Ti 濃度定量による結晶化温度と U-Pb 年代から導く花崗岩中のジルコンの形成プロセス：EPMA によるジルコン中のチタン濃度の定量

Zircon growth in a granitic pluton with specific mechanisms, crystallization temperatures and U-Pb ages: Quantitative determination of Ti concentration in zircon by EPMA

湯口貴史[1*]・加藤丈典[2]
Takashi Yuguchi[1*] and Takenori Kato[2]

[1]山形大学理学部地球環境学科・[2]名古屋大学宇宙地球環境研究所
[1]Department of Earth and Environmental Sciences, Yamagata University, 1-4-12 Kojirakawa, Yamagata 990-8560, Japan.
[2]Institute for Space-Earth Environmental Research, Nagoya University, Chikusa, Nagoya 464-8601, Japan

**Correspondence author. E-mail: takashi_yuguchi@sci.kj.yamagata-u.ac.jp*

Abstract

Zircon crystals of the Toki granitic pluton provide evidence of serial growth events with specific mechanisms, crystallization temperatures, and U-Pb ages. With this evidence we can deduce details of the sequential formation process from intrusion through emplacement to crystallization/solidification. Serial growth events have been identified by: 1) a study of the internal structure of zircon using cathodoluminescence observation, 2) crystallization temperatures using Ti-in-zircon thermometry of the internal structure, and 3) U-Pb age dating of the internal structure. This paper expounds a procedure for quantitatively determining the titanium concentration in zircon and the results of crystallization temperature measurements based on a Ti-in-zircon thermometer. The magmatic zircons collected from Toki granite display two different internal structures: LLC (low luminescence core) and OZ (oscillatory zonation). The LLC crystallized due to interfacial reaction-controlled growth with the cooling granitic magma under temperature conditions ranging from about 910^{o}C to about 760^{o}C. The OZ developed due to diffusion-controlled growth within a cooling magma chamber under temperature conditions from about 850^{o}C to about 690^{o}C.

Keywords: *Zircon growth mechanism; Ti-in-zircon thermometry; EPMA; Toki granitic pluton.*

Introduction

The zircon U-Pb dating has been used ubiquitously for igneous granitic rocks (e.g. Sano et al., 2002; Schmitt et al., 2002). However, there is the question of equating the thermal conditions in the formation history (from intrusion through emplacement to crystallization / solidification) of a granitic pluton with the zircon U-Pb age. The closure temperature of the zircon U-Pb age is in excess of 900^{o}C based on diffusion characteristics of Pb determined in natural and synthetic zircons (Cherniak and Watson, 2001). Crystallization temperatures of zircons are depending on the lithology, e.g. ranging from about 900^{o}C for mafic rocks to about 600^{o}C for felsic rocks (Fu et al., 2008). In granitic rock, the proposed closure temperature of zircon U-Pb (> 900^{o}C) is often higher than the crystallization temperature of the zircon, indicating that the U-Pb system in the granitic zircon crystal closed under the temperature condition and timing in which the zircon crystallized from the granitic magma. Therefore, the zircon U-Pb age means the timing of magmatic crystallization of the zircon (Yuguchi et al., 2016).

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名古屋大学宇宙地球環境研究所共同利用・共同研究プログラム
「一般共同研究」280409（代表者：湯口貴史）

