

**Microstructures of presolar Al<sub>2</sub>O<sub>3</sub> grains.** A. Takigawa<sup>1\*</sup>, R. M. Stroud<sup>2</sup>, L. R. Nittler<sup>3</sup>, C. M. O'D. Alexander<sup>3</sup>, and A. Miyake<sup>1</sup>, <sup>1</sup>Department of Geology and Mineralogy, Kyoto University, Kyoto, Japan, <sup>2</sup>Naval Research Laboratory, Washington DC, USA, <sup>3</sup>Department of Terrestrial Magnetism, Carnegie Institution of Washington, Washington DC, USA. \*E-mail: takigawa@kueps.kyoto-u.ac.jp

### Introduction:

Presolar grains are rare components of primitive chondrites that formed in the outflows of evolved stars, such as asymptotic giant branch (AGB) stars, red giants, and supernovae (SNe) [e.g., 1]. Their chemical compositions, morphologies, and crystal and interior structures potentially reflect their formation and evolutionary histories, and their isotopic compositions indicate their stellar origins.

Previously, we have performed sequential analyses including detailed morphological observations with field-emission scanning electron microscopy, energy dispersive X-ray spectroscopy, electron backscattered diffraction, cathodoluminescence spectroscopy, and oxygen isotopic measurements with SIMS on 260 Al<sub>2</sub>O<sub>3</sub> grains identified from acid residues of unequilibrated ordinary chondrites. Of these 260 grains, we found 18 presolar grains [2-5]. In this study, we observed the interior microstructures of several presolar Al<sub>2</sub>O<sub>3</sub> grains in detail.

### Experiments:

Presolar and solar Al<sub>2</sub>O<sub>3</sub> grains, which showed anomalous and normal oxygen isotopic compositions, respectively, identified from acid residues of the unequilibrated ordinary chondrites QUE 97008 (LL3.05), Roosevelt County (RC) 075 (H3.1), and Bishunpur (LL3.15) [2-3] were examined in this study.

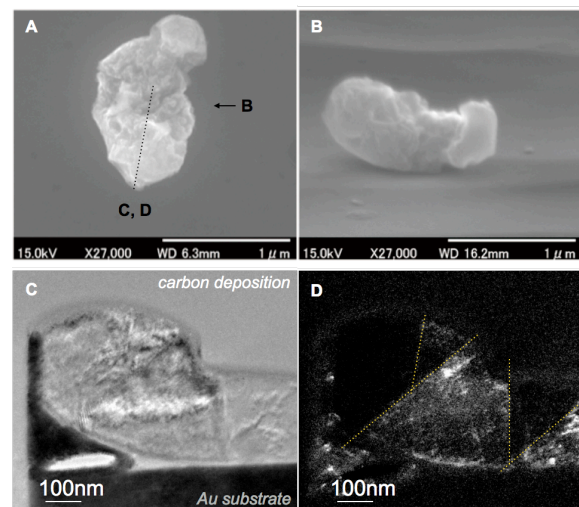
Ultra-thin sections of the 15 presolar and 4 solar Al<sub>2</sub>O<sub>3</sub> grains were prepared by a focused ion beam (FIB) lift-out technique with field emission scanning electron microscopes (FIB-SEMs): an FEI Nova 600 FIB-SEM and a Zeiss Auriga FIB-SEM at the Naval Research Laboratory (NRL) and the Carnegie Institution of Washington, respectively. Each thin-section was observed with transmission electron microscopes: a JEOL JEM-2200FS FE-STEM at NRL and a JEOL JEM-2100F FE-TEM at Kyoto University.

### Results and Discussions:

Fourteen presolar and four solar Al<sub>2</sub>O<sub>3</sub> grains were determined to be corundum by TEM electron diffraction. One grain (Bis60-44) had been almost completely sputtered by the primary beam of the SIMS, and only a damaged layer due to the SIMS primary beams remained.

Corundum twins were observed within a presolar grain, RC58-22, from RC 075 (Fig. 1). The twinning structures could have formed during condensation, crystallization of amorphous Al<sub>2</sub>O<sub>3</sub>, or

as a result of shock or high pressures. The morphology of RC58-22 is very irregular (Fig. 1), which indicates that this grain may have condensed as amorphous Al<sub>2</sub>O<sub>3</sub> and crystallized to be corundum afterward. It is thus likely that the observed twins formed during crystallization of amorphous Al<sub>2</sub>O<sub>3</sub>.



