

**Solidification depth and crystallization age of the Shiaidani  
Granodiorite: constraints to the average denudation rate of the Hida  
Range, central Japan**

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## **Abstract**

Solidification pressure and crystallization age of the ca. 5 Ma Shiaidani Granodiorite (Hida Mountain Range, central Japan) are determined based on Al-in-hornblende geobarometry and U-Pb zircon dating. Al-poor patchy replacements developed in amphiboles are common in this granite and petrographic study revealed that the replacements include chloritized biotite and albitic plagioclase. These are probably the hydrothermally recrystallized domains, and should not be used for solidification pressure estimates. Magmatic rim of amphibole is characterized by  $\text{Si} < 7.3$  a.p.f.u. ( $\text{Al}_{\text{IV}} > 0.7$  a.p.f.u.), and utilized in solidification pressure estimate that yielded 0.17-0.29 GPa. The solidification age of the granite is estimated as ca. 5.6-5.2 Ma using U-Pb zircon dating. From these data, the lower limit of an average denudation rate after ca. 5.6-5.2 Ma for the area where Shiaidani Granodiorite is exposed is estimated as 0.93-2.5 mm/yr.

**Keywords:** granite, exhumation, hornblende, patchy zoning

## **1 Introduction**

Emplacement depth of granitoids in the upper continental crust in combination with their solidification ages are useful in reconstructing the complex exhumation and tectonic processes of the region where the granitoids are currently exposed. The Kurobe area, Hida Mountain Range, central Japan is

characterized by the exposure of the youngest granites in the world (Ito et al., 2013, 2017), and exhumation history of the young granites have been of great interest (Yamada, 1997; Yamada & Harayama, 1999; Spencer et al., 2019). However, results of low-temperature geochronological methods are often thermally affected by the late-stage granite intrusions (Yamada & Harayama, 1999) and a method to reliably constrain the depth and age information was needed.

The Al-in-hornblende barometer (Anderson & Smith, 1995; Hammarstrom & Zen, 1986; Mutch et al., 2016; Schmidt, 1992) has long been used to constrain the solidification pressure of granitoid plutons. In applying the Al-in-hornblende barometer, choosing a right composition of hornblende is very important, because the barometer uses the Al content of amphibole that crystallized at the granite solidus. Mutch et al. (2016) recommended to use amphibole analyses taken from the rims of grains, in contact with plagioclase and in apparent textural equilibrium with the rest of the mineral assemblage at temperatures close to the haplogranite solidus, as determined from amphibole–plagioclase thermometry. However, using the rim composition of amphibole is not a successful criterion in the case of hornblende with patchy Al zoning (e.g., Yamaguchi et al., 2003; Hartung et al., 2017), which is probably a result of post-magmatic hydrothermal alteration. As a criterion of amphibole crystallized under magmatic or subsolidus conditions, Chivas (1981) proposed that amphiboles with  $\text{Si} > 7.3$  a.p.f.u. for  $\text{O} = 23$  ( $\text{Al}_{\text{IV}} < 0.7$  a.p.f.u.) are not truly magmatic and crystallized under subsolidus conditions in the presence of a fluid phase. On the other hand, in the study of the

Takidani Pluton (Hida Range), [Hartung et al. \(2017\)](#) considered low-Al and high-Al amphiboles observed as patchy zones could have formed under near-isobaric conditions and difference in Al may be related to the temperature and chemical variability of the melt from which they crystallized ([Hartung et al., 2017](#)). Therefore, careful microstructural observation on the coexisting minerals of each amphibole domain is required to understand the development of patchy zones in amphibole, and to finally constrain the solidification pressure of the pluton.

This study examines mode of occurrence and chemical composition of amphibole in the Shiaidani Granodiorite in detail to constrain the formation timing of the patchy domains. We petrographically constrain suitable amphibole domains/compositions that coexisted with the necessary phases to apply the Al-in-hornblende barometry (e.g., [Mutch et al., 2016](#)) and estimate the solidification pressure of the Shiaidani Granodiorite. In combination with the U-Pb zircon dating of the granodiorite, average denudation rate after ca. 5.6-5.2 Ma is finally constrained for the area where Shiaidani Granodiorite is exposed. In this study, we follow the definition of “exhumation” and “denudation” by [Ring et al. \(1999\)](#), [Reiners and Brandon \(2006\)](#) and [Sueoka and Tagami \(2019\)](#). “Exhumation” is the vertical distance traveled by rocks relative to Earth’s surface and “denudation” takes into account the lateral movement of the exhumed rocks.

## 2 Geological setting

The Shiaidani Granodiorite crops out in the Kurobe area, Hida Mountain Range, central Japan (**Figure 1**). It is exposed as elongate-shaped pluton with a NS length of ~12 km and maximum EW width of ~3 km ([Harayama, 2015](#)). The world's youngest granite, the Kurobegawa Granite (ca. 0.8 Ma), is exposed along the Kurobegawa River ([Ito et al., 2013; 2017; Spencer et al., 2019](#)). It is accompanied by young volcanics such as 1.7-1.6 Ma Jiigatake Volcanics (mainly rhyolitic welded tuff and andesite and rhyolite lavas) and 1.6 Ma Shirakawa-tengu Volcanics (mainly rhyolitic welded tuff) to the east (**Figure 1**; [Harayama et al., 2015](#)). The Kurobegawa Granite intrudes the Jiigatake Volcanics ([Harayama et al., 2010](#)). The Shiaidani Granodiorite is exposed roughly to the west of Kurobegawa River. It was previously considered as a part of the Kurobe-bessan Granite ([Harayama et al., 2010](#)), which was divided into the Shiaidani Granodiorite (ca. 5.5-5.4 Ma) and the Kuranosuke Granite (ca. 9.5-9.1 Ma) based on the difference in U-Pb zircon ages ([Harayama, 2015; Ito et al., 2013](#)). The Kuranosuke Granite is distributed to the west of the Shiaidani Granodiorite (**Figure 1**). The Shiaidani Granodiorite is also dated to be ca. 7.1-5.1 Ma and ca. 5.1-4.3 Ma by K-Ar dating of hornblende and biotite, respectively ([Ogata et al., 1983; Yamada & Harayama, 1999; Harayama, 2015; Harayama et al., 2010](#)). The zircon fission track age is dated at ca. 1.5 Ma ([Harayama et al., 2010](#)). The Kuranosuke Granite, on the other hand, is dated at ca. 5.1 Ma by K-Ar dating of biotite ([Harayama et al., 2010](#)). Based on the existence of a decussate structure of biotite, [Harayama et al. \(2010\)](#) considered that recrystallization due to thermal metamorphism was evident in the Kurobe-bessan Granite.

### 3 Analytical methods

Quantitative analyses of rock-forming minerals and X-ray elemental mapping of thin section samples were performed by a JEOL JXA-8105 superprobe at Kyoto University. Analytical conditions for quantitative analyses were 15.0 kV acceleration voltage, 10 nA beam current, and 3  $\mu$ m beam diameter. The counting time for the peak and backgrounds were 30 s and 15 s for Cl, 60 s and 30 s for F, and 10 s and 5 s for other elements. Natural and synthetic minerals were used as standards and the ZAF correction was applied. Representative mineral analyses are given in [Table 1](#). Recalculation of ferric iron in amphibole and calculation of amphibole formula are based on [Holland and Blundy \(1994\)](#).

Two granite samples were crushed in a rod mill and stainless steel mortar. Zircon grains were separated by panning, magnetic separation and using heavy liquid at Kyoto Fission-Track Co. Ltd. After handpicking under a stereomicroscope, zircon grains were mounted in epoxy resin (Struers Specifix-20). Cathodoluminescence (CL) images of zircon grains were obtained by using a field emission electron microprobe JEOL JXA-8530F at Japan Atomic Energy Agency, Tono Geoscience Center (JAEA, TGC). Analysis points were selected to avoid cracks and inclusions using CL images. U-Pb isotopic analysis by laser ablation–inductively coupled plasma–mass spectrometry (LA–ICP–MS) was performed in JAEA, TGC using Thermo Fisher Scientific Neptune-*Plus* coupled with Photon-Machines Analyte G2 Excimer laser on separate zircon grains. For U-Pb isotope analysis

to estimate the zircon crystallization ages, the 91500 zircon (Wiedenbeck et al., 1995, 2004) was used as the primary reference and standard material to correct the mass bias effect on  $^{206}\text{Pb}/^{238}\text{U}$  and  $^{207}\text{Pb}/^{206}\text{Pb}$ , respectively. Duplicate measurements of the secondary reference materials of OD-3 (33.0  $\pm$  0.1 Ma: Iwano et al., 2013) were carried out to assess the age data obtained from the unknown samples. Details of the analytical conditions are given in Table S1. Isoplot 4.15 (Ludwig, 2012) was used to create concordia diagrams and calculation of a weighted mean age.

In order to obtain accurate crystallization ages for young (<2 Ma) zircon, it is necessary to consider the contributions of common Pb and initial disequilibrium caused by Th/U and Pa/U fractionation in the zircon-melt system (Sakata et al., 2017; Sakata, 2018). We performed the correction for common Pb by a single correction based on a modified Tera-Wasserburg concordia diagram (modified  $^{207}\text{Pb}$  method; Sakata, 2018). In order to confirm the initial disequilibrium on the zircon data obtained in this study, we also made correction of the initial disequilibrium effect and compared it with the equilibrium results. For the initial disequilibrium correction, we used the average Th/U ratio of analyzed zircon (0.67) for the Th/U ratio of zircon grains at the time of zircon crystallization  $[(\text{Th}/\text{U})_{\text{Zircon}}]$ . Th/U ratio of the melt  $[(\text{Th}/\text{U})_{\text{Melt}}]$  was assumed to be 4.8, which was an average derived from river sand samples collected in the vicinity of the Kurobegawa Granite (Imai et al., 2004; Ito et al., 2013). Then, we used a Th/U fractionation factor  $[f_{\text{Th/U}} = (\text{Th}/\text{U})_{\text{Zircon}}/(\text{Th}/\text{U})_{\text{Melt}}]$  of 0.123 with 30% of estimation error for the initial Th/U fractionation correction. In this study, we

assumingly used general value of Pa/U fractionation factor between melt and zircon [ $f_{\text{Pa/U}} = (\text{Pa/U})_{\text{Zircon}}/(\text{Pa/U})_{\text{Melt}}$ ] of 3.36 with 30% of estimation error (compilation value based on Rioux et al., 2015; Sakata et al., 2017; Schmitt, 2011), because we did not determine a Pa/U partitioning factor.)