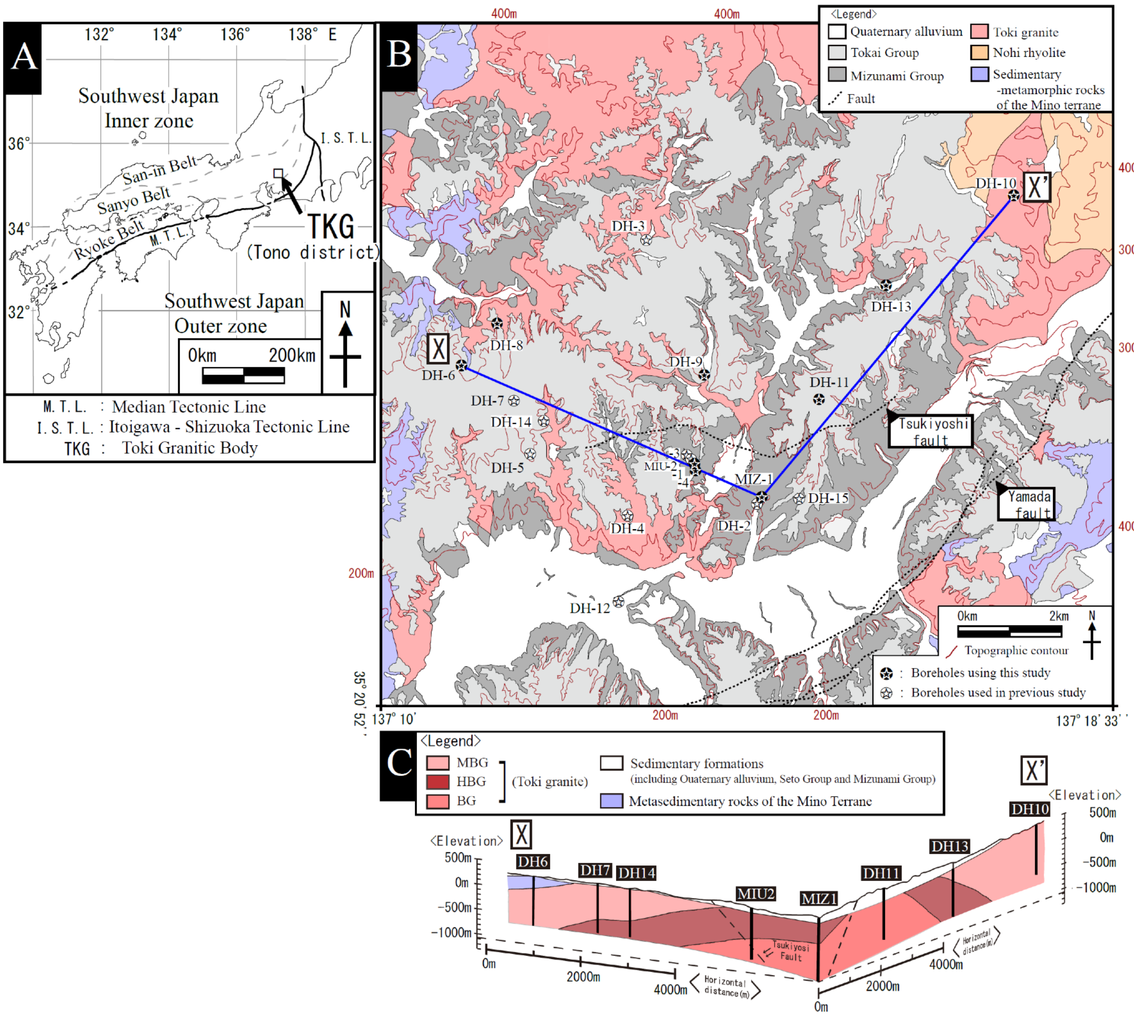

Fig. 1 The Toki granitic pluton. (A) Location map showing the Toki granite in the Tono district, central Japan (square symbol). (B) Geological map of the Tono district surrounding the Toki granite, after Itoigawa (1980). The topographic contours are based on the Geographical Survey Institute, 1:25,000 topographic maps entitled “Ontake”, “Takenami”, “Toki” and “Mizunami”. (C) Rock facies cross-sections for the Toki granite along the line from X to X’ in the geological map (Fig. 1B) (Yuguchi et al., 2010).

The identification of the zircon U-Pb age as the crystallization age gives a clue to clarify what event in formation process of a granite corresponds to the analyzed zircon U-Pb age. In particular, relationships among the zircon internal structure, the crystallization temperature and the U-Pb age will provide the classification of zircon U-Pb ages into some events in the formation process of a granitic pluton by the combination of the following analyses;

1) Clarification of the internal structure based on cathodoluminescence (CL) observations, leading to the recognition of different events in zircon crystallization

2) Characterization of crystallization temperature derived from the Ti-in-zircon thermometry (Watson et al., 2006; Ferry and Watson, 2007) in each part of the internal structure, giving the thermal conditions for each event

3) Characterization of U-Pb age in each part of the internal structure, revealing the formation ages of each event

Classification of zircon U-Pb ages into some events will clarify the ‘sequential’ processes from intrusion of granitic magma through emplacement of magma chamber to crystallization / solidification of the Toki granitic pluton, central Japan (Yuguchi et al., 2016). This paper expounds procedures and results of quantitative determination of titanium concentration of zircon within the published paper of Yuguchi et al. (2016) “Zircon growth in a granitic pluton with specific mechanisms, crystallization temperatures and U-Pb ages: Implication to the ‘spatiotemporal’ formation process of the Toki granite, central Japan. *Journal of Mineralogical and Petrological Sciences*, **111**, 9−34”.

