

IntCal13は何が画期的なのか Why is the IntCal13 special?

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放射性炭素年代が較正を必要とすることは広く認識されている。世界のいくつかの研究グループが較正モデルを提案しているが、その中でもっとも広範に用いられているのは IntCal と呼ばれるモデルである。IntCal は 1998 年に最初のバージョンが公表され (IntCal98)、その後 2004 年 (IntCal04)、2009 年 (IntCal09) と改訂を重ねてきた。だが 2013 年に公表された最新版の IntCal13 は、それまでの IntCal とは大きく一線を画す画期的なモデルであると評価されている。いったい IntCal13 の何が特別であるのか、また IntCal13 とそれ以前のデータセットとの比較から何が明らかになったのか、主として放射性炭素年代測定の専門家以外を対象として概説する。

キーワード: IntCal13, 放射性炭素年代測定, 放射性炭素年代較正, 年縞堆積物, 水月湖, 海洋リザーバー効果

Keywords: IntCal13, Radiocarbon dating, Radiocarbon calibration, Varved sediment, Lake Suigetsu, Marine reservoir effect

14C年代値に対する酸-アルカリ-酸洗浄法のアルカリ洗浄段階の影響評価 An evaluation of the effect on 14C dating (AMS) by alkaline treatment of the ABA method on charcoal sample

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地球科学や考古学の分野では、¹⁴C年代測定に用いる炭化物の前処理に、酸-塩基-酸処理法 (Acid-Base-Acid: ABA method or Acid-Alkali-Acid; AAA method) が広く用いられている。しかし、酸-塩基-酸 (Acid-Base-Acid: ABA) 処理法は¹⁴C年代測定法における木炭試料の前処理の基本であるにもかかわらず、同法の処理条件についての化学的指標に基づく研究例は少ない。そこで本研究は、¹⁴C年代測定法の問題点として、従来、詳細は未解明であった前処理法による誤差の発生を明らかにすることを目的とする。発表者はこの目的の為に3種類の実験を行った。第一に年代値の再現性確認実験。第二に最適なアルカリ洗浄時間の確認実験。第三に洗浄に最適なアルカリ溶液の濃度の確認実験である。

第一の結果: NaOH 洗浄済みの試料の年代値群は、 $T=0.45$ (自由度 3; 5% 危険率: $T > 12.59$) と高い収束性を示すのに対して、NaOH 未洗浄試料の年代値は $T=10.74$ (自由度 4; 5% 危険率: $T > 9.49$) と発散が大きく、NaOH 未洗浄試料 5 試料の中で有意の差が現れた。

第二の結果: 目視によってアルカリ抽出終了と判断された後にも、三次元蛍光測定が NaOH 溶液中にフミン酸を検出し、かつ、¹⁴C年代測定結果 (渥美ほか, 2009) はフミン酸による影響を示している。さらに、これらの結果はアルカリ抽出終了判定に目視は不適切であり、かつ、三次元蛍光測定が溶存有機汚染物質の存在を監視する上で効果的であることを示唆している。

第三の結果: ある考古遺跡中の同層準の 3 木炭試料を 8 つの別々の濃度の NaOH 溶液で洗浄した試料の年代測定結果を χ^2 検定で評価した。その結果、1.2 mol/l で処理した試料間で最小値を示した。三次元蛍光測定の結果では 2.1 mol/l の NaOH 洗浄溶液中の腐植物質の抽出特性と 0.5~0.001 mol/l の溶液の抽出特性との間に明確な傾向の差が出た。すなわち、0.5~0.001 mol/l NaOH 溶液は低励起光波長領域の汚染の抽出効率が相対的に弱い。この現象が年代値の収束性の差を生んでいると考えられる。結論として、¹⁴C年代測定の前処理には 1.0~1.5 mol/l NaOH 溶液の使用を推奨する。

これらの結果は、いずれも前処理の化学的条件の差異が引き起こすフミン酸の残留度の差が¹⁴C年代値に影響を及ぼすことを示している。

キーワード: 放射性炭素年代測定法, 酸-アルカリ-酸前処理法, 三次元蛍光分析, フミン酸, 木炭試料
Keywords: 14C dating, ABA pretreatment, 3-D fluorescent spectroscopy, Humic acid, Charcoal sample

沖縄トラフ海底熱水域の熱水性鉱石中の重晶石のESR及び放射非平衡による年代測定 Dating of sea-floor hydrothermal barite collected at the Okinawa Trough by ESR and radioactive disequilibrium

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The temporal change of submarine hydrothermal activities has been an important issue in the aspect of the evolution of hydrothermal systems which is related with ore formation (Urabe, 1995) and biological systems sustained by the chemical species arising from hydrothermal activities (Macdonald et al., 1980). Dating methods using disequilibrium between radioisotopes such as U-Th method (e.g. You and Bickle, 1998), ²²⁶Ra-²¹⁰Pb and ²²⁸Ra-²²⁸Th method (e.g. Noguchi et al., 2011) have been employed for such studies.

Okumura et al., (2010) made the first practical application of ESR (electron spin resonance) dating technique to a sample of submarine hydrothermal barite to obtain preliminary ages, while Kasuya et al. (1991) first pointed out that barite can be used for ESR dating. Toyoda et al. (2011) determined the optimum ESR condition while Sato et al. (2011) confirmed that the signal is thermally stable enough for an age range of several thousand years. Takamasa et al. (2013) obtained U-Th and ESR ages which are roughly consistent with each other.

The samples were taken by research cruises operated by JAMSTEC. Barite (BaSO₄) was extracted from hydrothermal sulfide chimney samples taken from two sites at the Okinawa Trough. Blocks of sulfide deposits were cut into pieces, and about 2.0g was crushed. The samples were soaked in 12M hydrochloric acid, left for approximately 24 hours. Then, 13M nitric acid was added. Finally, after rinsing in distilled water, the sample was filtered and dried. Impurities were removed by handpicking. An X-ray diffraction study was made to confirm that the grains are pure barite. After gamma-ray irradiation at Takasaki Advanced Radiation Research Institute, Japan Atomic Energy Agency, they were measured at room temperature with an ESR spectrometer (JES-PX2300) with a microwave power of 1mW, and the magnetic field modulation amplitude of 0.1mT. The bulk Ra concentration was measured by the low background pure Ge gamma ray spectrometer. Assuming that Ra is populated only in barite, the dose rate was calculated with the alpha effectiveness of 0.043 (Toyoda et al., 2012), where the decay of Ra (a half life of 1600 years) was also taken into account.

