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(Mis)Identification of magmatic and exhumation ages by detrital zircon U-Pb and He double dating: a case study from the Bergell-Gonfolite system (European Alps)

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Abstract

Reliable interpretation of detrital thermochronometric datasets requires correct attribution of these ages as either the record of exhumational cooling or the record of post-magmatic cooling independent of tectonic or erosional exhumation. A classic approach for identifying magmatic cooling ages is through double dating leveraging paired high- and low-temperature geo/thermochronologic systems, which should yield, within error, indistinguishable ages from the same grain. On the contrary, low-temperature thermochronometric ages that are younger than their corresponding crystallization ages are mostly invariably interpreted to record exhumation. Here, we test this last assumption by applying a detrital zircon U-Pb and (U-Th)/He double-dating approach to a well-constrained source-to-sink system in the southern European Alps, archiving the progressive unroofing of the Bergell-Novate volcanic-plutonic complex and associated country rocks. We depth-profile U-

Pb dated unpolished detrital zircon grains and performed (U-Th)/He analysis on non-volcanic/plutonic grains. Of the double-dated grains, 40% yielded (U-Th)/He ages overlapped in age with Bergell-Novate magmatism (32-30 and 27-24 Ma). Despite yielding (U-Th)/He ages younger than their corresponding U-Pb ages, these ages record post-magmatic cooling within the contact aureole and not exhumation. Our findings indicate that only a fraction of the grains yielding magmatic He ages can be identified by double dating, and that the assumption that all the remaining grains constrain exhumation can be potentially misleading. Finally, we present improved criteria for the interpretation of detrital zircon thermochronometric double-dating results and conclude that many previous interpretations based on a classic double-dating approach should be reconsidered in syn-magmatic orogenic systems.

Keywords: Zircon double dating; detrital thermochronology; post-magmatic cooling; exhumation; lag-time analysis; European Alps.

1. Introduction

Detrital thermochronometric analyses of apatite and zircon grains are increasingly employed to develop quantitative models of deep-time landscape evolution and constrain rates of erosional exhumation (e.g., Govin et al. 2020; Lang et al. 2020; Lossada et al. 2020; Stockli and Najman 2020). Many of these studies are based on the lag-time approach, which compares thermochronometric ages recorded by mineral grains in sedimentary strata to the corresponding stratigraphic ages, which are independently determined (e.g., Garver et al. 1999; Bernet et al. 2006). However, a crucial assumption for the validity of this application is the correct discrimination between thermochronometric ages, recording cooling due to exhumation, i.e., the motion of parent rocks towards Earth's surface (England and Molnar 1990) and thermochronometric ages that record cooling independent of exhumation, such as post-magmatic cooling of volcanic and shallow-level plutonic rocks (Malusà and Fitzgerald 2019a). A classic approach for the identification of magmatic crystallization ages in detrital thermochronometric studies is by double dating, combining U-Pb and low-temperature thermochronometric dating on the same mineral grain (Carter ad Moss 1999; Reiners et al. 2005) (Fig. 1). Magmatic zircon from volcanic or shallow-level plutonic rocks should display within error indistinguishable U–Pb and fission-track or (U–Th)/He ages (ZUPb, ZFT and ZHe hereafter), due to rapid post-crystallization cooling in the upper crust where country rocks are at temperatures below closure isotherms or the partial annealing (or retention) zones of the ZFT or ZHe systems (Malusà et al. 2011; Saylor et al. 2012). Zircon grains crystallized at high temperatures (i.e., greater depths) record cooling during subsequent exhumation and hence should yield ZFT and ZHe ages younger than their corresponding ZUPb ages and may reveal the long-term exhumation history of the source terrane. These cooling ages can be leveraged in the lag-time approach, provided that a range of fundamental assumptions are met and can be properly evaluated (Malusà and Fitzgerald 2020).

Implicit assumption of many detrital double-dating thermochronology studies is that all the grains yielding magmatic ages can be readily identified by their ZUPb=ZFT or ZUPb=ZHe fingerprint (1 in Fig. 1), and that all the remaining grains (2 and 3 in Fig. 1) provide meaningful constraints on the exhumation history of the source terrane (e.g., Najman et al. 2010; Stevens et al. 2013; Jourdan et al. 2013, 2018; Bootes et al. 2019; Lu et al. 2020; Pujols and Stockli 2021). However, this may be an inappropriate assumption in magmatic terranes or at least an oversimplification (Reiners et al. 2005). In this study, we explore the possibility that a portion of the magmatic ages in detrital thermochronometric dataset, for example those "magmatic ages" recording country-rock cooling within a contact aureole, might be systematically overlooked by a double-dating approach with major implications for lag-time interpretations in certain geologic settings. To explore this issue and its implications, we apply ZUPb and ZHe double dating to detrital zircon grains of the Gonfolite Group, an Oligocene-Miocene proximal foreland basin sedimentary succession largely derived from erosion of the Bergell/Bregaglia volcanic-plutonic complex and associated country rocks (Wagner et al. 1979; Bernoulli et al. 1993). The Bergell-Gonfolite system is an ideal field laboratory for our study because its mineral-age stratigraphy, i.e., the specific combinations of crystallization and exhumation ages of different thermochronometers at different depths in the crust, is extremely well constrained (Malusà et al. 2011, 2016; Fitzgerald et al. 2019).

We dated with the ZHe method, zircons of the Gonfolite Group with Paleozoic U-Pb ages from the zircon rim. In this way, we could be sure that the grains come from the country rock and not from the Bergell pluton and associated magmatic rocks. As these zircons come from stratigraphic units in which it is known from the literature that the AFT ages in country-rock cobbles are pre-intrusion, and because the AFT system is a lower temperature chronometer than the ZHe system, the expectation was to obtain pre-intrusion ZHe ages. Despite this, we found that 40% of the grains show ZHe ages overlapping with the age of the intrusions. We interpret these ZHe ages as recording post-magmatic cooling within the contact aureole of the plutons and not exhumation.

Based on our new results, we define improved criteria for correct identification of post-magmatic and exhumational cooling ages in detrital thermochronometry studies. These criteria are illustrated by applying them to high-quality published double-dating datasets from the literature. Our findings suggest that the interpretation of many previously published detrital thermochronology datasets should be carefully reevaluated and potentially reconsidered.

