

**DISCOVERY OF ABUNDANT PRESOLAR SILICATES IN SUBGROUPS OF ANTARCTIC MICROMETEORITES.** T. Yada<sup>1,2</sup>, F. J. Stadermann<sup>1</sup>, C. Floss<sup>1</sup>, E. Zinner<sup>1</sup>, C. T. Olinger<sup>1,3</sup>, G. A. Graham<sup>4</sup>, J. P. Bradley<sup>4</sup>, Z. Dai<sup>4</sup>, T. Nakamura<sup>5</sup>, T. Noguchi<sup>6</sup>, M. Bernas<sup>7</sup>, <sup>1</sup>Lab. for Space Sci., Phys. Dept., Washington Univ., St. Louis, MO 63130-4899, USA. <sup>2</sup>Dept. Earth Planet. Sci., Grad. Sch. Sci., Univ. Tokyo, Tokyo 113-0033, Japan. <sup>3</sup>Los Alamos Nat. Lab., Los Alamos, NM 87545, USA. <sup>4</sup>Inst. Geophys. Planet. Phys., Lawrence Livermore Nat. Lab., Livermore, CA 94550, USA. <sup>5</sup>Dept. Earth Planet. Sci., Grad. Sch. Sci., Kyushu Univ., Fukuoka 812-8581, Japan. <sup>6</sup>Dept. Material. Biol. Sci., Ibaraki Univ., Ibaraki 310-8512, Japan, <sup>7</sup>FEI Company, USA. E-mail: tyada@physics.wustl.edu

**Introduction:** Stardust found in chondrites and interplanetary dust particles (IDPs) has given us important information on stars that existed before the formation of our solar system. Recent studies have expanded the range of presolar grain types to silicates, the most dominant constituent of planetary materials [1-4]. Previous searches of micrometeorites, extraterrestrial dust grains collected from polar ice and snow [5-8], yielded no presolar grains [9-11]. Here, we report newly discovered presolar silicates from Antarctic micrometeorites (AMMs). A part of this study has been presented previously [12].

**Samples and methods:** AMMs analyzed in this study were collected at Cap-Prudhomme, West Antarctica in 1988 [6] and at Tottuki Point, East Antarctica in 1998 and 2000 [8, 13]. Twelve AMMs, AWU01-3, -9, -11, -14, -16, -24, -25, T98-G6, -H3, -H5, -G6, -NF2, and TT54B397, were used for this study. AMMs from the AWU01 series was first mounted in acrylic plugs and polished. Their cross sections were studied in a scanning electron microscope equipped with an energy dispersive X-ray spectrometer (SEM-EDS). Subsequently, they were extracted from the mounts and split for further analyses. One piece was pressed into Au foil and analyzed in the ims3f ion microprobe [14], the other was analyzed for Ne isotopes by noble gas mass spectrometry [15]. AMM from the T98 series were analyzed by SEM-EDS, after their bulk mineralogy were determined by synchrotron X-ray diffraction (XRD). They were then divided into two pieces, one pressed into Au foil for NanoSIMS analysis while the other is preserved for other analyses. TT54B397 was analyzed by SEM-EDS on a crystal-bonds mount, and was coated with Au for NanoSIMS analysis. All 12 of these AMMs were analyzed with the NanoSIMS at Washington University, using a ~1pA Cs<sup>+</sup> primary ion beam of ~200nm diameter that was accelerated to 16kV and rastered over the samples. Negative secondary ions were extracted at 8kV, sent through a double-focusing mass spectrometer and detected by a multi-collector system of electron multipliers. The mass resolving power was ~6,000. <sup>12</sup>C<sup>-</sup>, <sup>13</sup>C<sup>-</sup>, <sup>16</sup>O<sup>-</sup>, <sup>17</sup>O<sup>-</sup>, and <sup>18</sup>O<sup>-</sup> (lately <sup>28</sup>Si<sup>-</sup> and <sup>24</sup>Mg<sup>16</sup>O<sup>-</sup> instead of <sup>12</sup>C<sup>-</sup> and <sup>13</sup>C<sup>-</sup>) were simultaneously detected by five electron multipliers. The AMMs were analyzed in isotopic imaging mode

with repeated scans over a 20x20μm<sup>2</sup> area for each analysis.

