

## Closure temperature of single grain biotite by laser step heating experiment

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The concept of closure temperature has been applied in estimating regional cooling and uplift in various geologic settings. It was first applied in K-Ar system, but several problems were pointed out such as complex geometry in diffusion, or distribution of domain sizes in minerals. These findings made difficult for geologists to apply the concept. As a result, it is applied in various cases without assuming an appropriate cooling rate, or a broad range of temperature is assigned (650 - 900°C in U/Pb zircon  $T_c$ ).

Closure temperature was calculated using  $^{40}\text{Ar}/^{39}\text{Ar}$  data in laser step heating experiments of single grain biotites assuming simple cylindrical geometry. The diffusion parameters were calculated from Arrhenius plot in the temperature range where dehydration effects are minimal (below 800°C). Although the characteristic radius  $r$  is about 250 microns, it always appears as  $D_0/r^2$ . Change of  $r$  does not affect the results.

Three types are recognized in Arrhenius plots, depending on the activation energy and dehydration effects. However, these differences do not seem to affect very much on the resulting closure temperature. Relation between the characteristics of diffusion and age spectra is discussed.

Keywords: closure temperature, laser step heating  $^{40}\text{Ar}/^{39}\text{Ar}$ , biotite

## Development of un-irradiated and un-spiked laser fusion K-Ar dating for single grain minerals (2nd report)

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A laser fusion K-Ar dating has been developed as an in-situ micro measurement of radiometric ages applicable for less-abundant minerals found on thin sections [1, 2]. Strong induced radio-activities in Fe-rich minerals by neutron irradiation prohibits collecting sufficient amount of mineral separates, which partly limits applications of Ar-Ar method[3]. Nonetheless, once K-Ar method establish for a single grain mineral containing trace K with <0.1 wt%, much wider applications are anticipated, e.g. fine minerals of hydrothermal origin. Thus, we have installed a laser fusion apparatus to GVI-5400He noble gas mass spectrometer of JAMSTEC, as a part of the TAIGA-project: Grant-in-aid for Scientific Research on Innovative Areas.

Quantitative determination of K for trace concentration, approx. 0.1wt%, using EPMA sometimes faces poor accuracy and/or precision for a requirement of accurate dating. Thus we have been tested single grain K measurement under a low blank protocol using graphite furnace atomizer to ensure high sensitivities. This K measurement follows a laser fusion Ar measurement applied to the retrieved single melted mineral grain itself. An accurate K-Ar age determination requires complete retrieval both K and radiogenic Ar. Thus, conditions laser irradiation and K quantitation have investigated using SORI-93 K-Ar standard [4].

Our preliminary results show a 15% older averaged age with more than 10% deviation for recommended age for SORI-93 (92.6+/-0.6Ma, [4]), which is still under investigation.

[1] Sato et al. (2008) Chikyukagaku, 42, 179-199.

[2] Sato et al. (2011) JpGU annual meeting 2011.

[3] Ishibashi et al. (2009) J. Geogr., 118, 1186-1204.

[4] Sudo et al. (1998) Geochemical J., 32, 49-58.

Keywords: K-Ar dating, laser fusion, single grain, SORI-93 K-Ar standard

## Evaluation of the influence of alteration on K-Ar dating for Hawaiian tholeiites

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To obtain reliable K-Ar ages, the lava samples need to meet various requirements, and lack of K and Ar loss during weathering or alteration is one of the most important considerations. It is desirable to choose fresh rock samples that have not been affected by weathering/alteration; however, such samples are generally not available among the tholeiitic lava of shields older than about 1 Ma. In order to evaluate the influence of alteration on K-Ar dating for Hawaiian tholeiites, unspiked K-Ar ages were measured for 21 samples from four lava flows with varying degrees of alteration collected from the Makapuu Head section of Koolau volcano, Hawaii. The samples were classified based on freshness of olivine phenocrysts and the groundmass olivine, and the presence of secondary minerals in vesicles. The age data was evaluated by means of  $K_2O/P_2O_5$  ratios,  $^{36}Ar$  volumes, and calculated atmospheric Ar contamination. The results indicate that the ages for samples with fresh groundmass olivine are reliable, even though olivine phenocrysts may be slightly altered (thin reaction rims) or secondary minerals may have crystallized in the vesicles.

Keywords: K-Ar dating, alteration, tholeiites, Hawaiian volcano

## Observation of radiation damage in zircon by atomic force microscope and its application to geochronology

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Fission track (FT) method is a dating technique based on the observation of damage (tracks) by spontaneous fission of <sup>238</sup>U 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. This research examines the potential of atomic force microscope (AFM) for FT dating using zircons after a short time etching.

