High-resolution *P-T-t* paths from $\delta^{18}O$ zoning in titanite: A snapshot of late-orogenic collapse in the Grenville of New York

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ABSTRACT

We characterize oxygen isotope zoning within single titanite crystals from the Carthage-Colton mylonite zone (CCMZ), Adirondack Mountains (New York State, United States), by ion microprobe. Smooth gradients of $\delta^{18}O$, up to 0.6‰ over 90 µm, resulted from diffusive exchange of oxygen during cooling from peak metamorphic temperatures of 650–700 C. Modeling of the observed profile indicates punctuated cooling rates of 30–60 C/m.y. along the CCMZ, set within long periods of much slower cooling. These results indicate a previously unrecognized period of rapid cooling along the CCMZ that is interpreted to result from the post-Ottawan collapse of the Grenville mountain belt and exhumation of the central Adirondack Highlands at ca. 1050 Ma.

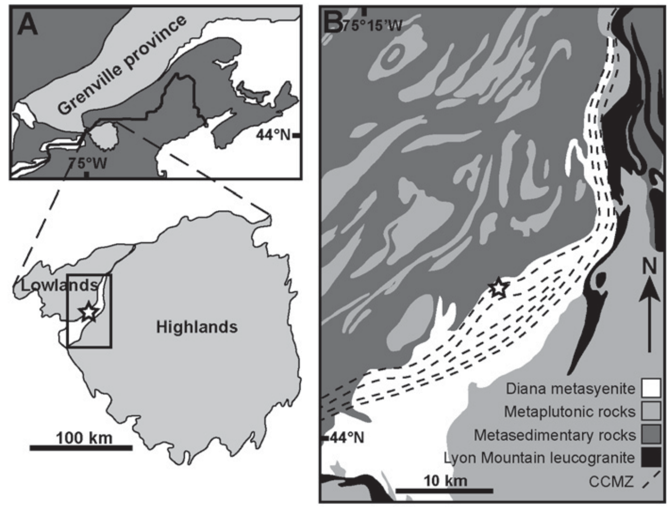

Figure 1. A: Location maps showing the study area within the Grenville province (upper) and the Adirondack Mountains (lower), New York, United States. B: Geologic map of the Diana metasyenite complex and the Carthage-Colton mylonite zone (CCMZ). Modified from Johnson et al. (2005). Star indicates Harrisville sample locations.

INTRODUCTION

Construction of detailed pressure-temperature-time (*P-T-t*) paths is critical to recognition and interpretation of short-lived magmatic and tectonic phenomena, such as pluton emplacement, fluid infiltration, or rapid exhumation. Typically, *P-T-t* paths for cooling and exhumation are constructed by dating the time of crystallization and/or diffusive closure of several minerals in combination with thermobarometry (e.g., Rivers, 2008). The temporal resolution along such a path depends on the duration of a metamorphic event relative to the time interval(s) between mineral crystallization and (diffusive) closure. In contrast, compositional variations within single minerals reflect changes in metamorphic conditions on the timescales of individual mineral growth or closure. High-spatial-resolution records of compositional variations within mineral grains have the potential therefore to provide high-temporal-resolution records of the tectonic and magmatic processes that affected metamorphic conditions (e.g., Page et al., 2010; Pollington and Baxter, 2010). Specifically, *P-T-t* paths determined from single-mineral chemical or isotopic zoning have the potential to resolve events that are one to two orders of magnitude shorter (e.g., Storm and Spear, 2005) than events resolved along *P-T-t* paths determined from multimineral thermochronology studies (e.g., Mezger et al., 1991).

In ancient, multiply deformed orogenic belts, *P-T-t* paths are important tools for interpreting the tectonic significance of structures (e.g., van der Pluijm et al., 1994; Dumond et al., 2007). This is exemplified by the thermochronologic studies of Mezger et al. (1991) and Cosca et al. (1992) that compare the cooling histories of distinct tectonic domains to determine kinematics and relative timing of motion along major, domain-bounding structures of the Grenville orogen. For this study, we revisit a controversial Grenville structure in the northwestern Adirondack Mountains (Fig. 1). The Carthage-Colton mylonite zone (CCMZ) retains evidence for a prolonged history of deformation and reactivation and is generally considered to be a key structure in accommodating late-Ottawan extension of overthickened Grenvillian crust (Streepey et al., 2000).

