

微隕石研究のための新しい分析スキームの開発と特異な鉱物学的特徴を持つ微隕石の発見

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

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

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

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

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

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

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南極雪中から回収された宇宙塵の化学組成

Chemical compositions of cosmic dust samples recovered from Antarctic snow

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

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

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

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

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

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Keywords: cosmic dust, micrometeorites, chemical composition

南極雪から回収された宇宙塵の希ガス同位体 Noble gas isotopes of micrometeorites collected from Antarctic snow

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

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

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

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

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南極とつぎ岬から回収した非溶融宇宙塵の鉱物学的研究

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

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

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

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

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

日本・ベルギー合同によるナンセン氷原での南極隕石探査

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

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これまで、セール・ロンダーネ山地方のナンセン氷原での組織的な隕石探査は JARE-29 および BELARE2010-2011 で行われているのみである。今回、JARE-54 夏隊と BELARE2012-2013 の合同チームによって、2012?2013 年シーズンにナンセン氷原での隕石探査を実施した。メンバー構成は JARE が FA1 名を含む 4 名および BELARE が FA1 名を含む 6 名であった。この結果、合計約 420 個、総重量約 76kg のあすか隕石を採集した。

自国出国は 2012 年 12 月初め、帰国は 2013 年 2 月中旬で、出入国期間は 66 日ほどであった。この期間のうち、2012 年 12 月下旬?2013 年 2 月初めの約 40 日間ナンセン氷原に滞在し、隕石探査を行った。Dronning Maud Land Air Network (DROMLAN) を利用し、航空機でベルギーの南極プリンセス・エリザベス (PE) 基地に入り、ここを起点とし、基地よりスノーモービルでナンセン氷原へアクセスした。前半 2013 年 1 月中旬まではナンセン氷原南西部エリア域の探査を行った。後半 1 月中旬以降はキャンプ地を移動し、東北部エリア域での探査を実施した。この間の気温は -15°C ? -30°C の間で推移し、多くの日に風速毎秒 10 m 以上のカタバ風が東より吹いた。キャンプには JARE は居住用モジュール、BELARE はコンテナを用いた。モジュール、コンテナ、および燃料槽の移動は雪上車で PE 基地より支援いただいた。

探査は、スノーモービル (Ski-doo) 10 台を用い、移動速度は時速 10 km 以下で、先頭を 1 名の FA が先導する V 字形状による方法を行った。各自が携帯 GPS のナビゲーション機能を活用し、隕石探査域の記録には航跡機能、隕石発見地点の記録には位置入力機能を利用した。今後、これらのデータの整理を行い、隕石の分布と重量の詳しい解析を行う予定である。ベースキャンプ地を移動直後より 12 日間連続の悪天候による停滞があるなど期間を通して約半分の期間が停滞であったが、当初の予想を上回り、多くの大きい隕石を採集することができた。最初のキャンプ地では約 15 日探査が実施でき、約 6.5kg の隕石を筆頭に約 360 個の隕石を回収した。キャンプ地の移動後は探査を実施出来たのは 5 日ほどであったが、約 60 個の隕石を採集した。これら合計約 420 個の隕石の多くは、コンドライトであり、この中に炭素質コンドライトや今回の探査では最大の 18 kg のコンドライトを含んでいる。また、エコンドライトも多く見つかった。今後、キュレーション作業は、国立極地研究所南極隕石ラボラトリにおいて行うことになる。なお、東南部エリアを中心に未探査エリアが残っており、近いうちにこの地域の探査を計画予定である。

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二次イオン質量分析法による流体包有物の同位体分析法の開発 I

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

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流体は惑星表層及び深部において元素移動や元素循環の媒体として働く主要な要素であり、流体の作用により地質現象がコントロールされる事も多い。地質現象を引き起こした過去の流体についてはそのとき生成した岩石の記録から間接的に推測されていた。しかしながら、過去の流体はしばしば鉱物中の流体包有物としてトラップされているので、もし流体包有物の分析ができれば過去の流体についての情報を直接得る事が可能になる。しかも、個々の流体包有物の鉱物内の位置関係は地質時間の経過と対応しているので、もし流体包有物を個別に分析できれば、一連の地質作用の間の流体の進化についての情報を得ることができる。特に、同位体分析は流体の起源についての有益な情報を与える。

