

Closure temperature of biotite and thermal history

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In previous report, closure temperature of biotite in K-Ar system has been estimated from the results in laser step heating ⁴⁰Ar/³⁹Ar experiment on a single grain. In the estimate, the cooling rate was assumed to be 1 K/1000 yr, and the data was taken from fraction less than 800 degrees steps where little effect of dehydration is expected. The closure temperature calculated were distributed from less than 0 degrees to over 300 degrees. The average was approximately 250 degrees, being slightly less than the estimate from bulk experiments, whereas the average of activation energy was not much different from the previous value, indicating that the closure temperature obtained from laser step heating experiments is good enough for a rough estimate.

In this paper, increasing the number of data and distinguishing slow (1K/1000 yr) and rapid cooling (1K/1yr) between "intrusives" (including metamorphics) and igneous "extrusives" (including tuffs), respectively, closure temperature and activation energy was compared. No significant difference was found in the activation energy, but the closure temperature estimates differed; 270 degrees in intrusives and 330 degrees in extrusives. The difference seems to reflect the effect of cooling rate in the Dodson's (1973) closure temperature formula. However, if we assume the cooling rate of the intrusives in extrusives, some of the samples show the closure temperature less than 200 degrees. Thus, the difference does not seem to be an apparent effect from the calculation.

There also were samples with closure temperature less than 100 degrees regardless of rock types. This often correlates with alteration, in particular chloritization of biotite, and reflected in age spectra. In most Arrhenius plots, the change of trend, reflecting the dehydration of biotite in bulk experiment was not found in laser step heating results. The difference is considered in view of heating scheme and slightly the lower estimate of closure temperature than bulk experiment.

Keywords: closure temperature, biotite grain, K-Ar system, ⁴⁰Ar/³⁹Ar, laser step heating

Reduction of extraneous ^{40}Ar contamination for accurate K-Ar age determinations: an experimental study in various sample

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A fundamental assumption of K-Ar dating is that the samples initially contained no radiogenic ^{40}Ar , but sometimes rocks contain radiogenic ^{40}Ar called extraneous ^{40}Ar . Some previous study reported argon isotopes of historical lavas had anomalously high $^{40}\text{Ar}/^{36}\text{Ar}$ ratios, and show old apparent ages. Since extraneous ^{40}Ar is likely contained in the phenocrysts and xenoliths, groundmass samples are generally prepared for analysis. Besides, Ozawa et al. (2005) showed fine-grained groundmass samples had less extraneous ^{40}Ar contamination, and suggested that extraneous ^{40}Ar is contained in fluid inclusions or vesicles and released during crushing. We measure argon isotopic ratios in various sizes of young lava samples, and investigated the reduction of extraneous ^{40}Ar contamination. The finer samples roughly showed lower $^{40}\text{Ar}/^{36}\text{Ar}$ ratios but more difficult to handling of the preparation such as mineral separation and wrapping in foils for isotopic measurements.

Keywords: K-Ar dating, extraneous ^{40}Ar , sample size

Results of Ar-Ar dating for basaltic rocks from Bowers Ridge, Bering Sea at site U1342A&D

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Basement rocks were drilled down to ca. 42 m into the volcanic sequence directly underneath the sedimentary section at Site U1342 on Bowers Ridge during the IODP Expedition 323 to the Bering Sea. This provided us an opportunity to describe the details of the sequence and to decipher the virtually unknown origin and evolution of the Bowers arc massif. There are two contrasting hypotheses for the origin of the arc, which include formation in the Pacific Basin well to the south of its present location during the Cretaceous and in-situ formation within the Bering Sea in Eocene.

The volcanic sequence recovered from Site U1342D was divided into six major lithological units: Unit 1, vesiculated andesitic lava flow; Unit 2, interbedded volcanic sandstones and polymict volcanic conglomerates; Unit 3: monomict volcanic conglomerates; Unit 4, interbedded volcanic sandstones and polymict volcanic conglomerates; Unit 5, monomict volcanic conglomerates; and Unit 6, polymict volcanic conglomerates. Units 3 and 4 represent hydroclastic volcanoclastics, while units 2, 4, and 6 are epiclastic volcanoclastics (Kawabata et al., 2011). We used the single grain ⁴⁰Ar-³⁹Ar dating method by step-wise laser fusion for Unit 1 basaltic andesite rocks. dating. We distinguish for the first time two stage (age groups) of activity (34-32Ma and 28-26Ma) from our Ar-Ar data, coupled with those from Wanke et al., (2012).

