U-Pb dating of zircon in the Bishop Tuff at the millennial scale

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ABSTRACT

Zircon from the Bishop Tuff (eastern California) was dated by the U-Pb isotope dilution thermal ionization mass spectrometry (ID-TIMS) method to evaluate time scales of magmatic evolution and applicability of the method to rocks younger than 1 Ma. The ²⁰⁶Pb/²³⁸U dates from 17 of 19 grains are equivalent with a weighted mean of 767.1 \pm 0.9 ka that overlaps with the published ⁴⁰Ar/³⁹Ar sanidine age, requiring that most zircon crystallized immediately before eruption. Dates from two grains that are 6 and 12 k.y. older indicate minimum times that a small portion of the grains resided in the magma. These findings are consistent with chemical zoning patterns in zircon that suggest a relatively simple crystallization history, yet contrast with previous results of ion microprobe dating of zircon that show 50–80 k.y. of magma residence. The weighted mean ²⁰⁷Pb/²³⁵U date is ~52 k.y. older than the ²⁰⁶Pb/²³⁸U date, a difference that is attributed to excess ²⁰⁷Pb from initial Pa/U disequilibrium. The results demonstrate that high-precision ID-TIMS geochronology can resolve magma chamber dynamics of <1 Ma silicic eruptions at the millennial scale.

Keywords: zircon, U-Pb geochronology, Bishop Tuff, magma residence, disequilibrium.

INTRODUCTION

Time scales of crystallization and eruption are crucial for understanding large silicic magmatic systems. Eruption ages are commonly determined by ⁴⁰Ar/³⁹Ar dates from sanidine, which record the time at which radiogenic Ar begins to accumulate following eruption and cooling below magmatic temperatures. In contrast, U-Pb and U-series disequilibrium dates from zircon in volcanic rocks show that zircon crystallization can significantly predate eruption (Reid et al., 1997; Lowenstern et al., 2000; Schmitt et al., 2003; Miller and Wooden, 2004; Bacon and Lowenstern, 2005; Charlier et al., 2005; Simon and Reid, 2005). This phenomenon is due to the closure temperature for the diffusion of radiogenic Pb in zircon being higher than typical magmatic temperatures (>1000 °C), and thus zircon can preserve dates that reflect (1) inheritance from the source and/or country rocks, and (2) protracted crystallization in the magma before eruption, termed "magma residence".

The ion microprobe is the primary tool used to unravel the histories of Tertiary and younger magmatic systems through U-Pb and U-series disequilibrium dating of zircon, with the latter limited to grains younger than ca. 400 ka. It has high spatial resolution and yields errors of $\pm 5\%$ -10% (2 σ) on individual U-Pb analyses of

500–1000 ka zircon. U-Pb isotope dilution thermal ionization mass spectrometry (ID-TIMS) is applied to single zircon grains or parts of grains with precision that is approximately an order of magnitude better, but it has not been widely used on <1000 ka grains.

During about six days at ca. 770 ka, ~600 km3 of Bishop Tuff was erupted from the Long Valley Caldera in California (Wilson and Hildreth, 1997). Cataclysmic eruption of the tuff was the largest phase of ~35 eruptive units of high-silica rhyolite that began ~400 k.y. before the Bishop Tuff (Hildreth, 2004). Recharge of the magma chamber by intrusion of mafic magma was proposed as the trigger for the major eruption (Wark et al., 2007). We present ID-TIMS dates from Bishop Tuff zircon that were obtained with the goals of evaluating (1) how long zircon resided in the magma before eruption through a comparison with 40Ar/39Ar sanidine dates, and (2) whether ID-TIMS dates can improve understanding of magmatic processes.