#### 4 Sample description

Two unweathered samples of the Shiaidani Granodiorite (Harayama, 2015) were collected from the outcrop exposures (Figure 1). These samples are likely affected by post-magmatic hydrothermal activity as indicated by complex patchy chemical zoning of amphibole, chloritization of biotite and partial sericitization of plagioclase. In order to check coexistence of mineral phases required for the application of the Al-in-hornblende geobarometry, a detailed mineral description is made for these samples.

Sample KRG16-07 is a hornblende-biotite granite (Figure 2), which was collected at the same outcrop as KRG-07 showing U-Pb zircon age of  $5.6 \pm 0.1$  Ma (Ito et al., 2013). Matrix mineral assemblage of this sample is amphibole + biotite (mostly chloritized) + plagioclase + quartz + K-feldspar + magnetite + titanite + zircon + apatite + allanite. Amphibole shows gradual core/rim chemical zoning accompanied by discontinuous patchy replacements; the core and patchy replacements are dark under the back scattered electron (BSE) images whereas the rim is slightly bright under the BSE images (Figure 2c). The core is weakly enriched in Mg (Figure 2f) and Na



(Figure 2h), while the rim is weakly enriched in Fe (Figure 2d) and K (Figure 2k). The patchy replacements are enriched in Mg (Figure 2f), Mn and Si, and depleted in Fe (Figure 2d), Al (Figure 2e), Cl (Figure 2g), Na (Figure 2h) and K (Figure 2k) compared to the core. The amphibole is mostly magnesiohornblende except for the final-stage rim and patches corresponding to actinolite (Figure 3). The amphibole core and rim enclose biotite, K-feldspar, plagioclase, ilmenite, magnetite, zircon and apatite. On the other hand, the patchy replacements include biotite (partly chloritized), plagioclase, titanite, K-feldspar, magnetite, zircon and apatite. Oscillatory-zoned plagioclase shows a decrease in anorthite content at the rim, and the plagioclase rim in contact with hornblende rim shows composition of An<sub>14-22</sub>. An albitic outermost rim (<An<sub>14</sub>) is locally developed on the An<sub>14-22</sub> plagioclase, and such plagioclase is commonly in contact with patchy replacements. The composition of albitic plagioclase enclosed in the patchy replacements is similar to the composition of the outermost albitic rim of matrix plagioclase (Figure 2e, i).

Sample KRG16-101 is a hornblende-biotite granite (Figure 4). Matrix mineral assemblage is amphibole + biotite + plagioclase + quartz + K-feldspar + magnetite + titanite + zircon + apatite + allanite. Chloritization of biotite is weaker compared to sample KRG 16-07, and is only limited to the rims and along the cleavages of biotite. Amphibole shows core/rim chemical zoning with patchy zoning under BSE images. The gradual core/rim zoning is recognized as darker core with brighter rim under the BSE images (Figure 4c). The discontinuous, commonly BSE-dark patchy replacements cut

the gradual zoning pattern. The rim is enriched in Fe (Figure 4e), Na (Figure 4i), K (Figure 4l) and Mn, and depleted in Mg (Figure 4g) compared to the core. The BSE-dark patchy replacements are more depleted in Mg (Figure 4g), Al (Figure 4f), Cl (Figure 4h) and Na (Figure 4i), and enriched in Si (Figure 4d) and Mn as observed by X-ray chemical mappings. The amphibole is mostly magnesiohornblende except for the final-stage patchy replacements that correspond to actinolite (Figure 5). The amphibole core and rim include plagioclase (rim with An18), K-feldspar, quartz, magnetite, ilmenite, apatite, zircon whereas the patchy replacements include albitic plagioclase, K-feldspar, quartz, chloritized biotite, magnetite, titanite, apatite, zircon. Composite pseudo-inclusion of K-feldspar and albitic plagioclase, which is connected with matrix via cracks, is commonly developed even in the core of amphibole. Oscillatory-zoned plagioclase in the matrix shows decrease in anorthite content at the rim, and the composition of the rim in contact with amphibole rim is An14-26. Locally, albitic film (<An14) is developed at the plagioclase rim. On the other hand, amphibole is enclosed in plagioclase showing various compositions of An39-52, An39-41 (core) and An16-17 (rim).

## **5 U-Pb zircon dating of KRG16-101**

Zircons in this sample is commonly euhedral, and present in the matrix and also enclosed in biotite, hornblende, quartz and K-feldspar. It is oscillatory- and sector-zoned under CL images (Figure 6, inset). Mineral inclusions such as apatite are common in zircon. Th/U ratio of oscillatory-zoned rim ranges

from 0.42 to 1.08 (0.65 in average). Dates of oscillatory-zoned rims are determined for sample KRG16-101, and the results are summarized in [Table S2](#). Concordia plots with  $1\sigma$  error ellipses show that the analytical results not corrected for the contribution of the common Pb and initial disequilibria are discordant ([Figure 6](#)). Common Pb-corrected weighted average of the 24 analysis spots yielded  $^{238}\text{U}$ - $^{206}\text{Pb}$  age of  $5.20 \pm 0.17$  Ma (95% confidence level, MSWD = 0.27, probability = 0.999). The initial disequilibrium correction resulted in ~2% difference in the weighted average age, *i.e.*,  $5.31 \pm 0.17$  Ma (95% confidence level, MSWD = 0.27, probability = 1.000). For simplicity, we prefer the former age assuming initial equilibrium in this study.

## **6 Amphibole composition and application of Al-in-hornblende geobarometry**

The chemical composition of amphiboles in sample KRG 16-07 is plotted in [Figure 3](#) and that in sample KRG16-101 is plotted in [Figure 5](#). As described above, amphibole represents gradual chemical zoning from the core to the rim, and discontinuous patchy replacements cut this texture ([Figures 3 and 5](#)). In creating [Figures 3 and 5](#), the cores, rims and replacements are classified based mainly on BSE images and partly on X-ray mappings. Therefore, discrimination between gradual core and rim was not always easy under the BSE images alone, and caused scattering of core points within rim-dominant compositional areas ( $\text{Si} > \sim 7.1$  a.p.f.u. and  $\text{Al}_{\text{IV}} < \sim 0.9$  a.p.f.u. areas of [Figures 3 and 5](#)). Nevertheless, it is important that original core/rim zoning and discontinuous patchy replacements are chemically

discriminated at around  $\text{Si} = 7.3$  a.p.f.u. and  $\text{Al}_{\text{IV}} = 0.7$  a.p.f.u. ( $\text{O} = 23$ ) for both samples (Figures 3 and 5).

Because patchy replacements include secondary minerals such as albitic plagioclase, chloritized biotite, and composite pseudo-inclusion of K-feldspar and albitic plagioclase that is connected with matrix via cracks (Figures 2 and 4; note inclusion minerals in Al-poor patches), it is considered that the patchy replacements are the recrystallized domains during hydrothermal alteration. The most extensive amphibole composition that patchy replacements show is actinolitic composition (Figures 3 and 5), and coexistence of such domains with albitic plagioclase and chloritized biotite also supports the alteration under subsolidus hydrothermal condition.

On the other hand, amphibole domains that show original core/rim zoning preserve amphibole composition of igneous stage as supported by presence of more calcic plagioclase inclusions ( $\sim\text{An}_{18}$ ) as well as higher Al contents of the amphibole domains compared to the replacements. The mineral inclusions in the igneous amphibole domains (plagioclase, K-feldspar, quartz, magnetite, ilmenite, apatite) and the matrix minerals in contact with amphibole rim (biotite, plagioclase rim) satisfy the mineral assemblage required for the application of Al-in-hornblende geobarometer (Mutch et al., 2016). Therefore, Al-in-hornblende geobarometer is applied to the amphibole rim to constrain the solidification pressure of the granite. Hornblende-plagioclase geothermometer (Holland & Blundy, 1994) is also applied to the amphibole rim and plagioclase rim

in contact with the amphibole rim. Composition of amphibole rim enclosed in plagioclase rim is also used for the  $P$ - $T$  estimate (Figure 7). Although  $P$ - $T$  estimates from patchy replacement is not considered to represent the solidification condition, they are also plotted in Figure 7 for comparison. The Mutch et al. (2016) calibration is preferred in this study, because it is experimentally calibrated for pressure down to 0.8 kbar and applicable to shallower plutons without extrapolation compared to previous calibrations (e.g., 2.5-13 kbar for Schmidt, 1992). Additionally, calibration dataset of Mutch et al. (2016) involves very wide range of plagioclase composition (An15-76), and applicable without extrapolation to the mineral assemblage with low-An plagioclase ( $\geq$  An15) as in the case of the studied samples.

Application of the Al-in-hornblende geobarometer (Mutch et al., 2016) and hornblende-plagioclase geothermometer (Holland & Blundy, 1994) to the amphibole rim and plagioclase rim pair resulted in 616-691°C and 0.17-0.27 GPa ( $\pm 0.04$  GPa) for KRG 16-07, and 620-702°C and 0.17-0.29 GPa ( $\pm 0.05$  GPa) for KRG16-101 (Figure 7). These  $P$ - $T$  estimates are plotted on the haplogranite solidus within error (Figure 7). Assuming rock density of 2700 kg/m<sup>3</sup> and lithostatic pressure gives solidification depths of  $6.3 \pm 1.0$  km to  $10.3 \pm 1.7$  km for KRG 16-07, and  $6.4 \pm 1.0$  km to  $11.0 \pm 1.8$  km for KRG 16-101.

## 7 Discussion

## 7.1 Discriminating magmatic and hydrothermally recrystallized amphibole

The Kurobe area exposes younger granite of ca. 0.8 Ma (Ito et al., 2013; 2017; Spencer et al., 2019), and estimation of denudation rate by low-temperature geochronology can be thermally affected by younger intrusions. On the other hand, estimation of exhumation rate based on Al-in-hornblende geobarometry is not strongly affected by later intrusions if the post crystallization, hydrothermal recrystallization of amphibole can be properly evaluated. The exhumation rate can be considered as the lowest estimate of an average denudation rate of the area, because denudation takes into account the lateral movement of the exhumed rocks (Batt & Braun, 1999; Ring et al., 1999; Reiners & Brandon, 2006; Sueoka & Tagami, 2019).

