Geochemical Journal, Vol. 52, pp. 281 to 286, 2018 doi:10.2343/geochemj.2.0508

NOTE

A practical method for calculating the U-Pb age of Quaternary zircon: Correction for common Pb and initial disequilibria

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(*Received July 26, 2017; Accepted October 29, 2017*)

To obtain accurate crystallization ages from Quaternary zircon, it is necessary to consider the contributions of common Pb and initial disequilibria caused by Th/U and Pa/U fractionation in the zircon-melt system. The disequilibrium in the melt also has influence on the resulting age. This study presents a method for calculating corrected U-Pb ages of Quaternary zircon. In the proposed method, initial disequilibria and common Pb are comprehensively accounted for by a single correction based on a modified Tera-Wasserburg concordia diagram (modified ^{207}Pb method). A bisection method is used to quantitatively evaluate the influence of all factors employed in the correction on the resulting U-Pb age and its uncertainties. If the contribution of Pa/U partitioning is ignored (i.e., when using the conventional ^{207}Pb method), the systematic age error for a 100 ka zircon is ~9.4%. Melt disequilibria on ^{230}Th and ^{231}Pa also have significant influence on the calculated age, when it is non-negligible (e.g., basaltic rocks). Consequently, we suggest that the dating of very young zircon (<100 ka) with the ^{207}Pb method requires precise estimation of both Th/U and Pa/U partitioning in the zircon-melt system.

Keywords: zircon U-Pb dating, common Pb correction, initial disequilibria, Quaternary zircon, melt disequilibria

INTRODUCTION

Zircon U-Pb dating for the Quaternary period is an important method for understanding the high-temperature cooling history of magmas and constraining the ages of tephras as important chronostratigraphic marker horizons (Schmitt, 2011). Laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) has evolved to one of the most distributed techniques for U-Pb dating, and is complemented by secondary ion mass spectrometry (SIMS) and thermal ionization mass spectrometry (TIMS) which have also found applications in Quaternary geochronology (Schaltegger *et al*., 2015). In dating of young zircon by LA-ICP-MS, there are some technical challenges, but recent technological advances in LA-ICP-MS now allow us to date Quaternary zircons (e.g., Sakata *et al*., 2014). Independent of the analysis method, to obtain accurate crystallization ages from young zircon, it is necessary to correct for the effect of initial disequilibria caused by intermediate nuclides in the ^{238}U and ^{235}U decay series (i.e., ^{230}Th and ^{231}Pa) (e.g., Wendt and Carl, 1985). To correct for this effect, estimating the magnitude of disequilibria from Th/U and Pa/U partitioning in zircon-melt system is common approach (Schmitt, 2011). Furthermore, in the case of dating young zircon, contributions from non-radiogenic Pb (common Pb) to the U-Pb age are non-negligible. There are several approaches to the common Pb correction, but the ^{207}Pb method (Williams, 1998) has some important advantages over other methods when applied to young zircon dating. This method does not depend on the measurement of ^{204}Pb, which occurs in minor isotopic abundances and suffers from isobaric interference from ^{204}Hg (LA-ICP-MS), and possibly HfC_2 (SIMS). Although the ^{207}Pb method assumes concordance between ^{238}U-^{206}Pb and ^{235}U-^{207}Pb ages, this assumption is reasonable for young zircon because Quaternary zircon crystals are simply too young to have undergone enough radiation damage to cause Pb mobility (Cherniak and Watson, 2001), unless some very unrealistic (e.g., high-U zircon) scenarios are invoked.

If the effect of initial disequilibria on ^{230}Th and ^{231}Pa is taken into account, the equations of ^{238}U-^{206}Pb and ^{235}U-^{207}Pb ages are more complicated than in the case of conventional U-Pb geochronology (Wendt and Carl, 1985). Furthermore, in the case of young zircon dating with the ^{207}Pb method, initial disequilibria and common Pb are interdependent and require an internally consistent correction. Therefore, the equation for the ^{207}Pb method

