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

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

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

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

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

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

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

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

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

Keywords: U-Th radioactive disequilibrium, seafloor hydrothermal vent

Problems related to the numerical age in a time table

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

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

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

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

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

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

Brittle fault dating -looking through a clutter of ages

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

Brittle fault illite K-Ar age data and δD from several studies in Europe, Scandinavia and Japan will be presented. All study areas are located within igneous or metamorphic rocks collected from tunnel or drill core samples, which offer a unique advantage as no detrital illite is present in the host rock, thus reducing potential contamination and weathering sources. The age data were obtained using a simplified and standarized method described by Zwingmann *et al.* (2010) [2]. Ages range from the Mesoproterozoic (1240±26 Ma) for the Finland to the Neogene (6.0±2.1 Ma) for the European Alps study. Fault gouges in Japan scatter around the Paleogene –early Eocene. The illite ages decrease with grain size, and are consistent with the cooling history of host rocks as bracketed by Ar-Ar, AFTA and ZFTA ages. The data indicates that the fault-rock samples formed within the stability field of illite and the main temperature field of brittle deformation (< ~ 300°C). The internal consistency of the illite K-Ar ages of fault gouges from samples, as well as their consistency with independent constraints from field relationships and existing geochronological data, demonstrate the potential of this simplified method for providing reliable data to constrain absolute timing of brittle deformation.

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

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

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

Diffusion in a mineral and unclosure temperature: sphere

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

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

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

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

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Hydrothermal activity is an essential phenomenon to drive geochemical differentiation on the Earth's surface, which should form polymetallic massive sulfide ores. Noble gas isotopes are regarded to be a powerful tracer even for the geochemical study on hydrothermal activity; however, there are abundant interference volatiles, e.g. halogens and hydrogen sulfide. Here, we designed a new preparation system to remove such volatiles using non-traditional gettering materials. This new preparation system consists of following components: the water preparations system, composite gettering system (halogen getter, sulfide getter and Ti-Zr getter), cryogenic pump, SAES-getter pump and charcoal trap to analyze the all noble gases (He, Ne, Ar, Kr and Xe). The three types of getters effectively remove halogens, sulfides, abundant water vapor and other active gases for respective gettering step. After the absorbing sea water by cold trap and collection Ar-Kr-Xe fraction in the charcoal trap, the gettering procedure were taken under the following orders: halogen gettering, sulfide gettering, water vapor and active gases. For the efficient removal of sulfides, an exposure to the halogen-getter of the extracted gases prior to the exposure to the sulfide getter is required. Then, respective He and Ne fraction, separated with cryogenic pump, was measured. Next, Ar, Kr and Xe fraction were further purified and separated with charcoal trap, respectively. Further, the new preparation system has compact volume, which needs small amount of seawater sample of 2-5 cc in volume. It is almost 1/20 compared to the typical requirement for He isotope measurement. This newly designed preparation system has been applied to the Patent filing (Application Number: 2015-234839).

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

Keywords: submarine hydrothermal fluids, noble gas, gettering system

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

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

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

Keywords: Radiocarbon Dating, ABA method, Charcoal

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

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The oxygen isotope records presented in natural archives such as tree ring cellulose, speleothems, leaf wax, and ice cores are widely accepted for climate reconstruction. Among other materials, these records are strongly influenced by the isotope value of precipitation as they developed. A better understanding of isotopic variability in precipitation can enable better reconstruction of past environments. For example, in the tropics, various observational and modeling studies have been carried out to provide a greater depth of the interpretation of natural isotopic variability, leading to a common conclusion that isotopic variation in tropical precipitation is related to the large-scale convective activity, rather than precipitation amount. This interpretation proposes that isotope records from the tropical region is used to reconstruct past large-scale convective activity. For Japan, intense observation studies have already done to identify climate drivers controlling isotopic variability. Kurita et al., (2015) showed that north-south displacements of the Baiu front are manifest in the isotopic composition of precipitation in central Japan. In this study, we reconstructed 100-year long records of oxygen isotopic composition of summer precipitation from tree-ring oxygen isotope records in central Japan. And, we discuss the past location of the Baiu front using reconstructed long-term precipitation isotope data. Since the oxygen isotope ratio of tree rings is not directly influenced by the isotopic composition of precipitation, using mechanistic model predicting the oxygen isotopic ratio of tree-ring, we calculated the isotopic ratio of source water that a tree used during the growing season. The modeled isotopic ratio of source water was exchanged to the oxygen isotopic ratio of summer precipitation using an experimental polynomial function of sunshine duration (SD).

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

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

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

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

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

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

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

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

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The temporal change of submarine hydrothermal activities has been an important issue in the aspect of the evolution of hydrothermal systems which is related with ore formation Urabe (1995) and biological systems sustained by the chemical species arising from hydrothermal activities (Macdonald et al., 1980). Takamasa et al. (2013) and Fujiwara et al. (2015) showed that ESR (Electron Spin Resonance) dating of barite ($BaSO_4$) is useful for investigation of history of hydrothermal activities.

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

Toyoda et al. (2012) investigated the alpha effectiveness for the ESR signal due to SO_3^- in barite by comparing the dose responses of the signal for gamma irradiation and for He⁺ ion implantation with an energy of 4 MeV, to obtain a value 0.043 ±0.018. However, the dose response was far from "good", where the number of points is not sufficient. The experiments of He⁺ ion implantation was repeated in the present study for several samples to determine the precise alpha effectiveness. A sample of hydrothermal barite, taken by the NT12-06 research cruise operated by Japan Agency for Marine-Earth Science and Technology (JAMSTEC) was used for the present study. The values were obtained to be 0.025±0.002 for synthetic barite, and to be 0.102(+0.014/-0.013) for barite extracted from a sea-floor hydrothermal sulfide deposit, being four times larger than the former. For the latter sample, it is possible that the slopes of the dose responses of the signal intensities around the zero dose value may not be estimated appropriately due to the extrapolation of the dose response curve. Tentatively, the value for the synthetic sample should be adopted.

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

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

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

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

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T. Fukuchi, H. Inamura, D. Taguchi & T. Hirose, 2016, Distribution of active faults and change of seismic activity in and around Japan South Alps. Bulletin of the Faculty of Education & Human Sciences, Vol.17, p.219-226.

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Keywords: Japan South Alps, Itoigawa-Shizuoka Tectonic Line, ESR dating, Electron spin resonance, Fault rock, Smectite Observation of zircon by atomic force microscope for establishing Alpha Recoil Track method

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

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

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

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Keywords: Alpha Recoil Track, Zircon, Atomic Force Microscope