Keywords: Ar-Ar dating, Bering Sea, Bowers ridge, U1342A&D

Fission track ages for baked country rocks adjacent to the mafic dikes in the Takato area, central Honshu

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We present new fission track (FT) ages for apatites and zircons separated from baked zones of country rock (granite) adjacent to dolerite dikes in the Takato area, Nagano Prefecture. The dolerite dikes form a dike swarm with a dominant NW-SE strike and vertical or subvertical dips, from which a minimum principal stress (σ_3) axis trending NE-SW is deduced. The country rock is the Takato granite of late Cretaceous age. There are a number of good exposures where the contacts between the dolerite dikes and the granite can readily be recognizable. In order to determine the age of this dike swarm by FT dating, rock samples were collected from three baked zone sites of the granite that are located adjacent to the dolerite dikes. At the baked zone sites, we carefully sampled tiny rock fragments and mineral grains within 8 mm from the contact. We determined FT ages of ca. 17-16 Ma for zircons from all the baked zone sites, compared with the zircon FT ages of ca. 55 Ma determined for granite samples far from dikes. Confined FT length measurements suggest that the zircon FT ages for the contact zones have totally been reset by the heat from dolerite dikes. These FT results indicate that the dolerite dike intrusion took place at ca. 17-16 Ma and that mafic igneous activity occurred in this area in the latest Early Miocene. This finding has an implication that the 17-16 Ma volcanic front probably lay through or close to this area of central Honshu. For apatites, consistent ages of ca. 4 Ma were determined for both the baked zone and distant sites. Such significantly young apatite FT ages can be explained by assuming (i) significant uplift and denudation in and around the Takato area after 4 Ma, or (ii) a local thermal event at that time.

Keywords: fission track age, dolerite dike, Takato granite, central Honshu, thermochronology, Miocene volcanic front

Apatite fission-track and (U-Th)/He ages of the Suzuka Range, southwest Japan, and their geomorphological implications

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The Suzuka Range is a fault block mountain distributed along the Isewan-Tsurugawan Tectonic Line, a tectonic boundary between the Kinki and Chubu districts. The Kinki district on the west of the Range is characterized by predominance of reverse faults and alternation of N-S trending mountain ranges and basins (Kinki Triangle; Huzita, 1962, 1983), whereas the Chubu district on the east of the Range has predominance of strike-slip faults and westerly tilting landforms (Chubu tilting block; Kuwahara, 1968). Miyoshi & Ishibashi (2008) mentioned that the Philippine Sea Plate slab beneath the region around the Suzuka Range has shallow subduction angle and form a convex shape (Isewan-Kohoku slab) and proposed this shallow slab resulted in the tectonic boundary between the Kinki and Chubu districts in the region. However, how the slab has affected the landform development and tectonics of the region is not well understood partly because vertical crustal movements in the past few million years are not estimated. Subsidence and its rates in the past few million years in the Ohmi and Nohbi basins can be estimated by the depths and ages of the layers of the Pliocene Kobiwako and Tokai Groups. On the other hand, estimating uplift and its rates of the Suzuka Range requires denudation and denudation rates.

We are attempting revealing denudation history of the Suzuka Range in the past few million years by using thermochronological methods. We used apatite fission-track and (U-Th)/He thermochronology which have low closure temperatures (90-120 deg. C and 55-80 deg. C, respectively) and are generally used to detect recent denudation events. In apatites of granitic samples collected at the Suzuka Range, fission-track densities do not vary systematically along the N-S profile. Assuming that uranium concentrations are homogeneous in the granitic samples, the Suzuka Range should have had spatially homogeneous denudational history. If the apatite fission-track ages reflect the denudation history of the Range in the past few million years, uplift of the Range might be spatially homogeneous although subsistence of the Ohmi and Nohbi Basins started from the south and propagated to the north (e.g., Okada, 1980). On the other hand, if the apatite fission-track ages reflect the denudation history of the Suzuka Range in the past few ten million years, the denudation might be mainly attributed to the regional peneplanation of the Kinki and Chubu districts since the Palaeogene or late Cretaceous time. In a presentation, we are planning to provide more fixed and detailed discussions from the results of apatite fission-track and (U-Th)/He ages.

