

U-Th radioactive disequilibria in sulfide minerals from sea floor hydrothermal systems

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Although the time scale of hydrothermal activity constrains the size of hydrothermal ore deposits and the evolution of chemosynthesis-based communities in a submarine hydrothermal system, limited systematic dating studies have been conducted.

Sulfide minerals from axial ridge with high spreading rate were mainly dated with radioactive isotopes with short half lives such as ^{210}Pb . ^{234}U - ^{230}Th ages were successfully obtained for sulfide minerals from off axial areas in the fast spreading rates with a spectroscopy (Lalou et al., 1985) and from the slow spreading mid-Atlantic Ridge using a thermal ionization mass spectrometry (You and Bickle, 1998). We will show the results of ^{234}U - ^{230}Th analyses with a MC-ICP-MS and also compare the results with ages obtained from other dating systems such as ESR dating.

Thorium is not soluble in sea water from which hydrothermal fluids originated, while uranium abundance is about 1 ppb. Thus when hydrothermal minerals precipitated without detrital component, $^{230}\text{Th}/^{234}\text{U}$ activity ratio is close to zero. In the minerals are kept in closed system $^{230}\text{Th}/^{234}\text{U}$ activity ratio increase with time and reaches secular equilibrium in 500ka. From $^{230}\text{Th}/^{234}\text{U}$ activity ratio, we can determine the age of the hydrothermal minerals.

Analyses are conducted as follows. A sulfide sample is decomposed with nitric acid after artificial tracers of ^{236}U and ^{229}Th are added for isotope dilution analyses. U and Th are separated from major elements with a solvent extraction chromatography resin, U TEVA resin (eichrom). U and Th are further purified with U TEVA resin and anion exchange resin (AG1-X8, Bio-rad). Isotope ratios of $^{234}\text{U}/^{238}\text{U}$, $^{236}\text{U}/^{235}\text{U}$, $^{230}\text{Th}/^{232}\text{Th}$, $^{229}\text{Th}/^{232}\text{Th}$ are measured to obtain radioactivity ratio of ($^{230}\text{Th}/^{234}\text{U}$). We can obtain precise ($^{230}\text{Th}/^{234}\text{U}$) ratio with several hundred milligram sample by MC-ICP-MS analyses.

Four hydrothermal sites of South Mariana Trough were dated yielding ages from <100 a to 10 ka. The results suggested that the oldest age from each site is correlated with the distance from the spreading axis of the Trough. The U-Th radioactive disequilibrium ages were roughly consistent with ESR ages.

Most of the samples from Okinawa Trough samples yielded younger age than South Mariana Trough. Some of them show inconsistent ages with ESR ages.

Sulfide minerals from some sites show large variations in U and Th abundances, suggesting an open system behavior.

We will report the results of leaching experiments in the presentation.

Keywords: U-Th radioactive disequilibrium, seafloor hydrothermal vent

Problems related to the numerical age in a time table

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To refer an age through the Earth's history, various kinds of time tables have been used and numerical ages are assigned. However, criteria to present such numerical ages are not always the same, depending on each time table and their values do not always represent effective numerical values.

Geologic time table has been constructed primarily on the basis of the stratigraphic classification, defined as relative ages. Then, numerical ages are assigned to each boundary between the two stratigraphic units based on the radiometric age data for rock and/or minerals which are included in each stratigraphic unit. However, in the geologic time table (ICS, 2009), three kinds of numerical ages are assigned depending on the age range. For the range of Precambrian ages, each geologic unit is defined based on the numerical age value. Hence, no error is included in these numerical values. For the range since Neogene to present, astronomical age values are assigned for each boundary. Since the errors in the astronomical age values are regarded to be too small to be assigned, no errors are attached in the numerical age values in this range. However, it should be noted that an astronomical age is a model age, whose uncertainties should be checked further. For the other range from the Paleogene to the Cambrian, the numerical age values are assigned with error for the boundary based on the radiometric age data.

In the case of magnetostratigraphic time table, it is constructed based on the paleomagnetic data for successive marine sediments. Numerical age values for each boundary are estimated based on interpolation of age values of some age standards, assuming a constant sedimentation speed. In the magnetostratigraphic time table by Cande and Kent (1995), numerical age values with 5 to 6 numerical figures are indicated. However, the effective numerical values for the age standards have only 3 numerical figures. Hence, even if the apparent numerical age values are shown with 5 or 6 numerical figures, their effective numerical values should be treated as 3 numerical figures.

The situation is the same for the other time table, as long as their key age standards were dated by the radiometric method. In the case of radiometric dating, their numerical age values have an effective numerical value of 4 numerical figures at most.

Thus, we should be very careful to treat numerical age values in a time table.

