A view into crustal evolution at mantle depths

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Abstract

Crustal foundering is an important mechanism in the differentiation and recycling of continental crust. Nevertheless, little is known about the dynamics of the lower crust, the temporal scale of foundering and its role in the dynamics of active margins and orogens. This particularly applies to active settings where the lower crust is typically still buried and direct access is not possible. Crustal xenoliths derived from mantle depth in the Pamir provide a unique exception to this. The rocks are well-preserved and comprise a diverse set of lithologies, many of which re-equilibrated at high-pressure conditions before being erupted in their ultrapotassic host lavas. In this study, we explore the petrological and chronological record of eclogite and felsic granulite xenoliths. We utilized accessory minerals – zircon, monazite and rutile – for coupled in-situ trace-element analysis and U-(Th-)Pb chronology by laser-ablation (split-stream) inductively coupled plasma mass spectrometry. Each integrated analysis was done on single mineral zones and was performed in-situ in thin section to maintain textural context and the ability to interpret the data

in this framework. Rutile thermo-chronology exclusively reflects eruption (11.17 ± 0.06 Ma), which demonstrates the reliability of the U-Pb rutile thermo-chronometer and its ability to date magmatic processes. Conversely, zircon and monazite reveal a series of discrete age clusters between 55-11 Ma, with the youngest being identical to the age of eruption. Matching age populations between samples, despite a lack of overlapping ages for different chronometers within samples, exhibit the effectiveness of our multi-mineral approach. The REE systematics and age data for zircon and monazite, and Ti-in-zircon data together track the history of the rocks at a million-year resolution. The data reveal that the rocks resided at 30-40 km depth along a stable continental geotherm at 720-750 °C until 24-20 Ma, and were subsequently melted, densified, and buried to 80-90 km depth – 20 km deeper than the present-day Moho – at 930 ± 35 °C. The material descended rapidly, accelerating from 0.9-1.7 mm yr⁻¹ to 4.7-5.8 mm yr⁻¹ within 10-12 Myr, and continued descending after reaching mantle depth at 14-13 Ma. The data reflect the foundering of differentiated deep-crustal fragments (2.9-3.5 g cm⁻³) into a metasomatized and less dense mantle wedge. Through our new approach in constraining the burial history of rocks, we provided the first time-resolved record of this crustal-recycling process. Foundering introduced vestiges of old evolved crust into the mantle wedge over a relatively short period (c. 10 Myr). The recycling process could explain the variability in the degree of crustal contamination of mantle-derived magmatic rocks in the Pamir and neighbouring Tibet during the Cenozoic without requiring a change in plate dynamics or source region.

Keywords: Zircon; Monazite; Rutile; Crustal evolution; Ti-in-zircon; U-Pb

1. Introduction

Subduction, subduction erosion, and foundering at active margins profoundly impact the evolution of continents and influence global geochemical cycles (e.g., Kay & Kay, 1993; Rudnick & Fountain, 1995; 96; Jull & Kelemen, 2001). Despite their importance in the evolution of Earth's lithosphere, these processes are relatively poorly characterized, and their rates and relative importance, in particular those of foundering, are not well constrained. Crustal xenoliths derived from deep regions of active margins have provided promising insight into deep-crustal dynamics (e.g., Ducea & Saleeby, 1996) and may prove of key importance in revealing how the lower crust and mantle interact. Nevertheless, analyzing this material and recovering its petrological and chronological record is non-trivial; they are rare, typically mafic and consequently depleted in ideal geochronometers, and highly altered through melt interaction. An exception to this is provided by centimeter- to decimeter-sized, mafic to felsic crustal xenoliths in ultrapotassic pipes in Dunkeldik magmatic field, Southern Pamir (e.g., Dmitriev, 1976; Ducea et al., 2003). These rocks are unique in that they are very well preserved, large (decimeter-sized), and of a large compositional variety ranging from felsic granulites to eclogites and ultramafic rocks (e.g., Lutkov, 2003; Hacker et al., 2005; Gordon et al., 2012). The xenoliths were derived from the deep root of the Pamir-Tibet Orogen, thus permitting direct insight into the evolution of the deepest section of this active orogeny. To obtain a high-resolution record of the history of these rocks, we utilized combined multi-method geochronology (U-Pb zircon; U-Pb and ^{232}Th - ^{208}Pb monazite), thermochronology (U-Pb rutile), petrology (REE), and thermometry (Ti-in-zircon; Zr-in-rutile). Each of these methods was done in-situ in thin sections enabling investigation of chronometric and thermometric data in relation to the petrological and textural context.

