Zircon U-Pb dating of plutonic rocks at the Tsukuba area, central Japan

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Abstract Late Cretaceous to Paleogene plutonic rocks occur at the Tsukuba Mountains and the surrounding area. The rocks are mostly granitic rocks with minor amounts of pyroxene gabbro and hornblende gabbro. A total of eight plutonic rocks were analyzed by U-Pb dating of zircon using Laser Ablation Inductively Coupled Plasma Mass Spectrometry. The obtained zircon U-Pb ages cluster at 61-64 Ma and one granite sample has an age of 80 Ma. Although the age data by K-Ar methods showed that all the granites belong to younger group, i.e. 58-63 Ma, and the gabbros belong to the older group, the present results showed that the gabbros belong to the younger cluster as well as most of the granite. Older granite occurs around the Tsukuba Mountains. The present age result will contribute to extension of the Late Cretaceous to Paleogene plutonic rocks in the Japanese Islands.

Key words: age, zircon, granite, gabbro, Tsukuba Mountains

Introduction

Since 1964, the K-Ar method has been widely used for age analyses of granitoids in Japan (e.g. Kawano and Ueda, 1964, 1965 and 1966). Based mostly on the K-Ar method of the Kawano group, radiometric age data of granitoids in the Japanese Islands were summarized by Nozawa (1975). From the K-Ar method, it is well known that Cretaceous granitoids are the most ubiquitous in the islands. In central and northern Japan, Cretaceous granitoids, partly Paleogene, with an age of around 65 Ma occur along the eastern part of the Asahi Mountains and Tsukuba-Nikko area. In western Japan, granitoids with age of 60-70 Ma occur linearly along the coastal zone of the Sea of Japan. Yokoyama et al. (2016) obtained the thorite and uraninite ages from about 400 granitoids in the Japanese Islands. Although their data are mostly similar to the K-Ar age data, the uraninite and thorite ages are much more concentrated in each province. The K-Ar method involves a few problems. One is Ar loss during later stage thermal event and alteration. The others are an age difference among minerals used for the age analyses. Uraninite and thorite ages also have a closure temperature problem as shown by Yokoyama et al. (2016). They are lower in closure temperature than zircon and monazite, but higher than micaceous minerals.

There are many other methods used to obtain the ages of granitoids. Among them, SHRIMP and Laser Ablation Inductively Coupled Plasma (LA-ICP-MS) are the most reliable and useful for granitoids due to the common presence of zircon, its high closure temperature and strong resistance against alteration. Watanabe et al. (2000) applied the SHRIMP method to Cretaceous granite in the Ryoke low-pressure type metamorphic belt at the Kinki District. The Tsukuba area belongs to the Ryoke Belt. Although the SHRIMP and LA-ICP-MS methods have been used for the age analyses of Cretaceous and older granite in Japan (e.g., Kon et al., 2015; Ishihara and Orihashi, 2015), the age methods have rarely applied to granites in the Ryoke Belt.

Geological setting and samples

Figure 1 shows a geological map around the Tsukuba Mountains (cf. Takahashi, 2006). Basement rock of the plutonic rocks belongs to the Yamizo Belt that is Jurassic to Early Cretaceous subduction complex. The Yamizo Belt in this area is more or less metamorphosed by low-pressure type metamorphism and is thought to be eastern extension of the Ryoke Metamorphic Belt. The plutonic rocks are mostly granite with a minor amount of gabbro. The granite is classified geologically and geographically



Fig. 1. Geological map of the Tsukuba area (partly revised after Takahashi, 2006). Numbers in black squares are analyzed samples.

into three bodies called as Inada, Mt. Kaba and Tsukuba granite bodies (Takahashi, 1982; Miyazaki *et al.*, 1996). There are two blocks of gabbro called as the Mt. Tsukuba and Dorokushin (Miyazaki *et al.*, 1996).

The Inada Body occurs at the northeastern part of the Tsukuba Mountain and is subdivided into three types based on grain size, mineral assemblage and texture such as equigranular and porphyritic. The Inada Body is represented by coarse-grained biotite granite. The Mt. Kaba Body occurs at the Kaba and Ashio mountains and is subdivided into four types. The Tsukuba Body occurs around the Tsukuba Mountain. It is also subdivided into several types. The Mt. Tsukuba Gabbro Body occurs at the upper part of the Tsukuba Mountain and shows layered structure with locally anorthositic part (Tagiri *et al.*, 2013). It is pyroxene gabbro with a small amount of hornblende. Intrusive relations of the gabbros and surrounding granites are observed on a wall rock of the Tsukuba-tunnel (Sasada *et al.*, 1987) and at an outcrop shown in Fig. 2. On the other hand, the Dorokushin Gabbro Body is composed mostly of hornblende gabbro occurring at northeastern part of the Tsukuba Mountain. It is elongated in shape with length of 4 km. Although it apparently seems to



Fig. 2. Outcrop showing gabbro-granite boundary at the western hillside of the Mt. Tsukuba (locality 4 and 7 in Fig. 1.). Scale: 1 meter.

intrude into the Mt. Kaba Granite Body (Fig. 1).

Geological relations of the granites and gabbros were summarized by Miyazaki et al. (1996). Figure 3 shows the geological relations of granites and gabbros, referring various age data based mainly on K-Ar age of biotite, Rb-Sr isochron of minerals and whole rocks (e.g. Kawano and Ueda, 1966; Miyazaki et al., 1996). Age of gabbro from the Tsukuba Mountain was obtained by K-Ar method of hornblende. They concluded that gabbros were formed at 75 Ma before the formation of granites of 58-63 Ma. Recently, Yokoyama et al. (2016) obtained uraninite and thorite ages from 400 granitoids in the Japanese Islands. Eleven samples of the granites in the Tsukuba area were analyzed. Most of the granites were ages from 65 to 70 Ma. In two samples from the Tsukuba and Mt. Kaba granite bodies, they obtained age around 80 Ma. They conclude that the combination with 65-70 Ma and 80 Ma may be correlated with granitic rocks from the southwestern part of the Asahi Mountains where is considered to be northern extension of the Ryoke Belt.