Keywords: time table, numerical age value, effective numerical value

Brittle fault dating –looking through a clutter of ages

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There have been numerous case studies in the last few years that successfully constrained the timeframe of brittle faulting through dating of clay-size fault gouge fractions. However, the radiogenic isotope systematics of fault rocks are complex due to the intimate mixture of minerals of different origins such as detrital phases, potentially from a variety of sources, as well as authigenic/synkinematic minerals. Consequently, it is often difficult to unambiguously interpret measured ages. Special sample preparation techniques involving freeze-thaw disaggregation to avoid overcrushing and extensive size separation to reduce the amount of detrital phases can address these issues [1]. Progressive size reduction down to submicron size fractions ($<0.1 \mu\text{m}$) increases the proportion of authigenic clay phases in the clay component and minimizes contamination suggesting that the most reliable isotopic ages for authigenic clay minerals are obtained for the finest size fractions.

Brittle fault illite K-Ar age data and δD from several studies in Europe, Scandinavia and Japan will be presented. All study areas are located within igneous or metamorphic rocks collected from tunnel or drill core samples, which offer a unique advantage as no detrital illite is present in the host rock, thus reducing potential contamination and weathering sources. The age data were obtained using a simplified and standardized method described by Zwingmann *et al.* (2010) [2]. Ages range from the Mesoproterozoic ($1240 \pm 26 \text{ Ma}$) for the Finland to the Neogene ($6.0 \pm 2.1 \text{ Ma}$) for the European Alps study. Fault gouges in Japan scatter around the Paleogene –early Eocene.

The illite ages decrease with grain size, and are consistent with the cooling history of host rocks as bracketed by Ar-Ar, AFTA and ZFTA ages. The data indicates that the fault-rock samples formed within the stability field of illite and the main temperature field of brittle deformation ($< \sim 300^\circ\text{C}$). The internal consistency of the illite K-Ar ages of fault gouges from samples, as well as their consistency with independent constraints from field relationships and existing geochronological data, demonstrate the potential of this simplified method for providing reliable data to constrain absolute timing of brittle deformation.

[1] Liewig *et al.*, 1987. *AAPG Bulletin* 71, 1467-1474.

[2] Zwingmann *et al.* 2010. *Geology*, v. 38, no 6, 487-490; doi10.1130/G30785.1

Keywords: brittle faults, illite clay dating and tracing, K-Ar and hydrogen stable isotopes

Diffusion in a mineral and unclosure temperature: sphere

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The concept of closure temperature (T_c) was proposed by Dodson (1973) from a view of a cooling rock unit, and it has been widely applied to interpret cooling ages in many geological settings. Although the derivation of the diffusion parameters is based on rigorous physicochemical experiments, some ambiguity remained in estimation of T_c . Dodson defined T_c as the temperature in which an isotopic clock practically starts due to drastic drop of diffusion coefficient by temperature change. However, it is unclear what value corresponds to the practical end of diffusion, and uncertainty still remains in cooling rate in spite of the resulting difference in T_c is within $\pm 50^\circ\text{C}$. Here, we consider a diffusion process due to a secondary heating as in a case of stepwise heating during $^{40}\text{Ar}/^{39}\text{Ar}$ experiments.

Exact solutions of a diffusing element at temperature of some duration are given for various geometries (Crank, 1975). A unclosure temperature of diffusion in a sphere is estimated since it is the most heat resistant geometry. When 99% of the starting quantity is lost, it is regarded as total reset. This determines the maximum of unclosure temperature. When nearly 20% is lost, the calculation by Turner (1968) shows that a sphere barely retains near-primary age in the highest temperature fractions. This defines the minimum of unclosure temperature. Both of the temperature is solved in terms of t , the duration using proper approximations. It is better to provide an expression of t in terms of T . This corresponds to the relation between reheating temperature and relaxation time in paleomagnetism (Pullaiah *et al.*, 1976). The closure temperature in cooling body lie between the curves of the minimum and maximum of the unclosure temperatures. The concept of relaxation time provides a clear view of temperature-time relation in geological settings.

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Keywords: diffusion, unclosure temperature, closure temperature, sphere, relaxation time

Trial data of noble gas analysis using new preparation system to measure submarine hydrothermal fluids: comparison for STD gas

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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 (He, Ne, Ar, Kr and Xe). The three types of getters effectively remove halogens, sulfides, abundant water vapor and other active gases for respective gettering step. After the absorbing sea water by cold trap and collection Ar-Kr-Xe fraction in the charcoal trap, the gettering procedure were taken under the following orders: halogen gettering, sulfide gettering, water vapor and active gases. For the efficient removal of sulfides, an exposure to the halogen-getter of the extracted gases prior to the exposure to the sulfide getter is required. Then, respective He and Ne fraction, separated with cryogenic pump, was measured. Next, Ar, Kr and Xe fraction were further purified and separated with charcoal trap, respectively. Further, 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. This newly designed preparation system has been applied to the Patent filing (Application Number: 2015-234839).

