

$^{40}\text{Ar}/^{39}\text{Ar}$ age of Haedean zircon

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Analyses of ages and closure temperatures using SHRIMP and LA-ICPMS U-Pb system of zircon, K-Ar system of hornblende and micas, and fission track method in zircon and apatite in the same rock sample are the standard techniques in thermochronology. Since zircon has no potassium, it has rarely been used in K-Ar geochronology. After intensive microscopic observation and electron microprobe analyses of Haedean zircons from the Labrador and Acasta gneiss, fine grain muscovite and other minerals as well as fluid inclusions are recognized. The presence of such inclusions suggests a possibility of a secondary event, and it might have caused disturbance in U-Pb system in the zircons, implying the zircons may be older. Utilizing susceptible characteristics of K-Ar system in a secondary hydrothermal event, laser step heating $^{40}\text{Ar}/^{39}\text{Ar}$ method was applied on the individual zircon grains to investigate the timing of formation of such inclusions in the zircons.

During laser step heating experiment, unirradiated Haedean zircons showed little release of ^{40}Ar below 1000°C. However, without exception, they released large amount of ^{40}Ar (an order of 10^{-7} ccSTP/g) above 1000°C in spite of a fact that zircon has no potassium. This suggested that either the zircons have excess argon trapped or a potassium phase. In $^{40}\text{Ar}/^{39}\text{Ar}$ experiment, the released argon isotopes were approximately 10^{-13} ccSTP (^{39}Ar) and 10^{-10} ccSTP (^{40}Ar) from a grain of 400 microgram. The volume of ^{39}Ar was very small, and the relative error was large. However, one of Labrador zircon gave 4.39 ± 0.34 Ga at a fusion step after 1000°C, and the ^{39}Ar fraction was over 70% of the total release. The age could be caused by an excess argon during initial stage of zircon formation or a secondary event. Because of localized nature of excess argon, it tends to produce various ages in the same area rather than a uniform age. The facts that the similar age was observed in another grain, and that the fraction is above 1000°C suggest that the age may have been preserved since the formation of the zircon. The results from other area will be discussed together.

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

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

Ar/Ar Geochronological analysis of Paka volcano, northern Kenya Rift

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The northern Kenya Rift constitutes one of the most prominent examples of young volcano-tectonic processes in a continental rift system. However, the limited amount of geochronologic information on different generations of lava flows and associated eruptive centers, fissures, and extensional faults has been a major problem in assessing the degree of activity in the inner trough of the rift. Here we report on 30 new ⁴⁰Ar/³⁹Ar ages of lava flows associated with the evolution of the composite Paka caldera-shield volcano in the northern sector of the Kenya Rift.

Paka is a composite caldera-shield volcano. It rises ~650 m above the rift floor and covers an area of ~280 km² with lava flows and pyroclastics of trachyte, mugearite or basalt (Dunkley et al, 1993). Dunkley et al. (1993) suggested that the principal volcanic eruptions on Paka occurred at 0.4 Ma, 0.2 Ma and 10 ka based on their four ⁴⁰Ar/³⁹Ar age determinations. The relationship between the pristine morphology of the summit crater and associated fault systems parallel to the rift orientation suggests that the volcano-tectonic activity at Paka is intimately associated with deformation processes in the inner rift trough, the youngest sector of volcano-tectonic processes in the rift. Thus, knowledge of the timing of volcanic eruptions and the generation of faults is crucial for a rigorous assessment of the youngest tectono-magmatic activity along the volcano-tectonic axis of the northern Kenya Rift.

Thirty-two samples were collected, mostly from lava flows or pyroclastic deposits, for ⁴⁰Ar/³⁹Ar dating at the geochronology laboratory at the University of Potsdam. Sampling was carried out based on the geologic map of Dunkley et al. (1993) and the morphology of lava flows on and around the flanks of Paka. Concentrated groundmass parts of the rocks after acid treatment were sent to Oregon State TRIGA Reactor for neutron activation of samples for four hours at the CLICIT facility, then ⁴⁰Ar/³⁹Ar analyses were performed at Potsdam. In most cases, high-resolution ages were successfully obtained that are broadly consistent with Dunkley's ages after careful evaluation of age spectra and isochrons obtained by stepwise-heating with a CO₂ laser.

