

冥王代ジルコンの $^{40}\text{Ar}/^{39}\text{Ar}$ 年代 $^{40}\text{Ar}/^{39}\text{Ar}$ age of Haedean zircon

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同じ岩石中からのジルコンの SHRIMP, LA-ICPMS による U-Pb 年代と角閃石, 雲母類の K-Ar 年代, ジルコン, アパタイトのフィッシュトラック年代を求め閉止温度から地域熱史を議論する方法は熱年代学の基本的手法として確立してきた。カリウムを含まないジルコンは、K-Ar 系年代測定では直接的対象とならない。しかしカナダ北東岸ラブラドル地域および北西準州アカスタ地域の冥王代片麻岩中のジルコンには細粒の白雲母等の鉱物や流体包有物がみられる。包有物の存在はジルコンの同位体系が流体等による二次的な擾乱を受けている可能性を示すものであり、冥王代のジルコンといえどもその影響を受けていないとは限らない。U-Pb 系に比べて熱作用に対してより敏感な K-Ar 系を使ってその影響を探ることはこれらのジルコン中の包有物の形成過程を知る上で重要である。段階加熱によって熱履歴をより詳しく検討する目的で単粒子ジルコンに対して $^{40}\text{Ar}/^{39}\text{Ar}$ 法が適用された。

中性子未照射のジルコンを段階加熱し ^{40}Ar 放出過程を測定したところ多くの粒子では 500-1000°C の温度領域ではあまり放出されないが、1000-1100°C を越えたフラクションで 10^{-7} ccSTP/g というカリウムを含まない鉱物としては比較的多量の ^{40}Ar が放出されることが分かった。そこでそれらのジルコン粒子を中性子照射し $^{40}\text{Ar}/^{39}\text{Ar}$ 測定を行った。多量の ^{40}Ar ($\sim 10^{-10}$ ccSTP) の放出に比べ、単粒子 400 マイクログラムのジルコンからの ^{39}Ar は 10^{-13} ccSTP 程度以下であった。誤差も同程度であるため精度の確保が課題となった。ラブラドルのジルコンからは 1000-1400°C の ^{39}Ar フラクションで全体の 70% を越えて 43.9 ± 3.4 億年という年代が測定された。現時点ではこの ^{40}Ar がジルコンの初期的形成過程もしくは二次的事象によって捕獲された過剰アルゴンでないと判別することは出来ない。しかし過剰アルゴンでは局所的な因子が強く働くため同一地域の岩石であっても、同一の過剰アルゴン年代を示しにくい。同年代の値が複数の単粒子から測定されたこと、また 1000°C 以上という高温のフラクションで観察されることはこれらの年代が偶然的ではなく、形成時から保持されてきたことを示唆する。他地域のジルコンの結果とも併せて検討する。

キーワード: 冥王代ジルコン, $^{40}\text{Ar}/^{39}\text{Ar}$ age

Keywords: Haedean zircon, $^{40}\text{Ar}/^{39}\text{Ar}$ age

英国・ウェールズ-イングランド地域に露出する沈み込み型造山帯中の二つの広域変成岩帯
Two regional metamorphic belts in assemblages of subduction-related orogens in Wales-England, U.K.

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Cryogenian-Devonian orogenic belt in the British Isles consists of accretionary complex, arc-related volcano-plutonic belt with regional metamorphic belt that have formed in relation to an oceanic-plate subduction in the Iapetus Ocean. This subduction-related orogeny plays an important role in understanding of the geotectonic history of the pre- Ordovician orogenic belt in Wales-England.

Wales-England consists of ca. 677-450 Ma calc-alkaline volcano-plutonic complexes and ca. 608-539 Ma accretionary complexes, suggesting that the subduction-related orogeny continued for ca. 200 My. Four metamorphic units related to this orogeny exist in northwest Wales. However, their metamorphic ages, except for one unit, have not been constrained yet. Therefore, we performed U-Pb dating on detrital zircon in psammitic schists and K-Ar dating on phengite in pelitic/mafic schists from the four metamorphic units in order to constrain their sedimentary and metamorphic ages, respectively.

The K-Ar analysis was carried out at Okayama University of Science, and the U-Pb ratio was determined with LA-ICP-MS at the Kyoto University. Three metamorphic units give K-Ar ages of 578-545 Ma, which overlaps the Ar-Ar age of 560-550 Ma reported by a previous work. During this metamorphic event, accretionary complex and calc-alkaline volcano-plutonic complexes also had been formed. On the other hands, one metamorphic unit, called as New Harbour Group, exhibits a younger K-Ar age of 474 ± 9 Ma. The youngest U-Pb age of detrital zircons from the New Harbour Group is 520 ± 31 Ma. These ages suggest that the New Harbour Group formed in the regional metamorphic event different from that of the above three units. Calc-alkaline igneous rocks of ca. 480-450 Ma exist in Wales-England, which coincide with the second metamorphic event. Therefore, we conclude that at least two regional metamorphic events occurred in the geotectonic history of Wales-England.

