

## Development on the post-ionization SNMS and application for the isotopic measurement of Presolar SiC grains

SUWA, Taichi<sup>1\*</sup>; TERADA, Kentaro<sup>1</sup>; KAWAI, Yosuke<sup>1</sup>; YABUTA, Hikaru<sup>1</sup>; TOYODA, Michisato<sup>1</sup>; ISHIHARA, Morio<sup>1</sup>; AOKI, Jun<sup>1</sup>

<sup>1</sup>Graduate School of Science, Osaka University

Isotopic composition of the individual presolar grains in primitive meteorites provide us the information on nucleosynthesis in Asymptotic Giant Branch stars (AGB stars) or supernova explosion that had occurred prior to the formation of the Solar system. So far, isotopic measurement of individual presolar grains often has been carried out by using Secondary Ion Mass Spectrometry (SIMS). However, since secondary ion yield of SIMS is less than 1 %, precise analysis of minor elements in individual grains has been difficult. That is, improvement of sensitivity is highly desired for better understanding of the nucleosynthesis.

Here, we report on the development on the post-ionization Sputtered Neutral Mass Spectrometry (SNMS) and an application for the isotopic measurement of presolar silicon carbide grains (SiCs). In this analytical system, the neutrals particles sputtered by Ga ion beam of which diameter is 40nm~2 μm was post-ionized by femt-second laser, and separated by the multi-turn time-of-flight mass spectrometer 'MULTUM' depending on their masses. Last year, we has achieved the about 1000 times higher secondary ion yield for Pb signals with high mass resolution (Nakabayashi et al. 2014).

Based on the preliminary Si isotope analysis using SNMS, we confirmed that current SNMS system shows good reproducibility of terrestrial Si isotopic ratios and figured out that the instrumental mass fractionation is -150~200 ‰/amu. Moreover, we successfully separate <sup>28</sup>Si (=27.977 amu) and <sup>29</sup>Si (=28.976 amu) peaks from interference peaks such as N<sub>2</sub> (=28.006 amu), CO (=27.995 amu) and <sup>28</sup>SiH (=28.985 amu) by using MULTUM. Finally, we also confirm the heavy isotopic anomaly of Si isotopes ( $\delta^{30}\text{Si}/^{28}\text{Si}=100\sim 200\text{‰}$ ,  $\delta^{29}\text{Si}/^{28}\text{Si}=130\sim 200\text{‰}$ ) of presolar SiC candidates extracted from Murchison meteorite, which are consistent with those of previous studies.

Keywords: SIMS, presolar grain, in-situ analysis, isotope anomaly, nuclear synthesis, meteorite

## Development of precise in-situ U-Th-Pb dating of phosphate using LA-MC-ICP-MS

SAKATA, Shuhei<sup>1\*</sup>; ITOH, Shoichi<sup>1</sup>; HIRATA, Takafumi<sup>1</sup>

<sup>1</sup>Laboratory for Planetary Science, Kyoto University

Phosphates in meteorites have high concentration of U and Th (<10 ug/g) and therefore, time-resolved history for planetesimal formation has been constrained based on U-Pb systematics using phosphates in chondrites<sup>1);2)</sup>. To establish a reliable chronological scenario, in-situ dating with high spatial resolution is important because these target material could possibly experience the gain or loss of Pb through the thermal effect or shock metamorphism by impacts among meteorites<sup>3)</sup> and interaction with water. To clarify the time interval for the formation of the chondritic parent bodies, required time resolution is a few million years at least<sup>1)</sup>. However, it is difficult to achieve the enough precision to discuss the planetesimal history using in-situ dating technique. Laser ablation method (LA) is flexible sampling technique in aspects that the spatial resolution can be readily changed and the various sampling protocols are possible. Recently, the highly-precise age determination has been reported by multiple collector ICP mass spectrometry couple with LA sample introducing technique (LA-MC-ICP-MS)<sup>4)</sup>. In this study, the development of precise in-situ U-Th-Pb dating method of phosphate using LA-MC-ICP-MS is carried out for the purpose of establishment of the time-resolved story for the thermal and accretion history of the chondritic parent bodies.

Problematic points which limit the analytical precision in in-situ U-Th-Pb age determination of phosphate using laser ablation ICP mass spectrometry (LA-ICP-MS) are as followed: (1)error associated with large counting statistics derived from deficit of amount of sampling, (2)absence of age-homogeneous standard reference material of phosphate desired for precise measurement, (3)heterogeneity of Pb isotopic composition in standard reference material caused by non-radiogenic Pb which might be initially distributed in the material, (4)matrix effect associated with the difference of volatility between measured elements, (5) the isobaric interference on <sup>204</sup>Pb from <sup>204</sup>Hg. To overcome these problems, in this study, we tested the following approaches listed below: (a)enhancement of the total sensitivity through the improvement of interface region of ICP mass spectrometer and the integration of target material using multiple-spot ablation method<sup>5)</sup>, (b)evaluation of the age homogeneity of Madagascar apatite<sup>6)</sup>, (c)evaluation of the common-Pb correction for the standard reference material based on <sup>207</sup>Pb method, (d)reduction of the matrix effect by utilizing the femtosecond laser ablation system and development of the method to evaluate the system closure using three isotope diagram for Pb, (e)test of isochron method based on normalization by <sup>208</sup>Pb. To identify the suitable phase for analysis, in addition, imaging mass spectrometry for U, Th and Pb isotopes in large area (cm x cm order) by LA-ICP-MS is also tested, and the possibility of age determination using laser ablation in liquid method<sup>7)</sup> is estimated as well.

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Keywords: Parent body, Phosphate, U-Th-Pb geochronology, LA-ICP-MS

## Hydrogen diffusion experiment of apatite crystal.

ITOH, Shoichi<sup>1\*</sup> ; HASHIGUCHI, Minako<sup>2</sup> ; SAKAGUCHI, Isao<sup>2</sup> ; SAKATA, Shuhei<sup>1</sup> ; HIRATA, Takafumi<sup>1</sup>

<sup>1</sup>Department of Earth and planetary sciences, Kyoto University, <sup>2</sup>National institute for materials science

Recently many studies focused on the origin of water in the solar system and Earth with Chondrite, differentiated meteorite, Moon and Mars (e.g., Greenwood et al., 2008; Greenwood et al., 2011; Alexander et al., 2012). Especially H<sub>2</sub>O and hydrogen isotopic compositions of apatite in the Moon and ordinary chondrites has been discussed about the origin of water in the magma of the Moon and at the Earth orbit material (Greenwood et al., 2011; Yanai et al., 2014). However, it is still controversial about the origin of water in the apatite grain since it is unclear about the investigation of H-diffusivity in the apatite during thermal metamorphism in the magma and parent body. In this study, we performed H-diffusion experiments using fluorapatite.