A total of thirty ⁴⁰Ar/³⁹Ar ages was obtained, except two negative ages. The age data show protracted volcanic activity during the past 0.6 Ma years, and also three phases of pronounced volcanic activity: (I) between 0.428 and 0.372 Ma, (II) between 0.160 and 0.126 Ma, and (III) between 0.039 and 0.012 Ma (Fig. 1). Based on these three periods, all rocks were grouped in three periods, as (1) 0.6-0.35 Ma, (2) 0.35-0.1 Ma and (3) 0.1-0 Ma. In addition, we obtained geochemical information of the samples and their spatial distribution was investigated. We found that several basaltic lava flows of Dunkley et al (1993) were trachyte or mugearite flows; in addition, we conclude that some stratigraphic positions should be modified. During period (1), only trachyte and mugearite erupted on the western rift floor and the northeastern flank of Paka; instead, the younger trachytes erupted more along a N-S trend on the flank of Paka. Many normal faults affecting flows are only found on the older lava flows in northeastern side of the volcano, which belong to period (1). The trace element ratio of Nb/Zr clearly shows different ratios among the three age groups, which implies a gradual change of magma composition over time. Overall, our new age data are meaningful and consistent with field observations of superposition and cross-cutting relationships and thus help to develop a structural evolution model for the inner trough region of the northern Kenya Rift.

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

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

SGC51-03

Room:A04

Time:May 24 15:45-16:00

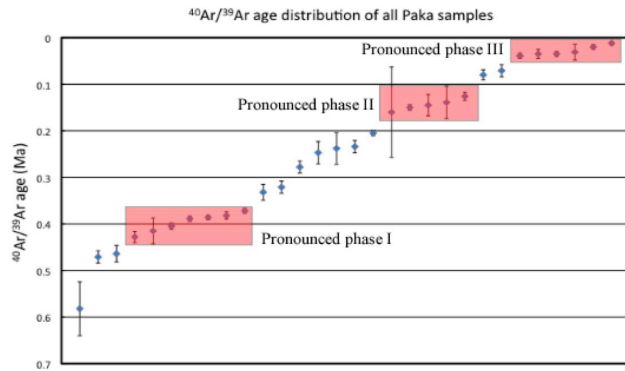


Fig 1. Distribution of $^{40}\text{Ar}/^{39}\text{Ar}$ ages of Paka samples and three inferred periods of pronounced volcanic activity.

Quantifying seawater assimilation across a single MORB pillow

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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 assimilate 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 assimilate 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

Keywords: Halogen, Noble gas, Seawater contamination, MORB

Halogen analysis of mantle xenoliths by extending Ar-Ar and I-Xe dating methods

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An extension of the Ar-Ar and I-Xe dating methods enables us to determine trace amount of halogens (Cl, Br, and I), K, Ca, Ba, and U. These elements are converted to corresponding isotopes of Ar, Kr, and Xe by neutron irradiation in a nuclear reactor. Noble gases in the irradiated samples are analyzed by noble gas mass spectrometry and the conversion factors from parent elements to noble gas isotopes are determined by analyses of standard samples [1]. The detection limits of this technique for Cl, Br, and I are 10^{-10} to 10^{-11} , 10^{-13} to 10^{-14} , and 10^{-14} to 10^{-15} mol, respectively, which are several orders of magnitude lower than those of conventional methods. Using this method, we are investigating halogen geochemistry in the mantle.