Chivas (1981) proposed that amphiboles with  $\text{Si} > 7.3$  a.p.f.u. ( $\text{Al}_{\text{IV}} < 0.7$  a.p.f.u.) are not truly magmatic and crystallized under subsolidus conditions in the presence of a fluid phase. In this study, chemical boundary between rim and discontinuous patchy replacements of amphibole, determined based on microtextural observation, is recognized at  $\text{Si} = 7.3$  a.p.f.u. and  $\text{Al}_{\text{IV}} = 0.7$  a.p.f.u. (Figures 3 and 5). The patchy replacements commonly enclose and coexist with albitic plagioclase and chloritized biotite, which is consistent with the interpretation of Chivas (1981) that it is the recrystallized domain under subsolidus conditions in the presence of a fluid phase. Some of the  $P$ - $T$  conditions estimated using the patchy replacements and the plagioclase composition in contact with them are plotted away from the haplogranite solidus (Figure 7), supporting the subsolidus origin of

patchy replacements. Therefore, criterion of [Chivas \(1981\)](#) is applicable to the Shiaidani Granodiorite as well, and patchy replacements should not be used in estimating the solidification pressure of the granite, although some of the patches yield pressure similar to that obtained from the magmatic amphibole rim ([Figure 7](#)).

## **7.2 Estimating the lower limit of the average denudation rate of the Shiaidani Granodiorite area after ca. 5.6-5.2 Ma**

The presence of magmatic oscillatory zoning and absence of secondary replacement microtextures in the dated zircon grains ([Figure 6a inset](#)) suggests that the U-Pb zircon ages of ca. 5.6-5.2 Ma can be interpreted as the crystallization ages of these samples. By dividing the estimated solidification depths by the U-Pb zircon age, average exhumation rate of the Shiaidani Granodiorite is estimated as 0.93-2.2 mm/yr from KRG 16-07 and 1.0-2.5 mm/yr from KRG 16-101. This is considered to be the lowest estimate of the average denudation rate since ca. 5.6-5.2 Ma for the area where the Shiaidani Granodiorite is exposed. Nonetheless, uplift and denudation in the northern Hida Range, including the Kurobe area, could be accelerated since ca. 1.5-1.0 Ma, considering depositional ages of granitic gravels sourced from the Hida Range to the Matsumoto basin to the east ([Oikawa & Wada, 2004](#)). The estimation in this study may provide the lower limit of the denudation rates since ca. 1.5-1.0 Ma, considering the higher denudation rates (several to 10 mm/yr at a maximum) in shorter timescales

estimated based on the sedimentary yields in catchments (Ohmori, 1978; Fujiwara et al., 1999) and terrestrial *in-situ* cosmogenic nuclide techniques (Matsushi et al., 2014).

## 8 Conclusion

We estimated the solidification depth of the Shiaidani Granodiorite utilizing Al-in-hornblende geobarometry and solidification age using U-Pb zircon dating. The Al-poor patchy replacements developed in amphiboles are probably hydrothermally recrystallized domains and should not be used for solidification pressure estimates. The lower limit of an average denudation rate after ca. 5.6-5.2 Ma for the area where Shiaidani Granodiorite is exposed is estimated as 0.93-2.5 mm/yr.

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# **Figure captions**

Figure 1 Simplified geological map of the Kurobe area modified after [Harayama et al. \(2010\)](#) and [Harayama \(2015\)](#). Sample localities are also shown. The approximate emplacement ages (Ma) are indicated by the subscript numbers attached to F, G and V following [Harayama \(2015\)](#). MTL: Median Tectonic Line, ISTL: Itoigawa-Shizuoka Tectonic Line.

Figure 2 Photomicrographs, back scattered electron (BSE) image and X-ray elemental maps of an amphibole in sample KRG 16-07. (a) Photomicrograph of an amphibole-bearing domain. Plane polarized light (PPL). (b) Crossed polarized light (CPL) of (a). (c) BSE image of the same area as (a) and (b). White square represents the area for the X-ray elemental mapping shown in (d)-(k). (d)-(k) X-ray elemental maps for (d) Fe, (e) Al, (f) Mg, (g) Cl, (h) Na, (i) Ca, (j) Ti and (k) K. Note that albitic plagioclase and chloritized biotite are enclosed in Al-poor patchy replacements. Amp: amphibole, An: anorthite, Ap: apatite, Bt: biotite, Chl: chlorite, Ilm: ilmenite, Kfs: K-feldspar, Mag: magnetite, Pl: plagioclase, Qtz: quartz, Ttn: titanite.

Figure 3 Chemical composition of amphibole (O = 23) in sample KRG 16-07. (a) (Na+K) in A-site vs

Si, (b) Cl vs  $Al_{IV}$ , (c)  $Mg/(Mg+Fe^{2+})$  vs Si, (d)  $Mg/(Mg+Fe^{2+})$  vs  $Al_{IV}$ . Solid black lines represent compositional boundaries of amphiboles. Blue broken lines represent  $Si = 7.3$  a.p.f.u. and  $Al_{IV} = 0.7$  a.p.f.u.

Figure 4 Photomicrographs, BSE image and X-ray elemental maps of an amphibole in sample KRG 16-101. (a) Photomicrograph of an amphibole-bearing domain. PPL. (b) CPL photo of (a). (c) BSE image of the boxed area in (b). (d)-(k) X-ray elemental maps for the boxed area in terms of (d) Si, (e) Fe, (f) Al, (g) Mg, (h) Cl, (i) Na, (j) Ca, (k) Ti and (l) K. Note that albitic plagioclase and chloritized biotite are enclosed in Al-poor patchy replacements.

Figure 5 Chemical composition of amphibole (O = 23) in sample KRG 16-101. (a) (Na+K) in A-site vs Si, (b) Cl vs  $Al_{IV}$ , (c)  $Mg/(Mg+Fe^{2+})$  vs Si, (d)  $Mg/(Mg+Fe^{2+})$  vs  $Al_{IV}$ . Solid black lines represent compositional boundaries of amphiboles. Blue broken lines represent  $Si = 7.3$  a.p.f.u. and  $Al_{IV} = 0.7$  a.p.f.u.

Figure 6 (a) Conventional (Wetherill) and (b) Tera–Wasserburg concordia diagrams for U-Pb zircon dating of KRG 16-101. Inset in (a) is an example of CL image of a dated zircon grain (spot KRG 16-101-09).



433

434 Figure 7  $P$ - $T$  diagrams showing the estimated solidification conditions for (a) KRG 16-07 and (b)  
435 KRG 16-101. A water-saturated haplogranite solidus line is from [Johannes and Holtz \(1996\)](#).

436

437 Table S1 Instrumentation and operational settings for LA-ICP-MS analysis.

438

439 Table S2 (a) Results of LA-ICP-MS U-Pb zircon dating with common Pb correction assuming  
440 initial equilibrium. (b) Results of LA-ICP-MS U-Pb zircon dating with common Pb correction  
441 assuming initial disequilibrium.

