

Zircon U–Pb ages of the granitic rocks in the Chikuihi area, central Kyushu, southwest Japan

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Abstract Zircons U–Pb ages were obtained from 5 granitoid samples in central Kyushu, southwest Japan. Two samples from Kikuchi Granite indicate ages of 106.6 ± 0.9 and 105.7 ± 1.2 Ma. Two samples from Tamana Granodiorite indicate ages of 105.7 ± 0.7 and 105.5 ± 0.9 Ma. The sample from Tsutsugatake Granite indicates an age of 106.0 ± 1.0 Ma. Errors are with 95% confidence interval. Including a previously reported age, zircon U–Pb ages in the study area concentrate around 106 Ma. The zircon ages are thought to be the plutonic age of the granitoids. Although 121–117 Ma of whole rock Rb–Sr ages was previously thought to be the plutonic ages of granitoids in this area, this assumption needs to be rechecked.

Key words: plutonic age, granitoid, Early Cretaceous, central Kyushu

Introduction

Kyushu is tectonically subdivided to three parts, northern, central and southern Kyushu, by the Matsuyama–Imari and Usuki–Yatsushiro tectonic lines (Fig. 1). Basement rocks in northern and central Kyushu consist of Permo–Jurassic accretionary complexes with Cretaceous granitoids. Basements in central Kyushu expose only the southernmost and northern parts called the Higo belt and Chikuihi area, respectively, due to subsidence in the Beppu–Shimabara Graben and covering of volcanics from Neogene to Quaternary volcanos.

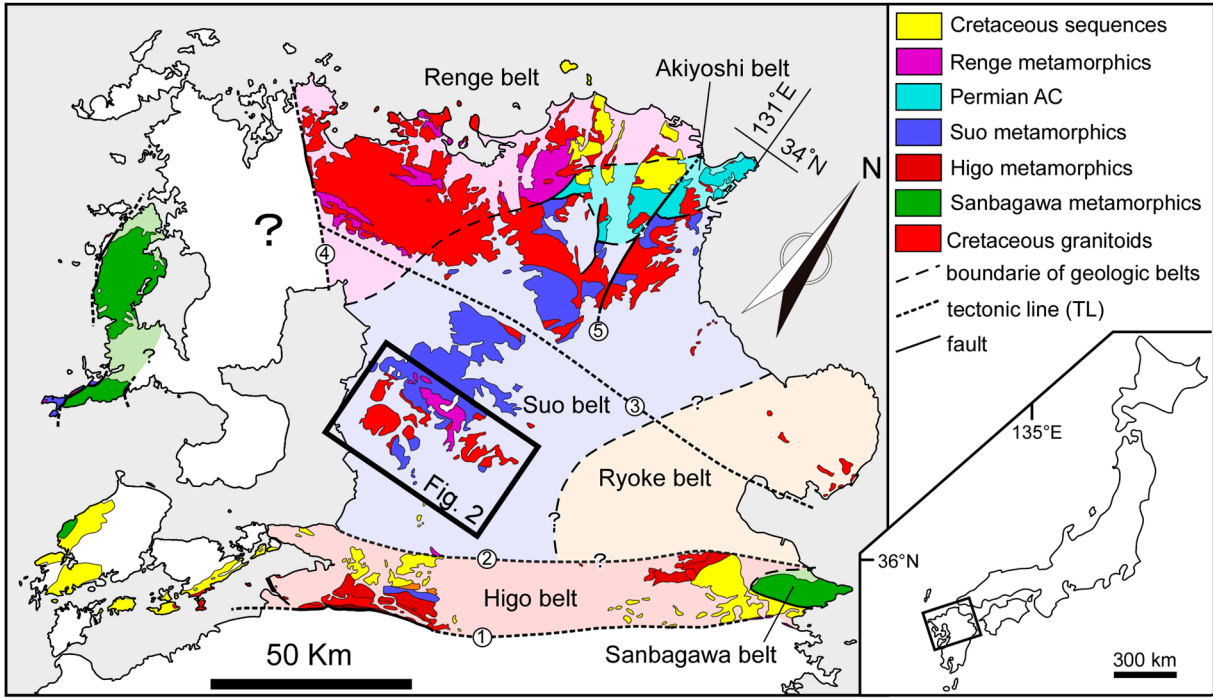
Because whole rock (WR) Rb–Sr age was believed to close from crystallization to cooling in igneous rocks (e.g. Moor bath, 1975; Roddick and Compston, 1977), it was thought to be suitable for plutonic age of granitic rocks. Hence, the plutonic age of Cretaceous granitoid in northern and central Kyushu had been discussed using WR Rb–Sr ages in spite of their large uncertainties. These days, zircon U–Pb method which has much higher precision, accuracy and closure temperature than WR Rb–Sr method has become common and it is thought to more accurately indicate plutonic age of granitoids.

