High-temperature Dust Condensation around an AGB Star: Evidence from a Highly Pristine Presolar Corundum

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Abstract

Corundum (α-Al2O3) and amorphous or metastable Al2O3 are common components of circumstellar dust observed around O-rich asymptotic giant branch (AGB) stars and found in primitive meteorites. We report a detailed isotopic and microstructural investigation of a unique presolar corundum grain, QUE060, identified in an acid residue of the Queen Alexandra Range 97008 (LL3.05) meteorite. Based on its O and Mg isotopic compositions, this 1.4 μm diameter grain formed in a low- or intermediate-mass AGB star. It has four developed rhombohedral {011} faces of corundum and a rough, rounded face with cavities. High Mg contents (Mg/Al > 0.004) are due to the decay of radioactive 26Al. No spinel (MgAl2O4) inclusions that might have exsolved from the corundum are observed, but there are several high-Mg domains with modulated structures. The subhedral shape of grain QUE060 is the first clear evidence that corundum condenses and grows to micrometer sizes in the extended atmospheres around AGB stars. The flat faces indicate that grain QUE060 experienced little modification by gas–grain and grain–gas collisions in the interstellar medium (ISM) and solar nebula. The Mg distribution in its structure indicates that grain QUE060 has not experienced any severe heating events since the exhaustion of 26Al. However, it underwent at least one very transient heating event to form the high-Mg domains. A possible mechanism for producing this transient event, as well as the one rough surface and cavity, is a single grain–grain collision in the ISM. These results indicate that grain QUE060 is the most pristine circumstellar corundum studied to date.

Key words: circumstellar matter – dust, extinction – methods: analytical – stars: AGB and post-AGB – stars: mass-loss – stars: winds, outflows

1. Introduction

Corundum, the only thermodynamically stable phase of alumina (Al2O3), is one of the most refractory dust species expected to condense from a gas of solar composition (e.g., Ebel 2006). Mid-infrared (MIR) spectroscopic observations have revealed that more than 90% of semi-regular variables and 20% of Mira variables, which are on the asymptotic giant branch (AGB), show single peaks at 13 μm (e.g., Speck et al. 2000; Sloan et al. 2003). Corundum is the most plausible dust species capable of producing this 13 μm feature (Zeidler et al. 2013; Takigawa et al. 2015). Amorphous or metastable alumina, which shows a broad MIR spectral feature peaking at 11–12 μm, is roughly as abundant as silicate dust around many O-rich AGB stars (e.g., Little-Marenin & Price 1986; Little-Marenin & Little 1990; Speck et al. 2000; Sloan et al. 2003). Recent radio observation of such an alumina-rich star, W Hya, shows that AIO gas molecules efficiently condense to dust while most SiO molecules remain in the gas (Takigawa et al. 2017). These authors also suggested that alumina dust grows and accumulates near W Hya from the observed gas distribution and dust mass. However, dust formation in the extended atmospheres of alumina-rich AGB stars is poorly understood because of the limited mineralogical and crystallographic information that is available. It is not even clear if circumstellar corundum is a direct condensate or if it forms by post-condensation transformation of amorphous or metastable alumina that formed in very rapidly cooling gases.

Presolar dust grains that must have formed prior to the birth of the Solar System are found in primitive chondritic meteorites and cometary dust, and have isotopic compositions indicating origins around evolved stars, such as AGB stars and supernovae. They may not be fully representative of all circumstellar dust grains, but because they can be studied in the laboratory with state-of-the-art microanalytical instrumentation, they can provide detailed quantitative information about the histories of individual dust grains that cannot be obtained by astronomical observations.