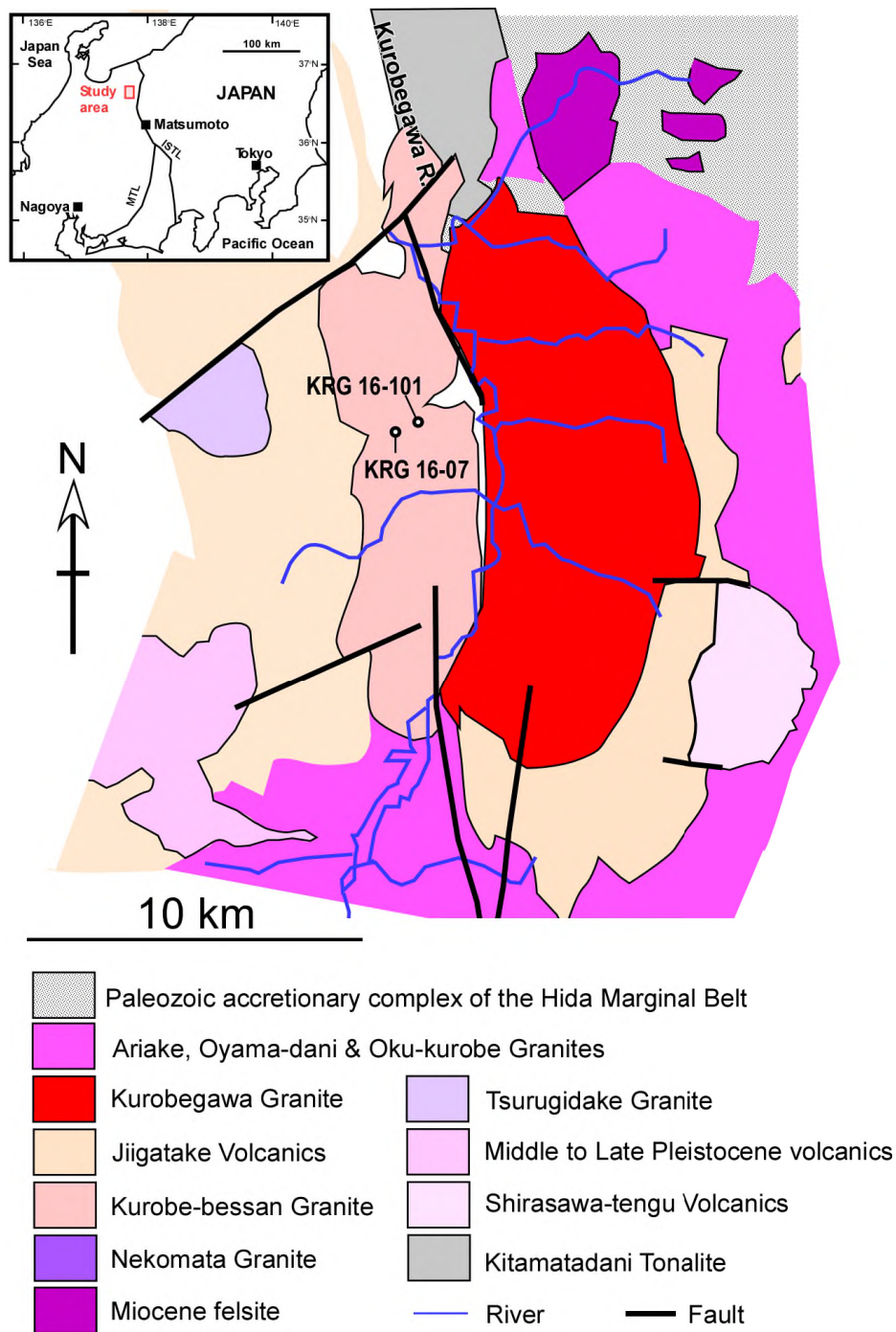


Fig. 1

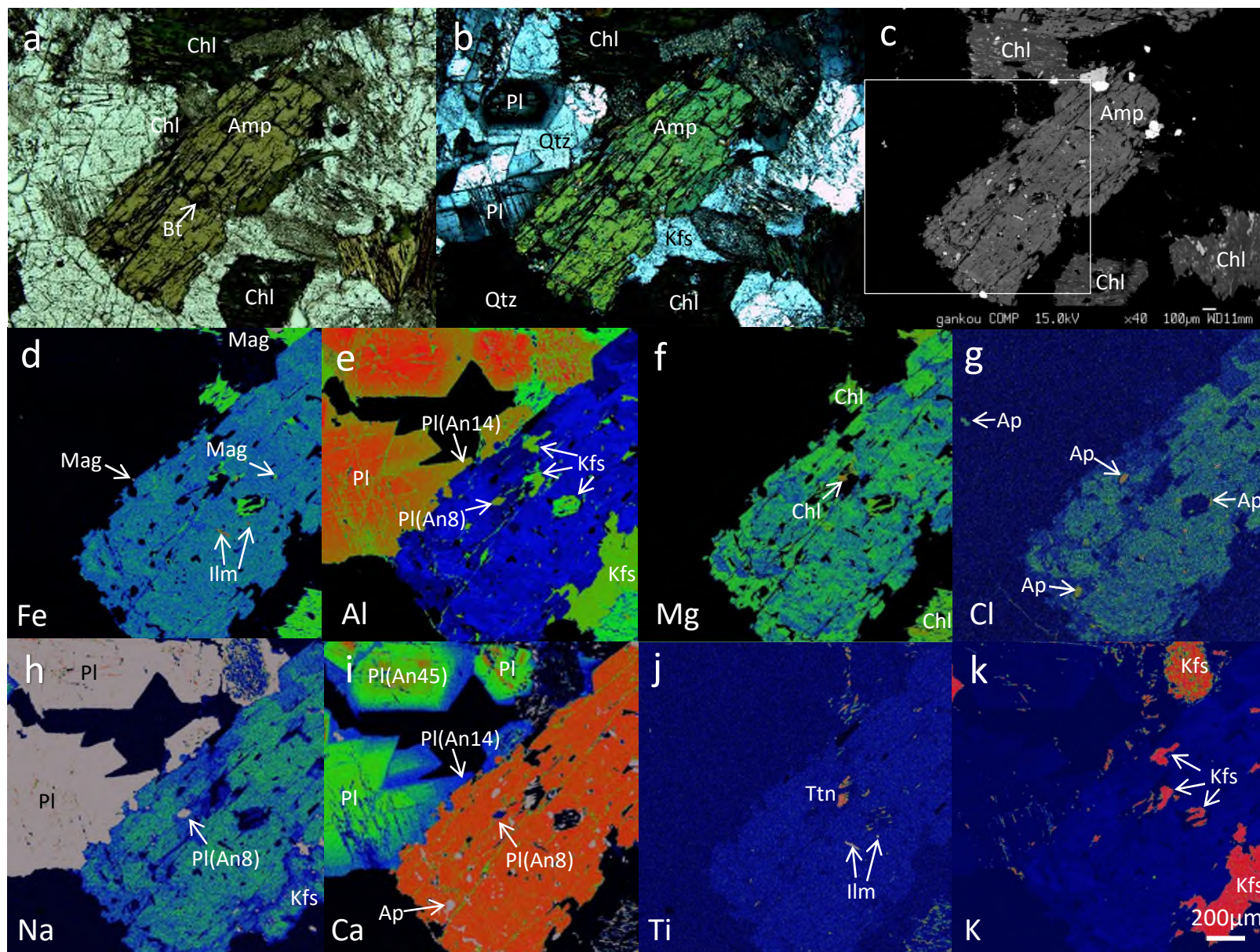


Fig. 2

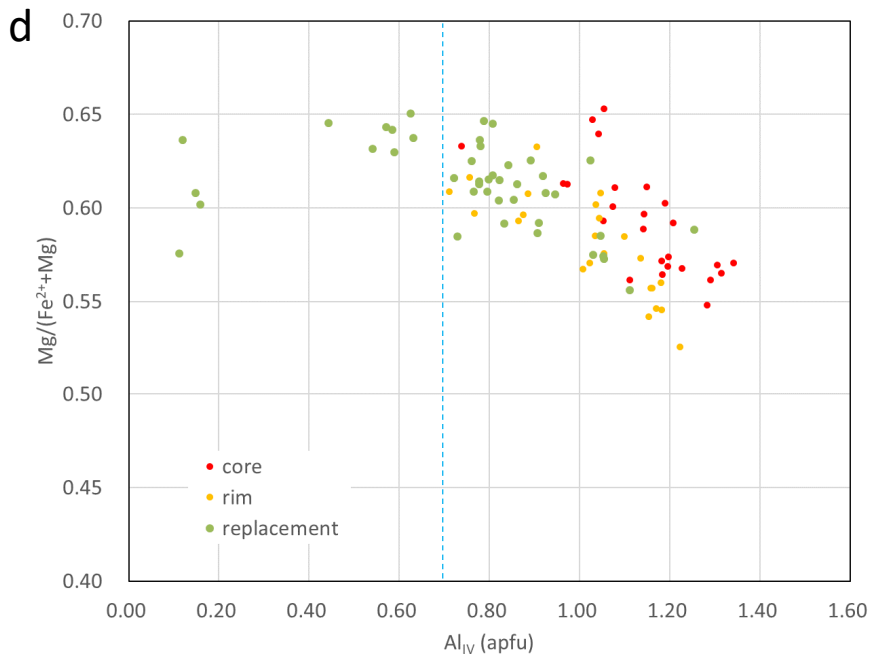
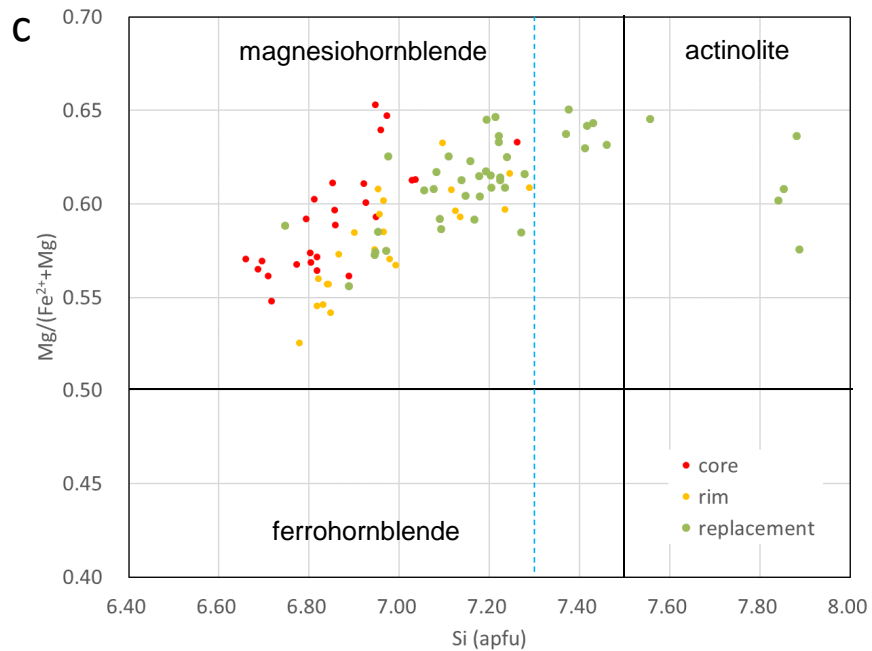
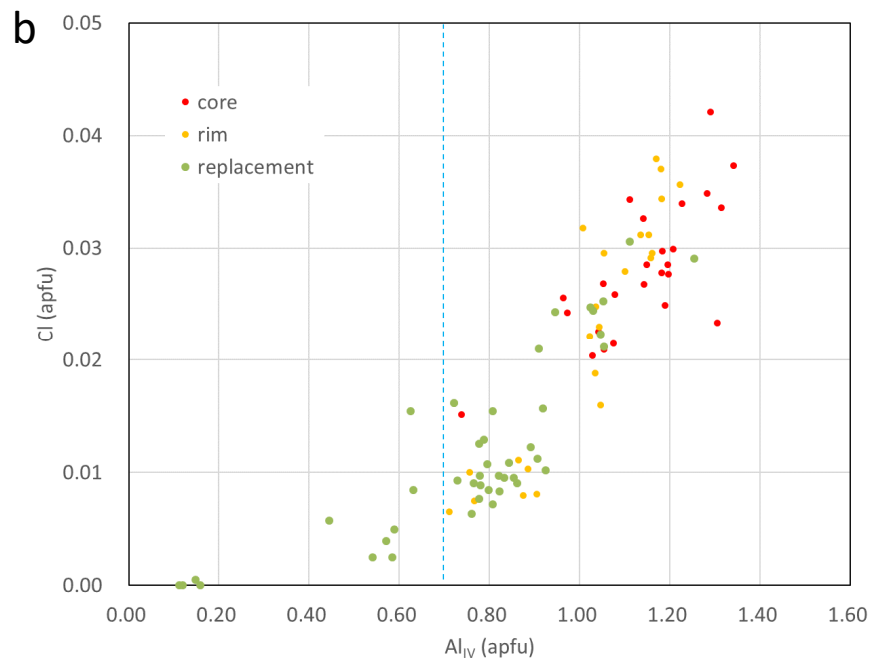
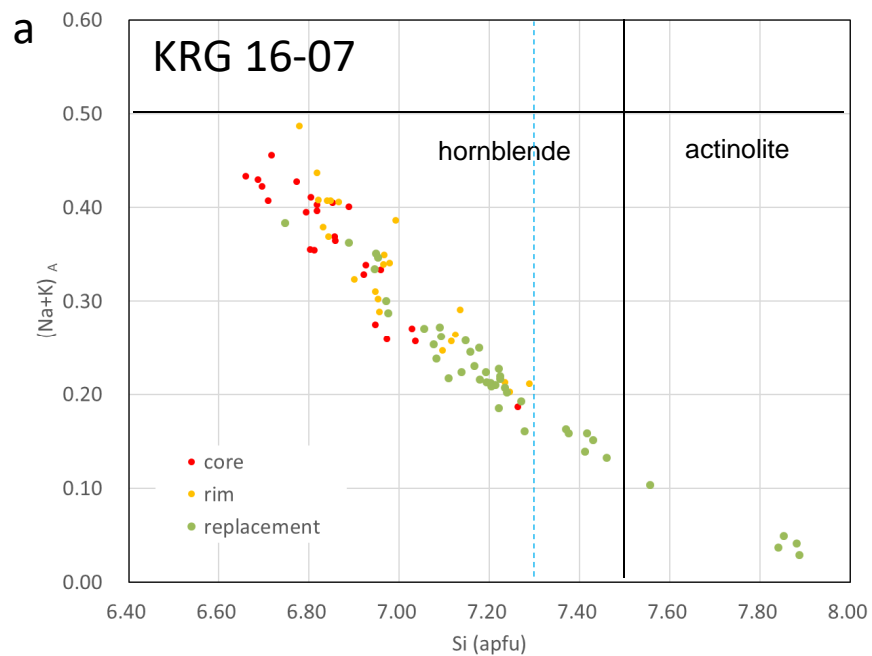