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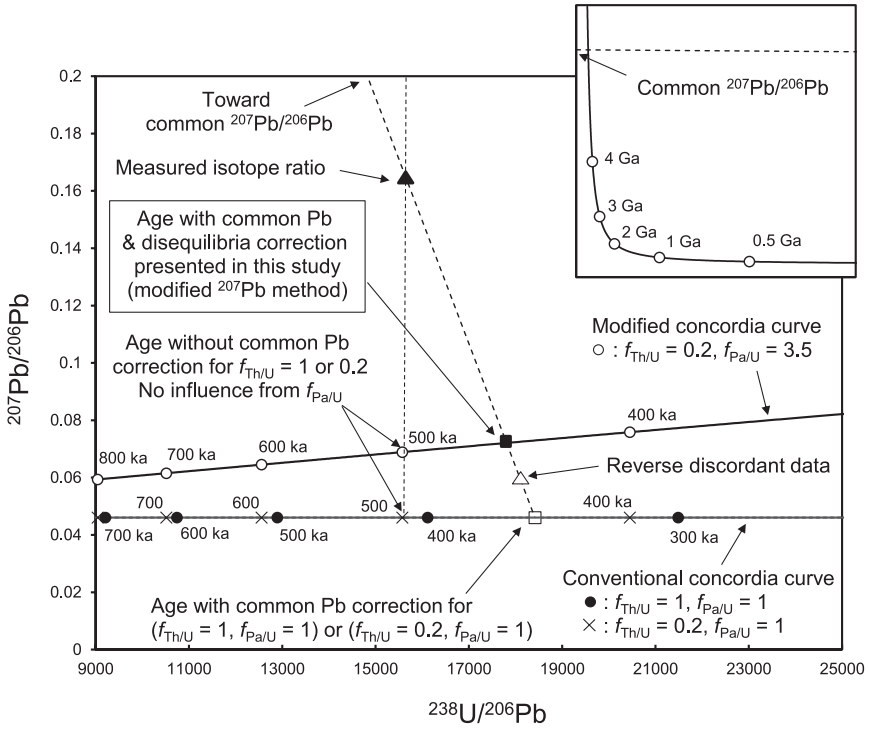

Fig. 1. Schematic diagram of the proposed corrections for the initial disequilibria effect on ^{230}Th and ^{231}Pa, and for common Pb.

(Williams, 1998) must be modified for Quaternary zircon dating. This study presents a practical method for calculating the ages of young zircon based on a new correction for common Pb and initial disequilibria which is termed the modified ^{207}Pb method.

Method for Common Pb and Disequilibria Correction

In young zircon dating, if disequilibria in melt associated with ^{230}Th and ^{231}Pa are reasonably neglected, the equations for calculating a U-Pb age can be simplified as follows (Sakata *et al*., 2017):

$$\left(\frac{^{206}\mathrm{Pb}^{*}_{\mathrm{Zircon}}}{^{238}\mathrm{U}_{\mathrm{Zircon}}}\right) = \left(e^{\lambda_{238}t}-1\right)+\frac{\lambda_{238}}{\lambda_{230}}\left(f_{\mathrm{Th/U}}-1\right)\left(1-e^{-\lambda_{230}t}\right)e^{\lambda_{238}t} \quad (1)$$

$$\left(\frac{^{207}\mathrm{Pb}^{*}_{\mathrm{Zircon}}}{^{235}\mathrm{U}_{\mathrm{Zircon}}}\right) = \left(e^{\lambda_{235}t}-1\right)+\frac{\lambda_{235}}{\lambda_{231}}\left(f_{\mathrm{Pa/U}}-1\right)\left(1-e^{-\lambda_{231}t}\right)e^{\lambda_{235}t} \quad (2)$$

where λ represents the respective decay constant and t the crystallization age of zircon. In Eqs. (1) and (2), $f_{\mathrm{Th/U}}$ and $f_{\mathrm{Pa/U}}$ are the fractionation factors of Th/U and Pa/U in a zircon-melt system, respectively, defined as follows:

$$f_{\mathrm{Th/U}} = \frac{\left(\mathrm{Th/U}\right)_{\mathrm{Zircon}}}{\left(\mathrm{Th/U}\right)_{\mathrm{Melt}}} \quad (3)$$

$$f_{\mathrm{Pa/U}} = \frac{\left(\mathrm{Pa/U}\right)_{\mathrm{Zircon}}}{\left(\mathrm{Pa/U}\right)_{\mathrm{Melt}}} \quad (4)$$