Presolar Al2O3 grains with isotopic compositions indicating AGB star origins have been identified in primitive chondrites (e.g., Hutcheon et al. 1994; Nittler et al. 1997; Takigawa et al. 2014). The sizes, morphologies, and crystal structures of presolar grains from AGB stars will reflect the condensation conditions in the circumstellar envelopes of their parent stars (e.g., pressure, temperature, cooling rate, and chemical composition of the envelope), but may also record alteration in the interstellar medium (ISM) and protosolar disk. Refractory presolar grains like SiC, graphite, Al2O3, and MgAl2O4 (spinel) can be isolated from their host meteorites via physical and chemical processes (Amari et al. 1994; Cody et al. 2002; Bernatowicz et al. 2003). However, most of the Al2O3 grains in meteorite residues have solar origins (Nittler et al. 1997, 2008; Choi et al. 1998; Takigawa et al. 2014), and presolar Al2O3 grains thus must be identified by secondary ion mass spectroscopic (SIMS) isotopic measurements. Because SIMS sputters and alters grain surfaces, determining grain...
instrumental mass fractionation and to determine the relative fractions. Morphological studies of pristine presolar Al2O3 grains are thus severely limited in number (Choi et al. 1998; Takigawa et al. 2014). Transmission electron microscopy (TEM) studies of presolar oxide grains were made possible with the development of focused ion beam (FIB) methods (Stroud et al. 2004), but the number of such studies is also small (Stroud et al. 2004, 2007; Zega et al. 2011, 2014). The microstructures of presolar Al2O3 grains reported to date include an amorphous grain, several corundum grains, and a crystalline Al2O3 tentatively identified as having a hexagonal crystal structure (Stroud et al. 2004, 2007).

Here we report on a unique subhedral presolar Al2O3 grain found in a residue of the Queen Alexandra Range (QUE) 97008 ordinary chondrite. From a systematic study of Al2O3 grains in acid residues that combined SEM, SIMS, FIB, and TEM, in that order, we discuss the origins of the morphology and interior microstructures, and thermal history of the grain.

2. Experiment

A residue of the primitive (LL3.05) QUE 97008 meteorite was prepared using the CsF procedure described in Cody et al. (2002) and Cody & Alexander (2005) to remove the bulk of the meteoritic material, followed by treatment with boiling perchloric acid to destroy carbonaceous material and chromite (FeCr2O4). Droplets of the residue suspended in isopropanol and MilliQ water were deposited onto a high-purity Au substrate so that on drying the grains were sparsely dispersed over its surface. Seventy-two Al2O3 grains were initially identified by energy dispersive X-ray spectroscopy (EDS) in a field-emission (FE)-SEM (JEOL JSM-6500F) at the Carnegie Institution of Washington (CIW) and then imaged from five directions by tilting and rotating the SEM stage. Electron backscatter diffraction (EBSD) measurements of grain surfaces were also made with a FIB-SEM (FEI Nova 600) with an HKL EBSD system at the US Naval Research Laboratory (NRL) on all grains to determine their crystal structures. Details of the EBSD method are described in Takigawa et al. (2014). Additional Al2O3 grains in acid residues of the ordinary chondrites Semarkona (LL3.0, 17 grains), Bishunpur (LL3.15, 23 grains), and Roosevelt County 075 (H3.1, 37 grains), previously identified by Takigawa et al. (2014), were also subjected to isotopic analysis.

The isotopic measurements were performed with the Cameca NanoSIMS 50L ion microprobe at CIW. The O isotopes of 149 Al2O3 grains were measured with a ∼100 nm Cs+ primary ion beam rastered over each of the grains with simultaneous detection of the secondary ions of 12C-, 16O-, 17O-, 18O-, 24Mg16O-, and 27Al16O-. The primary beam sputtering of the grains was minimized by halting the isotopic measurements as soon as isotopic anomalies were clearly detected. This resulted in increased statistical errors for the isotopic compositions of the presolar grains compared to the grains of Solar System origin (i.e., those without clear O isotopic anomalies). An ∼400 nm O- primary ion beam was used to measure the Mg–Al isotopic compositions of the presolar and some solar Al2O3 grains. Secondary ions of 24Mg+, 25Mg+, 26Mg+1, and 27Al+ were simultaneously measured. Micron-sized Burma spinel grains were used as a standard to correct for the instrumental mass fractionation and to determine the relative sensitivity factor of secondary Mg and Al ions. The standard

\[ \text{Figure 1. Oxygen isotopic compositions of presolar Al}_2\text{O}_3 \text{ grains from ordinary chondrites RC 075, Bishunpur, Semarkona, and QUE 97008 are shown in Figure 1.} \]

26Mg/24Mg of 0.13932 (Catanzaro et al. 1966) and a sensitivity factor of 1.16 were used to calculate the initial 26Al/27Al ratio.