Fig. 3. Intrusive relationships of the Tsukuba plutonic bodies and radiometric ages (after Miyazaki *et al.*, 1996). Solid arrows show the relationships between two units confirmed at the outcrops. Dotted arrows show intrusive relationships estimated from the areal distributions of the rocks. Radiometric ages were from Kawano and Ueda (1966), Shiba *et al.* (1979), Arakawa and Takahashi (1988), Shibata and Uchiumi (1992) and Miyazaki *et al.* (1996)).

Sample No.	Complex and unit*	Rock type	U-Pb age (Ma) MSWD		The number of culcu- lated zircon grain	
1	Ka1	Medium-grained biotite granite	63.9 ± 1.2	2.9	18	
2	Ts3	Porphyritic biotite granite	79.6 ± 1.3	1.5	16	
3	Ts5	Porphyritic biotite muscovite granite	61.1 ± 1.3	1.04	14	
4	Ts5	Porphyritic biotite muscovite granite	62.5 ± 1.9	1.5	6	
5	Gb1	Anorthositic pyroxene gabbro	61.1 ± 1.9	0.18	3	
6	Gb1	Pyroxene gabbro			0	
7	Gb1	Pyroxene gabbro	63.1 ± 0.8	1.16	28	
8	Gb2	Hornblende gabbro	62.1 ± 1.1	1.6	27	

Table 1. Rock- types and U-Pb zircon ages of the analyzed plutonic rocks in the Tsukuba area.

*In: Inada Granite Body, Ka: Mt. Kaba Granite Body, Ts: Tsukuba Granite Body, Gb1: Mt. Tsukuba gabbro Body, Gb2: Dorokushin gabbro Body.

The unit numbers of Ka1, Ts3-5, Gb1-2 are based on Miyazaki et al. (1996).

Analytical methods of U-Pb dating and results

Totally eight samples were collected from the Tsukuba area (Table 1). Both granites and gabbros



are mostly coarse-grained. All samples were apparently fresh and any alteration has not been observed. After careful cleaning of the samples by ultrasonic washing machine, around 200 g and more than 1 kg of granites and gabbros, respectively, were crushed by the selective fragmentation device using high-voltage pulsed electric discharges (selFrag-Lab) installed in the National Museum of Nature and Science to maximize the yield of zircon recovery. In granite samples, euhedral zircon grains for LA-ICP-MS analysis were handpicked from heavy fractions separated by heavy liquid (diiodomethane). Although zircon was not found in one gabbro sample, sample 7, from the Tsukuba Mountain, subhedral or partly broken zircon grains were recovered from two pyroxene gabbros from the Tsukuba body and one hornblende gabbro from the Dorokushin body.

Zircon grains from the samples, the zircon standard FC1 (206 Pb/ 238 U = 0.1859; Paces and Miller, 1993), and the glass standard NIST SRM610 were mounted in an epoxy resin and polished till the surface was flattened with the center of the embedded grains exposed. Both the backscattered electron and cathodoluminescence images were used to select the sites for analysis (Fig. 4). U-Pb dating of these samples was carried out using an LA-ICP-MS that was assembled by NWR213 (Electro Scientific Indus-

^{Fig. 4. Back-scattered image (BEI) of the analyzed zircon grains from the representative plutonic rocks. A: granite from the Mt. Kaba body (sample 1), B: granite from the Tsukuba body (samples 2), C: pyroxene gabbro from the Tsukuba gabbro body (samples 7). Small circles show analytical position in zircon grain. Scale bar: 100 μm.}



Fig. 5. Tera-Wasserburg concordia diagrams and age distribution plots of the analyzed granite samples. Left: Tera-Wasserburg concordia diagrams, right: age distribution plots. A & A': granite from the Mt. Kaba Body, sample 1. B & B': granite from the Tsukuba Body, sample 2. C & C': granite from the Tsukuba Body, sample 3. D & D': granite from the Tsukuba Body, sample 4. Error ellipses represent 68.3% confidence intervals. Quoted ages are weighted means of ²⁰⁷Pb-corrected ²⁰⁶Pb/²³⁸U spot ages (n analyses, errors at 95% confidence intervals). Solid ellipses, analyses used in weighted mean age calculation; dotted ellipses, excluded analyses.

tries) and Agilent 7700x (Agilent Technologies) that is installed at the National Museum of Nature and Science, Japan. The experimental conditions and the procedures followed for the measurements were based on Tsutsumi *et al.* (2012). The spot size of the laser was approximately $25\,\mu$ m. A correction for common Pb was made on the basis of the measured 207 Pb (207 Pb correction) or 208 Pb and Th/U ratio (208 Pb correction) (e.g. Williams, 1998) and the model for common Pb compositions proposed by



Fig. 6. Tera-Wasserburg concordia diagrams and age distribution plots of the analyzed gabbro samples. Left: Tera-Wasserburg concordia diagrams, right: age distribution plots. A & A': anorthositic pyroxene gabbro from the Mt. Tsukuba Body, sample 5. B & B': pyroxene gabbro from the Mt. Tsukuba Body, sample 7. C & C': hornblende gabbro from the Dorokushin Body, sample 8. Error ellipses represent 68.3% confidence intervals. Quoted ages are weighted means of ²⁰⁷Pb-corrected ²⁰⁶Pb/²³⁸U spot ages (n analyses, errors at 95% confidence intervals). Solid ellipses, analyses used in weighted mean age calculation; dotted ellipses, excluded analyses.