where $(\mathrm{Th/U})_{\mathrm{Zircon}}$ and $(\mathrm{Th/U})_{\mathrm{Melt}}$ are the ratios at the time of zircon crystallization. In the case of Quaternary zircon, $(\mathrm{Th/U})_{\mathrm{Zircon}}$ can be approximated by the present-day Th/U, which can be directly measured from the target zircons. The Th/U ratio of the melt can be estimated from that of the bulk rock (e.g., Reid *et al*., 1997), melt inclusions in host rocks (e.g., Schmitt *et al*., 2003), glass in volcanic ash (Matthews *et al*., 2015), or by comparing U-Pb and Th-Pb ages (e.g., Sakata *et al*., 2017). Although determining the Pa/U ratio in zircon and melt is difficult because of the low concentrations of Pa, several studies directly and indirectly determined a Pa/U partitioning factor ($f_{\mathrm{Pa/U}}$) between melt and zircon from volcanic rocks (2-sigma errors): $f_{\mathrm{Pa/U}}$ = 0.9–2.2 of Salton Buttes rhyolite

(Schmitt, 2007), $f_{Pa/U}$ = 3.8 ± 0.8 of China Hat rhyolite (Blackfoot Volcanic Field, Idaho) by Schmitt (2011), $f_{Pa/U}$ = 3.66 ± 0.89 of Kirigamine rhyolite (Wadatouge Pass, Nagano Prefecture, Japan), $f_{Pa/U}$ = 3.1 ± 1.2 of Bishop tuff (Inyo County, California), $f_{Pa/U}$ = 3.04 ± 0.99 of Toga pumice (Oga Peninsula, NE Japan) by Sakata *et al*. (2017), and $f_{Pa/U}$ = 2.2–3.8 for mid-ocean ridge zircons (Rioux *et al*., 2015). The available $f_{Pa/U}$ values roughly show agreement within uncertainties and their weighted mean is $f_{Pa/U}$ = 2.9 ± 1.0 (2σ, MSWD = 5.1, n = 6). Given that direct measurement of Pa/U in host rock is not conducted in Schmitt (2007) and the calculated MSWD value is relatively large, better estimation of $f_{Pa/U}$ in natural zircon can be calculated using the other five values except for that by Schmitt (2007): $f_{Pa/U}$ = 3.36 ± 0.40 (2σ, MSWD = 0.77, n = 5).

In the conventional ^{207}Pb-based method for the common Pb correction, it is assumed that the deviation of one data point from the concordia curve in a Tera-Wasserburg concordia diagram can be explained by the contribution from common Pb. However, a modified concordia curve must be defined in the case of Quaternary samples to account for the initial disequilibria effect (Wendt and Carl, 1985). A modified concordia curve can be defined using the fractionation factors $f_{Th/U}$ and $f_{Pa/U}$, if other short-lived intermediate products in the ^{238}U and ^{235}U decay series are reasonably ignored based on their negligible effects on Pb accumulation (Wendt and Carl, 1985). A U-Pb age of Quaternary zircon corrected for initial disequilibria and common Pb corresponds to a lower intercept between the modified concordia curve and the line defined by common Pb composition and measured isotope ratios (Fig. 1). Therefore, the age of Quaternary zircon can be calculated using the following equation:

$$F(t)=\frac{\left(1/R^{U}\right)\left[\left(e^{\lambda_{235}t}-1\right)+\frac{\lambda_{235}}{\lambda_{231}}\left(f_{Pa/U}-1\right)\left(1-e^{-\lambda_{231}t}\right)e^{\lambda_{235}t}\right]-\left(R_m^{76}-R_c^{76}\right)/R_m^{86}}{\left(e^{\lambda_{238}t}-1\right)+\frac{\lambda_{238}}{\lambda_{230}}\left(f_{Th/U}-1\right)\left(1-e^{-\lambda_{230}t}\right)e^{\lambda_{238}t}}-R_c^{76} \quad (5)$$