Some slices from a durango apatite grain were used in the H-diffusion experiment. These slices polished with diamond are with mirror surface and then were annealed under D<sub>2</sub>O/O<sub>2</sub> gas at 400°C-700°C for several hours. H-diffusion coefficients in the samples were determined using depth profiles of D concentration of the samples obtained by secondary ion mass spectrometry (SIMS) (Cameca ims-4f-E7 at Kyoto university and Cameca ims-4f at NIMS). The water content of these slices of a apatite grain were measured and minor impurities of apatite were also measured using SIMS.

In this talk we will report H-diffusion coefficient in the apatite using H-diffusion experiment and discuss about investigation of H-diffusivity in the apatite.

Keywords: Hydrogen, apatite, diffusion coefficient, early solar system, SIMS

## Relationship between oxygen diffusivity and Ca/Ti ratio in perovskite

HASHIGUCHI, Minako<sup>1\*</sup> ; SAKAGUCHI, Isao<sup>1</sup>

<sup>1</sup>National Institute for Materials Science (NIMS)

<Introduction> Calcium-aluminum-rich inclusions (CAIs) in carbonaceous chondrites are composed of refractory minerals, such as mellilite, spinel, and perovskite and believed to be the first solid in the early solar system. Previous studies reported heterogeneous oxygen (O)-isotopic compositions among the CAI minerals (Clayton et al. 1973). The O-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). However, it is unclear that the heterogeneous O-isotopic composition is resulted from whether environment where the mineral crystalized or diffusion during heating event after the crystallization. To discuss this issue, investigation of O-diffusivity of minerals is necessary.

Perovskite crystals show several order of larger O-diffusion coefficients than other CAI minerals (Gautason and Muehlenbachs 1993; Ryerson and McKeegan 1994; Sakaguchi and Haneda 1996). Thus, their O-isotopic compositions can provide us an important key to understand O-diffusivity of CAI minerals and thermal histories of CAIs. Diffusivity of solids is influenced by impurity in the sample (e.g. Sakaguchi et al. 2010). In this study, we performed O-diffusion experiments perovskite with different Ca/Ti ratio to understand O-diffusivity in perovskite.

<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. Different of Ca/Ti ratio of the samples from initial value due to a contamination of ZrO<sub>2</sub> from PSZ balls was calibrated within the error of ~0.001. The samples were polished by a chemical mechanical polishing and then were annealed under <sup>18</sup>O<sub>2</sub> gas at 750-1000 degC for 20 min~several hours. O-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 (SIMS) (Cameca ims-4f). Distributions of <sup>18</sup>O ions in cross section of the samples were observed using high spatial-resolution ion image obtained by NanoSIMS (Cameca NanoSIMS 50).

<Results and discussion>Ca-rich sample and Ti-rich sample showed different O-diffusivity. In Ti-rich sample, two contributions to the O-diffusion mechanism are observed in depth profiles of <sup>18</sup>O concentration obtained from Ti-rich perovskite samples. One starts near surface (diffusion I) and the other starts from about a few 100 nm depth (diffusion II) of the sample. In Ca-rich perovskite samples, only one diffusion mechanism was observed. O-diffusion coefficients (D) in Ca-rich samples were larger than that of Ti-rich samples, in both diffusion I and II. D value of 950 degC in the Ti-rich sample (Ca/Ti=0.098) was 7x10<sup>-13</sup>cm<sup>2</sup>/s (diffusion I) and 1x10<sup>-11</sup>cm<sup>2</sup>/s (diffusion II), whereas the value of a Ca-rich sample (Ca/Ti=1.002) was 8x10<sup>-10</sup>cm<sup>2</sup>/s.

High resolution <sup>18</sup>O concentration image of Ti-rich samples showed decrease of <sup>18</sup>O concentration around the grain boundaries, indicating formation of blocking layer for O-diffusion. Such blocking layer has also been reported in BaTiO<sub>3</sub> with perovskite structure (Watanabe et al. 2014).

This study revealed difference of O-diffusivity in perovskite by slight change of Ca/Ti ratio and larger O-diffusivity of Ca-rich perovskite than Ti-rich perovskite. Larger O-diffusion coefficients of perovskite than that of other CAI minerals suggested that perovskite record the final thermal process, which the CAI experienced. This study suggests that shorter timescale required for change of O-isotopic composition for Ca-rich perovskite than previously considered. We will also discuss defect chemistry and mechanism of the O-diffusion in this presentation.

Keywords: perovskite, oxygen diffusivity

## Metamorphism of a type B CAI from the reduced CV3 chondrite Efremovka.

ENOKIDO, Yuma<sup>1\*</sup>; FAGAN, Timothy<sup>1</sup>; ARAGANE, Haruki<sup>1</sup>

<sup>1</sup>Waseda University

Calcium-aluminium-rich inclusions (CAIs) are among the first rocks to form in the solar nebula and are abundant in CV chondrites [1,2]. Most CAIs have been altered and the understanding of metamorphic effects is essential for interpreting the metamorphic conditions of parent bodies and formation conditions in the solar nebula. Especially, the CV3 chondrite Allende has undergone relatively strong metamorphism, and previous studies have addressed effects of metamorphism on Allende CAIs [3,4]. On the other hand, the CV3 chondrite Efremovka has undergone less metamorphism than Allende [5]. Therefore study of Efremovka is useful for understanding more primitive conditions. In this study, we describe secondary minerals, their textures and modal abundances in a type B CAI from Efremovka (CGI-10), using elemental mapping, quantitative EPMA, cathodoluminescence, and Raman spectroscopy. We compare secondary features of CGI-10 with Allende CAIs, particularly with the Allende type B CAI 4022 (see [3]).