Stacey and Kramers (1975). The pooled ages presented in this study were calculated using Isoplot/ Ex software (Ludwig, 2003). The uncertainties in the mean ²³⁸U-²⁰⁶Pb* ages represent 95% confidence intervals (95% conf.). ²⁰⁶Pb* indicates radiometric ²⁰⁶Pb. Most of the analyzed zircon grains in the granite samples exhibit magmatic oscillatory zonings (e.g. Fig. 4) and lack of textural evidence for secondary modification, suggesting that the obtained age for each sample represents the timing of magmatic crystallization of the zircon. On the other hand, subhedral or partly broken zircons from the gabbros have a weak zoned texture as shown in Fig. 4, different from those from the granite samples. We obtained zircon U-Pb mean ages from four granite and three gabbro samples. Tera-Wasserburg Concordia plots of granites and gabbros and weighted mean ²⁰⁷Pb-corrected ²⁰⁶Pb/²³⁸U ages are shown in Fig. 5 and 6. Age and detailed isotope data are summarized in Table 1 and 2. Three granites have a narrow range from 61 Ma to 64 Ma. One granite from the Tsukuba Body has an older age: 80 Ma. The younger and older granites were recognized from the Tsukuba Granite Body. Ages of three gabbro samples from the Mt. Tsukuba and Dorokushin bodies are well concentrated in a range from 61 to 63 Ma, similar to young cluster of the granite samples (Table 1).

Table 2. Detailed data for the U-Pb ages of zircons from Tsukuba plutonic rocks.