Keywords: fission-track thermochronology, (U-Th)/He thermochronology, Suzuka Range, denudation

The alpha effectiveness for formation of SO_3^- in barite : an application to ESR dating

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While Kasuya et al. (1991) first pointed out that ESR (electron spin resonance) dating of barite (BaSO_4) is possible, the method was first practically applied by Okumura et al. (2010) to a sample formed by the submarine hydrothermal activity. A subsequent study by Sato et al. (2011) studied the thermal stability of the signal and concluded that the dating signal due to SO_3^- is stable so that dating method is applicable up to at least several thousand years.

Barite crystals formed by submarine hydrothermal activities contains large amount of Ra which replaces Ba in the crystal lattice where all dose rate is due to radiation from Ra. Okumura et al. (2010) reported a concentration of 7.7 Bq/g of Ra in a hydrothermal sulfide including barite where the internal alpha dose rate in barite contributes 40 to 60 % of total dose rate. 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. A value 0.043 was obtained for a sample from Morocco.

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.

As results, a value of 0.0012 was obtained from a sample from Morocco, and 0.00045 from one from Funaoka mine. The results of further repeated analysis will be presented.

Keywords: barite, electron spin resonance

ESR dating of barite in sea-floor hydrothermal sulfide deposits

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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. (in press) obtained U-Th and ESR ages which are roughly consistent with each other.

The samples were taken by NT11-20 and NT12-06 research cruises operated by JAMSTEC. Barite (BaSO₄) was extracted from hydrothermal chimney samples (HPD#1331G01, HPD#1331G03, and HPD#1333G06) taken from two sites at 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 ages of the pieces of HPD#1331G01 (Hatoma Knoll) were obtained to be 2600 to 4000 years, where outer pieces tend to be older. The ages of HPD#1331G03 are older to a direction, from 2.2 ka to 10 ka (Hatoma Knoll). HPD#1331G06 (Yoron Knoll) showed much younger ages around 100 years where they are older to a direction.

The results, the ages of the Hatoma Knoll is older than the Hatoma Knoll, are consistent with the landscape observation from the submarine vehicle, which gave such impression such as by number of dead chimneys and amount of sediments on the sulfide deposits, and with the diversity of the creatures inhabiting in the area.

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

ESR dating of tephra with dose recovery test for impurity centers in quartz

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Determining the age of tephra is an important issue for reconstructing the history of environmental change during the Quaternary epoch. To this end, we consider dating of quartz using the electron spin resonance (ESR) method. Quartz was first found to be useful for ESR dating of fault gouge while the mineral was also used for dating of tephra, heated flints, and sediments.

The first investigation pertaining to ESR dating of tephra using quartz was published using the Al center (a hole trapped at Al site replacing Si). Subsequently, several other successful results on tephra have been reported (e.g. Imai and Shimokawa, et al., 1988, Imai et al., 1992, Toyoda et al., 1995, and Yokoyama et al., 2004). Buhay et al. (1992) reported that the ESR age (45-49 ka) of a tephra from New Zealand is consistent with the ¹⁴C age (42-44) within statistical errors.

However, in other studies, systematic discrepancies were observed between the ages obtained using the Al center and Ti-Li center (an electron trapped at a Ti atom replacing Si, accompanying a Li ion as a charge compensator). Toyoda et al. (2006) systematically investigated the ESR and RTL (red thermoluminescence) ages of tephra with a known age range of 30 to 900 ka, and found that ESR dating has problems in obtaining the equivalent doses. Using the same dose rate, the RTL ages were consistent with the expected ages while the ESR based results were inconsistent and involved large scatter in data. The scatter in ESR ages was found to increase with age. Toyoda et al. (2009) proposed a new protocol, the multiple-aliquot regenerative-additive dose method, which provides equivalent doses estimates with smaller errors than the traditional additive dose method.