Fig. 3



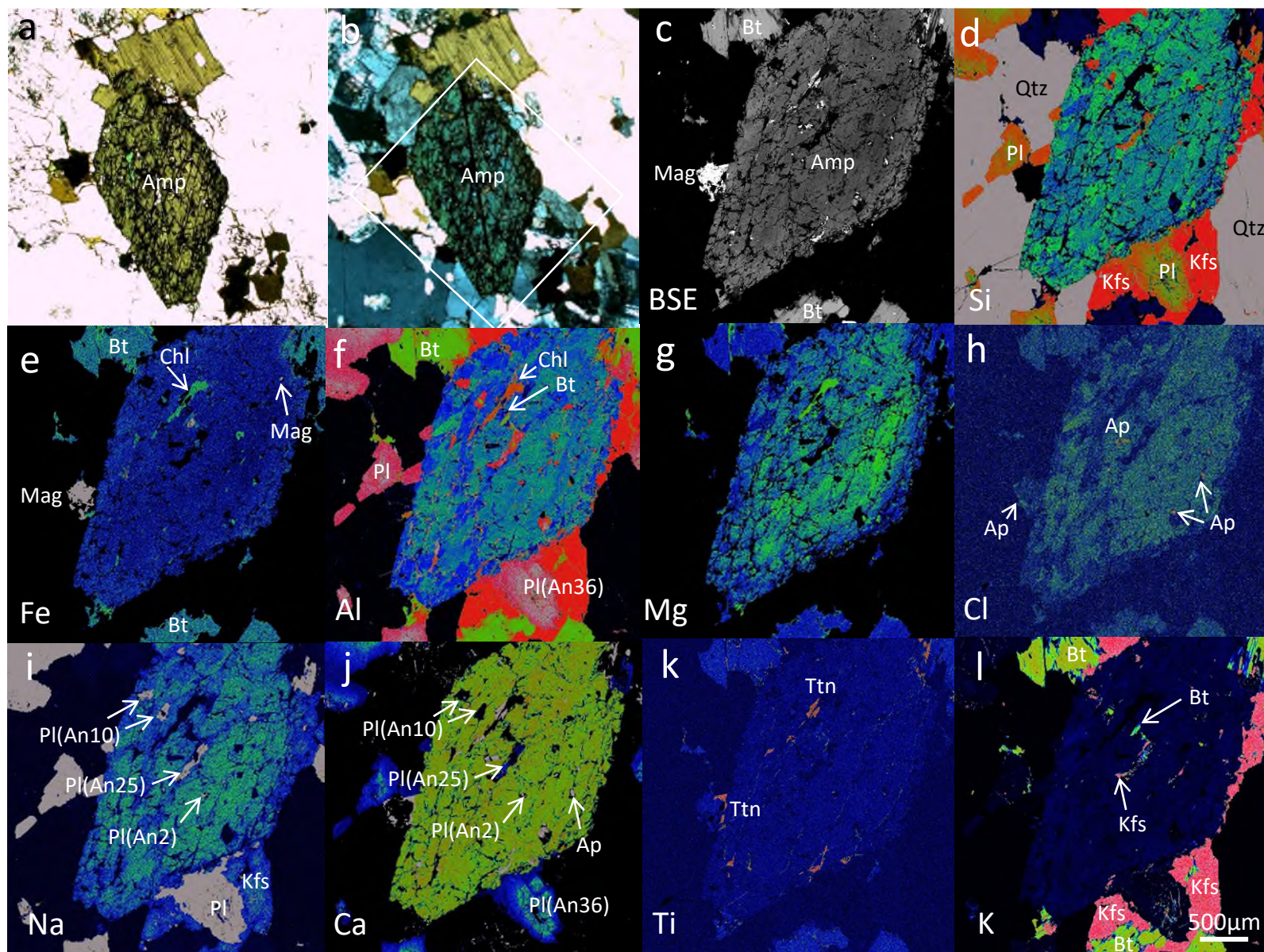


Fig. 4

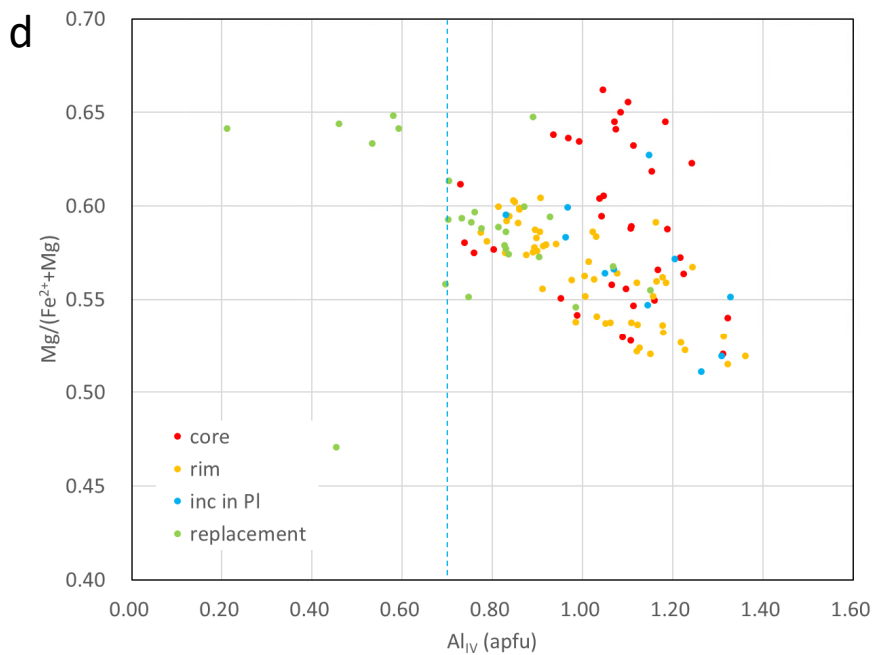
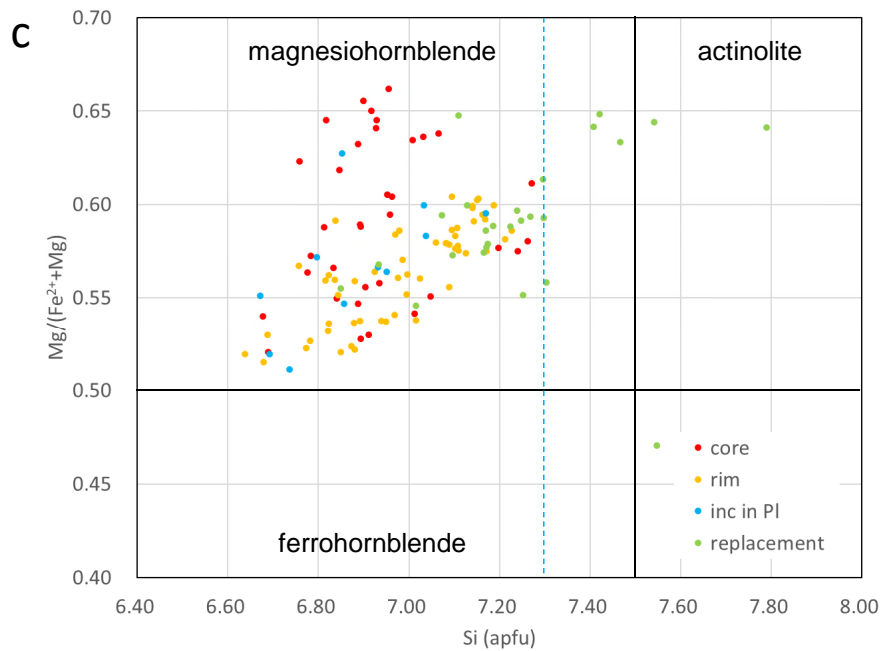
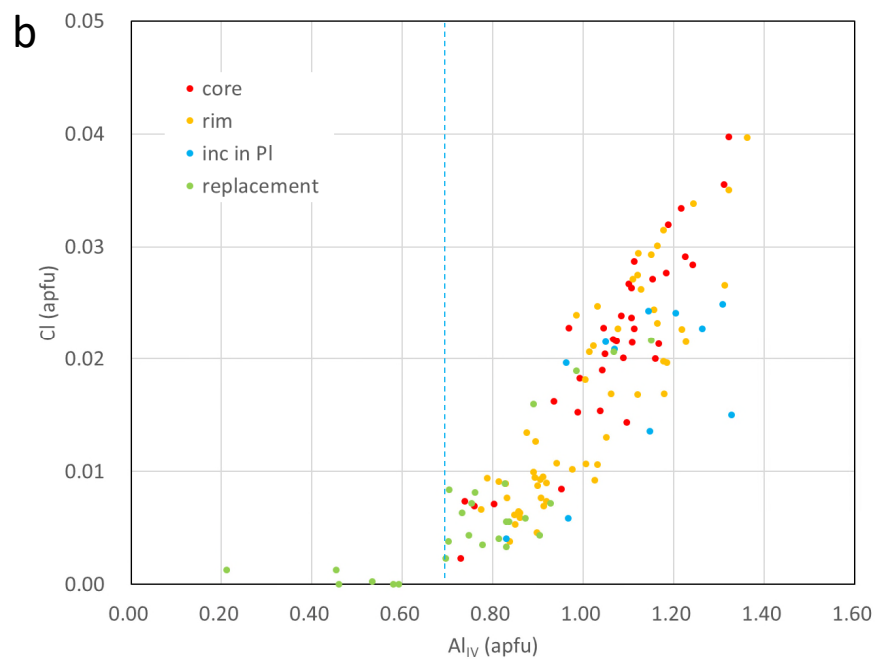
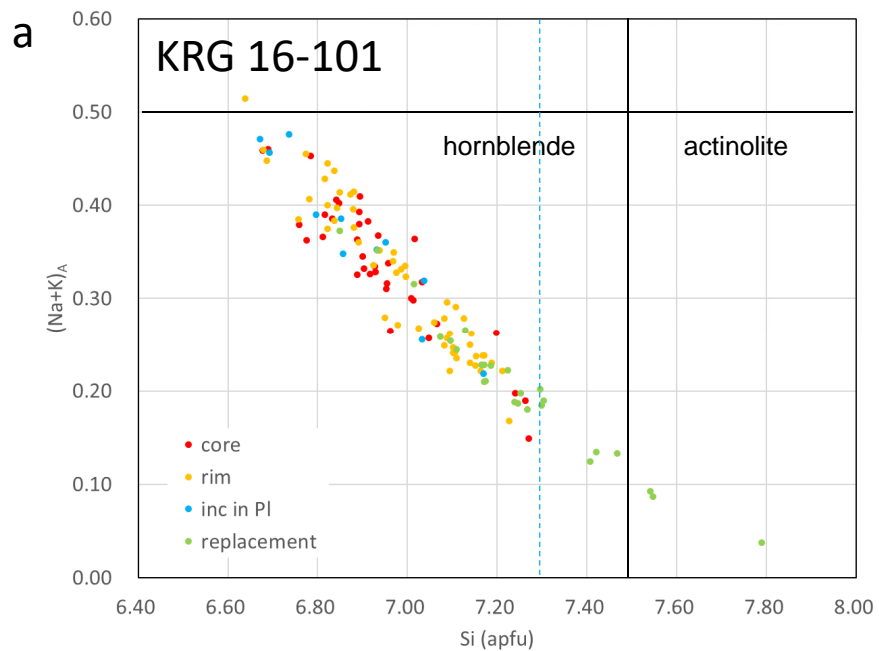