where R^{U}, R^{76}, and R^{86} represent the ^{238}U/^{235}U, ^{207}Pb/^{206}Pb, and ^{238}U/^{206}Pb ratios in zircon, respectively. Here, "m" and "c" denote measured values and the common Pb composition, respectively. The R^{U} can be assumed to be constant, given that the analytical uncertainty for most methods exceeds the heterogeneity of ^{238}U/^{235}U in natural zircon crystals (e.g., ^{238}U/^{235}U = 137.82, Hiess *et al*., 2012). The isotopic composition of common Pb in magma can be estimated either by measuring the bulk rock or Pb-bearing minerals from the host rock (e.g., K-feldspar), or by predicting the isotopic evolution of terrestrial Pb in the continental crust using Stacey and Kramers' (1975) two-stage model. When Eq. (5) is equivalent to zero, t is equivalent to the zircon crystallization age. In this study, t and its uncertainty are determined by the bisection method. For details, actual procedure of the calculation is described in Supplementary Materials, and, if necessary, a Microsoft Excel spreadsheet for this calculation is available (please contact author by e-mail: shuhei.sakata@gakushuin.ac.jp).

Quantitative Evaluation of the Modified ^{207}Pb Method

In this section, the potential change of the final age calculated by the modified ^{207}Pb method is discussed. If only ^{230}Th-disequilibrium correction is conducted, the resulting age is older than that by the modified ^{207}Pb method by *ca*. 40 kyrs in the case described in Fig. 1. Age calculation is also influenced associated with magnitudes of initial disequilibria and isotopic composition of common Pb. Figure 2 shows how the corrected age changes depend on $f_{Th/U}$, $f_{Pa/U}$, and $R^{76}{}_{c}$, compared with hypothetical zircon assumed to have crystallized in typical silicic magma with 20% common ^{206}Pb (i.e., $f_{Th/U}$ = 0.2 and $f_{Pa/U}$ = 3.5, f_{206} = 0.2; f_{206} = $^{206}Pb_{common}$/$^{206}Pb_{total}$). In the case of $f_{Th/U}$ and $f_{Pa/U}$ (Figs. 2(a) and (b)), younger zircon analyses are more severely affected by uncertainties of these factors than results for older zircon. If these fractionation factors have 20% relative uncertainty for 100 ka zircon, the age deviations are >4.2% and >2.6%, respectively. The conventional ^{207}Pb method only considers Th/U fractionation (i.e., $f_{Pa/U}$ is assumed to be unity). If this method is used, the systematic errors for zircon with ages of 1000 and 100 ka are ~0.7% and 9.4%, respectively. This suggests that great care must be taken regarding not only Th/U partitioning in the zircon-melt system but also Pa/U partitioning, if the ^{207}Pb-based correction is applied to very young zircons (<100 ka). On the other hand, older zircon is more sensitive to uncertainties in $R^{76}{}_{c}$. Although temporal changes in ^{207}Pb/^{206}Pb in continental crust is less than 1‰ throughout the Quaternary period according to the Stacey and Kramers' (1975) model, the ^{207}Pb/^{206}Pb evolution of the source reservoir, from which zircon-crystallizing evolved melts are generated, generally depends on the extent and timing of U/Pb chemical fractionation. Moreover, depletion or enrichment of U/Pb in the source reservoir could cause sys-

tematic changes in $^{207}Pb/^{206}Pb$ of melt. Hence, common $^{207}Pb/^{206}Pb$ in melt should be estimated carefully, taking account of the geological and geochemical characteristics of the study area.

In the discussion so far, the assumption that $^{230}Th/^{238}U$ and $^{231}Pa/^{235}U$ reached secular equilibrium in the melt from which zircon crystallized is employed. The melt disequilibria are reasonably neglected in the case of silicic melt (Condomines and Sigmarsson, 1993; Simon *et al*., 2008). However, significant $^{230}Th/^{238}U$ and $^{231}Pa/^{235}U$ disequilibria have been detected in basaltic rocks (e.g., Avanzinelli *et al*., 2012). In the case of the melt being in disequilibrium at the time of zircon crystallization, isotopic evolutions of radiogenic $^{206}Pb^*$ and $^{207}Pb^*$ can be expressed as follows (Sakata *et al*., 2017):

