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Zircon age-temperature-compositional spectra in plutonic rocks

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ABSTRACT

Geochronology can resolve dispersed zircon dates in plutonic rocks when magma cooling time scales exceed the temporal precision of individual U-Pb analyses; such age heterogeneity may indicate protracted crystallization between the temperatures of zircon saturation (T_{sat}) and rock solidification (T_{solid}). Diffusive growth models predict asymmetric distributions of zircon dates and crystallization temperatures in a cooling magma, with volumetrically abundant old, hot crystallization at T_{sat} decreasing continuously to volumetrically minor young, cold crystallization at T_{solid}. We present integrated geochronological and geochemical data from Bergell Intrusion tonalites (Central Alps, Europe) that document zircon compositional change over hundreds of thousands of years at the hand-sample scale, indicating melt compositional evolution during solidification. Ti-in-zircon thermometry, crystallization simulation using MELTS software, and U-Pb dates produce zircon mass-temperature-time distributions that are in excellent agreement with zircon growth models. These findings provide the first quantitative validation of longstanding expectations from zircon saturation theory by direct geochronological investigation, underscoring zircon's capacity to quantify supersolidus cooling rates in magmas and resolve dynamic differentiation histories in the plutonic rock record.

INTRODUCTION

Zircon is an inimitable chronicler of the temporal, thermal, and compositional evolution of many crustal magmatic systems. U-Pb zircon geochronology by chemical abrasion–isotope dilution–thermal ionization mass spectrometry (CA-ID-TIMS; Mattinson, 2005) is the most robust method available for constraining the tempo of pluton assembly and the longevity of magma reservoirs in deep time, having been applied to discern the incremental nature of composite intrusive suites (Coleman et al., 2004). As a result of steadily improved analytical precision, CA-ID-TIMS can resolve heterogeneous U-Pb zircon dates, or zircon spectra, in many igneous rocks at the hand-sample scale (Miller et al., 2007; Schoene et al., 2012; Broderick et al., 2015). Such studies demonstrate that zircon age distributions can reflect protracted zircon crystallization time scales, supplanting convention that zircon retains a singular emplacement age of its host rock (e.g., see discussion in Samperton et al., 2015). Therefore, zircon age information combined with complementary petrologic data from zircon and other phases can be used to constrain the time scales of magma emplacement, calculate supersolidus cooling rates, and track magma compositional evolution.

An important step in using zircon to further quantify magmatic processes is to compare zircon geochronology of natural systems with theoretical and experimental models for zircon saturation and growth (Watson, 1996; Harrison et al., 2007; Ferry and Watson, 2007; Boehnke et al., 2013; Bindeman and Melnik, 2016). *Ab initio* diffusive saturation calculations predict noninstantaneous, nonlinear volumetric zircon growth during monotonic magma cooling, with an asymmetric distribution of zircon mass crystallized as a function of time (t) or temperature (T) (Watson, 1996). This model predicts high initial crystallization rates at the system's zircon saturation temperature (T_{sat}) and continuous, near-exponentially decreasing growth to the solidus temperature (T_{solid}). While the treatment of Watson (1996) has been employed to describe zircon dissolution during thermal rejuvenation (Frazer et al., 2014), no attempt has been made to quantitatively compare theoretical expectations with measured zircon age spectra.

If a zircon population is the product of crystallization in a cooling, closed system, then the following criteria may be observed: (1) resolvable dispersion in U-Pb zircon dates, reflecting protracted crystallization time scales; (2) trends in zircon composition through time, reflecting evolving melt composition during fractional crystallization; (3) a systematic decrease in Ti-in-zircon crystallization temperature with time, reflecting magma undercooling (Ferry and Watson, 2007); and (4) asymmetric distributions of zircon mass crystallized as functions of time and temperature (Watson, 1996). While such features have been previously documented individually (e.g., Ickert et al., 2011; Tierney et al., 2016), no prior study has demonstrated these criteria simultaneously. As such, the geochronological, experimental, and modeling perspectives of zircon crystallization have yet to be concisely unified, and our ability to link U-Pb geochronology, petrology, and numerical modeling in magmatic systems thus remains limited. Here we address this shortcoming through geochronological, geochemical, and thermometric characterization of zircon from mid-crustal granitoids.