Ultra-thin sections of a few identified presolar grains were prepared with the FIB-SEM at NRL. TEM studies were carried out with field-emission scanning transmission electron microscopes (STEM) at NRL (JEOL JEM-2200FS) and Kyoto University (JEOL JEM-2100F equipped with JED 2300T). Chemical compositions were measured by STEM-EDS. The EDS map was acquired with 0.98 sr at ∼140 pA for ∼1300 sec and quantified with the K-factor method (Cliff & Lorimer 1975). We focus here on the TEM data from one particularly interesting grain, QUE060; results for the other grains will be reported elsewhere.

3. Results

3.1. Isotopic Compositions

The O isotopic measurements revealed six presolar grains from the acid residue of QUE 97008, and one from that of RC 075 (Figure 1 and Table 1). No new presolar grains were identified from the residues of Semarkona and Bishunpur. Combined with the grains reported by Takigawa et al. (2014), the presolar grain fraction in Al2O3 grains of residues from Semarkona, Bishunpur, RC 075, and QUE 97008 are 0/25, 2/72, 8/87, and 6/72, respectively.

Grain QUE060 is enriched in 17O compared to terrestrial by a factor of 2.8, but depleted in 18O by a factor of 25 (Table 1), which identifies it as a Group 2 grain according to the classification scheme for presolar oxide grains (Nittler et al. 1997). A large excess of 26Mg was detected due to the decay of radioactive 26Al, with an inferred initial 26Al/27Al ratio of 0.0126 (23) (Table 1). The O and Al–Mg isotopic compositions indicate that grain QUE060 originated either in an ∼5 M_Sun AGB star undergoing hot-bottom processing (Lugaro et al. 2017), or a lower-mass (<2 M_Sun) AGB star undergoing cool-bottom processing (Nittler et al. 2008).
The mean of the longest and shortest dimensions of grain QUE060 is about 1.4 μm. Four flat faces are clearly observed on the grain (faces a–d in Figure 2). Such flat and smooth faces were not observed for any other presolar Al₂O₃ grains examined here, nor have they been reported in previous studies (Choi 1998; Takigawa et al. 2014). The area e shows a rough and rounded surface with

### Table 1

| Meteorite | ¹⁸O/¹⁶O  | ¹⁶O/¹⁶O | ²⁵Mg/²⁴Mg | ²⁴Mg/²⁴Mg | ²⁴Mg/²⁷Al | ²⁶Al/²⁷Al |
|-----------|---------|---------|-----------|-----------|-----------|-----------|
| QUE018    | 8.41 (6) × 10⁻⁴ | 1.87 (2) × 10⁻⁴ | 0.123 (1) | 0.140 (1) | 2.88 (1) × 10⁻² | 1.80 (1) × 10⁻² |
| QUE053    | 2.43 (4) × 10⁻⁴ | 1.36 (4) × 10⁻⁴ | 0.213 (48) | 0.200 (5) | 3.33 (33) × 10⁻⁴ | 2.03 (52) × 10⁻² |
| QUE060    | 1.07 (1) × 10⁻³ | 7.99 (1.69) × 10⁻³ | 0.113 (3) | 99.0 (8.6) | 1.27 (11) × 10⁻⁴ | 1.26 (23) × 10⁻² |
| QUE067    | 9.13 (15) × 10⁻⁴ | 5.60 (4) × 10⁻⁴ | 0.129 (15) | 6.73 (31) | 4.63 (20) × 10⁻⁴ | 3.07 (19) × 10⁻³ |
| QUE088    | 4.14 (7) × 10⁻⁴ | 2.16 (1) × 10⁻⁴ | 0.135 (14) | 0.110 (14) | 1.80 (74) × 10⁻⁴ | ... |
| QUE124    | 7.52 (12) × 10⁻⁴ | 3.96 (2) × 10⁻⁴ | 0.176 (86) | 263 (53) | 2.95 (59) × 10⁻⁵ | 7.75 (2.19) × 10⁻³ |
| QUE137    | 1.35 (1) × 10⁻³ | 3.31 (1.36) × 10⁻⁵ | 0.139 (7) | 7.40 (15) | 2.19 (4) × 10⁻³ | 1.59 (4) × 10⁻² |
| RC075 58-02 | 3.21 (6) × 10⁻⁴ | 1.66 (1) × 10⁻³ | 0.138 (31) | 0.0810 (245) | 5.67 (50) × 10⁻⁵ | ... |

**Note.**

All errors are 1σ.