In the present paper, we have analyzed the same Nm-Sb tephra and A-Fm tephra and Ikezuki tuff to check the reproducibility of dating results and to test if known doses can be recovered using the multiple-aliquot regenerative-additive dose procedure.

Keywords: ESR dating, quartz, tephra

Estimate of the origin of the river sediment in the Kurobe River basin using TL and the ESR

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While the ESR signals of the E1' center in quartz was used to investigate the origin of the loess in MIS 1 and 2 (Toyoda and Naruse, 2002) and these diments in the Sea of Japan (Nagashima et al., 2007), Shimada (2008) showed that TL CI (thermiluminescence color image) may be useful for similar qualitative study on river sediments. In the present study, the wavelength-temperature two dimensional thermoluminescence measurement was employed, together with the ESR measurements, to investigate the origin of the river sediments quantitatively.

Sediment samples were collected from the 23 locations at the prefectural border of Nagano and Toyama and the Kurobe River basin. Eight samples of these were sieved to obtain four grain size fractions of 2-1mm, 1-0.5mm, 0.5-0.25mm, 0.25-0.125m. Quartz grains were extracted using chemicals, heavy liquid, and an is dynamic separator. The obtained quartz grains were heated at 300 degree celsius for 1 hour to erase the inherited signals. Each sample was then separated into 9 subsample aliquots for gamma ray irradiation up to 2640 Gy, which are for ESR measurements. Another separate aliquot for TL measurement was given a dose of 857 Gy where the sample glass tube was wrapped by Al foil to prevent from giving any light.

TL measurements were performed the two dimensional TL apparatus which measures the TL emission spectra during heating up to 450 degree celsius. Red emission (538 to 658 nm) was observed between 90 and 390 and Blue emission (379 to 538 nm) was between 70 and 370 . The integrated counts were taken as the intensities of the red and blue emissions. The intensities of the blue emission are roughly constant for all samples of river sediments and river terrace samples while red emission tends to increase with age, i.e., lower in higher terraces and higher in lower terraces and present river sediments. The results of ESR measurements will be given in the presentation together with the TL results.

Keywords: ESR, TL(thermiluminescence), Quartz, river sediment

Eruption age determination of Kannabe scoria cone using multi-dating method

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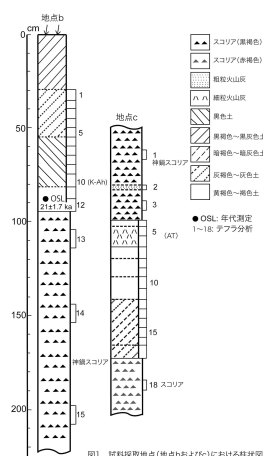
We determined eruption age of Kannabe scoria cone, which is located in southwest Japan. Although the eruption age had been estimated using K-Ar and loess stratigraphy, there is room for improvement in precision of the age determination.

We applied optically stimulated luminescence (OSL) dating, paleomagnetic measurement and tephrochronology on sediments and basaltic rocks associated with the Kannabe scoria cone. The sediment above the Kannabe basalt was formed at 21+/-1.7 ka (OSL dating). The eruption age was tephrochronologically estimated as 7.3-29 ka because the lava exists between two widespread tephra: Aira-Tn ash (ca. 26-29 ka) and Kikai-Akahoya ash (ca. 7.3 ka). The eruption age of the Kannabe scoria cone was before 21 ka and until ca. 29 ka.

We evaluated the eruption age of the Kannabe basaltic by detailed paleomagnetic investigation. The paleomagnetic data of 23 rock samples from 6 locations in the Kannabe basaltic field showed good agreement with each other. The averaged declination and inclination were respectively, 7.5° and 65.9°, which was in accordance with the geomagnetic secular variation of sediments in Lake Biwa at ca. 25 ka.

Consequently we proposed that the Kannabe basalt erupted at ca. 25 ka.

