

## Age-cytometry : new approach for meteorite anatomy

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The chronology for meteorite samples has played an important role to decode the early sequence of the solar system. Among the chronometers based on the radio-active decay of long-lived nuclides, because of the high time resolution of the resulting age data, the U-Pb chronometry has been widely used to define the timing of formation of refractory inclusions or to understand the formation sequence of the chondrite parental body. The high time resolution on the U-Pb chronometers was achieved by the small contribution of the analytical uncertainties in the isotope ratio measurements onto the resulting age data, and also because of the well-defined decay constants for <sup>238</sup>U, <sup>235</sup>U and <sup>232</sup>Th nuclides. Moreover, the U-Pb isotope systematics has further advantages of evaluating of system closure since the sample formation or crystallization of minerals, and therefore, reliability of the resulting age data can be rigorously tested. Using the U-Pb chronometer, the resulting time-resolution can become as small as 0.2 ? 1 Ma range for chondritic materials, but this could not be high enough to understand the planetary formation during the runaway growth or to understand the timing of the core formation. To overcome this, we are trying to measure in-situ <sup>238</sup>U/<sup>235</sup>U ratio from individual minerals by means of combination of two ion collectors. Details of the instrumentation and operational conditions would be demonstrated in this talk.

Cytometry is the quantitative analysis of cells and cell systems. Cytometry measures optical properties of cells, and most often uses fluorescence to measure specific antigen molecules, intracellular ions and DNA/RNA using antibodies, indicator dyes, or nucleic acid-specific probes. Cells may be live or fixed, depending on the application, and individual cells can often be physically sorted. ? Advantage of the cytometry are the analysis speed, detection sensitivity, the ability to measure many parameters simultaneously, and the ability to sort individual cells, and therefore, mechanism or process of elemental metabolism could be precisely evaluated based on the extensive number of cells (e.g., Benfall et al., Science, 2011; Bodenmiller et al., Nature Biotechnology, 2012). This approach can also be applied to understand the solar system evolution based on the numerous number of age data. In recent ten years, we have demonstrated the unique study approach using the distribution pattern of sample ages based on the series of precise age data collected from large number of samples (i.e., age-cytometry) [e.g., Rino et al., PEPI, 2008; Iizuka et al., Geology, 2008; Iizuka et al., Iizuka et al., Chem. Geol., 2009; Iizuka et al., GCA, 2010]. With the high-time resolution age data obtained by present analytical technique using the LA-ICPMS, further precise and quantitative discussion could be made on the solar system evolution through the age-cytometry. The newly developed high-resolution and high-throughput age determination system using a laser ablation-ICP mass spectrometry has a potential to become a significant tool to promote an age-cytometry.

Keywords: meteorite anatomy, cytometry, solar system evolution, multiple collector mass spectrometry, laser ablation-ICPMS, ICP-mass spectrometry

## $^{147}\text{Sm}$ - $^{143}\text{Nd}$ and $^{146}\text{Sm}$ - $^{142}\text{Nd}$ chronology of a basaltic eucrite, Juvinas

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Eucrites are interpreted to have originated from the asteroid 4-Vesta which differentiated into crust and mantle. The chronology of eucrites is important to understand the formation and differentiation of planets/ planetesimals in the early Solar System. The combination of two chronometers, short-lived  $^{146}\text{Sm}$ - $^{142}\text{Nd}$  system ( $T_{1/2}=6.8\times 10^7\text{yr}$ ) and long-lived  $^{147}\text{Sm}$ - $^{143}\text{Nd}$  ( $T_{1/2}=1.06\times 10^{11}\text{yr}$ ), is useful to decode the thermal history of the eucrite parent body, because they have the same closure temperature [1]. To obtain precise Sm-Nd ages for eucrites and other achondrites, it is indispensable to develop highly precise Nd isotope analysis.

We investigated the  $^{147}\text{Sm}$ - $^{143}\text{Nd}$  and  $^{146}\text{Sm}$ - $^{142}\text{Nd}$  chronometers for a brecciated basaltic eucrite, Juvinas. We examined samples of whole rock powder (W.R.), 400 mesh grains, plagioclase (Pl), and pyroxene (Px). Plagioclase and pyroxene grains were obtained by handpicking. Samples were dissolved using a mixture of concentrated pure acid ( $\text{HClO}_4$ , HF, and  $\text{HNO}_3$ ) and heating at 195 °C. About ten percent of the solution was removed and spiked with  $^{149}\text{Sm}$ - $^{145}\text{Nd}$  in order to precisely measure Sm and Nd concentrations by ID-ICP-MS (Thermo X-series II at Tokyo Tech). We separated Nd from the rest of the solution via a four-step column chemistry. Nd isotope ratios in W.R., 400mesh, Pl, and Px were analyzed by TIMS (Thermo TRITON-plus at Tokyo Tech) with the dynamic multicollection method.

The  $^{147}\text{Sm}$ - $^{143}\text{Nd}$  mineral isochron diagram of Juvinas, yielded an age of  $4610\pm 410\text{Ma}$ . In contrast, the  $^{146}\text{Sm}$ - $^{142}\text{Nd}$  systematic for Juvinas yielded an initial  $^{146}\text{Sm}/^{144}\text{Sm}$  ratio of  $0.0157\pm 0.0074$ . This gives  $4618^{+38}_{-63}\text{Ma}$  for the age of Juvinas when an initial solar system ratio of  $^{146}\text{Sm}/^{144}\text{Sm}=0.0094$  at 4568 Ma is assumed [1]. The self-consistency of the  $^{147}\text{Sm}$ - $^{143}\text{Nd}$  and  $^{146}\text{Sm}$ - $^{142}\text{Nd}$  ages for Juvinas supports early crust-mantle differentiation on the eucrite parent body as was proposed by previous chronological studies on basaltic eucrites (e.g., Pb-Pb, Al-Mg, Mn-Cr, and Hf-W; [2-5]).