ANALYTICAL METHODS

Bishop Tuff was collected from the Los Angeles Department of Water and Power upper powerhouse road that descends the west wall of Owens River gorge and exposes a 150 m thick section. The sample is from the dark gray, densely welded, eutaxitic zone that corresponds to the early part of the eruption. Nineteen zircon grains were dated as single crystals using ID-TIMS at the Massachusetts Institute of Technology (MIT) following Schoene et al. (2006), with a few differences described here. Internal zoning patterns in nine dated grains were revealed by cathodoluminescence (CL) and backscattered electron (BSE) imaging. Two dated grains and two other grains were analyzed for Zr, Si, Hf, Th, and U by electron microprobe at MIT. Fourteen grains were treated with the chemical abrasion method (Mattinson, 2005) by annealing for 60 h at 900 °C and leaching in HF acid at 180 °C for 12 h. All common Pb in the zircon analyses was assumed to be procedural blank (average is 0.54 pg; see Table DR1 in the GSA Data Repository1 for composition and error). Fifteen analyses used the EARTHTIME ²⁰⁵Pb-²³³U-²³⁵U tracer solution with the current working calibration and four used the MIT ²⁰⁵Pb-²³³U-²³⁵U tracer solution with the calibration discussed in Schoene et al. (2006). Errors were calculated with the algorithm of Schmitz and Schoene (2007) and are reported at 2σ . Weighted mean dates were calculated with ISOPLOT (Ludwig, 2003).

Most zircon has a deficit of ²⁰⁶Pb due to initial Th/U disequilibrium caused by exclusion of ²³⁰Th (Schärer, 1984; Parrish, 1990). Correction of ²⁰⁶Pb/²³⁸U dates for the deficit requires estimates of Th/U of zircon (Th/U[zircon]) and Th/U of the magma from which zircon crystallized

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¹GSA Data Repository item 2007278, U-Pb and compositional data from zircon from the Bishop Tuff, is available online at www.geosociety.org/pubs/ ft2007.htm, or on request from editing@geosociety. org or Documents Secretary, GSA, P.O. Box 9140, Boulder, CO 80301, USA.

(Th/U[magma]). Corrections are significant for young zircon, for example, increasing our dates from the Bishop Tuff by 11% (~89 k.y.). Because there are no previously published attempts to quantify the uncertainties associated with the correction and propagate them into errors on the dates, we have done so as described here and in equations in the GSA Data Repository. Th concentration in Th/U[zircon] was modeled based on the amount of ²⁰⁸Pb measured by ID-TIMS and assuming concordance between the ²⁰⁸Pb/²³²Th (not measured) and 206Pb/238U systems. U concentration in Th/U[zircon] was measured by ID-TIMS. The average error on Th/U[zircon] is ±0.01 (Table DR1). Th/U[magma] is more difficult to estimate and has a larger effect on dates. We used published ion microprobe measurements of melt inclusions in quartz phenocrysts that are uniform for the entire Bishop Tuff eruptive cycle with Th/U of 2.81 \pm 0.32 (2 σ) for 12 analyses (Anderson et al., 2000). We cannot estimate the initial 230Th/238U of the Bishop Tuff magma and have thus assumed secular equilibrium. Charlier et al. (2005) found that 11 wholerock samples from a <~65 ka silicic magmatic system were nearly in secular equilibrium. If the Bishop Tuff magma was, for example, 2% out of secular equilibrium, our corrected 206Pb/238U dates are inaccurate by 400 yr.

CHEMICAL ZONING

CL and BSE imaging shows that chemical zoning in Bishop Tuff zircon is remarkably simple and consistent with sector zoning superimposed upon oscillatory zoning (Fig. 1). About half the grains are cored by central domains that are sector-zoned or unzoned (labeled CD in Fig. 1). Central domains are interpreted as being regions of early growth around which the sector- and oscillatory-zoned external domains formed, rather than xenocrystic cores, based on (1) concordance between the outer boundary of the domain and oscillatory zoning in the external domains, and (2) U-Pb dates (below) that show no age difference between grains with central domains and those without. Electron microprobe traverses across four grains show compositional differences between the central and external domains (Table DR2). External domains have higher U concentrations (average of 3400 ppm versus 1170 ppm; Fig. 1), Th concentrations (average of 2260 ppm versus 510 ppm), and Th/U (average of 0.65 versus 0.43). An ion microprobe study by Simon and Reid (2005) showed that zircon from the earlier, more evolved part of the eruption has U concentrations similar to the exterior domains in our study.

