
SGL42-01

Room:419

Time:April 29 09:00-09:30

Why is the IntCal13 special?

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Please see Japanese abstract (the presentation will be in Japanese).

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

An evaluation of the effect on ^{14}C dating (AMS) by alkaline treatment of the ABA method on charcoal sample

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Charcoal treatment by means of the Acid-Base-Acid (ABA) method (or Acid-Alkali-Acid; AAA method) has been widely used for radiocarbon (^{14}C) dating in the Earth Science and Archaeological field. Although the ABA method is a basic charcoal pretreatment method for ^{14}C dating, the evaluation of processing conditions of the ABA method based on any chemical indicator does exist few until today. This study aims to clarify the error of ^{14}C dating caused by the alkaline pretreatment which is not studied hitherto. The author performed 3 types of experiments for the purpose. The first experiment was performed for confirmation of the reproducibility of ges. The second experiment was performed for confirmation of optimal treatment time of an alkaline solution. The third experiment was performed for confirmation of the optimal concentration of alkaline solution for the ABA pretreatment.

The first experiment: X^2 test of the results shows $T=0.45$ ($df=3$;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 variability.

The second experiment: even after the visual judgment of the completion of alkaline extraction, 3-DF detected humic acid in the retrieved NaOH solution, and Atsumi et. al. (2009) showed that radiocarbon (^{14}C) dating was influenced the existence of humic acid. These results suggest that visual observation is inadequate for the judgment of the completion of alkaline extraction, and that 3-DF is more effective for monitoring the presence of dissolved organic contamination.

The third experiment: three charcoal samples from a single archaeological context were split into 8 aliquots respectively, and treated with 8 different concentrations of NaOH solutions ranging from 0.001 to 2.0 mol/l. Dating results and X^2 tests showed minimum convergence at 1.2 mol/l. This is supported by 3-dimensional fluorescent (3-DF) analysis, which clearly shows different leaching characteristics between 2.0-1.0 and 0.5-0.001 mol/l. 0.5-0.001 mol/l NaOH solutions were too weak in humic leaching capacity at low excitation ranges, which is thought to be the phenomenon that generates the scattering of dates. We recommend using from 1.0 to 1.5 mol/l NaOH for radiocarbon pretreatment.

These results show that the ^{14}C age is affected by difference of the residual of humic acid caused by the difference of chemical conditions of the pretreatment .

Keywords: ^{14}C dating, ABA pretreatment, 3-D fluorescent spectroscopy, Humic acid, Charcoal sample

Dating of sea-floor hydrothermal barite collected at the Okinawa Trough by ESR and radioactive disequilibrium

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

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

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

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

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

Thermoluminescence property of calcite

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In earth science, the date of past event is very important. To determine accurate age, we have to select suitable dating method for an analyzed sample.

Thermoluminescence dating method calculates a date from equivalent dose estimated by emitted luminescence when mineral is heated. Thermoluminescence dating has been often applied to carbonate minerals because it emits strong luminescence and wider age range applicable than C14 dating method is an advantage of thermoluminescence dating.

However, thermoluminescence dating of calcite is sometimes problematic; e.g., sensitivity change of calcite occurred through repeated heating of samples, possible anomalous fading, difference in characteristics of luminescence response against different kinds of radiation (e.g., gamma-ray, beta-ray, alpha-ray, and x-ray), and so on.

In this study, calcite from Philippine, Mongol, and synthetic calcite are analysed to understand luminescence characteristics of calcite. Mgnesite is also analyzed to see the effect of chemistry. Luminescence was detected by Photon Multiplier (R649, HMA-MATSU) with filter of 600-650nm. Chemical composition, especially impurity concentration was measured by LA-ICP-MS.

First, we evaluate X-ray induced thermoluminescence property of each sample.

Second, we measured luminescence caused by alpha-ray, beta-ray and gamma-ray and compare it to the luminescence induced by the x-ray. .

Finally, the relationship between luminescence characteristics (namely a-x-value, b-x-value and c-x-value) and impurity concentration is examined.

As a result;

1. Most calcites have thermoluminescence peak at 80 and 230 degrees Celsius.
2. In thermoluminescence peak of calcite at 80 degrees Celsius, fading is detected, while at 230 degrees peak is stable.
3. Results show negative or positive relationship between luminescence efficiency factors (a-x-value, b-x-value and c-x-value) and Mg, Mn, Fe and Sr concentrations.
4. The concentration of Fe has a correlation with a luminescence emitting efficiency.

Fe plays an important role for thermoluminescence of calcite. Thermoluminescence property of calcite may be subject to multiple chemical factors (ex; Mg, Mn and Sr), therefore, further analyses on calcites with the variety of impurity is necessary to evaluate a relation between multiple impurity concentration and thermoluminescence properties quantitatively.

Keywords: thermoluminescence, calcite, dating

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

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Fission track (FT) method is a dating technique based on the observation of damage (tracks) by spontaneous fission of ²³⁸U left in a mineral. The number of tracks is counted under an optical microscope after etching (chemical expansion of a track). However, as FT density per unit area rises, it becomes difficult to count the number of tracks. This is due to the fact that FTs overlap one another and are unable to be readily distinguished. The atomic force microscope has a potential to observe FT with high track density after a short time etching. However, when etching time is too short, the number of counted tracks were increased probably due to difficulties in recognizing the FT among structures other than FT (e.g., alpha recoil tracks). In the observation of young zircons collected from modern volcanic product, the surface structures found in old zircons do not exist, and a hole with the depth of ~10nm can be found on the smooth surface. These countable holes may lead us to the alpha recoil track dating.

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

Excess argon in phengite from the Sanbagawa eclogites: Constraints on argon behavior during subduction zone metamorphism

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

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

Keywords: Sanbagawa belt, eclogite, phengite, excess argon

Deciphering early Earth's differentiation using short-lived isotope systematics

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

Keywords: Hadean, early differentiation, extinct radionuclides, non-chondritic Earth

Formation age of Fengtien Nephrite, Taiwan: Dating low-temperature thin (<20 μm) zircon rims by NanoSIMS

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

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

Significance of external morphology and zircon chemistry for precise U-Pb zircon dating

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

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

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

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

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

LA-ICP-MS U-Pb dating of Oki Dozen volcano using non-polished zircons

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LA-ICP-MS U-Pb dating using zircon is now widely used with great success. The normal dating process includes polishing zircons. This process is good in terms of avoiding surface contamination of common Pb and/or Pb loss and is essential for SHRIMP, in which only small fraction (drilling depth of 1-2 μm) is used for dating. Compared to SHRIMP, LA-ICP-MS ablates a much larger volume (drilling depth of $>10 \mu\text{m}$) of zircon. This means that it is easy to date zircons from the surface to the inner core of the crystal and examine the existence of inherited cores and potentially investigate the timespan of crystallization. Here we dated non-polished zircons for some reference samples (Fish Canyon Tuff and OD-3) and samples from Oki Dozen volcano. Zircons were ablated for 30 seconds using 213 nm Nd-YAG laser with 10 Hz repetition rate and 4-5 J/cm^2 energy density. Final drilling depth for 5 J/cm^2 was 27 μm and the $^{206}\text{Pb}/^{238}\text{U}$ ratio from 9-18 μm depth were used to determine ages. It was found that non-polished zircons yield reliable ages because of agreement with reference ages. The Oki Dozen samples yielded 6-7 Ma ages, in agreement with or slightly older than K-Ar ages of 5.4-7.4 Ma.

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

Crustal noble gases anomaly associated with fault movement and aftershock the 3.11 Northeast Japan Earthquake

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

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

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

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