It has been suggested that cumulate eucrites provide internal isochron ages younger than basaltic eucrites due to a longer history in the deep crust or late thermal disturbance during later meteorite bombardment event(s) [6]. Because of the limited Sm/Nd variation in the meteorite components analyzed, the Sm-Nd ages obtained in this study have uncertainties several times larger than those in previous studies [6]. The  $^{147}\text{Sm}$ - $^{143}\text{Nd}$  age and the initial  $^{143}\text{Nd}/^{144}\text{Nd}$  ratio for Juvinas are consistent with those obtained by the mineral isochrons of three cumulate eucrites within analytical error ( $4546\pm 8\text{Ma}$ ; [6]). However, our  $^{146}\text{Sm}$ - $^{142}\text{Nd}$  age is older than the proposed  $^{147}\text{Sm}$ - $^{143}\text{Nd}$  age for cumulate eucrites, indicating that the crystallization of basaltic eucrites predates the timing when the Sm-Nd systematics for cumulate eucrites reached a closure temperature. It should be noted that Juvinas is a brecciated basaltic eucrite which may not record a correct Sm-Nd age. Further investigation is required to obtain more precise Sm-Nd ages utilizing unbrecciated basaltic eucrite to reveal the thermal history on the eucrite parent body.

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[6] M. Boyet et al., (2010) *EPSL* 291, 172.

## Al-Mg mineral isochron of a Type C CAI from Allende

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Ca-Al-rich inclusions (CAIs) show the record of <sup>26</sup>Al, which is a short-lived radionuclide with a half-life of 0.73 Myr (e.g., MacPherson et al., 1995), thus a relative chronometer with Al-Mg systematics is applicable for determining a precise time interval of individual CAI formation process. In this work, we report full petrologic and mineralogical studies of a Type C CAI from Allende, EK1-04-2, with more detailed analyses of O isotopic distributions and Al-Mg systematics than presented by Ito et al. (2000).

FE-SEM equipped with EDS and EBSD system (JEOL JSM-7000F; Oxford X-Max 150; Oxford HKL) was used for petrologic and mineralogical studies. O and Al-Mg isotopic compositions were measured by SIMS (Cameca ims-1270).

The EK1-04-2 is a CAI fragment with a size of ~2 mm across. The CAI mainly consists of spinel, anorthite, olivine and diopside. The CAI has mantle and core structure. In the core part, euhedral spinel crystals are enclosed by other mineral grains. Anorthite and olivine grains show euhedral or subhedral shape. Diopside grains show anhedral shape and include spinel and olivine grains. Thus, crystallization sequences of core minerals are spinel, anorthite, olivine and diopside. The mantle part has a same mineral assemblage as the core part, however, amount of spinel is lower than the core part, and anhedral spinel and anorthite grains are present, and the diopside is more Mg-rich and Ti-poor comparing with core diopside.

O isotopic compositions of the minerals are distributed along CCAM line ( $\delta^{18}\text{O} = -44$  to  $+9$  permil). Spinel is <sup>16</sup>O-rich ( $\delta^{18}\text{O} \sim -43$  permil), while anorthite is <sup>16</sup>O-poor ( $\delta^{18}\text{O} \sim 9$  permil). Any differences of O isotopic compositions are not observed for these minerals among core and mantle parts while olivine and diopside show different O isotopic compositions between core and mantle parts. Olivine and diopside in the core have an intermediate O isotopic composition between spinel and anorthite ( $\delta^{18}\text{O} \sim -15$  permil). In contrast, the oxygen isotopic compositions of olivine and diopside in the mantle are not homogeneous and distributed to <sup>16</sup>O-poor direction compared with those in the core ( $\delta^{18}\text{O} = -13$  to  $-4$  permil). Olivine and diopside grains in the mantle are not in chemically equilibrium with those in the core. The O isotopic distributions among mineral grains indicate that the CAI experienced multiple and individual crystallization events: a crystallization of spinel, a crystallization of core olivine and diopside, and a crystallization of mantle olivine and diopside.

On the Al-Mg isochron diagram, spinel grains are plotted on a line of  $^{26}\text{Al}/^{27}\text{Al}_0 = (3.52 \pm 0.15) \times 10^{-5}$ , while olivine, diopside and anorthite grains of core are plotted on a line of  $^{26}\text{Al}/^{27}\text{Al}_0 = (5 \pm 5) \times 10^{-7}$ . The difference of the initial values corresponds to a relative age of ~4.6 Myr. In contrast, olivine and diopside grains in mantle are plotted below the line of olivine and diopside in core. The <sup>16</sup>O-poor and low  $\delta^{26}\text{Mg}_0$  compositions of mantle olivine and diopside suggest mixing of Al-rich chondrule-like materials. The composition of the mantle diopside is consistent with this scenario.

The petrographic, oxygen isotopic and chronological studies indicate that the CAI experienced multiple heating events after a precursor CAI formation. After ~4.6 Myr later than the precursor CAI formation, the CAI partially melted at ~1600K. The melt exchanged oxygen isotopes with the surrounding <sup>16</sup>O-poor solar nebular gas. <sup>16</sup>O-poor olivine, diopside and anorthite grains recrystallized from the partial melt. Subsequently, Al-rich chondrule-like materials accreted on the CAI and experienced partial melting and recrystallization, again. Distinctive <sup>16</sup>O-poor composition of anorthite is a result of a thermal metamorphism at the Allende parent body.

Our study revealed that the CAI has retained in the protosolar nebula at least for 4.6 Myr and experienced multiple melting events.