2. Geologic and thermochronologic setting

2.1. The Bergell-Gonfolite source-to-sink system

The Bergell pluton is exposed in the Central Alps and was intruded at 32-30 Ma into Alpine metamorphic rocks (von Blanckenburg 1992; Ji et al. 2019). It consists of a tonalitic–granodioritic main body and a tail-shaped feeder zone parallel to the Insubric Fault (Fig. 2a). The contact aureole of the pluton is largely eroded away and is exclusively preserved around its eastern part, where it is 1.5-2 km wide (Trommsdorff and Connolly 1996). Estimates of unroofing depth provided by hornblende geobarometry (Davidson et al. 1996) range from ca 20 km in the Bergell main body, to ca 26 km in its western tail, consistent with K-Ar biotite ages that are systematically younger than the Bergell intrusion age (Villa and von Blanckenburg 1991). A smaller younger pluton, the Novate granite, was intruded on the western side of the Bergell main body at 27–24 Ma (Liati et al. 2000, Ji et al. 2019) (Fig. 2a).

Progressive erosion of these magmatic rocks together with their country rocks provided detritus to the Oligocene–Miocene gravity flow deposits of the Gonfolite Group that are exposed in the proximal southern foreland basin of the Alps (Wagner et al. 1979; Giger and Hurford 1989; Malusà et al. 2011) (Figs. 2a, 3a) and to the more distal foredeep units now accreted in the Northern Apennines (Malusà et al. 2016). The linkage between the source and the sink, long recognized by compelling lithological and geochronological evidence (Wagner et al. 1979; Giger and Hurford 1989), is confirmed by (reverse) mineral-age stratigraphy (Malusà and Fitzgerald 2019b) and by Hf isotopic composition of magmatic detrital zircon grains (Ji et al. 2019; Lu et al. 2019) (Fig. 2b). Widespread volcanic detritus at the base of these stratigraphic successions (Fig. 3a) indicates that volcanic eruptive centers were present atop the Bergell magmatic complex (Malusà et al. 2011; Anfinson et al. 2016). Volcanic detritus was initially funnelled into submarine canyons, possibly corresponding to morphologic depressions now occupied by pre-alpine lakes (Garzanti and Malusà 2008) and bypassed the South Alpine units triggering southward-directed gravity flows. The first significant detrital pulse in the Gonfolite basin is dated biostratigraphically at ~25 Ma (Gelati et al. 1988) and appears to mark the onset of rapid unroofing of the Bergell area. This main erosional pulse was delayed by ~5 Ma relative to the main magmatic pulse, which was associated with negligible erosion and starved sedimentation in the final sink (Garzanti and Malusà 2008) (Fig. 3a). Oligocene-Miocene erosion was less prominent in the South Alpine units compared to the Axial Alps, as attested by ZFT ages ranging from 223 to 49 Ma, indicative of minor Cenozoic exhumation (Bertotti et al. 1999) (Fig. 2b).

2.2. Mineral-age stratigraphy

Published apatite and zircon fission-track, biotite K-Ar, and zircon U-Pb ages in cobbles in the Gonfolite Group derived from the Bergell and Novate plutons and from associated country rocks are summarized in Fig. 3b. These thermochronometric ages define a complex but fully predictable mineral-age stratigraphy, which allows definition of three distinct mineral-age units (A to C in Fig. 3) characterized by specific combinations of stationary and moving age peaks (Malusà and Fitzgerald 2020).

In unit A, cobbles derived from the Bergell and Novate plutons (blue solid dots) yield syn-intrusion apatite fission-track (AFT) ages that record the cooling history of the magma, whereas country-rock cobbles (blue open dots) yield older AFT ages that record the pre-intrusion history of the country rock. AFT ages in units B and C are the same for plutonic and country-rock cobbles and define a moving age peak that gets increasingly younger upsection (Fig. 3b). These AFT ages, either marked with solid or open blue dots, are systematically younger than the intrusion age of the Bergell and Novate plutons and record the erosional exhumation of the plutons and surrounding country rocks.

ZFT analysis in units A and B yield syn-intrusive ZFT ages in cobbles derived from the Bergell and Novate plutons (purple solid dots), and pre-intrusion ZFT ages in cobbles derived from the country rocks (purple open dots). The first moving age peak defined by ZFT ages, recording the erosional exhumation of the plutons and surrounding country rocks, only appears in mineral age unit C, as denoted by the young (28-24 Ma) ZFT ages in country-rock cobbles (purple open dots in Fig. 3b).

Some authors have interpreted the thermochronometric ages in the Gonfolite Group within a simplistic thermochronological framework by considering only the youngest age peak (e.g., Bernoulli et al. 1993; Carrapa, 2009). This led to suggest superfast early Oligocene erosion of the Bergell pluton at a rate exceeding 6 mm/a (Giger and Hurford, 1989; Carrapa and Di Giulio, 2001), which raised the paradox of superfast erosion without detrital counterparts in the adjacent foreland basins (Garzanti and Malusà 2008; Malusà et al. 2011). Such interpretation is demonstrably incorrect, not only because it clashes against compelling stratigraphic evidence (see accumulation rates in Fig. 3a), but also because it fails to explain a significant part of the detrital thermochronology dataset, i.e., all the data points located to the right of the vertical grey bars in Fig. 3b, representing pre-intrusion mineral ages in country-rock cobbles. More details on these topics and on the mineral-age stratigraphy on the

Gonfolite Group can be found in Malusà et al. (2011), Fitzgerald et al. (2019), and Malusà and Fitzgerald (2019a, 2020).

3. Methods

For our study, four sandstone samples (S1 to S4) were collected for detrital ZUPb (Malusà et al. 2016) and ZHe analyses (this work) from the lowermost part of the Gonfolite Group, corresponding to the mineral age unit A and lower part of mineral age unit B (Fig. 3a). These stratigraphic levels were fed by progressive erosion of the uppermost levels of the Bergell volcanic-plutonic complex and surrounding country rocks (Giger and Hurford 1989; Malusà et al. 2011) and are characterized by a progressive decrease in volcanic detritus and a progressive increase in plutonic detritus moving up section (Fig. 3a). AFT and ZFT ages on magmatic and country rock cobbles in these stratigraphic levels are independently known (Fig. 3b). Because the temperature range corresponding to the partial retention zone of the ZHe system (~180-140ºC; Wolfe and Stockli 2010) is in between the temperature ranges of the partial annealing zones of the ZFT (~240-180ºC) and AFT (~115-60ºC) systems (e.g., Gleadow and Fitzgerald 1987), one would expect that the analyzed zircon grains should yield either ZHe ages constraining the age of magmatism or ZHe ages constraining the pre-intrusion history of the country rock, but not the post-intrusive erosional exhumation history. An additional sample (S5) consisting of a single cobble of country rock was collected from the mineral age unit A to evaluate the intra-sample dispersion of single-grain ZHe ages.