CGI-10 is a type B CAI with primary spinel, anorthite and fassaite, all with 16O-rich compositions, and melilite that is 16O-poor [6]. The main secondary minerals in CGI-10 are sodalite, nepheline, Fe-bearing spinel and an elongate secondary mineral (ESM) with composition  $\text{CaAl}_2\text{Si}_2\text{O}_8$ . These secondary minerals are concentrated in FeO-alkali-rich domains near the CAI rim. Similar minerals and textures are observed in Allende CAIs [3]; however, FeO-alkali-rich domains are more abundant in Allende CAIs than in CGI-10 (approx. 1.5 mode% in CGI-10 vs. 8% in CAI 4022) and ESM is more abundant in the FeO-alkali-rich domains of Allende CAIs than in CGI-10 (trace in CGI-10 vs. 2.5 mode% in 4022). Furthermore, the main ESM in Allende appears to be dmisteinbergite [7,8], whereas the main ESM in Efremovka CGI-10 appears to be anorthite, based on the similarity of its cathodoluminescence (CL) to the CL of primary anorthite. Dmisteinbergite occurs in coal heaps in Chelyabinsk, Russia that underwent pyrometamorphism at low pressure (Earth surface), over a short time (10 to 15 years), at temperatures ranging up to 1200C and in the presence of gases of varying compositions and variable  $f(\text{O}_2)$  [9]. Some crystallization in the coal heaps apparently occurred during annealing while the heaps cooled from peak temperatures [9]. Although peak metamorphic temperatures were lower in Allende than in the Chelyabinsk coal heaps, it is possible that Allende shared some similarities in metamorphic conditions (low pressure, variable gas compositions, short heating & cooling times) that favored the formation of dmisteinbergite instead of anorthite.

In contrast with Allende CAIs [4], no grossular-rich veins were identified in CGI-10, and the mode of secondary minerals in CGI-10 (~2%) is much less than in Allende type B CAIs (~30%), consistent with the interpretation that Allende was metamorphosed at higher temperature than Efremovka [5]. On the other hand, the concentration of Na<sub>2</sub>O in CGI-10 is similar to that of a similar Allende CAI (both approx. 0.3 wt%). The similarity in Na<sub>2</sub>O-abundances suggests that infiltration of Na into CAIs during metamorphism is not due completely to metamorphic temperature, but is related to other factors (for example, composition and abundance of fluid available during metamorphism). In any case, CGI-10 has undergone some metamorphic recrystallization. Thus, when Efremovka CAIs are used to interpret conditions from the solar nebula, metamorphic effects should also be considered.

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Keywords: Ca-Al-rich inclusions, CV3 chondrites, metamorphism

## Hydro thermal experiments of Allende CV3 chondrite under reducing condition

IIKUNI, Tsuneyuki<sup>1</sup> ; SETO, Yusuke<sup>1\*</sup> ; TOMEOKA, Kazushige<sup>1</sup> ; MIYAKE, Akira<sup>2</sup> ; ICHIMURA, Shun<sup>1</sup>

<sup>1</sup>Kobe Univ. Sci., <sup>2</sup>Kyoto Univ. Sci.

### Introduction

Of a range of small asteroidal bodies, aqueous alteration is one of the most important processes affecting early cosmic materials including carbonaceous chondrites. Aqueous fluids in the chondrite parent bodies modified the primary mineralogical characteristics formed in the solar nebula, and resulted in formation of a variety of secondary phases, including phyllosilicates, magnetite and carbonates. CI and CM chondrites are widely recognized as extensively aqueous-altered meteorites. Other carbonaceous chondrites of types 3 (CR, CO, and CV) and some unequilibrated ordinary chondrites have also clear evidences of various degrees of aqueous alteration. The diversity in alteration assemblages among various chondrites likely reflect the aqueous environment (e.g., temperature, dissolved ion, water/rock ratio,  $fO_2$ , etc.) of the parent bodies. Although many mineralogical, isotopic and theoretical studies has been made to unravel the complex effects of aqueous alteration in the carbonaceous chondrites, re-productive experimental approaches using chondritic materials were not really examined. Here, in order to elucidate the actual behavior of chondrite toward aqueous fluid, we report hydrothermal alteration experiments of Allende meteorites.

### Methods

We use Allende CV3 carbonaceous chondrites as starting materials. We cut out block-shaped Allende meteorites (2.5 mm × 2.5 mm × 6 mm) by diamond blades, and never crushed ones in order to examine micro-textures before and after aqueous alteration experiments. All blocks were preliminary observed by an SEM, and blocks containing extremely large chondrules, dark inclusions and fusion clasts were not used as starting materials. Hydrothermal alteration experiments were performed with PTFE double-vessels (1 ml and 25 ml) loaded into a steel autoclave. To maintain a reducing ambient during experiments,  $H_2$  gas was generated in an outer vessel (25 ml) by reaction with HCl solution and magnesium metal, while the starting materials (Allende block and reaction solution) were set in a small (1ml) inner vessel with screw-on lid. All hydro-thermal experiments were carried out at 200 degree C and pressure of liquid-vapor saturation (~15 bar) for 168 hours. Four different pH solutions (7.0, 8.5, 10.0, 14.0) were reacted with the Allende block with three different water/rock (W/R) ratios (0.5, 2.0, 8.0 vol./vol.), and total 12 runs were performed. After the recovered samples dried for 24 hours in an oven maintained at 60 degree C, these were analyzed by a scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectrometer (EDS), synchrotron X-ray diffraction (XRD), and a transmission electron microscope (TEM).