						0		1		
			20((2)						²³⁸ U/ ²⁰⁶ Pb*	²³⁸ U/ ²⁰⁶ Pb*
Sample	Labels		$^{200}\text{Pb}_{c}^{(2)}$	U	Th	Th/U	²³⁸ U/ ²⁰⁶ Pb* ⁽²⁾	²⁰⁷ Pb*/ ²⁰⁶ Pb* ⁽²⁾	age (2)	age $^{(3)}$
No.			(%)	(ppm)	(ppm)				(Ma)	(Ma)
		(1)							()	()
1	T12_01.1	$iz^{(1)}$	0.78	353	82	0.24	4.12 ± 0.08	0.1184 ± 0.0033	1400.4 ± 23.2	1357.4 ± 22.8
1	T12_01.2		0.00	1348	406	0.31	101.13 ± 2.37	0.0478 ± 0.0035	63.4 ± 1.5	63.4 ± 1.5
1	T12_02.1		0.00	1855	408	0.23	96.78 ± 1.94	0.0468 ± 0.0027	66.3 ± 1.3	66.3 ± 1.3
1	T12 03.1		0.00	1350	323	0.25	98.39 ± 2.10	0.0468 ± 0.0031	65.2 ± 1.4	65.2 ± 1.4
1	T12_04.1	iz	0.00	193	126	0.67	2.47 ± 0.04	0.1306 ± 0.0027	2192.2 ± 28.1	2192.2 ± 28.1
1	T12_04.2		0.00	1224	344	0.29	$101 01 \pm 2.40$	0.0492 ± 0.0038	635 ± 15	63.4 ± 1.5
1	T12_05_1		0.00	1211	434	0.37	96.47 ± 2.12	0.0420 ± 0.0043	66.5 ± 1.5	66.5 ± 1.5
1	$T12_{061}$		0.00	2627	591	0.23	10658 ± 180	0.0502 ± 0.0013	60.2 + 1.0	60.0 ± 1.0
1	$T12_00.1$ $T12_07.1$	17	1 51	775	261	0.25	84.71 ± 2.23	0.0302 = 0.0027 0.0344 ± 0.0074	75.7 + 2.0	76.8 ± 1.0
1	$T12_07.1$ T12_08_1	IZ	0.00	1020	572	0.55	64.71 - 2.23 106.09 + 2.67	0.0344 ± 0.0074 0.0402 ± 0.0051	75.7 ± 2.0	70.8 ± 1.9
1	T12_00.1	·	0.00	241	120	0.37	100.96 ± 2.07	0.0403 ± 0.0031	00.0 - 1.3	00.0 - 1.3
1	T12_09.1	1Z	0.00	341	138	0.41	82.48 ± 3.25	0.0411 ± 0.0058	77.7 ± 3.0	77.7 ± 3.0
1	112_10.1 T12_11_1		0.00	1532	1//	0.12	100.82 ± 2.47	0.0504 ± 0.0039	03.0 ± 1.3	03.4 ± 1.0
1	112_11.1	1Z	0.15	720	61	0.09	3.24 ± 0.05	0.1258 ± 0.0022	$1/33.5 \pm 25.0$	1699.2 ± 24.8
1	T12_11.2		0.00	1311	323	0.25	96.75 ± 2.19	0.0504 ± 0.0041	66.3 ± 1.5	66.0 ± 1.5
1	T12_12.1		0.15	1713	477	0.29	97.33 ± 1.87	0.0505 ± 0.0048	65.9 ± 1.3	65.6 ± 1.3
1	T12_13.1	iz	0.00	518	27	0.05	4.34 ± 0.07	0.1148 ± 0.0023	1336.8 ± 18.6	1295.8 ± 18.4
1	T12_14.1		0.00	1295	305	0.24	96.33 ± 2.16	0.0455 ± 0.0040	66.6 ± 1.5	66.6 ± 1.5
1	T12_15.1		0.00	1808	393	0.22	98.35 ± 1.96	0.0460 ± 0.0031	65.2 ± 1.3	65.2 ± 1.3
1	T12 16.1		0.00	1098	380	0.36	106.74 ± 2.63	0.0548 ± 0.0046	60.1 ± 1.5	59.5 ± 1.5
1	T12 ^{17.1}		0.00	3803	1645	0.44	97.03 ± 1.45	0.0597 ± 0.0026	66.1 ± 1.0	65.1 ± 1.0
1	$T12^{-17.2}$		0.44	1498	544	0.37	103.41 ± 2.54	0.0438 ± 0.0055	62.0 ± 1.5	62.3 ± 1.5
1	T12 18 1	iz	0.00	293	172	0.60	3.02 ± 0.06	0.1487 ± 0.0030	1841.9 ± 30.7	17754 ± 302
1	T12_18.2	iz	0.00	1686	310	0.19	91.25 ± 1.86	0.0482 ± 0.0027	70.3 ± 1.4	70.2 ± 1.4
1	T12_10.2	12	0.00	175	106	0.62	96.30 ± 5.13	0.0390 ± 0.0120	66.6 ± 3.5	66.6 ± 3.5
1	T12_19.1		0.11	1309	321	0.02	$102\ 27+2\ 22$	0.0370 = 0.0120 0.0444 ± 0.0049	62.7 ± 1.4	62.8 ± 1.3
1	$T12_{19.2}$ T12_20.1	17	0.11	1309	207	0.23	102.27 = 2.22 21.22 ± 0.62	0.0444 = 0.0049 0.0572 ± 0.0076	02.7 = 1.4 202.2 + 4.1	02.8 = 1.3 201.5 + 2.0
1	$112_{20.1}$	IZ	0.41	1025	420	0.85	31.22 ± 0.03	$0.03/2 \pm 0.00/0$ 0.0491 ± 0.0020	203.2 - 4.1 64.0 + 1.4	201.3 ± 3.9
1	112_20.2		0.15	1655	430	0.24	98.81 ± 2.08	0.0481 ± 0.0039	04.9 - 1.4	04.0 - 1.4
2	T10 01.1	iz	1.43	225	148	0.68	59.48 ± 2.41	0.0453 ± 0.0141	107.5 ± 4.3	107.9 ± 4.2
2	T10_01_2		0.37	512	233	0.47	81.64 ± 2.45	0.0397 ± 0.0094	785 ± 23	78.8 ± 2.2
2	T10_02_1	17	0.00	232	111	0.49	28.22 ± 0.71	0.0397 = 0.0094 0.0496 ± 0.0043	70.5 = 2.5 224.5 ± 5.6	70.0 - 2.2 2245 + 56
2	$T10_02.1$ $T10_02.2$	12	0.00	053	155	0.49	20.22 = 0.71 70 24 + 1 70	0.0414 ± 0.0043	224.5 = 5.0 01.1 + 2.3	018 + 23
2	$T10_{02.2}$	12	0.74	404	008	1.99	70.24 = 1.79 72.21 + 2.22	0.0414 = 0.0002 0.0474 ± 0.0050	91.1 = 2.3 87.5 + 2.6	91.0 = 2.3 97.5 + 2.6