従来の質量分析法では流体包有物の測定は抽出法により実施されており、この方法は1mm以上の大きな流体包有物まで測定可能であるので、適用対象が限定されていた。岩石中では流体包有物の大きさは数から数十ミクロンのものが多いのでこの微小な流体包有物に適用可能な質量分析法の開発が重要である。また、その場分析が可能になれば、流体進化についての研究も飛躍的に進展すると考えられる。このような空間分解能を持つ質量分析法として二次イオン質量分析法(SIMS)が考えられる。しかしながらSIMSでは試料を真空中に露出する必要があるため、流体の分析は不可能である、本研究では、流体包有物のSIMS分析を行う事を目標とし、SIMS分析用試料調製法の開発研究を行った。

SIMS分析を安定に行うためには以下の試料条件を整える必要がある:(1)表面が平滑な鏡面平面であること(2)流体包有物が研磨面上に露出していること(3)試料表面が導電性を有すること。

条件(1)(2)を満足させるため、流体包有物を凍結させた状態で研磨ができる冷凍研磨機を開発した。冷凍研磨機は液体窒素浴を内蔵する研磨盤を有し、研磨中盤上は-100℃で温度管理可能である。常温から-100℃に約14分で到達する。この研磨盤上にアルミナ研磨シートを置き、流体包有物の乾燥冷凍研磨を可能にした。

研磨面への大気中の水蒸気凝縮を防止し、流体包有物露出面を導電状態に加工する(条件(3))ため窒素雰囲気グローブボックスを整備し、グローブボックス中に冷凍研磨機とイオンコーター装置(サンヨー電子SC-701AT)を設置した。イオンコーター試料台は冷却状態で使用でき、金薄膜作成中に試料を-100℃以下に保持した状態で研磨面に直接金薄膜を形成でき、条件(3)を満足する。また、研磨面の研磨状態を観察できる冷却試料台を有した反射顕微鏡も同時に設置した。現在の問題点は、凍結流体の試料面露出状態の簡便な観察判定法の確立であり、試行錯誤をくり返している。

キーワード: 流体包有物, 二次イオン質量分析法, 冷凍研磨, 同位体

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

LL5-6 普通コンドライト隕石中のリン酸塩鉱物の岩石学的研究, H₂O 含有量及び水素同位体組成

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

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

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

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

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

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

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

キーワード: アパタイト, 水素同位体, LL 普通コンドライト, リン酸塩鉱物

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

アエンデ隕石 CAI のメリライト結晶の高精度酸素同位体比イメージングによる太陽系星雲ガスの酸素同位体組成の推定 Oxygen isotopic composition of the solar nebula gas inferred from high-precision isotope imaging of melilite crystals

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

キーワード: コンドライト, メリライト, CAI, SIMS, 酸素同位体イメージング, 太陽系星雲ガス
Keywords: Chondrite, Melilite, CAI, SIMS, Oxygen isotope imaging, Solar nebula gas

Fluffy type A CAI 中メリライト結晶の酸素同位体ゾーニングと結晶成長の関係 Relationship between oxygen isotope zoning and crystal growth in melilite crystals from fluffy type A CAI