### Results and Discussion

Most of recovered sample blocks retained their original shapes, and no brown or reddish coloration on their surface was developed. A variety of alteration phases (e.g., magnetite, anhydrite and calcite) have been precipitated on the surface, which are ubiquitously distributed regardless of substrates. SEM observations of the inner texture showed that, in all 12 runs, serpentine formed as a main secondary phase at interstitial space of olivine grains in the matrices. Smectite coexists with serpentine only in low W/R (0.5) and strong alkaline (pH 14) condition. A silica-rich amorphous material is observed in low W/R (0.5) and neutral solutions (pH 7) condition. From analyses of SR-XRD and image processing of SEM, these phyllosilicates are generally more abundant as pH value or W/R ratio increases. TEM observations show serpentine and smectite are topotaxially crystallized on the surface of olivine grains, with the crystallographic relation of (010)olivine parallel to (001)serpentine and (001)smectite. The present study indicate that i) W/R ratios affect the mineral assemblage and abundance of secondary phases and ii) hydrated mineral formation under a reducing ambient easily proceeds (even in neutral solution) compared with the previous studies.

Keywords: carbonaceous chondrite, hydrothermal experiment, Allende, phyllosilicate, serpentine

## Characterization of aqueous alteration of the C-complex asteroids by reflectance spectra of carbonaceous chondrites

SATO, Yudai<sup>1\*</sup> ; NAKAMURA, Tomoki<sup>1</sup> ; NAKATA, Airi<sup>1</sup> ; MATSUOKA, Moe<sup>1</sup>

<sup>1</sup>Department of Earth and Planetary Materials Sciences, Faculty of Science, Tohoku University

C-complex asteroids are a key object to elucidate the evolution of volatile materials in the solar system, because they are enriched in volatiles and most abundant asteroids. Therefore, we need to investigate the mineralogy and the degree of aqueous alteration of the C-complex asteroids using reflectance spectroscopy. Each absorption band observed in reflectance spectra of C-complex asteroids should be characterized and assigned based on reflectance spectra of carbonaceous chondrites which are thought to derive from the C-complex asteroids (e.g. Beck et al. 2010, Takir et al. 2013). Therefore, we need to measure reflectance spectra of hydrated carbonaceous chondrites whose mineralogy and petrology are well characterized. In this study, we measured reflectance spectra of 16 CM carbonaceous chondrites under vacuum (2hPa) using a FT-IR (Fourier Transform Infrared) spectrometer and investigated relationships between features of reflectance spectra and mineralogical properties, especially between the degree of aqueous alteration and properties of  $3\mu\text{m}$  and  $0.7\mu\text{m}$  absorption bands that are related to hydrous minerals. All CM chondrite samples were characterized in detail by synchrotron X-ray diffraction and electron microscopy (Nakata et al. 2014).

There is a weak correlation between the  $3\mu\text{m}$ -band absorption position and subtype of the meteorites that expresses the alteration degrees (Rubin et al. 2007). Samples with the absorption position at shorter wavelengths are more altered, while those with the absorption position at longer wavelengths are less altered. This trend is consistent with previous studies (Beck et al. 2010, Takir et al. 2013). However, samples with the absorption position at middle wavelengths show a wide range of alteration degrees. Therefore, it is the case that the absorption position at shortest or longest wavelengths corresponds to greatest or least alteration, respectively. In addition, Fe/Mg ratios of matrix phyllosilicates and the  $3\mu\text{m}$ -band absorption position show a weak correlation. The absorption position shifts from long to short wavelengths with decreasing Fe contents.

On the other hand,  $0.7\mu\text{m}$ -band depth shows no correlation to subtype and thus the  $0.7\mu\text{m}$ -band properties seem to have no relation to the degrees of aqueous alteration. The  $0.7\mu\text{m}$ -band depth also shows no correlation to Fe/Mg ratio of matrix phyllosilicates. Since the  $0.7\mu\text{m}$  band is attributed to  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  charge transfer in phyllosilicates, the lack of the correlation suggests no clear relation between the alteration degrees and  $\text{Fe}^{3+}/\text{Fe}^{2+}$  ratios of matrix phyllosilicates.

In this study, we investigated relationships between  $3\mu\text{m}$  and  $0.7\mu\text{m}$  band properties and the mineralogy of CM carbonaceous chondrites, and we show that it is possible to characterize to some extent the degrees of aqueous alteration of C-complex asteroids using reflectance spectroscopy.

Keywords: aqueous alteration, C-complex asteroids, reflectance spectra

## Experimental reproduction of textures of radial pyroxene chondrules by a gas-jet levitation

NOMURA, Itsuro<sup>1\*</sup>; NAKAMURA, Tomoki<sup>1</sup>; KIMURA, Yuki<sup>2</sup>; TSUKAMOTO, Katsuo<sup>3</sup>; MIURA, Hitoshi<sup>4</sup>; UESUGI, Masayuki<sup>5</sup>; UESUGI, Kentaro<sup>6</sup>; HOSHINO, Masato<sup>6</sup>

<sup>1</sup>Graduate School of Science, Tohoku University, <sup>2</sup>Institute of Low Temperature Science, Hokkaido University, <sup>3</sup>Graduate School of Engineering, Osaka University, <sup>4</sup>Graduate School of Natural Sciences, Nagoya City University, <sup>5</sup>Japan Aerospace Exploration Agency, <sup>6</sup>Japan Synchrotron Radiation Research Institute

Chondrules, igneous silicate spherules, formed by transient heating and rapid cooling, are ubiquitously contained in primitive chondrites. But, the formation conditions of chondrules in the protoplanetary disks are still unclear. In order to constrain the formation conditions of chondrules, a number of reproduction experiments in a electric furnace have been performed (e.g., Tsuchiyama et al., 1980). In these experiments, a melt droplet sample was hung on a platinum wire and thus the melt did contact with a platinum loop wire, possibly leading to heterogeneous nucleation near equilibrium temperatures. While, Tsukamoto et al. (2000) started a non-contact method, namely acoustic levitation method and microgravity levitation for the first time for chondrule synthesis, followed by Nagashima et al. (2006) who employed a gas jet levitation method. In both cases, space environment was simulated because crystallization of a melting silicate sphere occurs in a levitated condition.

In this study, we performed experiments to reproduce the textures of radial pyroxene chondrules using the gas jet levitation furnace used in Nagashima et al. (2006). Samples are 1-2mm spheres and chemical compositions of the samples are similar as of the natural radial pyroxene chondrules excluding iron. The sample was completely melted at about 1600-1800 °C with a 100W CO<sub>2</sub> laser and cooling rate after heating was about 10<sup>4</sup>-10<sup>6</sup>K / hr. We used an argon gas in order to levitate samples. After the experiment, the samples were analyzed by optical microscope and scanning electron microscope. Three dimensional images of the internal texture were also obtained after crystallization using X-ray computed tomography at SPring-8 (BL20B2).