Keywords: Al-Mg, CAI, SIMS, oxygen isotopes, solar nebula

## Water contents and hydrogen isotopic compositions of phosphate minerals from LL4-6 ordinary chondrites.

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The origin of water on the earth is discussed by many researcher. The hydrogen isotopic compositions of earth's water were also studied to discuss the origin of water for Earth, Lunar and comet through the planetary scale in the solar system [Greenwood et al., 2011]. One of significant possible precursor of H<sub>2</sub>O in Earth's orbitary could be cometary ice but it is unclear. In preliminary results, we reported the hydrogen isotopic compositions of phosphate minerals (Merrillite and apatite) from Ensisheim LL6 ordinary chondrite (OC) on 2012 at JPGU and NIPR meeting in Japan. These D/H ratio are extremely deuterium-rich value ( $\delta D \sim 15000$ permil). In addition, Deloule and Robert (1995) also reports that the hydrogen isotopic compositions of phyllosilicate from LL3.0 Semarkona OC is D-rich ( $\sim 4000$ permil) and suggest the origin of this D-rich isotopic compositions come from the interstellar space or in the outer regions of the solar nebula, like cometary ice. These results suggest that, as the Itokawa S-type asteroids with Earth's orbitary, the heavily hydrogen isotopic compositions of LL OCs resulted from cometary ice close to the earth. However, there is no systematic study of hydrogen isotopic compositions of LL4-6 OCs because it is difficult to estimate the planetary hydrogen isotopic compositions of water due to very low water contents and contamination from adsorbed water. In this study, we applied the in-situ measurement technique of water content and hydrogen isotopic compositions of phosphate minerals from LL4-6 OCs by SIMS. All D/H ratio in the phosphate minerals are D-rich ( $\delta D \sim +2000$  to  $+25000$ ). The D/H ratio in the phosphate minerals from LL4 Soko-Banja and LL5 Tuxtuac could be resulting from degassing of H<sub>2</sub> during Fe-water oxidation reaction but it is difficult to apply it for those of LL6 Ensisheim and Bandong LL6. These results suggest that the origin of D-rich hydrogen isotopic compositions of LL6 phosphate mineral is resulting from extra-planetary with cometary ice because of extremely heavy hydrogen isotopic compositions.

Keywords: Hydrogen isotope, SIMS, phosphate, apatite

## Oxygen diffusion in perovskite with different Ca/Ti ratio

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<Introduction> Calcium-aluminum-rich inclusions (CAIs) in chondrites are believed to be the first solid formed in solar nebula and are composed of refractory minerals, such as spinel, melilite, anorthite, and perovskite (CaTiO<sub>3</sub>). Previous studies have been reported heterogeneous oxygen (O)-isotopic compositions among the CAI minerals [Clayton et al. (1973)]. The isotopic compositions are considered to be a marker of O-isotopic composition in the solar nebula [Yurimoto et al. (1998); Itoh and Yurimoto (2003); Park et al. (2012)]. Diffusion processes are important processes to affect the O-isotopic compositions of CAI minerals. To understand the effect, oxygen diffusivity of minerals should be investigated.

Perovskite crystals showed several order of larger oxygen diffusion coefficients than other CAI minerals [Gautason and Muehlenbachs (1993); Ryerson and McKeegan (1994); Sakaguchi and Haneda (1996)]. Thus, its O-isotopic compositions can provide us an important key to understand whether O-isotopic compositions of the CAI minerals have been modified or not. However, there still were a few reports on the oxygen diffusivity of perovskite, and the previously reported values are different by about one order of magnitude [Gautason and Muehlenbachs (1993); Sakaguchi and Haneda (1996)]. In this study, we focus on Ca/Ti ratio of perovskite and determined oxygen diffusion coefficients in perovskite with different Ca/Ti ratio experimentally.

<Experimental> Polycrystalline perovskite samples were prepared by a conventional sintering technique. High-purity reagent-grade powders of CaCO<sub>3</sub> and TiO<sub>2</sub> were used as the starting materials. These powders were mixed as Ca/Ti=0.098-1.002 by ball milling with PSZ balls. The powders were pressed as a sheet and were sintered at 1350 degC for 2 h in the atmosphere. Then, perovskite samples with different Ca/Ti ratio were obtained. Because of a contamination of ZrO<sub>2</sub> from PSZ balls, Ca/Ti ratio of the samples may differ by 0.001-0.0015 from initial value.

The samples were polished and finished by a mechanical polishing using the several grade of diamond pastes. To remove damages by the polishing, the samples were annealed at 1200 degC for 1h in the atmosphere. The samples were annealed under <sup>18</sup>O<sub>2</sub> gas at 750-1050 degC for several hours. Oxygen diffusion coefficients in the samples were determined using depth profiles of <sup>18</sup>O concentration of the samples obtained by secondary ion mass spectrometry (Cameca ims-4f).

<Results and discussion> Two contributions to the oxygen bulk diffusion mechanism are observed in depth profiles of <sup>18</sup>O concentration obtained from Ti-rich perovskite samples. One starts near surface (diffusion A) and the other starts from about a few 100 nm depth (diffusion B) of the sample. In Ca-rich perovskite samples, only a diffusion mechanism is observed.

We found that the oxygen bulk diffusion coefficients ( $D_b$ ) in Ca-rich perovskite samples were larger than that of Ti-rich samples, in both diffusion A and B. The  $D_b$  value of 950 degC in a perovskite sample prepared by a powder with Ca/Ti=0.098 was  $7 \times 10^{-13}$  cm<sup>2</sup>/s (diffusion A),  $1 \times 10^{-11}$  cm<sup>2</sup>/s (diffusion B), whereas the value of a sample prepared by a powder with Ca/Ti=1.002 was  $7 \times 10^{-10}$  cm<sup>2</sup>/s.

Previous reports showed <sup>16</sup>O-poor [Ito et al. (2004)] or <sup>16</sup>O-rich [Park et al. (2012)] perovskite in carbonaceous chondrites. It has been suggested that the former was changed from its original isotopic composition during thermal processes, the later was escaped the secondary processes. Larger oxygen diffusion coefficients of perovskite than that of other CAI minerals suggested that perovskite record the final thermal process, which the CAI experienced. Moreover, this study may suggest shorter timescale required for change of O-isotopic composition of perovskite than previously estimated and also indicates the importance of investigation on Ca/Ti ratio of perovskite to understand its oxygen diffusivity.