**Results and discussion:** In total, a ~37000μm<sup>2</sup> surface area of the twelve AMMs was analyzed with the NanoSIMS in 86 individual imaging measurements, each lasting more than six hours. Six grains with anomalous O isotopic ratios were found in three AMMs, AWU01-16, TT54B397, and T98-H5 (Fig. 1). Four of them belong to Group 1 presolar oxide grains, which are considered to have formed in stellar outflows from red giant (RG) or asymptotic giant branch (AGB) stars [16]. Two of them fall in the region of Group 4 presolar oxides, whose origin is still not unambiguously established; although high-metallicity AGB stars [16] and Type II supernovae [17] have been proposed as possible sources. All six presolar grains were identified as silicates on the basis of SEM-EDS analyses and their <sup>28</sup>Si signal during the NanoSIMS measurements.

Figure 2 shows a <sup>16</sup>O/<sup>17</sup>O ratio map of the first discovered presolar silicate, AWU01-16 grain C-3-P1. It has an isotopically anomalous area of ~5x3μm<sup>2</sup> with <sup>17</sup>O/<sup>16</sup>O=1.41±0.05x10<sup>-3</sup> and <sup>18</sup>O/<sup>16</sup>O=2.42±0.03x10<sup>-3</sup>. This phase was also analyzed for Mg isotopes with the NanoSIMS, but no isotopic anomalies were observed. This indicates that the silicate formed in stellar wind from a RG or an AGB star before third dredge-up [16]. Also, its highly enriched <sup>17</sup>O and slightly enriched <sup>18</sup>O indicate an origin from a star with higher-than-solar metallicity (Z > 0.02) and a mass of ~1.2 or ~7 M<sub>⊙</sub>.

AWU01-16 grain C-3-P1 was further analyzed by a field emission SEM (FE-SEM) (Fig. 4). A section containing it was extracted using FIB (focused ion beam) recovery [18]. A 30keV FIB/STEM image indicates that the volume of the presolar phase is similar to that of a ~2μm sphere (Fig. 4). This grain size is about that of larger presolar SiC grains. TEM studies of the section showed that it is composed of poorly crystallized ferro-magnesium silicates, possibly hydrous, and sub-micron clusters of Fe-Ni metal/oxide grains that are texturally similar to GEMS. One intriguing possibility is that AWU01-16 is an aqueously altered, GEMS containing AMM.

Based on the cross section areas of the presolar silicates and the total analyzed area of the AMMs in this study, the abundance of presolar silicates in the

AMMs is calculated to be  $\sim 300$ ppm. Although the statistics are still limited, this value is higher than that in C chondrites ( $\sim 40$ ppm) [3, 4], but comparable to that in anhydrous IDPs ( $\sim 400$ ppm) [2]. This indicates that the parent bodies of micrometeorites resemble more those of anhydrous IDPs than those of C chondrites.

AWU01-16 has a heterogeneous, fine-grained internal texture with no discernible vesicles and also a Ne isotopic composition which is dominated by implanted solar wind [12], both of which indicate that it was not severely heated during atmospheric entry. TT54B397 is a carbon-rich AMM according to SEM-EDS analysis. In contrast the third AMM with presolar silicates, T98-H5, appears to be very similar to ordinary, fine-grained AMMs based on its SEM-EDS image and XRD spectrum.