Fig. 5

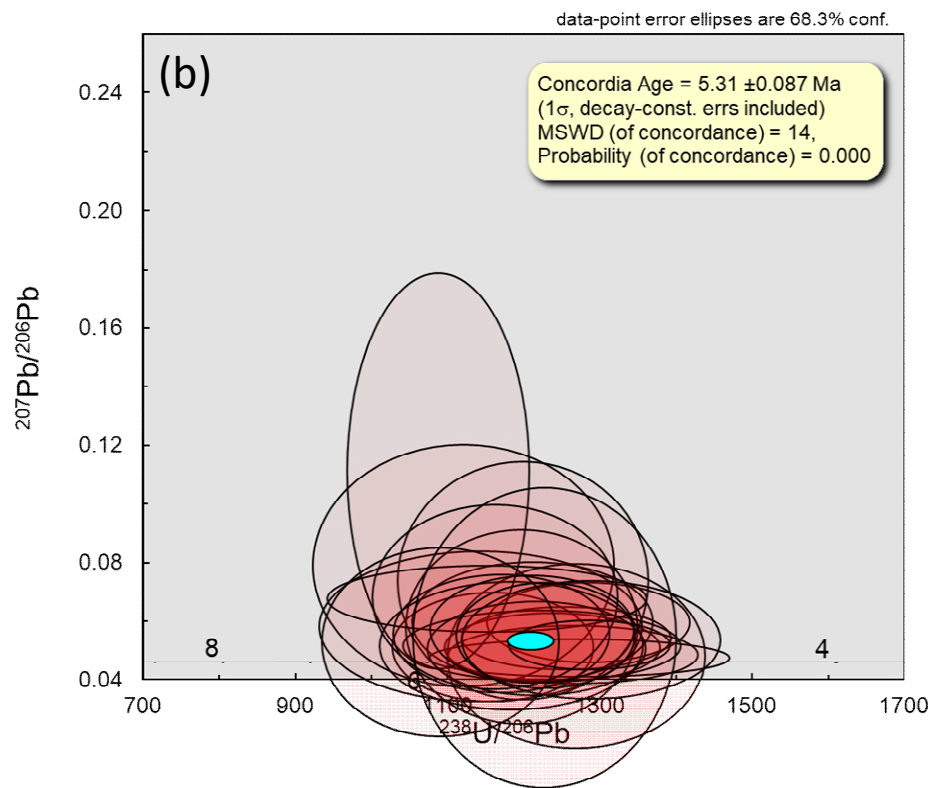
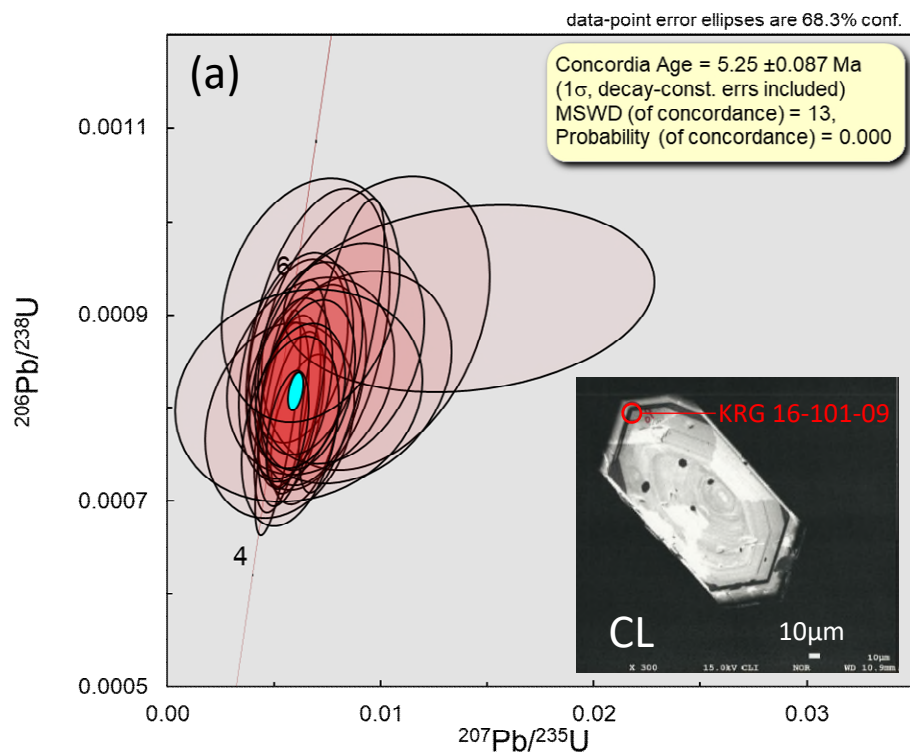


Fig. 6

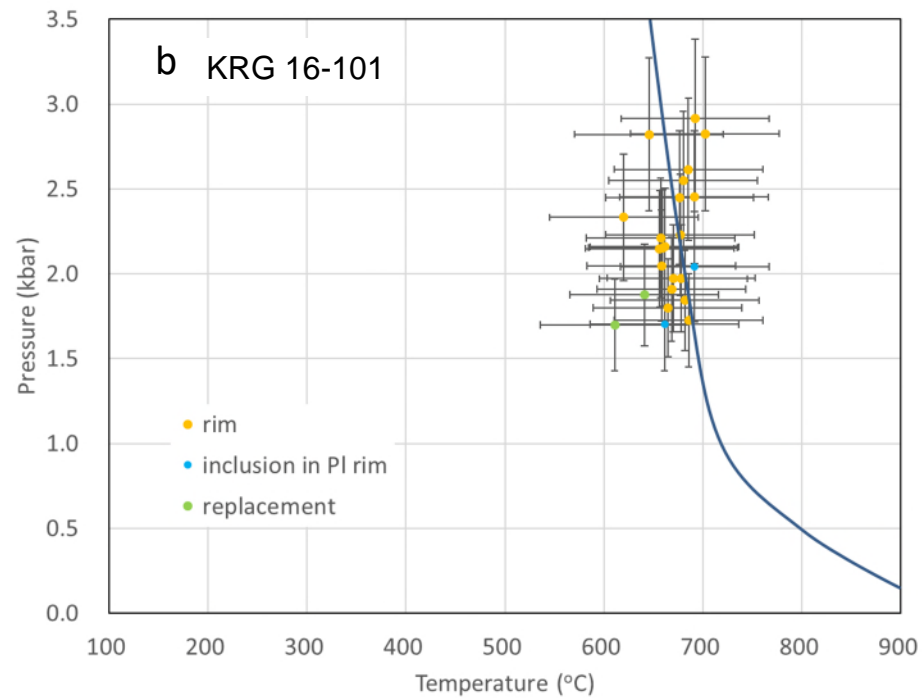
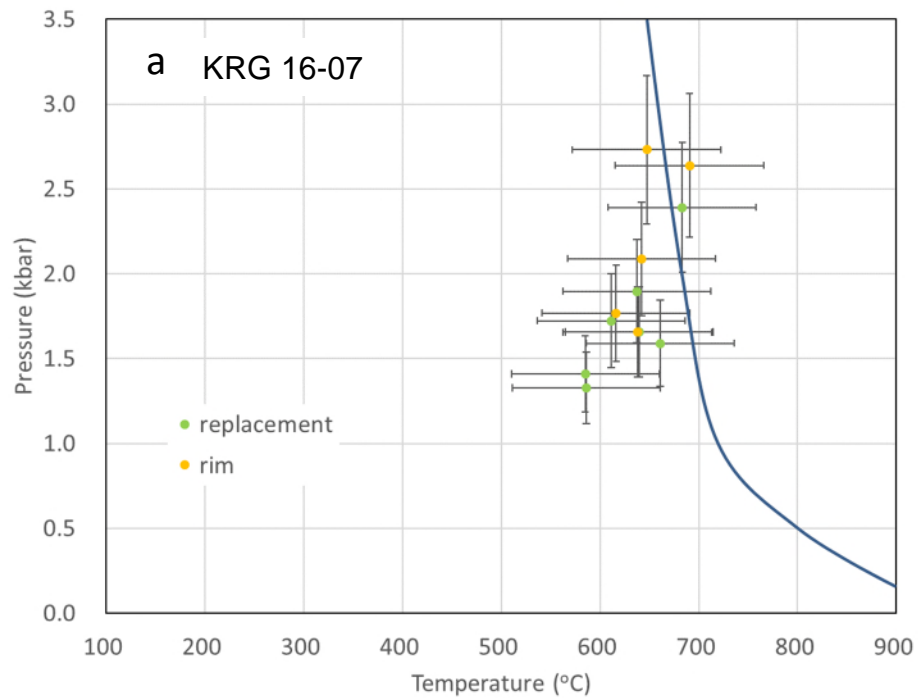