The obtained ages range from 4.1 to 16000 years, being consistent with detection of ²²⁸Ra in younger samples and radioactive equilibrium/disequilibrium between radium and daughter nuclei. The variation of the ages within each sample is mostly within the statistical error range. The relative order of the ages is consistent with the result of ²²⁶Ra-²¹⁰Pb method, where the difference in absolute ages would be explained by several hydrothermal events that form the chimney. It was found that Yoron Hole field is the youngest, then, Daiyon-Yonaguni Knoll field, Hatoma Knoll field, being nearly equal to Iheya North Knoll field, then Izena Hole field, which is consistent with the direct observation from the submersible.

キーワード: 重晶石, 海底熱水活動, 電子スピン共鳴, 年代測定

Keywords: barite, hydrothermal activities, electron spin resonance, dating

方解石の熱ルミネセンス特性 Thermoluminescence property of calcite

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地球科学において用いられる各種現象に対しての時間軸を与える要素を年代と呼ぶ。年代値をもとにして、地球や惑星さらには人類の進化等をさぐる事が可能である。そのため年代値は地球科学において最も基本的なデータの1つである。

熱ルミネセンス年代測定法とは、放射線を受けた鉱物を加熱するときに出るルミネセンスの量から年代を算出する方法である。炭酸塩鉱物を用いた熱ルミネセンス年代測定は、¹⁴C年代測定で欠落している年代を補填できることから、利用されてきた。

しかし、方解石の熱ルミネセンス年代測定には様々な問題がある;例えば、加熱中の相変化による感度変化やフェーディング、各放射線(α 線、 β 線、 γ 線やX線)に対する熱ルミネセンス特性の差などの問題がある。

本研究では、方解石熱ルミネセンス年代測定法の精度確度を向上させることを目的とし、微量元素濃度の異なる様々な方解石(フィリピン, モンゴル, 合成方解石)を用い以下の研究を行った。

1. 各方解石のX線誘起熱ルミネセンス特性の測定。
2. 各放射線に対する方解石熱ルミネセンス特性の差を測定(X線に対する α , β , γ 線のルミネセンス効率をa-x-value, b-x-value, c-x-valueとする)。
3. 各不純物濃度と方解石ルミネセンス特性の相関関係を考察する。
結果は以下に示す。
 - i) ほとんどの方解石熱ルミネセンスは80と230℃のピークを示す。
 - ii) 方解石熱ルミネセンスの80℃ピークはフェーディングの影響を受ける。
 - iii) a-x-value, b-x-value, c-x-valueはMg, Mn, Fe, Srと相関関係を示す
 - iv) Feにはルミネセンス抑制効果が存在する。

Feは方解石熱ルミネセンスに大きな影響を与える。また方解石熱ルミネセンスの特性は複数の要因(Mg, Mn, Sr)によって決定されることが考えられる。様々な不純物濃度を示す方解石の分析を行い不純物濃度と方解石熱ルミネセンス特性の関係を評価することが必要である。

キーワード: 熱ルミネセンス, 方解石, 年代学

Keywords: thermoluminescence, calcite, dating

原子間力顕微鏡によるジルコンの観察：フィッショントラック？ それとも α リコイルトラック？

Zircon observation by atomic force microscope: Fission track or alpha recoil track?

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フィッショントラック (FT) 法は鉱物中に含まれる ^{238}U が自発核分裂を起こすことによって生じた損傷の計数に基づく年代測定法である。FT の密度が高くなるにつれ FT 同士が重なり合い計数が困難になるため、現在行われている光学顕微鏡を用いた FT 法では、必要なエッチング時間と分解能の限界により、測定できる密度がある程度制限される。原子間力顕微鏡 (Atomic Force Microscope: AFM) はナノオーダーの高い分解能を有し、より高密度の FT を計数することができる。しかし密度が高くなるのに合わせてエッチング時間を短くすると、期待される年代から予測される FT 数より多いトラックが計数される。これはエッチングが短いために、 α リコイルトラックと FT の形状の違いがでるに至らない状態で観察することになり適切な計数が行えないことが原因であると思われる。一方若い火山岩から採取したジルコンには、古いジルコンで観察される α リコイルトラックが原因と思われる表面構造がなく、そこでまれに見つけられる、10nm 深さの、計数可能な凹は α リコイルトラックである可能性があり、年代測定に結びつけられるかもしれない。

キーワード: ジルコン, フィッショントラック, アルファリコイルトラック, 原子間力顕微鏡

Keywords: zircon, fission track, alpha recoil track, atomic force microscope

三波川エクロジヤイト中フェンジヤイトの過剰アルゴン：沈み込み帯変成作用におけるアルゴン挙動についての制約
Excess argon in phengite from the Sanbagawa eclogites: Constraints on argon behavior during subduction zone metamorphism

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K-Ar system dating of phengitic mica is a powerful tool to determine cooling ages of HP/UHP metamorphic rocks. However, discordant ages in a same metamorphic unit have been often reported, particularly from rocks in Alpine-Himalayan type collisional metamorphic belts. For example, UHP-metamorphosed continental crust materials of the Dola Maira massif (western Alps, Italy) show the discrepancy due to the existence of excess argon in metamorphic minerals that has been inherited from the precursor rocks with polyphase metamorphic records. Over the last two decades, we have addressed an excess-argon free hypothesis in oceanic petrogenesis of Pacific-type convergent margins. According to the hypothesis, metamorphosed oceanic materials in Pacific-type HP metamorphic belts with only a monophase metamorphic records do not contain significant amount of excess ⁴⁰Ar; in other words, the K-Ar system in syn-metamorphic phengitic mica is significantly reset during fluid-induced metamorphic recrystallization at a Pacific-type convergent margin. Well-documented geological examples are of schists from Sanbagawa, Suo and Renge metamorphic belts in SW Japan, and from Otago metamorphic belt in New Zealand. Ar-Ar phengite analyses of HP-UHP metamorphosed oceanic lithologies of the Lago di Cignana (western Alps, Italy) also show negligible excess ⁴⁰Ar in eclogite-facies syn-metamorphic phengitic mica.