2. Geological Setting and Sample Description

The xenoliths are from the Dunkeldik magmatic field in the East Pamir (Fig. 1), which comprises a series of Miocene ultrapotassic pipes. The magma in these pipes consists of trachyte, syenite, alkali-basalt and carbonatite, which erupted at 11.3 ± 0.2 Ma (Hacker et al. 2005). The xenoliths consist of felsic granulite, mafic granulite, eclogite and websterite. This association of rocks compositionally equilibrated at pressure (P) and temperature (T) conditions of 2.5-2.8 GPa and 1,000-1,100 °C (Hacker et al., 2005; Gordon et al., 2012), corresponding to depths of 75-90 km, 10-25 km below the present-day Moho depth (Mechie et al., 2012). Detrital zircon age data indicate that the rocks sample a deep section of the Early Cenozoic Hindu Kush–Pamir–Karakorum continental arc (Ducea et al., 2003; Schwab et al., 2004). Following equilibration on a 30 K/Ma pre-Neogene geotherm (Schmidt et al., 2011), the mid-crustal section of the same crustal column was exhumed in large domes during the Miocene (Stübner et al., 2013a,b; Stearns et al., 2013). Seven lower-crustal xenoliths were chosen for this study – two eclogites (DK03, DK60), four felsic granulites (DK20, DK33, DK42, DK62), and one sample comprising felsic and mafic layers (DK19; thin section photos in Fig. 2). All samples come from the largest pipe complex in Dunkeldik Valley (Fig. 1).

The sanidine eclogites (DK03, DK60; Fig. 2) are heterogeneous aggregates comprising centimetre-scale patches rich in omphacite, garnet and quartz, separated by patches made up largely of sanidine and quartz. Accessory minerals include kyanite, apatite, pyrrhotite, zircon, and rutile. Zircon occurs as small (<0.1 mm) inclusions in garnet and omphacite, and throughout the rock matrix. Rutile occurs as large (0.2-0.7 mm) matrix crystals between, and locally intergrown with, the rock-forming phases. The specific eclogites analysed here are different from other eclogites among the xenolith suite in the sense that the latter are homogeneous and

intensely foliated garnet-omphacite rocks. The reason for selecting these two samples is that they – in contrast to the other eclogites – contain abundant accessory minerals.

The felsic granulite xenoliths (DK20, DK33, DK42, DK62; Fig. 2) comprise millimetre- to centimetre-sized garnet, kyanite, plagioclase, quartz and sanidine. Accessory minerals are hematite, biotite, apatite, zircon, monazite, and rutile, all of which occur as matrix phases and as inclusions in garnet and other major minerals. Garnet modal abundance reaches up to 55 vol% and grains are up to cm-scale in size in samples that lack biotite. Rutile is also abundant in these biotite-free areas. Observations of biotite dehydration melting textures (Fig. 3F) indicate that this systematic relates to the occurrence of the reaction $\text{biotite} + \text{albite} + \text{kyanite} + \text{quartz} \rightarrow \text{garnet} + \text{sanidine} + \text{hydrous granitic melt}$. Sample DK33 exhibits a lineation defined by aligned kyanite blades. Garnet in contact with other garnet, or with kyanite blades in this sample shows a weak shape preferred orientation parallel to L_{ky} , indicating growth during or possibly after deformation. Zircon and monazite grains occur in each of these textures, as well as in the rock-forming phases. The grains are typically round to elliptic and show variable grain size up to 0.2 and 0.09 mm, respectively. Rutile is mostly elongate and coarse-grained (0.2-0.5 mm), and includes ilmenite lamellae.

Sample DK19 is a mixed lithology consisting of layers rich in omphacite separated by layers made up entirely of quartz and feldspar (Fig. 2). The rock differs compositionally from the eclogites in that it has a higher proportion of felsic phases and it contains monazite. All samples interacted with the alkali-basaltic host melt, causing minor corrosion of grain boundaries, and replacement of rock forming minerals by cryptocrystalline aggregates of silicates and oxides. Overall, less than 2 vol% of the samples is affected by such alteration.

3. Analytical Methods

To obtain texturally controlled chemical and chronological data, zircon, monazite and rutile were analyzed in-situ in polished thin sections (30 μm thickness). Back-scattered electron imaging of rutile and monazite and cathodoluminescence (CL) imaging of zircon were done using a FEI Quanta 400f field-emission scanning electron microscope (SEM) to reveal internal structures and zoning. The results are given in figure 3. Minor elements in rutile – Zr, Nb, Fe, and Al – were measured using a CAMECA SX-100 electron probe micro-analyzer (EPMA) operating at 20 kV and 80-nA beam current (data in Appendix A). Zirconium-in-rutile thermometry was conducted using a P-dependent calibration (Ferry & Watson, 2007).

Accessory mineral isotope and trace-element analyses were done in thin section by laser-ablation inductively coupled plasma mass spectrometry (LA-ICPMS) using a Photon Machines 193 nm Excimer LA system coupled to a Nu Instruments AttoM high-resolution sector-field ICP-MS (HR-SF-ICPMS; REE in zircon and monazite) and a Nu Instruments Plasma HR multi-collector ICP-MS (MC-ICPMS; U-Pb and Th-Pb in zircon and monazite; Ti in zircon). Zircon U-Pb age data, and REE and Ti concentrations were obtained from individual CL zones in zircon. To enable analysis tailored to the requirements of each of these methods, the analyses were conducted on separate small spots (<25 μm) in individual CL zones. Using this approach, we obtained U-Pb and Ti data (LA-MC-ICPMS), and REE concentrations (HR-SF-ICPMS) for all targeted zones. Zircon was ablated for 20 s using 4 mJ, and pulsing at 4 Hz on spots of 15-20 μm . The 91500 standard (1062.4 ± 0.4 Ma; Wiedenbeck et al., 1995) was used for normalization and indicated external reproducibility of 0.13 %RSD ($n = 52$). Accuracy was assessed by analyzing the zircon standards Plešovice (337.1 ± 0.4 ; Sláma et al., 2008), Peixe (564 ± 4 Ma; G. Gehrels, unpublished data), and GJ-1 (608.4 ± 0.4 Ma; Jackson et al., 2004) and 601.7 ± 1.3 Ma;