The Toki granite

The Toki granite in the Tono district, central Japan, is one of the Late Cretaceous plutonic intrusives in the Sanyo Belt, the Inner Zone of Southwest Japan (Fig. 1A; Ishihara and Chappell, 2007). The Toki granite is a stock, about 14 × 12 km in areal extent (Yuguchi et al., 2010), intruding into Jurassic sedimentary rocks of the Kamiaso unit in the Mino Terrane (Sano et al., 1992) as well as into the late Cretaceous Nohi rhyolite (Sonehara and Harayama, 2007) (Fig. 1B). The rock body has three rock facies, which grades from muscovite-biotite granite (MBG) at the margin, through hornblende-biotite granite (HBG) to biotite granite (BG) in the interior (Fig. 1C).

Analytical procedures

Rock samples (N = 10, drill core) used for this study were collected from eight boreholes in the Toki granite (Fig. 1B). Zircon crystals were separated from rock samples by a combination of grain size separation, magnetic separation using a Franz Isodynamic Separator and density separation using a conventional heavy liquid.

Cathodoluminescence (CL) imaging

The CL images were collected using a JEOL JSM7001F field emission scanning electron microscope equipped with an Gatan mini-CL detector housed at Kumamoto University, operating at an accelerating voltage of 15 kV and a beam current of 1.0 nA. The CL pattern gives the internal structure of a zircon crystal.

Titanium concentration in zircon

Titanium concentrations in zircons can be quantitatively determined by electron probe microanalysis (EPMA) to reveal the crystallization temperatures with respect to the internal structure. The analysis points were determined on the basis of core – rim relationships deduced from the CL pattern (3–15 points in each zircon and a total of 203 points for 31 zircons; Fig. 2).

Analyses of Ti and Ca in zircon crystals were carried out using a JEOL JCXA-733 electron microprobe fitted with four wavelength-dispersive crystal spectrometers (WDS), housed at the Institute for Space-Earth Environmental Research, Nagoya University. Calcium is also determined to assess the validity of the in-situ Ti concentration because the measured concentration value having excessive Ca implies the unexpected analysis for (fluid) inclusion in the zircon cystal (cf. Suzuki and Kato, 2008).

Each spectrometer equips JEOL 733-PET-J analyzing crystals (30 × 12 mm in crystal size) and Xe sealed proportional counters. Ti Kα X-rays were simultaneously collected through four PET crystals and four intensities were averaged to obtain the concentrations. Instrumental operating conditions for spot analysis were 25 kV accelerating voltage, 500 nA probe current, 5-8 μm beam diameter and 1200 seconds counting time, respectively. Careful configuration and validation of spectrometer characteristics showed that no background hole problem (Self et al., 1990; Donovan et al., 2011; Kato and Suzuki, 2014) was observed on the Ti Kα X-ray line. Off-peak background offsets were carefully determined to avoid the background holes near the Ti Kα

X-ray line. Matrix corrections were performed using the Bence and Albee (1968) method with α-factors by Kato (2005), and which enable us to perform quantitative analysis with the same accuracy as the 'PAP' model (Pouchou and Pichoir, 1991). Synthesized TiO_2 and $CaSiO_3$ were used as standard materials for determination of infinitesimal Ti and Ca, respectively (cf. Donovan et al., 2011). The detection limit of titanium at the 2σ confidence level is less than 3 ppm. The relative error in the titanium determination is about 3% at the 10 ppm concentration level and is much better for higher concentrations.

Results

Internal structure derived from the CL pattern

The CL observation allows us for recognizing the differences in the internal structure of a zircon crystal, which clarifies the different stages in zircon growth process and thus in plutonic formation process. The internal structure of magmatic zircon crystal is classified into two categories. Category 1 includes the low-luminescence cores (LLC) with the oscillatory zonal (OZ) surroundings (Fig. 2), which is observed most frequently in all samples. The LLC shows homogeneity without CL zonation and also has the angular and linear shape boundaries in contact with the OZ surroundings similar to euhedral zircon (Fig. 2A and B). The OZ surroundings exhibit growth banding with/without sector zonation. Category 2 is defined as a zircon crystal consisting of oscillatory zonation without LLC capable of identifying core – rim relationship (Fig. 2C, and D).