In northern Kyushu, the plutonic ages of granitoids had been thought to be 118–88 Ma based on WR Rb–Sr age (118 ± 11 Ma to 88 ± 18 Ma; 2σ ; Osanai *et al.*, 1993; Owada *et al.*, 1999). The ages of granitoids have since been updated as 107–98 Ma by zircon U–Pb age (107.4 ± 0.8 Ma to 97.9 ± 1.4 Ma, 95% conf.; Adachi *et al.*, 2012; Miyazaki *et al.*, 2018; Yuhara *et al.*, 2019).

In the Chikuihi area, only one zircon U–Pb age has been reported (Miyazaki *et al.*, 2018). In this paper, five new zircon U–Pb ages of granitoids are obtained. The main purpose of this work is improvement of the age data of granitic rocks in central Kyushu. The result will contribute to clarify the role of Kyushu in the tectonic framework of Japanese Islands.

Geological setting

Cretaceous granitic rocks in the Chikuihi area (Fig. 2) are divided into “older” and “younger” types (Owada *et al.*, 1999; Kamei *et al.*, 2002). The older type granitoids mostly comprise granodiorite to tonalite containing hornblende, whereas the younger type granitoids comprise peraluminous granodiorite to granite containing white mica and sometimes including garnet.



①Usuki-Yatsushiro TL ②Oita-Kumamoto TL ③Matsuyama-Imari TL ④Ushizu-Kariya TL ⑤Kokura-Tagawa TL

Fig. 1. Distribution map of the pre-Paleogene rocks in northern Kyushu.