In the year 2000, as a preparation to guide participants for the IEC Conference in Japan, we have determined K-Ar ages of phengite and paragonite from the eclogite-facies Sanbagawa metamorphic rocks in Shikoku; the twenty-two results were obtained from four localities including Seba (84-89 Ma), Gongen (123-136 Ma) and Western Iratsu (78-80 Ma), and Kotsu/Bizan (82-88 Ma). Excepting for the quartz-rich kyanite eclogite from Gongen (GO), phengite and/or paragonite yields similar cooling-age ranges of metasedimentary rocks of the Sanbagawa metamorphic rocks in central Shikoku. Phengite K-Ar ages of GO eclogites are significantly older than syn-metamorphic zircon U-Pb ages at the same unit. These old ages are interpreted as the presence of excess ⁴⁰Ar in phengitic mica. The bulk-rock compositions of GO eclogites suggest a sedimentary protolith such as greywacke. When, where and how has the excess argon been trapped in phengite crystals? Considering the geological fact that the GO eclogites are closely associated with the Higashi-Akaishi (HA) meta-peridotite body, the false age obtained from phengite were likely attributed to an interaction between the meta-sediment (GO eclogite) and the meta-peridotite (HA peridotite) at eclogite-facies depth. We postulate that the fluids exchange between deep-subducted sediments and mantle material enhanced a hydration of peridotite and mantle-derived noble gas (including extreme ⁴⁰Ar) was diffused from mantle material to the sediments. During the exhumation of them, the rigid HA peridotite might have prevented a ductile deformation of GO eclogite and consequently mantle-derived argon gained from HA peridotite in GO eclogite might have been inherited by the limited-argon-depletion due to less deformation. This is not only very rare example of false K-Ar age of metamorphosed oceanic materials but also remarkable observation to explain argon behavior during sediments/peridotite interaction at a deep portion of subduction zone environment.

キーワード: 三波川帯, エクロジヤイト, フェンジヤイト, 過剰アルゴン
Keywords: Sanbagawa belt, eclogite, phengite, excess argon

短寿命放射性同位体系列を用いた初期地球分化の研究 Deciphering early Earth's differentiation using short-lived isotope systematics

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Knowledge of the timescale and nature of early Earth's differentiation is central to understanding the evolution of the young Earth. Here I discuss short-lived isotope systematics of terrestrial samples that extended our knowledge of early Earth's differentiation. Recent high-precision W isotopic studies revealed positive ^{182}W anomalies of up to 0.15 epsilon unit in ca. 3.8 Ga Itsaq rocks from West Greenland and 2.8 Ga Kostomuksha komatiites from Russia. I explored the geologic significance of the ^{182}W anomalies by combining with trace element and other isotopic data. In this context, the W isotopic data are interpreted to reflect early silicate differentiation events on Earth. Under the assumption that the bulk silicate Earth has a 5% higher Sm/Nd than the chondrite average, the ^{182}W - ^{142}Nd - ^{143}Nd chronometry constrains the age of the source mantle differentiation for the Itsaq samples to 4.53-4.49 Ga. The age may reflect the timing of silicate differentiation during a sequence of magma ocean solidification.

キーワード: 冥王代, 初期分化, 消滅核種, 非コンドライト質地球
Keywords: Hadean, early differentiation, extinct radionuclides, non-chondritic Earth

台湾中央山脈東部、豊田ネフライトの形成年代：NanoSIMSを用いたジルコンの低温リム (<20 μm) の年代測定
Formation age of Fengtien Nephrite, Taiwan: Dating low-temperature thin (<20 μm) zircon rims by NanoSIMS

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Nephrite in the Fengtien area is associated with serpentinites within the subduction-accretionary complex in eastern part of the Central Mountain Range, Taiwan. In addition to nephrite, there are also other metasomatic rocks, such as diopsidefels and epidotite, present between serpentinites and their country rocks (metapelites and metapsammites). Among these metasomatic rocks, diopsidefels is the most common one observed, while nephrite and epidotite are less frequently present in association with diopsidefels. When all these rocks are present at one outcrop, the common lithologic sequence is serpentinite-nephrite-diopsidefels-epidotite- metasedimentary rocks. Nephrite, diopsidefels and epidotite were interpreted resulting from fluid-rock (serpentinite+country rocks) interactions during subduction metamorphism. Field occurrence and petrographic observations clearly showed that while nephrite and diopsidefels are mainly metasomatic products after serpentinite, epidotite is after metapsammite. The formation temperature has been estimated to be 300 - 400 °C based on regional geology and thermodynamic calculations by previous studies. Timing of these metasomatic processes, however, has not been constrained, although the hosting subduction-accretionary complex was thought to be of late Cretaceous in age due to paleo-Pacific subduction beneath the Eurasia continent and to be correlated with the Sambagawa belt in Japan. Zircons were separated from one epidotite sample in this study. Most of these detrital zircons were shown to have a thin zircon rim, which is less than 15 - 20 μm in thickness. These zircon rims were considered to be newly formed during metasomatic interactions between serpentinite and country rocks, which also led to nephrite/diopsidefels/epidotite formation. The CAMECA NanoSIMS NS50 at AORI, the University of Tokyo was employed to date these low-temperature thin zircon rims with a ~ 5 nA O- primary beam confined to a ~ 15 μm diameter. Sample surface was pre-ablated for 5 minutes to remove the surface Au coating and any possible surface contaminants. Data acquisition time was 500 seconds. The resulting $^{238}\text{U}/^{206}\text{Pb}$ - $^{204}\text{Pb}/^{206}\text{Pb}$ inverse isochron gave a young age of 3.3 ± 1.7 Ma (MSWD = 2.1, n = 5). The date clearly showed that the Fengtien nephrite would have formed during the (initial) exhumation of the subduction-accretionary complex, which should be of late Cenozoic in age related to subduction of the South China Sea plate beneath the Luzon arc. The present study gave a good example that NanoSIMS is able to date zircon rims with a thickness about 15 μm formed under low temperature conditions only a few million years ago. The instrument has a great potential in future studies dating various low-temperature hydrothermal, metasomatic or metamorphic zircon overgrowths.