In this time, we tried to analyze He isotopes the Kaminoyama hot spring source of water (sampled at November, 2011) included with high sulfide components, environmental water included with high chloride components, which are compared with Kaminoyama STD gas made at 1984 in Yamagata Univ. ($K_S \cong 5.7Ra$, Tamura et al., 2005, now kept in Okayama Science Univ.) and Helium standard of HESJ made in Osaka Univ. (Matsuda et al., 2000, $Ra = 20.63 \pm 0.10$). In this study, 2011 Kaminoyama hot spring Helium gas measured about 7Ra, which is little higher the data of Horiguchi et al., (2010) reported value, Togatta $\cong 6.1Ra$ (sampled at 2006). Thus, the Helium isotope is changed since 2006 before 2011 and relative higher than 1984, which is possible to be connected with activity of the Zao volcanism after Mega-Earthquake. Here, we reported trial data of Kaminoyama hot spring water using a new preparation system compared to the noble gas standards of Kaminoyama gas and HESJ.

Keywords: submarine hydrothermal fluids, noble gas, gettering system

An assessment of the reliability of carbon samples for ^{14}C dating.

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Charcoal samples are considered to be ones of the most reliable samples in the radiocarbon dating. Also, straws and wheat are considered to be ones of the most reliable samples because they have no annual structures. However, all of them have no chemical criteria for verification based on chemical composition and processing conditions. In the present study, we focus on the alkaline treatment stage of the Acid-Base-Acid pretreatment method generally used, and Carbon/Nitrogen ratio for the evaluation of the quality of carbon samples. Results are; 1. Charcoal samples which were left out-side environment and on which photochemical reaction occurred, melt away in 1M NaOH solution; 2. Charcoal samples which melt away in 1M NaOH solution but leave residues in low concentration NaOH solution, have 10-30 C/N ratios which means litter decomposition specified in forest soil science; 3. Dates of those samples described above show significant errors beyond 1 substrata(2010, Atsumi).

Results described above are effective for reliability criteria of samples for the radiocarbon dating. That is, 1. Carbon samples should leave residues against 1M concentration of NaOH solution; 2. C/N ratio of carbon samples should be over 30(2010, Atsumi). The presentation discusses systematically about technological sampling and sample selection. In addition, those 3 reasons derive that straws and wheat which melt away in 1M NaOH solution or C/N ratio of which are 10-30, are not reliable samples for ^{14}C dating.

Keywords: Radiocarbon Dating, ABA method, Charcoal

Reconstruction of past Baiu activity using oxygen isotope ratio of tree rings in central Japan

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The oxygen isotope records presented in natural archives such as tree ring cellulose, speleothems, leaf wax, and ice cores are widely accepted for climate reconstruction. Among other materials, these records are strongly influenced by the isotope value of precipitation as they developed. A better understanding of isotopic variability in precipitation can enable better reconstruction of past environments. For example, in the tropics, various observational and modeling studies have been carried out to provide a greater depth of the interpretation of natural isotopic variability, leading to a common conclusion that isotopic variation in tropical precipitation is related to the large-scale convective activity, rather than precipitation amount. This interpretation proposes that isotope records from the tropical region is used to reconstruct past large-scale convective activity. For Japan, intense observation studies have already done to identify climate drivers controlling isotopic variability. Kurita et al., (2015) showed that north-south displacements of the Baiu front are manifest in the isotopic composition of precipitation in central Japan.

In this study, we reconstructed 100-year long records of oxygen isotopic composition of summer precipitation from tree-ring oxygen isotope records in central Japan. And, we discuss the past location of the Baiu front using reconstructed long-term precipitation isotope data. Since the oxygen isotope ratio of tree rings is not directly influenced by the isotopic composition of precipitation, using mechanistic model predicting the oxygen isotopic ratio of tree-ring, we calculated the isotopic ratio of source water that a tree used during the growing season. The modeled isotopic ratio of source water was exchanged to the oxygen isotopic ratio of summer precipitation using an experimental polynomial function of sunshine duration (SD).