Keywords: Perovskite, Oxygen diffusion

## Newly identified hibonite-bearing FUN inclusions with low Al/Mg ratios

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It has been recognized that a minor group of CAIs named FUN (*F*ractionation and *U*nknown *N*uclear effects) and some types of hibonite (CaAl<sub>12</sub>O<sub>19</sub>) inclusions show isotopic anomalies in <sup>48</sup>Ca, <sup>50</sup>Ti, and no or small excesses in <sup>26</sup>Mg from the decay of <sup>26</sup>Al. The existence of isotopic anomalies in Ca and Ti suggests that these inclusions formed at the earliest stage of the solar system evolution, when isotopic heterogeneity still existed. The lack of <sup>26</sup>Al may also be interpreted as their formation before the homogenization of <sup>26</sup>Al distribution in the early solar system. Hence, FUN inclusions and hibonite-bearing inclusions may have significant importance in studying the earliest stage of the solar system evolution.

In order to better understand the isotopic homogenization process(es) in the early solar system, I have conducted multiple isotopic analyses of three hibonite-bearing inclusions from the Murchison (CM2) meteorite using two ion microprobes (Cameca ims-1270 & NanoSIMS 50). I identified three new hibonite-bearing FUN inclusions (MC037, MC040, and MC003), which exhibit extremely large mass-dependent fractionation in Mg (up to ~55 ‰/amu) but almost no excess in <sup>26</sup>Mg, and have resolvable isotopic anomalies in <sup>48</sup>Ca and <sup>50</sup>Ti. The results suggest that these inclusions formed during the isotopic homogenization process(es). The results of Mg isotopic compositions (extremely large isotopic fractionation) and elemental abundances (rather low Al/Mg ratios of 20-150) indicate that the precursors of these inclusions might have more Mg-rich (less refractory) compositions than the previously reported hibonite-bearing F(UN) inclusions (Al/Mg ratios from ~500 up to ~60000). In addition, their oxygen isotopic compositions are plotted on a mass-dependent fractionation line with Δ<sup>17</sup>O values of ~-23 ‰, similar to the value for the majority of typical CAIs. This suggests that oxygen isotopic compositions of their precursors are also <sup>16</sup>O-rich (δ<sup>17,18</sup>O ~-50 ‰), identical to those of typical CAIs. Furthermore, the textual signatures suggests that a molten precursor of MC040 may have been quenched. Although the origin of FUN inclusions is still not known, the present results and previous works show that there are further variations in their precursor compositions, isotopic anomalies, and thermal processes.

Keywords: FUN inclusion, hibonite, ion microprobe, Mg isotopes, Ca and Ti isotopes, oxygen isotopes

## O-16-rich olivine in igneous rim from NWA 3118 (CV)

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Ca-Al-rich inclusions (CAIs) and chondrules in chondrites show mass independent oxygen isotopic fractionation. Normally, CAIs are enriched in O-16 whereas chondrules are depleted in O-16. However, olivine grains having O-16-rich composition were reported in chondrule rims from CR2 chondrites (Takeda et al., 2002; Nagashima et al., 2013). Existence of O-16-rich grains in the rims indicates that the chondrule rims preserve information about chondrule precursor components with oxygen isotope in the chondrule formation region. In addition, abundance and distribution of O-16-rich olivine in chondrule rims have not been studied for other chondrite groups. Therefore, we study petrology and oxygen isotopic mapping of an igneous chondrule rim from a CV3 chondrite in order to reveal the distribution of O-16-rich materials.

The sample used in this study is a polished thin section from NWA 3118 CV3 chondrite. The petrographic observation and chemical analysis were performed by FE-SEM-EDS (JEOL JSM-7000F + Oxford X-Max 150). Crystal orientation analysis was studied by EBSD (Oxford HKL). Isotope mapping technique for oxygen was applied by an isotope microscope (Cameca ims-1270 + SCAPS).

The chondrule studied here has 1.4 millimeters in diameter and Mg-rich (type I) porphyritic texture mainly composed of forsterite, low-Ca pyroxene and feldspathic mesostasis. The chondrule is surrounded by rim that shows an evidence of igneous process with the thickness of up to 400 micrometers. The rim is mostly composed of ferromagnesian olivine and also contains low-Ca pyroxene, high-Ca pyroxene, Fe-Ni metal and sulfide. The Fe-rich olivine grains often show Fe-Mg zoning, suggesting that diffusional Fe-Mg exchange has occurred during metamorphism on the parent body.

In this study, seven O-16-rich olivine grains with 10-30 micrometers in diameter were found in the igneous rim. O-16-enrichments are observed in core of the olivine crystals. The oxygen isotope heterogeneity and the chemical composition are not correlated. This result suggests that these O-16-rich parts are relict and overgrown by O-16-poor olivine crystallized from melt during rim formation.

The existence of O-16-rich olivine in the rim from CV chondrite indicates that both O-16-rich and O-16-poor materials exist in the chondrule formation region.

Keywords: chondrule rim, chondrule, oxygen isotopes, carbonaceous chondrite, SIMS

## Internal structure of chondrules and their possible origin

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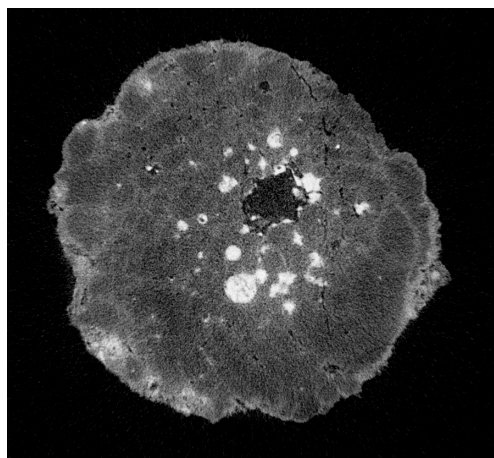
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In order to clarify 3D shapes and internal structure of chondrules in Allende chondrite, we have separated 180 chondrules grains and investigated them with X-ray CT apparatus (Scan Xmate-D180RSS270) recently installed at the Museum of Natural History, Tohoku University (Tsuda et al., JPGU 2013). 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 and oblate-spheres. Chondrules with porphyritic textures distribute in all shape categories. 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 are consistent with the shock-wave heating model (e.g., Miura & Nakamoto, 2002, 2008).