U-PB ZIRCON DATES

Nineteen ²⁰⁶Pb/²³⁸U dates corrected for initial Th/U disequilibrium with Th/U[magma] of 2.81 (Anderson et al., 2000) range from 779 to



Figure 1. CL images of Bishop Tuff zircon with ID-TIMS $^{206}Pb/^{238}U$ dates corrected for initial Th/U disequilibrium using Th/U[magma] = 2.81 ± 0.32. U concentrations (in ppm) from electron microprobe (EMP) traverses across two grains are shown. CD—central domains, the unzoned or sector-zoned regions in the middle of about half the grains.



Figure 2. ID-TIMS 206 Pb/ 238 U dates from Bishop Tuff zircon corrected for initial Th/U disequilibrium using Th/U[magma] = 2.81 ± 0.32. Weighted mean date based on the 17 youngest dates is shown. MSWD—mean square of weighted deviates.

765 ka (Figs. 1 and 2; Table DR1). The average internal error before propagating uncertainties in Th/U is ±1.2 k.y. Propagating the uncertainties in Th/U increases the average error to ±3.0 k.y. Using these errors, the 17 youngest ²⁰⁶Pb/²³⁸U dates are equivalent with a weighted mean of 767.1 ± 0.9 ka (MSWD = 1.3) (Fig. 2). Including a tracer calibration uncertainty of ±0.025% and decay constant uncertainties (Jaffey et al., 1971) increases the error on the weighted mean to ± 1.9 k.y. There is no relationship between date and the presence of central domains seen in the CL and BSE images. However, we recognize that the slight differences in dates could reflect subtle differences in growth histories that are smaller than analytical uncertainties.

The ²⁰⁷Pb/²³⁵U dates from the grains with the 17 youngest ²⁰⁶Pb/²³⁸U dates are equivalent with a weighted mean of 819.0 \pm 4.3 ka (MSWD = 0.5). The average internal error is \pm 27.0 k.y. Including the tracer calibration and decay constant uncertainties increases the error on the weighted mean to \pm 5.6 k.y. Possible explanations for the ~52 k.y. bias between the weighted mean 207 Pb/ 235 U and 206 Pb/ 238 U dates are discussed below.

MAGMA RESIDENCE TIME

The eruption age must be known before the amount of time that zircon resided in the magma can be determined. Based on ~70 single- and multigrain sanidine analyses from 11 samples of Bishop Tuff collected from five localities, an average 40 Ar/³⁹Ar date of 758.9 ± 3.6 ka was interpreted to approximate the eruption age (Sarna-Wojcicki et al., 2000). Recalculating this date using the commonly accepted age for the neutron fluence monitor, sanidine from the Taylor Creek rhyolite (Renne et al., 1998), yields a date of 770.4 ± 3.6 ka. The recalibrated dates from the 11 samples range from 783 to 761 ka, and the weighted mean date overlaps with all but the oldest two ²⁰⁶Pb/²³⁸U zircon dates.

Comparisons of ${}^{40}\text{Ar}/{}^{39}\text{Ar}$ and U-Pb dates from the same samples that range from 0.1 to 4.5 Ga show that ${}^{40}\text{Ar}/{}^{39}\text{Ar}$ dates are typically ~1% younger than 206 Pb/ 238 U dates (Min et al., 2000; Schoene et al., 2006). For the Bishop Tuff, the weighted mean of 17 of the 19 206 Pb/ 238 U dates overlaps with the 40 Ar/ 39 Ar date. Pb loss in zircon is unlikely to have decreased the dates, as most grains were treated with the chemical abrasion method that effectively eliminates Pb loss. Also, it is unlikely that Pb loss would have affected each zircon to the same extent, as would be required given the equivalence of the dates. The discrepancy between 40 Ar/ 39 Ar and U-Pb dates should decrease for very young samples if it is due only to decay constant inaccuracies, although this is the first high-precision comparison in <1000 ka rocks.