Condon, unpublished data). We obtained $^{206}\text{Pb}/^{238}\text{U}$ ages of 339.3 ± 1.1 Ma ($n = 24$, MSWD = 0.60), 562.1 ± 1.8 Ma ($n = 24$, MSWD = 0.93), and 601.8 ± 2.1 Ma ($n = 40$, MSWD = 0.90), indicating reliable accuracy. Common Pb was not corrected for ‘in-run’, but was instead used in $^{207}\text{Pb}/^{206}\text{Pb}$ - $^{238}\text{U}/^{206}\text{Pb}$ or ‘Tera-Wasserburg’ space to calculate Concordia ages using a common Pb composition as constrained from low U/Pb rutile (constructed using Isoplot 3.00; Ludwig, 2003). Uncertainty on the upper intercept does not significantly affect calculated ages, because these are young and near-concordant.

High-precision Ti concentration analysis was done by ion counting on m/z corresponding to ^{49}Ti using static MC-ICPMS and normalizing to the matrix-matched standard 91500 (c. 6.40 ppm; Yuan et al., 2004) yielding 0.85% RSD. Replicate Ti analyses of the GJ-1 zircon yielded 5.2 ± 0.9 ppm, which is within uncertainty of the preferred value of 4.3 ± 0.5 ppm (Jochum et al., 2005). Titanium-in-zircon thermometry was conducted using a P dependent calibration (Ferry & Watson, 2007). Concordia diagrams are given in Fig. 3, whereas the integral dataset can be found in Appendix B. Correlations between $^{206}\text{Pb}/^{238}\text{U}$ age, and the CI-chondrite-normalized REE ratios (Gd/Yb_N) (subscript ‘N’ denoting normalization) and Eu_N/Eu^* (where Eu^* is the average of Sm_N and Gd_N) are shown for 4 samples in figure 5. Chondrite normalization is done using the CI-chondrite composition of Boynton (1984). Trends of REE systematics with $^{206}\text{Pb}/^{238}\text{U}$ age for sample DK60 are visualized in figure 6.

Monazite was analyzed by LA-‘split-stream’-ICPMS (LA-SS-ICPMS; Kylander-Clark et al., 2013) to simultaneously yield U-Th-Pb age and REE data. This phase was ablated for 20 s applying pulsed laser light of 4 mJ at 4 Hz on spots of 7-10 μm . The Pb/U and $^{208}\text{Pb}/^{232}\text{Th}$ data were normalized to replicate analyses of the 44069 monazite (424.9 ± 0.4 Ma, ID-TIMS $^{206}\text{Pb}/^{238}\text{U}$ age; Aleinikoff et al., 2006), which indicated external reproducibility of 0.25 %

relative standard deviation (%RSD) during the analytical series ($n = 34$). To assess accuracy, we performed analyses of Stern (512.1 ± 1.9 Ma; Palin et al., 2013), FC-1 (55.6 ± 0.4 Ma; Horstwood et al., 2003) and Trebilcock (278 ± 2 Ma; Tomascak et al., 1996) monazite during the sample runs. The obtained weighted mean $^{206}\text{Pb}/^{238}\text{U}$ ages were 509.4 ± 1.7 Ma ($n = 34$, MSWD = 1.9), 56.76 ± 0.27 Ma ($n = 26$, MSWD = 2.4), and 276.18 ± 0.70 Ma ($n = 32$, MSWD = 1.06). Monazite REE concentrations were obtained by running the AttoM in e-scan (deflector) mode and using the matrix-matched standard 44069. Standard analyses indicated an average 2 %RSD for heavy REE concentrations and accuracy to within 10% for middle and heavy REE (MREE; HREE). Diagrams of $^{206}\text{Pb}/^{238}\text{U}$ versus $^{208}\text{Pb}/^{232}\text{Th}$ are given in figure 7. The integral monazite age and REE concentration data is provided in Appendix C.

Rutile micro-analysis was done using 7 mJ and 4 Hz for 20 s on 40-50 μm -wide spots. The Pb/U data were normalized to R10 standard rutile (1095 ± 5 Ma; Luvizotto et al., 2009), which reproduced to within 0.12 %RSD. Accuracy was assessed by analyzing R19 (489.5 ± 0.9 Ma; Zack et al., 2011) and SP-1 (911 ± 2 Ma; Mezger et al., 1989). We obtained $^{206}\text{Pb}/^{238}\text{U}$ ages of 496.5 ± 5.1 Ma ($n = 24$) and 900.1 ± 7.8 Ma ($n = 24$) for these materials, demonstrating accuracy to within 1%. Rutile age data for 4 samples are shown in Concordia diagrams in figure 8. The integral rutile age dataset is included in Appendix D. All accessory mineral data were reduced with Iolite v. 2.15 (Paton et al., 2010), applying a non-linear down-hole fractionation correction and a 1st order polynomial function to correct for drift in U-(Th-)Pb analyses.