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

キーワード: FTA, SIMS, oxygen isotope, CAI

Keywords: FTA, SIMS, oxygen isotope, CAI

Be-B 法を用いた melilite に富む CAI の年代決定の試み

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

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

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

キーワード: Be-B 法, CAI, メリライト

Keywords: Be-B system, CAI, melilite

アエンデ隕石中のコンドリュールの内部組織 Internal structure of chondrules from Allende CV3 chondrite

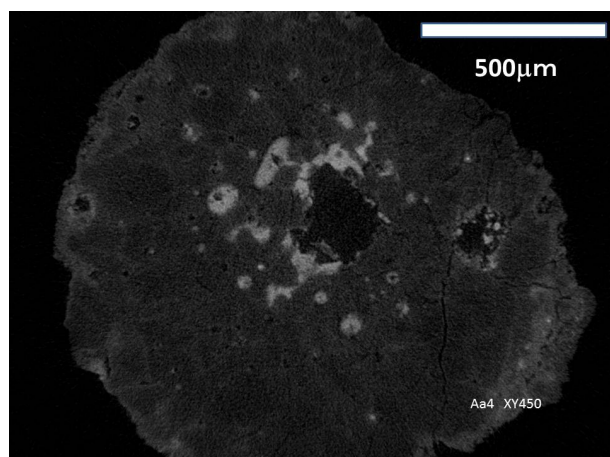
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In order to clarify 3D shapes and internal structure of chondrules in Allende CV3 chondrite, we have separated 180 chondrules grains and investigated them with X-ray CT apparatuses (Scan Xmate-D180RSS270, Scan Xmate-D160TSS105/11000) recently installed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

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

キーワード: アエンデ隕石, コンドリュール, 内部組織, 金属とシリケートの分離
Keywords: Allende, Chondrule, internal texture, metal-silicate separation



Tagish Lake 炭素質隕石中のコンドリュール、CAIs, forsterite aggregates を囲む細粒リムの形成メカニズム Formation mechanism of fine-grained rims surrounding chondrules, CAIs and forsterite aggregates in Tagish Lake

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

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

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

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

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

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キーワード: Tagish Lake 炭素質隕石, コンドリュール・リム, 水質変成, 角礫岩化

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

Acfer331 CM2 コンドライト隕石のコンドルールを覆う細粒リムの岩石学的記載 Petrography of fine-grained rim surrounding chondrule in the Acfer331 CM2 chondrite

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(イントロダクション)

コンドライト隕石中のコンドルールは、一般的に細粒リムによって囲まれている。細粒リムは星雲中のダストの付着成長によって形成された (Metzler et al. 1992), あるいは母天体内の変質作用により形成された (Tomeoka and Tanimura, 2000) と考えられている。どちらの起源であってもこの細粒リムは、水質変質によって組織や化学組成が変化してしまうため、その起源を明らかにするためには、水質変質の程度の違いにより、細粒リムにどのような変化が起こるのかを評価することが重要である。そこで、本研究では、Acfer 331 隕石の水質変質の異なる領域においてマトリクスと細粒リムの岩石組織学・化学組成学的研究を行い水質変質作用の影響を評価した。

(実験方法)

本研究での実験試料は、Acfer331 CM 2 コンドライト隕石を用い、薄片試料を作成した。マトリクス部の主要構成鉱物は、含水鉱物である粘土鉱物であるため、水を用いた研磨作業により、膨張、変形、破壊が起こり、元の組織が変化する可能性がある (Oohashi et al., 2008)。そのため、水を使用した研磨試料、水を使用しない研磨試料を作成して、組織や化学組成の違いを評価し、薄片作成方法を検討した。ダイヤモンドブレードで切り出しただけの試料と比較したところ、元々マトリクス部には、空孔は存在せず、水を使用した研磨試料では選択的に Fe に富む粘土鉱物が破壊され、組織が変化している可能性が高いと評価された。そのため、水を使用しない研磨方法を採用し、岩石学的研究を行った。岩石学的観察には、北海道大学設置の FE-SEM (JEOL JSM-7000 + EDS 検出器 (Oxford INCA Energy)) を用いて、隕石薄片全体 (12 x 14mm) の X 線元素マッピング及び微小領域の 30 x 30 ミクロンのパルク定量分析を行い、水質変質の程度及び元素組成の比較を行った。

(結果と議論)