We interpret the weighted mean 206 Pb/ 238 U date of 767.1 ± 0.9 ka from the 17 youngest zircon grains as closely approximating the eruption age. In this scenario, the two grains with dates that are 6 and 12 k.y. older indicate a short period of magma residence for some zircon. These are minimum estimates of residence for the oldest parts of each grain if the grains are composites of older and younger domains. However, given the narrow range of dates, it is unlikely that the oldest part of any grain we dated resided in the magma for more than a few tens of thousands of years. More work is required to precisely determine the abundance and age of older grains.

The 206Pb/238U dates from ion microprobe studies of the Bishop Tuff zircon are generally older than 40Ar/39Ar sanidine dates (Sarna-Wojcicki et al., 2000) and our 206Pb/238U ID-TIMS zircon dates (Fig. 3). Reid and Coath (2000) obtained a weighted mean date of 823 ± 28 ka (MSWD = 0.8) from 22 analyses from 20 grains from the early phase of the Bishop Tuff. The average error on each ion microprobe date is ±~100 k.y., which is 33 times higher than the average ID-TIMS error that includes the uncertainties from the Th/U disequilibrium correction. The date is ~53 k.y. (7%) older than the ⁴⁰Ar/³⁹Ar sanidine age that represents eruption, and thus it was concluded that Bishop Tuff zircon had short magma residence, having crystallized within a few tens of thousands of years before eruption. Simon and Reid (2005) obtained ion microprobe ²⁰⁶Pb/²³⁸U zircon dates from two samples of the early and late Bishop Tuff. Dates from the late Bishop Tuff were subdivided to yield a minimum age of 753 ± 22 ka (n = 7) and a mean pre-eruption age of 849 ± 20 ka (n = 13). The early Bishop Tuff yielded a mean pre-eruption age of 853 ± 18 ka (n = 21), and the youngest dates yielded a weighted mean of 767 \pm 60 ka (n = 4). Mean pre-eruption ages from the early and late Bishop Tuff are ~80 k.y. (9%) older than the 40Ar/39Ar sanidine age.

While further work is required to understand the discrepancy between ion microprobe dates that indicate 50–80 k.y. of magma residence in most grains and ID-TIMS dates



Figure 3. Dates from the Bishop Tuff (BT). The ID-TIMS $^{206}Pb/^{238}U$ dates agree with the eruption age known from $^{40}Ar/^{39}Ar$ sanidine dating, yet are younger than ID-TIMS $^{207}Pb/^{235}U$ dates and mean pre-eruption ages from ion microprobe dating. The ID-TIMS $^{206}Pb/^{238}U$ dates are corrected for initial Th/U disequilibrium using Th/U[magma] = 2.81 ± 0.32. The $^{40}Ar/^{39}Ar$ sanidine dates are recalculated using the commonly accepted age for the neutron fluence monitor, sanidine from the Taylor Creek rhyolite (Renne et al., 1998).

that indicate little to no magma residence in 19 grains, there is general agreement between the methods that magma residence in the Bishop Tuff was rather short. It is clear that high precision offered by ID-TIMS dates can reveal important details about the magmatic histories of young rocks that are not resolvable with the ion microprobe.

Pa/U DISEQUILIBRIUM

The ²⁰⁷Pb/²³⁵U dates from <50 Ma zircon are seldom used due to difficulties in measuring small amounts of ²⁰⁷Pb from decay of ²³⁵U. However, the equivalent 207Pb/235U dates in our study with a weighted mean of 819.0 ± 4.3 ka are considered meaningful. The date is ~52 k.y. (7%) older than the weighted mean ²⁰⁶Pb/²³⁸U date and ~39 k.y. (5%) older than the oldest ²⁰⁶Pb/²³⁸U date. There are several possible explanations for the bias. It cannot be due to initial Th/U disequilibrium because a difference of 24 k.y. remains after making the largest possible correction on the oldest 206Pb/238U date. It is unlikely to be related to the composition or calibration of the tracer solution because two independently calibrated tracers with different isotopic compositions were used. It cannot be an artifact of HF acid leaching during chemical abrasion because five grains were purposely not subjected to it. The possibility of an isobaric interference at mass 207 was considered. However, because each analysis was run for 2-3 h until exhaustion and the dates agree despite differences in measured ratios and beam intensities, we conclude that it is not the cause. The only analytical variable that could have a large effect is ²⁰⁷Pb/²⁰⁴Pb of the procedural blank. The bias can be eliminated if a 207Pb/204Pb of 16.5 is arbitrarily used. However, we do not attribute the bias to inaccuracy in the isotopic composition of the blank because this value is substantially higher than the 207Pb/204Pb of 15.6 measured in blanks and there is no correlation

between ²⁰⁷Pb/²³⁵U date and ²⁰⁷Pb/²⁰⁴Pb in the analyses (Table DR1).