PREVIOUS GEOCHRONOLOGY AND HYPOTHESIS

We use the Bergell Intrusion, Central Alps, Europe (Fig. 1), as an excellent locale to test the four criteria presented here. Thermal insulation of

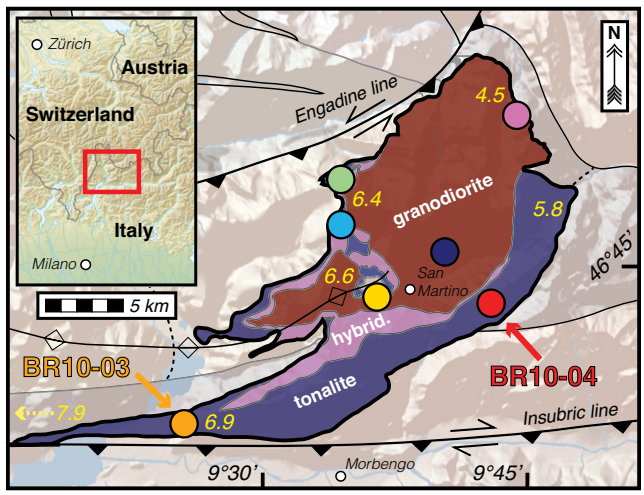

Figure 1. Geologic map of the Bergell Intrusion (northern Italy–southeast Switzerland) adapted from Samperton et al. (2015), with sample locations and key tonalite samples BR10-04 and BR10-03 specified. Representative Al-in-hornblende pressures (kbar) are given in yellow (Davidson et al., 1996). Hybrid.—hybridized.

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this magma reservoir at mid-crustal depths (Davidson et al., 1996) resulted in slow cooling and protracted zircon growth histories; its young age (ca. 32–30 Ma) makes resolving growth histories tractable by CA-ID-TIMS U-Pb geochronology; and minimal postemplacement mixing and/or remobilization of tonalites resulted in zircon spectra and geochemistry that are attributable to primary magmatic zircon growth (Samperton et al., 2015).

A multistep analytical workflow was applied by Samperton et al. (2015) to describe Bergell zircon systematics, including *in situ* trace element geochemistry and high-precision U-Pb CA-ID-TIMS-TEA (TEA—trace element analysis) geochronology and solution geochemistry (Schoene et al., 2010). Trends in tonalitic zircon composition on 10^5–10^6 yr time scales were interpreted to reflect zircon crystallization during magma evolution; e.g., hand-sample–scale trends of decreasing zircon Th/U as a function of U-Pb date reflect growth coeval with magmatic allanite + epidote on the basis of known trace element partitioning behavior (Figs. 2A and 2B; Oberli et al., 2004; Samperton et al., 2015). From these data, it was concluded (Samperton et al., 2015) that Bergell tonalitic zircon populations are overwhelmingly primary, i.e., the result of pseudo-closed system crystallization during postemplacement cooling, thus satisfying criteria 1 and 2. Comparatively, Bergell zircons from hybridized and granodioritic samples are a more complex amalgam of basement-derived xenocrysts, zircon entrained from earlier magma pulses, and primary magmatic domains. Criterion 3 dictates that Ti-in-zircon thermometry of tonalitic zircons should yield a strong correlation between temperature and a differentiation proxy (Th/U), confirming time-temperature-compositional evolution during down-liquidus crystallization. Here we test this hypothesis both through Ti-in-zircon thermometric characterization of Bergell zircon and, in tandem with the integrated U-Pb geochronological-geochemical data set in Samperton et al. (2015), compare empirical zircon data to a theoretical and an experimentally based zircon crystallization model (thus assessing criterion 4).

METHODS AND RESULTS

We present results of *in situ* zircon geochemical analyses by secondary ion mass spectrometry (SIMS), including Ti-in-zircon thermometry (Ferry and Watson, 2007), on a targeted subset of zircon populations described in Samperton et al. (2015; see the GSA Data Repository[1]). This approach permits determination of zircon temperature-geochemical trends by SIMS (Figs. 2C and 2D) that can be directly related to zircon time-geochemical trends resolved by U-Pb CA-ID-TIMS-TEA (Figs. 2A and 2B). Zircon time-Th/U trends parallel zircon temperature-Th/U trends in tonalite samples BR10-04 and BR10-03 (Fig. 2), indicating progressively decreasing crystallization temperatures through time and validating the interpretation that Bergell zircon spectra record protracted growth during magma cooling. Trends are corroborated by changes in complementary crystallization indicators with temperature (e.g., Ce/Ce* and Lu/Hf; Fig. DR1 in the Data Repository). Comparatively, SIMS analyses of granodioritic zircon yielded complex temperature-geochemical relationships (Fig. DR1), consistent with pervasive zircon inheritance in this Bergell lithology (Samperton et al., 2015).