$$\left(\frac{^{206}Pb^{*}_{Zircon}}{^{238}U_{Zircon}}\right)=e^{\lambda_{238}t}-1+\frac{\lambda_{238}}{\lambda_{230}}\left[f_{Th/U}\left\{1+e^{(\lambda_{238}-\lambda_{230})T}\left(f_{Th/U}(p.m.)-1\right)\right\}-1\right]\left(1-e^{-\lambda_{230}t}\right)e^{\lambda_{238}t} \quad (6)$$

$$\left(\frac{^{207}Pb^{*}_{Zircon}}{^{235}U_{Zircon}}\right)=e^{\lambda_{235}t}-1+\frac{\lambda_{235}}{\lambda_{231}}\left[f_{Pa/U}\left\{1+e^{(\lambda_{235}-\lambda_{231})T}\left(f_{Pa/U}(p.m.)-1\right)\right\}-1\right]\left(1-e^{-\lambda_{231}t}\right)e^{\lambda_{235}t} \quad (7)$$

$$f_{Th/U}(p.m.)=\frac{D^{Th}_{Solid/Initial\ Melt}}{D^{U}_{Solid/Initial\ Melt}}=\frac{(Th/U)_{Solid}}{(Th/U)_{Initial\ Melt}} \quad (8)$$

$$f_{Pa/U}(p.m.)=\frac{D^{Pa}_{Solid/Initial\ Melt}}{D^{U}_{Solid/Initial\ Melt}}=\frac{(Pa/U)_{Solid}}{(Pa/U)_{Initial\ Melt}} \quad (9)$$

where, $f_{Th/U}(p.m.)$ ($f_{Pa/U}(p.m.)$) and $f_{Th/U}$ ($f_{Pa/U}$) are the partitioning factor of Th/U (Pa/U) at the time of partial melting and zircon crystallization, respectively, T is the pre-crystallization magma residence time (i.e., the time between partial melting and zircon crystallization), and t is the zircon crystallization age. The $(Th/U)_{Solid}$ ($(Pa/U)_{Solid}$) and $(Th/U)_{Initial\ Melt}$ ($(Pa/U)_{Initial\ Melt}$) are the respective Th/U (Pa/U) values of the melt source, such as crust, and the initial melt at the time of partial melting. The D represents distribution coefficient for Th, U, or Pa in melt source (solid)-initial melt system. The zircon crystallization age accounting for common Pb via ^{207}Pb, as well as disequilibrium effects from melting and crystallization processes, is calculated analogous to Eq. (5) from following equation.

$$G(t)=\frac{\left(1/R^{U}\right)\left[e^{\lambda_{235}t}-1+\frac{\lambda_{235}}{\lambda_{231}}\left[f_{Pa/U}\left\{1+e^{(\lambda_{235}-\lambda_{231})T}\left(f_{Pa/U}(p.m.)-1\right)\right\}-1\right]\left(1-e^{-\lambda_{231}t}\right)e^{\lambda_{235}t}\right]-\left(R^{76}_{m}-R^{76}_{c}\right)/R^{86}_{m}}{e^{\lambda_{238}t}-1+\frac{\lambda_{238}}{\lambda_{230}}\left[f_{Th/U}\left\{1+e^{(\lambda_{238}-\lambda_{230})T}\left(f_{Th/U}(p.m.)-1\right)\right\}-1\right]\left(1-e^{-\lambda_{230}t}\right)e^{\lambda_{238}t}}-R^{76}_{c}. \quad (10)$$

Based on Eq. (10), the contribution of the melt disequilibria to the modified ^{207}Pb method can be quantitatively estimated (Fig. 3) (see Supplementary Materials for detailed calculation). Generally, when T becomes longer, the melt disequilibria is weakened and the resulting age is closed to be that by Eq. (5). Depletion of $^{230}Th/^{238}U$ and enrichment of $^{231}Pa/^{235}U$, compared to the equilibrium values, in the melt at the time of zircon crystallization make the age older. The radioactivity ratio of young volcanic rocks is roughly distributed in range of 0.8–1.2 for $^{230}Th/^{238}U$ and 1–2 for $^{231}Pa/^{235}U$ (e.g., Avanzinelli *et al*., 2012; Condomines and Sigmarsson, 1993; Pickett and Murrell, 1997). If the zircon sample crystallized from the disequilibrium melt is measured, the potential systematic error of the modified ^{207}Pb method based on Eq. (5) could be ca. 10% at a maximum. Therefore, this correction method should be carefully used for the zircon crystallized from melts with high degrees of U-series disequilibrium.