マトリクス部と細粒リム部の主要構成鉱物は、どちらも細粒な層状ケイ酸塩で構成されていた。隕石薄片全体の X 線元素マッピングにより、MgO の化学組成が異なる領域や CaSO₄·2H₂O の脈を示す領域が観察された。この化学組成の異なる特徴を示す領域は、これまでの研究により、水質変質の程度が異なることが考えられている (Howard et al., 2009)。この MgO に富んだ分布及び CaSO₄·2H₂O の分布を示す領域に注目し、水質変質の程度の異なる 2 つの領域を評価し、細粒リムを持つコンドルールの岩石学的記載をこれら 2 つの領域に対応させ比較を行った。

領域 (1) は、CaSO₄·2H₂O や MgO に富んだ脈のない領域とした。領域 (1) に存在する細粒リムは、inner rim と outer rim に分かれた層構造になっていた。Fe-Si-Al-Mg 三角ダイアグラムにおいて inner rim は、MgO に富む組成範囲にプロットされ、それと比較し、outer rim とリム周辺部のマトリクス物質は、MgO に乏しい組成範囲にプロットされた。outer rim とリム周辺部のマトリクス物質は、同じ組成範囲にプロットされ、組成の違いはみられなかった。

領域 (2) は、CaSO₄·2H₂O や MgO に富んだ脈のある領域とした。細粒リムは、領域 (1) でみられたような inner rim と outer rim の層構造を示さなかった。Fe-Si-Al-Mg 三角ダイアグラムにおいて細粒リムとマトリクスは、どちらも同じような化学組成範囲を示し、その組成範囲は、領域 (1) の細粒リムやマトリクスでみられた化学組成範囲をあわせもつ範囲にプロットされた。

領域 (2) のマトリクス部は、CaSO₄·2H₂O の脈があり、MgO に富む特徴を示したことから、領域 (1) と比較して、水質変質度が高いと考えられる。また、領域 (1) の inner rim の細粒リムは、周囲のマトリクス部と比較して MgO に富む特徴を示したが、領域 (2) の細粒リムでは、水質変質を強く受けていると考えられるマトリクスと同じ化学組成範囲となり、層構造を示さず、変質の影響により化学組成が周りのマトリクスと同じような分布となっていることを示唆した。以上より、領域 (2) の細粒リムや周囲のマトリクスで観察された岩石学的組織の変化は、母天体内部での局所的な mm-scale の水質変質の影響によるかもしれない。

キーワード: 水質変質, 細粒リム, 炭素質コンドライト, マトリクス

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

Mg/Si 比を変えた MgO-SiO₂ 系非晶質珪酸塩の水質変成実験Hydrothermal alteration experiments of amorphous silicates in the system of MgO-SiO₂ with different Mg/Si ratios高橋 竜平^{1*}, 土山 明¹, 松野 淳也¹, 瀬戸 雄介²Ryohei Takahashi^{1*}, Akira Tsuchiyama¹, Junya Matsuno¹, Yusuke Seto²¹ 京都大学大学院理学研究科地球惑星科学専攻, ² 神戸大学大学院理学研究科¹Division of Earth and Planetary Sciences, Graduate School of Science, Kyoto University, ²Graduate School of Science, Kobe University

隕石は太陽系初期の物質進化過程を記録しており、太陽系の初期進化を理解するにあたって重要な物質である。炭素質コンドライトは、その化学組成が揮発性元素を除いた太陽大気のスเปクトル分析から得られた化学組成と類似するため、これらの中でも最も始原的であると考えられている。多くの炭素質コンドライトは serpentine, saponite などの層状珪酸塩や炭酸塩鉱物を含み、母天体での水質変成を受けていると考えられる。とくに、CI、CM、CR コンドライトは強く水質変成を受けている。このような水質変成過程を理解することは、太陽系の物質進化過程を理解する上で重要である。このため、水質変成を模擬した実験的研究が行われている。たとえば Ohnishi and Tomeoka [1] は enstatite 結晶を出発物質として水質変成実験を行い、serpentine や saponite の生成条件を議論した。一方、太陽系を作った固体原材料物質は非晶質珪酸塩であると考えられ、非晶質珪酸塩の水質変成作用を理解することが重要である。