### 3.2. Morphology and Surface Structure

Figure 2 shows secondary electron images of grain QUE060 on an Au substrate that were acquired prior to modification of its surface morphology by the SIMS isotopic measurements. The tilting and rotating angles of Figures (A)–(E) are shown in Figure (F). A FIB section was prepared along the dash lines in (T, R) = (0°, 0°) image of Figure (F). A small subgrain (<100 nm) is attached to the area e. The faces of a, b, c, and d were indexed to rhombohedral (011), (110), (101), and (101) faces of corundum.
no clear edge and seems to have cavities (Figures 2(B)–(D)). The rough surface structure of area e is similar to the characteristic surface structures observed on most previously studied presolar Al$_2$O$_3$ grains (Choi et al. 1998; Takigawa et al. 2014).

The EBSD patterns taken prior to the SIMS measurements on multiple locations on face b (Figure 2) were indexed to that of corundum. The EBSD analysis shows that at least the very surface layer (<30 nm in depth) sampled by the measurements, which was subsequently lost during the SIMS measurements, originally had the corundum crystal structure, which is consistent with that of the grain interior as shown in the following section.

3.3. Interior Microstructures

An FIB lift-out section of grain QUE060, cut perpendicular to the Au substrate along the dashed lines in Figure 2(F), was prepared for TEM analysis (Figure 3). The SEM image in Figure 2(D) was taken almost parallel to the TEM images in Figure 3. The flat face indicated by the arrow in Figure 3(A) corresponds to face a in Figure 2. Comparing the initial shape and size (Figure 2(D)) with those in the FIB section (Figure 3(A)), most of the grain volume was preserved after the SIMS measurements. A cavity seen on area e of Figure 2 was also observed on the left part of the grain facing the Au substrate (Figure 3(B)). The ion beam damage caused by the SIMS measurements and FIB lift-out cannot be responsible for the cavity formation because the cavity was not exposed to the electron and ion beams.

Very similar electron diffraction patterns of corundum (Figure 3(B)) were obtained from most locations across the whole grain (the few exceptions will be described later), which indicates that this grain is a single crystal of corundum. From the EBSD pattern and the electron diffraction patterns along the three different zone axes, the faceted faces of a, b, c, and d in Figure 2 are indexed to rhombohedral (011), (110), (101), and (010) faces, which are crystallographically identical {011} planes and generally called r-planes (Hartman 1980). Note that we took the smallest rhombohedral unit cell to describe the crystal faces and directions of corundum in this Letter.

The bright-field (BF) TEM image in Figure 3(A) can be seen to be darkening from right to left. Electron diffraction patterns taken at the center of the grain and the edge of the cavity showed a rotational difference of ~0°.3. The gradual change of the contrast in the BF-TEM image from left to right (large-scale contrast) is thus due to rotational differences of the crystallographic orientation. Both the BF and the dark-field (DF) TEM images (Figures 3(A) and (D)) show small-scale (<30 nm) brightness contrast throughout the grain, which indicates that distortions are distributed everywhere in the grain. The selected-area electron diffraction patterns from the area b in Figure 3(A) showed satellite spots (Figure 3(C)),

