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Electron Microscopy of *In Situ* Presolar Silicon Carbide

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Data gathered from structural, chemical and isotopic studies of presolar grains help astronomers constrain models of stellar evolution. The isotopic properties of presolar SiC grains are very well studied and indicate that most of the grains formed in the outflows of carbon-rich Asymptotic Giant Branch (AGB) stars [1,2]. Microstructural studies of presolar SiC are more sparse, but such studies can provide important information about the nebular environment in which the presolar grains formed [e.g., 3]. Initial studies by transmission electron microscopy (TEM) and Raman spectroscopy indicated that most, if not all, isotopically anomalous SiC grains have the face-centered cubic β-SiC structure [4-6]. More recently, Daulton *et al.* [7] have shown that a significant fraction of sub-micron presolar SiC grains are of the hexagonal 2H polytype (α-SiC). All of the preceding studies were performed on SiC grains isolated from their host meteorites by harsh chemical treatments. These treatments have been shown to significantly alter the surface morphology of the grains [8], and might break apart cracked or fissured grains and preferentially destroy less resistant minerals or organic phases associated with the SiC. Here we report the first TEM microstructural study of a presolar SiC grain identified *in situ* in a polished meteorite section by x-ray mapping, and extracted for TEM analysis using the focused ion beam lift-out technique [9].

We implemented an x-ray mapping technique, similar to that developed by [10], on a JEOL JXA-8900 electron microprobe. This method exploits the higher Si K-α x-ray yield from SiC compared with other Si-bearing phases in chondrite matrices. In contrast to [10], we used a multi-detector wavelength-dispersive x-ray system, resulting in a higher signal-to-noise ratio. Elemental maps of C, Mg, Si, S and Ca (or Al) were obtained and potential SiC grain candidates identified by high counts of Si and C and low counts of the other elements. Accelerating voltage, beam current and integration times were chosen to optimize detection efficiency of micron-sized SiC grains while minimizing the number of false candidates. We obtained nine 500×500 μm maps (2.25 mm^2) from a polished section of the Cold Bokkeveld meteorite. One SiC grain, of size ~2.5×2 μm, was identified.

Diffraction studies of the SiC grain show it to be the cubic β-SiC phase, consistent with prior reports for the majority of presolar SiC grains > 1μm [4-6]. In contrast, most commercially available synthetic SiC powders have hexagonal structures. The selected-area diffraction pattern (SADP) from the [110] zone (Fig. 1a) shows heavy streaking along <111> directions, indicative of a high density of stacking faults, also consistent with prior observations. Extra spots are present that may indicate twinning. In addition, the SADP shows two rings, at 0.342 nm and 0.212 nm, corresponding to the (002) and (100) d-spacings of graphite, respectively. Dark field imaging (g = (111)) of the SiC grain (Fig. 1b) highlights the high density of stacking faults, and also reveals the presence of sub-grains. Some of these sub-grains are believed to be the graphite indicated by the rings in the SAD pattern. Other sub-grains, which appear elongated in the (001) direction of the primary SiC are likely SiC twins. The possibility of other carbides, e.g., TiC, is refuted by energy dispersive x-ray measurements, which showed only Si and C.

The SiC grain boundaries are sharply faceted along <111> directions. There is no evidence for an oxide layer at the grain boundaries, although the possibility of a very thin oxide layer (< 1 nm) cannot be ruled out. The lack of a thick oxide layer indicates that the grain was not exposed to hot (>1000K) nebular gas for any significant length of time [11]. The surrounding matrix consists primarily of sub-micron phyllosilicate grains, with no apparent crystallographic relationship to the SiC. This confirms the lower-resolution SEM observation of [10] that presolar SiC is sited as isolated grains in the matrix without other associated mineral phases.

The TEM results indicate that the SiC formed by rapid vapor-phase condensation, trapping pre-existing graphite grains in random orientations. This crystallization sequence, *i.e.*, graphite followed by other carbides, is expected for the C/O ratios and pressures found in the outer atmospheres of AGB stars [3]. However, to our knowledge, this is the first observation of graphite enclosed in presolar SiC [12].

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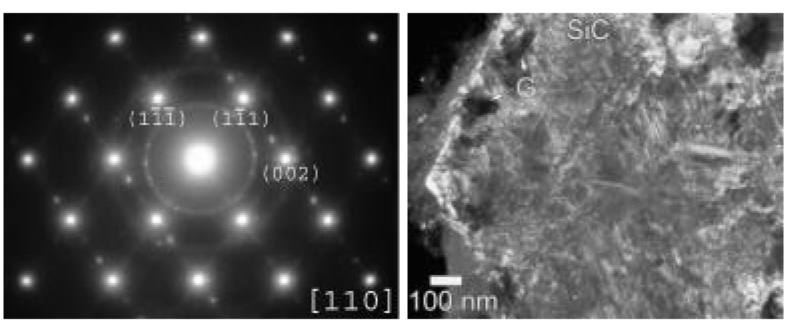

FIG. 1. (a) Selected area diffraction of a presolar SiC grain. (b) Dark field image of a presolar SiC grain. Possible graphite sub-grains are indicated by "G".

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