All mineral separations and isotopic analyses were carried out in the UTChron Facility in the Department of Geological Sciences at the University of Texas at Austin. Samples were crushed and ground and detrital zircon grains were separated using standard heavy mineral and magnetic separation techniques. For ZUPb LA-ICP-MS analysis, all detrital zircon grains were mounted without polishing on one-inch round epoxy pucks with double sided tape, which made it possible to perform subsequent ZHe analysis on the very same grains. All grains were depth-profile ZUPb dated using a Photon Machines Analyte G2 ATLex 300si ArF 193 nm Excimer Laser, equipped with a

Helix two-volume ablation cell, and a Thermo Fisher Element2 single collector, magnetic-sector ICP-MS. The reference material GJ1 (Jackson et al. 2004) was used as a primary reference and Plešovice (Sláma et al. 2008) was used as a secondary reference to monitor data quality. $^{206}Pb/^{238}U$ ages are reported for ages younger than 1000 Ma and $^{207}Pb/^{206}Pb$ ages are reported for ages older than 1000 Ma. For further details on the ZUPb analytical procedures see Malusà et al. (2016), where ZUPb ages for samples S1 to S4 were originally presented.

U-Pb dated zircon grains with ablation pits were selected for ZHe analysis following the criteria outlined in Farley (2002) and Hart et al. (2017). We exclusively chose grains for double-dating with pre-Alpine ZUPb ages not derived from the Bergell complex or other Periadriatic magmatic rocks (shown in Fig. 2b). We also avoided rounded grains that may have experienced removal of the grain rim by abrasion during sediment transport with consequences for data interpretation, either due to overcorrection for α-ejection that may lead to ZHe ages that are too old (Reiners 2005), or due to unwanted comparison between ZHe ages recording magmatic crystallization and ZUPb ages from inherited xenocrystic cores, which may lead to misinterpretation of magmatic ages in terms of exhumation (Malusà and Garzanti 2019).

Selected grains were morphometrically characterized and packed into a Pt packet for in-vacuo laser heating (~1300°C for 10 min) and complete degassing. Cryogenically purified and gettered He was spiked with a ^{3}He tracer and analysis by quadrupole mass spectrometer. Degassed grains were removed from the Pt packets and dissolved using a two-step HF-HNO_3 and HCl pressure vessel digestion procedures. U, Th, and Sm concentrations were determined by isotope dilution using a Thermo Fisher Element2 single collector, magnetic sector ICP-MS. Following Reiners (2005), Fish Canyon Tuff zircons were run with unknown grains to monitor data quality. A standard error of 8% was applied to all measurements. Individual analyses were excluded if the grain was partially broken during unpacking from platinum packets or if there was evidence of incomplete dissolution. More details on the analytical procedures for ZHe analysis are described in Wolfe and Stockli (2010).

4. Results

All ZUPb ages measured in each sample by LA-ICP-MS depth profiling and the corresponding kernel density estimates are shown in Fig. 4a-e, at the bottom of each frame. The scatter plots summarize the ZUPb and ZHe ages in double-dated grains not derived from Periadriatic magmatic rocks (see Supplementary Table S1 for details). No grains plot on the ZUPb = ZHe line.

The polymodal ZUPb age distributions of samples S1 and S2, collected from mineral-age unit A, are dominated by ZUPb ages pre-dating Periadriatic magmatism. Sample S1 (Fig. 4a) is dominated by Caledonian and Precambrian ages, and a minor Variscan-Permian peak and a very small Periadriatic peak at 32-37 Ma. Grains selected for double dating were characterized by ZUPb rim ages between 282 and 457 Ma. The corresponding ZHe ages range from 29.2±2.3 Ma to 278.9±22.3 Ma.

Sample S2 (Fig. 4b) is dominated by Caledonian ZUPb ages, with abundant Precambrian and Variscan-Permian ages, and a more prominent Periadriatic peak at 30-38 Ma than in sample S1. Grains selected for double dating displayed ZUPb ages between 287 and 461 Ma with corresponding ZHe ages ranging from 28.8±2.3 Ma to 238.8±19.1 Ma.

In contrast, the polymodal ZUPb age distributions of samples S3 and S4, collected from the lower part of mineral-age unit B, are dominated by Periadriatic ZUPb ages, with only minor Variscan-Permian, Caledonian and Precambrian age peaks. In sample S3 (Fig. 4c), Periadriatic ZUPb ages exceed 60% of the dataset and range in age from 27 to 35 Ma. However, grains selected for double dating were characterized by ZUPb rim ages between 272 and 296 Ma. The corresponding ZHe ages ranged from 26.0±2.1 Ma to 32.3±2.6 Ma. In sample S4 (Fig. 4d), Periadriatic ZUPb ages exceed 70% of the dataset and range from 29 to 34 Ma. Grains selected for double dating yielded ZUPb ages between 274 and 465 Ma. The corresponding ZHe ages ranged from 24.0±1.9 Ma to 129.6±10.4 Ma.

The country rock cobble collected from mineral-age unit A (sample S5, Fig. 4e) yielded a single unimodal ZUPb age peak with a Caledonian age. Crystals selected for double dating yielded ZUPb ages between 427 and 460 Ma. ZHe ages range from 108.8±8.7 Ma to 189.4±15.1 Ma, corresponding to a central age of 146.8±8.7 Ma.

ZHe ages vs. effective Uranium concentrations (e[U]) are summarized in the diagram of Fig. 4f. Notably, many double-dated zircon grains yielded ZHe ages overlapping Bergell and Novate magmatism, irrespective of their effective Uranium concentration ranging from ~100 to >1300 ppm (dark grey dots in Fig. 4f).

5. Interpretation

According to the long-established mineral-age stratigraphy of the Gonfolite Group (Fig. 3b), zircon grains collected from mineral-age unit A and lower part of unit B should give: *(i)* ZHe ages overlapping the Bergell and Novate intrusion age, if zircon were derived from erosion of Bergell-Novate magmatic rocks; or *(ii)* ZHe ages older than the Bergell and Novate intrusions, if zircon were derived the country rocks that were not thermally overprinted by the intrusion. As all selected double-dated zircon grains from samples S1 to S4 gave ZUPb ages between 272 and 465 Ma, they were derived from erosion of the country rock. However, only a portion of these zircon grains yielded ZHe ages older than the Bergell and Novate intrusions (open purple dots in Fig. 3b), whereas a substantial portion of the ZHe ages yielded ages that overlap the Bergell and Novate magmatism (yellow solid dots in Fig. 3b). This observation provides compelling evidence for derivation of some of the double-dated zircon grains from the Bergell-Novate thermal contact aureole. Hence, these youngest ZHe ages provide no direct constraints on the exhumation rate of the country rock exposed in the source area for the sediment, but only record country-rock cooling in the contact aureole after magma emplacement. ZHe ages in a contact aureole are expected to become progressively older, due to partial resetting, moving away from the intrusion (Calk and Naeser 1973; Malusà and Fitzgerald 2019a), which may explain the occurrence of a few slightly older ZHe ages of 37-39 Ma in samples S1, S2 and S4.