Keywords: British Isles, Subduction-related orogeny, Regional metamorphic belt, K-Ar phengite ages

北部ケニアリフト, パカ火山の Ar/Ar 年代測定による活動分析 Ar/Ar Geochronological analysis of Paka volcano, northern Kenya Rift

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北部ケニアリフトは、大陸地溝系における若い火山性かつテクトニックな活動の最も顕著な一例である。しかしながら、異なる時代の溶岩流や、関連する火口や断層についての限られた年代情報から、リフト内におけるそのような活動について正確に評価することは困難である。今回、北部ケニアリフトに位置し、複合カルデラ盾状火山であるパカ火山を構成する溶岩流の 30 個の ⁴⁰Ar/³⁹Ar 年代測定結果を得たので報告する。

パカ火山はリフトの底面から比高 650m の山体であり、trachyte, mugearite, basalt からなる溶岩流や火砕物で 280km² が被われている (Dunkley et al., 1993)。Dunkley et al. (1993) は、4 試料の ⁴⁰Ar/³⁹Ar 年代測定から、少なくとも 0.4 Ma, 0.2 Ma, 10 ka に火山活動があったとしている。新鮮な山頂火口の地形とそれに伴うリフト伸張方向に沿う断層群の関係は、パカ火山における火山性かつテクトニックな活動が、リフト内で、特に最も年代の若い部分の変形過程に関連している可能性を示している。このような火山の噴火と断層系生成時期を知ることが、北部ケニアリフトの軸に沿うテクトニック及び火山性の活動の正確な評価のためにきわめて重要である。

今回、ポツダム大学での ⁴⁰Ar/³⁹Ar 年代測定のために、溶岩流から主に 32 試料が採取された。試料の採集は Dunkley et al. (1993) の地質図とパカ火山及び周辺の溶岩地形を考慮し、各溶岩流から得られた。各岩石から石基を濃集した物を酸処理した後、オレゴン大学 TRIGA 原子炉の CLICIT 施設で 4 時間速中性子照射した試料を、ポツダム大学で ⁴⁰Ar/³⁹Ar 年代測定を行った。CO₂ レーザーを用いた段階加熱の結果を年代スペクトラムとアイソクロンから十分に検討し、ほとんどの場合で精度の良い年代値が得られ、かつ Dunkley の年代値ともよく一致した。

マイナス年代を示した 2 試料を除いてトータルで 30 個の年代が得られた。それらは過去 0.6 Ma の間火山活動が続き、かつ特に 3 時期; (I) 0.428 から 0.372 Ma, (II) 0.160 から 0.126 Ma, そして (III) 0.039 から 0.012 Ma において活動が多かったことを示した (Fig. 1)。この 3 時期を基にし、全ての岩石をつぎの 3 時期に分類した; (1) 0.6 から 0.35 Ma, (2) 0.35 から 0.1 Ma, そして (3) 0.1 から 0 Ma。これに従って全岩化学組成と火山活動の空間分布を調べた。最初に Dunkley et al のいくつかの basalt が trachyte あるいは mugearite であることが分かった。また層序にも変更を要するところがあった。(1) の時期には trachyte と mugearite のみが西側のリフト底面と山体の北東側に噴出した一方、それらより若い trachyte は山体中で N-S 方向に沿って噴出した。溶岩流中に形成された多くの正断層は、(1) の時期に含まれる山体の北東方向の古い溶岩流の中のみ見られた。微量元素比の Nb/Zr は、3 時期の間で明瞭に異なる元素比を示し、マグマの組成が活動期間中に徐々に変化したことを示唆した。今回の我々の有用な年代データは、フィールドにおける断層系の観察事実ともよく合い、これからの北部ケニアリフト内の構造の発達モデルの構築に貢献することが期待される。

Reference: Dunkley P. M., M. Smith, D. J. Allen and W. G. Darling (1993): International Series, Research Report SC/93/1, 185pp, British Geological Survey

キーワード: ケニアリフト, アルゴンアルゴン年代測定, 第四紀火山岩, テクトニクス, 全岩化学組成

Keywords: Kenya Rift, Ar/Ar dating, Quaternary volcano, tectonics, whole rock chemistry

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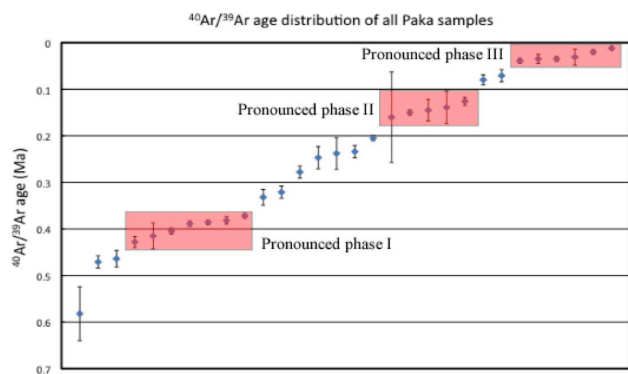


Fig 1. Distribution of $^{40}\text{Ar}/^{39}\text{Ar}$ ages of Paka samples and three inferred periods of pronounced volcanic activity.