Concluding Remarks

This study provides a useful method for calculating accurate U-Pb ages of Quaternary zircon. This technique includes corrections for the initial disequilibria effect on ^{230}Th and ^{231}Pa, and for common Pb. It should be noted that Pa/U partitioning in zircon-melt systems has a significant influence on the corrected age, if the zircon of interest is very young (<100 ka). Furthermore, the contribution from melt disequilibria on ^{230}Th and ^{231}Pa should be carefully estimated, especially in the case of measuring the zircon crystallized from melts with high degrees of U-series disequilibrium. If appropriate partitioning factors for Th/U and Pa/U are available, the modified

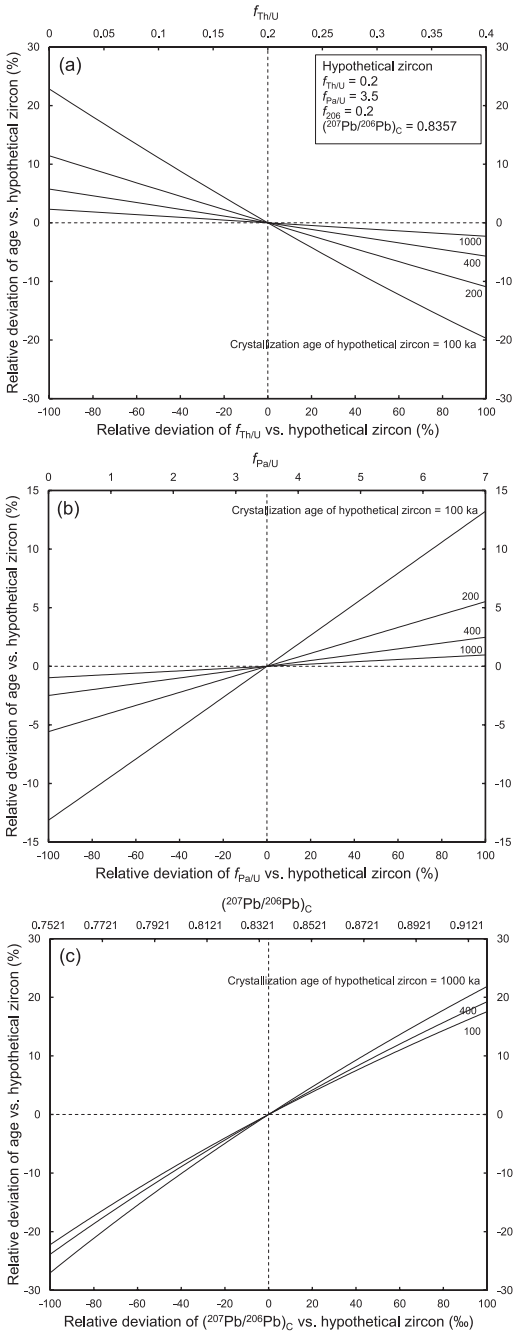

Fig. 2. Quantitative evaluation of the correction technique. The same hypothetical zircon is assumed in (a), (b), and (c). The f_{206} is defined as the fraction of common ^{206}Pb relative to the total ^{206}Pb. The $(^{207}Pb/^{206}Pb)_C$ is the isotopic composition of common Pb. Typical values in silicic magma are used to determine $f_{Th/U}$ and $f_{Pa/U}$ ($f_{Th/U}$ = 0.2 and $f_{Pa/U}$ = 3.5), and $(^{207}Pb/^{206}Pb)_C$ is calculated based on the two-stage model by Stacey and Kramers (1975) at the age of zero for a hypothetical zircon. Relative deviation (RD% and RD‰) is defined as follows: RD% = [(value)/(value)$_{hyp.}$ − 1] × 100, and RD‰ = [(value)/(value)$_{hyp.}$ − 1] × 1000, where "value" denotes the corrected U-Pb age, $f_{Th/U}$, $f_{Pa/U}$, or $(^{207}Pb/^{206}Pb)_C$. "hyp." denotes values for a hypothetical zircon.