Keywords: Kannabe scoria cone, eruption age, paleomagnetic dating, OSL dating, tephrochronology



An assessment for alkaline treatment in ABA method to charcoal sample for ^{14}C dating (AMS)

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The Acid-Base-Acid (ABA) method is one of charcoal treatment methods for ^{14}C dating. The evaluation of processing conditions of the ABA method based on any chemical indicator does not exist until today. An assessment against wood charcoal using Raman spectrometry has been tried, but the result only suggests that it is possible to detect the inclusion of humic acid in charcoal samples by Raman spectrometry but the relation between pretreatment efficiency and ^{14}C dates has not been investigated. Therefore, this study aims to confirm the error in ^{14}C dating generated by the alkaline pretreatment which is not clarified hitherto. The results show that medians of dates of samples treated with NaOH solution are scattered in the range of 57 ^{14}Cyr whereas medians of dates of samples untreated with NaOH solution are scattered in the range of 216 ^{14}Cyr . The results of chi-squared test show $T=0.45$ ($df=3$; 5% risk rate $T > 12.59$) for the treated samples which means high convergent validity, while $T=10.74$ ($df=4$; 5% risk rate $T > 9.49$) for the untreated samples which means large scattering and significant variation. In addition, dates of the untreated samples include younger (3589 plus-minus 41 BP) date and older (3805 plus-minus 40 BP) date in comparison to the average date of the treated samples (3701 plus-minus 43 BP), which suggests that ^{14}C dates are made younger or older by the pollution or contamination of charcoal. Those results stated above partially attests the effectiveness of the ABA method.

Keywords: radiocarbon dating, charcoal, archaeological sample, ABA method

Application of isotope-geology to ichnology: paleoecology of the Phymatoderma-producer based on carbon-isotope analysis

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The present study shows a case study that applies isotopic analysis to ichnology. The ichnogenus *Phymatoderma* is a subhorizontal branching burrow system consisting of radiating tunnels filled with fecal pellets. This ichnogenus has been interpreted as a product of a deposit-feeding organism, but the question of whether the *Phymatoderma*-producer was a subsurface deposit feeder or a surface deposit feeder is still a topic of controversy. Here I present evidence for the surface deposit-feeder hypothesis, based on carbon-isotope analyses, for the trace fossil *Phymatoderma granulata* from the lower Toarcian black shale in southern Germany. Carbon-isotope ratios of organic carbon in the pelletal infill of *P. granulata*, the surrounding black shale, and the overlying gray mudstone are -26.64 permil, -28.49 permil, and -26.27 permil, respectively. The difference between the pelletal infill and overlying mudstone in terms of C-isotope ratio is much smaller than that between the fillings and black shale; therefore, these data clearly indicate that the *Phymatoderma*-producer ingested the surface sediments and subsequently excreted fecal pellets into the subsurface sediments. Such a surface deposit-feeding style would be an effective way of absorbing nutrients, because surface sediments contain much fresh organic material, whereas organic matter in subsurface deposits consists mostly of refractory material that is poorly utilized by most marine benthos.

Correction of initial-disequilibrium on U-Th-Pb system for Accurate Zircon Dating

