CONTINUED STUDIES OF ULTRAMICROTOMED PRESOLAR SIC X GRAINS.

K. M. Hynes, S. Amari, T. J. Bernatowicz, T. K. Croat, and A. F. Mertz, Laboratory for Space Sciences and Department of Physics, Washington University, St. Louis, MO 63130, USA, khynes@hbar.wustl.edu.

Introduction: Approximately 1% of the total presolar SiC population is thought to originate in supernova (SN) outflows and is classified as type X grains [1]. The primary indicator of a SN origin for SiC X grains is their large ^{28}Si excesses, although they also have significant C and N anomalies and can show evidence of the initial presence of radionuclides, such as ^{26}Al and ^{44}Ti, in the form of ^{26}Mg and ^{44}Ca [2]. While over 500 SiC grains have been studied in the transmission electron microscope (TEM), these grains were primarily mainstream SiC and most did not have correlated isotopic data [3, 4]. Microstructure and phase information with corresponding isotopic data has been obtained for only a few disordered mainstream grains [4] and for six X grains [5, 6]. Here we present preliminary results from TEM studies of three additional SiC X grains.

Experimental: X grains from the KJG fraction (3 μm observed average size [7]) of the Murchison meteorite were first located using the IMS-3f. Their C and Si isotopic ratios were subsequently measured using the NanoSIMS to confirm their SN origin. Three of these X grains were then selected for study in the TEM. These grains were removed from the Au NanoSIMS mount, placed in resin, and sliced into ~70 nm sections with a diamond ultramicrotome. The slices were then studied in a JEOL 2000FX TEM equipped with a NORAN Energy Dispersive X-ray Spectrometer (EDXS).

Results and Discussion: The three grains exhibit isotopic ratios (summarized in Table 1) that are characteristic of X grains. All show significant ^{28}Si excesses, with lower δ^{30}Si than $\delta\,^{29}$Si, and fall near the slope of 0.67 defined by most X grains on a δ^{29}Si versus δ^{30}Si three-isotope plot [8]. The C anomalies also fall within the previously reported range ($10 < {}^{12}C/^{13}C < 6800$), with all the grains showing $^{12}C/^{13}C$ > solar, which is typical for X grains.

Table 1. Isotopic results for KJG SiC X grains for Si (permil) and C (both with 1σ errors).

Grain	δ^{29}Si	δ^{30}Si	$^{12}C/^{13}C$
KJG-185-1	-296 ± 2.6	-384 ± 3.6	127.1 ± 0.5
KJG-263-1	-249 ± 2.4	-384 ± 3.4	155.8 ± 0.6
KJG-585-2	-310 ± 2.6	-495 ± 3.7	408.5 ± 2.3

Each X grain slice is ~1.7 – 3.0 μm in diameter and is composed of many small crystal domains. All three grains contain crystals that range in size from ~60 – 300 nm (geometric mean diameter ~160 nm). This is similar to the other previously studied X grains, except for one grain that contained only very small crystals ~10 nm in size [5]. The small crystal size of SiC X grains is markedly different from that of mainstream SiC grains, in which the entire grain is normally composed of a single crystal (320 – 700 nm) [3]. Figure 1 shows a typical cross-sectional slice of an ultramicrotomed SiC X grain, for which several slices were available for each grain. Due to its hardness, SiC shatters when sliced. However, because the crystal domains are smaller than the SiC fragments (typically ~250 – 600 nm in size), the crystal size and structure can be observed independently of any fragmentation (Figure 2).

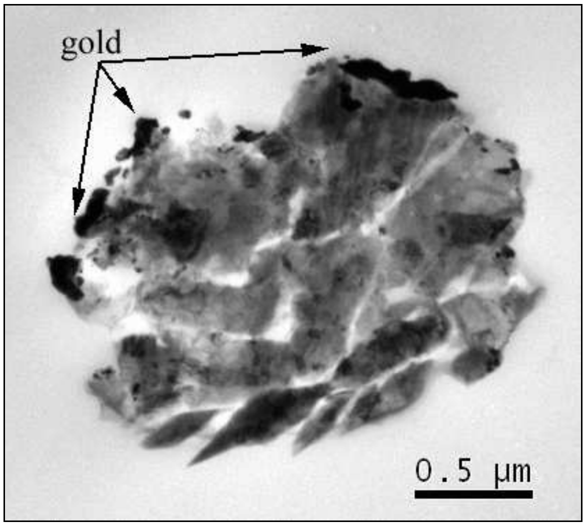

Figure 1. Bright-field (BF) TEM image of a slice of SiC X grain KJG-585-2 showing its general condition after ultramictrotomy. The black areas at the top of the slice are Au deposited during NanoSIMS analysis. Individual crystal domains can also be seen within the grain.

The small crystal size observed in SiC X grains may be the result of rapid formation. This agrees with isotopic evidence of the incorporation of live ^{49}V into SiC X grains, which requires X grains to condense over a timescale of months [9], in contrast to the years that are available for the formation of mainstream grains in AGB outflows [10]. However, microstructural investigations of mainstream grains are needed on a larger size fraction to confirm that the small crystal size observed in X grains is a result of faster formation conditions.