Quantifying seawater assimilation across a single MORB pillow Quantifying seawater assimilation across a single MORB pillow

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Halogens and noble gases are critical tracers of volatile interaction between geological reservoirs. The incompatibility of the heavy halogens (Cl, Br, I) coupled with their unique elemental composition and concentrations within surface reservoirs make them suitable tracers of volatiles recycled back into the mantle [1]. However, as the halogens are concentrated within seawater and other marine reservoirs it is possible that they are also incorporated within submarine basalts during eruption, causing the original mantle volatile signature of the basalt to be overprinted [2].

This study combines halogen (Cl, Br and I), noble gas, K and H₂O concentrations within a single pillow basalt to quantify the amount of seawater assimilation during eruption, and to further elucidate the mechanisms of assimilation. In order to determine the scale and heterogeneity of seawater contamination within the pillow, multiple sections taken at 2.5mm intervals were analysed along a transect from the glass rind to the crystalline interior.

The two outer sections of the glass rim show enrichment in the halogens compared to the deeper glass and crystalline section of the pillow, which have distinctly MORB-like I/Cl and Br/Cl ratios. Lower K/Cl and Br/Cl ratios measured within the outermost sections (#1 and #2) indicate the glass has incorporated a Cl rich seawater component. The H₂O/Cl ratios within sections #1 and #2 show that the assimilation is a high salinity brine produced by hydrothermal boiling and phase separation of seawater caused by the eruption of the basalt.

The extent of brine assimilation within the glass ranges between 8% in section #1 to >50% in section #2 indicating brine assimilation within basaltic glasses is considerably heterogeneous. The high level of brine contamination within the glass as calculated from the halogens is in contrast with the noble gases within the glass, which still retain their magmatic signatures. This suggests that the halogen and noble gases within submarine basalts may be decoupled, with loss of atmospheric noble gases from the assimilation during hydrothermal boiling.

The crystalline interior shows an increase in the Xe and I concentration relative to the glass rim. The ¹³²Xe/³⁶Ar ratio and elevated iodine concentrations when compared to the rest of the pillow indicate the pillow contains a sedimentary component within its interior, most likely due to incorporation of fine sediments during eruption.

Combined halogen, noble gas, K and H₂O data presented here for a single pillow basalt demonstrates that contamination from the marine environment can be variable and extensive. This combined analytical approach demonstrates the need for careful characterization of samples to ensure only samples free from contamination are chosen for analysis of mantle volatiles.

[1] Sumino et al (2010) EPSL, 294, 163 - 172 [2] Kumagai and Kaneoka (1998) Geophys. Res. Lett. 25(20), 3891 - 3894

キーワード: Halogen, Noble gas, Seawater contamination, MORB
Keywords: Halogen, Noble gas, Seawater contamination, MORB

Ar-Ar および I-Xe 年代測定法を応用したマントル捕獲岩のハロゲン分析 Halogen analysis of mantle xenoliths by extending Ar-Ar and I-Xe dating methods

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Ar-Ar および I-Xe 年代測定法を応用することにより、極微量ハロゲンの定量が可能となる。原子炉内において試料に中性子を照射すると、ハロゲン (Cl, Br, I) や K, Ca, Ba, U から特定の Ar, Kr, Xe 同位体が生成する。これらを高感度希ガス質量分析を用いて定量することにより、上記の複数の親元素を同時に定量することが可能である [1]。ただし希ガス同位体への変換率は、組成が既知の標準試料と同様に中性子照射、希ガス同位体分析して決定する。この手法によるハロゲンの検出限界は Cl, Br, I についてそれぞれ 10^{-10} - 10^{-11} 、 10^{-13} - 10^{-14} 、 10^{-14} - 10^{-15} mol であり、地球化学において通常用いられるような手法よりも数桁低い。

ハロゲンは F を除き、不適合元素であり水に強く濃集する性質をもつ。地球においては表層部に高濃度で存在し、マントル内での濃度は低い。また、各リザーバーにおいて大きく異なる元素組成を示す。このような性質から、ハロゲンはマントルへと沈み込む水の有用なトレーサーとなることが期待されている。また、ハロゲンは LIL (large-ion lithophile) 元素の輸送に大きく関与していることも示唆されている。島弧マグマに特徴的に多く含まれる LIL 元素は、スラブから放出される水流体とともにマントルウェッジに供給されていると考えられている。ハロゲンが水流体中に存在することで、LIL 元素は最大で 10 倍程度強く水流体に濃集することが実験的に示唆されている [2]。このようにハロゲンはスラブからマントルへ放出される流体において最も重要な元素の 1 つであり、マントル捕獲岩に含まれる流体包有物はマントルに供給された流体が持つ情報を最もよく保存していると期待される。しかし、マントル中のハロゲン濃度は低く、地球化学において通常用いられる手法での定量は困難である。そのため、マントル捕獲岩のハロゲンに関する研究例は極めて少ない。