In this study, we characterize oxygen isotope zoning within single titanite crystals from the CCMZ by ion microprobe. The resulting spatially resolved, high-precision records of intragrain $\delta^{18}O$ variations are the first of their kind for titanite. We observe grain-scale, core-to-rim $\delta^{18}O$ zoning that reflects oxygen diffusion during cooling. Because the metamorphic history of the Adirondacks is well characterized, we can use geologic and experimental data to model the observed profiles and determine the cooling rate. The modeling results can be used to interpret the deformation history of the CCMZ and its implications for the rheological state of the crust during orogenesis.

OXYGEN ISOTOPE DIFFUSION

Oxygen, the most abundant element in Earth's crust, is a pervasive and readily measured tracer of geochemical and mass transfer processes. In many crystalline rocks, diffusion is the primary mass transfer process for intracrystalline exchange. Oxygen isotope diffusion is a kinetic phenomenon driven by gradients in isotopic composition that arise from differences in $^{18}O/^{16}O$ fractionation between phases (see Cole and Chakraborty, 2001). In metamorphic rocks, diffusion commonly reflects small changes in equilibrium fractionations between phases as a result of temperature variations or fluid infiltration (see Valley, 2001). Unlike chemical diffusion of cations, tracer diffusion (isotope exchange) does not require complex coupled substitutions. Ions diffuse through crystalline lattices and/or dislocation networks within crystals, and along grain boundaries. Grain boundaries are "fast" diffusion pathways along which diffusion is orders of magnitude faster than diffusion through crystal lattices (Eiler et al., 1992). The fast grain boundary (FGB) diffusion model proposes that under high-temperature conditions isotopic equilibrium is established and maintained at grain boundaries by rapid grain boundary diffusion (Eiler et al., 1992). The FGB model, which can be quantitatively implemented through a numerical program (Eiler et al., 1994), has proven a successful tool for modeling the effects of intergrain diffusion on intragrain isotopic distribution in high-temperature systems (Eiler et al., 1995).

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GEOLOGIC SETTING AND BACKGROUND

The central Adirondack Mountains (New York State, United States) expose multiply deformed metaigneous and metasedimentary rocks of the Ottawan phase of the Grenville orogeny (1090–1000 Ma; McLelland et al., 2004). The northwest Adirondack Lowlands and central Adirondack Highlands are juxtaposed across the 1–15 km wide, NNE-striking CCMZ. The CCMZ is a major geochronologic boundary, separating rocks that experienced upper amphibolite facies metamorphism at ca. 1150 Ma in the Lowlands from rocks that reached granulite facies conditions nearly 100 m.y. later at ca. 1050 Ma in the Highlands (Mezger et al., 1992). The relative positions of the Highlands and Lowlands during the early Ottawan phase and the timing of their current structural juxtaposition remain controversial (e.g., Wiener, 1983; Baird and MacDonald, 2004; Johnson et al., 2005); however, the CCMZ ultimately accommodated northwest-directed displacement of the northwest Adirondack Lowlands and consequent exhumation of the central Highlands (Mezger et al., 1992; Streepey et al., 2001; Selleck et al., 2005). Regional thermochronologic studies are consistent with ≥200 m.y. of slow cooling in both the Lowland and Highland domains at an average rate of 1–5 °C/m.y. and convergence of the two thermal histories after 1000 Ma (Mezger et al., 1991, 1992).

Samples for this study were collected from well-exposed roadcuts in the Diana metasyenite along Route 3 west of Harrisville, New York (Fig. 1). These roadcuts contain a pervasive but variably developed protomylonitic fabric, which is crosscut by more than 100 steeply dipping ultramylonitic shear zones. Cartwright et al. (1993) documented variable whole-rock and mineral $\delta^{18}O$ values from within the shear zones and the adjacent protomylonitic wall rocks. Titanite is a widespread accessory phase throughout the Diana metasyenite with petrographic relations that indicate growth at the expense of ilmenite during and after the peak metamorphism. U-Pb dating of titanite and metamorphic zircon from the Harrisville roadcuts yields a range of ages spanning Ottawan deformation with a distinct age population at ca. 1050 Ma, near the proposed Ottawan thermal peak (Mezger et al., 1991; Johnson et al., 2005; Chappell et al., 2006). Titanite, which has low oxygen diffusivity relative to the modally dominant feldspar and quartz of the metasyenite, is thus a good candidate for the preservation of intragrain oxygen isotopic zoning that developed in response to late-Ottawan thermal events.