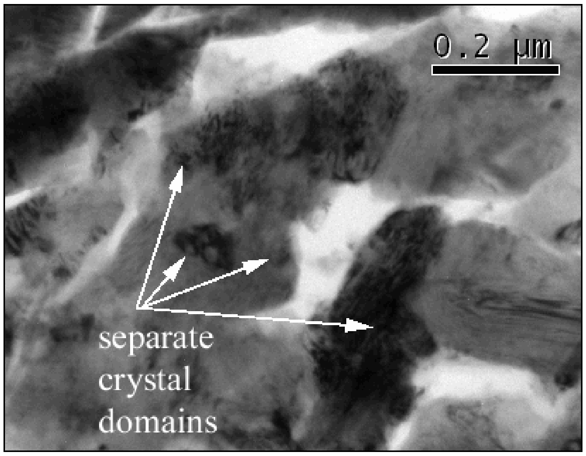

Figure 2. BF image of an area of SiC X grain KJG-585-2 showing multiple crystal domains within the grain. Intensity variations between domains are due to orientation-dependent diffraction contrast.

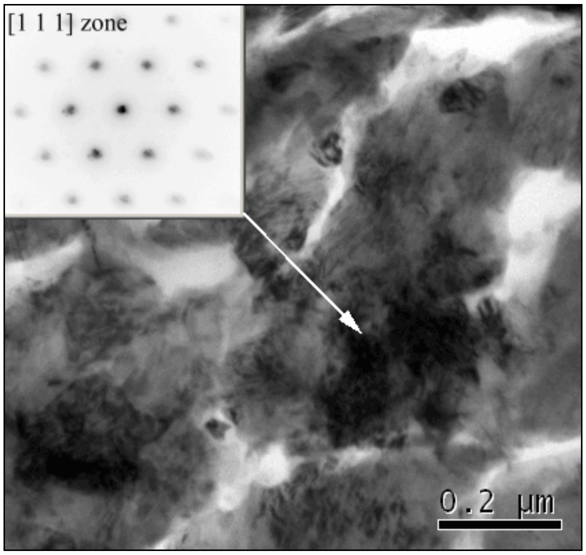

Figure 3. BF image of a 3C-SiC crystal with it corresponding [1 1 1] microdiffraction pattern.

Using microdiffraction, the polytypes of several crystal domains from each of the three X grains have been determined, mainly from <1 1 0> zones. All of the patterns are consistent with the 3C-SiC polytype (cubic; a = 4.35 Å; Figure 3). This was the polytype most commonly observed in the previously studied X grains [5, 6] and is also seen in 79% of mainstream grains [3]. Also observed are the 2H-SiC polytype and an intergrowth between the 2H and 3C polytypes. Both SiC mainstream and X grains appear to form the same polytypes, which are the lowest temperature polytypes to form. However, because higher densities exist in SN outflows, it is possible that higher order hexagonal or rhombohedral polytypes could exist in X grains, although none have yet been observed.

EDXS measurements of two X grains show significant quantities of Mg and Al. Both the Mg and Al appear to be uniformly distributed in the grains. Grain KJG-185-1 shows the highest measured Mg/Al ratio, with an approximate concentration of $Si_{88.5}Al_{6.5}Mg_5$ (C is excluded due to background from the grid), and a Mg/Al ratio of 0.74 ± 0.05. Grain KJG-585-2 had a slightly lower concentration, with $Si_{97}Al_{1.7}Mg_{1.3}$ and a Mg/Al ratio of 0.7 ± 0.1. Sufficient data are not yet available on KJG-263-1. Because an insignificant amount of Mg condenses in SiC during its formation (Mg/Al < 0.05 in mainstream SiC [11]), the presence of detectable amounts of Mg is likely due to the decay of ^{26}Al. If the Mg present in the X grains is ^{26}Mg, then the Mg/Al ratios are analogous to inferred $^{26}Al/^{27}Al$ ratios that are consistent with the highest $^{26}Al/^{27}Al$ ratios inferred from isotopic measurements of other SiC X grains [8]. These high concentrations of Mg are also consistent with three previously studied X grains [6].

Internal subgrains in SiC X grains were first observed in an ultramictrotomed grain [6]. Five Fe-Ni subgrains were found, one of which also contained Ti. TEM diffraction data from these subgrains are inconsistent with all previously observed Fe-Ni phases and may be silicides. The presence of Fe-Ni subgrains was confirmed by NanoSIMS analysis, which found evidence of subgrains in all of the grains in which Fe anomalies were measured [12]. While no subgrains have yet been found in the three new X grains, many ultramicrotome sections of these grains remain to be examined, and further searches for internal subgrains are ongoing.

Acknowledgements: We thank Roy Lewis (Enrico Fermi Institute/University of Chicago) for providing the Murchison KJG samples.

References: [1] Amari S. et al. (1992) *Astrophys. J., 394*, L43-L46. [2] Nittler L. R. et al. (1996) *Astrophys. J., 462,* L31-L34. [3] Daulton T. L. et al. (2003) *Geochim. Cosmochim. Acta, 67,* 4743-4767. [4] Daulton T. L. et al. (2006) *Meteoritcs and Planet. Sci, 41,* A42. [5] Stroud R. M. et al. (2004) *Meteoritcs and Planet. Sci, 39,* A101. [6] Hynes K. M. et al. (2006) *Meteoritcs and Planet. Sci, 41,* A83. [7] Amari S. et al. (1994) *Geochim. Cosmochim. Acta, 58,* 459-470. [8] Nittler L. R. et al. (1995) *Astrophys. J., 453,* L25-L28. [9] Hoppe P. and Besmehn A. (2002) *Astrophys. J., 576,* L69-L72. [10] Bernatowicz T. J. et al. (2005) *Astrophys. J., 631,* 988-1000. [11] Amari S. et al. (1995) *Meteoritics, 30,* 679-693. [12] Marhas K. et al. (2007) *LPSC XXXVIII,* Abstract #1338.