

Development of a new analytical scheme for micrometeorites and discovery of micrometeorites with intriguing mineralogy

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Introduction: Combined mineralogical and isotopic studies of individual Wild 2 cometary particles revealed that the materials formed in the inner solar system had been transported to the outer solar system before the formation of the Wild 2 comet [e. g. 1,2,3,4]. We sought another analytical scheme for MM (micrometeorite) studies. In this new analytical scheme, we have two objectives: identification of nonchondritic MMs and identification of asteroidal MMs with mineralogy indistinguishable from CP IDPs, which are regarded as cometary grains [5].

Samples and methods: MMs used in this study were found in the surface snow collected near the Dome Fuji Station, Antarctica in 2003 and 2010. The surface snow was melted and filtered in a clear room. After identification of MMs, we performed SR-XRD, FIB section preparation, TEM, micro-Raman, SEM, EPMA, INAA, and/or noble gas mass spectroscopy for each MM.

Results and discussion: We investigated twelve MMs and could classify them into five types based on their mineralogy: refractory MMs, chondrule-like MMs, fine-grained polycrystalline MMs, coarse-grained crystalline MMs, and phyllosilicate-rich MMs. Here we show the mineralogical results of two MMs with intriguing mineralogy.

Refractory MM A MM is composed mainly of anorthite, diopside, and spinel. A BSE image of the cross section of the MM showed that it has an amoeboid structure, in which small (<5 micrometer) Al-rich and Ti-bearing diopside exists on its surface and its interior and anorthite fills the interstices of diopside. The major minerals are similar to those in type C CAIs [6]. Although its amoeboid structure suggests low degrees of melting during the formation event, the MM has a compact interior, in which diopside and anorthite have triple junctions with $\sim 120^\circ$ angles. Therefore, the refractory object was probably formed by low degrees of melting and subsequent prolonged cooling. It is different from meteoritic type C CAIs that experienced intense melting and crystallization from melt droplets. Fine-grained material attached on the refractory object is composed of GEMS (glass with embedded metal and sulfide)-like objects, olivine, pyrrhotite, and carbonaceous material. Because GEMS-like objects have not been identified among meteorites yet, this MM may have derived from a comet.

Fine-grained polycrystalline MM A MM is composed of small (<400 nm) crystals of Fe-bearing olivine, Fe-free low-Ca pyroxene, Fe-Ni metal, Fe sulfide, amorphous silicate, and interstitial carbonaceous material. Although these crystals have often triple junctions with $\sim 120^\circ$ angles suggestive of recrystallization, both olivine and low-Ca pyroxene show almost no compositional zoning. The low-Ca pyroxene crystals elongate near the a-axis direction and are composed of a unit cell-order mixture of ortho and clino low-Ca pyroxene with stacking disorders. Its microstructure indicates rapid cooling ($>20\text{-}30\text{ K hr}^{-1}$) from above 1275K [7]. Both olivine and low-Ca pyroxene in this MM contain abundant tracks with $\sim 5 \times 10^{10}\text{ cm}^{-2}$, which corresponds to $\sim 10^4$ -year exposure to the solar wind [8]. Tracks in olivine are erased by flash heating above $\sim 600^\circ\text{C}$ [9]. Because typical cometary IDPs are heated to $>720^\circ\text{C}$ [10], it is plausible that the MM was derived from an asteroid instead of a comet although any materials similar to this MM have not been found among meteorites.

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Keywords: micrometeorites, TEM, noble gas, FIB, INAA

Chemical compositions of cosmic dust samples recovered from Antarctic snow

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We investigated four micrometeorites (MMs) recovered from Antarctic ice: D10IB034, D10IB103, D10IB179, and D10IB187. Bulk mineralogy was investigated by SR-XRD, TEM, FE-EPMA, and INAA. Analytical procedure for INAA was essentially the same as used for Itokawa grains [1]. The MMs were irradiated with neutron (total neutron fluence: 6.1×10^{18} n/cm²) along with reference standards at Kyoto University Research Reactor Institute (KURRI). After cooling for a few days, the MMs were measured for gamma-ray activity by using germanium semiconductor detectors at KURRI.

A total of eight elements (Na, Sc, Cr, Fe, Co, Ni, Sm and Ir) were determined at least for one MM sample. Their abundances relative to CI chondrite and Fe contents of individual samples show that siderophiles (Co, Ni, Ir) are more variable and depleted compared with lithophiles (Na, Sc, (Sm)). Compared with literature data for bulk and matrix samples of CM and CR meteorites, an agreement is not good within the limited data set. Even in the comparison with MMs from Antarctic ice, the four dust MMs from Antarctic snow seem to have distinct chemical features.

The FeO/Sc ratio can be used for judging the origin of planetary materials. When these ratios for the four MMs of this study are compared with data for bulk chondrites, Earth, Moon, Mars, olivine and pyroxene separates from ordinary chondrites and the Itokawa grain samples [1], the four MM samples along with chondritic materials and Itokawa grains have higher FeO/Sc ratios than those for terrestrial samples. This suggests that the four MMs are extraterrestrial in origin although their compositions are not chondritic and are not similar to MMs from ice.

Two MMs (D10IB034 and D10IB179) yielded definite values for both Co and Ni. Their abundances and ratios give us reliable judgement for the origin of planetary materials. If their contents relative to Fe contents are compared with those for Itokawa grains [1] and some constituent minerals of L and LL chondrites, the two MM from Antarctic snow are on the line defined by the CI Co/Ni ratio, clearly indicating their extraterrestrial origin. This further suggests that these MMs contain tiny metal particles or primitive materials having high and unfractionated Co and Ni abundances. Crustal materials of differentiated planets like Earth and 4 Vesta (HED parent body) also plotted in lower left region off the CI line. One MM (D10IB103) seems to be also in such region.

Only one MM sample (D10IB034) was observed to have a distinct value of Ir. When its ratio relative Co and Ni is compared with data for chondrules from ordinary chondrites (mostly unequilibrated ordinary chondrites; [2]), bulk chondrites and chondritic metals in addition to the Itokawa grain [1], bulk chondrites and chondritic metals mostly converge around the cross defined by CI ratios for Ir/Ni and Ir/Co. These elements are typical siderophile elements and, hence, they are not largely fractionated in bulk chondrites, chondritic metals and even in iron meteorites. Instead, chondrules show a large spread in Ir/Ni and Ir/Co ratios [2]. CI-normalized Ir/Fe and Ir/Co ratios of chondrules are arrayed on the line having a slope of 1, implying that chondrules have unfractionated (chondritic) Co/Ni ratios with a large variation of Ir abundances. The Itokawa grains also fit on this line [1]. It is observed that the MM D10IB034 also stay on the same line. The difference between the Itokawa grain and the Antarctic dust sample is on the Ir content. We interpret that the MM D10IB034 contains an early condensate in which Ir condensed but Co and Ni scarcely did.