Figure 3. (A) BF-TEM image of the FIB section of grain QUE060. An arrow indicates flat face a in Figure 2. SAED patterns of the areas indicated by the dashed circles (selected area apertures) a and b are shown in panels (B) and (C), respectively. They are taken in the [11̅1] orientation of corundum (written in the rhombohedral setting). (D) DF-TEM image using a diffraction 011 spot. (E) DF-TEM image from spots indicated by a circle (objective aperture) in panel (C). (F) Al (red) and Mg (green) overlay map obtained with STEM-EDS.
indicating modulated structures in the corundum crystal. These satellite spots were observed from several domains corresponding to the bright areas in the DF-TEM image (Figure 3(E)). These domains are <130 nm in diameter. The average Mg/Al ratio in the grain measured with STEM-EDS is 0.014(1). The detected Mg is essentially pure radiogenic \(^{26}\text{Mg}\) based on the measured Mg isotopic composition (Table 1), and the ratio obtained with EDS is consistent with the \(^{26}\text{Mg}/^{27}\text{Al}\) of 0.0126(15) determined by NanoSIMS. The Mg/Al ratios are not uniform within the grain, but are always greater than Mg/Al ~0.004(1) (2000 ppm Mg by weight), which is significantly higher than the Mg detection limit of 0.001 in our EDS measurements. The domains showing satellite spots contain a higher amount of Mg (Figure 3(F)). The Mg/Al ratios in such domains are up to 0.038(3), which is significantly higher than those in the surrounding regions. The DF image of satellite spots shows that the modulated structure is not present in the lower Mg-content regions (Figure 3(E)).

4. Discussion

4.1. Formation of Grain QUE060

The existence of presolar corundum grains does not rule out the possibility that the grains originally condensed as amorphous or metastable alumina and later transformed to corundum at >1000°C (Levin & Brandon 1998). Had grain QUE060 initially condensed as amorphous alumina around its parent AGB star, the faceted surfaces would need to have formed later by surface diffusion of molecules following crystallization. Because amorphous \(\text{Al}_2\text{O}_3\) formation needs very rapid cooling of the gas (e.g., Dragoo & Diamond 1967), reheating is required for crystallization and surface diffusion. Heating events in the outflows of AGB stars beyond the dust formation regions are mainly due to shock wave propagation. These are transient events, whereas sustained high temperatures are needed for faceted face formation by surface diffusion. Melting or surface diffusion in the protosolar disk can be ruled out because isotopic exchange with the surrounding gas is very efficient at high temperatures and would have erased the isotopic anomalies. Thermal alteration in the parent body of QUE 97008 is also unlikely because QUE 97008 (LL3.05) shows very little evidence of aequous alteration or thermal metamorphism (Grossman & Brearley 2005). Hence, the faceted faces on grain QUE060 suggest that this grain originally condensed as crystalline corundum.

Corundum grains found naturally in the Earth or synthesized with flux methods in laboratories generally show well-developed \{111\} faces (c-planes). However, the most developed faces theoretically predicted using attachment energies and from ab initio calculations are \{110\} (Hartman 1980, 1989; Rohl & Gay 1995) as observed for grain QUE060. Hartman (1989) argued that the development of \{111\} faces is due to adsorption of atoms on the \{111\} faces, where the density of unsaturated bonds is highest, and that \{110\} faces should develop at high temperature and low supersaturation conditions. It is, therefore, most likely that the faceted faces observed on grain QUE060 developed during the condensation and growth of crystalline \(\text{Al}_2\text{O}_3\) around its parent AGB star. There is no evidence of reaction between grain QUE060 and the surrounding gas, which indicates that the grain remained and grew within the high temperature region near the central star where no other mineral was thermodynamically stable. The grain was then transported rapidly to the low temperature and gas density regions along with the accelerated outflow so that other refractory grains could not grow on it or form by gas–grain reaction.

4.2. Formation of the Rough Surface and Cavities

The uniqueness of the very flat \{110\} faces on grain QUE060 indicates that this grain did not spend a very long time in the diffuse ISM. The rough surface area on grain QUE060 (area \(a\) in Figure 2) may be a secondary feature possibly formed by dust–gas or dust–dust collisions in the ISM or protosolar disk. Corundum is not dissolved even by more harsh acid treatments using HF, but amorphous and metastable alumina phases do dissolve into HF-HCl or HClO\(_4\) (Takigawa et al. 2014). If partial amorphization or deformation of the crystal structure occurred as the result of such a collision, the damaged areas could have been dissolved during the isolation procedures.

A grain–grain collision (Jones et al. 1996) is also a possible mechanism for forming the cavity observed in the FIB section. In addition to forming a crater, a high-velocity collision would have generated a shock wave that propagated through the grain (Jones et al. 1996). The rotational misorientation from left to right in the FIB section may be explained by such a shock wave propagating from the cavity.