Disequilibrium partitioning of intermediate daughter nuclides in the 235U-207Pb decay chain during crystallization is a possible explanation. The only long-lived intermediate daughter product that may significantly perturb the equilibrium systematics is ²³¹Pa with a half-life of 33 k.y. Effects of initial Pa/U disequilibrium should be of lesser magnitude than those for Th/U disequilibrium because of the shorter half-life of 231Pa relative to 230Th, unless zirconmagma fractionation is much greater for Pa than for Th. Analysis of Holocene zircon by ion microprobe indicates that zircon-magma fractionation of ²³¹Pa is not significant, and thus only small excesses are expected (Schmitt, 2007). Schmitt (2007) assumed reasonable Pa/U[melt] values to show that Pa/U[zircon]/ Pa/U[melt] is likely to be 0.9-2.2 for most zircon, which yields an excess of 207Pb that equates to 5-57 k.y. Because the ~52 k.y. bias between 207Pb/235U and 206Pb/238U dates from the Bishop Tuff in our study falls within this range, we propose that much of the bias is due to initial Pa/U disequilibrium. This raises the question of how common excess 207Pb is in zircon. Substantial excess of 207Pb has been reported only once (Anczkiewicz et al., 2001), and a few examples of subtle excess have been reported (Mattinson, 2005; Schoene et al., 2006). Small excess similar to the ~52 k.y. documented here may thus be common but would not be easily detected in >10 Ma samples due to the relatively large absolute errors on ²⁰⁷Pb/²³⁵U dates.

SUMMARY

The biggest obstacle to obtaining precise and accurate ID-TIMS $^{206}Pb/^{238}U$ dates of Pleistocene zircon growth is the correction for initial Th/U disequilibrium. Although internal errors on dates from Bishop Tuff zircon are as low as ± 1 k.y., there is a 29 k.y. interval over which

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dates could vary if the values associated with the correction are poorly known. Th/U of the magma from which the zircon crystallized has the largest effect on the date, and thus accurate estimates are required. Another possible obstacle is the mixing of multiple growth domains within the same zircon grain, one formed just prior to eruption and others formed in the magma significantly before eruption. Chemical domains can be identified through CL and BSE imaging and microbeam analysis before isolation for ID-TIMS dating.

The ${}^{207}\text{Pb}/{}^{235}\text{U}$ dates from Bishop Tuff zircon are ~52 k.y. older than ${}^{206}\text{Pb}/{}^{238}\text{U}$ dates. We propose that the difference is due to the rarely quantified excess of ${}^{207}\text{Pb}$ from initial Pa/U disequilibrium. Similar small excesses in zircon may be rather common but would not be detected in >10 Ma grains due to the relatively large absolute errors.

Bishop Tuff zircon crystallized rapidly, with 206Pb/238U dates indicating most grains grew within 5 k.y. A short magma residence is shown by the overlap between the weighted mean date of 767.1 \pm 0.9 ka and the eruption age determined by ⁴⁰Ar/³⁹Ar dating of sanidine. These findings are consistent with the relatively simple crystallization history of zircon suggested by the internal chemical zoning, yet contrast with previous ion microprobe studies that proposed 50-80 k.y. of magma residence in most zircon grains in different samples. The results also show that despite the potential for silicic eruptions to contain zircon with long residence times, not all eruptions are plagued by this problem. ID-TIMS dating in conjunction with ⁴⁰Ar/³⁹Ar sanidine dating can evaluate zircon residence in Pleistocene magma chambers at the millennial scale.

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