本研究では、Ar-Ar および I-Xe 年代測定法を応用した手法を用いて、アジア東縁の沈み込み帯 (カムチャッカ、フィリピン、東北日本) やヨーロッパ (アイフェル)、北アメリカ (サンカルロス、キルボーンホール) で産出したマントル捕獲岩に含まれるハロゲンを分析した。試料から希ガスを抽出する際には、試料を物理的に破碎することにより流体包有物のハロゲン組成を、加熱熔融することにより試料全体のハロゲン組成をそれぞれ得た。

火山フロントで産出したマントル捕獲岩に含まれる、 H_2O に富む流体包有物のハロゲン組成は高い I/Cl 比と比較的一定な Br/Cl 比を示し、海底堆積物中の間隙水によく似た組成である [3]。全岩組成は流体包有物の組成に比べやや Cl に富み、Cl を多く含む相が存在することを示しているが、やはり間隙水の強い寄与がみられる。間隙水的なハロゲン組成は三波川変成帯で産出したかんらん岩にも見出されていて、間隙水に由来するハロゲンが、沈み込む海洋プレート中の蛇紋石等の含水鉱物に取り込まれマントルへ沈み込んでいることが示唆されている [4]。ハロゲン濃度は低いが、同様な組成が背弧側のマントル捕獲岩にも一部みられた。ヨーロッパと北アメリカで産出したマントル捕獲岩の I/Cl、Br/Cl 比は、MORB の組成を含むような共通の相関関係を示した。

沈み込み帯でみられたハロゲン組成は、沈み込んだ海底堆積物中の間隙水起原のハロゲン [4] が火山フロント直下のマントルにおいて支配的であることを示している。また、その影響は小さいながらも背弧側まで及んでいることが示唆される。プレート内火山の活動によって地表にもたらされたヨーロッパと北アメリカのマントル捕獲岩にみられた I/Cl、Br/Cl 比の相関関係は、MORB 源マントル的な組成からの分別に起因すると考えられ、これらの地域の大陸下マントルにおいてハロゲン組成を同様な傾向で変化させるメタソマティズムが起きた可能性を示している。

[1] Turner (1965) JGR 70, 5433. [2] Kawamoto et al. (2014) EPS 66, 1. [3] Kobayashi et al. (2014) JpGU Meeting 2014, SCG62-04. [4] Sumino et al. (2010) EPSL 294, 163.

キーワード: ハロゲン, 希ガス, マントル捕獲岩

Keywords: halogen, noble gas, mantle xenolith

伊豆小笠原弧火山岩に見られる島弧横断方向の希ガス・ハロゲン組成変動 Across-arc variation in noble gas and halogen compositions of volcanic rocks from the Izu-Ogasawara subduction zone

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Recently, subducted halogens and noble gases with seawater and sedimentary pore-fluid signatures were discovered in exhumed mantle wedge peridotites and eclogites from the Sanbagawa-metamorphic belt, southwest Japan [1,2]. These findings along with seawater-derived heavy noble gases (Ar, Kr, and Xe) in the convecting mantle [3] provide observations that allow us to investigate the processes that control the return of volatile and highly incompatible elements into the mantle. Serpentinized lithosphere of subducting oceanic plates can transport noble gases and halogens acquired from pore water in the overlying sediment [1,2,4]. To verify whether and how such subduction fluids modify the composition of the mantle beneath subduction zones, we determined the noble gas and halogen compositions of olivines in arc lavas of the northern Izu-Ogasawara subduction zone and IODP sediments and basalts recovered from the northwestern margin of the Pacific plate. Trace amounts of halogens (Cl, Br, and I) in the olivine samples were measured by a combination of neutron irradiation and noble gas mass spectrometry [5].

The ³He/⁴He ratios of samples from the volcanic front (Izu-Oshima, Miyakejima, Mikurajima, Hachijojima, and Aogashima) and rear-arc (Nijima, Higashi-Izu monogenetic volcanoes, Nanzaki, and en-echelon seamount chains) regions are in the range of the mid-ocean ridge basalt (MORB) value, without systematic differences among the regions. This indicates a considerably low contribution to the mantle wedge beneath the arc of radiogenic ⁴He in the subduction fluids observed in the Sanbagawa samples exhumed from a depth ranging from 30 to 100 km [1,2].

In contrast, the ⁴⁰Ar/³⁶Ar ratios for each volcano (299-620) are significantly lower than the MORB source (up to 32,000 [3]), indicating significant involvement of atmospheric Ar (⁴⁰Ar/³⁶Ar = 296) in the magmas. Systematically higher ⁴⁰Ar/³⁶Ar ratios in the rear arc than in the volcanic front, and a comparison with those of subducting materials, suggest that subduction of seawater-derived Ar significantly affects the noble gas composition of the magma-generation region. A simple mass balance calculation of subducted and mantle-derived Ar isotopes reveals that higher subduction flux than that of seawater-derived Ar in the pore fluids in the subducting sediment/crust is required. The serpentinized lithosphere in the subducting slab is the best possible carrier of seawater-derived Ar with high subduction flux.

Although the halogen compositions of most of the olivines are close to that of MORB-source mantle, some samples from the en-echelon seamount chains show a significant contribution from pore-fluid-derived halogens. Combined with the noble gas results, halogen-poor fluid associated with atmospheric noble gases may be dominantly released from the subducting slab beneath the Izu-Ogasawara arc, while halogen-rich fluid significantly contributes to the magma generation region beneath some volcanoes in the rear arc.