Insights into exhumation rates are potentially provided by the oldest ZHe ages of the dataset, ranging from 62 to 279 Ma and largely fitting the distribution of single grain-ages in the analyzed country rock cobble, which yielded a central age of ~147 Ma (sample S5). Because detrital thermochronometric ages within a stratigraphic successions, unless reset by burial, must be equal to or older than the cooling ages now observed in bedrock within the potential source areas (Malusà and Fitzgerald 2020), these oldest ZHe ages support provenance from the core of the central Alps or from South Alpine rocks close to the Insubric Fault, yielding a ZFT age ~49 Ma. Most of the South Alpine units, instead, yielded ZFT ages from 135-177 Ma to 223 Ma in the present-day exposure level (Fig. 2a) and can be safely excluded as a potential source.

The pie charts in Fig. 3c summarize the ratio of country-rock derived zircon grains from outside the Bergell and Novate contact aureole vs those derived from within the contact aureole, which yield ZHe ages overlapping the Bergell and Novate intrusion ages. We observe that the percentage of ZHe ages reflecting country-rock cooling in the contact aureole (in yellow in Fig. 3c) progressively increases up section in the Gonfolite stratigraphic succession. This is expected because sedimentary successions record the mineral-age structure of eroded bedrock in reverse order, and contact aureoles are best developed at deeper structural levels surrounding large plutons rather than adjacent to subvolcanic feeder dikes near the Earth's surface. Note that the percentage of syn-intrusive ZHe ages recorded by country-rock derived zircon grains is an underestimate, as the partially-reset 37-39 Ma ZHe ages were not included in the budget.

6. Revised criteria for the interpretation of double-dated mineral grains

6.1 Expected thermochronologic age combinations

Our findings delineate a tripartite scenario, in terms of age combinations expected for high-temperature (e.g., ZUPb) and low-temperature (e.g., ZHe) geo/thermochronometric systems, when zircon grains eroded from a shallow-level volcanic-plutonic complex and associated country rocks are correctly analyzed via double-dating including ZUPb analysis that targets the zircon rim. These three cases are illustrated in the upper row of Fig. 5 (Cases 1 to 3), which shows an unpolished depth-profiled zircon grain similarly to the approach applied in this work: *Case (1)* - Zircon grains derived from shallow-level magmatic rocks; *Case (2)* - Zircon grains derived from the contact aureole; and *Case (3)* - Zircon grains derived from country rocks not thermally affected by the intrusion. The lower row of Fig. 5 (Cases 4 to 6) shows instead, for comparison, a polished zircon grain with an ablation pit on the center of the grain, similarly to the approach followed in several previous works (e.g., Shen et al. 2012; Jourdan et al. 2013, 2018; Stevens 2013; Bootes et al. 2019). Notably, if the ablated central part of the grain includes a xenocrystic core (e.g., Corfu et al. 2003), this will provide a core $ZUPb_{(C)}$ age that can be remarkably older than the rim $ZUPb_{(R)}$ age of the depth-profiled zircon rim.

Zircon grains derived from shallow-level magmatic rocks (Case 1 in Fig. 5) are expected to yield ZHe ages that record ultra-rapid cooling following at the time of intrusion (T_i) and that are indistinguishable within error from the rim $ZUPb_{(R)}$ age of the depth-profiled zircon rims. Zircon grains derived from the contact aureole (Case 2 in Fig. 5) are instead expected to yield ZHe ages = T_i that reflect country-rock cooling immediately after intrusion, and that are younger than the pre-intrusion rim $ZUPb_{(R)}$ ages of the zircon. These ZHe ages are expected to become progressively older moving away from the intrusion. Finally, zircon grains derived from country rocks not affected by the intrusion (Case 3 in Fig. 5) should yield ZHe ages > T_i that are generally younger than the rim $ZUPb_{(R)}$ age of the corresponding zircon rim.

Note that the ZHe system provides constraints to the age of magmatism both for Case 1 and Case 2. However, only for Case 1 do zircon grains show $ZUPb_{(R)}$ = ZHe or a fingerprint that can be exploited for the identification of magmatic ages. Zircon grains derived from the contact aureole show instead $ZUPb_{(R)}$ > ZHe, which implies that those ZHe ages cannot be identified as magmatic ages by a classic approach to double dating. Because only part of the grains yielding magmatic ZHe ages can be identified by double dating (i.e., $ZUPb_{(R)}$ = ZHe), the assumption that all the remaining grains preserve an exhumation signal invariably leads to interpretations that are prone to be incorrect.

6.2 Zircon core vs zircon rim U-Pb ages

Another important point underlined in Fig. 5, cases 4 to 6, is that core ZUPb$_{(C)}$ ages are always > ZHe ages in all the three cases considered. Therefore, a double-dating approach targeting zircon cores is not suitable for detecting magmatic ages in detrital zircon grains, unless the occurrence of xenocrystic cores can be safely excluded. Hence, it is critical to determine the youngest magmatic age zone of a zircon for double dating. So far, only few detrital thermochronometric studies have targeted the grain rim for ZUPb analysis in the context of double dating. For example, Rahl et al. (2003) performed LA-ICP-MS analysis in a single pit ~30 μm in diameter and ~20 μm deep on the exterior of unmodified grains mounted on tape. Najman et al. (2010) performed ZUPb analysis on grain external surfaces with ablation crater depths of ~4-10 μm. Pujols and Stockli (2021) mounted unpolished zircons on acrylic disks for LA-ICP-MS depth profiling to recover the ZUPb age of the youngest growth zone from each grain. Many other works, for example Stevens et al. (2013) and Jourdan et al. (2013, 2018) explicitly targeted the core of zircon grains for their ZUPb analyses. Bootes et al. (2019) preablated the grains with a 40-μm diameter spot followed by a 20-μm diameter spot that penetrated to a depth of ~12 μm for data collection at zircon cores, whereas Lu et al. (2020) performed LA-ICP-MS ZUPb dating through multiple 30 μm spots located within the same area where ZFT were counted, also including the core of the zircon grains. The double-dating approach that was applied in this second category of studies likely precluded a correct identification of magmatic ages in those grains with a xenocrystic core, despite their potential derivation from shallow-level magmatic rocks. Additional complications may arise due to removal of grain rims by abrasion during sediment transport. In this case, zircon grains derived from erosion of magmatic rocks may only preserve the ZUPb age of an inherited core even in the most external parts of the grain (Malusà and Garzanti 2019). As a result, magmatic ZHe ages are prone to be incorrectly interpreted in terms of exhumation, because ZUPb$_{(C)}$ > ZHe. Crucial for a correct interpretation is therefore an independent knowledge of the age T_i of the main magmatic events in a study region. This information, although sometimes not available *a priori*, can be easily retrieved in detrital thermochronology studies by identification of the ZUPb$_{(R)}$ = ZHe fingerprint in double-dated zircon grains.