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Keywords: cosmic dust, micrometeorites, chemical composition

Noble gas isotopes of micrometeorites collected from Antarctic snow

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As a comprehensive study [e.g., 1], we have been investigating micrometeorites (MMs) collected from Antarctic snow in 2003 and 2010 by JARE. MMs used in this study were recovered by filtering melted snow water at 20 °C in a clear room at Ibaragi Univ. Each MM was investigated by several analytical methods: identification using SED-EDS, followed by characterization by SR-XRD, TEM, micro-Raman, SEM, and EPMA analyses, and additionally by INAA, and/or noble gas mass spectroscopy. We classified the MMs investigated into five types based on their mineralogical features: 1) refractory, 2) chondrule-like, 3) fine-grained polycrystalline, 4) coarse-grained crystalline, and 5) phyllosilicate-rich MMs. Here, we report results of noble gas analyses for the MMs and show the relation between their noble-gas and mineralogical features.

Each of the samples mounted on Mo sheets was removed using acetone, and packed into an Al cup. The Al cups were set in a sample holder of a small furnace (designed for submicrogram to milligram samples) equipped with a purification line of the noble gas mass spectrometer at Kyushu Univ. The samples were heated *in vacuo* at 150 °C for 1 day to reduce adsorbed atmospheric gas contamination. Each sample was heated stepwise at 400, 650, and 1800 °C to extract noble gases. Absolute abundances and ratios of noble gas isotopes were calibrated by measuring known amounts of atmospheric gas and a He standard gas with ³He/⁴He of 1.71E-4. Blank levels of He and Ne at every extraction temperatures are reasonably low but those of the other elements are comparable to those released from MMs: e.g., ⁴He = 5E-12, ²⁰Ne = 5E-13, ³⁶Ar = 1E-12, ⁸⁴Kr = 1E-14, ¹³²Xe = 3E-15 cm³ STP for the 1800 °C blank run.

Samples studied are two chondrule-like MMs, one fine-grained MM, and three coarse-grained MMs, and are 30-60 micrometers in diameter. Isotopic ratios of He and Ne are indicative of solar wind (SW) origin, while elemental ratios of ⁴He/²⁰Ne are <90, lower than SW value (~650 [2]). Release profiles of SW noble gases are different among MMs: two out of seven begin to release noble gases at 400 °C, three at 650 °C, and two at 1800 °C. This indicates that they were heated to varying degrees during atmospheric entry. Plotting ⁴He concentrations against ⁴He/²⁰Ne ratios, a positive correlation is observed in the MMs, as is the case with Itokawa particles [3], IDPs [4], unmelted MMs [5, 6], and cosmic spherules [7]. The largest amounts of He and Ne among the MMs studied were obtained from a fine-grained MM (D03IB67), which consists mainly of sub-micron olivine and low-Ca pyroxene grains. This MM releases noble gases at 400-1800 °C, indicating that this MM was not heated above 400 °C during atmosphere entry. The TEM observation revealed that this MM contains abundant solar flare track with ~5E10 /cm² density, which corresponds to >1E4-years exposure [8] to solar wind and flare. The presence of the flare track is consistent with the noble gas release temperature, since the track can be erased by flash heating above ~600 °C [9].

A chondrule-like MM (D03IB057) and a single olivine MM (D10IB170) contain only small amounts of He and Ne with the lowest ⁴He/²⁰Ne ratios (1.4 and 0.4, respectively). They release detectable amounts of noble gases only at 1800 °C. Both of the MMs show signs of higher degrees of heating: the uppermost surface layer of D03IB057 is partly melted, while that of D10IB170 is decomposed into magnetite and glass.

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Keywords: micrometeorites, noble gas, solar wind

Mineralogical study of phyllosilicate-rich unmelted micrometeorites recovered from "Cape Tottuki" in Antarctica

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In the present study, seventy six particles recovered from ice at "Cape Tottuki" in Antarctica were identified as Antarctic micrometeorites (AMMs) by SEM/EDS chemical analysis. Among them, twenty eight relatively less heated AMMs were analyzed by synchrotron X-ray diffraction (S-XRD) using Gandlfi camera, which reveals bulk mineralogy of individual AMMs. Based on the results of S-XRD, we have selected four primitive AMMs. They were embedded in resin and were sliced to be ultrathin sections by ultra microtomy. The ultrathin sections were analyzed by 200-KeV FE-TEM equipped with EDS, which is able to determine nano-scale mineralogy. Rest of the particles, which remained in the resin, were polished and analyzed by EPMA/WDS, which is able to determine the local and bulk chemical composition. We accomplished this multistage detailed analysis, and revealed the mineralogy and the physicochemical properties of each small cosmic dust particle.

The results of the multistage analysis indicated that KTP2H6 is a member of Tagish Lake type micrometeorites, which are believed to come from D-type asteroids in the outer main asteroid belt. HTP2H6 consists mainly of fine-grained saponite and magnetite. The survival of hydrous mineral saponite indicates that the particle almost escaped deceleration heating when it entered the Earth's atmosphere, because saponite is decomposed at 700 degrees Celsius during the brief heating. AMMs had been stayed deeper in the blue ice field in Antarctica for about 30 thousand years, however, the crystal structure was well preserved. Thus, KTP2H6 preserve material characteristics of D-type asteroids.

KTP3I6 was also identified as Tagish Lake type micrometeorites, but it was slightly heated in the atmosphere. Although its phyllosilicate and carbonate phases were decomposed to amorphous phase and magnesiowustite, respectively, we could know the type of phyllosilicate from chemical composition. Si-rich compositions of phyllosilicates indicate that saponite is a major phyllosilicate before decomposition in the atmosphere. Magnesiowustite showed very Fe-rich composition, indicating that the particle had had Fe-rich carbonate. Fe/Mg ratios of the carbonates in KTP3I6 are the highest among carbonates found in Tagish lake meteorite and Tagish Lake-type micrometeorites. This means that the particle is identified as D-type asteroids material, but its formation process is different from the Tagish Lake meteorite. Therefore, we propose KTP3I6 is one of the varieties of D-type asteroids property. In addition, two anhydrous AMMs were also investigated in detail, however, they were highly decomposed during the atmospheric entry and also altered in the Antarctic ice layer.