Fig. 7



<b>Laboratory</b>	JAEA- Toki Geochronology Research Laboratory
<b>Analyst</b>	S. Kagami, T. Yokoyama
<b>Laser ablation system</b>	
<b>Model</b>	Photon-Machines Analyte G2
<b>Laser type</b>	Excimer 193 nm
<b>Energy density</b>	2.0 J cm <sup>-2</sup>
<b>Crater size</b>	20 μm circle
<b>Repetition rate</b>	10 Hz (200 shots)
<b>Carrier gas</b>	He
<b>He gas flow rate</b>	1.0 L/min
<b>ICP-MS</b>	
<b>Model</b>	Thermo Fisher Scientific Neptune- <i>Plus</i>
<b>Forward power</b>	1200 W
<b>Carrier gas</b>	Ar
<b>Ar gas flow rate</b>	1.1 L/min
<b>Scanning mode</b>	Multi-collector Static
<b>Data acquisition protocol</b>	Time resolved analysis
<b>Integration time</b>	0.066s × 700 ratios
<b>Monitor isotopes</b>	<sup>202</sup> Hg (CDD), <sup>204</sup> Pb (CDD), <sup>206</sup> Pb (SEM), <sup>207</sup> Pb (SEM), <sup>208</sup> Pb (SEM), <sup>232</sup> Th (FC), <sup>238</sup> U (FC) *CDD: Compact Discrete Dynode, SEM: Secondary Electron Multiplier, FC: Faraday Cup
<b>Primary standard</b>	91500
<b>Secondary standard</b>	OD-3

Table S2a. Results of LA – ICP – MS U – Pb zircon dating with common Pb correction assuming initial equilibrium.

analysis no.	microtexture	$^{207}\text{Pb}/^{235}\text{U}$	error (1σ)	$^{206}\text{Pb}/^{238}\text{U}$	error (1σ)	Error correlation	$^{207}\text{Pb}/^{206}\text{Pb}$	error (1σ)	common $^{207}\text{Pb}/^{206}\text{Pb}$	error	$f_{\text{Th/U}}$	error (30%)	$f_{\text{Pa/U}}$	error (30%)	Common Pb corrected age (Ma)	error (1σ)	Th (ppm)	U (ppm)	Th/U	* excluded from weighted average
KRG16-101-1	unzoned mantle	8.40462	0.76642	0.07334	0.00608	0.90954	0.83109	0.03150	0.83594	0	1	0	1	0	456.3	36.6	n.d.	n.d.	n.d.	*
KRG16-101-2	oscillatory-zoned rim	0.00639	0.00106	0.00078	0.00005	0.41295	0.05964	0.00901	0.83594	0	1	0	1	0	4.9	0.3	629	739	0.85	
KRG16-101-3	oscillatory-zoned rim	0.00531	0.00085	0.00079	0.00007	0.53741	0.04874	0.00655	0.83594	0	1	0	1	0	5.1	0.4	600	893	0.67	
KRG16-101-4	oscillatory-zoned rim	0.00568	0.00096	0.00081	0.00006	0.44623	0.05104	0.00774	0.83594	0	1	0	1	0	5.2	0.4	391	674	0.58	
KRG16-101-5	unzoned mantle	0.00610	0.00153	0.00086	0.00006	0.29956	0.05130	0.01231	0.83594	0	1	0	1	0	5.5	0.4	269	379	0.71	
KRG16-101-6	oscillatory-zoned rim	0.02174	0.00997	0.00326	0.00146	0.97558	0.04835	0.00487	0.83594	0	1	0	1	0	20.9	6.5	n.d.	n.d.	n.d.	*
KRG16-101-7	unzoned mantle	0.00597	0.00089	0.00080	0.00006	0.48540	0.05444	0.00707	0.83594	0	1	0	1	0	5.1	0.3	1453	1132	1.28	
KRG16-101-8	unzoned mantle	25.41318	6.34566	0.21903	0.05350	0.97819	0.84149	0.04364	0.83594	0	1	0	1	0	1276.8	289.3	n.d.	n.d.	n.d.	*
KRG16-101-9	oscillatory-zoned rim	0.00515	0.00070	0.00078	0.00008	0.75091	0.04759	0.00427	0.83594	0	1	0	1	0	5.0	0.5	1064	1846	0.58	
KRG16-101-10	unzoned	9.41347	1.10182	0.08136	0.00890	0.93451	0.83913	0.03496	0.83594	0	1	0	1	0	504.2	53.3	n.d.	n.d.	n.d.	*
KRG16-101-11	oscillatory-zoned rim	0.00973	0.00353	0.00089	0.00010	0.32086	0.07917	0.02724	0.83594	0	1	0	1	0	5.5	0.6	404	949	0.43	
KRG16-101-12	oscillatory-zoned rim	0.00601	0.00177	0.00084	0.00008	0.32705	0.05166	0.01438	0.83594	0	1	0	1	0	5.4	0.5	169	327	0.52	
KRG16-101-13	oscillatory-zoned rim	0.00702	0.00222	0.00087	0.00011	0.38820	0.05825	0.01693	0.83594	0	1	0	1	0	5.5	0.6	221	343	0.64	
KRG16-101-14	oscillatory-zoned rim	0.00575	0.00129	0.00082	0.00006	0.34910	0.05082	0.01064	0.83594	0	1	0	1	0	5.3	0.4	317	541	0.59	
KRG16-101-15	oscillatory-zoned rim	0.00523	0.00230	0.00079	0.00007	0.20054	0.04816	0.02073	0.83594	0	1	0	1	0	5.1	0.4	203	221	0.92	
KRG16-101-16	oscillatory-zoned rim	0.00659	0.00165	0.00085	0.00007	0.31936	0.05597	0.01331	0.83594	0	1	0	1	0	5.4	0.4	296	426	0.69	
KRG16-101-17	oscillatory-zoned rim	0.00682	0.00164	0.00082	0.00007	0.35975	0.05997	0.01344	0.83594	0	1	0	1	0	5.2	0.4	321	420	0.76	
KRG16-101-18	oscillatory-zoned rim	0.00811	0.00257	0.00086	0.00008	0.28748	0.06846	0.02080	0.83594	0	1	0	1	0	5.4	0.5	204	256	0.80	
KRG16-101-19	oscillatory-zoned rim, crack	0.00849	0.00317	0.00083	0.00008	0.24292	0.07388	0.02676	0.83594	0	1	0	1	0	5.2	0.5	206	190	1.08	*
KRG16-101-20	oscillatory-zoned rim, crack	0.00654	0.00146	0.00083	0.00006	0.32817	0.05749	0.01211	0.83594	0	1	0	1	0	5.2	0.4	341	461	0.74	*
KRG16-101-21	oscillatory-zoned rim	0.00668	0.00259	0.00083	0.00007	0.21654	0.05806	0.02196	0.83594	0	1	0	1	0	5.3	0.4	116	224	0.52	
KRG16-101-22	oscillatory-zoned rim	0.00558	0.00066	0.00078	0.00004	0.45816	0.05208	0.00550	0.83594	0	1	0	1	0	5.0	0.3	879	1840	0.48	
KRG16-101-23	unzoned rim	0.01410	0.00574	0.00092	0.00007	0.17825	0.11140	0.04459	0.83594	0	1	0	1	0	5.4	0.5	124	254	0.49	
KRG16-101-24	oscillatory-zoned rim	0.00573	0.00151	0.00078	0.00007	0.33107	0.05362	0.01333	0.83594	0	1	0	1	0	4.9	0.4	275	477	0.58	
KRG16-101-25	unzoned	4.92068	0.77426	0.04267	0.00642	0.95639	0.83631	0.03844	0.83594	0	1	0	1	0	269.4	39.8	n.d.	n.d.	n.d.	*
KRG16-101-26	unzoned	8.65497	1.09942	0.07489	0.00879	0.92402	0.83815	0.04071	0.83594	0	1	0	1	0	465.6	52.9	n.d.	n.d.	n.d.	*
KRG16-101-27	unzoned	7.94481	1.06792	0.06892	0.00861	0.92955	0.83603	0.04143	0.83594	0	1	0	1	0	429.7	52.2	n.d.	n.d.	n.d.	*
KRG16-101-28	unzoned	8.76559	1.69260	0.07486	0.01396	0.96585	0.84928	0.04249	0.83594	0	1	0	1	0	465.3	84.3	n.d.	n.d.	n.d.	*
KRG16-101-29	oscillatory-zoned rim	0.00812	0.00132	0.00087	0.00010	0.73813	0.06786	0.00746	0.83594	0	1	0	1	0	5.4	0.6	596	1076	0.55	
KRG16-101-30	oscillatory-zoned rim	0.00669	0.00276	0.00092	0.00009	0.22815	0.05299	0.02129	0.83594	0	1	0	1	0	5.9	0.5	114	195	0.58	
KRG16-101-31	oscillatory-zoned rim	0.03772	0.01384	0.00105	0.00013	0.33810	0.26046	0.08995	0.83594	0	1	0	1	0	4.9	0.9	555	565	0.98	
KRG16-101-32	oscillatory-zoned rim	0.00620	0.00139	0.00081	0.00005	0.28156	0.05527	0.01186	0.83594	0	1	0	1	0	5.2	0.3	340	566	0.60	
KRG16-101-33	unzoned	6.85524	0.64235	0.05887	0.00498	0.90367	0.84459	0.03389	0.83594	0	1	0	1	0	368.7	30.4	n.d.	n.d.	n.d.	*
KRG16-101-34	unzoned, crack	9.95498	1.52518	0.08506	0.01234	0.94689	0.84878	0.04181	0.83594	0	1	0	1	0	526.3	73.7	n.d.	n.d.	n.d.	*
KRG16-101-35	oscillatory-zoned rim	0.00581	0.00106	0.00082	0.00008	0.53712	0.05157	0.00797	0.83594	0	1	0	1	0	5.2	0.5	569	896	0.63	
KRG16-101-36	oscillatory-zoned rim	0.00613	0.00383	0.00081	0.00008	0.14815	0.05458	0.03377	0.83594	0	1	0	1	0	5.2	0.5	82	147	0.56	

n.d.: not determined

Table S2b. Results of LA – ICP – MS U – Pb zircon dating with common Pb correction assuming initial disequilibrium.