It remains unclear whether noble gases and halogens in the subducting materials are completely released beneath the rear arc. However, the presence of seawater-like noble gases in the convecting mantle [3] implies that a small portion of seawater-dissolved atmospheric noble gases, carried in the serpentine, might be transported to greater depths in the deeper mantle.

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キーワード: 希ガス, ハロゲン, 沈み込み帯, 伊豆小笠原弧, 間隙水

Keywords: Noble gas, Halogen, Subduction zone, Izu-Ogasawara arc, Pore fluid

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希ガス同位体比；太陽系形成のパラメター
Noble gas isotopes; Solar evolution parameter

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太陽系の進化のリファレンス・パラメター

キーワード: 希ガス同位体, 惑星進化, 隕石
Keywords: noble gas isotopes, planetary evolution, meteorite

地球外物質希ガス測定のための新しい分析方法とシステム New analytical methods and a system for noble-gas measurements of extraterrestrial materials

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Noble gas isotopes in extraterrestrial materials, meteorites, cosmic dust (micrometeorites), and spacecraft-return samples, are one of the most useful tools of cosmochemistry. Radiogenic (e.g., ⁴⁰Ar, ¹²⁹Xe, and ¹³⁶Xe) and cosmogenic (²¹Ne, ⁸¹Kr, ⁸³Kr, and ¹²⁸Xe) isotopes are used for dating when gases have begun to be accumulated in the relevant minerals or phases. Primordial or trapped (neither radiogenic nor cosmogenic) isotopes are informative to infer the origin of the materials. Conditions of physical processes, such as evaporation and condensation, will be discussed based on elemental/isotopic fractionation effects.

The mass spectrometry system at Kyushu Univ. consists of a conventional mass spectrometer (MS) and a resonance ionization MS, called RING (Resonance Ionization Noble Gas mass spectrometer). The conventional MS is mainly used to measure light noble gases (He, Ne, and Ar) in micro- or submicro-gram samples enriched in solar-wind noble gases, such as mineral separates from regolith breccia meteorites, Antarctic micrometeorites [1], lunar soils, and the Itokawa dust particles returned by the Hayabusa spacecraft [2]. Extraction of noble gases is performed using infrared radiation lamps for 50-200 °C and a resistant furnace (called Pot-pie furnace) for 200-2000 °C. In addition, the MS system is equipped with a Nd:YAG laser that produces energy of 200 mJ/pulse (with a 10 nsec width) at 1064 nm and a spot with ~50 μm in diameter. In the ablation of samples, plasma lights are emitted due to the high energy density of the laser pulse, and have wavelengths depending on the chemical composition of the fused area. The plasma emission spectra are measured using a LIBS (Laser-induced Breakdown Spectroscopy, e.g., [3]) system prepared for the laser extraction system. Combination of the conventional MS and LIBS enables to determine K-Ar radiometric ages for the specific areas (e.g., shock-melt veins or feldspathic phases) of polished sections. Experimental analyses using terrestrial rocks containing K-rich phases (e.g., K-feldspar) and equilibrated ordinary chondrite samples are now on going.

The RING mass spectrometer is designed to measure extremely small amounts of Kr and Xe (>100 atoms), as modeled after RELAX (Refrigerator Enhanced Laser Analyzer for Xenon) developed by the Manchester group (e.g., [4] and [5]). In our system, a dye laser equipped generates 3.5 mJ/pulse at ~216 and ~256 nm of wavelengths that ionize resonantly Kr and Xe, respectively. At this moment, we have got mass spectrum of Xe isotopes but not those of Kr yet.

In addition to the improvements and developments of instruments for noble-gas mass spectrometry, sample preparation methods have also been developed for combinational (multidiscipline) analyses for individual samples with micro- and submicro-gram masses. For example, we have performed TEM and noble gas analyses for individual micrometeorites [1] and the Itokawa dust particles [2].

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キーワード: 希ガス, 地球外物質, 共鳴イオン化質量分析計, レーザ誘起ブレイクダウン分光装置, K-Ar 年代法

Keywords: noble gas, extraterrestrial material, resonance ionization mass spectrometry, laser-induced breakdown spectroscopy, K-Ar dating

イトカワ試料の形態的特徴と希ガス同位体組成との関係 Relationships between morphological features and isotopic signatures of noble gases of four Itokawa particles

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Introduction: Fine-grained particles of the asteroid Itokawa preserve the effects related to the processes on and near the surface of an asteroid. Various types of surface modification were found on the very surface (<100 nm) [1-6]. The modification textures suggest that irradiation by solar wind (SW) plays an important role for their formation. Number density of solar flare tracks in them ranges from a few $\times 10^9$ to a few $\times 10^{10}$ tracks/cm² [2, 5], which are considerably lower than expected. These data suggest the mechanisms and periods of the surface modification of Itokawa grains are still unresolved. In addition to the SW irradiation, there seems to have been another mechanism that modified the morphology and surface features of the grains. Matsumoto et al. [3] found some Itokawa grains were aggregates composed of subgrains with stepped surfaces, which suggests evaporation and/or condensation. Noble gas mass spectrometry is another tool to infer the individual history of each Itokawa grain. Nagao et al. [7] argued that a grain experienced multiple processes: exposure to SW, burial in the regolith layer, and removal of its external surface. We intend to investigate the relationships among morphology, surface modifications, and noble gas isotopic signatures to discuss the history of each Itokawa particle from various perspectives.