Keywords: micrometeorites, Tagish Lake meteorite, D-type asteroids, carbonaceous chondrites

The search for Antarctic meteorites in the Nansen Ice Field by the joint team of JAPAN and BELARE

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So far, the meteorite search on the Nansen Ice Field spreading at the south of the Sor Rondane Mountains has been carried out only by the 29th Japanese Antarctic Research Expedition (JARE-29) and Belgian Antarctic Research (BELARE) 2010-2011. We carried out the meteorite search here by the joint team of JARE-54 summer members and BELARE2012-2013, consisting of ten members, that is, four (including one field assistant, FA) from JARE and six (including one FA) from BELARE. As a result, we collected about 420 Asuka meteorites with about 76 kg in total weight.

The total period was ~66 days, from the beginning of December 2012 to the middle of February 2013. The staying period in the Nansen Ice Field was 39 days, from the end of December 2012 to the beginning of February 2013. We used Dronning Maud Land Air Network (DROMLAN). Entering in Novo from Cape Town by Ilyushin D5 flight (5 Dec., 2012), in PE station from Novo by Basler Turbo (9 Dec., 2012). Leaving PE station to Novo by Basler Turbo (8 Feb., 2013) and Novo to Cape Town by Ilyushin D10 flight (8 Feb., 2013).

We accessed to the Nansen Ice Field from the Princess Elisabeth (PE) Station using ten snowmobiles (Ski-doo). The searched area was focused on the south-west part of the Nansen Ice Field for the first half period from the beginning until the middle of January, where we stayed at Base Camp 1 (BC1) located on 72 52 19.6 S and 24 20 28.0 E, 2909 m in elevation, and then the north-east part of the Nansen Ice Field for the latter half period since the middle of January, where we stayed at BC2 located on 72 42 04.3 S and 24 46 40.3 E, 2841 m in elevation. The camp system consists of two module containers for JARE and three containers for BELARE (among two BELARE containers are connected in line). The transportation of Japanese modules, BELARE containers, and a fuel sledge was carried out by the support from the PE station.

We formed the V-shaped system by ten snowmobiles for the method of the meteorite search, where a FA was at the top with the average moving speed of normally less than ~10 km/h. Mobile-typed small GPSs were used for the navigation of the planned route, for the recording of trajectories of the searched area, and for the positioning of found meteorites. This will be useful for the clarification of the meteorite distribution on the field. Although just after the moving camp from BC1 to BC2, we experienced the bad weather of the half period including the long-period bad weather of the continuing 12 days, the collected number of meteorites and the total weights were both larger than expected. Most meteorites appear to be ordinary chondrites, but carbonaceous chondrites and the largest meteorite of 18 kg at the present meteorite search are included among them. Possible achondrites such as eucrites and diogenites are also recognized. The detailed studies for classification and curation will be carried out in the laboratory at NIPR. In near future we are planning a detailed meteorite search of the south-east part where we could not carry out due to the restricted periods.

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Keywords: meteorite search, Asuka meteorites, JARE-54, BELARE2012-2013

Development of isotope analysis of fluid inclusions by secondary ion mass spectrometry I

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Fluids play important role for element migration and circulation on the surface and interior of planets, and often control geological phenomena. The fluid caused geological phenomenon was indirectly assumed from the record of rock formed at the time. On the other hand, fluid inclusions trapped in minerals give the direct information of the fluid. Moreover, individual analysis for each fluid inclusion can reveal fluid evolution during a series of geological action because spatial position of fluid inclusions in the mineral corresponds to course of geological time. Especially, isotope analysis provides useful information for the origin of fluid.

In traditional mass spectrometry, analysis of fluid inclusions was conducted by extraction procedure method. The candidate for application of this method was limited because this method can measure fluid inclusions with a size of 1mm or larger. The fluid inclusions in rock have often several micrometers to several dozen micrometers in size. Therefore, development of mass spectrometry applicable to such minute fluid inclusions is important. In-situ analysis of fluid inclusions is expected to make significant progress for the study of evolution of fluid. Secondary ion mass spectrometry (SIMS) is an analysis method having such spatial resolution. However, SIMS can not analyze fluid because the sample should be exposed in high vacuum. In this study, we developed a sample preparation method in order to analyze fluid inclusions by SIMS.

The requirements for stable analysis of SIMS are (1) flat and smooth mirror surface, (2) fluid inclusions are exposed to polished surface, (3) conductive sample surface.

In order to satisfy (1) and (2), we developed freezing polishing instrument to polish the sample keeping fluid inclusions in a frozen state. The instrument has a lapping table including liquid nitrogen bath. The temperature of the table during polishing is controlled at -100 degree by automatic temperature controller. It takes approximately 14 minutes to cool the lapping table from room temperature to -100 degree. Freeze and dry polish of fluid inclusions is realized with alumina polish sheet on the lapping table.

In order to prevent condensation of water vapor in the atmosphere on the polished surface and make conductive surface (condition (3)), the polishing instrument and an ion coater (SANYU SC-701AT) are put in a glove box with nitrogen atmosphere. The sample stage of ion coater usable in cooling state satisfy condition (3) by making gold thin film on the polished surface keeping the temperature of sample below -100 degree during gold coating. A reflection microscope having a cold stage to observe surface condition is also put in the glove box. We are currently developing a simple observation method to check the exposed surface of frozen fluid inclusions.

Keywords: fluid inclusions, secondary ion mass spectrometry, freezing polish, isotope

Petrography, H₂O contents and hydrogen isotopic composition of phosphate minerals from LL5-6 ordinary chondrites

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Ordinary chondrites of petrologic types 4 to 6 exhibit a sequence of progressive chemical and textural equilibration, and contain Cl-rich apatite grains (e.g., Jones et al., 2011).

Previous study reported that the water contents of chlorapatite are very low (<100ppm), even though apatite grains in ordinary chondrites seem to have a significant H₂O contents based on a deficit of anions (Jones et al., 2011). As results, they suggest that apatite grains were formed with a dry and halogen-bearing fluid that has been derived by degassing of chondritic melts.