analysis no.	microtexture	$^{207}\text{Pb}/^{235}\text{U}$	error (1 $\sigma$ )	$^{206}\text{Pb}/^{238}\text{U}$	error (1 $\sigma$ )	Error correlation	$^{207}\text{Pb}/^{206}\text{Pb}$	error (1 $\sigma$ )	common $^{207}\text{Pb}/^{206}\text{Pb}$	error	$f_{\text{Th/U}}$	error (30%)	$f_{\text{Pb/U}}$	error (30%)	Disequilibrium & Common Pb corrected age (Ma)	error (1 $\sigma$ )	Th (ppm)	U (ppm)	Th/U	* excluded from weighted average
KRG16-101-1	unzoned mantle	8.40462	0.76642	0.07334	0.00608	0.90954	0.83109	0.03150	0.83594	0	0.122271	0.036681	3.36	1.008	456.4	36.6	n.d.	n.d.	n.d.	*
KRG16-101-2	oscillatory-zoned rim	0.00639	0.00106	0.00078	0.00005	0.41295	0.05964	0.00901	0.83594	0	0.122271	0.036681	3.36	1.008	5.0	0.3	629	739	0.85	
KRG16-101-3	oscillatory-zoned rim	0.00531	0.00085	0.00079	0.00007	0.53741	0.04874	0.00655	0.83594	0	0.122271	0.036681	3.36	1.008	5.2	0.4	600	893	0.67	
KRG16-101-4	oscillatory-zoned rim	0.00568	0.00096	0.00081	0.00006	0.44623	0.05104	0.00774	0.83594	0	0.122271	0.036681	3.36	1.008	5.3	0.4	391	674	0.58	
KRG16-101-5	unzoned mantle	0.00610	0.00153	0.00086	0.00006	0.29956	0.05130	0.01231	0.83594	0	0.122271	0.036681	3.36	1.008	5.6	0.4	269	379	0.71	
KRG16-101-6	oscillatory-zoned rim	0.02174	0.00997	0.00326	0.00146	0.97558	0.04835	0.00487	0.83594	0	0.122271	0.036681	3.36	1.008	21.0	6.5	n.d.	n.d.	n.d.	*
KRG16-101-7	unzoned mantle	0.00597	0.00089	0.00080	0.00006	0.48540	0.05444	0.00707	0.83594	0	0.122271	0.036681	3.36	1.008	5.2	0.3	1453	1132	1.28	
KRG16-101-8	unzoned mantle	25.41318	6.34566	0.21903	0.05350	0.97819	0.84149	0.04364	0.83594	0	0.122271	0.036681	3.36	1.008	1276.9	289.3	n.d.	n.d.	n.d.	*
KRG16-101-9	oscillatory-zoned rim	0.00515	0.00070	0.00078	0.00008	0.75091	0.04759	0.00427	0.83594	0	0.122271	0.036681	3.36	1.008	5.2	0.5	1064	1846	0.58	
KRG16-101-10	unzoned	9.41347	1.10182	0.08136	0.00890	0.93451	0.83913	0.03496	0.83594	0	0.122271	0.036681	3.36	1.008	504.3	53.3	n.d.	n.d.	n.d.	*
KRG16-101-11	oscillatory-zoned rim	0.00973	0.00353	0.00089	0.00010	0.32086	0.07917	0.02724	0.83594	0	0.122271	0.036681	3.36	1.008	5.6	0.6	404	949	0.43	
KRG16-101-12	oscillatory-zoned rim	0.00601	0.00177	0.00084	0.00008	0.32705	0.05166	0.01438	0.83594	0	0.122271	0.036681	3.36	1.008	5.5	0.5	169	327	0.52	
KRG16-101-13	oscillatory-zoned rim	0.00702	0.00222	0.00087	0.00011	0.38820	0.05825	0.01693	0.83594	0	0.122271	0.036681	3.36	1.008	5.7	0.6	221	343	0.64	
KRG16-101-14	oscillatory-zoned rim	0.00575	0.00129	0.00082	0.00006	0.34910	0.05082	0.01064	0.83594	0	0.122271	0.036681	3.36	1.008	5.4	0.4	317	541	0.59	
KRG16-101-15	oscillatory-zoned rim	0.00523	0.00230	0.00079	0.00007	0.20054	0.04816	0.02073	0.83594	0	0.122271	0.036681	3.36	1.008	5.2	0.4	203	221	0.92	
KRG16-101-16	oscillatory-zoned rim	0.00659	0.00165	0.00085	0.00007	0.31936	0.05597	0.01331	0.83594	0	0.122271	0.036681	3.36	1.008	5.5	0.4	296	426	0.69	
KRG16-101-17	oscillatory-zoned rim	0.00682	0.00164	0.00082	0.00007	0.35975	0.05997	0.01344	0.83594	0	0.122271	0.036681	3.36	1.008	5.3	0.4	321	420	0.76	
KRG16-101-18	oscillatory-zoned rim	0.00811	0.00257	0.00086	0.00008	0.28748	0.06846	0.02080	0.83594	0	0.122271	0.036681	3.36	1.008	5.5	0.5	204	256	0.80	
KRG16-101-19	oscillatory-zoned rim, crack	0.00849	0.00317	0.00083	0.00008	0.24292	0.07388	0.02676	0.83594	0	0.122271	0.036681	3.36	1.008	5.3	0.5	206	190	1.08	*
KRG16-101-20	oscillatory-zoned rim, crack	0.00654	0.00146	0.00083	0.00006	0.32817	0.05749	0.01211	0.83594	0	0.122271	0.036681	3.36	1.008	5.3	0.4	341	461	0.74	*
KRG16-101-21	oscillatory-zoned rim	0.00668	0.00259	0.00083	0.00007	0.21654	0.05806	0.02196	0.83594	0	0.122271	0.036681	3.36	1.008	5.4	0.4	116	224	0.52	
KRG16-101-22	oscillatory-zoned rim	0.00558	0.00066	0.00078	0.00004	0.45816	0.05208	0.00550	0.83594	0	0.122271	0.036681	3.36	1.008	5.1	0.3	879	1840	0.48	
KRG16-101-23	unzoned rim	0.01410	0.00574	0.00092	0.00007	0.17825	0.11140	0.04459	0.83594	0	0.122271	0.036681	3.36	1.008	5.5	0.5	124	254	0.49	
KRG16-101-24	oscillatory-zoned rim	0.00573	0.00151	0.00078	0.00007	0.33107	0.05362	0.01333	0.83594	0	0.122271	0.036681	3.36	1.008	5.1	0.4	275	477	0.58	
KRG16-101-25	unzoned	4.92068	0.77426	0.04267	0.00642	0.95639	0.83631	0.03844	0.83594	0	0.122271	0.036681	3.36	1.008	269.5	39.8	n.d.	n.d.	n.d.	*
KRG16-101-26	unzoned	8.65497	1.09942	0.07489	0.00879	0.92402	0.83815	0.04071	0.83594	0	0.122271	0.036681	3.36	1.008	465.7	52.9	n.d.	n.d.	n.d.	*
KRG16-101-27	unzoned	7.94481	1.06792	0.06892	0.00861	0.92955	0.83603	0.04143	0.83594	0	0.122271	0.036681	3.36	1.008	429.8	52.2	n.d.	n.d.	n.d.	*
KRG16-101-28	unzoned	8.76559	1.69260	0.07486	0.01396	0.96585	0.84928	0.04249	0.83594	0	0.122271	0.036681	3.36	1.008	465.4	84.3	n.d.	n.d.	n.d.	*
KRG16-101-29	oscillatory-zoned rim	0.00812	0.00132	0.00087	0.00010	0.73813	0.06786	0.00746	0.83594	0	0.122271	0.036681	3.36	1.008	5.5	0.6	596	1076	0.55	
KRG16-101-30	oscillatory-zoned rim	0.00669	0.00276	0.00092	0.00009	0.22815	0.05299	0.02129	0.83594	0	0.122271	0.036681	3.36	1.008	6.0	0.5	114	195	0.58	
KRG16-101-31	oscillatory-zoned rim	0.03772	0.01384	0.00105	0.00013	0.33810	0.26046	0.08995	0.83594	0	0.122271	0.036681	3.36	1.008	5.0	0.9	555	565	0.98	
KRG16-101-32	oscillatory-zoned rim	0.00620	0.00139	0.00081	0.00005	0.28156	0.05527	0.01186	0.83594	0	0.122271	0.036681	3.36	1.008	5.3	0.3	340	566	0.60	
KRG16-101-33	unzoned	6.85524	0.64235	0.05887	0.00498	0.90367	0.84459	0.03389	0.83594	0	0.122271	0.036681	3.36	1.008	368.8	30.4	n.d.	n.d.	n.d.	*
KRG16-101-34	unzoned, crack	9.95498	1.52518	0.08506	0.01234	0.94689	0.84878	0.04181	0.83594	0	0.122271	0.036681	3.36	1.008	526.4	73.7	n.d.	n.d.	n.d.	*
KRG16-101-35	oscillatory-zoned rim	0.00581	0.00106	0.00082	0.00008	0.53712	0.05157	0.00797	0.83594	0	0.122271	0.036681	3.36	1.008	5.3	0.5	569	896	0.63	
KRG16-101-36	oscillatory-zoned rim	0.00613	0.00383	0.00081	0.00008	0.14815	0.05458	0.03377	0.83594	0	0.122271	0.036681	3.36	1.008	5.3	0.5	82	147	0.56	

n.d. : not determined