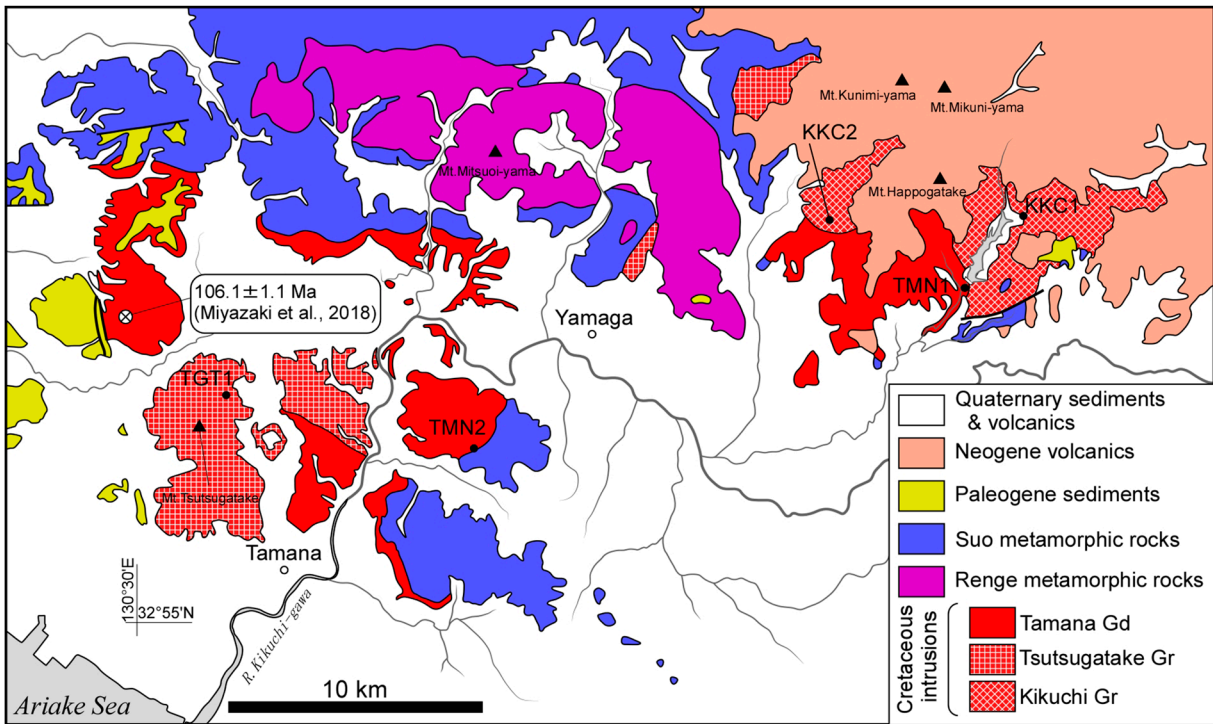


Fig. 2. Geological map of the study area showing the sampling localities, modified after Hoshizumi *et al.* (2004). Gd: granodiorite, Gr. Granite.

Granitoids in the Chikui area are divided into three bodies (e.g. Kamei *et al.*, 2009; Kamei and Osanai, 2010); Tamana granodiorite which is the older type, and Kikuchi and Tsutsugatake granites which are the younger type. The Tamana granodio-

rite is coarse to medium grained hornblende-biotite granodiorite. Biotite and hornblende K–Ar ages in the granodiorite are 99.7 ± 2.5 Ma and 106.0 ± 2.6 Ma, respectively (Tomita *et al.*, 2008). Recently, the zircon U–Pb age of 106.1 ± 1.1 Ma (95% conf.;

Miyazaki *et al.*, 2018) was reported. The Kikuchi granite comprises fine to medium grained biotite granite to granodiorite sometimes containing white mica and/or garnet. The WR Rb–Sr isochron age is 121.3 ± 8.4 Ma (2σ ; Osanai *et al.*, 1993), whereas the K–Ar age of muscovite is 95.3 ± 4.8 Ma (Sasada, 1987). Tsutsugatake granite is fine to medium grained two mica-garnet granite. The WR Rb–Sr isochron age is 116.8 ± 12.7 Ma (2σ ; Osanai *et al.*, 1993), whereas biotite and muscovite K–Ar ages are 95.0 ± 4.8 Ma and 95.1 ± 4.8 Ma, respectively (Sasada, 1987). According to classical interpretation based on closure temperatures of minerals used for dating, granitoids in this area were thought to be formed at 121–117 Ma, and had been cooled until ~ 95 Ma (Kamei and Osanai, 2010).

Analytical methods

At first, the rock samples were scrubbed, and washed in an ultrasonic bath for ten minutes to avoid surface zircon contaminants. Fragmentation of the rock sample was conducted by a high voltage pulse power selective fragmentation equipment, SELFRAG Lab (Selfrag AG). The zircon grains were handpicked from heavy fractions that were separated through heavy-liquid techniques. Zircon grains from the samples, the zircon standards TEMORA2 (416.78 Ma; Black *et al.*, 2004) and OD-3 (33 Ma; Iwano *et al.*, 2013), 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. Before the mounting and polishing, secondary electron (SE) images of each grain were taken for morphological note. After the mounting and polishing, backscattered electron (BE) and cathodoluminescence (CL) images of zircon grains were taken. Scanning electron microscope-cathodoluminescence equipment, JSM-6610 (JEOL) and a CL detector (SANYU electron), were used for SE, BE and CL images. The images were used to select the sites for analysis. U–Pb dating of the samples was carried out using laser ablation inductively coupled plasma mass spectrometry using an NWR213 (Elemental Scientific Lasers) and Agilent 7700x (Agilent Technologies). All processes for sample preparation and analysis were conducted at the National Museum of