キーワード: NanoSIMS, ジルコン, U-Pb 年代測定, ネフライト, 中央山脈, 台湾

Keywords: NanoSIMS, zircon, U-Pb dating, Nephrite, Central Mountain Range, Taiwan

高精度 U-Pb ジルコン年代測定における結晶形態観察および微量元素存在度分析の意義：石鎚コールドロンに産する火成岩類を例に Significance of external morphology and zircon chemistry for precise U-Pb zircon dating

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Improvement of U-Pb zircon dating by microbeam analysis has been provided opportunity of discussion about more detailed geological events. Recent analytical precision of less than 2% at Paleogene zircon allows us to investigate shorter period events such as crystallization differentiation in magma chamber. However, the highly precise U-Pb age data yield an importance of confirming their accuracy and assaying disturbance of U-Pb system and incorporation of exotic components. In this study, we introduce data processing method of the highly precise ages based on zircon morphology, trace element abundances as well as statistics.

The precise U-Pb zircon dating by using a sensitive high-resolution ion microprobe (SHRIMP II) at National Institute of Polar Research, Japan, was applied to igneous rocks of the Tertiary Ishizuchi Cauldron in the Setouchi volcanic belt of Miocene age in northwestern Shikoku. A primary ion beam of about 10 nA was used to sputter an analytical spot of about 40 μm diameter. A retardation lens system was utilized as a means to increase signal-to-noise ratio, and a secondary ion optics including slits of source and collector was adjusted to maximum transmission of the secondary ion under suitable mass resolution avoiding isobaric interferences on Pb isotopes. The surfaces of grain mounts were carefully washed with diluted HCl and ultra pure water to remove Pb contamination. A correction for common Pb was made on the basis of the measured ^{204}Pb and the model for common Pb composition.

Weighted mean ages were calculated from $^{206}\text{Pb}/^{238}\text{U}$ ratios corrected by ^{207}Pb . In order to ensure the accuracy of U-Pb age, age known zircon, OD-3, was analyzed together with unknown sample. Concentrations of Hf and rare earth element (REE) in zircons were also measured at the same analytical spot of U-Pb dating by SHRIMP.

Zircon grains from the Bansyodani-biotite-rhyolite were divided to two types based on the external morphology: sharply euhedral type and relatively rounded edge of prism and pyramid type. $^{206}\text{Pb}/^{238}\text{U}$ data of whole zircon grains were widely scattered beyond analytical uncertainty and show a weighted mean of 14.78 ± 0.18 Ma (mean square weighted deviation, MSWD: 3.4). On the other hand, the euhedral zircons yielded the weighted mean of 14.21 ± 0.19 Ma (MSWD: 1.0), whereas the relatively rounded zircons were older than the euhedral zircons, which suggests the incorporation of exotic components.

Zircon chemistry supported the classification by the morphology and the U-Pb dating. An average of Hf contents of the euhedral zircons were 9523 ppm ranging from 8883 to 10496 ppm and those of the relatively rounded zircons were 8475 ppm ranging from 7616 to 8803 ppm. Hf contents of the euhedral zircons were higher than those of relatively rounded zircons. C1 chondrite-normalized REE patterns of the euhedral zircons were characterized by a large fractionation between light REE and heavy REE, large positive Ce anomalies and large negative Eu anomalies. In contrast, those of the relatively rounded zircons were enrichment of light REE, weaker anomalies of Ce and Eu. Difference of the zircon chemistry between the euhedral zircons and the relatively rounded zircons reflects source melt composition. Therefore, the external morphology, Hf content, and REE abundance are useful criteria for the data processing of the highly precise U-Pb age data.

ジルコンの外部面を用いた LA-ICP-MS による U-Pb 年代測定：隠岐島前火山への適用 LA-ICP-MS U-Pb dating of Oki Dozen volcano using non-polished zircons

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LA-ICP-MS によるジルコンの U-Pb 年代測定法は、今や広く使われ、大いに成功している。この方法は、通常、ジルコンを研磨した状態で用いるが、これは、恐らく、common Pb や Pb loss といった結晶表面付近で生じるマイナス要因を避けるためであり、また、SHRIMP のような極少量の体積（深さ 1~2 μm が掘削される）を消費する分析法では必須である。SHRIMP に比べると、LA-ICP-MS は格段に多くの体積を消費する（掘削される深さは 10 μm 以上）ため、このことは、逆に、ジルコンの表面から内部までを容易に年代測定できることを意味する。従って、研磨しないジルコンを利用すれば、結晶内部の inherited core の存在やジルコンのマグマ中での成長速度の検討にも適用可能と思われる。今回、研磨しないジルコンを対象に標準試料（Fish Canyon Tuff と OD-3）と隠岐島前火山の試料について LA-ICP-MS による U-Pb 年代測定を行った。ジルコンは波長 213nm の Nd-YAG レーザー（repetition rate: 10Hz, エネルギー密度: 4~5 J/cm²）で 30 秒間アブレーションを行った。エネルギー密度 5 J/cm² では、最終的な掘削深度は 27 μm になり、深さ 9-18 μm の ²⁰⁶Pb/²³⁸U 比を用いて年代値を求めた。その結果、研磨しないジルコンを利用した場合でも信頼性の高い年代が得られることが標準試料の年代値が一致することから確かめられた。また、隠岐島前試料は 6~7 Ma の年代を示し、既存の K-Ar 年代（5.4~7.4 Ma）と一致もしくは少し古い年代が得られたことを報告する。

キーワード: U-Pb 年代測定, ジルコン, LA-ICP-MS, 隠岐島前火山

Keywords: U-Pb dating, zircon, LA-ICP-MS, Oki Dozen volcano

東北地方太平洋沖地震の余震による大気のAr同位体変化 Crustal noble gases anomaly associated with fault movement and aftershock the 3.11 Northeast Japan Earthquake

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Noble gases have unique characteristics that they are rarely combined with other chemicals as their very stable nature. Because its main reservoir is atmosphere, their isotopic composition is well defined and believed to be uniform all over the world insensitive to disturbance from anthropogenic and/or natural emission of geologically trapped noble gases in the earth interior. Based on our preliminary friction experiment, however, detectable amount of noble gases seem to be emitted accompanied with a fault motion (Sato et al., 2009). After the extreme Northeast Japan Earthquake occurred on March 11, 2011, extraordinary increase of seismic activity as numerous aftershocks e.g. over 4000 felt earthquakes in four months, which may be a source of non-atmospheric component preserved in the earth interior. In terms of anthropogenic component, Nuclear Power Plant) is a potential source, which is frequently monitored by radioactive species of noble gases.