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During recent years, the improvement of analysing technique provides a more precision in case of dating Quaternary zircons. Major analytical problems associated with age determination of the young zircons are (1) the analytical difficulty to measure extremely low Pb/U and Pb/Th ratios (e.g., $^{206}\text{Pb}/^{238}\text{U} < 0.0001$), and (2) initial-disequilibrium in the U-Th-Pb decay systems through the crystallization of zircon in source magma. The ability to measure isotope ratios with high-dynamic ranges could be improved by the suppressor device for ion counting systems in the ICPMS instrument, in which the gain of the ion counting could be changed during the fast mass scanning, and the resulting precision and accuracy for the Pb/U and Pb/Th ratio measurements was dramatically improved. However, correction of the initial-disequilibrium is highly desired to obtain reliable age data for young (<1Ma) zircons. Because of the different distribution coefficient ($D_{\text{zircon/magma}}$) between U and Th, isotope equilibrium was disturbed at the crystallization of zircons in source magma. Among the uranium series decay products, the initial disequilibrium of ^{230}Th can become a major source of systematic error in the resulting ages. To evaluate and correct the contribution of the initial disequilibrium on ^{230}Th , the ratio of the distribution coefficient for Th and U ($f_{\text{Th/U}} = D^{\text{Th}}/D^{\text{U}}$) must be defined [2]. To achieve this, we have determined both the ^{238}U - ^{206}Pb and ^{232}Th - ^{208}Pb ages were obtained for three tephra zircon samples collected from Kirigamine rhyolite, Bishop tuff and Toga pumice (^{40}Ar - ^{39}Ar ages are 0.945 ± 0.005 Ma, 0.7589 ± 0.0036 Ma, and 0.42 ± 0.01 Ma reported by [3], [4], and [5], respectively) using a LA-ICPMS. The resulting ^{232}Th - ^{208}Pb ages were 0.938 ± 0.026 Ma (Kirigamine), 0.757 ± 0.008 Ma (Bishop), and 0.428 ± 0.004 Ma (Toga), respectively, demonstrating that the resulting ages were consistent with the previously reported values. The $f_{\text{Th/U}}$ values could be calculated based on the measured $^{206}\text{Pb}/^{238}\text{U}$ ratio and the resulting ^{232}Th - ^{208}Pb ages, and the calculated $f_{\text{Th/U}}$ values were 0.50 ± 0.26 for Kirigamine, 0.51 ± 0.10 for Bishop, and 0.55 ± 0.07 for Toga zircons. The resulting three $f_{\text{Th/U}}$ values agreed well within the analytical uncertainties. The disequilibrium-corrected ^{238}U - ^{206}Pb age can be calculated under the assumption that the $f_{\text{Th/U}}$ value did not vary significantly among the zircons. To evaluate this, we have measured the ^{238}U - ^{206}Pb and ^{232}Th - ^{208}Pb ages for zircons from Sanbekisuki tephra [6]. The $f_{\text{Th/U}}$ values used for the correction was based on the weighted mean of three $f_{\text{Th/U}}$ values obtained here ($f_{\text{Th/U}} = 0.53 \pm 0.05$). The corrected ^{238}U - ^{206}Pb age was 86.2 ± 2.1 ka, which agreed with the ^{232}Th - ^{208}Pb age (90.1 ± 2.6 ka) within the analytical uncertainties. It should be noted that the ^{238}U - ^{206}Pb dating after the correction of the initial disequilibrium can provide accurate and precise chronological information. To evaluate the reliability of the present correction technique for the U-Pb dating, we have developed a pseudo concordia diagram (plot of $^{206}\text{Pb}/^{238}\text{U}$ ratio against the $^{208}\text{Pb}/^{232}\text{Th}$ ratio). In this diagram, most of the U-Th-Pb isotope data fall close to a concordia curve, suggesting that the Sanbekisuki zircon did not suffered from significant Pb-loss. In conclusion, we can construct more accurate and effective dating tool based on the U-Th-Pb decay systems based on the $f_{\text{Th/U}}$ value defined in this study, especially, for the young zircons.

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Keywords: zircon U-Pb dating, disequilibrium, Quaternary zircon, precise isotopic analysis, laser ablation, ICPMS

SHRIMP geochronology of the Hida gneiss in the Wada-gawa area, Toyama Prefecture

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The Hida belt, situated at the northern part of southwestern Japan, consists of low P/T metamorphic rocks such as paragneisses, orthogneisses, amphibolite and marble with multiple episodes of metamorphism evident, and Permo-Triassic granitoids. Previous works suggested that an earlier metamorphism occurred at ca. 350Ma under the granulite-facies conditions, and was overprinted by the amphibolite-facies metamorphism at 240-220 Ma (e. g., Arakawa et al., 2000) but these data were probably distributed by the Funatsu-type granites intrusion at about 180 Ma.

In regard to protolith, Sano et al. (2000) reported U-Pb zircon ages peaked at about 3420 Ma, 2560 Ma, 1840 Ma, 1130 Ma, 580 Ma, 400 Ma, 360 Ma, 285 Ma, and 250 Ma from the Hida gneiss at Amo area. Asano et al. (1990) also reported protolith ages of 415 +/- 189 Ma (Sm-Nd whole rock isochron) and 413 +/- 60 Ma (Sm-Nd mineral isochron) from basic metamorphic rocks and amphibolite at the Wada-gawa area, respectively. However, there is no precise geochronological data for protolith and the timing of the metamorphism. In this study, the Hida gneisses collected from the Wada-gawa area were analyzed by SHRIMP (Sensitive High-Resolution Ion Microprobe) to discuss about the protolith and the timing of the metamorphism of the Hida belt.