Nature and Science, Tsukuba, Japan. The experimental conditions and the analytical procedures used for measurements followed Tsutsumi *et al.* (2012), with the additional devices of a buffered type stabilizer (Tunheng and Hirata, 2004) and TwoVol2 sample cell also applied. The spot size of the laser was 25 μm . A correction for common Pb was made on the basis of the measured $^{207}\text{Pb}/^{206}\text{Pb}$ ratio (^{207}Pb correction), $^{208}\text{Pb}/^{206}\text{Pb}$ and Th/U ratios (^{208}Pb correction) (e.g. Williams, 1998) and the model for common Pb compositions proposed by Stacey and Kramers (1975). In this paper, we adopt ^{207}Pb correction for age discussion because it is effective for calculating Phanerozoic ^{238}U – $^{206}\text{Pb}^*$ age compared to ^{208}Pb correction (e.g. Williams, 1998). ^{208}Pb corrected $^{238}\text{U}/^{206}\text{Pb}^*$ and $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ ratios are used for concordia plots. Pb^* indicates radiometric Pb. The pooled ages presented in this study were calculated using Isoplot/Ex software (Ludwig, 2012). The data of secondary standard OD-3 zircon obtained during analysis yielded weighted mean ages of 32.3 ± 1.1 Ma ($n = 8$; MSWD = 0.35; when KKC1, KKC2 and TMN1 were analyzed) and 31.7 ± 1.4 Ma ($n = 5$; MSWD = 1.13; when TMN2 and TGT were analyzed). MSWD is acronym of mean square weighted deviation, which is calculated from square root of χ^2 value.

Sample descriptions and age results of zircon

Table 1 lists zircon data in terms of the fraction of common ^{206}Pb , U, and Th concentrations, Th/U, $^{238}\text{U}/^{206}\text{Pb}^*$ and $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ ratios, and radiometric $^{238}\text{U}/^{206}\text{Pb}^*$ ages of the samples. All errors are 1σ level. All zircons in the samples show rhythmic oscillatory and/or sector zoning in CL images (Fig. 3), which is commonly observed in igneous zircons (e.g. Corfu *et al.*, 2003). Errors of weighted mean zircon U–Pb ages are at 95% confidence interval (95% conf.). Concordia and age distribution diagrams are shown in Fig. 4 and 5, respectively. The obtained weighted mean ages and sample localities are summarized in Table 2.

All rock samples are stored in the National Museum of Nature and Science. The registration number of each sample can be found from the rock specimen number in the collection database of the

Table 1. Continued

Labels	$^{206}\text{Pb}_c^{(1)}$ (%)	U (ppm)	Th (ppm)	Th/U	$^{238}\text{U}/^{206}\text{Pb}^*^{(1)}$	$^{207}\text{Pb}^*/^{206}\text{Pb}^*^{(1)}$	$^{238}\text{U}/^{206}\text{Pb}^* \text{ age}^{(1)}$ (Ma)	$^{238}\text{U}/^{206}\text{Pb}^* \text{ age}^{(2)}$ (Ma)	Remarks
TGT1_21.1	0.02	446	103	0.24	57.76 ± 1.29	0.0508 ± 0.0051	110.7 ± 2.4	110.3 ± 2.5	
TGT1_22.1	0.00	380	184	0.50	61.68 ± 1.41	0.0436 ± 0.0033	103.7 ± 2.3	103.7 ± 2.3	
TGT1_23.1	0.40	119	105	0.91	56.76 ± 2.22	0.0497 ± 0.0151	112.6 ± 4.4	112.4 ± 4.2	
TGT1_24.1	0.01	360	119	0.34	60.14 ± 1.34	0.0539 ± 0.0056	106.3 ± 2.3	105.6 ± 2.3	
TGT1_24.2	0.00	2104	678	0.33	60.97 ± 0.86	0.0465 ± 0.0013	104.9 ± 1.5	104.9 ± 1.5	
TGT1_25.1	0.42	420	129	0.32	60.14 ± 1.29	0.0532 ± 0.0051	106.3 ± 2.3	105.6 ± 2.3	
TGT1_26.1	0.00	110	74	0.69	55.51 ± 2.01	0.0294 ± 0.0053	115.1 ± 4.1	115.1 ± 4.1	D, N
TGT1_27.1	0.00	112	103	0.95	63.95 ± 2.27	0.0573 ± 0.0074	100.0 ± 3.5	98.9 ± 3.6	

Errors are 1-sigma; Pb_c and Pb^* indicate the common and radiogenic portions, respectively.