Zircons with track densities of about 4, 6.5, 10, and 20 ( $10^6/\text{cm}^2$ ) are observed. The clearer image for a short time etching is obtained after polishing with colloidal silica solution. Several tracks were found connected through step-etching. Thus, to measure the exact track density, correction in number of tracks is necessary by comparing the images before and after etching. FT ages were calculated using the corrected track densities, and agreed with the ages obtained by conventional methods.

In addition to fission tracks, there were numerous topographic lows in an AFM image. Due to these topographic lows, the cross sections shows cyclic waves. Number of these lows are estimated from the wavelength of the cyclic waves under the assumption that these topographic lows distribute on the surface evenly. As the result, the value obtained from wavelength of 0.10 micron was matched in the order to the expected number of alpha recoil tracks.

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

## ESR dating of tephra with quartz: inconsistency between signals

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Determining the age of tephra is important 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 <sup>14</sup>C 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 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 Ft to check the reproducibility of dating results and to test if known doses can be recovered using the multiple-aliquot regenerative-additive dose procedure.

The age obtained from Ti-H center is consistent with the age reference for Nm-Sb while the ones from Al and Ti-Li center overestimate. The ages obtained from Al and Ti-H center are consistent with the age reference for A-Fm while the ones from Ti-Li center overestimate. The dose recovery test indicates that the equivalent dose estimate based on the Ti-H center of Nm-Sb and A-Fm agrees within error of the expected dose (370 Gy). We consider the dose recovery test to be a useful procedure for choosing the signal appropriate for dating.

Keywords: quartz, ESR, dating, tephra

## Re-evaluation of ABA pretreatment for $^{14}\text{C}$ dating of fossil charcoal from late Holocene layer

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It had been suspected and had been tried to clarify the effect of the contamination by exterior organic matter on the  $^{14}\text{C}$  date from the beginning of development of radiocarbon dating. A comparison between ABA method and Acid-Base-Oxidation (ABOX) method confirmed the effect on the  $^{14}\text{C}$  date of contaminated carbon using AMS. However, the research didn't examine objectively of the chemical quality of late Holocene charcoal samples. And, there exists no research with the objective method for the effect of residual external organic carbon after pretreatment on the  $^{14}\text{C}$  date. Furthermore, in the researches of quality control of samples and pretreatment as described above, there were many cases where alkaline extraction time were less than 3 hours hence it was strongly suspected of the remnant of humic acid in dating samples. Thus, the discussion on what is reasonable and reliable sample in the  $^{14}\text{C}$  dating method and the discussion on the most suitable chemical treatment condition are still left unsettled. Recently, a few researches are reported which based on objective chemical indicator for the ABA method. The assessment for charcoal using raman spectrometry has attempted, and the result shows the existence of humic acid in charcoal samples. But, the relation between the pretreatment and the  $^{14}\text{C}$  dates has not been investigated yet. The mechanism of humification has been deduced from the comparison of fossil and recent charcoal. But it only shows humification under special desert environment, and has not investigated the effect of humification on the  $^{14}\text{C}$  date. Moreover, there exists no reliable chemical method to distinguish external organic matter and humified charcoal of sample charcoal. But we know empirically that charcoal's characteristics to resist against NaOH solution are different by burial and preserved states. Therefore, many researchers are using NaOH solution of low concentration when charcoal's characteristics to resist against NaOH are weak. The problem on the conventional ABA method is that what degree of concentration of NaOH solution is the most effective to the  $^{14}\text{C}$  dating samples. There exists no consensus on the problem. Here, we require the adequate condition of sample and the adequate pretreatment condition to obtain reliable  $^{14}\text{C}$  dates. However, as far as we know, there exist few data and researches on conditions of ABA pretreatment. In the present research, we try 5 steps pretreatment using alkaline solution increasing concentrations stepwise in order to search the optimum condition of alkaline treatment stage of ABA method. We make comparisons by the  $^{14}\text{C}$  dates among residual charcoal of the individual pretreatment stages, and compare emission intensity of dissolved organic matter and absorbance of the extracted solutions of the individual pretreatment steps. Namely, the present research aims to clarify the problem of ABA method and its practical solutions.

The results of the  $^{14}\text{C}$  dates, UV-vis and 3-DF (3-dimensional fluorescence) show that there exists no correlation between the visible color of the solution and the intensity of humic emission of the solution. And, the results show that the multiple step treatment using NaOH solution of concentration from 0.001 M to over 1.2 M can enhance the effectivity of humic extraction in relatively short time (18-20 days). Furthermore, the multi-step extractions from low concentration of 0.001 M to high concentration of 1.2 M are necessary, because we can't predict the concentration of NaOH solution which yields high humic extraction effect. In addition, 3 DF meter must be necessary to confirm treatment effect by ABA method indirectly. The dating results show that the  $^{14}\text{C}$  dates converge or saturate between from 1.0 to 2.0 M in concentration of NaOH solution.