We widely collected atmosphere samples all over Japan from Hokkaido, Honsyu, Chugoku and Kyusyu Is. The atmospheres have been sampled into vacuumed containers, Isotube®, at each sampling site to evaluate time-series changes. The elemental and isotopic compositions of the samples were analyzed mainly by quadrupole residual gas analyzers (RGA-200, SRS Co.) and partly confirmed by sector-type mass spectrometers (GVI-5400, GV instruments). In the duplicated analyses of the selected a few samples, the measured elemental and isotopic compositions were consistent within analytical uncertainties.

The relative elemental abundances were changed at least in heavier noble gases. Argon was enriched to pre-3.11 Earthquake atmospheres associating with a high $^{40}\text{Ar}/^{36}\text{Ar}$ ratio. It might be contributed by emission of crustal Ar at aftershock earthquakes, deformation and fault movements. In addition, a frictional melting was occurred in a >M5 earthquake as reported by Kanamori et al. (1998). Further, radioactive Ar isotopes (^{42}Ar and ^{39}Ar) were slightly abundant than those in "pre" 3.11 Earthquake atmospheres. These radioactive Ar isotopes were regarded to be detected limitedly in neutron irradiated geological samples especially in Ar - Ar dating. These altered atmospheric Argon isotopic composition in Eastern Japan area were observed until typhoon season.

Keywords: noble gas, 3.11 Northeast Japan Earthquake, aftershock earthquake, nuclear power plant disaster, Ar Isotope

Ar-Ar及びI-Xe年代測定法の拡張による極微量ハロゲン・希ガス多元素同時分析 Ultrahigh-sensitive simultaneous determination of halogens and noble gases by an extension of Ar-Ar and I-Xe methods

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Noble gas isotope ratios in various geochemical components in the Earth are significantly different, making them useful tracers to constrain origin of volatiles in the mantle. The development of noble gas mass spectrometry during the last two decades has enabled us to detect less than 10000 noble gas atoms (e.g., [1]).

An extension of Ar-Ar and I-Xe dating methods allows us to simultaneously determine trace amounts of noble gases, halogens, K, Ca, Ba, and U by use of ultrahigh-sensitive noble gas mass spectrometry on neutron-irradiated samples. This method has several advantages: (i) detection limits for halogens are three or four orders of magnitude lower than those of other conventional analytical methods, (ii) several components of different origin can be distinguished based on their relations with specific noble gas isotopes such as mantle-derived ³He and by using various noble gas extraction methods such as laser microprobe [2], and (iii) in-situ production of radiogenic noble gas isotopes (such as ⁴He and ⁴⁰Ar) after the entrapment of the noble gas component of interest in the sample can be corrected by the simultaneously determined their parent elements, such as U and K, when the age of the entrapment is known or can be assumed.

We have developed a new noble gas mass spectrometric system for this method based on an Ar-Ar and I-Xe dating system [3]. Accuracy and precision of our method were examined by analyzing GSJ and USGS reference materials, their original rocks, and scapolite standards [4] and by comparing the halogen data with those obtained with ion chromatography and ICP-MS followed by pyrohydrolysis extraction [5].

By using this method, we analyzed halogens and noble gases in exhumed mantle wedge peridotites and eclogites from the Sanbagawa-metamorphic belt, southwest Japan and those in mantle-derived xenoliths from Kamchatka and N. Philippines, in all of which relicts of slab-derived water are contained as hydrous mineral/fluid inclusions. The striking similarities of the observed noble gas and halogen compositions with marine pore fluids [6,7] challenge a popular concept, in which the water flux into the mantle wedge is controlled only by hydrous minerals in altered oceanic crust and sediment (e.g., [8]).

On the other hand, halogen ratios of olivines in lavas from the northern Izu-Ogasawara arc [9] indicate insignificant contribution to the mantle wedge of pore fluid-derived halogens. This implies a relatively small amount of the pore water subduction fluids would be released from the Izu slab at a sub-arc depth resulting in further subduction to great depths in the mantle, possibly resulting in the seawater-like heavy noble gas composition of the convecting mantle [10].

Based on the relation with ¹²⁹Xe produced from decay of short-lived nuclide ¹²⁹I during stepwise heating noble-gas extraction of the Allende and Shallowater meteorites, intrinsic I and U to the meteorites were distinguished from those of terrestrial contamination origin.

These results demonstrate that simultaneous determinations of noble gases, halogens, K, Ca, Ba, and U in mantle-derived rocks and meteorites provide important information about their origins.

[1] Sumino et al. (2001) *J. Mass Spectrom. Soc. Jpn.* 49, 61-68. [2] Sumino et al. (2008) *J. Volcanol. Geotherm. Res.* 175, 189-207. [3] Ebisawa et al. (2004) *J. Mass Spectrom. Soc. Jpn.* 52, 219-229. [4] Kendrick (2012) *Chem. Geol.* 292-293, 116-126. [5] Muramatsu & Wedepohl (1998) *Chem. Geol.* 147, 201-216. [6] Sumino et al. (2010) *Earth Planet. Sci. Lett.* 294, 163-172. [7] Kobayashi et al. (2013) *Mineral. Mag.* 77, 1484. [8] Schmidt & Poli (1998) *Earth Planet. Sci. Lett.* 163, 361-379. [9] Sumino et al. (2013) *Mineral. Mag.* 77, 2285. [10] Holland & Ballentine (2006) *Nature* 441, 186-191.