The reconstructed oxygen isotope ratios of summer precipitation during the past 100 years exhibit weak interdecadal variability, with the highest values in the mid-1930s, but inter-annual fluctuations are more considerable. During the period of availability of the reanalysis dataset (1958--2005), the inter-annual and long-term variability in isotopic ratios are closely linked to the Baiu front activity as follows: (1) there is a positive negative correlation ($R=0.37$, $p<0.01$) with the onset days of Baiu rainfall in central Japan, (2) the decreasing trend of the onset days in the later half of the 20th century corresponded to the long-term decreasing trend of the modeled isotopic ratio, (3) the center of Baiu frontal zone is shifted southward, apart from the coast of Japan during the years with low oxygen isotope ratio. These indicate that we can discuss the past Baiu activity beyond the period of availability of the reanalysis dataset using the reconstructed oxygen isotope ratio in precipitation.

Keywords: Tree-ring oxygen isotope ratio, Oxygen isotope ratio in precipitation , Baiu frontal zone

Oxygen isotopic compositions of zircons from modern intra-oceanic arc and arc collision zone granites

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The Izu-Bonin-Mariana (IBM) Arc is an active intra-oceanic arc, where silicic to intermediate granitic crust is being generated through subduction zone magmatism (e.g. Tani et al., 2015 EPSL). It has been active since ~52 Ma and for the last 15 million years has been colliding end-on with the Honshu Arc at the Izu collision zone (ICZ). As a result of this collision, voluminous syn-collisional granitic plutons are exposed in the ICZ that have attained geochemical features more akin to average continental crust through crustal modifications during the arc collision (e.g. Saito et al., 2007 J. Pet.; Tani et al. 2010, Geology). The collective understanding of silicic crust formation in modern intra-oceanic arcs and their successive modification during arc-arc collision is important, as they may be the modern analogue for continental crust formation during the early Earth history.

Since oxygen isotope ratios are sensitive to low-temperature geological processes and zircon is one of the most robust crystals to preserve initial isotopic and trace element compositions of the coexisting melt, zircon oxygen isotopic compositions are commonly used as a key tool to estimate the onset and degree of crustal recycling in the Archean crust (e.g. Valley 2003 RMG). However, it is unclear whether the zircon oxygen isotope systematics of the modern intra-oceanic arcs and arc collision zones are concordant with what is assumed for juvenile crustal formation and successive crustal modification in the Archean.

We have conducted zircon oxygen isotope analyses of representative IBM and ICZ granitic rocks using the CAMECA IMS-1280HR installed at the Kochi Institute, JAMSTEC. The analyzed IBM granitic rocks range from Eocene (~49 Ma, Torishima forearc tonalite) to modern (<1 Ma Niijima tonalite xenolith, Tani et al., 2011 EPSL) and ICZ granitic rocks range from 15 Ma (Kaikomagatake granite, Saito et al., 2012 CMP) to 4 Ma (Tanzawa tonalite, Tani et al., 2010). The average δ^{180} values of IBM granites are generally low from 4.93 to 5.26, except for one diorite sample from Oligocene Omachi Seamount (~38 Ma) that has high $\delta^{180} = 7.03$. ICZ granitic rocks, on the other hand, show a wider range of δ^{180} values from 4.77 to 6.40, which most likely reflects various degrees of incorporation of mature sediments from the Honshu Arc and/or interactions with meteoric water during the emplacement of the granitic magma at shallow levels during the arc collision. These data will provide important geochemical constraints on the crustal development processes in intra-oceanic arcs and arc collision zones and whether crustal development in intra-oceanic arcs and arc collision zones is a modern geochemical analogue of Archean crustal formation.

Keywords: Zircon oxygen isotope, intra-oceanic arc, arc collision zone

The alpha effectiveness of the dating ESR signal in barite: a revision

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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). Takamasa et al. (2013) and Fujiwara et al. (2015) showed that ESR (Electron Spin Resonance) dating of barite (BaSO_4) is useful for investigation of history of hydrothermal activities.

Barite crystals formed by sea-floor hydrothermal activities contains large amount of Ra which replaces Ba in the crystal lattice where the internal alpha dose rate in barite contributes 40 to 60 % of total dose rate (Okumura et al., 2010). As the LET of alpha particles is much larger than beta and gamma rays, causing high-density ionization, the probability of recombination which do not contribute to the generation of the signal is larger, therefore, generating smaller amount of signals. Determination of alpha effectiveness is thus the one of the essential factors for improving the precision of dating of barite by ESR.

Toyoda et al. (2012) investigated the alpha effectiveness for the ESR signal due to SO_3^- in barite by comparing the dose responses of the signal for gamma irradiation and for He^+ ion implantation with an energy of 4 MeV, to obtain a value 0.043 ± 0.018 . However, the dose response was far from "good", where the number of points is not sufficient. The experiments of He^+ ion implantation was repeated in the present study for several samples to determine the precise alpha effectiveness.