4. Results

4.1. Zircon

Zircon grains in all samples typically comprise cores and rims, or exhibit more complex zoning (Fig. 3). In contrast to cores, rims typically show no zoning under cathodoluminescence (CL). Ages between 22-11 Ma ($n = 130$; Fig. 4) were obtained from matrix zircon in all samples, and from inclusions in omphacite and garnet in the eclogites. These results were obtained from rims, patches along edges and cloudy mantles in zircon. In the felsic rocks, zircon additionally demonstrated an inherited age component of 767 ± 27 Ma in oscillatorily zoned cores (DK42; $n = 11$, MSWD = 1.2; Fig. 4F), and $^{206}\text{Pb}/^{238}\text{U}$ ages of 55.3 ± 1.8 (DK33, $n = 2$), 37.3 ± 1.7 (DK20, $n = 2$), and 30.7 ± 1.9 Ma (DK42, $n = 5$, MSWD = 1.1; Fig. 4; Appendix B). Of the Cenozoic ages, the oldest was obtained from zircon included in kyanite (Fig. 3E), whereas the other ages are from grains included or partially embedded in garnet, kyanite, and sanidine. Eclogite DK60 yielded $^{206}\text{Pb}/^{238}\text{U}$ ages of 103.3 ± 2.2 ($n = 6$, MSWD = 0.8), 53.1 ± 1.1 ($n = 4$, MSWD = 0.2; Fig. 1A), and 41.8 ± 0.7 Ma ($n = 4$, MSWD = 0.5; Fig. 3A, 4G). The first and last population are also represented in DK03 zircon through a single concordant age each (Fig. 4A), with the older age found in a single zircon core. Most zircon from eclogites provided distinct age clusters in the Miocene; a large proportion of analyses for sample DK60 overlap with the age of eruption (12.5-11.1 Ma; $n = 18$; Fig. 4H).

In the felsic rock samples, zircon $(\text{Gd}/\text{Yb})_{\text{N}}$ is typically 0.15-1.2, with Miocene zircon showing the highest values. $\text{Eu}_{\text{N}}/\text{Eu}^*$ does not correlate with $(\text{Gd}/\text{Yb})_{\text{N}}$ or age. Two felsic samples that show a range in these values (DK20 and DK42) are included in figure 5. In contrast to felsic samples, the two eclogites exhibit a correlation between age and $(\text{Gd}/\text{Yb})_{\text{N}}$ (0.022-0.053) and $\text{Eu}_{\text{N}}/\text{Eu}^*$ for zircon. Sample DK60 provided a range of $^{206}\text{Pb}/^{238}\text{U}$ ages. We used the data from this sample to investigate trends of REE systematics with age (Fig. 6A). In this sample, zircon older than 21 ± 3 Myr shows low $(\text{Gd}/\text{Yb})_{\text{N}}$ (0.022-0.053) and $\text{Eu}_{\text{N}}/\text{Eu}^*$ (0.10-

0.33; increasing with decreasing age). In contrast, zircon formed between 14.4-11.1 Ma exhibits (Gd/Yb)_N up to 0.78 and high Eu_N/Eu* of 0.54-0.82 (Figs. 5, 6A). A similar systematic is observed for eclogite DK03, where highest Eu_N/Eu* (0.83) occurs at 15.0 ± 0.3 Ma (Fig. 5A).

Zircon Ti concentrations are 9-42 ppm in felsic rocks and 8-52 in mafic rocks. Titanium concentrations are low (<20 ppm) in zircon older than 20 Ma in all samples. Zircon grains in felsic rocks contain up to 42 ppm in those with ages between 20-15 Ma, and 18-22 ppm in those yielding 15-12 Ma. Zircon of 20-14 Ma in eclogites contains <20 ppm. Younger zircon shows elevated Ti concentrations. Zircon that yielded 11.2 ± 0.2 Ma – identical to the age of eruption (11.3 ± 0.2 Ma; Hacker et al. 2005) – in eclogite sample DK60 contained up to 52 ppm Ti (Fig. 4H).