Keywords: ABA(AAA) pretreatment,  $^{14}\text{C}$  dating, late Holocene

## Thermochronological study of the dip-slip displacement and timing of initiation of the Atera fault

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Fission-track analysis was performed for 5 samples collected from 5 surface outcrops and 6 samples from the Kawaue boring core around the Atera fault, which is a large-scale, left-lateral active fault with dip-slip component of displacement. The results were ~76 Ma of mean zircon fission-track age from SW of the fault, 69 Ma from NE of the fault, ~42 Ma of mean apatite fission-track age from outside of the fault zone and ~22 Ma from inside of the fault zone. Mean track length from U-4 zircon, collected from the fault zone, was only shrunk and the length distribution was apparently bimodal. An interpretation of the distribution was that this sample would have been slowly cooled down from ~300°C, since around 40-60 Ma, derived from some forward calculations with annealing kinetics. In addition, we calculated the excess erosion of the hanging wall (NE of the fault) and true dip displacement of the fault, assuming that the age difference across the fault was caused by dip-slip displacement of the fault and difference of exhumation rate across the fault. The results led us to obtain the following three conclusions. 1. The dip-slip displacement of the Atera fault after 70 Ma is ~1km. 2. The present fracture zone along the fault was widely heated at after 20 Ma but before at least the Quaternary. The displacement is similar to the geomorphologically estimated displacement of basement rocks or topography across the fault. 3. Some minor intrusions or other heating and slow cooling events would have occurred only near U-4 at ~40-60 Ma. Therefore conclusion 1 is consistent with previous studies that indicated the present Atera fault activity restarted after the early Quaternary. Conclusion 2 indicates that the fracture zone already existed at from ~20 Ma to at least the Quaternary and perhaps the ancient fault activity had already initiated.

Keywords: Atera fault, dip-slip displacement, timing of initiation of active fault, fission-track



## Lower boundary of the Caledonian Barrovian metamorphic belt at Loch Leven, Scotland: Phengite K-Ar ages of metapelites

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Since the Barrow's report (1893), the Barrovian metamorphic belt in the Caledonides of Scotland has become one of the best-studied orogenic belts in the world. Based on many geological and isotopic studies, this belt has long been considered to be formed by arc-continental collision in the Ordovician-Silurian (the Grampian orogeny: ca. 480-430 Ma) (Dewey and Bird, 1999; Oliver, 2008). However, many controversies and fundamental uncertainties remain. For example, in terms of the polarity of subduction, Ryan and Dewey (1991) and Oliver (2008) assumed that the polarity of subduction was toward the NW, but Cliff et al. (2004) proposed the opposite. Also, the main reason for the "orogeny" and metamorphism for several decades has been just burial at depth caused by thrusting, which we consider to be a dated, unlikely concept.

The Barrovian metamorphic rocks in Loch Leven belong to the biotite zone, have been folded and thrust northwards (e.g., Treagus, 1974; Piasecki, 1980), and they overlie weakly metamorphosed rocks (Eilde Flags). Inferring from our understandings of the occurrence and exhumation of HP rocks in Japan (Aoki et al., 2008; 2009), and in many Barrovian orogenic belts worldwide (Agard et al., 2009), we predicted that the lower tectonic boundary of the Barrovian metamorphic rocks should have occurred at Loch Leven. In order to examine this model, we determined K-Ar ages of phengite-rich mineral separates from 6 metapelites in the area.

Our new phengite K-Ar ages are 398.2 +/- 10.4 (sample no. LL46), 406.4 +/- 10.6 (LL20), 405 +/- 10.5 (LL19), 399.7 +/- 10.5 (LL24) and 445.0 +/- 11.6 Ma (LL16). One sample (LL13) has an "anomalously young" age of 340 +/- 8.9 Ma, probably because of its low K<sub>2</sub>O content (1.3 wt%). The main results indicate a K-Ar age-gap between ca. 445 Ma and ca. 400 Ma, which, when integrated with previous metamorphic-age (e.g., Oliver, 2008) and structural data from Loch Leven (Rovers, 1976; Atherton, 1977) suggest that the lower boundary of the Barrovian metamorphic belt formed at 445-400 Ma.

