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Preliminary SIMS Zircon Age of Pelitic Gneiss from Oki-Dogo Island, Southwest Japan

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Abstract: Single zircon U-Pb analytical technique by SIMS (secondary ion mass spectrometry) was applied to metasediment from Oki-Dogo Island, southwest Japan. $^{207}Pb^{*}/^{206}Pb^{*}$ age of zircons comprise three generations; 2.3, 2.0, and 1.7 Ga. Well-rounded crystal shape of the analyzed zircons suggests that the sediments are detritus of a continental land mass which was Proterozoic in age. The age dispersion may be attributed to at least three individual magmatic activities in the Proterozoic continent.

Key words: geochronology, U-Pb age, zircon, SIMS, pelitic gneiss, metamorphism, Oki-Dogo Island

INTRODUCTION

Zircons have found widespread application in geochronology because of their high concentration of U and Th and low inherent concentration of Pb, and their high resistance to geochronological resetting during later metamorphic, melting, and other geological events. Another merit of zircon is its common occurrences in felsic to intermediate rocks and even in sedimentary rocks. Although there are several methods to date old rocks, single grain zircon dating by secondary ion mass spectrometry (SIMS) is now considered to be the most reliable dating technique for Precambrian/Paleozoic rocks. The advantage of SIMS compared to the conventional U-Pb method is its in situ and almost non-destructive analysis of small areas (<25 μm in diameter) within a single zircon. This enables us to analyze composite population of zircons with multi-stage histories and separate them into different age groups by dating several spots per one grain. Accordingly, zircon U-Pb ages have given us valuable information about early crustal evolution (e.g. Compston *et al.*, 1984).

This paper presents preliminary zircon ages of pelitic gneiss from Oki-Dogo Island, Shimane Prefecture, by analyzing with a commercial SIMS instrument. Although the rock has yielded several chronological data, the ages vary from 20 Ma (million years) to 3000 Ma (3 Ga). The wide variation is mainly due to different dating technique on different materials, while it may be caused by analytical uncertainties in bulk rock or bulk mineral analyses. The bulk analyses may have a problem because we cannot obtain correct age of metamorphism and/or crystallization of each mineral. The present SIMS U-Pb method is thus useful for determination of U-Pb age even from single zircon crystal. Another purpose of this paper is to test reliability of SIMS U-Pb technique presented by Tsunogae and Yurimoto (1995). This sample offers a suitable testing ground for evaluating present zircon ages with available K-Ar, Rb-Sr, and Sm-Nd ages.

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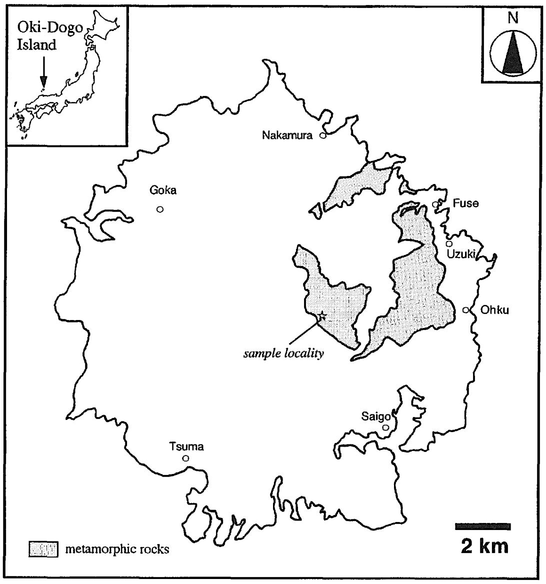

Fig. 1. Simplified location map of the Oki-Dogo Island with sample locality of pelitic gneiss. Shaded area denotes distribution of high T/low P metamorphic rocks (simplified from Ohta, 1963). Open circles are major towns.