In the preliminary study, we reported the petrography, H₂O contents and hydrogen isotopic compositions in chlorapatite from Mocs L5-6 and Ensisheim LL6 ordinary chondrites using secondary ion mass spectrometry (SIMS). The H₂O contents of apatite grains in Ensisheim (LL6) and Mocs (L5-6) are estimated to be ~60 ppm and ~35 ppm, respectively. These H₂O contents are similar to the previous reported values from LL chondrites (Jones et al., 2011). The hydrogen isotopic compositions of apatite grains from Ensisheim are extremely D-rich (dD= ~ +17000 permil). Remarkably, we found the reaction texture between apatite grain and olivine grain from Ensisheim LL6 chondrite, which reported that of texture from St. Severine LL6 chondrite (Jones et al., 2011). However, the origin of extremely D enrichment of those of Ensisheim LL6 is unclear whether this is resulting from LL6 general characteristics of thermal process of parent body or from unique results of late reaction by fluids only for Ensisheim LL6 chondrite, because of limited study of preliminary examination.

In this study, we will report the petrography of phosphate minerals with other thin sections, LL5-6 (Ensisheim LL6 and Tuxtuac LL5) to compare it with those of Ensisheim LL6 chondrite. Thin section (Tuxtuac) loaned from Natural History Museum, London (NHM) were used in this study. Phosphate grains were identified using elemental X-ray mapping using scanning electron microscope (FE-SEM JEOL JSM 7000-F) and energy dispersive X-ray spectrometry (EDS Oxford INCA Energy) at Hokkaido University.

Phosphate minerals (merrillite and apatite) from Tuxtuac show the reaction texture at the boundary of olivine and phosphate minerals. However, the chemical compositions (P, Cl and Ca) of olivine grain are homogeneously distributed using 5kV, even if those of distribution are enriched in the rim of olivine grain with 15kV X-ray mapping. This suggests that the reaction texture with BSE image might be resulting from the effect of edge crystal. On the other hand, the Cl-zonation in the reaction texture between olivine and apatite from Ensisheim LL6 chondrite with D-enrichment signature are identified with 5KV X-ray mapping.

In the future, we will further examine the petrography of phosphate minerals from other LL4-6 chondrites has already been loaned thin sections from NHM and compare with the petrography, hydrogen isotopic compositions of phosphate minerals to give an constraint for the origin of D-enrichment of phosphate minerals in the Ensisheim LL6 chondrite.

Keywords: apatite, Hydrogen isotope, LL ordinary chondrite, phosphate mineral

Oxygen isotopic composition of the solar nebula gas inferred from high-precision isotope imaging of melilite crystals

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High-precision isotope imaging analyses of reversely zoned melilite crystals in the gehlenitic mantle of Type A CAI ON01 of the Allende carbonaceous chondrite reveal that there are four types of oxygen isotopic distributions within melilite single crystals: (1) uniform depletion of ¹⁶O ($\delta^{18}\text{O} = -10$ permil), (2) uniform enrichment of ¹⁶O ($\delta^{18}\text{O} = -40$ permil), (3) variations in isotopic composition from ¹⁶O-poor core to ¹⁶O-rich rim ($\delta^{18}\text{O} = -10$ permil to -30 permil, -20 permil to -45 permil, and -10 permil to -35 permil) with decreasing akermanite content, and (4) ¹⁶O-poor composition ($\delta^{18}\text{O} > -10$ permil) along the crystal rim. Hibonite, spinel, and perovskite grains are ¹⁶O-rich ($\delta^{18}\text{O} = -45$ permil), and adjoin ¹⁶O-poor melilites. Gas-solid or gas-melt isotope exchange in the nebula is inconsistent with both the distinct oxygen isotopic compositions among the minerals and the reverse zoning of melilite. Fluid-rock interaction on the parent body resulted in ¹⁶O-poor compositions of limited areas near holes, cracks, or secondary phases, such as anorthite or grossular. We conclude that reversely zoned melilites mostly preserve the primary oxygen isotopic composition of either ¹⁶O-enriched or ¹⁶O-depleted gas from which they were condensed. The correlation between oxygen isotopic composition and akermanite content may indicate that oxygen isotopes of the solar nebula gas changed from ¹⁶O-poor to ¹⁶O-rich during melilite crystal growth. We suggest that the radial excursions of the inner edge of the protoplanetary disk gas simultaneously resulted in both the reverse zoning and oxygen isotopic variation of melilite, due to mixing of ¹⁶O-poor disk gas and ¹⁶O-rich coronal gas. Gas condensates aggregated to form the gehlenite mantle of the Type A CAI ON01.

Keywords: Chondrite, Melilite, CAI, SIMS, Oxygen isotope imaging, Solar nebula gas

Relationship between oxygen isotope zoning and crystal growth in melilite crystals from fluffy type A CAI

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The oxygen isotopic microdistributions within melilite measured using in situ secondary ion mass spectrometry correspond to the chemical zoning profiles in single melilite crystals of a fluffy Type A Ca-Al-rich inclusion (CAI) of reduced CV3 Vigarano meteorite. The melilite crystals show chemical reverse zoning within an individual single-crystal from the akermanite-rich core to the akermanite-poor rim. The composition changes continuously with the crystal growth. The zoning structures suggest that the melilite grew in a hot nebular gas by condensation with decreasing pressure. The oxygen isotopic composition of melilite also changes continuously from ¹⁶O-poor to ¹⁶O-rich with the crystal growth. These observations suggest that the melilite condensation proceeded with change consistent with an astrophysical setting around the inner edge of a protoplanetary disk where both ¹⁶O-rich solar coronal gas and ¹⁶O-poor dense protoplanetary disk gas could coexist. Fluffy Type A CAIs could have been formed around the inner edge of the protoplanetary disk surrounding the early sun.

Keywords: FTA, SIMS, oxygen isotope, CAI

Beryllium-bron measurement of a melilite-rich calcium-aluminium-rich inclusions in the NWA5958 CM chondrite

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CAIs (Ca, Al-rich Inclusions) are the oldest object in the solar system. In CAIs, there are evidence of now-extinct isotopes like ²⁶Al and ¹⁰Be. They have a short-half-life (<100Ma) and because of short-half-life, they are expected to indicate an accurate relative age. In order to estimate a relative age using now-extinct isotopes, it is required that they were homogeneously distributed in the solar nebula. In terms of ²⁶Al, it has been almost already proven that they were homogeneously distributed in the solar nebula, while ¹⁰Be has not proven yet. ²⁶Al decays by electron capture to ²⁶Mg with a half-life of 0.71Ma. ¹⁰Be *B*-decays to ¹⁰B with a half-life of 1.5Ma. In terms of Al-Mg chronology, it is difficult to detect excess of ²⁶Mg in CAIs which experienced late-stage alteration (e.g reheating in the nebula or asteroids), because the Mg diffusion in anorthite where Al-Mg measurements were made is fast. The B diffusion, however, in melilite where Be-B measurements were made is slow. Therefore, it is suggested that Be-B chronology is effective way to measure a relative age of CAIs which experienced late-stage alteration. Hence, it is significant to confirm that ¹⁰B was distributed in solar nebula homogeneously or heterogeneously.