Due to the density contrast, textures of Fe-FeS phase are most easily observed by X-ray tomography. By melting, coagulation and growth of Fe-FeS phase occur in chondrules. In some chondrules, coagulation resulted in mini-core formation (driven by surface energy minimization, see Fig.1). It is suggested that duration of heating episodes could be estimated by coagulation textures of Fe-FeS. If chondrules were formed by shock heating mechanism (e.g., Miura & Nakamoto, 2002, 2008), separation of molten silicate and coagulated Fe-FeS droplets would have taken place during acceleration and slow down of chondrules. Accordingly, significant chemical fractionation took place by dynamic processes during chondrules formation.

In X-ray CT images, many chondrules are surrounded by 50-200 micron thick rims. Coagulation and growth of Fe-FeS grains are observed in chondrules-rims as well as inside of chondrules indicating that temperature may have exceeded ~1000C (eutectic point in the Fe-FeS) in the chondrule-rims. The common appearance of growth texture of Fe-FeS grains supports high-temperature origin of chondrule-rims rather than metasomatic origin. Clear textural distinction between chondrules and their ambient rims may correspond with presence or absence of melting in silicates. Most abundant element that volatilized below 1000C is sulfur. It is suggested that sulfur would have acted as a glue to coagulate dust particles onto molten droplets (chondrules) during shock heating episodes.

Keywords: meteorite, chondrule, texture, origin, X-ray CT





## Artificial cosmic spherules produced by heating and quenching experiments

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Micrometeorites (MMs) are extraterrestrial fine particles derived from asteroids and comets and continuously falling to the Earth. Depending on their velocity, mass and entry angle, micrometeorites have undergone various degrees of heating during the atmospheric entry within a few seconds. This heating lead to significant textural, mineralogical and chemical modifications to MMs. The MMs larger them 70 micron meters in diameter show variously melted textures. In particular, completely melted micrometeorites are known as cosmic spherules. Cosmic spherules have experienced large degrees of melting of primary phases during atmospheric entry, and form molten droplets. In this study, we carried out rapid heating and quenching experiments on fine particles of three kinds of meteorites (CV, CM and H chondrite) to reproduce cosmic spherules by atmospheric entry.

The run products of meteorites as starting materials show quite analogous textures to cosmic spherules including porphyritic olivine and barred olivine. The most of molten particles show spherical shape due to surface tension of the silicate melt. The outside shape of the particles is various depending on melt fraction of the particle. We successfully reproduced artificial cosmic spherules with remarkably analogous textures to natural ones. We can compare textural variations of cosmic spherules to run products and possible precursors of cosmic spherules. Analogy of the run products to cosmic spherules can be applied from textural, mineralogical and chemical modifications during atmospheric entry to estimate abundance of the interplanetary fine particles in the vicinity of the Earth's orbit.

Keywords: micrometeorite, carbonaceous chondrite, H chondrite, atmospheric heating, cosmic spherule

## Experimental reproduction of microtextures of chondrules and CAIs by reducing-gas levitation technique

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Chondrules are the most abundant component in chondrites. They are mm-sized round (or irregularly) shaped particles mainly composed of silicates, which formed by the rapid cooling of droplets of molten or partially molten rock in space before they accreted. They show unique and diverse internal micro-textures (e.g., porphyritic olivine, barred olivine, radial pyroxene, etc.), even if they have same bulk compositions. These internal textures, therefore, should reflect not only starting material compositions, but also nebular conditions, such as gas species and their partial pressures, heating and cooling rate. CAIs are another major component in chondrites, which are also known to show the evidence of molten and quenching textures. The conditions of chondrule formation, however, remain poorly constrained, because the reproduction of the chondrule formation processes in a laboratory is experimentally difficult, especially in terms of container-less arrangement and reducing (low-fO<sub>2</sub>) ambient. In the present study, we conducted gas-levitation and laser heating experiments in order to reproduce micro-textures of chondrules and CAIs, and constrain their formation conditions.

As starting materials, we used (i) natural olivine (Fo<sub>90</sub>) from San Carlos, USA, (ii) matrices of Murchison CM meteorite, and (iii) mixed compounds of melilite (Ca<sub>2</sub>Al<sub>2</sub>SiO<sub>7</sub>) + spinel (MgAl<sub>2</sub>O<sub>4</sub>) which are analogue to CAIs. The molten-quenched droplets of these samples were obtained using the gas-levitation and laser-heating experiments, which avoided both contamination of the molten samples and heterogeneous nucleation by crucible surfaces, at SPring-8 BL04B2. 1-2 mm-sized droplets were heated by a continuous-wave CO<sub>2</sub> laser, and were levitated in 96% Ar + 4% H<sub>2</sub> gas to achieve reducing ambient. The temperature during heating was monitored by a pyrometer. Surfaces and internal textures of the recovered samples were analyzed using SEM-EDX and -EBSD (JEOL, JSM-6480LAI and JSM-7100F).

(i) As a result of experiments using San Carlos olivine, olivine was re-crystallized under any condition. They show micro-porphyritic texture consisting of fine grained (1 to 5 μm) olivine, which are equal in composition to the starting material. Magnetite (Fe<sub>3</sub>O<sub>4</sub>) or hematite (Fe<sub>2</sub>O<sub>3</sub>) was completely absence, which suggests the low fO<sub>2</sub> (at least MFQ buffer) was maintained during the present experiments. (ii) The recovered sample of Murchison CM chondrite shows barred-olivine like texture. The platy (10 μm thickness) olivine crystals (Fa<sub>20-40</sub>) are embedded in a FeO-SiO<sub>2</sub>-rich glass. Idiomorphic fine (1 μm) magnetite are also observed in the glass. EBSD analyses revealed that most of the olivine plates are normal to b-axis, and the crystallographic orientations in a parallel platelet domain are identical. (iii) As a result of experiments using melilite and spinel compounds, dendritic spinel was always crystallized from surfaces to core irrespective of sample size and cooling rates, and they are embedded in Al-rich glass phase. The demonstrations of the present study show that reducing-gas levitation experiments is a powerful technique to simulate the molten-quenched texture of early solar materials.