The Hida gneiss sample, WD090810-3, is composed of biotite, orthopyroxene, plagioclase, quartz and other minor mineral, such as prehnite, titanite, zircon, apatite and opaque minerals. Some biotite is chloritized. Most of plagioclase is also altered and fresh ones were partly observed. Zircon grains of the sample are rounded to well-rounded morphologies. Cathodoluminescence images reveal existence of overgrowth rim.

U-Pb dating of the zircon core yielded five age peaks centered at about 2526 Ma, 1864 Ma, 760 Ma, 553 Ma and 316 Ma, which indicates that the protolith is probably a sedimentary rock. Some age peaks are consistent with those reported by Sano et al. (2000). The youngest age peak suggests that the protolith was formed after 316 Ma. The overgrowth rim yielded weighted mean of ²⁰⁶Pb-²³⁸U ages of 247.7 +/- 0.5 Ma (MSWD = 1.18), which indicates the timing of metamorphism in the Hida belt. This age is first report of precise age of the Hida metamorphism and we will discuss about thermal history of the Hida belt with U-Pb titanite ages of the Hida gneiss.

U-Pb dating of Eoarchaeon zircon using a NanoSIMS

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Volatile compositions, such as hydrogen and/or sulfur which are included in the Eoarchaeon igneous rock, have crucial information to reveal the evolution of interior of the early Earth. Apatite and/or glass inclusions, found in the zircon crystal, are expected to preserve a "primitive" information of such volatile elements, though a high sensitive and high resolution analytical method are required for it. NanoSIMS is one of the most powerful tools to analyze such volatile compositions in micro-scale inclusions in zircon crystals with precise in situ U-Pb dating.

For the first step of this purpose, we performed ²³⁸U-²⁰⁶Pb and ²⁰⁷Pb-²⁰⁶Pb zircon dating using a NanoSIMS 50 ion microprobe, with the method developed by our group [1]. A 5 nA O⁻ primary beam, with 20 micrometers in diameter spot size, was used for ionization of sample surface, and secondary positive ions were collected in multicollector for mass analyses. The detector system was modified to measure ³⁰Si⁺, ⁹⁰Zr⁺, ¹⁶O⁺, ²⁰⁴Pb⁺, ²⁰⁶Pb⁺, ²³⁸U¹⁶O⁺, and ²³⁸U¹⁶O₂⁺ ions simultaneously in ²³⁸U-²⁰⁶Pb dating session. In ²⁰⁷Pb-²⁰⁶Pb dating session, ²⁰⁴Pb⁺, ²⁰⁶Pb⁺, and ²⁰⁷Pb⁺ ions were collected in one detector by scanning the magnetic field. A multicrystal zircon, QGNG (zircon extracted from Quartz-Gabbro-Norite-Gneiss from South Africa) with a U-Pb age of 1842 Ma, was used for standard of U-Pb dating [2].

The targeted zircons were separated from tonalite which was from Eoarchaeon Nuvvuagittuq supracrustal belt, Superior Craton, Canada. The reported U-Pb age of this tonalite is 3661 +/- 4 Ma by using LA-MC-ICP-MS [3]. Euhedral to subeuhedral zircons were picked up to measure. The size distribution of zircons was from approximately 50 micrometers to 200 micrometers as the long axis of each crystal. Some of them have inclusions of glass or apatite whose size were 10 to 30 micrometers in diameter. Dating measurements were done avoiding such inclusions. Also, some of measured zircons have zonal structure. In such case, spot measurements were done by zone by zone for each zoning crystal.