BRIEF GEOLOGY OF OKI METAMORPHIC ROCKS

General geology and metamorphism

Fragments of high-T/low-P regional metamorphic complex crop out in the northeastern part of the Oki-Dogo Island (Fig. 1). The complex consists mainly of upper amphibolite to lower granulite facies metasedimentary gneisses with minor mafic and calcareous gneisses, and post-tectonic granite (Tomita, 1936; Ohta, 1963; Hoshino, 1979). In the area around Choshi River, metamorphic rocks are dominated in migmatized pelitic gneiss which is composed of quartz, plagioclase, K-feldspar, garnet, biotite, muscovite, chlorite, graphite, and ilmenite with or without cordierite, sillimanite, and andalusite (Fig. 2a). Owing to the absence of orthopyroxene in the rock, very few mineral assemblages and reaction textures are amenable to detailed thermobarometric analysis. As a result, pressure and temperature history of the terrane cannot be unraveled in great detail except several P-T estimates done by Hoshino (1979, 1981), Hamada (1995MS), and Tsunogae *et al.* (1995). Petrographical observations of leucocratic veins, which are probably products of partial melting of pelitic gneiss, show a pressure range of about 4 to 7 kbar for prograde stage. As the pelitic gneisses generally lack orthopyroxene, peak temperature was estimated to be $<820℃$ which corresponds to a temperature of the beginning of biotite dehydration melting. It is consistent with a temperature estimate of 830℃ from two-pyroxene assemblage in mafic gneiss. The pelitic gneiss was subsequently affected by retrograde garnet breakdown reaction to cordierite and biotite + sillimanite (or andalusite). Available geothermobarometric and thermodynamic data indicate that hydration of garnet + K-feldspar to biotite + aluminosilicate + quartz occurred around a boundary between sillimanite and andalusite fields.

Previous geochronologic data

The gneiss of Oki-Dogo previously gave several geochronologic data. Shibata and Nozawa (1966) first reported ages of the gneiss as 165 and 173 Ma by K-Ar method of biotite, whereas slightly older age was reported by Hayase and Ishizaka (1967) by Rb-Sr method as 187 Ma. On the other hand, mafic gneiss has yielded Sm-Nd model ages of 2000 and 166 Ma (Tanaka and Hoshino, 1987), while no detailed analytical procedure has been discussed in the paper. Recently Suzuki and Adachi (1994) presented CHIME (chemical Th-U-total Pb isochron) ages for pelitic gneisses as 350 Ma to 3 Ga for zircons and 250 Ma for monazites. They concluded that Oki terrane is composed of >350 Ma sediments and has suffered single metamorphic event at about 250 Ma. The gneisses are occasionally intruded by "younger granite" which gives a K-Ar age of 19.7 Ma (Tainosho *et al.*, 1991).

SAMPLE DESCRIPTION

Sample of pelitic gneiss (sample 1-8D) in which the analyzed zircons occur was collected from a quarry about 1.5 km northeast of Harada Township (Fig. 1). The rock forms a melanocratic part of banded migmatitic gneiss and contains quartz-plagioclase-biotite-muscovite-chlorite-garnet assemblage with minor zircon, apatite, graphite, and ilmenite (Figs. 2b and c). Four zircons which contain few inclusions were analyzed in this study. They are colorless to pale brownish transparent crystals, euhedrally zoned, and 40 to 250 μm in size (Fig. 2d). Their edges are generally well-rounded, implying detrital origin of the zircons (Figs. 2e and f). It suggests that they crystallized from granitic melt, and suffered subsequent rounding owing to erosion and sedimentation to form protolith of pelitic gneiss.

ANALYTICAL PROCEDURE

The Pb and U isotopic compositions of zircon grains were measured by a commercial CAMECA IMS-3F SIMS instrument equipped with the electrostatic peak switching system at the University of Tsukuba. The analytical procedures used on 3F were similar to those reported in Tsunogae and Yurimoto (1995) and Koike *et al.* (1993). Briefly, analyzed zircons were separated from sample, mounted in epoxy disk, and polished until they were approximately sectioned in half so that their cores were exposed. The polished disk was coated with ~30 nm thick of gold film for SIMS analysis. 3F was operated at 6000 mass resolution to eliminate all significant isobaric interferences. An 11 kV primary beam of negative oxygen ions ($^{16}O^-$) was focused on a small ($\leqq$25 μm in diameter) area from which sputtered positive secondary ions were extracted. Pb isotopic compositions were measured directly and not corrected for possible mass discrimination. Progressive changes in Pb^+/U^+ during sputtering were compensated by using an empirical quadratic relationship between Pb^+/U^+ and UO^+/U^+ determined on a standard Sri Lankan zircon (SL13). A radiogenic $^{206}Pb^*/^{238}U$ of 0.0928 (equivalent to an age of 572 Ma) was assumed for SL13 on the basis of independent isotope dilution measurements and was confirmed to be constant all over the grain (Kinny *et al.*, 1990). Sensitivity of the machine during data acquisition was about 1 cps/ppm for ^{206}Pb.