A sample of hydrothermal barite, taken by the NT12-06 research cruise operated by Japan Agency for Marine-Earth Science and Technology (JAMSTEC) was used for the present study. The values were obtained to be 0.025 ± 0.002 for synthetic barite, and to be $0.102(+0.014/-0.013)$ for barite extracted from a sea-floor hydrothermal sulfide deposit, being four times larger than the former. For the latter sample, it is possible that the slopes of the dose responses of the signal intensities around the zero dose value may not be estimated appropriately due to the extrapolation of the dose response curve. Tentatively, the value for the synthetic sample should be adopted.

Keywords: barite, hydrothermal activities, ESR dating, alpha effectiveness

ESR dating of the Itoigawa-Shizuoka Tectonic Line located at the northeast edge of Japan South Alps

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The Gofukuji fault is a part of the Itoigawa-Shizuoka Tectonic Line (ISTL) Active Fault System (AFS), and its risk of earthquake disaster has risen since the 2011 off the Pacific coast of Tohoku Earthquake (M9.0). According to hypocenter maps in and around Japan South Alps, after the 2011 Earthquake seismic activity has become more active along the Gofukuji fault, while it has hardly been observed along the ISTL-AFS located at the southeast extension of the Gofukuji fault before and after the 2011 Earthquake. More active seismic activity occurs along the north-northwest - south-southeast (NNW-SSE) line, which is an extension direction of the strike of the Gofukuji fault plane, so that in the future large earthquakes may occur along this line rather than the ISTL-AFS (Fukuchi et al., 2016).

Recent studies of fault rocks revealed that the Hoozan fault located at the southeastern extension of the Gofukuji fault may have moved during the Quaternary period (Fukuchi, 2015). As a result of the ESR dating of the Hoozan fault gouge collected from the Ishiutorogawa outcrop, the age (T) of the latest fault movement of the Hoozan fault is estimated as $T < 0.6 \pm 0.1$ Ma, from the hyperfine structure of the Al center in quartz. This result suggests that the ISTL as a geological tectonic line located at the southward extension of the Hoozan fault may be still active during the Quaternary. Thus we carry out ESR dating of the ISTL distributed at the Dondoko-sawa outcrop, Nirasaki city, central Japan. The distribution maps of hypocenters indicate that the seismicity of micro-earthquakes is more active along the ISTL around the Dondoko-sawa outcrop. The X-ray diffraction analysis of fault gouge collected at the Dondoko-sawa outcrop indicates that the black fault gouge on the fault plane contains smectites, which can stably exist below about 110 degree C in natural environments (Yoshimura, 2001). We estimate the formation age of the smectites in the black gouge from the formation depth of smectites by assuming the average geothermal gradient of 3 degree C/100m and the average upheaval rate of 2-3mm/y. As a result, the ISTL around the Dondoko-sawa may have moved between about 1.7-2.5 Ma BP. The ESR dating of the black gouge shows that the age of the latest fault movement is estimated as $T < 0.55 \pm 0.12$ Ma. We conclude that the ISTL around the Dondoko-sawa is still active during the Quaternary period.

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Keywords: Japan South Alps, Itoigawa-Shizuoka Tectonic Line, ESR dating, Electron spin resonance, Fault rock, Smectite

Observation of zircon by atomic force microscope for establishing Alpha Recoil Track method

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ART method calculates a date by measuring U and Th concentrations and the number of ARTs which are proportional to the time passed. ART method for zircon can date samples of hundreds to 100 hundreds years old (it depends on U and Th concentrations). For the size of ART is smaller than FT, it is difficult to observe ART by using optical microscope, but by using atomic force microscope (AFM), it is possible because it has high resolution of the nano-order. Previous works observed concaves which are clearly different from FT in zircons with the age of, 20a (Ito, 2014) and 58ka (Kozaka, 2015). In the previous work (Kozaka,2015) on a zircon(HKS08, 58ka), the areal ART density was estimated by counting the number of ART.

First, U-Th concentrations on HKS08 were surveyed by LA-ICP-MS. U and Th concentrations of three area, A, B and C in the zircon crystal were 150~270ppm and 86.5~210ppm, respectively. Because areal track density was estimated for the area close to point B, we calculate the age with U and Th concentrations of point B, 211.9ppm and 178.3ppm, and estimated age was 13.8ka, younger than expected HKS08 age (58ka).

Second, zircon from Aira-Ito pyroclastic flow (ITO) was observed. This is a wide-spread tephra and its expected eruption age is 22ka to 25ka (Machida and Arai, 1992). Six zircons were observed and area ART densities were measured. The results were 0.001~0.116/ μm^2 . U and Th concentrations were measured on other ITO zircons. U and Th concentrations were 130~270ppm and 60~160ppm, respectively. As a result, these estimated ages were younger than the expected ITO age (22~25ka). It might be caused by inappropriate factor in age equation or wrong criteria for ART identification. For establishing ART method, it is necessary to discuss whether the concave is ART or not by additional step-etching and observing many samples with known age.