4.2. Monazite

Monazite was found in all samples, except the eclogites. Concordant monazite ²⁰⁶Pb/²³⁸U and ²⁰⁸Pb/²³²Th ages yielded a Cretaceous inherited component (99.1 ± 2.6 Ma, *n* = 8); 39.7 ± 0.7 Ma (*n* = 10; Figs. 3C, 7) for inclusions in garnet in DK42, and 39.0 ± 0.3 Ma (*n* = 28) for inclusions in garnet and kyanite in DK33; 21.5 ± 0.2 Ma for a texture reflecting biotite melting (DK62; Fig. 3F); and a range of near-concordant age clusters between 22-11 Ma (Appendix C). Slight over-concordance is attributed to excess amounts of ²⁰⁶Pb, which can occur in young monazite due to the presence of initial ²³⁰Th (Schärer, 1984). As such, ²⁰⁸Pb/²³²Th ages are used in the evaluation of the monazite data. Each age population identified in the zircon dataset is present in the monazite data. Interestingly, there is a lack of overlapping ages for different chronometers within samples underscoring the importance of our multi-mineral approach. Correlations between monazite age and texture resemble those observed for zircon, with 40-36

Ma monazite typically being included in garnet and kyanite, and younger monazite occurring in the matrix (Fig. 3). Values of $(\text{Gd/Yb})_{\text{N}}$ generally increase with decreasing age (Appendix C). Similar to the young zircon in eclogites, monazite dated at 12.0 ± 1.6 Ma in the compositional intermediate DK19 lacks a strong negative Eu anomaly ($\text{Eu}_{\text{N}}/\text{Eu}^* = 0.43\text{-}0.62$; Fig. 6B).

4.3. Rutile

Rutile is common in all samples in textural equilibrium with, and as inclusions in, minerals of the main rock-forming assemblage (e.g., garnet, clinopyroxene in eclogites; garnet, feldspars, kyanite in felsic granulites). Rutile thermochronology exclusively provided lower-intercept dates that were identical to the established age of eruption (11.3 ± 0.2 Ma; Hacker et al. 2005; Fig. 8). The weighted mean of all concordia intercept results is 11.17 ± 0.06 Ma ($n = 201$; Appendix D), with all discordant analyses caused by common Pb. The data show the exceptional potential of rutile in xenoliths to date eruptions of volcanic rocks; no data-point shows any evidence for inherited radiogenic Pb. Rutile Zr concentrations are between 1,020-1,760 ppm and 1,750-2,440 ppm for eclogites and felsic granulites, respectively (Appendix A). For the possible range of pressures (0.9-2.5 GPa), rutile in felsic rocks yielded 820 ± 30 °C to 960 ± 25 °C. In the eclogites, rutile is a constituent of the high-pressure assemblage for which pressures of 2.5-2.8 GPa were estimated (Hacker et al., 2005). Assuming these pressures, Zr-in-rutile thermometry yielded 890 ± 40 °C ($n = 87$).

5. Discussion

5.1. Evolution of the lower-crustal xenoliths in time and space

Rutile in all samples provided age data corresponding exclusively to the eruption of the xenolith host lava (11.17 ± 0.06 Ma). This demonstrates the reliability of the rutile U-Pb thermometer and highlights the potential of using this system in xenoliths to date volcanic processes. Zircon and monazite yielded a very different set of age data, comprising a series of age populations before and, for the most part, during the Cenozoic. Together with the trace-element characteristics of these phases, this record provides a detailed look into the petrological processes that occurred within the Pamir lower crust during the Cenozoic. The comprehensive record from zircon is particularly useful. The concentration of Ti in this mineral is a function of temperature (T) and to a lesser extent pressure (P) of zircon (re-)crystallization at a given activity of TiO_2 and SiO_2 (Ferry & Watson, 2007). Zircon shows a very strong retentivity for both Pb and Ti, even at ultrahigh temperatures (e.g., Kooijman et al., 2011). This is evidenced here by the preservation of a wide range of age populations and Ti concentrations, even in single crystals (Figs. 3, 9). As such, both $^{206}\text{Pb}/^{238}\text{U}$ ages and Ti-in-zircon concentrations reflect the crystallization or recrystallization of zircon, and can be linked directly to reconstruct the evolution of P - T conditions with time (t). This is particularly powerful in cases such as the one presented in this study, in which the geochronological record is very detailed and includes a range of distinct age populations, each with a different Ti signature. We thus resolve trends in U-Pb ages versus Ti-in-zircon concentrations for the Pamir xenoliths (Fig. 9) and explore their implications with regards to dynamics and evolution of the deep crust in the region.

An important point to consider when estimating temperatures from Ti concentrations is knowledge of the activity of TiO_2 . This may be difficult to estimate for mafic rocks, because rutile would not likely have been present at low pressures. Rutile stability can only be assumed with certainty for the eclogitic stage. The value of $\text{Eu}_\text{N}/\text{Eu}^*$ in zircon provides an opportunity to

