

**RAMAN AND ISOTOPIC STUDIES OF LARGE PRESOLAR SiC GRAINS.** B. Wopenka<sup>1</sup>, M. Jadhav<sup>2</sup>, E. Lebsack<sup>2</sup>, and E. Zinner<sup>2</sup>, <sup>1</sup>Department of Earth and Planetary Sciences, <sup>2</sup>Laboratory of Space Sciences and the Physics Department, Washington University in St. Louis, One Brookings Dr., St. Louis, MO 63130, USA. [bwopenka@levee.wustl.edu](mailto:bwopenka@levee.wustl.edu).

**Introduction:** In a previous study, we obtained Raman spectra of presolar SiC grains with our 1<sup>st</sup> generation single-channel Raman microprobe [1] and correlated them with isotopic measurements. We are returning to Raman studies of SiC grains because the 3<sup>rd</sup> generation Raman microprobe we use today (HoloLab Series 5000 Raman microscope from KOSI equipped with an axial spectrograph with volume holographic transmission gratings) is superior to the instrument we had used ~25 years ago. Most notably, the HoloLab 5000 allows us to simultaneously obtain a spectrum from 100 to 4200  $\Delta\text{cm}^{-1}$  within minutes (rather than in 8-12 hours), and this capability makes it possible to extend Raman measurements to many SiC grains of different types. Here we report first results from this effort. In addition, the HoloLab has excellent throughput, spectral resolution (2  $\text{cm}^{-1}$ ), peak position accuracy ( $\pm 0.5 \text{ cm}^{-1}$ ), and peak position precision ( $\pm 0.1 \text{ cm}^{-1}$ ).

**Experimental:** Twenty large SiC grains from the SiC-rich Murchison separates LS and LU [2] were mounted on a Au foil and pressed into it with a sapphire disk. Grain sizes range from 3 to 30  $\mu\text{m}$ . We performed the Raman microprobe analyses (in selected 1  $\mu\text{m}$  spots) before X-ray analysis and SEM imaging. This allows us to confirm that Raman analysis did not do any damage to the grains. Subsequently, we measured the C and Si isotopic ratios of the grains (as well as N isotopic ratios in a few selected grains) with the NanoSIMS.

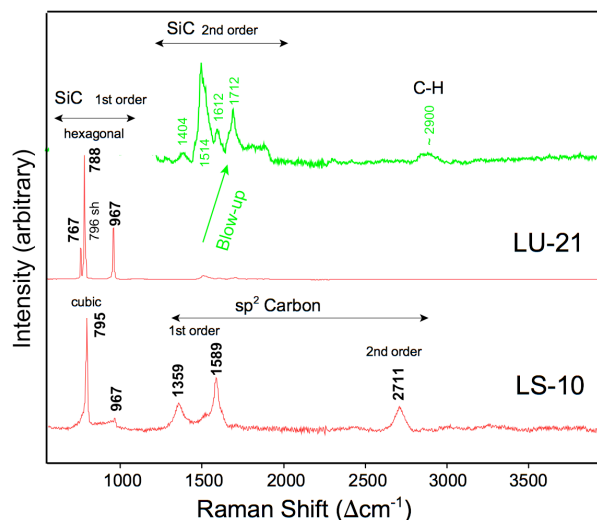


Figure 1

**Results:** Raman results are consistent with previous Raman [1] and TEM [3-4] studies, i.e., SiC grains can be cubic (characterized by one single peak of TO mode  $\sim 790 \text{ cm}^{-1}$ ) or non-cubic (the TO peak is split into three peaks). See Fig. 1. The exact peak positions as well as the peak widths, however, can be quite variable for different grains. We observed both the 1<sup>st</sup> and 2<sup>nd</sup> order modes for both cubic and hexagonal SiC grains, and found one grain, LS-10, a mainstream grain with unusually large  $^{29}\text{Si}$  and  $^{30}\text{Si}$  excesses (Fig. 2) and  $^{12}\text{C}/^{13}\text{C} = 51$ , that contains disordered carbonaceous material in addition to cubic SiC (Fig. 1).

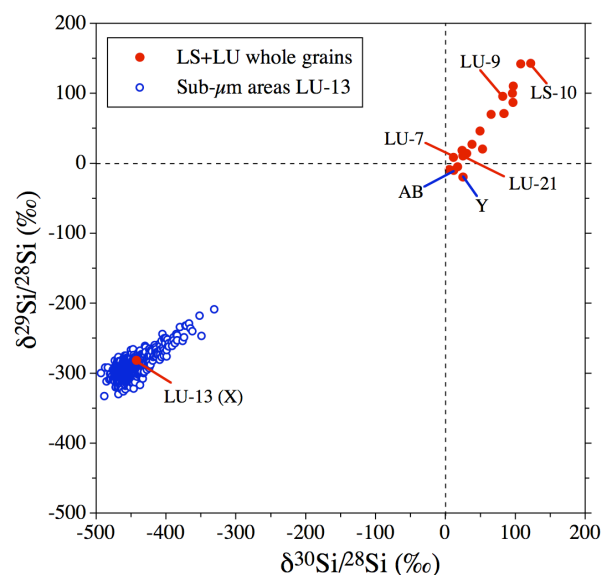


Figure 2

Figure 2 shows the Si isotopic ratios of the grains. One grain each is of type AB, X and Y, the rest are mainstream grains or have normal isotopic compositions. Figures 3 shows examples of the different types of Raman spectra that we obtained on four selected grains (labeled in Fig. 2) together with their SEM images. Among the 19 non-X grains, there seems to be a relationship between the Raman spectrum and the grain morphology. Most euhedral grains have spectra similar to that of mainstream grain LU-9, with a narrow single peak at  $\sim 796 \text{ cm}^{-1}$  for the TO mode (Fig. 3a). The Raman spectra of the "glassy-looking" grains, however, are quite different from the spectra of euhedral grains. LU-21 (Fig. 3b) is a large blocky grain with smooth surfaces. While its C isotopic ratio is normal, its Si isotopic ratios are

