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 <s>a </s>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 Atrea fault showed remarkably lower ages than those of cataclasite<s>. </s>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 <sup>38</sup>Ar/<sup>36</sup>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 are found to be collected through the method of Amli 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 Sowth 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

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Development for in-situ radiogenic <sup>4</sup>He analysis in zircon

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Development for in-situ radiogenic <sup>4</sup>He 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 <sup>4</sup>He 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 <sup>4</sup>He analysis in current U-Th-He dating. Since a typical range of an  $\alpha$ -particle is ~20 µm, the conventional apparatuses for <sup>4</sup>He analysis cannot accurately evaluate an escape of radiogenic <sup>4</sup>He from minerals by $\alpha$ -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 shold 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  $\alpha$ -particle because a spatial resolution of LIMAS is less than 1 µm in case of analysis for trace amount of <sup>4</sup>He (Bajo et al., 2015). LIMAS can measure depth profile, and analyze distribution of the 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 <sup>4</sup>He 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 <sup>4</sup>He 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 <sup>4</sup>He was supposed to be 55 ppm based on the U and Th concentrations and U-Pb age.

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

Keywords: zircon, U-Th-He dating, 4-He, 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 <sup>238</sup>U and <sup>235</sup>U 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  $U0^{+}/U^{+}$ (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 x  $({}^{254}(U0)^+/{}^{238}U^+)^2$ . 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<sup>+</sup>, U<sup>+</sup>, U0<sup>+</sup>, and U0<sub>2</sub><sup>+</sup> was tested using TEMORA2 zircon. The precision of Pb<sup>+</sup>/U<sup>+</sup> ratios seems to be improved by using the correlation between Pb<sup>+</sup>/U0<sup>+</sup> and U0<sub>2</sub><sup>+</sup>/U0<sup>+</sup>, which is probably derived from similarity of energy distribution between Pb<sup>+</sup> and U0<sub>2</sub><sup>+</sup> rather than between Pb<sup>+</sup> and U<sup>+</sup>. 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