ION MICROPROBE $\delta^{18}O$ ANALYSIS AND DATA

Ion microprobe samples were prepared as thin sections to preserve the microstructural context of titanite grains and ensure the preservation of the grain rims. We performed a series of *in situ* $\delta^{18}O$ analyses along traverses across titanite grains using an IMS-1280 at the WiscSIMS lab, University of Wisconsin–Madison (Kita et al., 2009; Valley and Kita, 2009), and a 10–15 µm spot size. Values of $\delta^{18}O$ are corrected for instrument bias using a linear correlation between Ti content in three titanite standards and bias relative to UWQ-1, a well-characterized quartz standard (Kelly et al., 2007; Appendix DR1 in the GSA Data Repository[1]). Analytical precision of $\delta^{18}O$ values is based on eight quartz standard analyses that bracket each group of 10–20 sample analyses and is reported at two standard deviations (Table DR1 in the GSA Data Repository).

Ion microprobe traverses were performed on 12 titanite grains with diameters ranging from 400 to 1000 µm. In the Harrisville outcrops, there are four microstructurally distinct populations of titanite. Analytical traverses across grains from the three populations with pre- and syn-shearing microstructures yield profiles with higher $\delta^{18}O$ in grain cores and lower $\delta^{18}O$ at grain rims (Fig. 2). The profiles are steep-sided, extending 80–120 µm into grains with approximately flat interior plateaus. This $\delta^{18}O$ zoning indicates modification of titanite oxygen isotope ratios by diffusive exchange with the surrounding rock in response to increased equilibrium fractionation relative to modally dominant quartz and feldspar during cooling. In this contribution, we highlight results from one pre-shearing porphyroblast that is related to the earliest deformational fabric and is therefore inferred to preserve the longest cooling history. All analyzed Harrisville grains with diffusion-related zoning preserve similar $\delta^{18}O$ gradients.

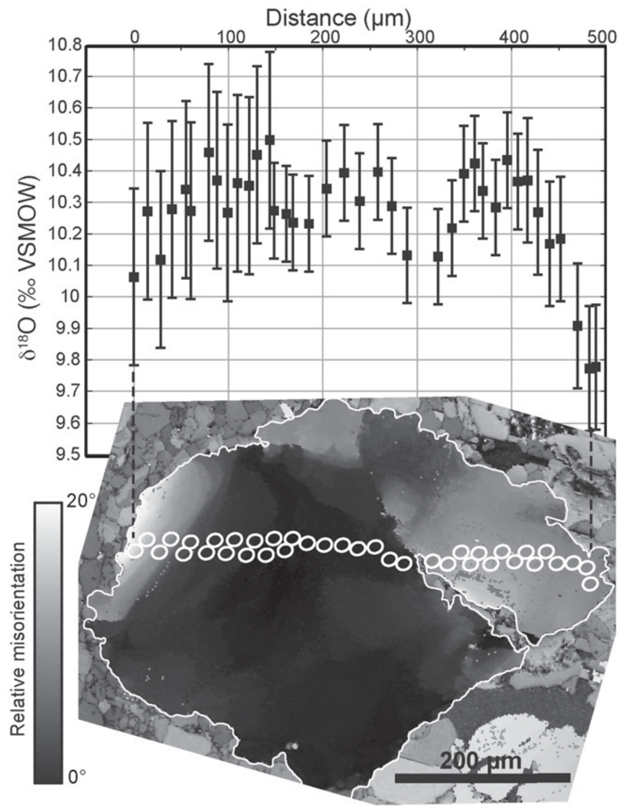

Figure 2. Electron backscatter diffraction relative misorientation map of a titanite grain and the $\delta^{18}O$ profile obtained by ion microprobe. Titanite grain is outlined in white. White ovals indicate locations of ion microprobe analysis pits. Fine-grained matrix is the feldspar-quartz–dominated metasyenite wall rock. Error bars are two standard deviations. See text for discussion. VSMOW—Vienna standard mean ocean water.

OXYGEN DIFFUSION MODELING

We model the $\delta^{18}O$ profile from a 490 µm long wall-rock titanite grain in the fast grain boundary program (Eiler et al., 1994) (Fig. 2; Appendix DR2). The profile is steep-sided with a total core-to-rim range in $\delta^{18}O$ of 0.6‰ (Table DR1). Electron backscatter diffraction (Fig. 2) reveals a correlation between subgrain boundaries and grain-interior $\delta^{18}O$ variations, suggesting that these boundaries acted as fast diffusion pathways. We therefore model only the right-hand, 180-µm segment of the diffusion profile (Fig. 3) that falls between the grain rim and a well-defined orientation boundary, which we infer was a fast pathway for oxygen exchange and delineated the right-hand part of the grain as a distinct diffusion domain. Within error, the left side of the grain is bounded by the same $\delta^{18}O$ gradients as the right side.