Remarks; D: discordant, N: not used for weighted mean age calculation

(1) Common Pb corrected by assuming $^{206}\text{Pb}/^{238}\text{U} - ^{208}\text{Pb}/^{232}\text{Th}$ age-concordance

(2) Common Pb corrected by assuming $^{206}\text{Pb}/^{238}\text{U} - ^{207}\text{Pb}/^{235}\text{U}$ age-concordance

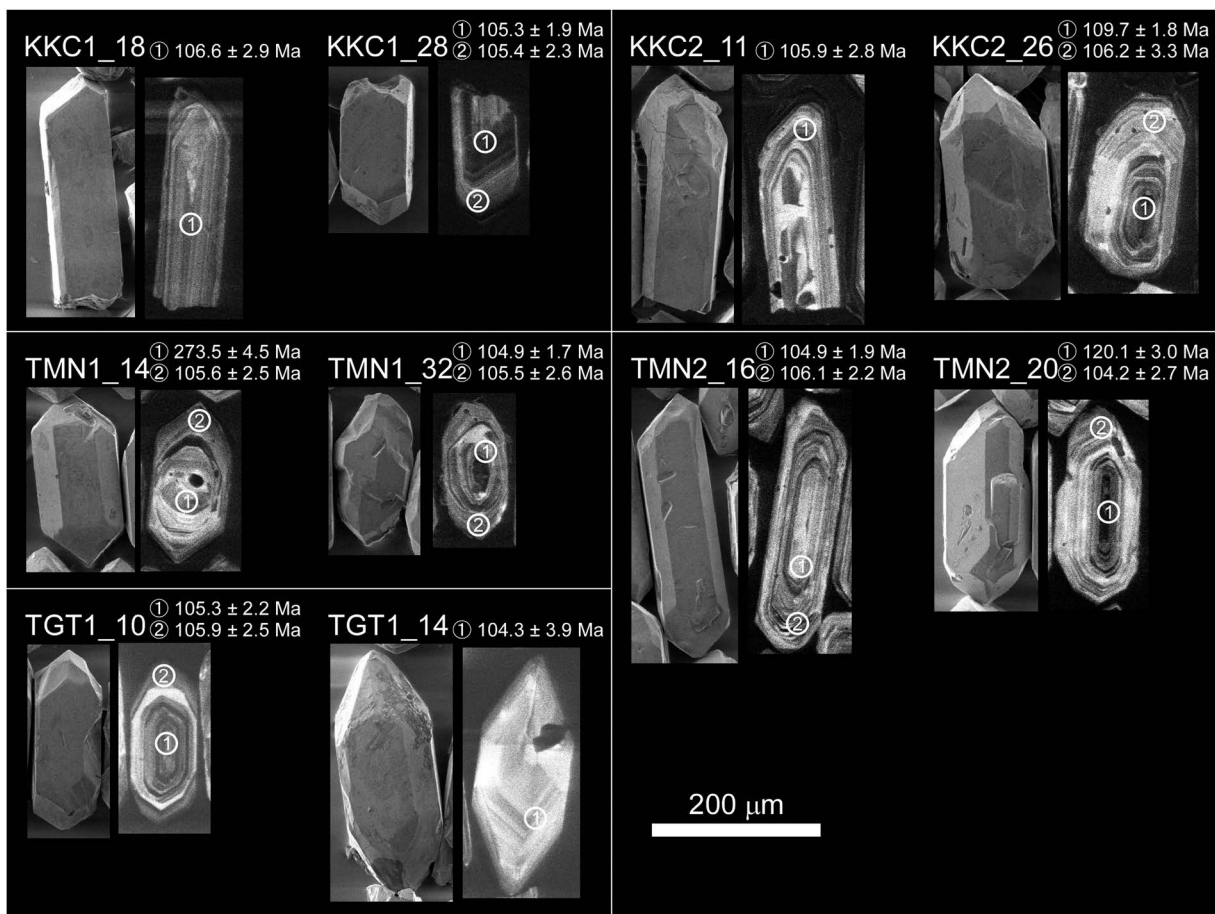


Fig. 3. Morphological secondary electron (SE) images before cement in resin and cathodoluminescence (CL) images of analyzing section of typical zircon grains from the samples. Circles on the images point to analyzed spots by LA-ICP-MS which diameter are 25 μm approx.

National Museum of Nature and Science (http://db.kahaku.go.jp/webmuseum_en/).