Since an intensity of $^{204}Pb^+$ was less than the detection limit, a correction for common

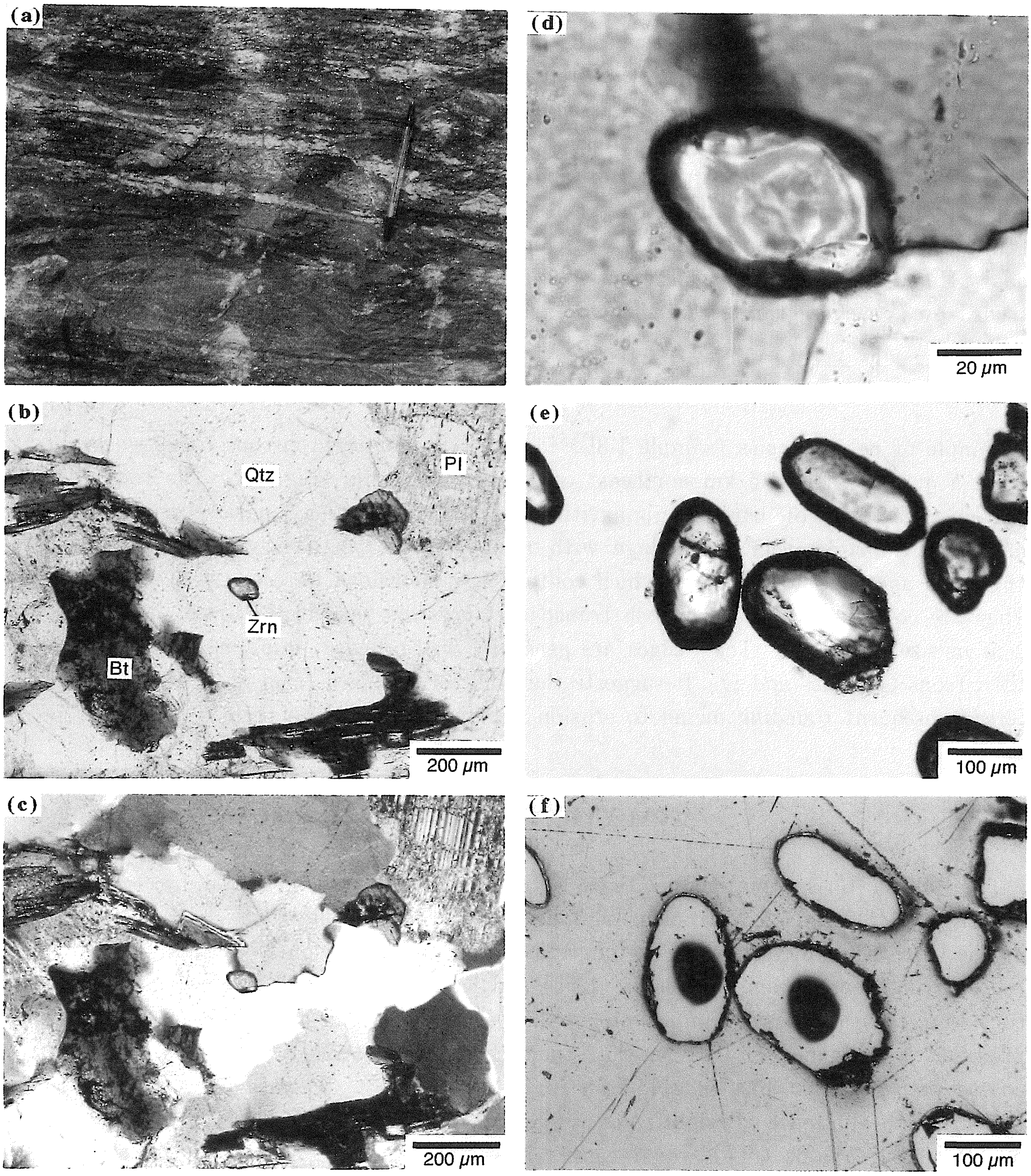

Fig. 2. (a): Typical outcrop of migmatized pelitic gneiss, Oki-Dogo Island, from which analyzed zircons were obtained. Compositional layering, defined by higher modal feldspar and quartz and coarser grain size in light-colored layers (leucosome), is typically parallel to the foliation defined by orientated biotite in dark-colored layers (mesosome). (b): Photomicrograph of small zircon in pelitic gneiss (sample 1-8D). Zircons are often included in quartz and plagioclase, and occasionally in biotite. Polarized light. (c): Same as (b). Crossed polars. (d): Enlarged photomicrograph

Table 1. Representative Pb/U analyses of zircons in pelitic gneiss from Oki-Dogo Island.