We input a whole-rock $\delta^{18}O$ value (13.8‰), grain diameter (180 µm), and the relative modal abundances of phases based on thin-section obser-

[1]GSA Data Repository item 2011281, Appendix DR1 (correction of $\delta^{18}O$ measured in titanite by ion microprobe, and Appendix DR2 (Fast Grain Boundary modeling), is available online at www.geosociety.org/pubs/ft2011.htm, or on request from editing@geosociety.org or Documents Secretary, GSA, P.O. Box 9140, Boulder, CO 80301, USA.

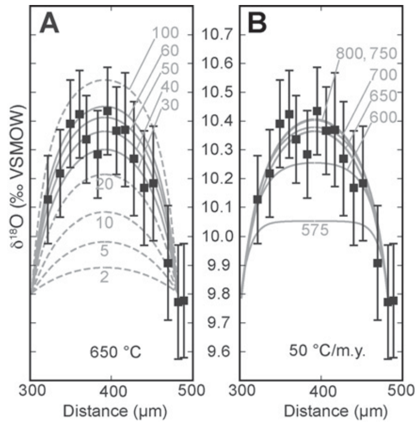

Figure 3. Values of δ18O from the right-hand (diffusion) domain of the titanite grain in Figure 2 with (A) fast grain boundary model profiles for a fixed peak temperature of 650 C and various cooling rates, and (B) fast grain boundary model profiles for a fixed 50 C/m.y. cooling rate and various peak temperatures. Models that best fit both the magnitude and shape of the δ18O gradient require 30–60 C/m.y. cooling rate (solid lines) from peak *T* of >600 C to 550 C. VSMOW—Vienna standard mean ocean water.

vations and measured δ18O of titanite. Arrhenius parameters (D_0 and Q) in "wet" titanite are taken from the experimental study of Zhang et al. (2006), which is consistent with an earlier study by Morishita et al. (1996). These studies showed no significant variations in diffusivity with crystallographic orientation, and titanite is accordingly modeled as isotropic. Equilibrium oxygen isotope fractionations involving titanite have been calibrated by King et al. (2001). Starting temperatures are selected from the range (650–700 °C) determined for peak metamorphism along the CCMZ (Kitchen and Valley, 1995). A final temperature of 550 °C reproduces the observed grain-rim δ18O value, indicating that diffusion at the micrometer scale was not significant below this temperature. We assume linear cooling rates of 2, 5, 10, 20, 30, 40, 50, 60, and 100 °C/m.y., and thus vary the time for temperature to decrease from the selected peak *T* to 550 °C. The resulting profiles are compared with the measured δ18O profile, and goodness of fit is determined by visual inspection. Modeling results for a peak *T* of 650 °C are shown in Figure 3A. The observed profile is best fit by cooling rates in the range 30–60 °C/m.y., and the observed δ18O gradients would not have been preserved at cooling rates below 30 °C/m.y.

DISCUSSION AND GEOLOGIC IMPLICATIONS

This is the first study to report δ18O zoning measured in natural titanite by ion microprobe. Improvements in instrumental techniques and sample preparation increase precision to the level of ≤0.3‰ for single spots and better for multiple measurements, and thus subtle isotopic variations are clearly resolved (Kita et al., 2009). The grain-scale, core-to-rim zoning in Figure 2 resulted from diffusion of oxygen in titanite. We can therefore use numerical modeling to determine the cooling rates that produced the observed δ18O diffusion profiles.

Figure 3 shows that cooling rates of 30–60 °C/m.y. fit the measured profile well, but that slower cooling would result in homogenization of the δ18O gradients by diffusion. These rates are 6–60 times faster than the regional rates of 1–5 °C/m.y. determined by Mezger et al. (1991) and Streepey et al. (2000). This apparent discrepancy arises from differences in the temporal resolution of the intragrain δ18O record and the Mezger et al. (1991) thermochronologic data. Modeling suggests that titanite retains a record of oxygen diffusion over a period of ≤5 m.y., whereas the Mezger et al. (1991) multimineral study spans ~200 m.y. Figure 4A shows that the new δ18O data are consistent with the long-term average cooling rates determined from bulk closure temperatures and dates for U-Pb and Ar-Ar geochronology. The δ18O record therefore complements the long-term record and resolves details of the CCMZ cooling history that were previously inaccessible.