To further evaluate the hypothesis that zircon age-temperature-compositional spectra reflect magma compositional evolution, we generated model zircon spectra from crystallization simulations using the thermodynamic modeling software MELTS (Ghiorso et al., 2002; http://melts.ofm-research.org), in which tonalitic whole-rock geochemistry was used as liquid compositions. The zircon solubility model of Boehnke et al. (2013) was employed to calculate the mass of zircon crystallized at each simulation step using the system temperature, melt compositional proxy (M value), and Zr concentration (Keller et al., 2017). To account for uncertainty in magmatic H_2O content, 5000 simulations were performed with H_2O content ranging between 0 wt% and 10 wt%; results were binned into 2.5 wt% increments and mean model zircon spectra calculated for each (Fig. 3; Fig. DR16). Simulations were also used to estimate the titania activity ($aTiO_2$) over the melt fraction interval of most tonalitic zircon crystallization (0.60–0.35) using the $aTiO_2$ solubility model of Hayden and Watson (2007), with calculated $aTiO_2$ increasing from ~0.25 to 0.65 (Fig. DR17). Mean $aTiO_2$ values of 0.44 (sample BR10-04) and 0.33 (sample BR10-03), in accordance with the estimated range of $aTiO_2$ in silicic magmas (0.3–0.9; Ghiorso and Gualda, 2013), were used to calculate Ti-in-zircon temperatures (Figs. 2 and 3). To address a widely recognized offset between MELTS and experimental temperatures, i.e., with MELTS regularly overestimating absolute temperatures by ~25–50 °C (Ghiorso et al., 2002; Gualda et al., 2012), a systematic minimum correction of –25 °C was applied to all model curves. Excellent correlation between the Ti-in-zircon temperature spectra and corrected MELTS models is observed (Fig. 3), with the former best matching relatively hydrous crystallization simulations (i.e., >2.5 wt% H_2O), consistent with evidence of H_2O-enriched Periadriatic parental magmas (Hürlimann et al., 2016).

Using the primary magmatic U-Pb zircon spectra interpreted in Samperton et al. (2015), we calculate cumulative distributions of Bergell zircon mass crystallized as a function of normalized time (and by extension, temperature; Fig. 4). In this scaling, x = 1 denotes the initiation of zircon crystallization at T_{sat}, and x = 0 the termination of crystallization at T_{solid}. Also included in this plot are the cumulative density functions of the aforementioned MELTS simulations and Ti-in-zircon temperatures (Fig. 3), as well as the zircon growth curve from Zr diffusion modeling of Watson (1996).

