CORRELATED HIGH SPATIAL RESOLUTION ELEMENTAL AND ISOTOPIC CHARACTERIZATION OF WILD 2 COMETARY SAMPLES. F. J. Stadermann, C. Floss, and M. Bose, Physics Department, Washington University, St. Louis MO, 63130, USA. fjs@wuphys.wustl.edu

Introduction: NASA's Stardust mission was successfully completed with the return of the first *bona fide* cometary samples for laboratory studies, and the preliminary examination yielded some unexpected results about the composition and origin of cometary matter [1]. From an isotopic point of view, the most important findings were (a) the fact that the majority of the samples are isotopically normal [2], (b) the observation of CAI-like (Ca-Al-rich inclusions) material with a ^{16}O-enriched composition similar to what has been seen in meteorites [2, 3], (c) the detection of D and N anomalies indicative of an interstellar heritage of some organics [2, 4], and (d) the discovery of one – but only one – incontrovertible circumstellar grain [2, 5].

While isotopic data alone can provide important clues about a given sample, essential insight can frequently only be gained by combining isotopic information with spatially correlated elemental and mineralogical data. Such a complementary analytical approach is especially mandatory in cases of rare or valuable samples such as the cometary matter from Wild 2. For samples that are heterogeneous on a sub-micrometer scale, the NanoSIMS is the optimal tool for a detailed isotopic characterization. Transmission electron microscopy (TEM) is the technique of choice for detailed mineralogical information and as such provides an ideal complement to the NanoSIMS [6]. Many types of samples, however, are not electron-transparent and thus not immediately accessible for TEM measurements. In such cases, however, it is still possible to use Auger spectroscopy for elemental imaging on a spatial scale of tens of nanometers, well below the working range of EDX characterization [7, 8]. Here we describe the application of complementary high resolution isotopic and elemental imaging for the analysis of cometary samples.

Experimental: Stardust Al foil strip C2118N contains a large impact crater with a diameter of 72 µm and was the object of NanoSIMS studies during the Stardust Preliminary Examination [5]. While most of the analyzed area in and around this crater is isotopically normal in C, N, and O, one moderately ^{14}N-enriched grain was located at the crater bottom (Fig. 1). This C- and N-rich grain has a ^{14}N/^{15}N ratio of 564 ± 97 vs. a terrestrial value of 272. This particle, with an original diameter of ~150 nm, was sputtered away during repeat measurements and it was not possible to gather additional compositional data [5]. Unfortunately, the Washington University Auger Nanoprobe only became available after the conclusion of the Stardust Preliminary Examination. We are convinced that non-destructive high resolution Auger elemental imaging measurements before and between NanoSIMS runs would have provided valuable additional information in this case.

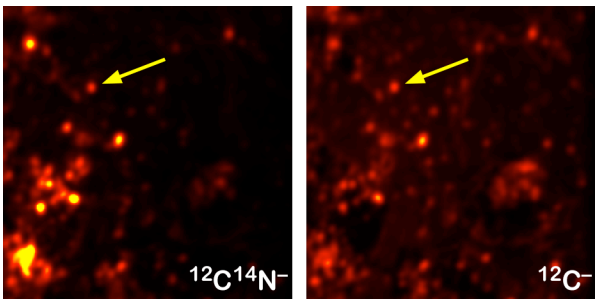

Figure 1: NanoSIMS secondary ion images of a 10 x 10 µm^2 area on the bottom of crater C2118N (c.f. Fig. 2). An isotopically anomalous C- and N-rich grain was found at the location shown.

To evaluate the applicability of the new analytical capabilities for the elemental characterization of Stardust samples, we have performed additional measurements in other areas on this crater that were previously studied with the NanoSIMS. A 5 x 5 µm^2 sub-region of the NanoSIMS imaging area was subsequently analyzed by high resolution elemental imaging with the new Washington University PHI 700 Scanning Auger Nanoprobe (Fig. 2). The resulting elemental distribution images clearly show that the cometary dust impact residue is heterogeneous on a submicrometer scale. Although there occasionally are larger 'islands' of silicate material, the debris does not cover a contiguous area on the crater rim or bottom, but consists instead of dispersed fragments, between which the Al foil is exposed ('Al' in Fig. 2). The elemental images identify, *e.g.*, the highlighted grains (Fig. 2) as Fe sulfide (grains 1 - 4), possibly troilite which is abundant in aerogel-captured Wild 2 material [3], carbonaceous matter (grain 5), as well as Mg-rich (grain 6) and Mg-poor (grain 7) silicates. Note that grains 1 - 5 are below 200 nm in diameter and, thus, too small for EDX characterization. Such particles, however, represent the typical size range for presolar grains that are routinely studied in NanoSIMS isotopic studies [e.g., 9]. A spatial correlation of the isotopic

and elemental maps makes it possible to determine individual grains' isotopic and elemental composition. In this particular area, no statistically significant isotopic variations were found in C, N, or O. However, the overall approach of combining high resolution elemental and isotopic imaging has proven suitable for the analysis of cometary impact residue in and around Stardust Al foil craters. Future NanoSIMS work on these foils will routinely be complemented by Auger analyses. In addition to qualitative elemental distribution images, it is also possible to perform quantitative analyses in point mode for select grains. The required database of Auger elemental sensitivity factors is currently being developed.