6.3 Implications for lag-time analysis

In the previous section we have underlined the importance of a double-dating approach targeting zircon rims for a correct identification of magmatic ages. For the sake of simplicity, in this section we assume that double-dated grains from previous studies contain no xenocrystic cores, to dismiss any potential problems due to an incorrect location of the ZUPb ablation spot. However, even under such favorable hypothetical circumstance, our results from the Bergell-Gonfolite system indicate that the assumption that all double-dated grains with a ZUPb > ZHe fingerprint preserve an exhumation signal is invariably misleading. Our findings are particularly relevant in detrital thermochronology studies that emphasize the importance of the youngest thermochronologic age peaks for lag-time analysis. Because ZHe = T_i for Case 2 and ZHe > T_i for Case 3 (Fig. 5), magmatic ZHe ages derived from the contact aureole (Case 2) are systematically younger than any potential exhumation ZHe age in nearby country rocks unaffected by the intrusion (Case 3). Therefore, zircon grains from a contact aureole always form the youngest ZHe age peak in the polymodal grain-age distribution derived from erosion of a single volcanic-plutonic source. Even if magmatic ages recognized by their $ZUPb_{(R)}$ = ZHe fingerprint (1 in Fig. 1) are systematically removed from the data set, the interpretation of the youngest ZHe (or ZFT) age peak in terms of exhumation remains prone to misinterpretations.

As a rule, zircon grains likely derived from country rock ($ZUPb_{(R)}$ > ZHe) but sharing the same ZHe age with zircon grains that are undoubtedly magmatic according to their ZUPb=ZHe fingerprint (3 in Fig. 1), should be excluded from any interpretation in terms of exhumation using the lag-time approach. If there is a suspicion that part of these ZHe ages may derive from a different source and may therefore represent exhumation ages, this must be demonstrated in a stratigraphic sequence where the contribution of ZHe magmatic ages (ZUPb=ZHe) is not present. Note that zircon grains derived from country rock and from much younger magmatic rocks may, and often do share the same ZHe exhumation age in specific intervals of a stratigraphic succession. However, exhumation ages are always younger than the intrusion age T_i of mineral grains derived from the same source (Malusà and Fitzgerald 2020). Therefore, these situations are readily identifiable because no grains lie on the ZUPb = ZHe (or ZUPb = ZFT) line.

7. Application to previous studies

The concepts described in section 6 can be applied to published high-quality double-dating datasets from the literature. For example, Figure 6a shows ZUPb and ZFT data from zircon grains of the Zhaguo Member, Tethyan Himalaya (Najman et al. 2010). ZUPb analysis was performed on grain external surfaces. Most grains have similar ZUPb and ZFT ages (marked red in Fig. 6a), which were interpreted as formation ages from an igneous source. The remaining grains have Precambrian ZUPb ages and were interpreted, based on their ZFT age, as being exhumed from depth during the Mesozoic (Najman et al. 2010). However, most of these grains (marked yellow in Fig. 6a) have ZFT ages systematically overlapping the ZFT ages of grains lying on the ZUPb=ZFT line, which are clearly magmatic. Therefore, these ZFT ages provide no direct indication on exhumation. Instead, these ZFT ages may either record country-rock cooling in a contact aureole, or crystallization of shallow-level magmatic rocks as recorded by zircon grains that include xenocrystic cores improperly analyzed for ZUPb dating. Only a single grain of the dataset (marked green in Fig. 6a) can possibly provide information on exhumation, albeit loosely constrained due to the large error bar of this ZFT age (Fig. 6a).

A similar situation is observed in Fig. 6b, showing a data set based on the analysis of modern sands of the Ordos Basin in north-central China (Stevens et al. 2013). The authors have specifically targeted zircon cores for their ZUPb analyses. They found a majority of grains showing Permo-Triassic ZFT ages independent on ZUPb ages. A number of grains showing overlapping ZUPb and ZFT age at ~250 Ma was interpreted as the indication of relatively shallow emplacement and rapid exhumation at the source. However, these ages (marked red in Fig. 6b) simply record crystallization of shallow-level magmatic rocks and cannot be used to deduce an exhumation rate. The same applies for the many grains with a similar ZFT age but a much older ZUPb age (marked yellow in Fig. 6b) which provide no useful constraints on exhumation. The greater percentage of zircon grains marked in yellow in Fig. 6b compared to Fig. 6a is likely the effect of a biased approach in ZUPb analysis targeting zircon cores rather than zircon rims, which may have precluded a correct identification of magmatic ages in many zircon grains with older xenocrystic cores. Only four grains on the entire dataset (marked green in Fig. 6b) can therefore potentially provide information on exhumation.

The occurrence of a majority of grains with an old ZUPb age and a young ZFT age that overlaps magmatic ZFT ages of grains yielding ZUPb=ZFT is common to several other studies (e.g., Shen et al. 2012; Jourdan et al. 2013, 2019). Jourdan et al. (2013) applied a double-dating approach including ZFT analysis and ZUPb dating of zircon cores in sedimentary rocks on either side of the Western Alps (Fig. 7). In the western Alpine foreland basin, they identified three zircon groups: (i) a first group with overlapping Paleogene ZUPb and ZFT ages representing ~10% of the dataset and interpreted as volcanically derived Paleogene zircon grains; (ii) a second group encompassing ~ 66% of the data set and including zircons with Paleogene ZFT ages and much older, mainly Variscan ZUPb ages; and (iii) a third group characterized by a wide range of Cretaceous–Jurassic ZFT ages and much older (Variscan or Panafrican) ZUPb ages. Jourdan et al. (2013) excluded from the dataset the ZFT ages of the first group zircons to obtain what they considered a pure exhumation signal. Based on ZFT ages ~30-31 Ma in the second group zircons from Oligocene sediments (marked by a blue star in Fig. 7a), they proposed a short-lived rapid exhumation event affecting the Western Alps during the early Oligocene, triggered by indentation of the Adriatic upper mantle beneath the eclogitic units of the Internal Zone (dark blue in Fig. 7c) and consequent surface uplift and rapid erosion of the Western Alps at rates exceeding 1.5–2 km/Ma. However, zircon grains with ZUPb > ZFT, but sharing the same ZFT age with zircon grains of the first group that are undoubtedly magmatic because ZUPb = ZFT, should be excluded from any interpretation in terms of exhumation. It may be argued that part of these zircon grains may derive from a different source compared to the zircon grains of the first group, and that their ZFT ages may potentially represent exhumation ages. However, as introduced in Sect. 6.3, this hypothesis must be tested in a stratigraphic succession where any potential contributions of magmatic ZFT ages can be safely excluded. The Monferrato sedimentary succession, exposed to the east of the Western Alps (Fig. 7b, c), provides a favorable opportunity to check whether detrital ZFT data supports the hypothesis of an Oligocene exhumation signal or not. These sediments were demonstrably fed by detritus shed from the Alpine Internal Zone (Elter et al. 1966; Polino et al. 1991). They contain ZUPb ages that are Jurassic or older, but no Paleogene ZUPb age, pointing to a negligible contribution of Paleogene magmatism to the detrital thermochronologic record. Because the youngest ZFT age peak in Oligocene sediments of the Monferrato spans from 39 to 78 Ma, but no peak ~30 Ma is observed (question mark in Fig. 7b), we conclude that detrital ZFT data from either side of the orogen provide no evidence of early Oligocene fast erosional exhumation in the Western Alps, unlike suggested by Jourdan et al. (2013). In the western Alpine foreland basin, ZFT ages of second group zircons thus record country-rock cooling in a contact aureole or crystallization of shallow-level magmatic rocks as recorded by ZUPb dating of old xenocrystic cores. Potential candidate sources for these zircon grains are magmatic complexes located to the south-west (e.g., Provence, Sardinia) or to the north-east (e.g., Biella) of the sampling location (Fig. 7c). These potential sources may have provided zircon grains to the western Alpine foreland basin, but apparently no zircon grains to the Monferrato.