KKC1: Kikuchi granite

The sample was collected from north of Lake Hanjaku in the northeastern part of Kikuchi City (lat: N $33^{\circ}03'32.0''$, long: E $130^{\circ}52'29.0''$). This is a medium to coarse grained granite. The major minerals of this rock are quartz, plagioclase, alkali feld-

spar, biotite, and muscovite. Plagioclase occurs as euhedral to subhedral crystal and exhibits indistinct albite twin and oscillatory zoning. Biotite is partly altered into chlorite. Undulatory extinction is observed in quartz. Zircon and opaque mineral are common accessory minerals. The registration number is 137666.

Most zircon grains are 150 to 300 μm in length, partly rounded with elongation ratio of 1.8 to 3.3.

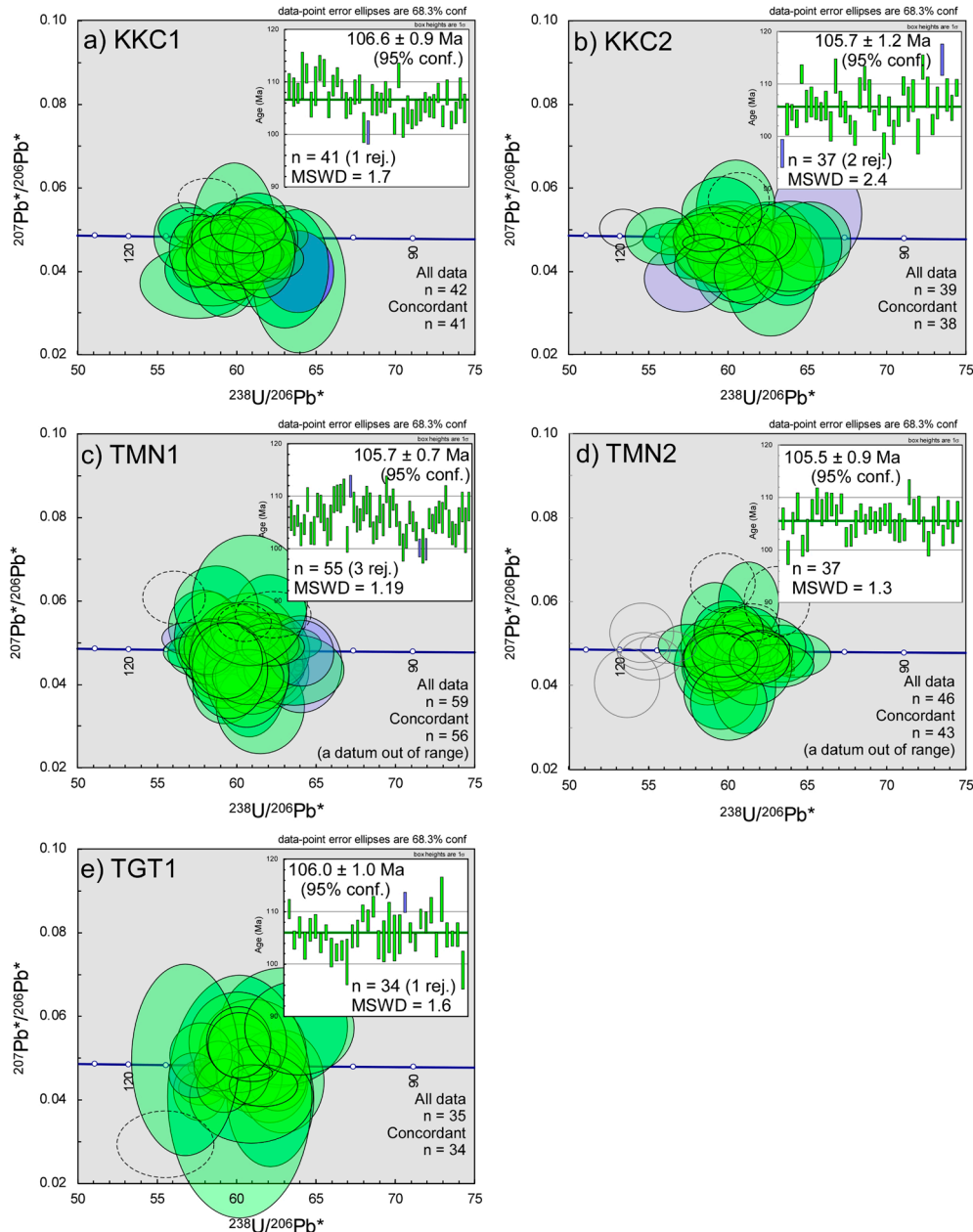


Fig. 4. Tera-Wasserberg U–Pb concordia diagrams and age distribution plot of zircons from the samples.