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Keywords: Alpha Recoil Track, Zircon, Atomic Force Microscope

New approach to OSL dating of fault gauge

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Several methods for fault dating, such as ESR and FT, have been used to attempt to determine the latest age of active faults in Japan. The investigations however have generally shown ages quite older than expected, which were estimated by examining the geological and geographical evidence.

We have determined the fault quartz ages from three locations of the Atera fault in central Japan using the OSL method as the reset temperature of the OSL signal (350 °C, 10s) is much lower than when measured by the above methods [1].

Heating tests using granitic quartz grains from the Atera fault have been carried out to study attained and reset temperatures; those were evaluated from the sensitivity change in the SAR method of OSL dating.

The heating experimental result showed that there was a good relationship between sensitivity and heating temperature and when a quartz sample was attained at a temperature of 340°Cx40s, OSL signals were completely reset.

The OSL dating of the gouge quartz grains from three locations of the Atera fault showed remarkably lower ages than those of cataclasites. Therefore it could be deduced that the gauges were attained a higher temperature than the cataclasites.

Keywords: quartz, OSL dating, reset temperature, Atera fault

Evaluation of influence on the radiocarbon dating by the difference in chemical pretreatment protocols

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Mass movements (e.g., slope failure, landslide and debris flow) and fault movements are natural phenomena to change the local topography greatly. Reconstruction of the movement histories and evaluation of long-term stability of topographies are crucial to assess the geological environment in future. History reconstruction based on radiocarbon dating of plant material recovered from sediment deposited by the past movements requires the compilation and the comparison of existing data measured by different analysts with different methods.

We compared the results of radiocarbon ages measured at different laboratories with various chemical pretreatments (acid-base-acid method, cellulose extraction, acid-base-oxidation-stepped combustion), using plant material samples (charcoal, old tree-trunks and roots; estimated ages of 600-2200 BP or 45000 BP) recovered from sediment in Tsuwano, Shimane Pref.

For three younger samples (600-2200 BP) measured at different laboratories, concordant ages within measurement uncertainty are yielded as long as the experimental protocol of each laboratory is applied. For five older samples (43000-47000 BP) measured under various experimental conditions, dispersion in ages (2100 - 3500 years; approximately 5-8 % of the ages) is greater than measurement uncertainty (200 - 900 years; 0.5-1.7 %). However, the order of weighted mean ages for each sample agrees well with stratigraphic sequence at the sampling site, and such dispersion near the limit of radiocarbon dating method can be observed in previous works (e.g., Reimer et al., 2013, Radiocarbon, 55, 1869-1887). It is therefore thought that the influence of the systematic error due to a specific experimental conditions is small.

Keywords: radiocarbon dating, ABA method, ABOx method, cellulose extraction

Argon Isotopic Composition on Mauna Loa Historical Lavas

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K-Ar and Ar-Ar dating are widely adopted method to the reconstructions for the eruption history of the active volcanoes. Regardless of the wide use, there is a fatal problem of its basis that the initial argon isotope ratios are not fully reset, in other words, incompletely equilibrated with the atmospheric Ar. Although this phenomenon has been occasionally reported, the factors controlling have not intensively been investigated yet. Thus in this study, a set of lava systematically sampled from a historical lava flow was analyzed: sixteen samples from 1935 lava flow on Mt. Mauna Loa. Ar isotope measurement was performed with GVI-5400He as well as their major element compositions with XRF (Simultix12, Rigaku Co.) at JAMSTEC. Most of the samples showed smaller $^{38}\text{Ar}/^{36}\text{Ar}$ compared with the atmospheric isotope ratio. It may be a result of some magmatic processes at depth as well as shallower eruption related ones. Regardless of an anticipated enrichment of heavier isotopes that correlates with vesicularity for the residual argon in vesiculation, all the samples showed enrichment of lighter isotopes. Further, there is no clear correlation with vesicularity, lava type nor distances from the vent of the flow, etc.