constrain the timing of this stage, because it is used to trace eclogitization through the breakdown of Eu-rich plagioclase during progression towards high pressure conditions (Rubatto, 2002). In our samples, $\text{Eu}_\text{N}/\text{Eu}^*$ is highest at c. 14 Ma (Fig. 5A). The concurrent increase in $(\text{Gd}/\text{Yb})_\text{N}$ around this age could be related to concomitant growth of HREE-sequestering omphacite and garnet. These observations indicate that eclogite facies conditions were reached by 14 Ma. Applying the Ti-in-zircon thermometer and assuming peak- P of c. 2.7 GPa for eclogitic zircon yielded 930 ± 35 °C (14.4-12.5 Ma), which is within uncertainty of the Zr-in-rutile temperatures obtained from the same assemblage (890 ± 40 °C). Ti-in-zircon thermometry of 11.2 ± 0.2 Ma eclogitic zircon yielded 980-1,090 °C (after Ferry & Watson, 2007; assuming peak P). The latter range is consistent with previous estimates of the magmatic temperature of the ultrapotassic host rocks (1,000-1,100 °C; Hacker et al., 2005) and indicates likely selective zircon (re-)equilibration with infiltrating melt. For pre-14 Ma zircon, activity of $\text{TiO}_2 < 1$ is possible and cannot be reliably corrected for. Therefore, to accurately constrain $\text{Ti}_{\text{zircon}}(\text{Age})$ before 14 Ma, we focus on the felsic rocks. Rutile is stable in felsic systems, even at low pressure, and is found as part of the main rock-forming assemblage in the felsic samples. These observations indicate that rutile has been part of the rock, even during the early stages of Cenozoic evolution where the rocks would not yet have reached high-pressure conditions. It is possible that TiO_2 activity may have been < 1 in micro-domains that were chemically isolated as a result of slow matrix diffusion. This can explain why, even in felsic rocks, Ti-in-zircon concentrations may show a range; lower Ti concentrations for a given age population could reflect local TiO_2 undersaturation, in which case the highest concentrations would be most representative of the P - T conditions subjected to the rocks at a given time. However, we do not make assumptions in this regard and treat the zircon dataset from felsic rocks as a whole when constraining $\text{Ti}_{\text{zircon}}(\text{Age})$ and its uncertainty.

To constrain the zircon evolution in time, the Ti-in-zircon concentrations were plotted against the $^{206}\text{Pb}/^{238}\text{U}$ age of the same CL zone. An average $\text{Ti}_{\text{zircon}}(\text{Age})$ curve was fitted through Ti-in-zircon data, applying uncertainty margins that include 95% of all data (Fig. 9). Subsequently, Ti-in-zircon isopleths corresponding to the extremes of this curve were established and plotted in P - T space (Fig. 10). These extremes are: 1) the Ti concentrations in zircon until c. 40 Ma (13 ppm), 2) the highest Ti concentration before the eruption stage (33 ppm), and 3) the highest Ti concentration upon eruption (52 ppm). A P - T and time evolution that represents that of the Pamir lower crust should satisfy these criteria to accurately represent the data obtained. One first-order deduction is that the low Ti concentrations of >30 Ma zircon (< 15 ppm) indicate significantly lower temperature conditions than estimated for the xenoliths so far. The intersection between the Ti-in-zircon isopleths and the Cenozoic (50 – 25 Ma) geotherm of the Pamir deep crust as established through research on the high-grade cores of the Pamir gneiss domes (Fig. 10; e.g., Schmidt et al., 2011; Stearns et al., 2015) suggests that the rocks resided at 720-750 °C and 0.6-0.9 GPa. These conditions are very similar to the peak conditions known for the local middle crust at or before 24-20 Ma (650–800 °C and 0.7–1.1 GPa; Schmidt et al., 2011; Stearns et al., 2015). This consistency indicates that the rocks represented by the xenoliths and represented by the gneiss domes underwent a common evolution prior to 30 Ma. Consistent with this inference, the 42-37 Ma ages obtained from zircon in prograde kyanite and garnet (Fig. 3) coincide with Lu-Hf garnet ages obtained from felsic gneisses in the most deeply derived parts of the domes (37.3 ± 0.5 Ma; Smit et al., 2014).

To reconstruct a realistic P - T - t path for the analyzed rocks, we first established a continuous $P(T)$ curve extending from the pre-Neogene geotherm (Schmidt et al., 2011) with uncertainty margins that was bracketed by the previously established Ti-in-zircon isopleths (yellow field in

Fig. 10). Criteria for this curve to cover were: 1) the range of P - T conditions known for the Pamir middle crust (650–800 °C and 0.7–1.1 GPa; Schmidt et al., 2011; Stearns et al., 2015); 2) the P - T conditions of the rocks shortly before their entrenchment in the ultrapotassic magma (Ti-in-zircon temperature of 930 ± 35 °C at 2.50-2.65 GPa); 3) the isopleths representing the maxima of the average Ti versus $^{206}\text{Pb}/^{238}\text{U}$ age relationship and its uncertainty margins (Fig. 9). To add age constraints to this curve, we solved $\text{Ti}_{\text{zircon}}(\text{Age})$ (Fig. 9) for age using the thermometric calibration – $\text{Ti}(P,T)$ – and $P(T)$ as suggested above. We did so by numerically tracing Ti concentrations with time plus their uncertainty (Fig. 9) at 2 Myr intervals and constraining the corresponding intersection of the P - T curve with the isopleths indicated by these concentrations. This yields a continuous modelled P - T - t curve as shown in Fig. 10. Although P and T may not co-vary in settings where tectonic processes are fast, we avoided any additional complexity such as intermittent cooling or decompression. No such complexity is yet known; the proposed curve represents the simplest model that explains all available data.