Sample	$^{207}Pb^*/^{235}U$ ± 1σ **	$^{206}Pb^*/^{238}U$ ± 1σ **	$^{207}Pb^*/^{206}Pb^*$ ± 1σ **	$^{208}Pb^*/^{206}Pb^*$ ± 1σ **	Common ^{206}Pb(%)	$^{207}Pb^*/^{206}Pb^*$ age (Ga)**
OK1	5.69 ± 18	0.339 ± 21	0.123 ± 10	0.0558 ± 23	0.0	1.98 ± 15
OK2a	4.89 ± 27	0.288 ± 24	0.124 ± 3	0.109 ± 2	1.3	2.01 ± 4
OK2b	4.48 ± 43	0.267 ± 28	0.123 ± 9	0.105 ± 6	0.1	1.99 ± 12
OK3a	3.59 ± 10	0.245 ± 9	0.107 ± 2	0.0544 ± 25	0.3	1.74 ± 4
OK3b	3.67 ± 65	0.249 ± 31	0.108 ± 8	0.0610 ± 22	0.1	1.75 ± 14
OK4	7.25 ± 42	0.363 ± 1	0.145 ± 9	0.158 ± 20	0.2	2.28 ± 11

* Radiogenic lead isotopes.

**Errors are shown for the least one or two figures.

Pb in zircon samples was made using $^{208}Pb^*/^{206}Pb^*$ and $^{232}Th/^{238}U$. The composition of the initial Pb in the zircons was assumed to be similar to that of average crustal Pb of the same age (Cumming and Richards, 1975). It has to be noted that the technique relies on the assumption that there was neither relative movement of Th and U nor differential movement of Pb isotopes within the zircon. An amount of non-radiogenic ^{206}Pb is shown in Table 1. The correction is normally small (less than 3% common Pb) and yielded an insignificant effect on determined ages. Regression treatment and calculation of concordia-discordia intercepts and errors followed the method of York (1969) and Ludwig (1980), respectively. The isotopic ages were calculated using decay constants recommended by the IUGS Sub-commission on Geochronology (Steiger and Jäger, 1977).

RESULTS

Obtained Pb^*/U and Pb^*/Pb^* with ages for zircons in the pelitic gneiss sample are shown in Table 1 and Fig. 3. One of the weakness of ion microprobe analysis is that, compared to conventional bulk zircon techniques, the determination of Pb isotopic composition is generally less precise. SIMS analyses in Table 1 are, however, still accurate and isotopic compositional differences in individual zircon grains can be well observed. When the data are plotted in a concordia diagram (Fig. 3), they show considerable dispersion. They lie close to concordia and thus imply little secondary movement of U and Pb within the crystals.

Grain OK4 is distinctly older than the rest of the population and has $^{207}Pb^*/^{206}Pb^*$ age as 2.28±11 Ga (1σ). In all cases the measurements were made on clear, unzoned cores, and this age can be therefore interpreted as reflecting the time of zircon crystallization during emplacement of original felsic magma. There is no evidence of significantly older zircon of >2.3 Ga.

Grains OK1, 2a, and 2b show slightly younger $^{207}Pb^*/^{206}Pb^*$ ages ranging from 1.98±15

of zircon in (c). The zircon shows euhedral magmatic zoning which is clearly displayed by color layering. (e): Transmitted light photomicrograph showing typical morphology of zircons dated in this study. Edge of the crystals are generally well-rounded, implying detrital origin of the zircons. (f): Same as (e). Reflective light. Black circles are analytical spots by sputtered primary beam irradiation.