In this study, we have carried out Be-B measurements in one CAI (Type-A CAI) from CM chondrite NWA5958. This CAI consists mainly of a large melilite crystal and small spinel and perovskite are contained as inclusions in the melilite. Our SIMS measurements at Tohoku University showed that this CAI has ¹⁰B excesses in melilite. The initial ¹⁰Be/⁹Be ratio inferred 4.6×10^{-2} . This ratio is much higher than those obtained in other CAIs from CV chondrites (e.g. 9.5×10^{-4} ; MacPherson et al. 2003, 7.2×10^{-4} ; Sugiura et al. 2001). We have also analyzed rare earth elements (REEs) abundance and oxygen isotopes ratios of this CAI. Melilite shows nearly flat (unfractionated) CI-normalized REEs pattern with anomalies in Eu and ¹⁶O-rich composition, whereas melilites in CV chondrites usually have ¹⁶O-poor composition (e. g. Clayton et al. 1977). REE pattern indicates that the large melilite in this CAI formed from a melt, thus the CAI experienced melting by reheating in the nebula. ¹⁶O-rich oxygen isotope ratios suggest that this CAI stayed nearer the Sun, while CAIs in CV chondrite stayed away from the Sun because of ¹⁶O-poor composition of melilite. Our result implies that the nebula gas near the Sun contained much more ¹⁰Be than the gas far from the Sun. This suggests that ¹⁰B was distributed in solar nebula heterogeneously and Be-B system can't measure a relative age.

Keywords: Be-B system, CAI, melilite

Internal structure of chondrules from Allende CV3 chondrite

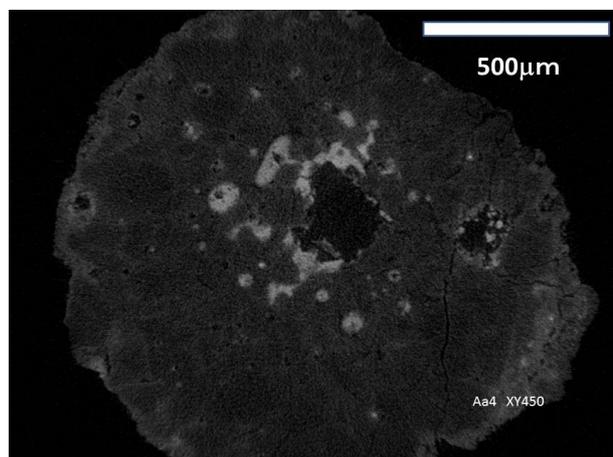
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In order to clarify 3D shapes and internal structure of chondrules in Allende CV3 chondrite, we have separated 180 chondrules grains and investigated them with X-ray CT apparatuses (Scan Xmate-D180RSS270, Scan Xmate-D160TSS105/11000) recently installed at the Museum of Natural History, Tohoku University. We also developed an optical device to measure 3D shape of chondrules or other spherical objects (Nishida et al. JPGU 2013). Our results revealed that chondrules shapes show wide distribution consisting of true spheres, prolate-spheres (rugby-ball shape) and oblate-spheres (pancake shape) (Tsuda et al. JPGU 2013). Chondrules with porphyritic textures distribute in all three shape categories. Chondrules with granular texture (lower melting degree than porphyritic) also show nearly homogeneous distribution. Chondrules with barred olivine texture (quenched from super-heated melt) show a distribution between true sphere and oblate-shape. Implication of the 3D shapes and internal texture of chondrules will be discussed from the shock-wave heating model (e.g., Miura et al., 2008).

Melting textures of Fe-FeS and silicates were observed by high-resolution X-ray CT (Scan Xmate-D160TSS105/11000). Coagulation processes of molten Fe-FeS melt in chondrules were observed. An example of cross section of a porphyritic chondrule is shown in Fig. 1, which shows "the smallest core formation process" in the early solar system. Our observation shows that 1) there are group of "chondrules" which have undergone melting of Fe-FeS only (peak temperature:1000-1200 degreeC), 2) coagulation and separation process of Fe-FeS and silicate takes place during short heating duration, 3) degassing of sulfur would be the main source of bubbles.

Keywords: Allende, Chondrule, internal texture, metal-silicate separation



Formation mechanism of fine-grained rims surrounding chondrules, CAIs and forsterite aggregates in Tagish Lake

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Introduction: Tagish Lake carbonaceous chondrite consists of two major lithologies: carbonate-rich and carbonate-poor [1]. Most chondrules and coarse-grained aggregates in Tagish Lake are surrounded by fine-grained rims [1]. The origin of fine-grained chondrule rims has been controversial [e.g. 2-7]. Nakamura et al. [6] suggested that the rims in the carbonate-rich lithology were formed during brecciation on the parent body (or bodies). In contrast, by studying the carbonate-poor lithology, Greshake et al. [7] concluded that formation of the rims by dust accretion in the solar nebula most convincingly accounts for their observations. Here we present the results of our mineralogical and petrological study of fine-grained rims surrounding chondrules and coarse-grained aggregates in the carbonate-poor lithology of Tagish Lake.

Results and Discussion: We found 87 chondrules, 14 forsterite aggregates, and two CAIs in the two thin sections (~114 microns²). These coarse-grained components are embedded in the dominant matrix (84.1 vol.%) consisting mainly of phyllosilicates with minor amounts of Fe-Mg carbonate, magnetite, forsteritic olivine, Ca carbonate, and Fe-(Ni) sulfides. Most chondrules consist of irregularly shaped cores composed of forsterite and enstatite and phyllosilicate-rich outer zones (POZs) (5-100 microns in thickness). Also, characteristic round pseudomorphs of opaque nodules that consist largely of phyllosilicates were commonly found in both core and the POZs. The observation suggests that the POZs are altered zones which were formed by replacing the peripheries of chondrules.

96 % of the chondrules are surrounded by fine-grained rims which are significantly less porous than the matrix. The volume abundances of the minerals in the rims are significantly different from those of the matrix (e.g. Ca carbonate is totally absent). Most rims contain characteristic fractures that run radially from the core/altered zone boundaries, penetrate both altered zones and rims, and terminate at the rim/matrix boundaries. All of the CAIs and forsterite aggregates are also surrounded by fine-grained rims which are identical to those of the chondrules in texture, mineralogy and chemical compositions.