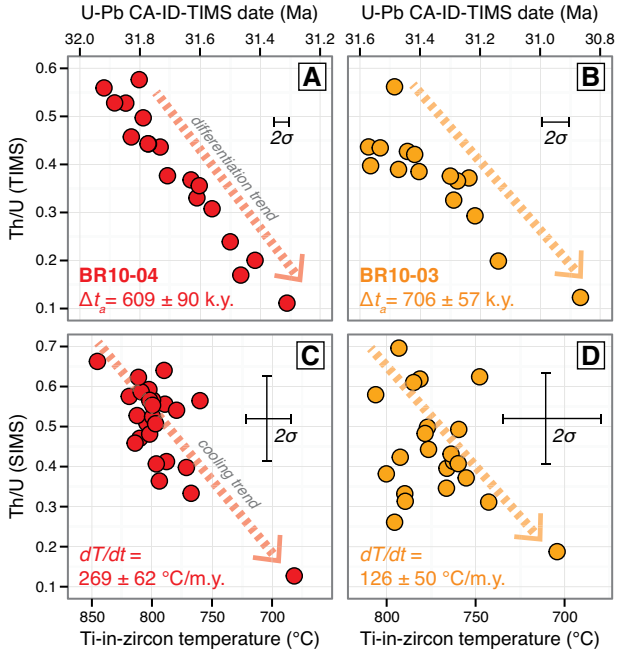

Figure 2. Zircon age-temperature-compositional trends from Bergell Intrusion (northern Italy–southeast Switzerland) tonalites BR10-04 and BR10-03. A, B: Zircon Th/U evolution as a function of $^{206}Pb/^{238}U$ date from chemical abrasion–isotope dilution–thermal ionization mass spectrometry (CA-ID-TIMS) geochronology, recording zircon compositional evolution (time, Δt_a) over 600–700 k.y. of magma crystallization (Samperton et al., 2015). C, D: Corresponding evolution in zircon Th/U as a function of Ti-in-zircon crystallization temperature (*T*) from secondary ion mass spectrometry (SIMS) microanalysis, indicating systematically decreasing growth temperatures through time during cooling and solidification. Magmatic cooling rate: *dT/dt* (°C/m.y.). Mean internal analytical uncertainties are 2σ.

[1]GSA Data Repository item 2017333, analytical details, modeling methods, SIMS and CA-ID-TIMS data tables, is available online at http://www.geosociety.org/datarepository/2017/, or on request from editing@geosociety.org.

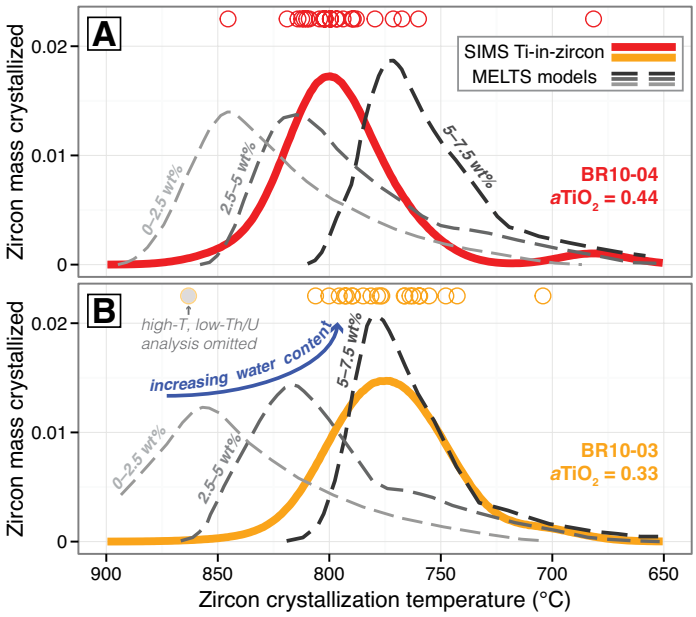

Figure 3. Ti-in-zircon temperature distributions as determined by secondary ion mass spectrometry (SIMS) microanalysis. A: From tonalite sample BR10-04. B: From tonalite sample BR10-03. Empirical temperature (*T*) distributions (solid curves) were calculated using the Ti-in-zircon calibration of Ferry and Watson (2007) and include analytical and calibration uncertainties. Circles show absolute Ti-in-zircon temperatures. Dashed curves are mean distributions of MELTS zircon crystallization simulations, binned by magmatic H_2O content. Mean titania activity, $aTiO_2$, values calculated from MELTS outputs and the TiO_2 solubility model of Hayden and Watson (2007). $aSiO_2 = 1$ is assumed for Ti-in-zircon calculations.