The lower boundary of the E-dipping, Barrovian metamorphic belt at Loch Leven has long been considered to be an extension of the 430-413 Ma Moine Thrust (MT) displaced by the Great Glen Fault. The shear sense of the E-dipping MT is top-to-the-WNW/NNW (Butler, 2004). Moreover, we have recently discovered that the upper boundary of the originally E-dipping Barrovian metamorphic belt crops out near Portsoy in NE Scotland, the shear sense of which was top-to-the-SW (Kawai et al., in prep.). In terms of the internal thermobaric Barrovian zones of Scotland, the grade of metamorphism decreases symmetrically upwards and downwards from a central highest-grade zone (Kennedy, 1948), which contains relicts of retrogressed HP rocks. Integration of all multi-disciplinary data provides robust evidence that the Barrovian metamorphic zones were exhumed from a HP depth by N-directed wedge extrusion. The lower wedge boundary was the Moine thrust-Loch Leven thrust, and the upper extensional boundary at Portsoy enabled the downward emplacement of the overlying, lower pressure Buchan metamorphic zonal belt. Thus, formation of the whole Caledonian orogenic belt of Scotland was controlled by wedge extrusion.



## U-Pb geochronological map of Unazuki area

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The Unazuki area, situated at the northeastern part of the Hida Belt, which is one of crucial sites for deciphering the Permo-Triassic orogeny in East Asia, has experienced the kyanite-sillimanite type metamorphism characterized by a clockwise P-T path. The Unazuki area is classified into the Hida gneiss region (Katakaigawa group), the Unazuki schist region (Unazuki group), and the Unazuki plutonic complex. Radiometric ages of the Unazuki schists, previously determined by Rb-Sr and K-Ar methods, are scattered from 248 Ma to 175 Ma primarily because of multi-phase metamorphism and deformation. Geochronological data for the Unazuki plutonic complex are limited. In this study, U-Pb geochronology of zircon and titanite was applied to the schists and the plutonic complex to discuss about timing of the kyanite-sillimanite type metamorphism and thermal history.

Protoliths of the Unazuki schists are sedimentary and felsic volcanic rocks. Probability of U-Pb data of detrital zircons in pelitic schist shows some peaks centered at 453, 365, 347, 320, 310, and 298 Ma, which suggests that protolith of the Unazuki metamorphic rocks were deposited after 298 Ma. U-Pb data of quartzo-feldspathic schist derived from felsic volcanics yield an eruption age of 258 +/- 2 Ma, indicating that regional metamorphism occurred after 258 Ma. On the other hand, U-Pb age of a granite in north part of the Unazuki area is 253 +/- 1 Ma. The granite contains some xenoliths of the Unazuki schist, in which staurolite is replaced by andalusite and cordierite due to thermal flux from granitic magma. Therefore, regional metamorphism occurred between 258 and 253 Ma, suggesting a rapid metamorphic progression. 251 +/- 1 Ma of gneissose quartz diorite containing the Unazuki schists supports the timing of the regional metamorphism.

The granite in central part contains Eoarchean-Paleoproterozoic zircon inheritance and shows 256 +/- 2 Ma, whereas that in south part is 250 +/- 1 Ma. Hornblende quartz diorite in central part yields 191.1 +/- 0.8 Ma, whereas biotite quartz diorite in south part is 275 +/- 2 Ma. Meta-gabbros in central part yield 260-256 Ma. Some parts of zircon in meta-gabbros were recrystallized at ca. 236 Ma.

Eboshiyama mylonite in the Katakaigawa group shows 250.0 +/- 0.4 Ma, which suggests that mylonitization of the Katakaigawa group occurred after the regional metamorphism of the Unazuki group.

Keywords: geochronological map, U-Pb age, zircon, Unazuki, Hida belt

## Age distribution of detrital monazites in the sandstones from the northern Borneo and its tectonic setting

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Late Cretaceous to Late Eocene turbidite is widely distributed in the northern part of Borneo. It is known as the Rajang Group. Heavy minerals were collected from the four rivers that flow through this stratum. Detrital monazites were analyzed by EPMA to obtain their ages. The monazite age distributions of the four rivers show three main peaks at 200-300 Ma, 400-500 Ma and 1850-1900 Ma, and a weak cluster at 700-1100 Ma. Such age distributions show that the detrital grains were not supplied from Southeast Asia, but from the southern part of China. There is a huge unconformity, called as Sarawak Orogeny, which occurs after deposition of the Rajang Group. The age is the Latest Eocene, close to the opening of the South China Sea which began as early as the Early Oligocene. Hence, it is probable that the Northern Borneo situated at oceanic side of the South China Sea have been moved to its current position during the opening of the South China Sea. There is no clear tectonic event after the opening of the South China Sea between the northern Borneo and western Indonesia Archipelago. Assuming that Borneo and western Indonesia Archipelago were moved at the time of the opening of the South China Sea, the reduced connection between the Indian Ocean and the Pacific Ocean at Miocene and also the Wallace line for fauna and flora may be explained more simply than the recent reconstruction models.

Keywords: monazite, age, Borneo