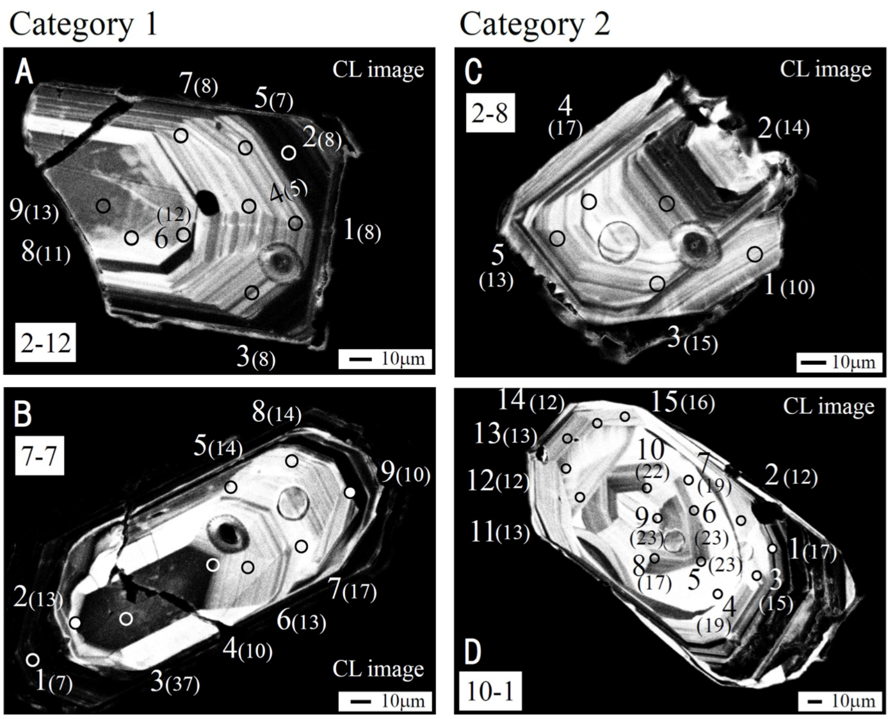

Fig. 2 Cathodoluminescence images of representative zircon crystals, showing the Ti analysis points in the internal structure (A: grain No. 2-12, B: grain No. 7-7, C: grain No. 2-8 and D: grain No. 10-1). Two kinds of internal structure are observed; low-luminescence core (LLC) with no zonation and oscillatory zonation (OZ). Circle symbols in the crystals denote analysis points for Ti concentration and the number corresponds to point number in Table 1. The number in parenthesis represents the Ti concentration (ppm).

Table 1 Titanium and calcium concentrations for zircon crystals from the Toki granite, and the crystallization temperature deduced from the Ti-in-zircon calibration of Ferry and Watson (2007).

BSE / CL images	Grain No.*[1]	Point No.*[1]	Loc*[2]	Ti ppm	Ti SD (1s) %	Ca ppm	Ca SD (1s) %	Temperature Ferry and Watson (2007) $a_{SiO2}=1$ $a_{TiO2}=1$
Fig. 2A	2-12	1	OZ	8	3.7	2	6.2	725 ± 37 ℃
	2-12	2	OZ	8	3.7	n.d.		725 ± 37 ℃
	2-12	3	OZ	8	3.8	n.d.		725 ± 37 ℃
	2-12	4	OZ	5	4.5	3	5.0	685 ± 35 ℃
	2-12	5	OZ	7	4.0	15	2.3	713 ± 36 ℃
	2-12	6	OZ	12	3.0	n.d.		763 ± 38 ℃
	2-12	7	OZ	8	3.6	22	1.9	725 ± 37 ℃
	2-12	8	LLC	11	3.3	n.d.		755 ± 38 ℃
	2-12	9	LLC	13	2.9	16	2.2	771 ± 39 ℃
Fig. 2B	7-7	1	OZ	7	3.8	n.d.		713 ± 36 ℃
	7-7	2	OZ	13	2.9	27	1.8	771 ± 39 ℃
	7-7	3	LLC	37	1.7	112	0.9	886 ± 44 ℃
	7-7	4	OZ	10	3.3	n.d.		746 ± 38 ℃
	7-7	5	OZ	14	2.8	n.d.		779 ± 39 ℃
	7-7	6	OZ	13	2.8	n.d.		771 ± 38 ℃
	7-7	7	OZ	17	2.5	n.d.		798 ± 40 ℃
	7-7	8	OZ	14	2.7	n.d.		779 ± 39 ℃
	7-7	9	OZ	10	3.2	n.d.		746 ± 37 ℃
Fig. 2C	2-8	1	OZ	10	3.3	n.d.		746 ± 38 ℃
Fig. 2C	2-8	2	OZ	14	2.8	n.d.		779 ± 39 ℃
	2-8	3	OZ	15	2.7	1	8.5	785 ± 39 ℃
	2-8	4	OZ	17	2.6	n.d.		798 ± 40 ℃
	2-8	5	OZ	13	2.9	n.d.		771 ± 39 ℃
Fig. 2D	10-1	1	OZ	17	2.5	n.d.		798 ± 40 ℃
	10-1	2	OZ	12	2.9	n.d.		763 ± 38 ℃
	10-1	3	OZ	15	2.6	n.d.		785 ± 39 ℃
	10-1	4	OZ	19	2.3	n.d.		810 ± 40 ℃
	10-1	5	OZ	23	2.1	n.d.		831 ± 41 ℃
	10-1	6	OZ	23	2.1	n.d.		831 ± 41 ℃
	10-1	7	OZ	19	2.4	n.d.		810 ± 40 ℃
	10-1	8	OZ	17	2.5	n.d.		798 ± 40 ℃
	10-1	9	OZ	23	2.1	n.d.		831 ± 41 ℃
	10-1	10	OZ	22	2.2	n.d.		826 ± 41 ℃
	10-1	11	OZ	13	2.8	n.d.		771 ± 38 ℃
	10-1	12	OZ	12	3.0	n.d.		763 ± 38 ℃
	10-1	13	OZ	13	2.9	n.d.		771 ± 39 ℃
	10-1	14	OZ	12	3.0	n.d.		763 ± 38 ℃
	10-1	15	OZ	16	2.6	n.d.		792 ± 39 ℃