The textures similar to natural radial pyroxene chondrules were reproduced at the cooling rates of about 10<sup>4</sup>K/hr. At faster cooling rates (10<sup>5</sup>-10<sup>6</sup>K/hr), samples became transparent glass without any crystals. These glass chondrules rarely exist in nature. The cooling rates (~10<sup>4</sup>K/hr) that successfully reproduced chondrules are slower than the calculated cooling rate (10<sup>6</sup>K/hr) of the melt whose temperature decreases by radiation alone in vacuum. When the nucleation center of the radial texture was observed by high magnification optical microscopy, there in most cases exists a tiny particle that is different from the radial pyroxene. This implies the importance of the heterogeneous nucleation from the tiny particles, which might be formed prior to the formation of the radial texture or impurities simply coming from dusts or the chemical reagent. These experimental data would lead to a discussion on the chondrule formation in the protoplanetary disk in the presence of gases that slowed down the cooling.

Keywords: chondrule, radial pyroxene chondrule, gas jet levitation



## Chondrule shapes as indicators of shock deformation in reduced vs. oxidized CV chondrites

AOKI, Ren<sup>1\*</sup>; FAGAN, Timothy<sup>1</sup>

<sup>1</sup>Waseda University

CV3 chondrites are classified into oxidized and reduced subgroups based mostly on the speciation of Fe into silicates and oxides (oxidized, CV3ox) or Fe-Ni metal (reduced, CV3red)[1,2]. Both oxidized and reduced subgroups are type 3, indicating low metamorphic grade; however, the CV3ox chondrite Allende has undergone metamorphism at higher temperatures and has experienced a greater degree of recrystallization than the CV3red chondrites Leoville and Efremovka [3]. It has been proposed that the lower extent of recrystallization in Leoville and Efremovka is due to an early impact event that deformed the CV3 parent body, causing lower porosities [4] and expelling ice from the part of the parent body where Leoville and Efremovka came from [5].

To identify evidence of deformation in the CV3 chondrites, we determined modal abundances of object types (chondrules, matrix, CAIs, etc.) and shapes of chondrules in three thin sections of CV3red chondrites (two of Efremovka and one of Leoville) and in two thin sections of CV3ox chondrites (both of Allende). Mosaics of the thin sections were prepared using the following types of images: plane polarized light and reflected light from petrographic microscope; back-scattered electron (BSE) and elemental (Na, Mg, Al, Si, P, S, K, Ca, Ti, Fe among others) maps collected by EPMA (JEOL JXA-8900 at Waseda University). Grids were overlain on the mosaic images and object types were identified at each grid node; between 1200 and 1700 points were counted on each thin section. Lengths and widths of chondrules were determined for each thin section. Ratios of (L-W)/L, where L = chondrule length and W = chondrule width, were calculated as indicators of two-dimensional chondrule shapes. As described below, distinct lengths vs. widths were identified in the CV3red chondrites, but Allende chondrules were more equant. Orientations of the chondrule lengths in the CV3red chondrites were measured and plotted in rose diagrams to evaluate similarities in chondrule orientations.

Modes show that the CV3red chondrites have lower proportions of matrix than the two CV3ox thin sections (Leoville and Efremovka matrix modes are 22-32%, compared to 34-40 mode % matrix in the two Allende thin sections) (also see [6]). Ratios of (L-W)/L tend to be near 0.1 in the chondrules from Allende, indicating circular shapes in the plane of the thin section. In contrast, (L-W)/L ratios in the Leoville and Efremovka thin sections tend to be near 0.4-0.5, indicating more elongate shapes. Chondrules in one Efremovka thin section are slightly more equant than in the other two CV3red thin sections (most [L-W]/L values 0.25-0.40 vs. 0.35-0.50), probably as a result of orientation of the thin section vs. fabric in Efremovka. Furthermore, rose diagrams of long axes of chondrules show that the chondrule long axes are clustered together along a common orientation in each thin section of Leoville and Efremovka.

Porous matrix would compress more easily than chondrules and CAIs, so the low abundances of matrix in the CV3red chondrites could be explained by compression during shock. Such compression of matrix is consistent with the low porosity of CV3red chondrites [4]. The elongation of chondrules along a common orientation also could result from shock deformation. Therefore, the modes, and chondrule shapes and orientations support the interpretation that CV3red chondrites were deformed by shock and that CV3ox chondrites were less affected by this impact event on the CV3 parent body [5].

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Keywords: CV chondrites, metamorphism, chondrules, shock deformation

## Attempt to Extract and Identify Weak-magnetic and Ferro/Ferri-magnetic Grains Included in Chondrites by Magnetic Force.

HISAYOSHI, Keiji<sup>1\*</sup> ; KUWADA, Kento<sup>1</sup> ; UYEDA, Chiaki<sup>1</sup>

<sup>1</sup>Institute of Earth and Space Science, Graduate School of Science, Osaka University

The effect of magnetic force caused by a field gradient has been commonly used to extract ferro (ferri)- magnetic materials from grain ensembles. The extraction was realized because the forces that operated on the spontaneous magnetizations of these materials were exceedingly large compared to terrestrial gravity. Chondrites are formed from ensemble of grains with different chemical composition, and the origin of grains is considered to be heterogeneous. At an initial stage of investigating this type of sample, it is desirable to extract and identify the material of individual particles included in the ensemble. Such method should be non- destructive and easily performed; it should be conducted by a well-established physical or chemical concept. In the case of analyzing a heterogeneous organic solution, the process of extraction and identification has been well established by introducing the technique of chromatography; such method has not been established as yet on a mixture of heterogeneous solid-grains.

A principle was proposed to identify the grain efficiently, which was driven by magnetic volume force[1][2]. When an ensemble of heterogeneous grains is released at a point located in a monotonously decreasing field, the grains are expected to be separated into multiple groups of ensembles as they translate toward an area of  $B = 0$ . Each ensemble is consisted by a single material, which is identified by comparing the measured  $\chi$  with a list of published values.