Halogens, except for F, are incompatible elements that are strongly partitioned into aqueous fluids. They are concentrated at the Earth's surface in distinct compositions and scarce in the mantle. These features enable us to trace water subducted into the mantle using halogens. Halogens are also thought to play an important role in transporting LIL (large-ion lithophile) elements. Enrichment of LIL elements in arc magma is explained by addition of slab-derived aqueous fluids. These elements are up to 10 times more strongly partitioned into aqueous fluids when the fluids contain halogens [2]. Because of these geochemical characteristics, halogens are among the most important elements in slab-derived fluids. The fluid inclusions of mantle xenoliths provide the most primary information on slab-derived fluids supplied to the mantle wedge. However, the halogen concentrations in such materials are too low to analyze by conventional methods and thus little research has been conducted on them.

We determined the halogen concentrations in mantle xenoliths from various geological settings: Western-Pacific subduction zones (Kamchatka, the Philippines, and N. Japan), Europe (Eifel), and North America (San Carlos and Kilbourne Hole). The halogen compositions of fluid inclusions were obtained by crushing extraction of noble gases. Melting extraction was also conducted to obtain those of whole samples.

The H₂O-rich fluid inclusions of the mantle xenoliths from volcanic fronts of subduction zones show high I/Cl ratios with relatively constant Br/Cl ratios, which are similar to those of marine sedimentary pore fluids (seawater trapped in pores of deep-sea sediments) [3]. Halogen compositions obtained from melting of the same samples show a small addition of Cl; however, they are still similar to sedimentary pore fluids. Similar signatures were found in mantle peridotites from the Sanbagawa metamorphic belt, suggesting subduction of sedimentary pore-fluid-derived halogens thorough incorporation into hydrous minerals such as serpentine in the subducting oceanic lithosphere [4]. Although the halogen concentrations are lower, some mantle xenoliths from rear-arc regions of subduction zones also show sedimentary pore fluid-like signatures. The I/Cl and Br/Cl ratios of mantle xenoliths from intraplate settings (Europe and North America) show common correlation, suggesting halogen fractionation from MORB source-like composition.

The halogen signatures of mantle xenoliths from subduction zones suggest that subduction of sedimentary pore-fluid-derived halogens [4] significantly extend into the mantle beneath volcanic front. These subducted halogens also extend to rear-arc regions, although their influence is small. The correlated I/Cl-Br/Cl ratios of mantle xenoliths from intraplate settings may have resulted from a metasomatic event, which could generally take place in subcontinental lithospheric mantle.

[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.

Keywords: halogen, noble gas, mantle xenolith

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 (Niijima, 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.

[1] Sumino et al. (2010) *Earth Planet. Sci. Lett.* 294, 163-172. [2] Sumino et al. (2011) *Mineral. Mag.* 75, 1963. [3] Holland & Ballentine (2006) *Nature* 441, 186-191. [4] Kendrick et al. (2011) *Nature Geosci.* 4, 807-812. [5] Turner (1965) *J. Geophys. Res.* 70, 5433-5445.

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

SGC51-07

Room:A04

Time:May 24 17:00-17:15

Noble gas isotopes; Solar evolution parameter

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Noble gas isotopic parameters are essential reference parameters for planetary evolution.

Keywords: noble gas isotopes, planetary evolution, meteorite

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].

References: [1] Okazaki et al. (2015) submitted to Earth Planet. Space. [2] Noguchi et al. (2015) this issue. [3] Loree and Radziemsky (1981) Plasma Chem. Plasma Process. 1, 271-279. [4] Gilmour et al. (1994) Rev. Sci. Instrum. 65, 617-625. [5] Strashnov et al. (2011) J. Anal. At. Spectrom. 26, 1763-1772.

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

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 grains 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.

References: [1] Noguchi et al. (2011) Science 333, 1121-1125. [2] Noguchi et al. (2014) Meteorit. Planet. Sci. 49, 188-214. [3] Matsumoto et al. (2013) 44th Lunar Planet. Sci. Conf. abstr. #1441. [4] Thompson et al. (2014) Earth Planet. Space 66, 89-99. [5] Keller and Berger (2014) Earth Planet. Space 66, 71-78. [6] Noguchi et al. (2014) Earth Planet. Space 66, 124-134. [7] Nagao et al. (2011) Science 333, 1128-1131. [8] Heber et al. (2009) Geochim. Cosmochim. Acta 73, 7414-7432.