*[1] Grain and point numbers correspond to the analysis points in Fig. 3. *[2] Location classifying into LLC (low-luminescence core) and OZ (oscillatory zoning).

Titanium concentrations

Titanium (and calcium) concentrations at the representative analysis points are listed in Table 1. The analysis numbers (grain and point No.) correspond to that of Fig. 2. All average concentrations are described at one sigma (1σ) level. Zircons typically include relatively higher titanium concentrations in their cores than their rims (Fig. 2). Titanium concentrations determined for the LLC (in the observed Category 1) range from 11 to 49 ppm. The OZ (the OZ surroundings in Category 1 and the OZ zircon in Category 2) has Ti concentrations ranging of 5–27 ppm.

Discussion

Clarifying the different events in zircon crystallization

Division of the internal structure on the basis of the CL pattern clarifies the different stages in zircon crystallization from the granitic magma. The Category 1 (Fig. 2A and B) indicates that the crystallization of LLC occurs prior to that of OZ surroundings, representing that the crystallization temperature of LLC is higher than that of OZ surroundings. The Category 2 (Fig. 2C and D) shows that the formation of OZ zircon can occur without the preceding LLC formation. The LLC and OZ formed in different events. That is, in the formation process from granitic magma to pluton, the U-Pb age and crystallization temperature derived from the LLC have quite different meanings from that obtained from the OZ area.

The LLC shows chemical homogeneity through the CL observation, and crystallized at higher temperature condition relative to the OZ. Thus, the LLC may have grown due to crystallization from a homogeneous magma under equilibrium conditions between crystal and melt. The OZ formation arose as a source of the nonlinearity in the crystal-growth kinetics in a magma chamber (L'Heureux and Fowler, 1994; Shore and Fowler, 1996). In particular, the OZ formation has been identified as a phenomenon that occurred within a magma chamber.

Characterization of the crystallization temperature in the internal structure.

Crystallization temperatures of zircon crystals are estimated by the Ti-in-zircon thermometry. Titanium concentration of zircon is highly sensitive to temperature along with quadrivalent Ti substituting for Si (Watson et al., 2006; Ferry and Watson, 2007; Anderson et al., 2008). The temperature condition of zircon crystallization in the Toki granite was estimated using the calibration of Ferry and Watson (2007). The precise temperature estimation of zircon crystallization requires determination of suitable activities of SiO_2 and TiO_2 at the time of zircon crystallization in granitic magma (e.g. Barboni et al., 2013). Abundant quartz occurrences in the Toki granite indicates SiO_2 saturation in the magma in equilibrium with zircon, which enable us to assume the activity of SiO_2 in the magma = 1. The Toki granite also includes rutile as high-temperature Ti-saturation phase, implying TiO_2 in equilibrium with zircon. And thus the activity of TiO_2 is defined as 1 in the magma.