Here we extend the above-mentioned method on paramagnetic silicate grains that compose the chondrites by extracting and identifying the individual grains by the difference of their Fe mol %. The concentration of Fe is considered as an indicator to estimate the extent of evaporation and condensation in the course of individual grain formation. The size of particles was about 100-800  $\mu\text{m}$ . Microgravity was generated by a compact drop shaft system having length of the shaft was 1.8m; duration of microgravity time was about 0.5 second. The experimental apparatus was set inside a rectangle box which had a size of 30 x 30 x 20 cm. Interior of the box consisted of a vacuum chamber equipped with an electric actuator, a sample holder controller, a magnetic circuit, a battery, and a high-vision video camera. The compact setup was realized by introducing a magnetic-circuit that was composed of small NdFeB permanent magnets. Maximum field intensity of the circuit was 0.7 T. The result achieved here on sub-mm-sized diamagnetic grain provides a technical basis to extend the analysis on micron-sized grains that compose the chondrites. It may be applied to search new types of pre-solar grains that are not identified as yet.

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Keywords: translational motion, microgravity, magnetic separation, magnetic extraction, nondestructive identification

## A Partial Melting Study of an Ordinary Chondrite Composition with Application to the Felsic Asteroidal Crust Formation

USUI, Tomohiro<sup>1\*</sup> ; JONES, John<sup>2</sup> ; DAVID, Mittlefehldt<sup>2</sup>

<sup>1</sup>Tokyo Institute of Technology, <sup>2</sup>NASA

Melting experiments of a synthesized, alkali-bearing, H-chondrite composition were conducted at ambient pressure with three distinct oxygen fugacity conditions (IW-1, IW and IW+2). Oxygen fugacity conditions significantly influence the compositions of partial melts. Partial melts at IW-1 are distinctly enriched in SiO<sub>2</sub> relative to those of IW and IW+2 melts. The silica-enriched, reduced (IW-1) melts are characterized by high alkali contents and have silica-oversaturated compositions. In contrast, the silica-depleted, oxidized (?IW) melts, which are also enriched in alkali contents, have distinctly silica-undersaturated compositions. These experimental results suggest that alkali-rich, felsic, asteroidal crusts as represented by paired achondrites Graves Nunataks 06128 and 06129 should originate from a low-degree, relatively reduced partial melt from a parent body having near-chondritic compositions. Based on recent chronological constraints and numerical considerations as well as our experimental results, we propose that such felsic magmatism should have occurred in a parent body that is smaller in size and commenced accreting later than those highly differentiated asteroids having basaltic crusts and metallic cores.

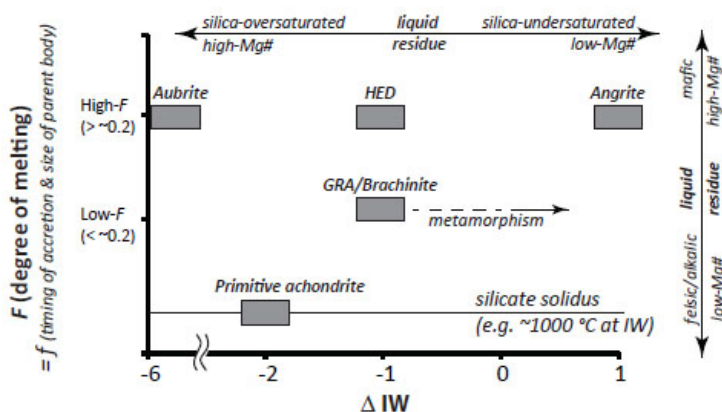
### Figure caption:

Summary of magmatic conditions of achondrite suites in terms of degree of melting and fO<sub>2</sub> (relative to IW). See [1] for details.

### Reference:

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Keywords: asteroid, crust, ordinary chondrite



## High-pressure polymorphs in Type 7 and 5 ordinary chondrites

SAITOU, Masahito<sup>1</sup> ; MIYAHARA, Masaaki<sup>1\*</sup> ; YAMAGUCHI, Akira<sup>2</sup>

<sup>1</sup>Department of Earth and Planetary Systems Science, Graduate School of Science, Hiroshima University, <sup>2</sup>NIPR

Most ordinary chondrites experienced shock metamorphism on their parent-bodies. Many kinds of high-pressure polymorphs occur in shocked ordinary chondrites, and their impact pressure conditions have been estimated based on a phase equilibrium diagram. Ordinary chondrite is classified into petrologic Type 3 to Type 7 based on the degree of thermal metamorphism. The parent-body of ordinary chondrite is expected to consist of Type 7, Type 6, Type 5, Type 4 and Type 3 from inner to outer portions based on Onion shell model. Many previous studies have tried to estimate impact pressure conditions recorded in Type 6 ordinary chondrite using high-pressure polymorphs. However, other Type ordinary chondrite such as Type 5 and 7 have been hardly investigated so far. A systematic investigation using all petrologic Types is required to reveal an impact history on the parent-body of ordinary chondrite. In this study, we tried to estimate impact pressure conditions based on high-pressure polymorphs in Type 7 and Type 5 ordinary chondrites.

Asuka 880844 H7, Asuka 880933 LL7, Yamato 790120 H7, Yamato 790960 H7, Jilin H5 and Bassikounou H5 ordinary chondrites are obtained for this study. Preliminary optical microscopic observations reveal that all samples except for Yamato 790120 H7 and Yamato 790960 H7 include shock-melt veins. So, we focused our investigations on Asuka 880844 H7, Asuka 880933 LL7, Jilin H5 and Bassikounou H5 samples. Both Type 5 and Type 7 consist mainly of olivine, Low-Ca pyroxene, plagioclase, nickel-iron alloy and troilite. Although shock-melt veins occur in all samples, high-pressure polymorphs were not identified. We expected that feldspars in and around the shock-melt veins become amorphous (maskelynite). However, Raman analyses indicate that only a few feldspar become maskelynite. It is expected that high-pressure and -temperature conditions were achieved during a dynamic event, and high-pressure polymorphs would be formed in and around the shock-melt veins once in Type 7 at that time. However, the high-pressure polymorphs would be back-transformed into their low-pressure polymorphs again because high-temperature condition was retained even if after the dynamic event. Accordingly, it is difficult to estimate shock pressure conditions recorded in Type 7 based on high-pressure polymorphs. We used only H type ordinary chondrite as a Type 5 sample. It is inferred that the parent-body of H type ordinary chondrite may be smaller in size compared to L and LL Type. The duration of high-pressure condition during a dynamic event depends on the size of an asteroid. It is likely that the duration of high-pressure condition on H type ordinary chondrite parent-body is very short so that a high-pressure polymorph could not form, implying that few high-pressure polymorphs form in H5 ordinary chondrite