Samples and methods: We fixed four Itokawa particles on tantalum plates by acetone-soluble glue. We observed the surface of the particles and prepared thin foils by FIB-SEM at Ibaraki University. After preparation of FIB foils, all the Ta plates with samples rinsed by acetone in a N₂ filled glove box. They were transferred to Kyushu University and noble gas mass spectrometry was performed for all the noble gas elements using a modified VG-5400. Noble gas extraction from each sample was conducted stepwise at 50, 100, 200, 300, 600 and 1400 °C.

Results and discussion: SEM observation of the four Itokawa grains investigated shows various morphology and surface features. Edges observed on the surfaces of three grains vary from sharp to dull. Additionally, one grain is composed of two subgrains with stepped surfaces. Morphology and surface features are related to the He release patterns. The three particles have similar ⁴He release patterns; they release most ⁴He below 200 °C fractions and a small amount of ⁴He above 600 °C. Although the gas-release profiles are similar to each other, there is a difference in the bulk concentrations of ⁴He; one of the three grain having dull edges contain more abundant ⁴He than the others having sharp edges. Because bulk ³He/⁴He ratios in the three grains are 2.5 to 6.5 $\times 10^{-4}$, which is comparable with that in the solar wind of 4.57 $\times 10^{-4}$ [8], it is likely that the implanted He in the grains increased as the surfaces were degraded by solar wind sputtering. On the other hand, the grain composed of subgrains with stepped surfaces shows a different ⁴He release pattern; ⁴He was released only below 200 °C. This fact suggests that deeply implanted He, which is probably related to He released above 600 °C, was completely reset by severe heating. The steps on the surfaces of this grain may have been formed by evaporation and/or evaporation and subsequent recondensation by a heating event, which released He from the interior of the grain. These results strongly suggest that micrometer-size surface features are related to the amounts and the release patterns of the implanted He.

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トンネルイオン化スパッタ粒子質量分析による局所希ガス同位体分析の現状 Present status for in-situ noble gas analysis by Sputtered Neutral Mass Spectrometry with tunneling ionization

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LIMAS (Laser Ionization MASS nanoSCOpe) was installed in 2011. In-situ analysis for trace elements in nanometer-scale is a concept of this apparatus. I have conducted a study on mechanism of solar wind (SW) implantation into extra-terrestrial materials such as Itokawa particles. Because depth profile of SW-He have not been determined by using conventional noble gas mass spectrometer (NG-MS) in a quantitative way, a depth profiling of the SW noble gas contributes to the great progress for understanding implantation and removing processes of the SW noble gases. The processes imply the space-weathering history of the materials on its asteroid surface.

LIMAS is mainly composed of Ga focused ion beam (FIB) system for primary ion, femtosecond (fs) laser for tunnel ionization of sputtered particles, and time-of-flight mass spectrometer. Depth profiling procedure is as follows. The FIB system exhibits high spatial resolution with large primary current. The smallest beam size is 8 nm at 3 pA, and the largest size is 600 nm at 30 nA. The fs laser can ionize the all element even He because of high energy density. The energy density is $8E15 \text{ W/cm}^2$ because the pulse energy and width is 6.3 W and 30 fs, respectively, and the laser pulse was focused on a volume of $50 \times 50 \times 50 \mu\text{m}^3$. This laser pulse induces tunneling ionization (Delone and Krainov, 1998). Helium depth profile analysis in a few micrometer has been developed with LIMAS and atomic force microscope for crater depth measurement.

LIMAS can detect tens ppm in number of atoms He in sub-micrometer area on solid surface. At present useful yield of He is 0.02% which should be up to 1% by optimizing laser focusing and irradiation position. The yield of 0.02% corresponds to 4000 He atoms for single He ion count. The back ground of He is much less than that of the NG-MS because LIMAS is used in dynamic operation under the ultra-high vacuum.

LIMAS can measure locally concentrated noble gas because the sputtered volume is quite small, which indicates that this measurement is almost non-destructive isotope analysis. This feature can play a role for analysis of tiny and precious samples such as the Itokawa particles. U,Th-He and K-Ar dating for single grain can be applied by the in-situ noble gas analysis. The in-situ dating may obtain a new insight of thermal history of igneous rocks by comparing conventional in-situ dating method such as U-Pb age.