Noguchi et al. [2] は CI コンドライト組成から Fe, Ni, S を除いた組成をもつ非晶質珪酸塩を出発物質として用いて、水質変成実験を行い、serpentine, saponite などの鉱物が反応性の高い非晶質物質と純水との反応で容易に生成することを明らかにした。しかしながら、反応初期に生成した層状珪酸塩は未同定であり、層状珪酸塩の生成プロセスもよくわかっていない。

本研究では非晶質珪酸塩からの層状珪酸塩の生成プロセスを理解するために、MgO-SiO₂-H₂O 系の単純系において水質変成実験を行った。とくに炭素質コンドライトの主要な水質変成鉱物である serpentine (Mg₃Si₂O₅(OH)₄) に着目し、出発物質である非晶質珪酸塩の Mg/Si 比の違いが serpentine の生成にどのような影響を及ぼすのかを考察した。

出発物質として用いた非晶質珪酸塩は熱プラズマ法で合成した粒径約 10nm の微粒子である。Mg/Si = 1.15, 1.25, 1.50, 1.75, 2.02 の 5 種類の組成のものに純水を加え (Water/Rock 比 (重量比) は 5.0)、密封したテフロン容器内で 150°C において 8, 24, 72, 168, 504 時間加熱した (圧力: 3.0 bar)。実験生成物は粉末 X 線回折法 (XRD) により鉱物同定を行い、電界放出型走査型顕微鏡 (FE-SEM) および透過型電子顕微鏡 (TEM) 観察と EDX による組成分析を行った。

すべての実験で層状珪酸塩が生成した。Mg/Si 比が 1.5 より大きいもの (Mg/Si=1.75, 2.02) では、168 時間以上で brucite (Mg(OH)₂) または magnesite (MgCO₃) が生成した (magnesite 中の C は容器として用いたテフロンに由来すると考えられる)。層状珪酸塩の底面反射はブロードであり、一般には出発物質の Mg/Si 比の値が大きくなるほど底面層間隔は連続的に短くなり (0.77-1.26nm)、また反射ピーク強度は大きくなった。Mg/Si=2.02 の組成では、実験時間の増加に従い層間隔が減少し、504 時間の実験では最も serpentine に近い層間隔を持つものが得られた。従って、serpentine と同じ Mg/Si 比 (1.5) を持つ非晶質珪酸塩から serpentine が生成しやすいわけではないことが分かった。

FE-SEM 観察では、実験生成物は数ミクロン以上の塊状物質になっており 10nm 粒子の痕跡は認められなかった。実験生成物の Mg/Si 比は出発物質より小さくなっており、Mg が選択的に水に溶出し、水溶液から層状珪酸塩や brucite (magnesite) の析出が起こったと考えられる。

実験生成物の Mg/Si 比と実験生成物の層間隔を考慮すると、実験生成物の層状珪酸塩は serpentine と stevensite (3 八面体のスメクタイトの一種) の不規則混合層鉱物になっている可能性が考えられる。TEM 観察では層状構造をもつものが観察されるがその詳細な構造は今後の課題である。

[1] Ohnishi and Tomeoka (2007) Meteoritics & Planetary Science, 42, 49-61. [2] Noguchi (2010) JpGU Meeting, PPS009-10.

キーワード: 水質変成過程, 炭素質コンドライト

Keywords: aqueous alteration process, carbonaceous chondrite

コンドライト母天体におけるサーペンティン、スメクタイトの生成条件の推定 Experimental study on serpentine and smectite formation on chondrite

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

キーワード: 炭素質コンドライト, 水質変成, 熱水変成実験, サーペンティン, スメクタイト

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

火星隕石中カンラン石の電子顕微鏡観察による鉄ナノ粒子観察 Investigation of iron-nanoparticles in Martian meteorite olivine using electron microscopy