Keywords: K-Ar method, mass fractionation, Mauna Loa volcano

The progress of the CHIME monazite dating on JXA-8530F FE-EPMA equipped with R = 100 mm spectrometers

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The CHIME (Chemical Th-U-total Pb isochron method, Suzuki and Adachi, 1991) dating has been performed on JEOL JXA-8530F FE-EPMA introduced in Tono Geoscience Center of Japan Atomic Energy Agency. The age spectrum of detrital monazite grains is a useful measure for the provenance analyses of clastic sediments. The CHIME is best suited for dating of detrital monazite where grains are not chronologically uniform and many analyses are required to characterize a population. Analyses of Pb on conventional R = 140 or 160 mm spectrometers have disadvantage in count rate. The R = 100 mm spectrometer is desirable for quantitative determination of trace Pb, giving an intrinsic response 3 to 5 times higher than that of the R = 140 mm spectrometer. JXA-8530F equipped with three R = 100 and two R = 140 mm spectrometers significantly shortens an analyzing time. Spectral interferences peculiar to R = 100 mm spectrometers are found to be collected through the method of Amlı and Griffin (1978) on the monazite analyses (Th, U, Pb, Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Er, P, Si, Ca, S, K, Na, Al and Mg). This interference correction and the pulse height discrimination (PHA) improve the spectral resolution of R = 100 mm spectrometer, which is comparable to that of conventional R = 140 mm spectrometer.

The CHIME monazite age for the Cooma granodiorite was determined to check the reliability of the data provided by this advanced procedure. The Cooma granodiorite is a 3 km x6 km pluton at the core of an N-S trending Cooma metamorphic complex in the Lachlan Fold Belt, New South Wales, southeastern Australia (Vernon et al., 2001). The procedure carried out 434 ±26 Ma for monazite from Cooma granodiorite, which was previously dated by SHRIMP as 432.8 Ma (Williams, 2001). The CHIME dating is applicable to monazite as young as ca. 60 Ma (Suzuki and Adachi, 1998). The Kojaku granite is exposed in the Tsuruga Peninsula and the northern side of the Lake Biwa, central Japan. The CHIME monazite age of the Kojaku granite is 68 ±8 Ma that accords well with the LA-ICP-MS U-Pb zircon age of 68.5 Ma (Sueoka et al., 2016, in press). Both of CHIME monazite age and U-Pb zircon age represent the formation age of rock body, because their closure temperatures are sufficiently high to record the formation of rock body, suggesting agreements of these ages guarantee accuracy of the monazite dating system. The age values in this study correspond to the values of the previous researches within the error range, thus the improved CHIME monazite dating using the JXA-8530F FE-EPMA has been established.

In our presentation we also show the CHIME age of the Steenkampskraal monazite, which was dated by SHRIMP as 1033 Ma (Knoper et al., 2000; Hokada and Motoyoshi, 2006). In addition, a quick heavy mineral identification method, which has been established as well, is introduced. In this method heavy minerals are identified based on their elemental compositions quantitatively analyzed by the FE-EPMA in contrast to the general conventional method depending on microscopic observations. The method is developed to enable the identification of several-hundred grains of heavy minerals in short time as possible, and by using this method the monazite grains for CHIME dating can be found quickly.

This study was carried out under a contract with Ministry of Economy, Trade and Industry of Japan as part of its R&D supporting program for developing geological disposal technology.

Keywords: CHIME dating, EPMA, monazite

Development for in-situ radiogenic ^4He analysis in zircon

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Development for in-situ radiogenic ^4He analysis in zircon

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Zircon is recently used for U-Th-He dating of various rocks because zircon contains high concentration of U and Th, and radiogenic ^4He which is produced by disintegration of U and Th (e.g., Reiners et al., 2004). The closure temperature of U-Th-He dating in zircon is low (~180°C). Recently, a low-temperature thermal history of their samples are revealed by using the U-Th-He dating of zircon, for example, the process of uplift and denudation of mountains (e.g., Sueoka et al., 2011).

There is a question about ^4He analysis in current U-Th-He dating. Since a typical range of an α -particle is ~20 μm , the conventional apparatuses for ^4He analysis cannot accurately evaluate an escape of radiogenic ^4He from minerals by α -decays due to a larger spatial resolution.

LIMAS (Laser Ionization Mass nanoScope) is an analytical system developed for analyzing noble gases in micro region, which should be a key instrument to solve the issue. In this system, neutral particles sputtered by Ga focused-ion-beam from sample surface are ionized by femtosecond laser for tunneling ionization, and produced ions are separated by the multi-turn time of flight mass spectrometer 'MULTUM II' depending on their m/z . LIMAS has a potential to trace a track of the α -particle because a spatial resolution of LIMAS is less than 1 μm in case of analysis for trace amount of ^4He (Bajo et al., 2015). LIMAS can measure depth profile, and analyze distribution of the ^4He concentration at a given depth. Thus, the depth profiling may tell us the further details of ^4He diffusion profile in minerals.