キーワード: 希ガス同位体, 局所分析, スパッタ中性粒子質量分析
Keywords: noble gas, in-situ analysis, Sputtered Neutral Mass Spectrometry

熱水の希ガス分析前処理装置の製作と質量分析計の改良 New preparation apparatus for noble gas isotope analyses to measure submarine hydrothermal fluids and renewed GVI-5400

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Hydrothermal activity is an essential phenomenon to drive geochemical differentiation on the Earth's surface, which should form polymetallic massive sulfide ores. Noble gas isotopes are regarded to be a powerful tracer even for the geochemical study on hydrothermal activity; however, there are abundant interference volatiles, e.g. halogens and hydrogen sulfide. Here, we designed a new preparation system to remove such volatiles using non-traditional gettering materials. This new preparation system consists of following components: the water preparations system, composite gettering system (halogen getter, sulfide getter and Ti-Zr getter), cryogenic pump, Saes-getter pump and charcoal trap to analyze the all noble gases. The new preparation system has compact volume, which needs small amount of seawater sample of 2-5 cc in volume. It is almost 1/20 compared to the typical requirement for He isotope measurement.

To design new noble gas measurement system in JAMSTEC, another critical problem is to be solved. One of widely used noble gas mass spectrometers, GVI-5400, is now not listed in commercial production models. Thus, all successor models of VG-5400 series have entirely finished their production support since several years ago. However, the VG-5400 series and its successor models still show satisfactory sensitivity and precision to determine noble gas isotope ratios. If we decide to use them much longer, we should maintain their control units, such as power supply, emission control or source control and so on, which requires replacement of essential electrical parts, including vacuum tube (!), regularly. At present, some of these parts are not supplied anymore and we are forced to choose between not replacing them with the new/second-hand electrical parts while they still worked, or renew their electrical circuits with currently manufactured electrical parts. By chance we had started the project to re-use old model of power supply unit. The first trial was applied to the very original VG5400 that was moved from Univ. Tokyo to Yamagata Univ. As the second case, we renewed the power supply and the emission control unit equipped for the JAMSTEC-2 noble gas mass spectrometer (GVI-5400He).

Here we report the better performance of new version of the renewed power supply unit, noiselessness and better stability, with the new noble gas preparation system.

キーワード: 希ガス, ゲッターポンプ, 海底熱水, 質量分析計, 抽出系

Keywords: noble gas, getter pump, hydrothermal activity, mass spectrometer, extraction system

Giese型イオン源を搭載した高感度希ガス質量分析計 High sensitive noble gas mass spectrometer equipped with a Giese-type ion source

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Although noble gas isotopes are powerful tracers in geosciences, their extremely low abundances in mineral and rock samples make their analysis quite difficult. For example, concentration of ^3He , which is a good indicator of mantle-derived component because of its primordial origin, is as high as 0.01 parts-per-trillion in volcanic rocks and mantle-derived materials. Such scarce noble gas isotopes are detected with a special mass spectrometer operated in static mode. We have made it possible to detect 10^3 to 10^4 atoms of noble gas isotopes by modifying a commercial sector-type single focusing noble gas mass spectrometer (VG5400), which is equipped with a double collector system to detect ^3He and ^4He simultaneously with a secondary electron multiplier and Faraday cup, respectively [1]. Here we report an attempt of further improvement of sensitivity of the mass spectrometer by installation of a new ion source (Giese-type source).

The Giese-type electron ionization (EI) ion source is equipped with two electrostatic quadrupole lenses [2]. This source has been reported to have up to two orders of magnitude higher sensitivity than conventional Nier-type EI source because of the absence of a beam defining slit to collimate the ion beam and thus high transmission [3]. We designed a Giese-type source to have an adequate resolution to separate $^3\text{He}^+$ from HD^+ and H_3^+ , to have the source housing volume as small as possible, and to be bankable at up to 300 °C to reduce outgas from the source materials. The ion and electron optics were based on a calculation by Lu and Carr [4] and refined using SIMION-3D software [5]. Prior to the installation on the mass spectrometer, the ion beam profile emitted from the source was monitored by a microchannel plate and phosphor screen to optimize the configuration of the quadrupole lens.

A sufficient mass resolution over 500 essential for $^3\text{He}/^4\text{He}$ analysis has been achieved with an improved sensitivity approximately three times higher than the previous condition. The amount of helium required to obtain a precision with $^3\text{He}/^4\text{He}$ ratio is two orders of magnitude smaller than that with the condition installed by the manufacture. However, total ion transmission is estimated to be about 30%, suggesting further refinement of the source condition is required to obtain the maximum sensitivity.

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キーワード: 希ガス, 質量分析計, 四重極レンズ, イオン源
Keywords: Noble gas, Mass spectrometer, Quadrupole lens, Ion source

熱水性石英中の流体包有物を用いた Ar-Ar 年代測定と試料の産状の比較 Ar-Ar dating by fluid inclusions in hydrothermal quartz, compared with its field and microscopic observations

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The chemical evolution history of the ocean must have been one of the most critical factors to unravel the evolution of life on the Earth. However, this topic has not fully understood yet. Recently, de Ronde et al. (1997) and some studies try to estimate seawater composition during Archean and Proterozoic using fluid inclusions trapped in hydrothermal quartz correlated with pillowed basalt, which is expected to erupt in sub-seawater condition. Lowe and Byerly (2003) claims that the hydrothermal minerals used by de Ronde et al. (1997) were formed at far younger age (Quaternary) than expected age of eruption of the matrix lavas (3.2 Ga), based on the only field and microscopic observations. This problem must be solved by dating of the fluid inclusions trapped in hydrothermal quartz and comparing the age of pillowed basalt as a matrix in addition to field and microscopic observations. We carried out Ar-Ar dating of the fluid inclusions with stepwise crushing techniques at Guangzhou Institute of Geochemistry, Chinese Academy of Sciences.