キーワード: 希ガス, ハロゲン, 質量分析, Ar-Ar年代測定法, I-Xe年代測定法

Keywords: noble gas, halogen, mass spectrometry, Ar-Ar dating, I-Xe dating

蔵王火山溶岩の感度法による K-Ar 年代測定 Unspiked K-Ar dating for lavas from Zao volcano

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蔵王火山は東北日本の火山フロントの中央部に位置する複成火山である。先行研究では、約 80 万年前に活動を開始し、主となる山体は約 30-10 万年前に形成され、その後、約 3 万年前から現在までの最新活動期が続くと考えられている。本火山は初めて K-Ar 法における質量分別補正の必要性が示された火山であり、約 50 試料の年代値が報告されているが、未調査地域も残っており、一部カリウム含有量が低い試料や過剰アルゴンの混入が疑われる試料等について層序や古地磁気データと矛盾する場合もあった。本研究では、蔵王火山の活動史の全体像を明らかにするために、これまで未調査だった地域を含めて調査・試料採取を行ない、感度法による K-Ar 年代測定を実施したので、その結果を報告する。

キーワード: 蔵王火山, K-Ar 年代測定
Keywords: Zao volcano, K-Ar dating

段階加熱による拡散実験と白雲母 Diffusion experiment by stepwise heating and muscovite

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一般に結晶水を含む鉱物のアルゴンの拡散パラメーターを決定するとき真空中での段階加熱実験は加熱中に結晶格子の破壊が起きるため正確な値が得られず不相当であるとされてきた。以前報告したレーザー段階加熱による単結晶黒雲母の結果では冷却速度を考慮すると実際の値と変わらない範囲に収まることがわかっている。一方、真空中での白雲母の拡散実験の結果は従来のデータとの不一致が大きく実用的な適用が難しいと考えられた。白雲母のレーザー加熱による実験での大きな問題は脱水反応もしくは構造相転移による脱ガスが 600°C 以上で急激に進行し、拡散現象では解釈できなくなる要素が大きい。真空中の実験の場合、拡散パラメーターを見積もる Arrhenius 実験では脱水の影響が顕著でない 600°C 以下でのデータを用いたが、拡散するガス量が少なく誤差が大きい問題があった。ほとんどの場合、活性化温度は 30-40 kcal/mole の領域であり、その値から得られる閉止温度は 300°C を超える値は少ない結果となった。むしろ 400°C 以上の高い温度は温度領域を 600-700°C にして急激な拡散を起こす状態でのデータから得られた。近年、熱水環境下での実験で活性化エネルギーは 63 kcal/mole、冷却速度、拡散半径に依存するが閉止温度は、400°C と見積もられている。(Harrison et al., 2009)。このデータは 600-700°C の値を使用し、構造的な変化が起きていないという保証はない。熱水環境下で仮に結晶格子が安定的に存在したとしても同じ温度領域で構造相転移がおきるとすれば拡散パラメーターは独立に分離できない。単に数学的側面からは Arrhenius plot で大きな活性化エネルギー E もしくは周波数因子 D_0/a^2 を得るには急な傾きすなわち急激な脱ガスが必要となる。これは実は拡散においてゆっくりとした振る舞い (E, D_0/a^2 が大きい) をすることは矛盾する領域で拡散現象を見ていることになる。一方、野外においては白雲母が高い閉止温度を示している証拠もみついている。すなわち単に実験室において条件をみただけでなく、野外での条件に基づく考察を平行して行う必要があり、白雲母の閉止温度の意味を再考する必要がある。

キーワード: 拡散実験, アルゴン, 閉止温度, 段階加熱, 白雲母
Keywords: diffusion experiment, argon, closure temperature, stepwise heating, muscovite

滋賀県余呉湖細粒堆積物のルミネッセンス年代測定と環境変動解析
Luminescence dating and analysis of environmental change of fine grained sediments
from Lake Yogo, Japan

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We applied optically stimulated luminescence (OSL), infrared stimulated luminescence (IRSL), post-IR IRSL (pIRIR) and ¹⁴C dating to the sediment core YG11-3 (294cm) from Lake Yogo, Japan. The fine grained quartz and polymineral sample are used for equivalent dose (D_e) estimation. As a result of several basically test, the preheat temperature of 200 °C for 10 s and a cut heat of 160 °C were suitable to all OSL measurements. The accepted aliquots are about 90 % per measurement discs and the range of D_e s are 0.3 ~3.5 (Gy). The bulk ¹⁴C ages are ca. 300 years older than these of plant residue. After subtracting this age difference from bulk ¹⁴C ages, the corrected ages agree with the OSL ages except the ages of sediments from some depths. Two excepted OSL ages are older than the corrected bulk ¹⁴C ages (YG11-3-245, YG11-3-343) and these layers include a lot of plant residue enough to analyze the plant residue ¹⁴C ages. It seems that these sediments from two layers have been transported quickly in muddy stream based on a temporary environmental event. Additionally, the result of the IRSL_{50/225} and pIRIR₂₂₅ age confirms the existence of this temporary event. By comparing the OSL ages with ¹⁴C, IRSL and pIRIR ages, the quartz from the small catchment area can be applied to reconstruct the age model of sediment core in Japan.