2	T10_03.1	IZ	0.00	494	908	1.00	75.21 ± 2.25	$0.04/4 \pm 0.0030$	0/.3 - 2.0	07.3 ± 2.0
2	T10_04.1		0.23	/03	227	0.31	80.32 ± 2.02	0.0407 ± 0.0004	79.8 ± 2.0	79.9 ± 2.0
2	110_05.1		0.00	646	230	0.36	75.35 ± 2.13	0.0494 ± 0.0038	85.0 ± 2.4	84.8 ± 2.4
2	110_06.1	1Z	0.00	2331	445	0.20	3.46 ± 0.05	0.1157 ± 0.0014	1636.9 ± 19.3	1611.7 ± 19.2
2	T10_07.1	1Z	0.00	1145	456	0.41	71.26 ± 1.65	0.0486 ± 0.0029	89.8 ± 2.1	89.7 ± 2.1
2	T10_08.1		0.00	1733	595	0.35	82.17 ± 1.75	0.0471 ± 0.0029	78.0 ± 1.6	78.0 ± 1.6
2	T10_09.1	iz	0.35	228	82	0.37	3.19 ± 0.05	0.1130 ± 0.0036	1757.9 ± 25.0	1748.8 ± 25.2
2	T10_09.2	iz	0.59	899	433	0.49	67.51 ± 1.60	0.0524 ± 0.0067	94.8 ± 2.2	94.3 ± 2.2
2	T10_10.1		0.00	569	229	0.41	79.93 ± 2.31	0.0480 ± 0.0048	80.1 ± 2.3	80.1 ± 2.3
2	T10_11.1		0.29	571	735	1.32	76.60 ± 2.36	0.0464 ± 0.0129	83.6 ± 2.6	83.7 ± 2.3
2	T10_12.1	iz	0.45	733	54	0.08	3.93 ± 0.05	0.1167 ± 0.0020	1461.6 ± 15.7	1423.8 ± 15.6
2	T10 12.2		0.00	609	214	0.36	75.47 ± 1.99	0.0525 ± 0.0045	84.9 ± 2.2	84.3 ± 2.3
2	T10 13.1	iz	0.00	511	535	1.07	67.96 ± 2.21	0.0516 ± 0.0052	94.2 ± 3.0	93.7 ± 3.1
2	T10_14.1		0.78	1179	493	0.43	79.69 ± 1.70	0.0371 ± 0.0060	80.4 ± 1.7	81.0 ± 1.7
2	T10 15 1		0.00	509	453	0.91	81.41 ± 2.85	0.0527 ± 0.0047	78.7 ± 2.7	78.2 ± 2.8
2	T10 15 2		0.00	839	425	0.52	82.20 ± 2.14	0.0501 ± 0.0039	77.9 ± 2.0	77.7 ± 2.0
2	T10_16_1	17	0.00	437	264	0.62	26.96 ± 0.56	0.0536 ± 0.0034	234.8 ± 4.8	234.0 ± 4.8
2	T10 17 1	12	0.48	812	1086	1 37	20.90 ± 0.30 81 99 ± 2 38	0.0451 ± 0.0110	78.1 ± 23	78.4 ± 2.0
2	$T_{10}^{17.1}$		0.40	1254	510	0.20	83.46 ± 1.45	0.0443 + 0.0019	76.9 + 1.2	76.9 + 1.2
2	$110_{1/.2}$ T10_10_1		0.00	500	204	0.39	03.40 - 1.43 77.80 + 2.00	0.0440 ± 0.0028	70.0 - 1.3 82.2 + 2.2	70.0 - 1.3 82.2 + 2.2
2	T10_10.1	:	0.00	309	200	0.41	11.07 ± 2.09	0.0437 ± 0.0030	02.2 - 2.2	02.2 - 2.2
2	110_19.1 T10_10.2	1Z	0.00	401	254	0.56	30.27 ± 0.72	0.0494 ± 0.0033	209.5 ± 4.9	209.5 ± 4.9
2	110_19.2		0.00	536	236	0.45	81.83 ± 2.48	0.0504 ± 0.0054	78.3 ± 2.4	78.0 ± 2.4
2	T10_20.1		0.00	144	176	1.25	82.54 ± 4.60	0.0584 ± 0.0182	77.6 ± 4.3	76.6 ± 4.6
2	T10_21.1		0.00	869	436	0.52	81.17 ± 1.98	0.0483 ± 0.0036	78.9 ± 1.9	78.9 ± 1.9
2	K00 01 1	17	2 1 2	169	110	0.67	3.80 ± 0.07	0.1061 ± 0.0050	1475 6 + 24 4	1456 8 + 22 0
2	K07_01.1	1Z	2.12	100	110	0.07	$3.07 \div 0.07$	$0.1001 \div 0.0000$	177.0 - 24.4	$1730.0 \div 23.9$
3	K09_01.2	1Z	0.09	567	6	0.01	29.24 ± 0.59	$0.04/0 \pm 0.0025$	216.8 ± 4.3	$21/.0 \pm 4.3$
3	K09_02.1		0.00	119	107	0.92	109.20 ± 6.06	0.0419 ± 0.0239	58.8 ± 3.2	58.8 ± 3.2
3	K09_02.2		0.00	276	257	0.96	104.60 ± 4.38	0.0548 ± 0.0089	61.3 ± 2.6	60.8 ± 2.6
3	K09_03.1		0.00	202	143	0.73	109.87 ± 5.32	0.0348 ± 0.0133	58.4 ± 2.8	58.4 ± 2.8
3	K09_03.2		0.00	221	168	0.78	105.66 ± 4.69	0.0438 ± 0.0095	60.7 ± 2.7	60.7 ± 2.7
3	K09_04.1		0.00	204	170	0.85	94.39 ± 4.28	0.0607 ± 0.0131	67.9 ± 3.1	66.8 ± 3.2
3	K09_05.1		1.55	1266	81	0.07	108.83 ± 2.57	0.0485 ± 0.0055	59.0 ± 1.4	58.9 ± 1.4
3	K09_06.1		0.76	171	135	0.81	103.27 ± 5.62	0.0274 ± 0.0235	62.1 ± 3.4	62.6 ± 3.0
3	K09_06.2		0.00	232	168	0 74	101.28 ± 4.55	0.0400 ± 0.0097	63.3 ± 2.8	63.3 ± 2.8
3	K09_07_1		0.00	345	397	1 1 8	106.28 ± 3.90	0.0262 ± 0.0057	60.4 ± 2.0	60.4 ± 2.3
ĩ	K09_08_1		0.00	150	182	1 24	102.67 ± 5.93	0.0398 ± 0.0164	625 ± 36	62.5 ± 3.6
5	1.07_00.1		0.00	1.20	102	1.47	102.01 - 5.75	5.0570 - 0.0104	52.5 - 5.0	52.5 - 5.0