Their CL images are relatively bright with distinct oscillatory and/or sector zoning. 42 spots from 33 grains were analyzed and the weighted mean age of 41 concordant data indicate 106.6 ± 0.9 Ma (1 datum rejected; MSWD = 1.7).

KKC2: Kikuchi granite

The sample was collected from east of Happogatake in the eastern part of Yamaga City (lat: N33°03'32.0", long: E 130°47'29.3"). This is a medium grained granite. Microscopic observation is similar to KKC1. The registration number is 137668.

Most zircon grains are 180 to 350 μm in length

with prismatic and elongation ratio from 1.8 to 4.0. CL image of the zircons are relatively bright with distinct oscillatory zoning. Although some darker CL cores were observed, there are no age differences beyond the error range. 39 spots from 33 grains were analyzed and 38 data are concordant. After an older datum was excluded, the weighted mean age of 37 data indicates 105.7 ± 1.2 Ma (2 data are rejected; MSWD = 2.4).

TMN1: Tamana granodiorite

The sample was collected from the left side bank of the Hazama River, downstream of the Ryumon Dam (lat: N 33°02'05.4", long: E 130°50'51.0").

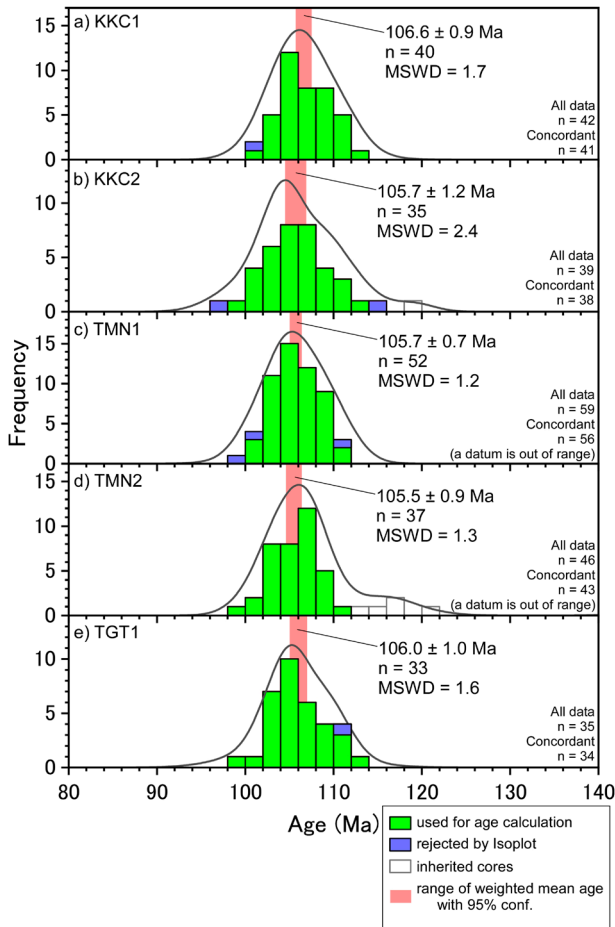


Fig. 5. Probability distribution diagrams and histogram of zircon ages in the samples.

This is a coarse grained granodiorite. The major minerals of this rock are plagioclase, quartz, biotite and amphibole. Plagioclase occurs as euhedral to subhedral crystal and exhibits indistinct albite twin and oscillatory zoning. Undulatory extinction is observed in quartz. Zircon and opaque mineral are common accessory minerals. The registration number is 137667.

Most zircon grains are 150 to 240 μm length with prismatic and elongation ratio from 1.5 to 3.5. CL images are relatively bright with distinct oscillatory zoning. 59 spots from 38 grains were analyzed and 56 data are concordant. After 1 older datum was excluded, the weighted mean age of all concordant data indicates 105.7 ± 0.7 Ma (3 data rejected; MSWD = 1.2).