4.3. Thermal History after \(^{26}\text{Al}\) Decay

The inferred initial \(^{26}\text{Al}/^{27}\text{Al}\) of grain QUE060 is about 0.01. Thus, the radiogenic \(^{26}\text{Mg}\) produced by \(^{26}\text{Al}\) decay should have initially occupied roughly one percent of the octahedral Al-sites in the corundum structure. The solubility limit of MgO in corundum is 130 ppm at 1600°C (Miller et al. 2006) and the abundance of Mg in grain QUE060 (≥2000 ppm) is much higher than this. Because spinel (\(\text{MgAl}_2\text{O}_4\)) and corundum show complete immiscibility, if it had been able to achieve thermodynamic equilibrium after the decay of the \(^{26}\text{Al}\) grain QUE060 would have decomposed to \(^{26}\text{Mg}\text{Al}_2\text{O}_4\) and corundum by the following reaction:

\[
^{26}\text{Mg}_{x}^{27}\text{Al}_{1-x}\text{O}_3 = x^{26}\text{Mg}^{27}\text{Al}_2\text{O}_4 + (1-2x)^{27}\text{Al}_2\text{O}_3 + 2x \times \text{O},
\]

where \(x\) denotes the initial ratio of \(^{26}\text{Al}/^{27}\text{Al}\) when the grain condensed. If all \(^{26}\text{Mg}\) diffused to form stoichiometric spinel, about 1.6 vol.% of the grain should have become spinel. No precipitate of Mg-bearing phases was observed even in the Mg-rich domains—the crystal structures in the Mg-rich domains are corundum with the modulated structure. These observations indicate that there was at least one heating event to form the modulated structures by Mg diffusion, but the grain did not gain enough energy to exsolve spinel.

Based on the sizes, Mg/Al ratios, and distribution of the Mg-rich domains, \(^{26}\text{Mg}\) atoms could not have diffused in grain QUE060 by more than 0.1 μm. We estimated the maximum temperature that grain QUE060 could have experienced after the decay of \(^{26}\text{Al}\) using the activation energy and pre-exponential terms for Mg self-diffusion of 874 kJ and 74.6 cm² s⁻¹, respectively (Sheng et al. 1992). Here we assume
that the diffusion coefficients of Mg in corundum and spinel are similar (MacPherson et al. 1995). Magnesium diffuses 0.1 μm within 20 Myr at >500°C, 1 hr at >1100°C, and 1 s at >1500°C. These estimated temperatures and durations are maximum values because the distortions in the corundum structure due to Mg atoms formed by 26Al decay in octahedral sites would have enhanced the diffusion rate. It is unlikely that the grain remained at a few hundred K for such a long time while retaining its O isotopic anomaly, and thus these results indicate that after the decay of 26Al, the grain may have experienced a transient heating event. This event must have occurred in the ISM or the early solar system because the timescale for grain injection into the ISM after formation around an AGB star is much shorter than the 720,000 year half-life of 26Al. A collisional event in the ISM that could also have been responsible for the crater formation discussed above, and is an attractive explanation for the heat source that enabled the Mg diffusion.

4.4. Implication for Astromineralogy

The faceted faces of the presolar corundum grain QUE060, indexed to {011} faces, provide strong evidence for the direct condensation of corundum around an AGB star. Because of its very refractory nature, corundum condenses from a hot gas within a few stellar radii from the central star, where acceleration of the stellar wind does not occur. The micron size of grain QUE060 indicates that the grain had to survive several pulsation periods to grow (Gobrecht et al. 2016). Small Al2O3 grains (<0.1 μm) are transparent to the stellar radiation, but micron-sized grains efficiently scatter the stellar light and contribute more to the acceleration of the stellar wind (Höfner 2008). Hence, grain QUE060 formed and grew for at least several years in the extended atmosphere of its parent star before being accelerated away from it by stellar radiation. The observation of AIO gas in the atmosphere of an alumina-rich AGB star, W Hya, suggests that the growth and accumulation of alumina dust contributes to the triggering of the acceleration that generates a stellar wind (Takigawa et al. 2017). Micron-sized presolar corundum grains like grain QUE060 may have been such triggers of dust-driven winds from alumina-rich AGB stars.

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