キーワード: OSL 年代測定, pIRIR 年代測定, 湖沼堆積物
Keywords: OSL dating, pIRIR dating, lake sediments

NanoSIMS を用いた太古代ジルコンの U-Pb 年代測定 -包有物中揮発性元素の測定を目指して U-Pb dating of Eoarchaeon zircon using a NanoSIMS -implication for the measurement of volatile in the inclusions

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太古代火成岩中の水素や硫黄などの揮発性成分の同位体比値を明らかにすることは、初期地球の内部進化を解釈する重要な手掛かりの内の一つである。火成岩中に産するジルコン中のアパタイトやガラスなどの包有物はそのような揮発性成分を保持していると期待されるが、これらはしばしばマイクロスケールで存在するため、高空間分解能な分析手法が求められる。同時に、そうした包有物が初生的な情報を残しているかを評価することも重要である。本研究では NanoSIMS50 を用いてこれらの問題へのアプローチを行っている。

測定は、カナダ、Nuvvuagittuq supracrustal belt のトーナル岩から分離されたジルコンを対象に行った。過去の研究で報告されているこのトーナル岩の年代は、LA-MC-ICP-MS を用いた U-Pb 年代測定法で 3661 ± 4 Ma である [1]。測定では、自形から半自形で結晶の長軸方向の長さが 50 から 200 μm 程度のジルコンを対象とした。いくつかのジルコンには直径 10 μm 以下のアパタイトやガラスの包有物がみられ、年代測定はこれら包有物を避けて行われた。

²³⁸U-²⁰⁶Pb 及び、²⁰⁷Pb-²⁰⁶Pb の 2 種類の年代測定を同スポットに対して行った。測定手法は Takahata et al.(2008) に準じた [2]。1 次イオンビームとして 5nA の酸素イオンを用いた。²³⁸U-²⁰⁶Pb の年代測定では、³⁰Si⁺, ⁹⁰Zr¹⁶O⁺, ²⁰⁴Pb⁺, ²⁰⁶Pb⁺, ²³⁸U¹⁶O⁺, ²³⁸U¹⁶O₂⁺ を多重検出器で同時に測定し、²⁰⁷Pb-²⁰⁴Pb の年代測定では ²⁰⁴Pb⁺, ²⁰⁶Pb⁺, ²⁰⁷Pb⁺ を一つの検出器で磁場を変化させながら測定した。

測定の結果、²⁰⁶Pb/²³⁸U 比は 0.4932 から 0.7993 とバリエーションをとり、²⁰⁷Pb/²⁰⁶Pb 比は 0.3052 から 0.3443 の値をとることが分かった。得られたそれぞれの値を Tera-Wasserburg コンコーディア図にプロットすると、過去の研究と良く一致する 3638 ± 19 Ma の年代が得られた。一方で、一部の試料において 2 つの年代値の不一致 (ディスコード) が見られた。こうした試料では鉛を失うような変成作用を経験しているため、包有物中の揮発性元素の始原性は失われている可能性が高いと考えられる。U-Pb 年代測定の結果をもとに、ジルコン中包有物の揮発性元素の分析を進めている。

[1] David et al., GSA Bulletin, 121, 150-163, 2009.

[2] Takahata et al., Gondwana Res., 14, 587-596, 2008.

キーワード: ウラン鉛年代測定, ナノシムス, ジルコン, 包有物, 太古代
Keywords: U-Pb dating, NanoSIMS, zircon, inclusion, Archaean

東南極ナピア岩体西部における原生代の年代的・地球化学的特徴 Geochronological-geochemical characterization of Proterozoic age, western part of the Napier Complex, East Antarctic

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The Napier Complex in East Antarctica has attracted considerable interest from a viewpoint of long Archaean crustal history from 3800 Ma to 2500 Ma (e.g., Harley & Black 1997) and >1000 °C ultrahigh-temperature (UHT) metamorphism in a regional scale (e.g., Sheraton et al., 1987; Harley & Hensen 1990). The timing of ultrahigh-temperature metamorphism is in argument either >2550 Ma or <2480 Ma (Kelly and Harley, 2005). However, some previous works reported relatively younger ages, such as 2380 Ma, ~2200 Ma, and ~1820 Ma (e.g., Grew et al., 2001; Owada et al., 2001; Suzuki et al., 2001, 2006; Carson et al., 2002; Hokada and Motoyoshi, 2006). In addition, Horie et al. (2012) reported similar ages in felsic orthogneiss from Fyfe Hills and quartzite from Mt. Cronus via zircon U-Pb dating. In this study, we try to characterize the "younger ages" in order to interpret thermal history after the UHT metamorphism in the Napier Complex.

A quartzo-feldspathic gneiss, YH05021606A, collected from Fyfe Hills by Y.H. during the field work at the 2004-2005 Japanese Antarctic Research Expedition was analyzed by using a high-resolution ion microprobe (SHRIMP II) at the National Institute of Polar Research, Japan. The zircon U-Pb ages of the YH05021606A sample are already reported in Horie et al. (2012). The sample shows multiple age peaks centered at ca. 3025, 2943, 2883, 2818, 2759, 2674, 2518, and 2437 Ma, and evidence of the "younger ages" has never been reported. In this study, primary ion beam was focused up to 10 μm in order to observe detailed zircon structure. The U-Pb analysis of zircon yielded similar age population to the previous work and revealed the "younger ages" of ca. 2273, 2195, 2106, and 1980 Ma. Distribution of the "younger ages" is consistent with those of a felsic orthogneiss, YH05021603A, in Fyfe Hills and those of a quartzo-feldspathic gneiss, YH05021701A, and a quartzite, YH05021701H, in Mt. Cronus (Horie et al., 2012). The "younger ages" in this sample could be found in overgrowth rim and single grain, which indicates that both of Fyfe Hills and Mt. Cronus had been affected by any geological events after the UHT metamorphism. Previous workers suggested that the ca. 2200 Ma age that they obtained for beryllium syn-metamorphic pegmatites reflects post-emplacement deformation and metamorphism (Grew et al., 2001), and a ca. 1930-1800 Ma U-Pb upper intercept age for zircons were affected by aqueous fluid from Paleozoic pegmatite (Carson et al., 2002). Horie et al. (2012) only suggests that these 2380-820Ma ages represent local fluid infiltration or a local deformation events. We will discuss about character of the "younger ages" zircon with trace element signature.

キーワード: 東南極, ナピア岩体, ジルコン, U-Pb 年代測定, 希土類元素, 変成作用

Keywords: East Antarctica, Napier Complex, zircon, U-Pb dating, rare earth element, metamorphism

変成炭酸塩岩中のストロンチウム、ネオジウム同位体を用いた古海洋の復元 —東西ゴンドワナ間のモザンビーク海— Sr and Nd isotope systematics of metacarbonate rocks as proxies for reconstructing extinct oceans: Mozambique Ocean

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Geochemistry of sedimentary rocks is widely used for understanding the depositional environment and tectonic setting, including source rock composition and paleo-ocean signature. In particular, chemically deposited carbonate rocks are directly precipitated from saturated seawater are supposed to hold key information of extinct paleo-oceans. An important geochemical tool that can lead to the identification of contemporaneous seawater is isotopic composition of strontium and neodymium in carbonate rocks, because these elements have distinct residence and mixing time in seawater and also characterized by surrounding continents.