If a complementary stratigraphic succession without magmatic ZFT ages is not available, further clues for a correct interpretation of double-dated mineral grains can be provided by the analysis of thermochronologic age trends through a stratigraphic succession (Fig. 7a) following the criteria illustrated in Malusà and Fitzgerald (2020). Thermochronologic ages that are set during episodes of magmatic crystallization define stationary age peaks that remain fixed up section (see in Fig. 7a the points that lies on the 30 Ma dotted line). These stationary age peaks can be detected in the detrital thermochronologic record starting from strata deposited synchronously with magmatism. Thermochronologic ages that are set during bedrock erosion define instead moving age peaks that gets progressively younger up section. Notably, the first appearance of a moving age peak that records erosional exhumation only occurs with a considerable time delay in the sedimentary succession fed by erosion, depending on the closure temperature of the thermochronologic system under consideration and the erosion rate. In fact, the whole rock pile with a thermochronologic fingerprint acquired before the onset of erosion must first be completely removed (Malusà and Fitzgerald 2020). For example, ZFT ages recording the onset of fast erosion at rates ~2 km/Ma starting at ~30 Ma would be only expected in sedimentary strata with depositional age of ~ 27-26 Ma or younger. Since the ZFT ages ~30-31 Ma (blue star in Fig. 7a) in the western Alpine foreland basin were detected in layers with a depositional age of ~30 Ma, we can conclude that these ages provide information on magmatism, not exhumation, consistent with the information previously provided by the analysis of the Monferrato complementary stratigraphic sequence (Fig. 7b).

8. Conclusions

ZUPb and ZHe double dating of selected detrital zircon grains from the sedimentary succession of the Gonfolite Group allows us to revise the criteria that had been previously applied in detrital thermochronometric studies in terms of identification and discrimination of magmatic and exhumation ages. In a detrital thermochronometric dataset, we found that only part of the grains yielding magmatic ZHe (or ZFT) ages can be identified by their $ZUPb_{(R)}$ = ZHe (or ZFT) fingerprint. A relevant percentage of syn-intrusive ZHe (or ZFT) cooling ages that record country-rock cooling in a contact aureole remain undetected because these ages are younger than the corresponding $ZUPb_{(R)}$ age in the same zircon grains. Because only part of the grains yielding magmatic ZHe or ZFT ages can be identified by a classic double-dating approach, the assumption that all the remaining grains may provide information on exhumation is invariably misleading. Interpretation is even more critical in those studies targeting zircon cores for ZUPb dating, because such approach is not suitable for systematically and reliably detecting magmatic ages in detrital zircon grains. Our findings are particularly relevant for detrital thermochronometric studies that emphasize the importance of the youngest thermochronologic age peaks for lag-time analysis, as zircon grains from a contact aureole always form the youngest thermochronologic age peak in polymodal grain-age distributions derived from erosion of the uppermost levels of a single volcanic-plutonic source. This is particularly pertinent for basin deposits that are sourced from orogenic hinterlands characterized by voluminous magmatism, such as magmatic arcs in convergent tectonics settings.

Based on our analysis, we illustrate improved criteria for the interpretation of double-dated mineral grains in detrital thermochronology studies. Their application to high-quality datasets of double-dated grains from the literature suggest that many previous geologic interpretations based on a classic double-dating approach should be proficiently reconsidered. Our results underline not only the potential of a double-dating approach that includes ZUPb analysis of unpolished depth-profiled zircon grains, but also demonstrate the importance of a proper analysis of thermochronologic age trends through a stratigraphic succession for a correct geologic interpretation of detrital thermochronology data.

CRediT authorship contribution statement

Marco G. Malusà: Conceptualization, Formal Analysis, Visualization, Writing – original draft, Writing – review & editing. *Owen Anfinson:* Conceptualization, Investigation, Formal Analysis, Writing – review & editing, Funding acquisition. *Daniel F. Stockli:* Methodology, Resources, Writing – review & editing, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary material

Supplementary Table S1: Sample locations, reduced (U-Th)/He data, and U-Pb data of double-dated zircon grains.

Figure 1

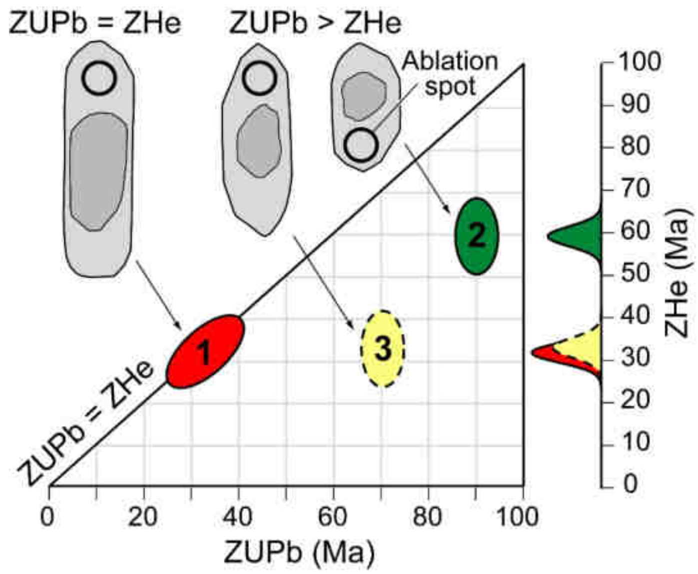

Figure 1: Rationale of the double-dating approach for the identification of magmatic ages in detrital zircon grains. Paired high-temperature and low-temperature geo/thermochronologic analyses, such as zircon U-Pb (ZUPb) and (U-Th)/He (ZHe) or fission-tracks, are applied to the same detrital grains (Carter and Bristow 2003; Reiners et al. 2005). Zircon grains yielding magmatic ZHe ages are readily identified by their ZUPb=ZHe fingerprint (1), provided that older xenocrystic cores are avoided during ZUPb analysis (Malusà and Fitzgerald 2020). The remaining grains, yielding low-temperature thermochronometric ages younger than the corresponding ZUPb age (2, 3) are often used to infer the exhumation history of the source rocks (e.g., Najman et al. 2010; Stevens et al. 2013; Jourdan et al. 2013, 2018; Bootes et al. 2019; Lu et al. 2020). Grains marked (3), unlike grains marked (2), share the same ZHe age with zircon grains marked (1) that are undoubtedly magmatic according to their ZUPb=ZHe fingerprint.

