images of a presolar silicate grain from ALHA77307. Top: secondary electron (SE) image; Bottom: composite RGB elemental Auger image (red = Fe, green = Mg, blue = Al). A presolar grain was found with the NanoSIMS at the center of the circle [3]. A distinct grain with a diameter of 250 nm can be seen at the same location in these images. The Auger analysis identifies this presolar grain as a Mg-rich silicate.

The sputtering process in SIMS also leads to some localized mixing of the sample material on a 10 nm scale. This results in a more blurred appearance of originally sharp-contoured features, but this is insignificant during the identification of presolar grains that typically have diameters larger than 100 nm. We have carried out Auger measurements of both sputtered and unsputtered areas of Renazzo matrix material. These measurements show that there

is little difference in the level of detail that can be resolved with Auger imaging, indicating that the SIMS sputtering is not significantly affecting our ability to identify specific grains in this type of material.

Qualitative identification of presolar grain types based on Auger elemental images is in many cases possible without extensive calibration of Auger signal intensities (Fig. 2). For quantitative measurements, however, it is necessary to determine mineral type specific elemental sensitivity factors. To test the feasibility of this, we have performed quantitative Auger measurements in a variety of mineralogically relevant silicate standards after they were sputtered in the NanoSIMS. Reasonable accuracy, *e.g.*, to distinguish between different types of silicates, can be achieved after applying the correct sensitivity factors (Fig. 3). We are continuing such systematic tests to determine the limits of Auger quantification of silicates grains *in situ* and determine the optimal measurement conditions. We will then apply this analytical approach to the study of presolar silicates and oxides to further constrain their stellar origins [2].

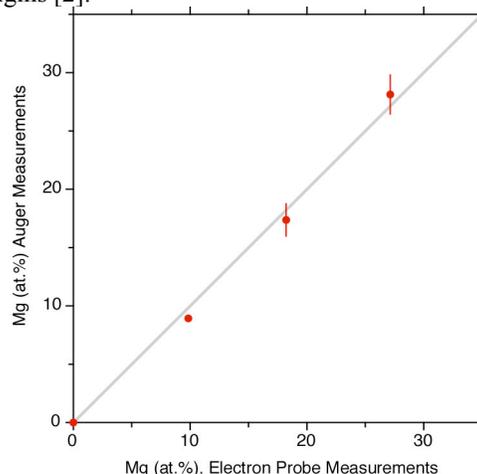


Figure 3. Comparison of Auger and electron probe quantitative measurements in four silicate standards with different Mg contents (plagioclase, clinopyroxene, orthopyroxene, olivine). The error bars of the Auger results show the standard deviation of repeated measurements.

References: [1] Stadermann F. J. et al. (2005) *Meteorit. Planet. Sci.* 40, A146. [2] Floss C. et al. (2005) *Meteorit. Planet. Sci.* 40, A49. [3] Nguyen A. et al. (2005) *Meteorit. Planet. Sci.* 40, A113. [4] Marhas K. K. et al. (2006) *this conf.* [5] Yada T. et al. (2006) *this conf.* [6] Floss C. et al. (2005) *GCA*, submitted.

This work was supported by NASA's cosmochemistry program. A portion of the research described herein was carried out at EMSL, a national scientific user facility sponsored by the DOE Office of Biological and Environmental Research and located at Pacific Northwest National Laboratory. We would also like to thank Physical Electronics in Chanhassen, Minnesota, for access to their Auger demonstration instrument.