Table 1 shows crystallization temperature conditions of the LLC and OZ in representative zircons (shown in Fig. 2) deduced from the Ti-in-zircon thermometer after Ferry and Watson (2007). Reported uncertainty of the temperature estimation includes both errors in titanium quantification and in Ti-in-zircon calibration. Crystallization temperatures collected from the LLC range from about 760^{o}C to about 910^{o}C (Table 1). Zircons with oscillatory zonation include crystallization temperatures ranging from about 690^{o}C to about 850^{o}C. Zircons in the observed Categories 1 and 2 typically show relatively-higher temperature at the cores and lower temperature at the rims.

The highest temperature in the OZ (about 850^{o}C) represents the onset temperature at which magma behaved as if in a chamber, and thus the temperature in which magma came to a standstill (i.e. emplacement of magma). The lowest titanium concentration in the OZ indicates the cessation temperature of zircon growth in the Toki granite (about 690^{o}C), which is consistent with the solidus temperature of granite as presented by Tuttle and Bowen (1958), meaning that the OZ growth continued until the magma reached the solidus temperature. Therefore, the OZ grew continuously with temperature decrease in a cooling magma chamber from emplacement to crystallization / solidification.

The LLC crystallization occurred in a chemically homogeneous granitic magma in the temperature range from about 910^{o}C to about 760^{o}C as the magma cooled, whereas the OZ developed in the temperature range from about 850^{o}C to about 690^{o}C. That is, the magmatic zircon growth has the two stages. The LLC was probably produced by 'interfacial reaction-controlled growth' and the OZ was possibly derived by 'diffusion-controlled growth', according to Fisher's (1978) classification of kinetic processes. The magma at higher temperature can maintain faster diffusion rates in the melt (magma) (e.g. Fisher, 1973, 1978). The interfacial reaction-controlled growth occurs predominantly in the magma at relatively higher temperature because of rate-determining process. The LLC indicates chemical homogeneity (Fig. 3) and the formation temperature condition of LLC was higher than that of OZ, and thus the LLC can be defined as an interfacial reaction-controlled structure. The magma with lower temperature involves a slower diffusion rate in the melt relative to the reaction rate with respect to zircon crystallization, which gives the diffusion-controlled growth of zircon. Such a kinetic effect is likely to form oscillatory zoning in the zircon crystal (Fig. 3). Thus, the diffusion rate deceleration with decreasing temperature of the magma triggered the transition from the interfacial reaction-controlled growth producing LLC to the diffusion-controlled growth forming OZ.

Conclusions

The internal structure of zircons observed by CL imaging and the titanium concentration provides the basis for classification of zircon growth into two events in the formation process of a granitic pluton. In particular, this paper described the procedures and results of titanium concentration determined by EPMA. The magmatic zircons collected from the Toki granite show the two kinds of internal structure: LLC (low luminescence core) and OZ (oscillatory zonation). The LLC crystallized due to interfacial reaction-controlled growth with the cooling granitic magma under temperature conditions of about 910^{o}C to about 760^{o}C. Under temperature conditions from about 850^{o}C to about 690^{o}C, the OZ developed due to diffusion-controlled growth within a cooling magma chamber.

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日本語要旨

カソードルミネッセンス象観察に基づくジルコンの内部構造の分類，Ti-in-zircon 温度計より内部構造ごとの結晶化温度の決定，内部構造ごとの U-Pb 年代の決定を組み合わせた研究は，時間・温度の観点からジルコンの成長プロセスを明らかにする．本論文では，2016 年に Journal of Mineralogical and Petrological Sciences に公表された Yuguchi et al. (2016) “Zircon growth in a granitic pluton with specific mechanisms, crystallization temperatures and U-Pb ages: Implication to the ‘spatiotemporal’ formation process of the Toki granite, central Japan.”に関して，EPMA を用いたジルコンのチタン濃度の定量そしてそこから得られた結果について解説を行った．

土岐花崗岩体中のマグマ起源のジルコンはカソードルミネッセンス像から 2 つの領域に区分できる；1 つは LLC(low luminescence core) であり，もう 1 つは OZ (oscillatory zonation)である．それぞれの領域に対して EPMA による Ti 濃度の定量分析を実施し，LLC は 910℃から 760℃の間の反応律速型成長によって，OZ は 850℃から 690℃までの拡散律速型成長により生じたことを明らかにした．