Keywords: Chondrule formation, Molten-quenched texture, Crystal growth, Containerless heating, Barred olivine

## A unique lithology in the NWA 1232 CO3 chondrite containing clasts of different metamorphic grades

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Northwest Africa 1232 (NWA 1232) is a CO3 carbonaceous chondrite consisting of three lithologies (A, B, and C) that went through different thermal histories [1,2]. Kiriishi et al. [3] found that lithology A contains many small clasts (100-400 micron in diameter) scattering throughout the lithology. These small clasts typically consist of one chondrule surrounded by matrix and show little evidence of thermal metamorphism. Such unique lithology has not been known in other CO3 chondrites and potentially provides new insights into the formation of CO3 chondrites. Here we report the results of detailed mineralogical and petrological study of NWA 1232. The study was performed using SEM-EDS, TEM (STEM)-EDS, EPMA, and SR-XRD.

The thin section of NWA 1232 studied consists of lithologies A (738 mm<sup>2</sup>), B (624 mm<sup>2</sup>), and C (196 mm<sup>2</sup>) that are separated by sharp boundaries. In lithology C, olivine phenocrysts in type I chondrules have relatively homogeneous compositions (Fa<sub>3-13</sub>) and exhibit weak Fe-Mg zoning; these correspond to metamorphic grade 3.4. The matrix consists mainly of fine grained (100-500 nm in diameter) olivine that is relatively homogeneous in composition (~Fa<sub>60</sub>). In lithology B, olivine phenocrysts are more Fe-rich (Fa<sub>19-42</sub>) and exhibit distinct Fe-Mg zoning; these correspond to metamorphic grade 3.7. The matrix is mainly composed of relatively coarse-grained (>500 nm in diameter) olivine that is very homogeneous (~Fa<sub>40</sub>).

In contrast, chondrules and matrix in lithology A exhibit considerable chemical and textural diversities. Olivine phenocrysts in chondrules vary widely in composition from Fa<sub>1</sub> to Fa<sub>43</sub>. Most of Mg-rich olivine phenocrysts in Mg-rich chondrules show almost no Fe-Mg zoning; these are similar to those in CO 3.0 chondrites. Their surrounding matrix consists mainly of very fine-grained Mg-Si-Fe-rich amorphous material. On the other hand, most of Fe-rich phenocrysts in Fe-rich chondrules show distinct Fe-Mg zoning and their surrounding matrix consists mainly of coarse-grained (>500 nm in diameter) Fe-rich (~Fa<sub>40</sub>) olivine; these are similar to those in highly metamorphosed CO 3.7 chondrite. There are also many other chondrules whose olivine phenocrysts have compositions intermediate between the Mg-rich and Fe-rich olivines described above. The matrix surrounding these chondrules mainly consist of fine-grained (100-500 nm in diameter) Fe-rich olivine similar to those mildly metamorphosed CO chondrites.

The chemical and textural heterogeneities observed in lithology A cannot be explained by thermal metamorphism of a single lithology. The results suggest that lithology A is composed of many clasts that underwent various degrees of thermal metamorphism in different locations of the parent body. The results further imply that CO chondrites had once experienced various degrees of thermal metamorphism in different locations in the parent body and subsequently went through extensive brecciation and mixing.

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Keywords: CO chondrite, thermal metamorphism, brecciation, clast, TEM, SR-XRD

## Hydrothermal alteration experiments of the Allende meteorite

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Among the processes that have affected early cosmic materials including carbonaceous chondrites, water perhaps played the most significant role in the chemical and mineralogical evolution of a range of small asteroidal bodies, by modifying the primary mineralogical characteristics of precursor materials. The obvious effect of aqueous alteration is the formation of secondary phases, such as serpentine and smectite. The diversity in alteration assemblages among various chondrites likely reflect the aqueous environment (e.g., temperature, dissolved ion, duration, water/rock ratio,  $fO_2$ , etc.) of the parent bodies. Although several hydrothermal experiments were made on olivine or pyroxene as starting materials<sup>#1-#2</sup>, little is known about the actual behavior of chondrite toward aqueous fluid<sup>#3</sup>. Here we report hydrothermal alteration experiments of Allende meteorites.

We use Allende meteorites as starting materials. In order to observe micro-textures before and after alterations, we cut out block-shaped samples (2.5 mm×2.5 mm×6 mm) and never crushed. Hydrothermal alteration experiments were performed with PTFE double-vessels (1 ml and 25 ml) loaded into a steel autoclave. In order to maintain a reducing ambient,  $H_2$  gas was generated in the outer vessel by reaction with HCl and magnesium,. All experiments were carried out at temperature of 200 deg.C, where the saturated vapor pressure reaches about 15 bar. As to reaction fluids, we use different pH solution (7, 8.5, 10, 14)<sup>#4</sup> with different water/rock (W/R) ratios (0.5, 2, 8 vol./vol.)<sup>#5</sup>. All run durations are 168 hours. Recovered products were analyzed by synchrotron X-ray diffraction (SR-XRD), scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectrometer (EDS).

As the results, except for condition of pH 7 and W/R 0.5, serpentine is observed in matrix, which formed at interstitial space of olivine grains of the matrix. As pH value increases, more abundance of matrix olivine are replaced by serpentine. Smectite is observed only under the condition of pH 14 and W/R 0.5. Calcite ( $CaCO_3$ ) is formed on the sample surface under almost all conditions. Under pH 7 condition, anhydrite ( $CaSO_4$ ) is also formed on the sample surface. The results of this study indicate that hydrated mineral formation easily proceeds compared with the previous studies where olivine or pyroxene were used as starting materials.