*Figure 1: Secondary electron images of RC58-02 taken from top (A) and the direction indicated by arrow (B) prior to the isotopic measurements (after [2]). C and D show a bright-field and a dark-field TEM image of a part of the FIB section (see A), respectively.*

A Group 3 (<sup>16</sup>O-rich) grain, RC59-07, was also identified from an acid residue of RC 075. In addition to aluminum and oxygen, titanium and chromium were detected from the whole of the FIB section (Fig. 2). No precipitates with high amounts of Ti or Cr were observed. Secondary electron images taken during thinning of the FIB section (FIB tomography) showed that several cracks exist in RC59-07 connecting with the grain surface. Several dark (low-Z) regions in the dark-field STEM image of the FIB section (Fig. 2) were observed, but no corresponding contrast was confirmed in the secondary electron images of the FIB section, which indicates that these dark regions are isolated voids. Iron and sulfur were detected from a small bright region indicated by a white arrow in Fig. 2, which may be iron sulfides. They were also detected from the dark regions (black arrows in Fig. 2), which suggests that small iron sulfide grains exist associated with voids.

Iron- and sulfur-rich grains were not observed in any other solar or presolar grains making

contamination unlikely. Exsolution is the most straightforward explanation for the association with voids but the conditions necessary to precipitate iron sulfides from corundum are unknown. Since iron sulfides are low temperature condensates, it is unlikely that they condensed earlier than or simultaneously with the host corundum or amorphous  $\text{Al}_2\text{O}_3$  condensation. We should note that exsolution of subgrains from a presolar  $\text{Al}_2\text{O}_3$  grain was also proposed to explain the Ti-oxide grains associated with voids within a presolar corundum, Bis60-35 (Bishunpur) [4]. Voids were observed within other presolar corundum grains, such as QUE137 and QUE060, and a solar corundum grain QUE132, but only Al and O were detected from these voids. The two types of voids (voids with and without subgrains) suggest that void and subgrain formation do not necessarily relate to each other, or that there are multiple processes that form voids and subgrains.

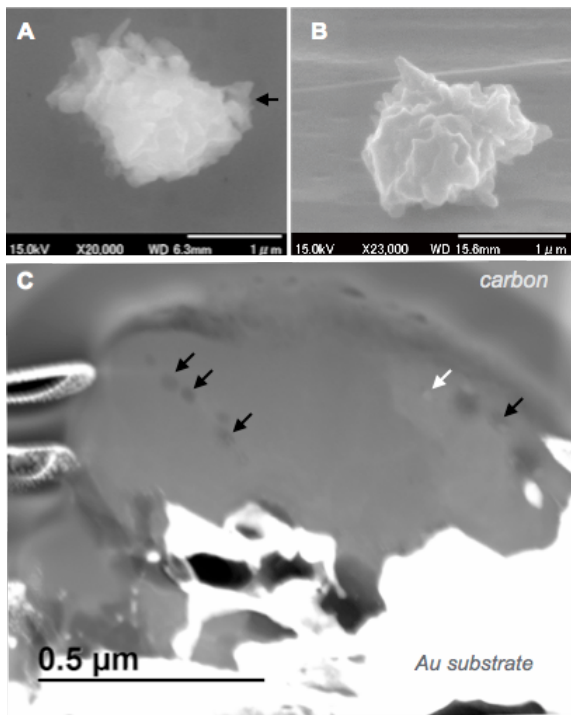


Figure 2: Secondary electron images of RC59-07 taken from top (A) and the direction indicated by arrow (B) prior to the isotopic measurements (after [2]). Dark-field STEM image of the FIB section of RC59-07 (C). Iron and sulfur were detected from regions indicated by arrows.

#### References:

- [1] Zinner, E. (2014) in 2<sup>nd</sup> edition of *Treatise on Geochemistry*, 1, pp.181-213. [2] Takigawa A. et al. (2014) *GCA*, 124, 309. [3] Takigawa A. et al. (2014) *LPS XLV*, 1465. [4] Takigawa A. et al. (2014) *M&M*, 1694. [5] Takigawa A. et al. (2013) *Meeting of the Met. Soc.*, A335.