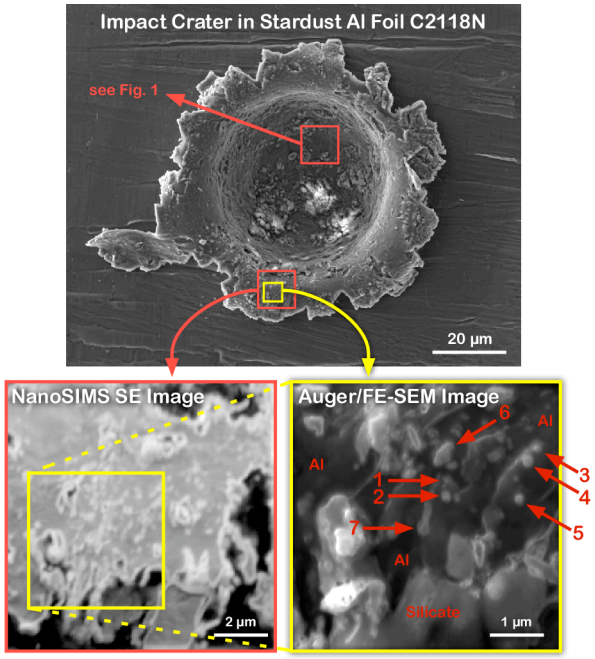

Figure 2: Details from the rim of crater C2118N. Boxes on the lower rim indicate areas studied for isotopic and elemental compositions. See text for details.

Discussion: Since non-destructive Auger elemental measurements can be performed without sample modification on any specimen that is analyzable with the NanoSIMS, this analytical procedure also has important implications for the isotopic study of aerogel-extracted cometary particles. During the Preliminary Examination we performed C, N, and O isotopic imaging measurements on a variety of sliced or pressed track particles. By obtaining detailed elemental maps of such samples before the NanoSIMS measurements, we will not only be able to better identify the possible carrier phases of isotopic anomalies, but it will also be possible to better optimize the inherently destructive SIMS measurements for a given sample. For example, it may be advantageous to focus on O and Mg/Al isotopic measurement first (instead of C and N) when the sample appears to be a refractory type mineral [3]. The minute sample amounts available will in many cases limit the number of possible consecutive isotopic measurements and prioritizing is essential for extracting a maximum of information. We note that obtaining detailed elemental maps of the sample during the NanoSIMS analysis stage is important even when subsequent FIB-TEM analyses are planned. Locating the exact position of isotopically anomalous hotspots for FIB extraction frequently requires difficult triangulation and any additional compositional data can be invaluable.

During the Stardust Preliminary Examination it was not possible to identify the carrier of the ^{14}N-enrichement mentioned here or the exact nature of the sole ^{17}O-rich circumstellar grain [2, 5]. However, we will continue the search for additional presolar and circumstellar components in Wild 2 cometary samples and, with the new analytical capabilities, we expect to attain more detailed information on the nature of the carrier phases. This will allow us to compare not only the abundances, but also the different types of isotopically anomalous matter among the cometary samples with what is typically seen in primitive meteorites and interplanetary dust particles. The analysis of the first comet samples has yielded many surprises already [1], but a more detailed look at the elemental, isotopic and mineralogical makeup of comets on a sub-micrometer scale will become available in coming years with the help of studies like the one described here. This will allow us to get a better understanding of the different sources contributing to Kuiper Belt comets, both from the inner solar system and the interstellar medium.

References: [1] Brownlee D. et al. (2006) *Science* 314, 1711. [2] McKeegan K. D. et al. (2006) *Science* 314, 1724. [3] Zolensky M. E. et al. (2006) *Science* 314, 1735. [4] Sandford S. A. et al. (2006) *Science* 314, 1720. [5] Stadermann F. J. et al. (2007) *Meteoritics & Planet. Sci.*, submitted. [6] Stadermann F. J. et al. (2005) *Geochim. Cosmochim. Acta* 69, 177. [7] Stadermann F. J. et al. (2006) *LPS XXXVII*, #1663. [8] Stadermann F. J. et al. (2005) *Meteoritics & Planet. Sci.* 40, A146. [9] Floss C. et al. (2006) *Geochim. Cosmochim. Acta* 70, 2371.
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