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Keywords: carbonaceous chondrite, hydrothermal experiments, hydrated minerals, Allende

## Electron Holography Reveals Nanometer Scale Magnetic Structure of Framboidal Magnetite and its Formation Process

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Small solar system bodies were formed as agglomerates of dust and ices 4.6 billion years ago. Several million years after asteroid formation [1], the ice melted due to radioactive heating inside the larger asteroids [2] and/or highly energetic impacts [3]. Then, water plays several major roles in the chemistry of asteroids, both in mineralization and in the formation of organic compounds. Currently, bulk liquid water no longer exists in meteorites. We see only the signature of water in ancient asteroids as veins of hydrothermally deposited minerals [4] or water trapped in salt crystals [5] in meteorites. The Tagish Lake meteorite, which is a unique Type 2 carbonaceous chondrite, has a signature of aqueous process in the matrix that is abundant micrometer-sized polyhedral particles of magnetite [6]. The framboids are three-dimensionally ordered colloidal crystals of magnetite nanoparticles. The uniformity of the size distribution and the similar morphology of the magnetite nanoparticles in each of the colloidal crystals suggest that they were formed through homogeneous nucleation from a highly supersaturated isolated solution in a single nucleation event.

Here we show evidence of how magnetite nanoparticles assembled into periodic structures based on a nanometer scale paleomagnetic method using electron holography in an examination of the framboidal magnetite in the Tagish Lake carbonaceous chondrite [7]. An attractive force such as magnetism never contributes to the formation of colloidal crystals [8], but the repulsive force caused by the surface charge of the magnetite is able to work. To overcome the repulsive force, the density of magnetite nanoparticles in a solution must be sufficiently high in an isolated solution as a water droplet parches in microgravity. We used electron holography to visualize the magnetization of the meteoritic minerals for the first time and found that magnetite grains in the framboid have no external magnetic force, i.e., they have a flux-closure vortex structure, which allowed the formation and preservation of the colloidal crystals. We conclude that these framboids formed in tiny water droplets with pH of 7-12 containing ions such as Ca<sup>2+</sup> and Mg<sup>2+</sup> at levels of 10<sup>-14</sup>-10<sup>-15</sup> mol m<sup>-2</sup>, just before exhaustion of water during thermal alteration in a hydrous asteroid.

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Keywords: Magnetite, Electron holography, Tagish Lake meteorite

## C-XANES analyses of carbonaceous solid inclusions from Monahans halite

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Monahans meteorite (H5) contains fluid inclusion-bearing halite (NaCl) crystals [1]. Microthermometry and Raman spectroscopy showed that the fluid in the inclusions is an aqueous brine and they were trapped near 25°C [1]. Their continued presence in the halite grains requires that their incorporation into the H chondrite asteroid was post metamorphism [2]. Abundant solid inclusions are also present in the halites. The solid inclusions include abundant and widely variable organics [2]. Analyses by Raman microprobe, SEM/EDX, synchrotron X-ray diffraction and TEM reveal that these grains include macromolecular carbon similar in structure to CV3 chondrite matrix carbon, aliphatic carbon compounds, olivine (Fo<sub>99-59</sub>), high- and low-Ca pyroxene, feldspars, magnetite, sulfides, lepidocrocite, carbonates, diamond, apatite and possibly the zeolite phillipsite [3]. Here we report organic analyses of these carbonaceous residues in Monahans halite using C-, N-, and O- X-ray absorption near edge structure (XANES).

Approximately 100 nm-thick sections were extracted with a focused ion beam (FIB) at JSC from solid inclusions from Monahans halite. The sections were analyzed using the scanning transmission X-ray microscope (STXM) on beamline 5.3.2.2 at the Advanced Light Source, Lawrence Berkeley National Laboratory for XANES spectroscopy. C-XANES spectra of the solid inclusions show micrometer-scale heterogeneity, indicating that the macromolecular carbon in the inclusions have complex chemical variations. C-XANES features include 284.7 eV assigned to aromatic C=C, 288.4-288.8 eV assigned to carboxyl, and 290.6 eV assigned to carbonate. The carbonyl features obtained by C-XANES might have been caused by the FIB used in sample preparation. No specific N-XANES features are observed. Various degrees of 1s-σ\*exciton shown in the C-XANES spectra indicate that the solid inclusions contain macromolecular carbon which experienced various degree of thermal processing. The natures of the macromolecular carbon in the solid inclusions observed by C-XANES are consistent with the previous studies showing that the solid inclusions have not originated from Monahans parent body [1-3].

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Keywords: C-XANES, Halite, Carbon, Chondrite, Organic matter

### 3D shapes of Itokawa regolith particles: comparison with lunar regolith particles

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Regolith particles were returned from the surface of asteroid Itokawa by the Hayabusa spacecraft. The sample analysis elucidated a variety of surface processes on the asteroid (e.g., [1]): (1) Formation of regolith by impacts of small objects, with selective escape of the finest-scale particles. (2) Implantation of solar wind into the uppermost particle surfaces and formation of space-weathering rims. (3) Grain abrasion, probably due to seismic-induced particle motion. Processes (5) and (6) might have been repeated. (7) Final escape of particles from the asteroid by impact within the past 8 million years (1-3 million years [3]).

During the course of the analysis, 3D size and shape features of the Itokawa particles were obtained by SR-based x-ray microtomography to understand the origin and evolution of the regolith particles on Itokawa's surface [2,3]. In particular, the particle shape distribution with respect to their three-axial ratios was obtained and compared with that of fragments formed by high-speed impact in laboratory experiments [4,5] and of lunar regolith samples [6]. The 3D shapes of the lunar samples have been examined by tomography [6] but not grain-by-grain as performed for the Itokawa samples. In addition, the procedure for measuring the three axial lengths was different between the regolith particles and the impact fragments: the former was obtained from 3D external particle shapes by ovoid approximation [2,3,6], while the latter by bounding box method using a calliper [4,5]. In order to make strict comparison between them, lunar regolith particles were examined by the same method as the Itokawa particles, and the three axial lengths were measured from the tomography data by bounding box method that was newly developed in the present study.

The 3D shapes of 70 particles from 105-250  $\mu\text{m}$  sieved fraction of Descartes highland (60501) and 74 particles from <1 mm sieved fraction of Mare Tranquillitatis (10084) were obtained by microtomography at BL47XU of SPring-8. Furthermore, the 3D shapes of new 24 Itokawa particles (3 of them are from Dr. M. Meier, personal communication) were also examined in addition to the previous 48 particles [3]. The three axial lengths were measured in the orders of short to long and long to short axes to compared with the data of [3] and [4], respectively. The shape distribution in a Zingg diagram was compared using the Kolmogorov-Smirnov test.