We found 55 clasts that consist of materials texturally and mineralogically identical to the fine-grained rims surrounding the coarse-grained components. We also found a large clast that contains three chondrules and three forsterite aggregates. The chondrules in this clast also have altered zones. The matrix of the clast exhibits fractures that run radially from the surfaces of the chondrules and forsterite aggregates and interconnect them. They always terminate at the clast/matrix boundary.

These observations suggest that the chondrules, CAIs, forsterite aggregates, and their rims and the clasts originated from a common precursor region in the meteorite parent body that was different from the location where the host meteorite was finally lithified. That is, the rims are remnants of matrix material of the precursor region. This model is essentially consistent with those previously proposed for the carbonate-rich lithology of Tagish Lake [6] and the hydrated clasts in the Vigarano and Mokoia CV3 chondrites [4, 8].

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Keywords: Tagish Lake carbonaceous chondrite, Chondrule rims, Aqueous alteration, Brecciation

Petrography of fine-grained rim surrounding chondrule in the Acfer331 CM2 chondrite

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Introduction

Chondrules in chondrites are commonly surrounded by fine-grained rim (FGR). It is widely believed that the rims were formed by accretion of dust in the solar nebula (Metzler et al. 1992) or in the parent body (Tomeoka and Tanimura, 2000). Because of changing in textures and chemical compositions by the physical processes in the parent body, such as aqueous alteration, in order to estimate the origin of FGR, it is important to estimate the changes of petrography for the FGR corresponding to the degree of aqueous alteration. In this study, we try to estimate the effect of aqueous alteration process to study about the petrography and chemical compositions of matrix and FGRs in the area for the different degree of aqueous alteration in the Acfer331 CM2 chondrite.

Analytical methods

The samples in this study were used in thin sections of Acfer331 CM2 chondrite. Since the major minerals in the matrix of Acfer331 chondrite are clay minerals consists of hydrous minerals, by polishing operation to make thin section using water, the expansion, deformation and fracture of matrix were occurred to destroy the original texture (Oohashi et al., 2008). Therefore, we made thin sections with and without water to estimate whether the original texture was destroyed or not. In order to estimate the original texture of matrix, cutting fragment of Acfer 331 CM2 chondrite with diamond blade are also compared with those of thin sections. As results, the petrography of matrix in a thin section with water indicates to be selectively destroyed a part of FeO-rich phyllosilicates and resulting from many voids in the matrix area relative to that of thin section without water and cutting fragments. A thin section without water were adopted. Petrographic observation, X-ray elemental mapping of thin sample (12 x 14 mm), and bulk quantitative analysis of the 30 x 30 micrometer were performed by FE-SEM-EDS system (JEOL JSM-7000F + Oxford INCA Energy) at Hokkaido University.

Results and Discussion

The major minerals in FGR and matrix consist of fine-grained phyllosilicates. From X-ray mapping of whole thin sections, there are different chemical distribution for vein of MgO-rich and CaSO₄ 2H₂O in the matrix. Previous study suggests that different MgO chemical compositions of matrix are corresponding to the degree of aqueous alteration (Howard et al., 2009). Using the distribution of MgO-rich and CaSO₄ 2H₂O, two different regions are divided. And the comparison of petrography of FGRs is corresponding to these two regions.

Region (1) are without vein of CaSO₄ 2H₂O and MgO-poor. FGRs in region (1) show layered structure with inner and outer rim. In the Fe?Si+Al?Mg ternary diagram, the quantitative analyses areas in the inner rim are plotted at MgO-rich area relative to that of outer rim and matrix surrounding FGR, which are plotted the same area.

Region (2) are with vein of CaSO₄ 2H₂O and MgO-rich. FGR in region (2) shows no layered structure with inner and outer rim. In the Fe?Si+Al?Mg ternary diagram, the FGR and matrix show the similar range of chemical compositions plotted for the total variation of FGR and matrix from region (1).

The degree of aqueous alteration from matrix at region (2) seems to be higher than that of region (1) because of MgO-rich and CaSO₄ 2H₂O vein. In addition, the inner rim of FGR from region (1) shows the MgO-rich variation compared with that of matrix surrounding FGR, but FGR from region (2) shows no obvious layered structure and no different chemical variation relative to that of matrix surrounding FGR. As results, this suggests that FGR from region (2) is subject to the aqueous alteration with mm-scale in the parent body.

Keywords: aqueous alteration, fine-grained rim, carbonaceous chondrite, matrix

Hydrothermal alteration experiments of amorphous silicates in the system of MgO-SiO₂ with different Mg/Si ratios

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Meteorites have information on evolution process of materials in the early solar system. Carbonaceous chondrites are the most primitive meteorite class. Most of them, particularly CI, CM, CR chondrites, contain layer silicates, such as serpentine and saponite and have experienced aqueous alteration in their parent bodies. In order to understand evolution process of the early solar system, it is important to understand aqueous alteration process. Experimental studies to understand aqueous alteration have been performed so far. For example, aqueous alteration experiments using enstatite crystals were carried out, and conditions for serpentine and saponite formation were discussed [1]. On the other hand, it has been accepted that amorphous silicates are original building bricks of solid materials in the solar system, and thus it is important to understand aqueous alteration process of amorphous silicates.

Aqueous alteration experiments of an amorphous silicate with Fe, Ni and S free CI chondritic composition were performed [2]. In the experiments, serpentine and saponite formed easily from the amorphous silicate with pure water. However, some of layer silicates formation was not been well understood.

In the present study, aqueous alteration experiments of amorphous silicates were performed in the simple system MgO-SiO₂-H₂O to understand process of layer silicate formation. We focused serpentine (Mg₃Si₂O₅(OH)₄) and examine effect of the Mg/Si ratio of the starting material to formation of serpentine (Mg/Si=1.5).

The starting materials are nano particles of amorphous silicates (~10 nm in diameter), which were synthesized by induction thermal plasma method. Five types of starting materials with different Mg/Si ratios (1.15, 1.25, 1.50, 1.75, 2.02) were prepared. They were sealed in a Teflon vessel with pure water (water/rock ratio: 5.0) and heated at 150°C for 8, 24, 72, 168, 504 hours (pressure: 3.0 bar). Run products were examined by X-ray diffraction (XRD) for mineral identification. Some of run products were observed by field emission scanning electron microscope (FE-SEM) and transmission electron microscope (TEM) and the chemical compositions were obtained by EDX equipped in FE-SEM and TEM.