Keywords: ordinary chondrite, high-pressure polymorph, Type 7, Type 5

## Effect of pressure loading path on PDFs orientation of planar deformation features (PDFs) in shocked quartz.

CHANG, Yu<sup>1\*</sup> ; TAJIKA, Eiichi<sup>2</sup> ; SEKINE, Yasuhito<sup>1</sup> ; SEKINE, Toshimori<sup>3</sup> ; KOBAYASHI, Takamichi<sup>4</sup>

<sup>1</sup>Earth & Planetary Sci., Univ. of Tokyo, <sup>2</sup>Complexity Sci. & Eng., Univ. of Tokyo, <sup>3</sup>Earth & Planetary Sci., Hiroshima Univ., <sup>4</sup>National Institute for Material Science (NIMS)

Shocked quartz is the most widely known and convincing evidence of asteroid impact. It is characterized by presence of amorphous shock metamorphic microstructures, i.e., planar deformation features (PDFs). The PDFs are detectable by the optical microscope as thin straight parallel lamellae, spacing less than 10  $\mu\text{m}$ , commonly orientated parallel to rational crystallographic planes of low Miller indices such as, (0001), {101n}, {112n}, {1122} planes. Previous shock recovery experiments show that the crystallographic orientations of PDFs (e.g., {1012}, {1013}, {1122} orientation) are formed at different shock pressure [Horz et al., 1968; Langenhorst & Deutch, 1994]. Therefore, the distribution and frequency of PDFs orientations of shocked quartz can be used to estimate average shock pressure, which is one of the most crucial parameters to constrain impact process and mechanism on the Earth and other solid bodies in the solar system.

The mechanism for PDFs formation is considered as local amorphization caused by the lattice collapse, on the shock front during shock wave passing through the quartz crystal [Goltrant et al., 1992]. The elastic instabilities in the shear modulus of specific planes occur with shock intensity. Therefore, they should be sensitive to the pressure loading path especially to the most intense first shock.

However, previous pressure calibration schemes are based on compilation of different shock experiments with different shock loading path (i.e., single shock method and reverberation method) [e.g., Stoffer & Langenhorst, 1994]. In this study, we therefore conducted a series of shock recovery experiments in order to clarify a characteristic features of PDFs for different pressure and different loading path.

The shock recovery experiments were conducted in the National Institute for Material Science (NIMS) with a one stage propellant gun. Start materials are natural and synthetic quartz crystals. The velocity range of flyer plate was 0.5 to 1.8 km/s, which produces peak shock pressure from 5 to 40 GPa for reverberation samples, and from 5 to 25 GPa for single shock samples, based on the impedance matching method. Recovered samples were mounted on epoxy resin. Orientations of PDFs were measured with 4-axis universal stage (U-stage) microscope.

In our experiments, shocked quartz grains show PDFs under the shock pressure over 10 GPa. At pressure above 30 GPa, grains are almost transformed to totally amorphous glass, but still remains their original crystal shapes (diaplectic glass). We will compare the characteristic features of PDFs orientation distribution for quartz grains shocked both by single shock and reverberation methods, and also discuss the sensitivity to the two different pressure loading paths, and revisit the previous pressure calibration schemes.

Keywords: shock metamorphism, shocked quartz, planar deformation features, shock recovery experiment

## Traces of water in lunar meteorite

KAYAMA, Masahiro<sup>1\*</sup>; TOMIOKA, Naotaka<sup>2</sup>; OHTANI, Eiji<sup>3</sup>; SETO, Yusuke<sup>1</sup>; FUKUDA, Jun-ichi<sup>4</sup>; SEKINE, Toshimori<sup>5</sup>; MIYAHARA, Masaaki<sup>5</sup>; OZAWA, Shin<sup>3</sup>; MIYAKE, Akira<sup>6</sup>; TOMEOKA, Kazushige<sup>1</sup>; FAGAN, Timothy<sup>7</sup>; NISHIDO, Hirotsugu<sup>8</sup>

<sup>1</sup>Department of Earth and Planetary Sciences, Faculty of Science, Kobe University, <sup>2</sup>Kochi Institute for Core Sample Research, Japan Agency for Marine-Earth Science and Technology, <sup>3</sup>Department of Earth and Planetary Materials Science, Graduate School of Science, Tohoku University, <sup>4</sup>Department of Geology and Geophysics, Texas A&M University, <sup>5</sup>Graduate School of Science, Hiroshima University, <sup>6</sup>Department of Geology and Mineralogy, Graduate School of Science, Kyoto University, <sup>7</sup>Department of Earth Sciences, School of Education, Waseda University, <sup>8</sup>Department of Biosphere-Geosphere Science, Okayama University of Science

Based on the Apollo mission, the lunar rocks from the Moon's surface to interior were considered to be anhydrous because of absence of hydrous minerals, lack of aqueous alteration effect evidence, and very low water contents below 1 ppb [1]. However, recent remote sensing observations, e.g. Chandrayaan-1, revealed that there exists molecular water and hydroxyl groups on the lunar surface by the refraction spectroscopy [2]. Furthermore, hydroxyl water was detected in volcanic glasses in lunar basalt at 46 ppm [3] and plagioclase in anorthosite at 5 to 6.4 ppm [4]. Previous study also founds hydroxyl-rich apatite (0.7 to 1.7 wt.%) in lunar meteorite NWA2977 [5]. These investigations had demonstrated that the water (or hydroxyl) contents on lunar surface are more abundant than expected previously, but there is still little evidence of water in the lunar interior. In this study, gabbroic and basaltic breccia lunar meteorites that have clasts interpreted as products of crystallization of late-stage magmatic residual liquids were studied using an electron microscope and spectroscopic analyses for the evaluation of their mineralogy and water contents.