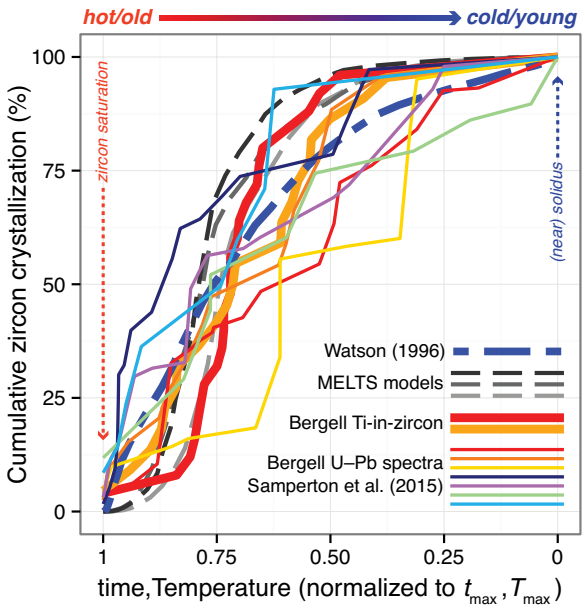

Figure 4. Summary of primary magmatic U-Pb zircon chemical abrasion–isotope dilution–thermal ionization mass spectrometry (CA-ID-TIMS) age spectra (Samperton et al., 2015), Ti-in-zircon thermometric data, MELTS (see text) zircon crystallization models, and zircon diffusive growth modeling (Watson, 1996), plotted as cumulative distributions (%) as functions of normalized time-temperature. Horizontal axis: 1 = zircon saturation temperature (T_{sat}) with old, high-*T* zircon domains; 0 = near solidus (T_{solid}) with young, low-*T* zircon domains. U-Pb spectra are color coded to sample locations in Figure 1, and MELTS and Ti-in-zircon curves are color coded to Figure 3.

DISCUSSION

Multitechnique application provides an opportunity to evaluate the efficacy of a given method. While the fidelity of Ti-in-zircon thermometry has been debated in certain applications (e.g., Fu et al., 2008), strong zircon age-temperature-compositional covariance in Bergell zircon on magmatic time scales provides compelling evidence in favor of Ti-in-zircon thermometry as a robust petrologic tool (Fig. 2). In general, due to the low volume of young, low-*T* zircon crystallized during cooling and relatively coarse CA-ID-TIMS sampling resolution, core-rim mixing will tend to skew geochronological data toward old, high-*T* domains. This may explain the subtle offset between MELTS simulations and zircon age spectra (Fig. 4), as the latter distributions may undersample the absolute latest, youngest, lowest-*T* stages of zircon growth. Similarly, SIMS analyses of unpolished zircon rims can ensure isolation of the outermost zircon domains for Ti-in-zircon thermometry (Tierney et al., 2016). Alternatively, that MELTS thermodynamic simulations do not capture potentially important zircon growth kinetics (e.g., Zr-limited zircon diffusive growth, major phase Zr bulldozing; Bindeman and Melnik, 2016) will also potentially contribute to deviations between data and models.

Despite these caveats, that the numerical crystallization model of Watson (1996), U-Pb zircon CA-ID-TIMS age spectra, Ti-in-zircon crystallization temperatures, and MELTS-based zircon crystallization modeling define independent, mutually consistent distributions is compelling evidence that these Bergell zircon populations record magma cooling and crystallization in action (Fig. 4). This study therefore provides a novel template for extracting the time-temperature-compositional history of an intrusive rock from its zircon population with high-precision geochronology, from which powerful information may be obtained. For example, magmatic cooling rates (*dT*/*dt*) calculated from the observed spread in U-Pb dates and Ti-in-zircon temperatures in the structurally higher BR10-04 sample are double that of the deeper BR10-03, consistent with interpretation of the Bergell Intrusion as being tilted (Figs. 1C and 1D; Davidson et al., 1996). Such knowledge is essential to informing and assessing increasingly sophisticated thermal models of magmatic systems (e.g., Gelman et al., 2013; Annen et al., 2015). A quantitative understanding of zircon spectra is also key to reconciling magma crystallization with incremental pluton assembly, wherein magma pulses are cyclically emplaced, cooled, and sometimes rejuvenated by later pulses (Coleman et al., 2004; Miller et al., 2007). It is interesting that the absence of strong zircon age-temperature-compositional covariance in many geochronological-geochemical studies necessitates incremental assembly as an important pluton-forming mechanism (Schoene et al., 2012; Broderick et al., 2015). Application of the tools described here to other plutons can therefore help further quantify models of incrementally emplaced plutons.

CONCLUSIONS

Few magmatic processes are truly instantaneous; the limitation imposed by analytical resolution can create the illusion of temporal, compositional, and thermal homogeneity in such systems. We have quantified post-emplacement crystallization and cooling histories in mid-crustal intrusive rocks on 600–700 k.y. time scales through characterization of zircon age-temperature-compositional spectra. Data and modeling in the current study corroborate U-Pb zircon spectra as reflecting resolvable time scales of melt residence and solidification in the middle crust, highlighting the power and necessity of treating zircon as a zoned, dynamic archive. Such data are essential for refining models of incremental pluton assembly and harmonizing temporal histories of magmatic systems inferred from geothermometry and geochronology versus those constrained by numerical modeling.

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