Layer silicates formed in all run products. Brucite (Mg(OH)₂) and magnesite (MgCO₃) formed in runs with the Mg/Si ratio of more than 1.5 (carbon in magnesite was probably from Teflon). Basal reflections of the layer silicates are broad. Generally, with increasing the Mg/Si ratio, the layer spacing continuously decreases, and the peak intensity increases. In runs with Mg/Si=2.02, the layer spacing decreases with increasing time. Layer silicate in 504 hr run has the layer spacing mostly close to that of serpentine, and serpentine did not easily form from the amorphous silicate with Mg/Si=1.5.

FE-SEM observation showed that run products are composed of aggregates more than a few μm in size, and signature of the original 10 nm particles disappeared. The Mg/Si ratio of run products are always less than that of the starting material. These results indicate that Mg in the starting material selectively dissolved into water, and layer silicates and brucite (and magnesite) formed from the aqueous solution.

The relation between the Mg/Si ratios of run products with the layer spacing suggest that layer silicates might be randomly interstratified mineral with serpentine and stevensite (a kind of smectite with the talc composition). In TEM observation, layer structure seemed to be observed, but more detailed observation is required for further study to confirm the layer silicate structures.

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Keywords: aqueous alteration process, carbonaceous chondrite

Experimental study on serpentine and smectite formation on chondrite

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Aqueous alteration is one of the significant processes that occurred widely in the early solar system. Indeed, several groups of carbonaceous chondrites contain abundant hydrous phyllosilicates, most of which formed by aqueous alteration of anhydrous silicates such as olivine. Mineral species of the phyllosilicates is known to be unique for groups of carbonaceous chondrite; CM and CO groups contain serpentine, while CV contains mainly smectite. In CI and CR groups, both serpentine and smectite are found. These differences should be reflected from chemical conditions on their parent body, and yet are still unknown. In the present study, we conducted the hydrothermal alteration experiments using olivines (Fo100, 80, 50, 20, 0) with various pH solutions (pH 0, 7, 10, 14) as starting materials. They were sealed into gold capsules, loaded into a test-tube-type hydrothermal apparatus, and heated at 300°C under 500 bar. Run durations are 1 week and 1 month. All recovered products were analyzed by powder X-ray diffraction (XRD), scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectrometer (EDS) and transmission electron microscope (TEM). As the results, serpentine was formed in various proportions by replacing Mg-rich olivine (Fo100, 80, 50) irrespective of pH conditions. On the other hand, smectite was formed replacing Fe-rich olivine (Fo20, 0) under alkaline condition (pH 14). Under the present conditions, we did not observe paragenesis of both serpentine and smectite, which might occur replacing Fo20~50 under alkaline conditions. EDS analyses showed that molar ratio of Fe/(Fe+Mg) in those phyllosilicates is significantly lower than that of olivine used as starting material. These results are basically consistent with previous studies [Ohnishi and Tomeoka (2007), MAPS; Zolensky et al. (1989), Icarus] where it has been suggested that aqueous alteration in the parent bodies occurred under high pH condition. The present study would provide more constraints on the aqueous-alteration conditions of the meteorites.

Keywords: carbonaceous chondrite, aqueous alteration, hydrothermal experiments, serpentine, smectite

Investigation of iron-nanoparticles in Martian meteorite olivine using electron microscopy

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1. Introduction

Olivine crystals in Martian meteorites are often dark-brown colored because they were heavily altered probably by a shock event to escape the planet. It is suggested that this change results from iron metal and magnetite nanoparticles segregated in olivine [1-3]. Mikouchi et al. [4] performed shock experiments and proposed that either iron metal or magnetite by the temperature difference during the shock event. However, the relation between the morphologies of them and shock condition is still unclear. The purpose of this study is to obtain new insights into the formation of the iron nanoparticles, through a detailed investigation of Northwest Africa 1950 shergottite which potentially includes both iron metal and magnetite nanoparticles, using advanced electron microscopic techniques.

2. Sample and Experimental methods

NWA1950 is a Iherzolitic shergottite and its dominant phases are olivine (~55wt%), pyroxenes (~35wt%) and maskelynite (~8wt%) [5].

Scanning electron microscopy (SEM), transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) were mainly used for the observation. In the SEM observation, identification of the crystallographic orientation using electron back-scatter diffraction (EBSD) was performed. The mineral species of nanoparticles observed in crushed olivine fragments and thin film specimens prepared by a focused ion beam (FIB) instrument were identified by nano-beam electron diffraction (NBED) and the measurement of lattice spacing in high-resolution TEM images. In the STEM observation, Z contrast images and composition images were acquired by the high-angle annular dark-field (HAADF) detector and STEM-EDS, respectively.

3. Results

During the observation of the crushed olivine fragments using TEM, relatively large (more than 100 nm) iron metal particles were frequently found, as well as spherical iron metal nanoparticles of a few tens of nanometers reported in the previous works. In addition, iron metal nanoparticles surrounded with magnetite-shell were discovered, although they were very rare. According to these findings, the distribution of such large iron particles was investigated by SEM. As a result, platy domains of olivine with a more iron content than the surrounding olivine matrix and Fe-abundant bright particles arranged inside the plates were observed. EBSD analysis in SEM revealed that these platy domains are parallel to several fundamental lattice planes.

Thin film specimens were prepared by FIB to investigate these domains and bright particles in detail. According to TEM/STEM observations, the bright particles observed in SEM-BSE images have a film-like form of a few tens of nanometers thick, obliquely formed in the platy olivine domains (Fig. 1). They were confirmed as iron metals by using NBED. Furthermore, very thin, thread-like iron metal particles as well as the fine spherical particles were found only outside the domains in the Z contrast images by HAADF-STEM (Fig. 1). Silica-abundant area was not found around metal iron by the composition STEM image either.

4. Conclusions

In this study, various forms of iron metal particles and their characteristic distribution in the parent olivine crystals were found by the combined observation using SEM, TEM and STEM. Moreover, an interesting structure, iron particles surrounded with magnetite-shells, was also discovered. These findings combined with further observation and analysis may be able to be applied to better understand remote sensing results and elucidation of temperature-pressure history by strong shock metamorphism.