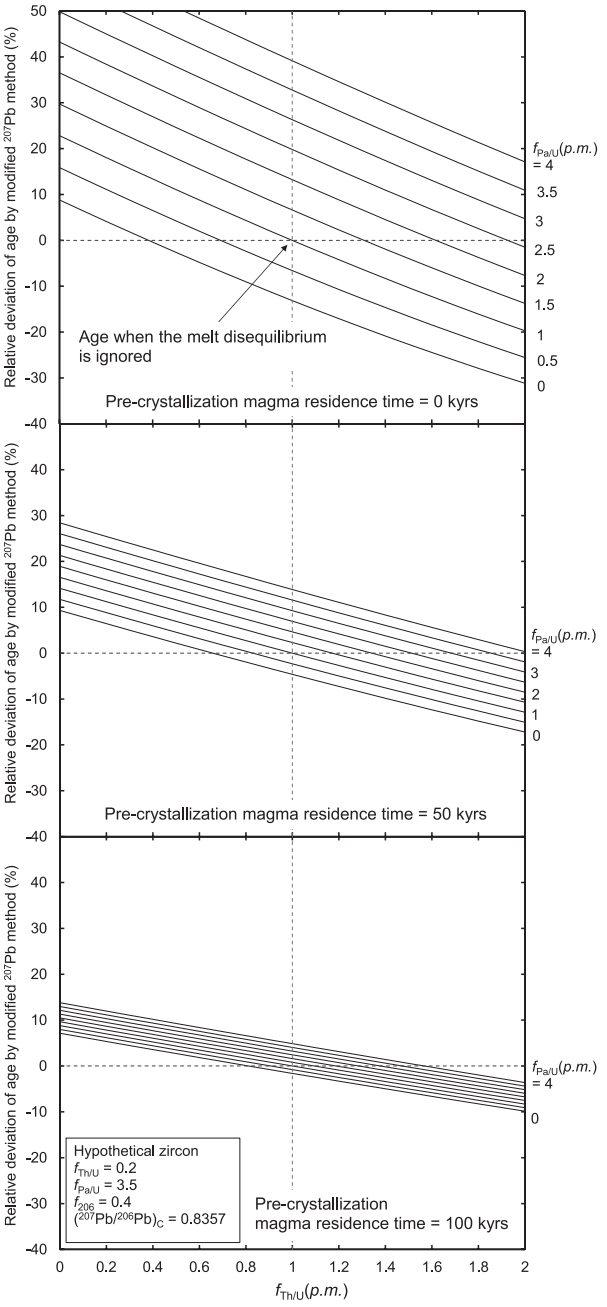

Fig. 3. Contribution of melt disequilibria to the age calculated by modified ^{207}Pb method. Pre-crystallization magma residence time is expressed by T, and it is defined as the time between partial melting and zircon crystallization. The same hypothetical zircon is assumed for three cases. Relative deviation (RD%) is defined as follows: RD% = [(age$_{Eq.(10)}$)/(age)$_{Eq.(5)}$ − 1] × 100, where "age$_{Eq.(10)}$" and "age$_{Eq.(5)}$" denote the respective U-Pb age calculated by Eqs. (10) and (5) using parameters of the hypothetical zircon. The $f_{Th/U}$(p.m.) and $f_{Pa/U}$(p.m.) represent the partitioning factor of Th/U and Pa/U in solid-liquid system at the time of partial melting, respectively.

^{207}Pb method could be useful for other U-bearing accessary minerals (e.g., monazite). The combination of this method with recent advances in analytical techniques enables investigations of complicated magma processes with a rigorous geochronometer.

Acknowledgments—Critical reviews by two reviewers greatly improved this manuscript, and editorial handling by Kentaro Terada is gratefully acknowledged. We thank Takeshi Ohno (Gakushuin University, Japan), Hideki Iwano and Tohru Danhara (Kyoto Fission-Track Co. Ltd., Japan), Shuji Matsu'ura (Ochanomizu University, Japan), Takafumi Hirata (The University of Tokyo, Japan) for scientific advice.

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Supplementary Materials

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