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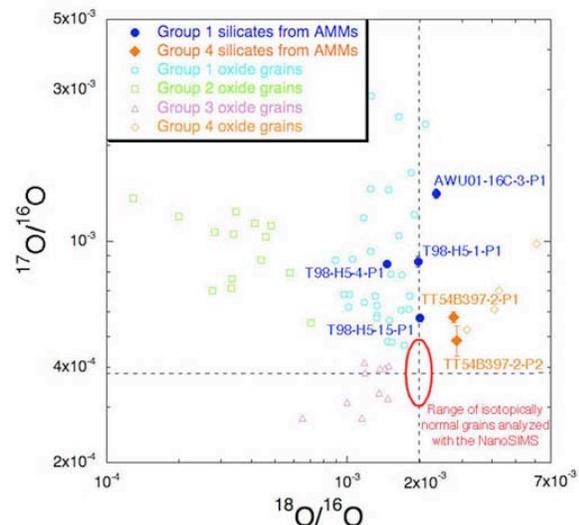


Fig. 1. Oxygen isotopic ratios of six presolar silicates found in AMMs. Data of Group 1-4 presolar oxides are shown for comparison [16]. Four grains plot in the region of Group 1, and two in that of Group 4. Dashed lines express solar isotopic ratios. Errors are  $1\sigma$ .

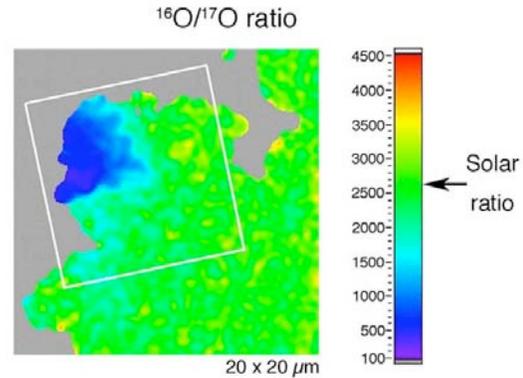


Fig. 2. A  $^{16}\text{O}/^{17}\text{O}$  ratio image with the first discovered presolar silicate, AWU01-16 grain C-3-P1. The isotopically anomalous area is  $\sim 5 \times 3 \mu\text{m}^2$ . The white square indicates for the area shown in Fig. 3.

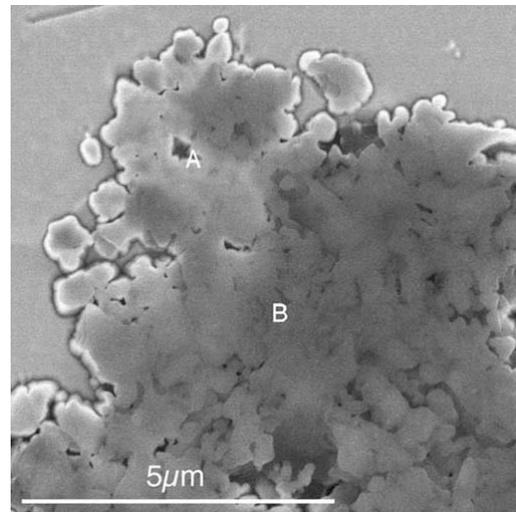


Fig. 3. FE-SEM image of AWU01-16 grain C-3-P1. It seems to be composed of  $\sim 10$ -20 grain. The letters shown in the image refer to the corresponding locations in the image in Fig. 4.

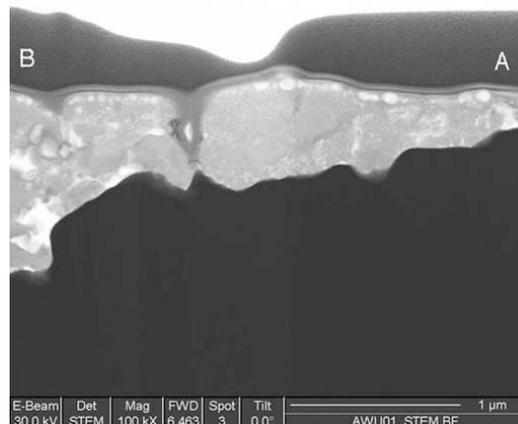


Fig. 4. STEM image of the cross section of AWU01-16 grain C-3-P1. Letters shown in the image refers to the corresponding location in the image of Fig. 3. The gray thin film shown on top of the sample is Pt deposited to protect the sample during slicing.