The shape distribution of the Itokawa particles cannot be distinguished from that of the impact fragments of [4] but can be distinguished from that of [3]. This may suggest that the Itokawa particles resulted from mechanical disaggregation, as a response to impacts with a specific condition. In contrast, the shape distribution of the lunar regolith particles can be distinguished from that of the Itokawa particles and the impact fragments although lunar regolith is the product of impact on the lunar surface. The lunar particle shapes are more equant than the others. The both lunar samples examined are matured (Is/FeO = 80 and 78 for 60501 and 10084, respectively [7]). These regolith particles should become equant from the shapes similar to the impact fragments by mechanical disaggregation or abrasion due to repeated impacts during a long residence time in the regolith layer although a specific process for the shape change is not known.

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Keywords: Hayabusa mission, particle shape, SPring-8, x-ray tomography, Apollo mission, impact

## Surface micromorphology of regolith particles from Asteroid Itokawa: Implication for space weathering of regolith

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Spectral features of airless bodies are known to be modified by processes called space weathering. From Analysis of lunar samples, the space weathering is considered to be caused by mainly nano phase iron (npFe<sup>0</sup>) formed by mainly vapor deposition produced by micrometeorite bombardments and solar wind irradiation [1]. Space weathering of asteroid is also considered to progress by mainly solar wind irradiation and micrometeorite bombardments [1]. Detailed Space weathering processes on asteroids are expected to be revealed by analysis of asteroidal regolith samples. Hayabusa spacecraft recovered regolith particles from S-type asteroid 25143 Itokawa. In previous study, implantation of solar wind to Itokawa regolith particles was detected by noble gas isotope analysis [2]. The space weathering rims including npFe<sup>0</sup> were observed on regolith particles using transmission electron microscopes [3][4]. Surface micromorphology of regolith particles are expected to have information to reveal space weathering processes related to surface activity on Itokawa and applied to. So far, general surface features of regolith particles have not yet been clearly understood. Therefore, this study investigates Itokawa regolith particles focusing on their surface micromorphology.

Three-dimensional (3D) external shapes of the regolith particles were analyzed by microtomography. Surface morphologies of the regolith particles were observed by field-emission scanning electron microscopy (FE-SEM). It is revealed that the regolith surfaces can be classified into fractured surfaces formed by impact and surfaces formed by condensation from vapor in micro-druses of original chondritic materials. Regardless of these surface types, there are matured surfaces, which have rounded edges. The matured surfaces are considered to be formed by abrasion processes on Itokawa [5]. Internal structures of space weathering rim of regolith particles was observed by transmission electron microscopy (TEM/STEM) and this was compared with the surface morphologies of the same surface observed by FE-SEM. Observed space weathering rim have vesicles and form blister structures. The blisters can be identified by FE-SEM, indicating that space weathering rims with blisters can be observed by FE-SEM without any destructive methods. Space weathering processes related to the surface activities on Itokawa is considered from observation of blister distribution on the regolith particles. It is revealed that there is no correlation between blister distribution and the roundness of the surface morphologies, indicating that dominant mechanism of the abrasion process is not solar wind sputtering but mechanical abrasion. The abrasion processes can peel off the space weathering rims. In addition, there are heterogeneous distribution of blisters suggesting migration and fragmentation of regolith particles. Two distinct time-scales for the spectral reddening of S-type asteroids due to space weathering were proposed [7]: solar wind irradiation for about 10<sup>6</sup> years and micrometeorite bombardment for about 10<sup>9</sup> years. This study proposes that spectral change of Asteroid Itokawa by space weathering would have gradually occurred for 10<sup>6</sup> years at the latest by regolith activities on Asteroid Itokawa such as mechanical abrasion, migration and fragmentation of regolith particles, while space weathering rims are developed on local surface of individual regolith particles for 10<sup>3</sup>-10<sup>4</sup> years[4].

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Keywords: Itokawa, regolith, space weathering



## Extraction and Identification of Primitive Grains Driven by Magnetic Volume Force.

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Magnetic volume force caused by a field gradient has been commonly used to extract ferro- ( or ferri- ) magnetic materials from weak magnetic materials. The separation was realized because the field-gradient forces that operated on the spontaneous magnetizations of the above category of materials were considerably large with respect to terrestrial gravity. It was considered that dynamic motion of a weak magnetic material generally require a strong field above  $B = 10T$  .

Primitive materials are generally obtained as an ensemble of grains with different elemental composition with heterogeneous origins. At an initial stage of investigating this type of material, it is important to extract and identify the material of individual particles included in the ensemble. Such method should be non-destructive and easily performed. Moreover, it should be based on a well-established physical or chemical concept. In the case of analyzing a fluid sample of organic molecules, the stage separation and identification has been established by introducing the technique of chromatography; such method has not been established as yet on mixture of solid samples. Here we propose a new principle of grain separation that is driven by magnetic volume force. By comparing the measured  $\chi_{DIA}$  of a particle by their published values, material of the particle is identified. This is because an intrinsic  $\chi_{DIA}$  value is assigned to a material.

Microgravity was generated using a compact drop shaft system, which can be introduced in an ordinary laboratory. The length of the shaft was 1.8m, and the duration of microgravity time was about 0.5 second. The sample is released in the field-gradient produced by a by a magnetic circuit composed by a NdFeB permanent magnet. Maximum field intensity of the circuit was 0.8 T. The experimental apparatus was set inside a rectangle box which had a size of 30x30x20cm. The vacuum chamber equipped with an electric actuator, sample releasing signal reception device, the sample holder controller, the magnet, the battery, and the high-vision video camera are installed in the above box. [1-3]

The present results achieved on sub-millimetre-sized diamagnetic grain have a large significance as a step to realize the extraction and identification of micron-sized grains that compose the primitive materials. The technique described is useful in the search for new types of pre-solar grains that are not identified as yet.

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Keywords: magnetic ejection,, nondestructive identification, magnetic extraction, microgravity, translational motion, magnetic volume force