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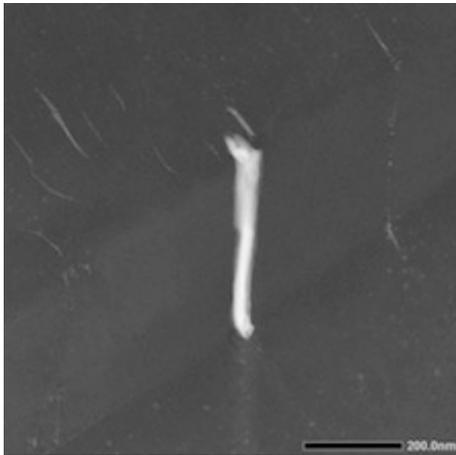
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Keywords: Martian meteorites, olivine, electron microscope, shock, nanoparticle

PPS24-P16

Room:Convention Hall

Time:May 23 18:15-19:30



Nondestructive identification of a single primitive-grain using the translational motion induced by field gradient

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Primitive chondrites are aggregates of small particles composed of different materials, and their origins are considered to be heterogeneous. It is desirable to identify the materials of individual grain before performing refined micro-probe analysis on isotopic, chemical or optical properties. The above method of identification should be non-destructive and easily performed. Moreover the method should be based on an established principle. We previously reported that a translation caused by field gradient force is induced on diamagnetic solid in common, in a condition that effect of gravity and viscous drag are negligible. A material possesses intrinsic diamagnetic susceptibility per unit mass. Therefore it is possible to identify the material of the translating particle by comparing the observed susceptibility with a list of published values. According to a Newton's equation of motional equation considered for a field gradient force, acceleration of particle was expected to be independent to mass of the particle; it is uniquely determined by the intrinsic susceptibility of material in a given field distribution. Accordingly, detection of susceptibility is possible for limitlessly small samples, and so as material identification.

It is noted that most of the particles that compose the chondrites are paramagnetic or ferro- (ferri-)magnetic materials; their size range from mm to sub-micron in diameter. In the present study field-induced ejections were newly observed for particles of diamagnetic, paramagnetic and ferri-magnetic materials; namely graphite, diamond, pyroxene and magnetite. Sample size was reduced to a level below 100 microns for the diamagnetic samples.

The field-induced motion is observable by the chamber-type drop box; the system was realized by introducing small Nd-Fe-B plates as a field generator. Using the above-mentioned box, material identification of a single grain that composes primitive materials becomes possible by a routine process that can be performed in an ordinary laboratory. The setup for observing the magnetic motion was attached inside a rectangular volume of 35x30x20 cm of a drop box. The setup was enclosed in a vacuum chamber; the sample motion was observable from the outside of the Pyrex wall of the chamber, using a high-vision video camera that had time resolutions of 0.033 s. Its spatial resolution was 0.004 cm. The pressure of the medium inside the chamber was P ? 100 Pa. Duration of microgravity inside the box was about 0.5 s, with residual gravity of 0.01G. .

Compared to the conventional methods to measure magnetic susceptibility in terrestrial gravity, the proposed principle based on the magnetic translation is free of a back ground signal of a sample holder; it is does not require a mass measurement of the sample. Hence susceptibility is obtainable for samples with a limitlessly small size, provided that translation of sample is detected. The lower limit of sample size may reach several microns by introducing an optical microscope in the compact drop box.

Keywords: magnetic ejection, nondestructive identification, microgravity, translational motion, magnetization measurement, body force

A Cleaning Method for Extraterrestrial Sample Holder

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The Hayabusa spacecraft captured particles at the surface of a near-Earth asteroid (25143)Itokawa [1]. The samples were stored in the reentry capsule of Hayabusa, and successfully brought to the Planetary Material Sample Curation Facility (PMSCF) of JAXA in 2010 [2,3,4]. They were extracted from the capsule and have been preserved in PMSCF[3,4]. Typical size of the sample is less than 100 micron, so handling of them is performed with a micro-manipulation system in the clean chamber filled with purified nitrogen [4]. Picked up samples are identified by FESEM-EDS observation, then each of them is placed on the gridded quartz glass plate in the sample holder for preservation.

Contamination control of extraterrestrial samples is essential matter, and it is important to handle and preserve samples without terrestrial contamination and alteration as possible. We hereby report the cleaning of quartz glass plates which contacts with samples directly.

The cleaning processes are performed in the clean room of PMSCF. A series of ultra sonic cleanings is a typical procedure for all materials such as metals and glasses. The first step of ultra sonic cleaning is to remove mainly organic impurities. Its solvent is typically 2-propanol. The cleaning in the solvent is repeated more than twice with 40 kHz frequency band for 20min, changing the solvent each time. The next step is to remove particles and ions. Its solvent is ultrapure water, overflowing from the ultrasonic bath to keep providing fresh water. The frequencies for the cleaning are 40, 100, and 1000 kHz bands. The cleaning is repeated twice at each frequency for 20 to 30 min. After the series of ultrasonic cleanings, water on the cleaned parts are removed by the purified nitrogen gas blow or air in clean booth.

For quartz glass, an additional acid and alkali treatment is performed after the series of ultra sonic cleanings. They are washed by heated alkali and acid solutions to remove organics, ions, and particles again. The washing method is a batch cleaning, and its procedure is our modified RCA method, originally for washing semiconductor wafer. The alkali and acid solutions are used twice respectively with ultrapure water rinse. After the cleaning and drying, the quartz glass plates is assembled to the cleaned holder or case with a cover, and stored in a desiccator filled with purified nitrogen.

The cleanness of washed quartz glass plate was evaluated with 4 methods. After our previous study [5], the detection limit was improved for metals and ions, and the analysis method was changed for organics.

1. Contaminant metals remained on the plate were extracted with 3 ml of HCl (0.35%) with H₂O₂ (0.3%), then 32 elements (B, Na, Mg, Al, K, Ca, Ti, Cr, Mn, Fe, Ni, Co, Cu, Zn, Ga, Sr, Y, Zr, Mo, Pd, Ag, Cd, In, Sn, Ba, La, Hf, Ta, W, Pt, Au, and Pb) were examined with inductively coupled plasma mass spectrometry. They were 1x10⁷ to 1x10¹⁴ atoms/cm² on the plate. Some contaminated elements are detected, but the amount of most of the elements does not affect the scientific analysis such as ion-probe measurement.

2. Contaminant ions were extracted with 60 ml of purified water, then 8 of ions (F⁻, Cl⁻, Br⁻, NO₂⁻, NO₃⁻, SO₄²⁻, PO₄³⁻ and NH₄⁺), lactic acid, acetic acid, formic acid and a few amines are examined with ion chromatography. They were less than 1x10⁻⁹ g/cm² on the plate.

3. Organics released by thermal desorption from the surface of the plate was collected to Tenax^(R) adsorbent resin. The collected organics are measured with gas chromatography mass spectrometer. Total amount of the organic carbon was less than 1x10⁻⁸ g/cm² on the plate.

4. Contaminant particles, larger than a few micron, were not observed by microscopes.

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Keywords: extraterrestrial sample, contamination control, cleaning, Hayabusa, Itokawa