The titanite δ18O zoning record indicates a period of rapid cooling within a few million years of peak Ottawan metamorphism (ca. 1050 Ma).

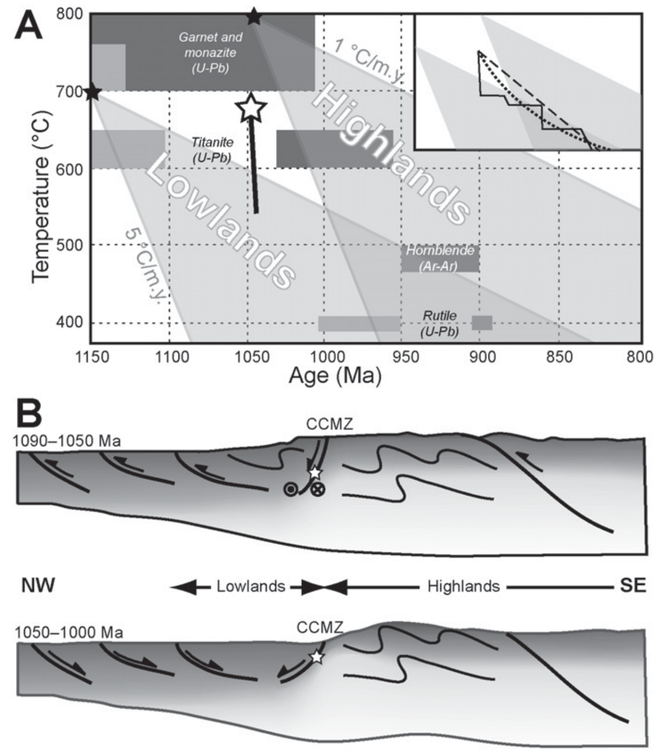

Figure 4. A: Temperature-time plot showing constraints on retrograde cooling in the Adirondack Mountains, New York. Note that different paths exist for the Highlands and the Lowlands. Rectangles are bulk-mineral closure constraints from regional thermochronology studies, as compiled by van der Pluijm et al. (1994). Triangular domains are cooling envelopes originating at the peak temperature for each domain (black stars) and bounded by the slowest (1 C/m.y.) and fastest (5 C/m.y.) cooling rates proposed by Mezger et al. (1991). Overlap of envelopes defines the earliest time of final Lowland-Highland juxtaposition after the Ottawan orogeny (Mezger et al., 1992; Streepey et al., 2001). White star and heavy black line indicate the peak *T* and rapid-cooling (≥30 C/m.y.) path segment, respectively, for rocks within the Carthage-Colton mylonite zone (CCMZ) as determined from titanite oxygen zoning in this study. Inset shows three schematic cooling paths—linear (steady-state), 1/*T* (gradually varying), and stepwise, where steep path segments indicate rapid cooling during active shearing, and isothermal segments indicate periods of limited deformation. B: Schematic cross sections of the western Adirondack Mountains during the Ottawan orogeny, modified from Heumann et al. (2006). Star shows location of study area rocks at depths of ~10–20 km. Early deformation along the CCMZ was likely oblique-slip (Streepey et al., 2001; Johnson et al., 2005). Later, dip-slip–dominated motion accommodated exhumation. Shading indicates the crustal thermal gradient. Rapid cooling may have been induced by juxtaposition of hotter and cooler crust across the CCMZ as shown in the lower cross section.

Rapid cooling for these samples implies rapid exhumation and shearing along the CCMZ. Thus, we infer short punctuated events within the slow regional cooling of the Highlands. Furthermore, rapid exhumation during the initial stages of the CCMZ cooling suggests either a final, culminating phase of crustal thickening, or, which we consider more probable, precipitous collapse of the Ottawan orogen (Fig. 4B).

The Grenville province represents a massive orogenic event on the scale of the modern-day Himalayan collision (e.g., Beaumont et al., 2006; Rivers, 2008), and the geologic evidence of a hot, weak crust (i.e., granulite facies metamorphism and partial melting) is at odds with the thermochronologic evidence of a strong, slowly deforming crust (i.e., prolonged steady-state cooling). The period of rapid cooling recorded by δ18O zoning in titanite suggests that this paradox may arise from differences in the time scales of rheological and mineralogical transformation. More detailed application of the techniques described in this study can be used to generate high-resolution *P-T-t* paths that better capture the evolving thermomechanical properties of orogenic crust.

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