If U-Th-He can be measured by LIMAS, the new U-Th-He dating which should be more reliable than previous measurements will be proposed. As the first step, we carried out quantitative analysis of radiogenic ^4He in a zircon standard (zircon91500), that have known data about U and Th concentrations and U-Pb age, by LIMAS.

We measured zircon91500 to detect radiogenic ^4He by using LIMAS. Concentrations of U and Th in zircon91500 are 80 and 30 ppm, respectively, of which U-Pb age is 1065 Ma (Wiedenbeck et al., 2004). The concentration of radiogenic ^4He was supposed to be 55 ppm based on the U and Th concentrations and U-Pb age.

We measured ^{28}Si ions and $^4\text{He}^+$ to estimate concentration of radiogenic ^4He . The radiogenic ^4He in zircon91500 was calculated to be 30 ± 5 ppm after a blank correction. The blank level of this study was 7 ± 3 ppm, which was derived from residual ^4He in the sample chamber of LIMAS.

Keywords: zircon, U-Th-He dating, ^4He , LIMAS

Review of calibration method of zircon Pb/U ratio obtained by SIMS

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U-Pb zircon geochronology has been widely applied due to zircon's high durability, high closure temperature (e.g., Cherniak 2010 and references therein), high concentration of parent element uranium, and its negligible incorporation of the daughter element Pb during crystallization. In addition, the paired decay scheme of ²³⁸U and ²³⁵U allows us to verify the determined U-Pb zircon age, using two geochronometers. Recent technical progress of microbeam analysis using secondary ion mass spectrometry (SIMS) and laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) has allowed us to obtain highly precise U-Pb zircon age from several mm to 25 mm. A calculation of the U-Pb zircon age obtained by SIMS analysis is generally complicated by largely attributed to differences in ion yield in Pb and U sputtered from zircon. In addition, the measured ion ratios are not even necessarily constant for a target of known composition from spot to spot and from session to session, often varying during a measurement session, for example in response to pressure, surface charging, primary beam intensity, spot shape, and sample temperature. To correct this effect, a suitable reference material is required to calibrate the secondary ion ratio (Pb^+/U^+) to the atomic abundance ratio (Pb/U). An empirical relationship between Pb^+/U^+ and UO^+/U^+ (calibration curve) has traditionally been used for this correction, and this relationship has been considered to obey a power law of the form $^{206}Pb^+/^{238}U^+ = a \times (^{254}(UO)^+/^{238}U^+)^2$. The statistically more reliable calibration scheme enables us to obtain highly precise and accurate U-Pb zircon age. In this presentation, several calibration schemes were compared by using a sensitive high resolution ion microprobe (SHRIMP-IIe).

Correlation among Pb^+ , U^+ , UO^+ , and UO_2^+ was tested using TEMORA2 zircon. The precision of Pb^+/U^+ ratios seems to be improved by using the correlation between Pb^+/UO^+ and UO_2^+/UO^+ , which is probably derived from similarity of energy distribution between Pb^+ and UO_2^+ rather than between Pb^+ and U^+ . Also, the correlation was assessed by several reference zircons such as FC1, OT4, OG1, and Mud Tank and the results will be reported.

Keywords: zircon, U-Pb dating, SIMS

U-Pb zircon geochronology and geochemistry of Utsubo granitic pluton in Hida Belt

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Geochronological and geochemical information is one of the essential consideration of the magmatic processes. Especially, plutonic complex with compositional zonation produced by a single magma intrusion provides good opportunity to understand detailed timescale of magmatic evolution. In this study, we discuss timescale of geochemical evolution in the Utsubo granitic pluton using highly precise U-Pb zircon dating and trace element analyses.

The Utsubo granitic pluton is situated in Hida belt, which is the northernmost geotectonic unit in the Inner Zone of Southwest Japan Arc. The granitic rocks in the Hida belt are Early Triassic to Early Jurassic, and are traditionally classified into Funatsu type and Simonomoto type. Recently, the calc-alkaline plutons in the Hida belt are divided into two types based on petrology and isotopic composition of Sr and Nd: Type-1 indicates a limited variation in initial Sr and Nd isotopic value and Type-2 indicates a wider range of isotopic value (Arakawa and Shinmura, 1995). The Utsubo granitic pluton, emplaced in the Hida gneiss, belongs to Type-1 pluton and shows a normal laterally compositional zoning, which has tonalite, granodiorite, pink coarse-grained granite, and fine-grained granite from its margin to center (Kano, 1990). Zircon grains collected from tonalite, granodiorite and coarse-grained granite were yielded ca. 192 Ma, 190 Ma and 188 Ma, respectively. The geochronological results indicate that time interval from tonalite to coarse-grained granite is about 4 Ma. In addition, geochemical data will be presented.

Keywords: zircon, U-Pb dating