Table 2. Continued.

Sample No.	Labels		²⁰⁶ Pb _c ⁽²⁾ (%)	U (ppm)	Th (ppm)	Th/U	²³⁸ U/ ²⁰⁶ Pb* ⁽²⁾	²⁰⁷ Pb*/ ²⁰⁶ Pb* ⁽²⁾	²³⁸ U/ ²⁰⁶ Pb* age ⁽²⁾ (Ma)	²³⁸ U/ ²⁰⁶ Pb* age ⁽³⁾ (Ma)
3	K09 09 1	iz	0.00	190	168	0.90	238 ± 0.04	0.1657 ± 0.0031	2264.6 ± 35.0	2215.7 ± 35.0
3	K09 09.2	iz	5.57	558	139	0.26	9.00 ± 0.23	0.1100 ± 0.0042	679.4 ± 16.3	642.7 ± 15.6
3	K09_09.3		0.51	648	71	0.11	97.47 ± 2.86	0.0513 ± 0.0058	65.8 ± 1.9	65.5 ± 1.9
3	K09_10.1		0.50	809	327	0.41	106.21 ± 3.33	0.0379 ± 0.0077	60.4 ± 1.9	60.7 ± 1.8
3	K09_10.2		0.00	291	160	0.56	105.01 ± 4.06	0.0615 ± 0.0084	61.1 ± 2.4	60.0 ± 2.4
3	K09_11.1 K09_12_1	17	0.00	62 72	54 62	0.90	104.83 ± 8.34 2 45 ± 0.06	0.0450 ± 0.0295 0.1533 ± 0.0065	61.2 ± 4.8 2208 1 + 45 3	61.2 ± 4.8 2177.7 ± 45.2
3	K09_12.1 K09_12.2	iz	0.00	1178	28	0.00	11.74 ± 0.18	0.1033 ± 0.0003 0.1047 ± 0.0021	527.0 ± 7.7	497.8 ± 7.4
3	K09 13.1	iz	0.14	552	194	0.36	85.66 ± 2.56	0.0566 ± 0.0081	74.8 ± 2.2	74.0 ± 2.2
3	K09_13.2	iz	0.00	2050	722	0.36	87.29 ± 1.65	0.0473 ± 0.0027	73.4 ± 1.4	73.4 ± 1.4
4	T15_01.1	iz	0.00	219	86	0.40	40.88 ± 1.03	0.0501 ± 0.0034	155.8 ± 3.9	155.6 ± 3.9
4	T15_02.1		0.65	811	479	0.61	104.92 ± 2.12	0.0434 ± 0.0051	61.2 ± 1.2	61.4 ± 1.2
4	T15_02.2	:	0.02	961 427	500	0.53	105.16 ± 1.97	0.0483 ± 0.0040	61.0 ± 1.1	60.9 ± 1.1
4	T15_03.1 T15_04_1	1Z	0.29	427	257	0.38	93.84 ± 2.27 99.56 + 2.48	0.0461 ± 0.0060 0.0427 ± 0.0064	68.3 ± 1.0 64.4 ± 1.6	68.4 ± 1.0 64.6 ± 1.5
4	T15_04.1	iz	1.55	832	211	0.49	78.65 ± 1.52	0.0427 = 0.0004 0.0418 ± 0.0036	81.4 ± 1.6	82.0 ± 1.6
4	T15 05.2		0.09	158	68	0.45	98.71 ± 3.50	0.0537 ± 0.0091	65.0 ± 2.3	64.5 ± 2.3
4	T15_06.1		0.29	204	201	1.01	98.15 ± 3.33	0.0446 ± 0.0137	65.3 ± 2.2	65.5 ± 2.0
4	T15_07.1		0.86	767	251	0.34	103.15 ± 2.26	0.0422 ± 0.0041	62.2 ± 1.4	62.6 ± 1.4
4	$T15_{08.1}^{+(4)}$	1Z	1.02	419	119	0.29	6.36 ± 0.11	0.1069 ± 0.0021 0.0502 ± 0.0074	940.9 ± 15.5	902.8 ± 14.9
4	T15_09.1 T15_09.2	iz	2.30	211	42 217	0.42	77.10 ± 2.99 85 75 ± 2 88	0.0393 ± 0.0074 0.0251 ± 0.0132	83.0 ± 3.2 74.7 ± 2.5	81.8 ± 3.2 765 ± 23
4	T15_09.2 T15_10.1	iz	0.27	249	128	0.52	73.45 ± 2.02	0.0231 ± 0.00132 0.0377 ± 0.0068	87.2 ± 2.4	70.3 = 2.3 87.4 ± 2.3
4	T15 11.1	iz	0.74	416	75	0.18	63.76 ± 1.94	0.0419 ± 0.0050	100.3 ± 3.0	101.1 ± 3.0
4	T15_11.2	iz	0.00	331	210	0.65	91.54 ± 2.43	0.0446 ± 0.0035	70.0 ± 1.8	70.0 ± 1.8
4	T15_11.3*	iz	0.00	347	199	0.59	90.30 ± 2.48	0.0597 ± 0.0053	71.0 ± 1.9	69.9 ± 2.0
4	T15_12.1	iz	1.16	609	62	0.11	54.54 ± 1.27	0.0431 ± 0.0033	117.1 ± 2.7	117.9 ± 2.7
4	115_12.2	1Z	0.56	631	246	0.40	90.62 ± 1.87	0.03//±0.0051	/0./± 1.5	/1.1 ± 1.4
5	T08_01.1	iz	0.28	655	224	0.35	91.86 ± 2.61	0.0523 ± 0.0074	69.8 ± 2.0	69.4 ± 2.0
5	T08_01.2	iz	0.14	633	227	0.37	85.00 ± 2.37	0.0375 ± 0.0072	75.4 ± 2.1	75.5 ± 2.0
5	108_02.1 T08_02_1		0.00	810	49 184	0.26	105.49 ± 5.20 106.00 ± 2.61	$0.046 / \pm 0.0109$ 0.0450 ± 0.0026	60.8 ± 3.0 60.5 ± 1.5	60.8 ± 3.0 60.5 ± 1.5
5	T08_03.1 T08_04.1		0.00	934	380	0.23	100.09 ± 2.01 104.03 ± 2.57	0.0430 ± 0.0030 0.0467 ± 0.0075	61.7 ± 1.5	61.7 ± 1.5
7	T13 01.1		2.14	130	145	1.14	107.97 ± 3.77	0.0222 ± 0.0155	59.4 ± 2.1	60.7 ± 1.8
7	T13 01.2		0.00	102	71	0.72	99.26 ± 2.79	0.0411 ± 0.0059	64.6 ± 1.8	64.6 ± 1.8
7	T13_02.1		0.00	362	588	1.66	100.09 ± 2.02	0.0474 ± 0.0031	64.1 ± 1.3	64.1 ± 1.3
7	T13_02.2		0.29	423	696	1.69	104.23 ± 2.53	0.0533 ± 0.0109	61.6 ± 1.5	61.1 ± 1.3
7	T13_02.3		1.75	521	564	1.11	102.26 ± 2.36	0.0403 ± 0.0086	62.7 ± 1.4	63.3 ± 1.3
7	T13_02.4		0.00	514	560	1.12	106.13 ± 2.08 104.02 ± 2.28	0.0493 ± 0.0030	60.5 ± 1.2	60.3 ± 1.2
7	T13_02.5 T13_03_1		0.57	183	91 72	0.51	104.92 ± 3.38 106.52 ± 5.56	0.0382 ± 0.0090 0.0379 ± 0.0130	61.2 ± 2.0 60.2 ± 3.1	61.5 ± 1.9 60.2 ± 3.1
7	T13_03.2		1.56	97	125	1.32	100.32 = 5.30 104.15 ± 5.13	0.0370 ± 0.0232	61.6 ± 3.0	62.6 ± 2.6
7	T13 03.3		0.00	136	149	1.12	102.17 ± 3.89	0.0418 ± 0.0071	62.8 ± 2.4	62.8 ± 2.4
7	T13_04.1		0.00	113	119	1.09	101.56 ± 3.77	0.0524 ± 0.0075	63.2 ± 2.3	62.8 ± 2.4
7	T13_04.2		0.87	129	152	1.21	97.12 ± 4.00	0.0444 ± 0.0143	66.0 ± 2.7	66.3 ± 2.6
7	T13_04.3		2.47	94	272	0.78	106.23 ± 4.88 102.16 ± 2.27	0.0563 ± 0.0208	60.4 ± 2.8	59.7 ± 2.6
7	T13_05.2		0.33	430	361	0.86	102.10 ± 2.27 97.93 ± 2.17	0.0390 ± 0.0080 0.0449 ± 0.0030	02.8 ± 1.4 65.5 ± 1.4	05.1 ± 1.5 65.5 ± 1.4
7	T13_06.1		0.00	47	58	1.27	96.09 ± 4.93	0.0498 ± 0.0141	66.7 ± 3.4	66.5 ± 3.6
7	T13 06.2		0.00	44	44	1.04	98.01 ± 5.18	0.0322 ± 0.0126	65.4 ± 3.4	65.4 ± 3.4
7	T13_07.1		0.00	53	47	0.90	100.71 ± 5.24	0.0575 ± 0.0111	63.7 ± 3.3	62.9 ± 3.4
7	T13_07.2		0.00	112	80	0.73	99.05 ± 3.29	0.0593 ± 0.0070	64.8 ± 2.1	63.8 ± 2.2
7	T13_07.3		1.50	90	113	1.28	103.44 ± 5.22	0.0423 ± 0.0230	62.0 ± 3.1	62.4 ± 2.8
7	T13_08.1 T13_08.2		0.00	91 162	92 174	1.03	103.70 ± 3.23 94.63 ± 2.03	0.0308 ± 0.0192 0.0408 ± 0.0055	60.7 ± 3.0 67.8 ± 2.1	01.9 ± 2.8 67.8 ± 2.1
7	T13_09.1		2.59	145	282	1.99	104.49 ± 4.62	0.0262 ± 0.0267	61.4 ± 2.7	63.0 ± 2.0
7	T13_10.1		0.00	131	145	1.14	94.46 ± 3.35	0.0527 ± 0.0067	67.9 ± 2.4	67.4 ± 2.4
7	T13_10.2		1.34	81	71	0.90	105.59 ± 4.76	0.0335 ± 0.0180	60.8 ± 2.7	61.6 ± 2.5
7	T13_11.1		0.00	415	542	1.34	93.51 ± 1.89	0.0520 ± 0.0033	68.6 ± 1.4	68.2 ± 1.4
/ 7	113_12.1 T13_12.2		1.10	570 187	467	1.30	94.06 ± 2.39 100 54 + 2.10	0.0406 ± 0.0104 0.0472 ± 0.0050	68.2 ± 1.7	63.8 ± 1.6
	T1J_12.2		1.50	40/		0.02	100.34 - 2.19	$0.0472 \div 0.0039$	$03.0 \div 1.4$	$03.0 \div 1.4$
8	T14_01.1		0.25	73	47	0.65	104.33 ± 5.61 109.39 ± 4.20	0.0210 ± 0.0192 0.0224 ± 0.0145	61.5 ± 3.3 58 7 + 2 2	61.6 ± 3.1
0 8	$T14_01.2$ T14_02_1		0.00	97	09 54	0.55	107.39 ± 4.20 100.71 ± 5.53	0.0334 ± 0.0143 0.0450 ± 0.0109	50.7 ± 2.2 63.7 ± 3.5	63.7 ± 3.5
8	T14 02.2		1.47	122	57	0.48	110.30 ± 5.19	0.0259 ± 0.0140	58.2 ± 2.7	59.0 ± 2.6
8	T14_03.1		4.45	339	171	0.52	103.05 ± 3.33	0.0480 ± 0.0125	62.3 ± 2.0	62.2 ± 2.0