Analyzed samples in this study are from 2.2 - 2.4 Ga Ongeluk Formation, Transvaal Supergroup, South Africa. Above Ongeluk Formation, two younger volcanic layers exist, one is mafic intrusions of the Malopo Farms complex formed at 2044 ± 24 Ma, the other is porphyritic andesite of the Hartley Formation formed at 1928 ± 4 Ma.

The analyzed samples are hydrothermal quartz with growth zoning texture, filling the primary shaped drainage cavities and interstitial spaces of lava flows of the Ongeluk Formation without quartz vein crosscut pillow of lava, suggesting the quartz had precipitated soon after eruption of the lava. We analyzed four of the most primary-rich samples (GU84, GU91, GU103a and GU103b), in terms of microscopic observations. However, secondary fluid inclusions are not excluded completely, in other words, argon gas extracted the quartz samples including some secondary fluid inclusions origin in the analysis.

Three samples, GU 84, GU103a and GU103b, yielded Ar-Ar isochron ages of 2688 ± 584 Ma, 1952 ± 279 Ma and 1967 ± 64 Ma Ar-Ar isochron ages, respectively. GU91 was also dated in the same manner as well, but failed to obtain a tight line of isochron with a diffused age (611 ± 517 Ma).

Comparing the age of the Ongeluk lava and younger two volcanisms with the results, the age from GU84 has a wide range of error, but is consistent with Ongeluk lava, GU103a has also a wide range of error, but is consistent with all three volcanisms, and GU103b has the narrowest range of error and is consistent with Malopo Farms complex and Hartley Formation, especially match with Hartley. Based on field and microscopic observations, it is unlikely to precipitate quartz with filling cavities 100-200 Myr after eruption of Ongeluk lava. Two probable mechanisms to explain the results, one is that fluid inclusions were re-equilibrated at younger volcanisms in terms of the argon with preserving primary textures. The other is that secondary fluid inclusion formed by younger volcanism driving hydrothermal circulations and that extracted argon gas in the analysis is mainly composed of the secondary inclusions. The secondary fluid inclusions have argon ratio suggesting younger age than primary fundamentally, suggesting that GU84 obtaining the oldest argon age is the most primary rich samples. Assuming that obtained younger ages than Ongeluk lava are resulted in argon gas from secondary fluid inclusions, the consistency with Ar-Ar age of GU84, the most primary-rich sample, and Ongeluk lava support that our field and microscopic observations are robust and that the primary fluid inclusions formed soon after Ongeluk lava eruption.

キーワード: 流体包有物, 年代測定, アルゴン, 石英, 海水
Keywords: fluid inclusion, Ar-Ar dating, quartz, seawater

宇宙線生成³⁹Arを使った月表面のK-Ar年代分布測定の可能性について Possibility of K-Ar age mapping on the moon using cosmogenic ³⁹Ar

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月表面探査において迅速にかつ広範囲に年代分布を調べるにはその場分析を行う必要がある。宇宙線照射により³⁹Kから³⁹Arが生成されていることは隕石の希ガス研究の初期から知られ、⁴⁰Ar/³⁹Ar年代測定法の開発のきっかけにもなった。宇宙線中の速中性子束密度が時間的にほぼ一定と考えられる環境では長い時間がたつと生成と放射性壊変の平衡状態が実現され、岩石中の³⁹ArはそのK濃度に応じて一定値をしめすようになる。太陽系での速中性子の供給源は主に太陽である。太陽の活動周期による変化で速中性子密度も変化するが³⁹Arの半減期の293年ではその供給はほぼ一定と見なすことは可能であろう。ほぼ同じ照射条件下にあるとみなすことができる隕石試料または月表面の岩石を用いて原理的にはその⁴⁰Ar/³⁹Ar年代が可能になる。すなわちそれらの一つを実験室で標準試料として正確な年代測定を行う。その年代を使った同じ試料の野外でのJ-値を求め、他の未知試料の⁴⁰Ar/³⁹Ar比を測定することで年代測定が可能になる。干渉同位体による³⁹Arの生成はCa/K比が極端に大きくない限り影響は大きくならない。中性子束密度は照射される物体の深さに応じて減衰するので余り深い位置にある試料に対しては有効でない。しかし⁴⁰Arのバックグラウンドや質量測定を必要としない点、³⁶Arを考慮する必要がほとんど無い点では地球上での測定より簡便で迅速な測定に向いている可能性がある。

キーワード: ³⁹Ar, 宇宙線生成, 年代分布, ⁴⁰Ar/³⁹Ar年代, 月面
Keywords: ³⁹Ar, Cosmogenic, age distribution, ⁴⁰Ar/³⁹Ar age, moon surface