The viability of the P - T - t curve can be tested by comparing the predicted and observed ages for important petrological processes. The $P(T, \text{Age})$ evolution curves intersect the biotite solidus for metapelites (840-850 °C at 1.0-1.6 GPa (Vielzeuf & Schmidt, 2001) and the vapour-absent solidus for garnet-quartz amphibolite (850 °C at 1.0-1.5 GPa; Wolf & Wyllie, 1994) between 22-19 Ma, and indicate the introduction of omphacite in mafic rocks at 880-980 °C and 1.8-2.5 GPa between 18-12 Ma (isopleths from mineral compositions; Hacker et al., 2005). These constraints agree with what is observed; monazite and zircon in biotite breakdown textures and other melt vestiges in the felsic granulites formed at 21.5-19.1 Ma (Fig. 3F), whereas zircon in the eclogites indicates eclogite-facies plagioclase breakdown at 14.4-12.5 Ma (Figs. 5A, 6). These

observations support the validity of the proposed $P(T, Age)$ relationship to accurately describe the progression of the rocks towards mantle depth.

5.2. Detailed Record of Crustal Foundering

The evolution of the rocks sampled by the xenoliths – established here through detailed and texturally controlled multi-mineral micro-analysis – is indistinguishable from that established for the mid-crustal complexes exposed across the Pamir before c. 30 Ma. After this time, however, the evolution of these two seemingly related sections of the Pamir crust became very different. The mid-crustal rocks entered a >10-Myr period of exhumation and cooling (Schmidt et al., 2011; Stübner et al., 2013b; Stearns et al., 2013; 2015). In striking contrast, the xenoliths indicate that the lower crust was instead heated to the point where felsic lithologies melted. These lithologies developed into the dense refractory residues with up to 55 vol% garnet as observed among the xenoliths. Approximately from this point on, downward transport accelerated with rates increasing by 3-6 times over a 10 Myr period (Fig. 10). By 14.4-12.5 Ma, the material descended beyond the present-day Moho depth (65 ± 5 km; Mechie et al., 2012) at a rate of 4.0-5.7 mm yr⁻¹ and continued on for at least another 20 km. This acceleration is ascribed to density inversion, with the dense composite of eclogite (3.2-3.5 g cm⁻³) and refractory felsic granulite (2.9-3.2 g cm⁻³) foundering into the highly metasomatized Asian mantle wedge (3.00 g cm⁻³; densities after Hacker et al., 2005). The process is captured in the geochemical record of a fraction of this material, which became entrained in mantle-sourced magma that pierced the crust at the time when the material had reached sub-Moho depth.

The history revealed here directly captures foundering of deep Asian crust (Ducea et al., 2003; Gordon et al., 2012) north of India's continental subduction and indicates that this process

enables the introduction of crustal material into the mantle at tectonic rates in an active mountain belt. This style of crustal recycling is different from continental subduction (e.g., Kufner et al., 2016), and has a different impact on mantle geochemistry. Continental subduction involves downward transport of large terranes. With eclogitisation lagging behind changes in P - T (Austrheim, 1987), and much of these terranes remaining internally undeformed and buoyant (e.g., Spencer et al., 2013), they are efficiently exhumed in the subduction zone as a rigid body (Chemenda et al., 1995) or relaminated at the base of the upper plate (Hacker et al., 2011). In the case of foundering, crustal material sinks into the mantle, which introduces vestiges of evolved, enriched crustal material, with its characteristically high $^{87}\text{Sr}/^{86}\text{Sr}$ and U/Pb, and variably low $^{143}\text{Nd}/^{144}\text{Nd}$ and $^{176}\text{Hf}/^{177}\text{Hf}$, into otherwise depleted mantle and magmatic source regions. With Sr, Pb, Nd, and Hf being at least 10 times more enriched in the lower crust relative to primitive mantle, a ‘domestic’ crustal contamination effect in the mantle due to the presence of foundered crust may be substantial. Felsic and mafic crustal xenoliths similar to those found in the Pamir were also observed among Cenozoic igneous complexes in the Kunlun Shan (N-Tibet; Jolivet et al., 2003), the Qiangtang terrane (central Tibet; Hacker et al., 2000; Ding et al., 2007) and just north of the Yarlung–Tsangpo suture zone (S-Tibet; Chan et al., 2009). These xenoliths are very similar to those in the Pamir in that they are hot, dry, dense, and hosted in mantle-derived ultrapotassic magmas. Interestingly, these and other Cenozoic igneous rocks show a large spatial and temporal variability in radiogenic isotope compositions, which is not always sufficiently accounted for by plate-tectonic processes. For instance, $^{143}\text{Nd}/^{144}\text{Nd}$ and $^{87}\text{Sr}/^{86}\text{Sr}$ for 60-0 Ma magmas vary between depleted-mantle compositions to those representing highly evolved crustal rocks exposed in the High Himalaya, and are not always isotopically consistent for a given location and age (Ding et al., 2003; Chung et al., 2005). Local contamination of the sub-

continental mantle with foundered crustal material could explain these observations without requiring secular changes in regional plate dynamics.