slightly anomalous, and its structure is hexagonal. LU-7 (Fig. 3c) has conchoidal fractures that were seen before in large SiC grains from LS+LU [1, 5]. Its C and Si isotopic ratios are normal within errors, and the Raman spectrum seems to be a "hybrid" of both cubic and hexagonal peaks. Both smooth grains LU-7 and LU-21 belong to what has been defined as Cluster I of grains with close-to-solar isotopic compositions [1]. It is still a topic of discussions whether Cluster I grains are presolar, of Solar System origin, or contamination with synthetic SiC (carborandum.)

LU-13 is a true bonanza. This X grain (Fig. 3d) is unusual because of its large size of 30  $\mu\text{m}$ . Most X grains previously studied are 3  $\mu\text{m}$  or smaller [e.g., 6-8]. TEM studies on other X-grains have shown that X grains are polycrystalline of cubic structure, with smaller crystal sizes than mainstream grains [9]. This seems to be also true of LU-13, because the SEM image shows that it consists of an agglomerate of many small crystals. The Raman spectrum is characteristic for cubic SiC with crystallographic disorder because the 789  $\text{Acm}^{-1}$  peak, as well as all the other 1<sup>st</sup> and 2<sup>nd</sup> order peaks are very wide.

In addition to the analyses of C, N, and Si isotopes of the whole LU-13 grain, we also obtained isotopic images of C and Si in multi-detection in order to investigate whether the grain has uniform isotopic composition. In Fig. 2 we plot the Si isotopic ratios of 985  $0.8\mu\text{m} \times 0.8\mu\text{m}$  areas (in blue), obtained by superimposing a grid on the image. The deviations of the data points from lines through the bulk LU-13 composition and compositions for mainstream grains are consistent with counting statistical errors for the individual areas. The stretched-out distribution of the Si isotopic ratios can thus be explained by contamination with small mainstream grains and we do not have any unambiguous evidence for Si isotopic heterogeneity of X grain LU-13.

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**References:** [1] Virag A. et al. (1992) *GCA* 56, 1715-1733. [2] Amari S. et al. (1994) *GCA* 58, 459-470. [3] Daulton T. L. et al. (2002) *Science* 296, 1852-1855. [4] Daulton T. L. et al. (2003) *GCA* 67, 4743-4767. [5] Gyngard F. et al. (2009) *ApJ* 694, 359-366. [6] Nittler L. R. (1996) *PhD Thesis, Washington University*. [7] Hoppe P. et al. (2000) *MAPS* 35, 1157-1176. [8] Lin Y. et al. (2002) *ApJ* 575, 257-263. [9] Hynes K. M. et al. (2008) *LPS XXXIX*, Abstract #2076.

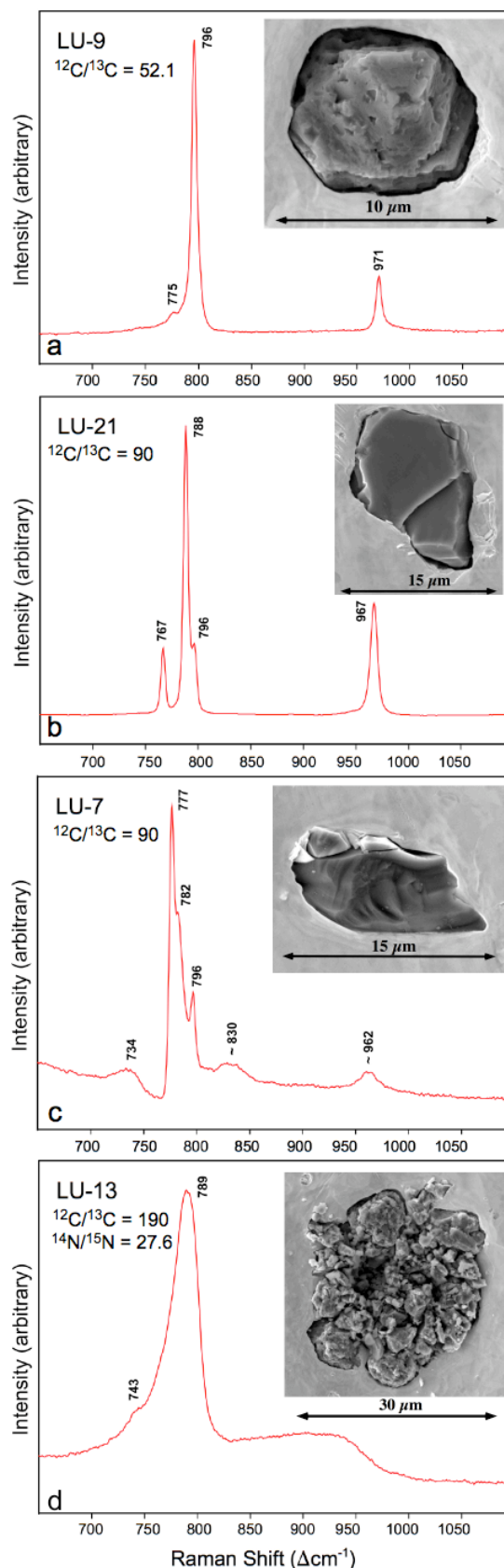


Figure 3