Sample No.	Labels	²⁰⁶ Pb _c ⁽²⁾ (%)	U (ppm)	Th (ppm)	Th/U	²³⁸ U/ ²⁰⁶ Pb* ⁽²⁾	²⁰⁷ Pb*/ ²⁰⁶ Pb* ⁽²⁾	²³⁸ U/ ²⁰⁶ Pb* age ⁽²⁾ (Ma)	²³⁸ U/ ²⁰⁶ Pb* age ⁽³⁾ (Ma)
8	T14 04.1	0.20	457	483	1.08	104.80 ± 3.00	0.0505 ± 0.0107	61.2 ± 1.7	61.0 ± 1.6
8	T14 05.1	0.73	263	134	0.52	110.18 ± 3.87	0.0413 ± 0.0103	58.2 ± 2.0	58.7 ± 2.0
8	T14 05.2*	0.00	159	82	0.53	100.88 ± 3.65	0.0708 ± 0.0092	63.6 ± 2.3	61.7 ± 2.3
8	T14 06.1	1.74	330	304	0.94	107.74 ± 3.32	0.0283 ± 0.0124	59.6 ± 1.8	60.6 ± 1.7
8	T14_06.2*	0.00	187	90	0.49	98.37 ± 4.01	0.0693 ± 0.0077	65.2 ± 2.6	63.4 ± 2.6
8	T14_07.1	0.75	285	223	0.80	107.08 ± 3.81	0.0391 ± 0.0119	59.9 ± 2.1	60.4 ± 2.0
8	T14 08.1	0.00	440	452	1.06	108.47 ± 2.92	0.0444 ± 0.0039	59.2 ± 1.6	59.2 ± 1.6
8	T14_08.2	1.16	127	62	0.51	99.22 ± 5.41	0.0232 ± 0.0164	64.6 ± 3.5	65.4 ± 3.4
8	T14 09.1	0.33	300	249	0.85	103.45 ± 3.60	0.0353 ± 0.0127	62.0 ± 2.1	62.2 ± 2.0
8	T14_10.1	1.21	119	95	0.82	102.50 ± 6.15	0.0283 ± 0.0231	62.6 ± 3.7	63.4 ± 3.5
8	T14_11.1	0.09	433	306	0.72	100.13 ± 2.61	0.0465 ± 0.0065	64.1 ± 1.7	64.1 ± 1.6
8	T14 12.1	0.00	349	300	0.88	105.12 ± 3.16	0.0399 ± 0.0044	61.0 ± 1.8	61.0 ± 1.8
8	T14_12.2	2.47	175	90	0.52	109.14 ± 4.62	0.0365 ± 0.0129	58.8 ± 2.5	59.6 ± 2.5
8	T14_13.1	0.98	771	958	1.27	105.81 ± 2.95	0.0416 ± 0.0107	60.6 ± 1.7	61.1 ± 1.6
8	T14_14.1	0.00	209	131	0.64	94.88 ± 3.14	0.0433 ± 0.0064	67.6 ± 2.2	67.6 ± 2.2
8	T14_15.1	0.00	384	356	0.95	103.26 ± 3.17	0.0495 ± 0.0045	62.1 ± 1.9	61.9 ± 1.9
8	T14_16.1	1.62	408	349	0.88	104.26 ± 3.49	0.0279 ± 0.0116	61.5 ± 2.1	62.5 ± 1.9
8	T14_16.2	0.63	152	85	0.57	104.16 ± 4.88	0.0432 ± 0.0135	61.6 ± 2.9	61.9 ± 2.8
8	T14_17.1	0.00	136	78	0.59	102.07 ± 4.06	0.0554 ± 0.0079	62.9 ± 2.5	62.2 ± 2.5
8	T14_18.1	0.00	321	199	0.63	96.23 ± 2.43	0.0435 ± 0.0039	66.6 ± 1.7	66.6 ± 1.7
8	T14_19.1	1.53	148	78	0.55	99.32 ± 3.42	0.0253 ± 0.0129	64.6 ± 2.2	65.6 ± 2.1
8	T14_20.1	0.00	150	83	0.57	94.42 ± 3.54	0.0404 ± 0.0073	67.9 ± 2.5	67.9 ± 2.5