Figure 2

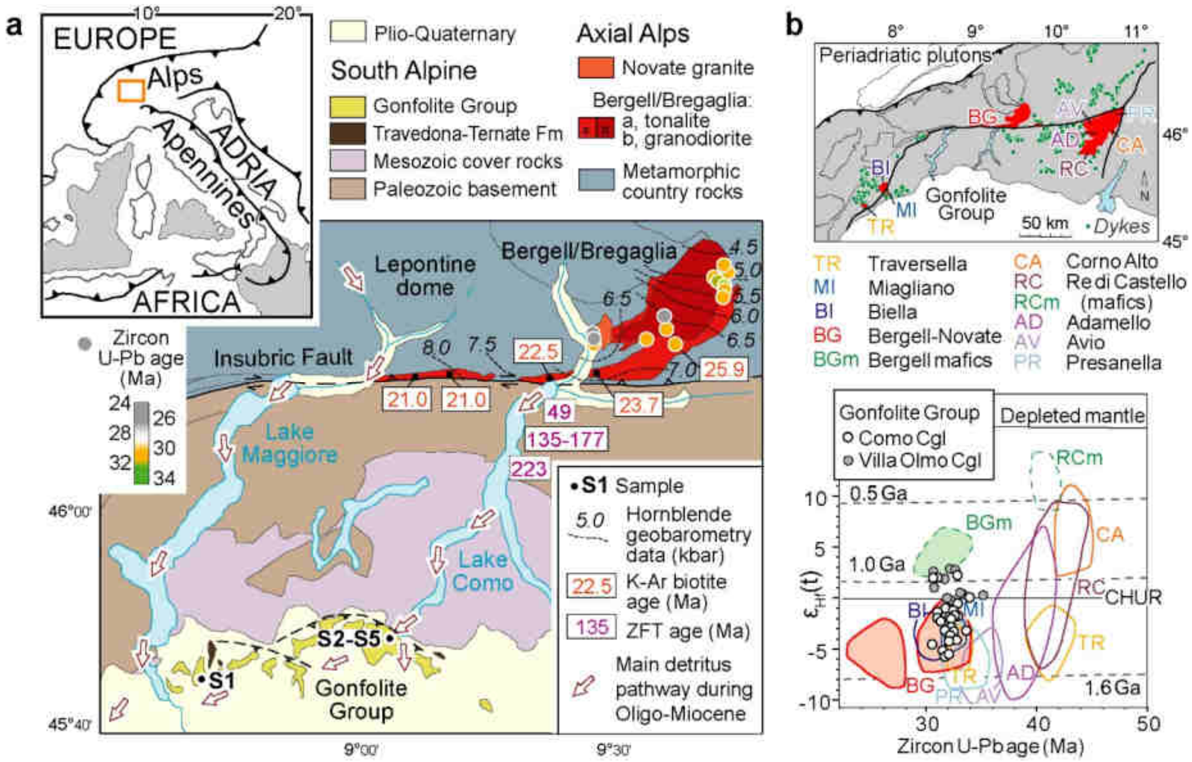

Figure 2: Tectonic framework. a Geologic sketch map of the Bergell-Gonfolite source-to-sink system (see location in the inset). Lakes Como and Maggiore occupy Oligocene-Miocene paleovalleys funnelling detritus towards the Gonfolite basin (after Malusà et al. 2011). Relevant bedrock zircon U-Pb ages (Ji et al. 2019), hornblende geobarometry data (Davidson et al. 1996), biotite K-Ar ages (Villa and von Blanckenburg 1991), and zircon fission-track (ZFT) ages (Bertotti et al. 1999) are also shown. S1 to S5 indicate samples of the Gonfolite Group analyzed in this work (the coordinates for each sample are indicated in the supplementary material, their biostratigraphic ages are reported in Malusà et al. 2016). **b:** U-Pb ages and Hf isotopic compositions of magmatic zircon grains of the Gonfolite Group compared to the reference fields of the potential Periadriatic sources shown in the map (after Malusà and Fitzgerald 2020, based on data from Ji et al. 2019 and Lu et al. 2019).