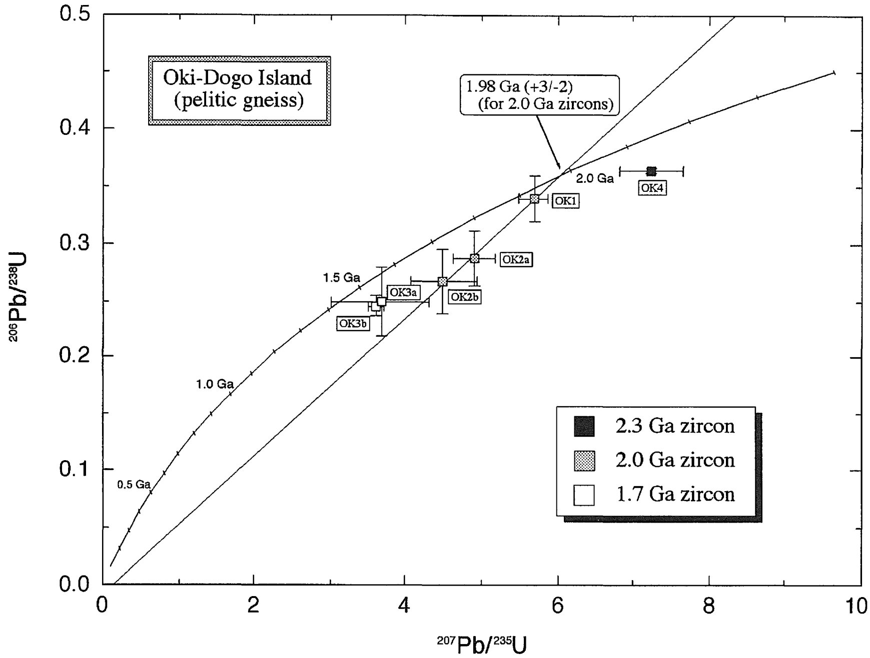

Fig. 3. Concordia diagram showing Pb/U ratios of three zircon generations. 2.0 Ga zircons define discordia line intersecting concordia at 1.98 +3/-2 Ga (1σ).

Ga to 2.01±4 Ga. Because the zircons show consistent $^{207}Pb^*/^{206}Pb^*$ ages, they can be fitted to a discordia line intersecting concordia at 1.98 +3/-2 Ga (1σ) and indicating recent Pb-loss (Fig. 3). It suggests redistribution of radiogenic Pb during later metamorphic or hydrothermal episodes.

The remaining two grains (OK3a and 3b) have $^{207}Pb^*/^{206}Pb^*$ ages of about 1.75 Ga. They plot above the 1.98 Ga discordia line in Fig. 3 and are clearly younger than the other zircons. Although the zircons apparently show recent Pb loss, discordia age was not calculated from the data because of few analysis.

DISCUSSION

It can be confirmed from new Pb/Pb data that zircons in metasediments of Oki-Dogo Island comprise three generations; 2.3, 2.0, and 1.7 Ga. Morphological characteristics of the analyzed zircons suggest that the pelitic gneisses are detritus of a continental land mass which was Proterozoic in age. The age dispersion may be attributed to at least three individual magmatic activities in the Proterozoic continent. The 2.0 and 1.7 Ga zircon generations are almost consistent with available CHIME age (Suzuki and Adachi, 1994) and also with unpublished conventional U-Pb zircon age by T. Yanagi (personal communication, 1994) on pelitic gneiss samples which yielded concordia intercept age of about 1.9 Ga, while this is the first report to date the 2.3 Ga magmatic event in the protolith continent.

Although this study failed to identify a timing of metamorphism, concordia diagram (Fig. 3) suggests possible modern (Phanerozoic?) metamorphic event and associated Pb-loss from the zircons.

These data also confirm a reliability of SIMS (CAMECA IMS-3F) dating technique introduced by Tsunogae and Yurimoto (1995). Until recently, zircon U-Pb chronological data have been restricted to works done by SHRIMP (sensitive high resolution ion microprobe) of Australian National University (ANU). This instrument differs from other SIMS mainly in its high sensitivity when operating at high mass resolution. In spite of the high reliability of isotopic data by SIMS, and SIMS has so far been installed in many institutions, it was not applied to zircon U-Pb method before preliminary work done by Tsunogae *et al.* (1992). As demonstrated in this paper, SIMS dating technique is enough to determine reliable U-Pb age at high mass resolution mode. We hope that single zircon dating by SIMS will become widespread and will stimulate chronological work on crustal evolution.

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二次イオン質量分析法による隠岐島後産泥質片麻岩のジルコン年代（予報）

角替　敏昭

二次イオン質量分析法(SIMS)によるジルコン単結晶のU-Pb年代測定法を隠岐島後の泥質片麻岩に応用し、年代を求めた。泥質片麻岩中のジルコンは自形の累帯構造を持つが外形は楕円形であり、砕屑性ジルコンであることを意味している。SIMS分析の結果、ジルコンは23億、20億、17億年の3つのグループに分けられる。形態的特徴から、これらジルコンは原生代地殻にて活動した酸性マグマ中で結晶化したものであり、泥質片麻岩はこの原生代地殻を起源として形成された堆積岩に由来する。