Table 2. Continued.

Errors are 1-sigma; Pb_c and Pb^* indicate the common and radiogenic portions, respectively. (1) iz: inheritad zircon

(2) Common Pb corrected by assuming ²⁰⁶Pb/²³⁸U-²⁰⁸Pb/²³²Th age-concordance

(3) Common Pb corrected by assuming ²⁰⁶Pb/²³⁸U-²⁰⁷Pb/²³⁵U age-concordance

(4) *: discordant

Discussion

Since 1965, the K-Ar method has been widely used for age analyses of granitic rocks in Japan (e.g. Kawano and Ueda, 1964, 1966) and radiometric age data of granitic rocks in the Japanese Islands were summarized by Nozawa (1975). Recently many authors have used SHRIMP and LA-ICP-MS methods for the age analyses in the Japanese Cretaceous granites (e.g. Ishihara and Orihashi, 2015; Kon *et al.* 2015; Tani *et al.* 2016). In the Ryoke metamorphic belt, however, the reliable age method was applied only to the Cretaceous granites at the Kinki District by Watanabe *et al.* (2000).

Basement rock of the Tsukuba area belongs to the Ryoke Metamorphic Belt. The area is an eastern end of the belt. In this paper, reliable age analyses were done for the plutonic rocks in the Tsukuba area. The age results are summarized in Fig. 7. It shows that igneous event of granite occurred at least twice in this area, different from the K-Ar age data. The younger cluster is roughly consistent with K-Ar ages of 58-63 Ma. The older granites with age around 80 Ma were found in the Tsukuba Granite Body. Our result is rather similar to the uraninite and thorite age data obtained by Yokoyama *et al.*

(2016). They concluded that the similar igneous events, i.e. 60-65 Ma and around 80 Ma, occurred at the western part of the Asahi Mountains where is considered to be northern extension of the Ryoke Belt. On the other hand, granites with 80 Ma occur sporadically in the Ryoke Belt from the Chubu to Kinki provinces. The older granites with age around 80 Ma may be related to the major event of the Ryoke Belt.

The most unexpected result was ages of gabbros in the Tsukuba and Dorokushin bodies. Ages of three gabbro samples are well concentrated from 61 to 63 Ma, corresponding to younger cluster of the granite samples with age from 61 to 64 Ma. As shown in Fig. 2, granite is apparently intruded into pyroxene gabbro of the Mt. Tsukuba body. The granite at the contact zone is 62.5 ± 1.9 Ma, faintly younger than nearby gabbro with 63.1 ± 0.8 Ma. Unfortunately, it is hard to conclude simply that gabbro was formed before the intrusion of the granite, considering of the standard deviation of the age. As probable explanations, zircons collected from gabbro were contaminated from the surrounding granite or zircons were affected by the thermal event of the later stage granitic activity. The zircons in gabbros, however, have different BEI texture,



Fig. 7. The radiometric ages of the Tsukuba plutonic rocks. White squares are from Kawano and Ueda (1966), Shiba *et al.*, (1979), Arakawa and Takahashi (1988), Miyazaki *et al.* (1966), Shibata and Uchiumi (1992). White stars are from Yokoyama *et al.* (2016). Black circles are from this study.

weakly zoned structure (Fig. 4c), from oscillatory zoned zircons in granites. Any zircon analyzed in this paper shows no secondary texture affected by later stage thermal event. Furthermore, age data from three gabbros have a narrow in range from 61 to 63 Ma. Hence, it is reasonable that the gabbros and younger granites were formed almost at the same period.

As a future work, it is important to analyze plutonic rocks in the Ryoke Belt by SHRIMP and LA-ICP-MS to correlate the relationships among the rocks. Even if only a little, the present study will contribute a solution to the problem of the plutonic activity in Japan.

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