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1. はじめに

火星隕石, 特にシャーゴットタイトに含まれるカンラン石は強い衝撃作用により黒色化しており, また吸収スペクトルや磁化率が元のカンラン石とは異なることが知られている. これらの変化の原因はカンラン石中に晶出した平均 20nm 程度の大きさの鉄ナノ粒子であることが指摘されている [1,2]. また, Van de Moortele et al. [2] の磁化率測定によると, 鉄ナノ粒子の鉱物種が金属鉄だけでなく磁鉄鉱の存在が示唆され, Kurihara et al. [3] は観察で実際に磁鉄鉱が観察されたことを報告した. また, 三河内他 [4] は衝撃実験によりその選択が上昇温度の違いによって起こることを示した. しかし, これらの鉄ナノ粒子の形成過程や衝撃圧・温度との関係は詳しくわかっていない. 本研究は金属鉄と磁鉄鉱の 2 種類の鉄ナノ粒子が共存すると言われているレールゾライト質シャーゴットタイト Northwest Africa 1950 (NWA1950) 隕石を, 最新の電子顕微鏡手法により詳細に調べることで, この問題に関して新たな知見を得ることを目的とした.

2. 試料・手法

今回用いた試料は NWA1950 隕石であり, 主要構成鉱物はカンラン石 (?55wt %) , 輝石 (?35wt %) , マスケリナイト (?8wt %) である [5]. カンラン石は黒色化しているが, その色づきにはそれぞれの粒子内でもばらつきがある.

観察には走査型電子顕微鏡 (SEM) , 透過型電子顕微鏡 (TEM) , 走査透過型電子顕微鏡 (STEM) を主に用いた. SEM の観察では電子線後方散乱回折 (EBSD) を用いた結晶方位の特定やエネルギー分散型 X 線分析 (EDS) による元素分析を行った. TEM では粉碎試料と収束イオンビーム (FIB) 法により切り出した薄膜試料を用いて制限視野電子回折やナノ電子回折, 高分解能 TEM 像による格子間隔測定により鉄ナノ粒子の鉱物種の同定を行った. STEM では広角円環状検出暗視野 (HAADF) 検出器により Z コントラスト像の観察や, EDS により組成像を取得した.

3. 結果

粉碎試料の TEM 観察では従来報告されていた数十 nm の鉄ナノ粒子とともに, 100nm を超える不定形?自形の大きな金属鉄粒子が観察された. 他にも周囲を殻状の磁鉄鉱で囲まれた金属鉄粒子がひとつの結晶片でのみ観察された. 次に岩石薄片の SEM 反射電子像によってこのような大きな金属鉄粒子の分布を調べた. その結果, カンラン石母結晶内に三次元的に見ると板状であると思われる Fe の多いカンラン石の領域と, その中にそれと斜めに交差するように鉄を多く含む粒子が配列する構造が観察された. EBSD で結晶方位を測定すると, この板状カンラン石の領域は母結晶の (100) などの結晶面に沿って形成されていた. この領域をより詳細に観察するために FIB 法で薄膜試料を作製し, TEM/STEM による観察を行った. これにより SEM で観察された粒子は厚さ数十 nm の薄膜状の形態を持ち, ナノ電子回折より金属鉄であることが確認された. さらに STEM の Z コントラスト像により, 薄膜状金属鉄を含む板状のカンラン石の領域には金属鉄ナノ粒子は含まれないこと, 板状のカンラン石の周囲には糸状の金属鉄微粒子の存在も確認された (図 1). また STEM による組成像でも金属鉄の周囲にシリカの多い領域等は見られなかった.

4. 結論

今回の観察では SEM と TEM , STEM を組み合わせることによって, 様々な金属鉄粒子の形態が見つかり, それらのカンラン石母結晶における分布などが明らかにされた. また金属鉄を包有する磁鉄鉱という興味深い構造も見つかった. これらの粒子のさらなる観察・解析により衝撃変成時における温度圧力履歴の解明やリモートセンシングの分野での応用が期待される.