TMN2: Tamana granodiorite

The sample was collected from the western end part of Yamaga City (lat: N $32^{\circ}58'40.9''$, long: E $130^{\circ}38'30.3''$). This is a medium to coarse grained

granodiorite. Microscopic observation is similar to TMN1. The registration number is 137670.

Most zircon grains are 150 to 300 μm length with prismatic and elongation ratio from 2.0 to 3.9. CL images are relatively bright with distinct oscillatory zoning. 48 spots from 27 grains were analyzed and 43 data are concordant. After 5 older data were excluded, the weighted mean age of all 38 data indicates 105.5 ± 0.9 Ma (MSWD = 1.3).

TGT1: Tsutsugatake granite

The sample was collected from the northeastern foot of Mt. Tsutsugatake (lat: N $32^{\circ}59'41.8''$, long: E $130^{\circ}32'08.8''$). This is a medium to coarse grained granite. The major minerals of this rock are quartz, plagioclase, alkali feldspar, biotite and muscovite. Plagioclase occurs as euhedral to subhedral crystal and exhibits indistinct albite twin and oscillatory zoning. Undulatory extinction is observed in quartz. Zircon and opaque mineral are common accessory minerals. The registration number is 137671.

Most zircon grains are 150 to 280 μm length with prismatic and elongation ratio from 1.8 to 4.2. CL images are relatively bright with distinct oscillatory zoning. 35 spots from 27 grains were analyzed and 34 data are concordant. The weighted mean age of all concordant data indicates 106.0 ± 1.0 Ma (1 datum rejected; MSWD = 1.6).

Discussion

Because of the high closure temperature of the decay system and the robust nature of zircon, U–Pb age is commonly interpreted to indicate the plutonic age of granitoids. In Chikuhi area, the zircon U–Pb ages of samples in this study and an age previously reported (Miyazaki *et al.*, 2018) are well-concentrated in a narrow range from 106.6 to 105.5 Ma. There is no relationship between zircon U–Pb ages and the classification of “older” or “younger” granitoids. It is thought that the plutonism in the Chikuhi area was concentrated in ca. 106 Ma. The WR Rb–Sr ages previously reported by Osanai *et al.* (1993) of the study area are 121.3 ± 8.4 and 116.8 ± 12.7 Ma. Present zircon U–Pb ages are in a range of their high 2σ data errors.

Zircon U–Pb ages of granitoids in the Higo belt, southern part of the central Kyushu, concentrated in

Table 2. Summaries of localities and weighted mean ages of each sample.

sample name	registration No. ¹⁾	locality	rock body	n of data			Age	MSWD
				All	Conc.	Calc.		
KKC1	137666	N33°03'32.0"E130°52'29.0"	Kikuchi granite	42	41	40	106.6 ± 0.9	1.7
KKC2	137668	N33°03'32.0"E130°47'29.3"	Kikuchi granite	39	38	35	105.7 ± 1.2	2.4
TMN1	137667	N33°02'05.4"E130°50'51.0"	Tamana granodiorite	59	56	52	105.7 ± 0.7	1.2
TMN2	137670	N32°58'40.9"E130°38'30.3"	Tamana granodiorite	46	43	37	105.5 ± 0.9	1.3
TGT1	137671	N32°59'41.8"E130°32'08.8"	Tsutsugatake granite	35	34	33	106.0 ± 1.0	1.6

Age errors are 95% conf.; Conc.: concordant, Calc.: used for age calculation.

1) The number of rock specimen in the collection database of the National Museum of Nature and Science (http://db.kahaku.go.jp/webmuseum_en/).

113–108 Ma (Nagata and Otoh, 2021 and references therein). In contrast, zircon U–Pb ages of northern Kyushu, northern than Matsuyama-Imari Tectonic line, mainly range from 105–98 Ma (Adachi *et al.*, 2012; Yuhara *et al.*, 2019; Miyazaki *et al.*, 2018). Granitoids in the Chikuhai area indicate zircon U–Pb ages of ca. 106 Ma which is intermediate between Higo belt and northern Kyushu areas. The trend in zircon age data is more noticeable than in WR data, i.e. Cretaceous granitoid in Kyushu roughly becomes younger northward.

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*English translation from the original written in Japanese.