Electron microscopic observation revealed that the lunar meteorites consist of gabbroic and basaltic clasts in their breccia matrix. The basaltic clasts contain quartz grains up to a several tens of micrometers in length. The quartz was crystallized above 40 MPa under a dry condition, which is corresponding to the depth below 10 km from the lunar surface [6]. It suggests that the lunar meteorites in the present study were originally formed in the lunar interior. As a result of synchrotron XRD analysis and Raman spectroscopy, the silica grains mainly consisting of moganite (monoclinic silica polymorphs) were observed in the breccia matrix, where the moganite content reaches >90 wt.% based on Raman spectroscopic methods [7-8]. These moganite grains were also present in shock veins, and they were surrounded by high-pressure polymorphs of silica, coesite and stishovite, implying that the moganite grains originally formed in the Moon, irrespective of terrestrial weathering nor back-transformation during an impact event. Terrestrial microcrystalline silica with >20 wt.% moganite content are known to be precipitated from alkaline fluid as evaporite [9]. Microcrystalline calcite also coexists with moganite in the lunar meteorites, and they possess traces of foaming in shock veins and close to fusion crusts with the melt glasses, implying their formations in the Moon. These facts suggest that a significant amount of water was present in the original magma in the lunar interior, where fluids probably assisted chemical/physical evolution of lunar inner materials. For quantitative estimation of water content in the magma, we will perform infrared spectroscopy for the constituent minerals such as olivine, pyroxene and feldspar in gabbroic and basaltic clasts of the lunar meteorites.

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## Insight into the thermochemical state of the Jovian core from ab initio thermodynamic integration MD

TANIUCHI, Takashi<sup>1\*</sup> ; TSUCHIYA, Taku<sup>1</sup>

<sup>1</sup>Geodynamics Research Center, Ehime University

The thermochemical state of the Jovian rocky core still remains unclear. Although the rocky components of the cores in gas giants are likely to be MgO, SiO<sub>2</sub> and Fe (Guillot, 1999), reported melting condition are still limited up to ~500-1500 GPa (Boates and Bonev, 2013; Millot *et al.*, 2015; Bouchet *et al.*, 2013), which are far lower than the Jovian core P,T condition (4 TPa and 20 kK) estimated based on the equations of state of gas materials determined by ab initio calculations (Nettelmann *et al.*, 2012), primarily due to experimental difficulty. Thermochemical state (e.g., solid or liquid) of these materials might influence the tidal dissipation of planets and the mixing of the core and envelope. In order to clarify the phase equilibria, we have developed a new technique for calculating free energies of liquid and crystalline states based on the thermodynamic integration method (Frenkel and Smit, 2001) combined with the ab initio molecular dynamics method.

We obtained the following new findings: (1) calculated density of the MgO-SiO<sub>2</sub>-Fe mixture is found highly consistent with the modeled Jovian core density, (2) although the melting temperatures of MgO and SiO<sub>2</sub> are higher than the modeled Jovian core temperature, the eutectic temperature is lower. These could serve for more detailed modeling of Jovian interior.

Keywords: ab initio calculation, thermodynamic integration method, Jovian core

## Investigation of organic matter in the matrix of Allende meteorite using FIB and compact STXM

SUGA, Hiroki<sup>1\*</sup> ; TAKEICHI, Yasuo<sup>2</sup> ; MIYAMOTO, Chihiro<sup>3</sup> ; MASE, Kazuhiko<sup>2</sup> ; ONO, Kanta<sup>2</sup> ; TAKAHASHI, Yoshio<sup>3</sup>

<sup>1</sup>Department of Earth and Planetary Systems Science, Hiroshima University, <sup>2</sup>Institute of Materials Structure Science, KEK, <sup>3</sup>Department of Earth and Planetary Science, The University of Tokyo

Allende meteorite (Allende), which classed as carbonaceous meteorite (CV3), fell in Pueblito de Allende, Chihuahua State, Mexico, on February 8, 1969. Allende is composed of chondrule, Calcium-aluminium-rich inclusions (CAIs), Amoeboid olivine aggregates (AOAs), Fe/Ni metal, and matrix. Organic matters (OM) is contained in matrix phase, however, distribution of OM in the Allende at the micrometer-scale has not investigated yet.

In this study, we analyzed a focused ion beam (FIB) milled thin section (approximately 150 nm) of Allende transected a region of fine-grained matrix employing carbon X-ray absorption near-edge structure (C-XANES) spectroscopy using compact scanning transmission X-ray microscope (cSTXM), which is installed to BL-13A at Photon Factory, high energy acceleration research organization. The cSTXM analysis revealed that the thin section of matrix of Allende contains c-rich grains, the C-XANES spectra were obtained from eight of these grains of which are similar to Allende's insoluble organic matter (IOM) reported by Cody et al. (2008). Some of eight c-rich grains has exciton peak of the diamond (288.6-288.9 eV). Existence of this peak indicates the possibility that nano-diamonds were contained in these grains. In addition, one of the c-rich grains has possibility of contain of C-N bond (nitrile or heterocyclic imine). As to diffuse OM, distributing like vein, whose C-XANES were carboxylic rich OM compared to grains. Diffuse OM was investigated in CM and CI chondrites by C-XANES, whose compositions were mainly labile and low molecules OM and which was carboxylic rich OM was reported by Le Guillou et al. (2014). However, C-XANES of diffuse OM, was investigated in this study, was different from already reported. This difference caused from the difference of empirical temperature between CV, CI, and CM.

From these results, OM was existed in matrix phase as small grain and diffuse OM. And cSTXM measurement was revealed that speciation of OM. Grains have IOM-like feature, and possibility of containing nano-diamonds and C-N compounds. Diffuse OM is carboxylic-rich OM. As to the future work, existence of nano-diamonds and C-N compounds should be confirmed by TEM observation and N-XANES, respectively. In addition, metamorphosis of OM between OM and inorganic minerals when thermal denaturation should be investigate using cSTXM and Raman microscope.

Keywords: Allende meteorite, Scanning Transmission X-ray Microscope, Organic matter, Focused ion beam, Carbon XANES, Characterization