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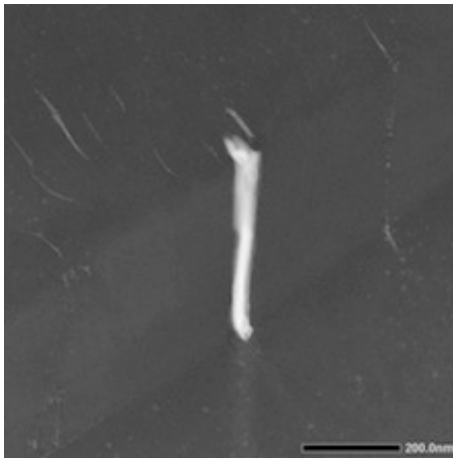
キーワード: 火星隕石, カンラン石, 電子顕微鏡, 衝撃, ナノ粒子

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

PPS24-P16

会場:コンベンションホール

時間:5月23日 18:15-19:30



磁場勾配によって誘導される並進運動を用いた始原的隕石構成粒子の非破壊同定 Nondestructive identification of a single primitive-grain using the translational motion induced by field gradient

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始原的隕石は、起源の異なる多様な粒子の集合体であるため、SIMS や顕微ラマンなど様々なマイクロプローブ技術を駆使して研究が進められている。このような分析に先立って、隕石を構成する個々の粒子の物質識別を、非破壊で効率よく実施する手段が望まれる。近年、私たちは磁場勾配力による並進運動を利用した新しい磁化測定法を確立し、これに基づく物質同定法を提案した。すなわち重力の無視できる希薄な空間に開放した固体粒子は、その内部に誘導される磁気的エネルギーのため、永久磁石レベルの低磁場で、並進や回転を引き起こす。この運動は体積力に由来するため、同一の磁場分布の中では、粒子の質量に依存せず物質固有の磁化率のみに依存する。物質はそれぞれ固有の磁化率を持つので、この方法で得た磁化率を文献値と対応することで、単一粒子の物質識別ができる。これまでに私たちは mm ~ sub-mm サイズの反磁性粒子について、微小重力環境下の磁場勾配中に試料を解放し、試料の並進運動で得た磁化率が文献値と一致することを確認した。しかしながら隕石を構成する粒子の大多数は、sub-mm サイズ以下の常磁性体あるいは強磁性(フェリ磁性)体である。そこで今回、これらの条件の粒子に関しても上記の原理で物質識別が可能か、検証するための実験を進めた。

微小重力は前回に引き続き、室内型の小型落下ボックスを用いて発生させた。落下距離は 1.8m、有効な微小重力継続時間は 0.5 秒以下である。上記のボックスに観測装置を収納する必要があるため、装置の大きさを直径 30cm 以下に小型化した。具体的には小型のネオジム磁石プレートを使用した磁気回路を新たに導入することにより、試料の磁気並進運動の距離を約 2cm に縮小した。さらに、この距離の縮小により、粒子が終端速度に到達するまでの所要時間を 0.5 秒以下に短縮できた。

現在、磁化測定は SQUID や VSM で行うことが一般的である。しかしこれらの方法では、試料サイズの減少とともに、試料ホルダーからのバックグラウンド信号の寄与が顕著となる。また、試料の質量測定が不可欠であるが、これも 10 μ g 以下では実施が難しい。これに対し、私たちが提案する方法では、原理上(試料の運動が観測可能な限り)無制限に小さい試料の磁化率を測定できる。

隕石に限らず、混合試料の分析の前段階として、物質の種類を非破壊で分離・同定することが望まれる。すでに有機化学・生化学の分野では、精密測定に先立ってクロマトグラフィにより、有機分子の混合物を分子量ごとに同定する技術が確立しているが、無機試料でも、有機物と同様の分析過程が望まれる。常磁性、強磁性およびフェリ磁性体で、磁気並進運動を用いた測定原理の有効性が確認されれば、ほぼ全ての固体物質で物質識別が実現することになる。

キーワード: 磁場放出, 非破壊同定, 微小重力, 並進運動, 磁化測定, 体積力

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

惑星物質試料ホルダの洗浄

A Cleaning Method for Extraterrestrial Sample Holder

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

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

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

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

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

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

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

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

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

References

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