6. Conclusions

In situ accessory mineral micro-analysis of Pamir crustal xenoliths enabled a detailed time-resolved reconstruction of the burial of crustal rocks to mantle depths. Rutile thermochronology exclusively dated eruption (11.17 ± 0.06 Ma), demonstrating its ability to date magmatic processes. Conversely, zircon and monazite reveal a series of distinct age clusters between 55-11 Ma. The lack of overlapping ages for different chronometers within samples show the effectiveness of a multi-mineral approach. The age data for zircon and monazite combined with REE concentrations and Ti-in-zircon temperatures track the history of the rocks at a million-year resolution. The data reveal that the rocks resided at 30-40 km depth along a stable continental geotherm at 720-750 °C until 24-20 Ma. Subsequently, the material descended rapidly, accelerating from 0.9-1.7 mm yr⁻¹ to 4.7-5.8 mm yr⁻¹ within 10-12 Myr, and became buried beyond Moho depth at 14-13 Ma and 930 ± 35 °C. The data reflect the foundering of differentiated deep-crustal fragments (2.9-3.5 g cm⁻³) into a metasomatized and less dense mantle wedge. This recycling process could explain the variability in the degree of crustal contamination of mantle-derived magmatic rocks in the Pamir and neighbouring Tibet without requiring secular changes in plate dynamics. Our new approach in constraining the burial history of rocks provided the first time-resolved record of this crustal-recycling process in an active orogen.

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Figure Captions

Fig. 1: Geological map of the Pamir with the Dunkeldik location in the inset. The map is based on that of Schmidt et al., (2011).

Fig. 2: Plane-polarized light photomicrographs of the samples analyzed here. All photomicrographs are of the thin sections subjected to in-situ analysis in this study.

Fig. 3: Backscatter electron (BSE) and cathodoluminescence (CL) images of zircon and rutile in eclogite samples DK60 (A) and DK03 (B; BSE image with CL overlay for zircon), and for zircon, monazite, and rutile felsic granulites (C, DK42; D, DK33; E, DK20; F, DK62). Monazite in DK62 (F) is included in sanidine formed by the dehydration melting of biotite. Grt – garnet, omp – omphacite, sd – sanidine, zrn – zircon, rt – rutile, mnz – monazite, ky – kyanite, bt – biotite.

Fig. 4: Zircon U-Pb data plotted in Tera-Wasserburg (A-E, G, H) and Wetherill (F) concordia diagrams. Cited age results represent the U-Pb concordia intercept age of corresponding data clusters. The data for samples DK33 and DK19 were few in number due to low zircon abundance and were hence merged into diagram B. The 31 ± 2 Ma age population in diagram E has a lower upper intercept, which indicates an inherited age component. The older age data for DK42 are shown in diagram F. The <12 Ma age data for DK60 are shown colour-graded according to Ti concentration if h. The data show an irregular increase of Ti with time leading up to eruption at c. 11 Ma.

Fig. 5: REE systematics plotted versus U-Pb concordia intercept age for the samples that yielded a substantial spread in ages as well as REE composition. The diagrams show Eu_N/Eu^* (A) and $(\text{Gd}/\text{Yb})_N$ (B) versus age. Both show an increase after c. 24 Ma.

Fig. 6: A) Chondrite-normalized REE plot for zircon in eclogite sample DK60. Zircon equal to or younger than 14.4 Ma exhibits recovery of the negative Eu anomaly and shows markedly

lower Gd/Yb, indicating omphacite stabilization. B) Chondrite-normalized REE plot for monazite in samples DK19, DK20, DK42. The negative Eu anomaly that is common among monazite of 17.9 Ma or older is nearly absent for spots yielding 12.0 Ma.

Fig. 7: Monazite $^{206}\text{Pb}/^{238}\text{U}$ and $^{208}\text{Pb}/^{232}\text{Th}$ data plotted in U-Th-Pb concordia space for the four felsic samples. Cited age results represent the weighted mean $^{208}\text{Pb}/^{232}\text{Th}$ age of corresponding data clusters.

Fig. 8: Rutile U-Pb data plotted in Tera-Wasserburg concordia space. All data cluster around the previously established age of eruption at c. 11 Ma.

Fig. 9: Titanium-in-zircon boxplots vs. age. The Ti(Age) curve was constructed on the basis of data from felsic samples only. The curve plots through the medians of Ti and U-Pb age data between 40-11 Ma. Uncertainty limits include 95% of all data.

Fig. 10: Reconstruction of the Pamir P - T evolution. Important reactions recorded by textures and assemblages in the various samples are marked with roman numbers as follows: I) albite_{pl} ↔ jadeite_{cp} + quartz on the basis of highest activity of albite (a_{ab}) estimated from measured mineral compositions (Hacker et al., 2005); II) albite_{pl} ↔ jadeite_{cp} + quartz on the basis of lowest a_{ab} ; III) biotite + albite + kyanite + quartz ↔ garnet + sanidine + hydrous granitic melt; IV) hornblende + plagioclase ↔ clinopyroxene + garnet + hydrous melt. Titanium-in-zircon isopleths in green are marked with numbers denoting corresponding Ti-in-zircon concentration at rutile saturation. Coloured contours within the P - T path denote age in Ma.

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10