Measured ²⁰⁶Pb/²³⁸U ratios range from 4.932E⁻¹ to 7.644E⁻¹. These ratios get smaller toward the edge of zoning crystal. The ²⁰⁷Pb/²⁰⁶Pb ratios range from 3.052E⁻¹ to 3.407E⁻¹. After the correction of common Pb, ²³⁸U/²⁰⁶Pb* and ²⁰⁷Pb*/²⁰⁶Pb* values were plotted on Terra-Wasserburg Concordia diagram. They showed a Discordia suggesting recent Pb loss. The intersection of Concordia and Discordia indicates that the age of this rock is 3633 +/- 35 Ma, which are agreed well with previous study. Now we are trying to measure the volatile compositions of inclusions in these zircons. They could provide us primary information about the evolution of the Early Earth.

References

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Keywords: NanoSIMS, zircon, U-Pb age, Pb-Pb age

Chemically estimated depositional and zircon ages from metacarbonate rocks in the Sor Rondane Mountains, East Antarctica

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Estimation of timing of carbonate deposition implies the presence of platform environment for the accumulation of sediments from surrounding continents. As a consequence the determination of deposition ages in metasedimentary sequences is important in understanding the tectonic history of continental collisions and closure of oceans to form supercontinents. In general, radiometric dating, such as U-Th-Pb, of key horizons or the interval between youngest protolith age and metamorphic age in zircon from metasedimentary rocks helps us to determine the sedimentation age. However, zircons in metasedimentary rocks will provide information of provenance of source rocks in a wide interval between opening and closure of ocean. For this reason, other methods have to be employed for estimating exact depositional ages. In this study we have selected a typical continental collision zone of the Sor Rondane Mountains, located in the African-Antarctic orogenic belt formed during the Neoproterozoic to Cambrian time. This region is composed of medium- to high-grade metasedimentary, metaigneous and intrusive rocks of diverse composition. Shiraishi et al. (2008) and other studies reported wide range of depositional ages that were estimated by detrital and metamorphic ages of zircon from ortho- and paragneiss. Recently, Otsuji et al. (2013), estimated the depositional ages of 880-850 Ma and 820-790 Ma (late-Tonian and early-Cryogenian age) for the metacarbonate rocks by using strontium and carbon isotope chemostratigraphy. The metacarbonate rocks are considered to have deposited chemically in the so-called the "Mozambique Ocean" that separated the continental blocks of Gondwana and possibly record geochemical signatures of contemporaneous seawater. However, according to the results by Otsuji et al. (2013), there are regional differences in their depositional timing. The determination of sedimentation ages may not be straight forward, and it has to be confirmed by the correlation with material derived from continental blocks. Here we present age information from zircon grains in impure metacarbonate rocks.

Petrographic observations of impure metacarbonate rocks, that contain relatively higher modal abundance of calc-silicate minerals, have shown that zircon is present in impure carbonate rocks from the Sor Rondane Mountains. Therefore it is possible that the zircons in impure metacarbonate rocks might be of detrital origin and record information about the provenance of pelitic components within the carbonate sediments. In contrast to the expected detrital ages, we obtained well-defined tight concordia U-Pb zircon ages of 545 +/- 1, 546 +/- 2 and 549 +/- 2 Ma, from three different layers in the Balchen region of the Sor Rondane Mountains. This age represent the latest phase of metamorphic age for this region, as reported in many recent studies. The zircons in metacarbonate rocks show hydrothermal re-equilibration texture on cathodoluminescence observations. Most of them have rounded shape, characterized by the absence of oscillatory growth texture, and shows dissolution-precipitation structures. Metacarbonate rocks are usually depleted in zirconium, however those in the Balchen region have abundant zircons. In general, zircon shows enriched heavy-REE pattern, whereas zircon in metacarbonate rocks from Balchen has flat REE pattern and low HREE concentrations, consistent with the rare earth pattern of zircons formed by hydrothermal activity. In addition to the high Cl-rich fluid activity around 600 Ma, our result shows that another important fluid activity was present in Balchen at around 545 Ma. Similar zircon age is reported from the matrix zircon in pelitic gneiss from Balchen (Higashino et al. 2013), implying that pelitic lithology also experienced the same fluid activity at around 545 Ma.

References; Shiraishi et al. 2008. *GSL special publications*, 308, 21-67; Otsuji et al. 2013. *PR* (in press); Higashino et al., 2013. *JpGU abstract*.

Keywords: depositional age, metacarbonate rocks, the Sor Rondane Mountains, zircon