In the Sør Rondane Mountains (SRMs), East Antarctica, metasedimentary rocks including metacarbonate rocks are widely distributed. These rocks were supposed to have formed in the paleo-ocean called as "Mozambique Ocean". SRMs are divided into two terranes, the SW and NE terranes, by the Main Tectonic Boundary (MTB). In the SW terrane, metaigneous rock that were formed at ca. 1000 Ma and metasedimentary rocks occur as main lithological units, which underwent metamorphic evolution along a anticlockwise *P-T* path, whereas the NE terrane is dominated by metasedimentary rocks, with a characteristic clockwise of *P-T* path. Additionally, metapelitic rocks in the SW terrane have similar detrital age population with the nearby metaigneous rocks, in contrast to those in the NE terrane show older detrital ages (ca. 3300Ma)(Osanaï et al., 2013). If it is possible to reveal the relationship between ocean and continents during depositional timing of carbonate rocks in both terranes, we will be able to put forward a model to explain the difference in depositional setting between SW and NE terranes. To achieve this, analyzed detailed study of Sr and Nd isotopic composition of metacarbonate and metamorphosed silicate rocks, such as pelitic, felsic, mafic and ultramafic rocks, from several important outcrops throughout SRMs were carried out. Based on these data, we discuss about the relationship with continent and depositional basin of carbonate sediments before the final amalgamation of Gondwana.

After geochemical screening for post-depositional alteration, using oxygen isotopes, trace elements and REE + Y patterns, strontium isotope chemostratigraphy was applied to the metacarbonate rocks from SRMs and depositional ages of 880-850 Ma and 820-790 Ma (late-Tonian and early-Cryogenian age) were estimated (Otsuji et al., 2013). Metacarbonate rocks in the Bratnipene and Tangarden regions in the SW terrane are showing typical seawater-rock mixing relationship in a ϵSr vs. ϵNd cross-plot indicating the deposition of metacarbonate rocks nearby meta-tonalitic and orthogneiss dominated continental arc. By contrast, the Perlebandet region exhibits an extremely different depositional setting of a seamount based on Nd model and depositional age and REE and ϵNd compositions. Moreover, the Balchen metacarbonate rocks show a signature of depositional setting surrounding a continent, based on the comparison of metacarbonate rocks with continental and oceanic derived rock units. A comparison of isotopic characteristics of Balchen carbonate rocks with the basement rocks from neighboring Gondwana regions suggested the presence of an ancient continent that is different from Kalahari and Dharwar Craton.

Thus, the Sr and Nd isotopic compositions of carbonate rocks deposited in the Mozambique Ocean have preserved important information about depositional setting of sedimentary rocks and relationship with surrounding basement and continents. In summary, geochemical proxies such as Nd and Sr isotopes of metacarbonate rocks can yield key information not only of paleo-oceans but also about the surrounding rocks during depositional timing, which can lead to a better understanding of oceanic closure during the formation of supercontinents.

Reference cited: Osanaï et al., 2013. PR, 234, 8-29. Otsuji et al., 2013. PR, 234, 257-278.

Keywords: Sr and Nd isotope ratios, metacarbonate rocks, the Sor Rondane Mountains, Mozambique Ocean, Gondwana

南インドダールワークラトン中の縞状鉄鉱層を用いた地球化学的研究 Geochemistry of Archaean Banded Iron Formations in the Chitradurga Schist Belt, Dharwar Craton, Southern India

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Banded iron formations (BIF) are marine chemical sediment rocks precipitated mostly in Archaean and early Paleoproterozoic between 2.7Ga and 2.3Ga. This time interval record profound changes in the redox state of the oceans and atmosphere, such as the Great Oxidation Event (GOE). We present here the geochemical data obtained from 3.0 Ga banded iron formation (BIF) in the Chitradurga Schist Belt, Dharwar Craton, Southern India. This region exposes the Archaean strata predominated by supracrustal greenstone belts, stratigraphically overlying the Peninsular gneiss. Chitradurga schist belt comprises of three important BIF layers distributed in the Bababudan and Chitradurga groups. We present here the salient geochemical characteristics and strontium and neodymium isotope results of the BIFs and discuss the depositional environment.

BIF contain very low content of Al₂O₃ (<1wt.% except 1 sample) indicating less detrital components. The PAAS-normalized REY patterns shows positive La and Eu anomaly, low concentration of rare earth element, depletion of light rare earth elements (LREEs) relative to heavy rare earth elements (HREEs). These features differ with other Archaean BIFs in terms of lack of positive Y anomaly. The large positive Eu anomalies in Archean BIF of Chitradurga schist belt attribute to high-T hydrothermal fluid fluxes (>250 °C), while the negative Ce anomaly reflects the lack of significant oxidizing agents.

Sr isotopic composition of BIF shows large variations suggesting post depositional alterations, whereas Nd isotope ratios gave consistent information. Most of the samples show $\epsilon\text{Nd}(3000\text{Ma})$ in the range of +2 to +4 and T_{DM} model age in accordance with sedimentation age. The $\epsilon\text{Nd}(3000\text{Ma})$ of depleted mantle is about +4, which suggests that most of the Chitradurga BIFs were deposited in an environment strongly affected by input from a depleted mantle. However samples with different REY pattern show higher $\epsilon\text{Nd}(3000\text{Ma})$ between +6 and +14 and their T_{DM} model age are not equal to the sedimentation age. The geochemical results thus suggest that the BIFs in the Chitradurga schist belt were deposited near possible ridges affected by hydrothermal activities.

キーワード: 縞状鉄鉱層, ダールワークラトン, 太古台, Nd 同位体

Keywords: Banded Iron Formations, Dharwar Craton, Archaean, Nd isotope