Figure 3

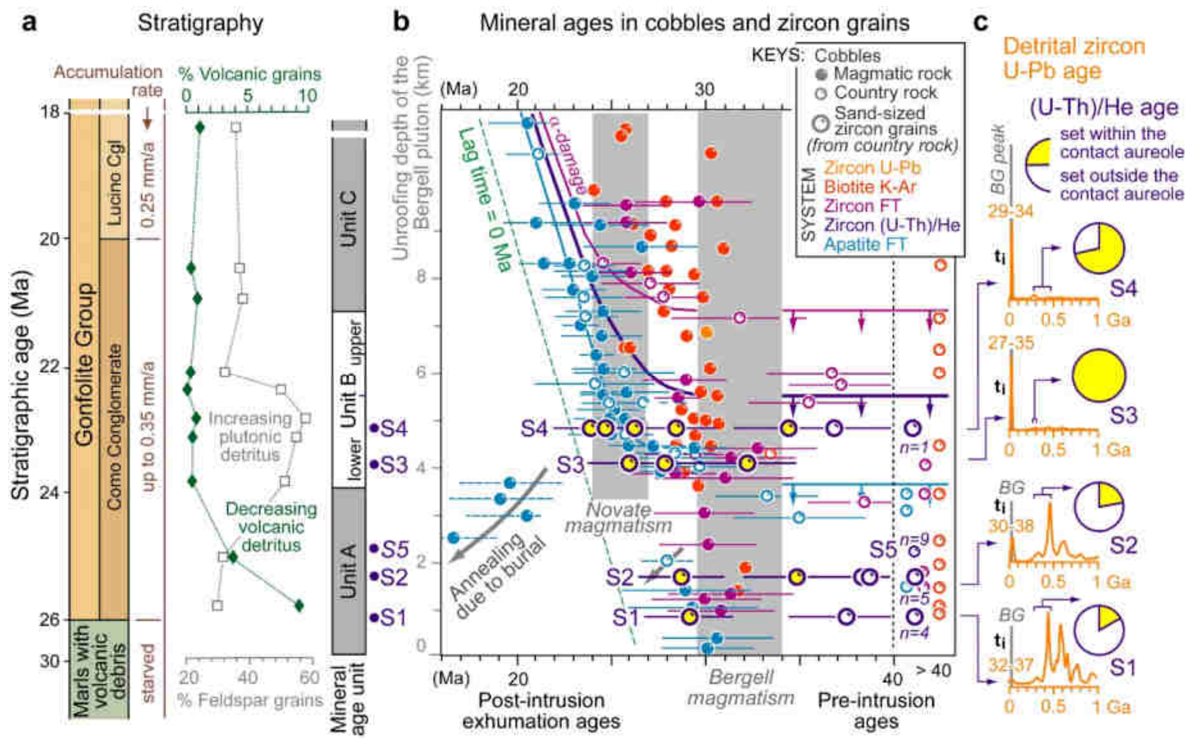

Figure 3: Mineral-age stratigraphic framework. a: Simplified stratigraphic column, accumulation rate, modal composition, and mineral-age units of the Gonfolite Group (from Malusà et al. 2011). **b:** Mineral ages in magmatic cobbles (small solid dots) and country rock cobbles (small open dots) provided by apatite and zircon fission-track, biotite K-Ar and zircon U-Pb dating (from Malusà et al. 2011 and references therein; see color coding on the top-right). The vertical grey bars indicate the age of Bergell and Novate magmatism (Ji et al. 2019). Continuous lines in color indicate predicted patterns of pre-intrusion and exhumation ages (Malusà et al. 2011; Malusà and Fitzgerald 2020). (U-Th)/He ages of double-dated zircon grains from samples S1 to S5 (this work) are also shown (larger dots, in violet). The yellow fill indicates zircon (U-Th)/He ages overlapping with Bergell and Novate magmatism. *n* is the number of double-dated zircon grains yielding (U-Th)/He ages older than 40 Ma in each sample. **c:** Zircon U-Pb kernel density estimates of samples S1 to S4. Numbers in orange indicate the age range of the youngest U-Pb age peaks (from Malusà et al. 2016). The pie charts summarize the ratio of country-rock derived zircon grains showing (U-Th)/He ages set within the Bergell and Novate contact aureole vs those derived from outside the contact aureole (this work; arrows mark the corresponding U-Pb age ranges). BG = Bergell-Novate.

Figure 4

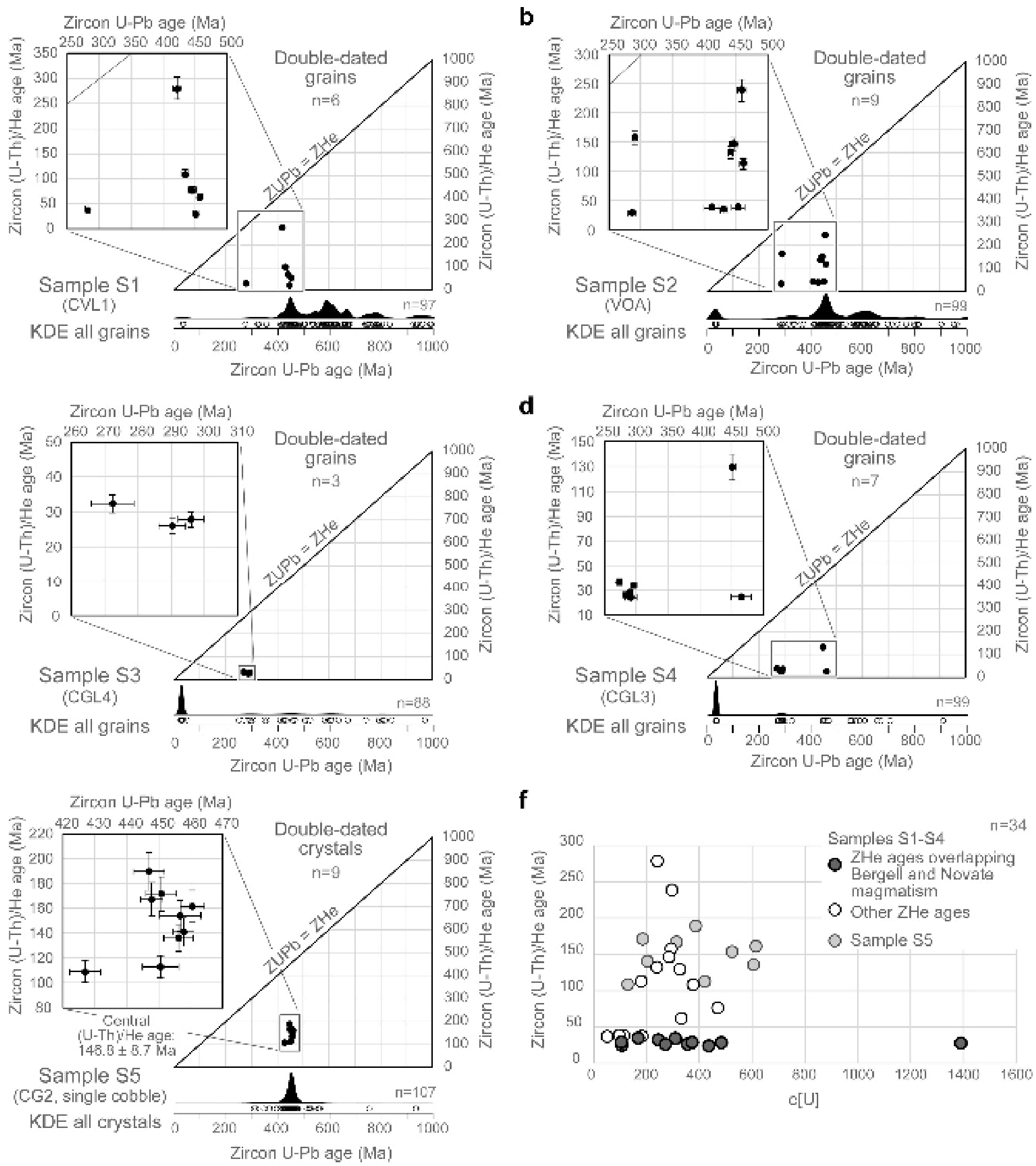

Figure 4: Double-dating results. a-d: Zircon U-Pb kernel density estimates (KDE) for detrital samples S1 to S4 (from Malusà et al. 2016, see stratigraphic locations in Fig. 3a). Open dots are single U-Pb ages measured by LA-ICP-MS depth profiling of unpolished zircon grains (n= number of dated grains). Scatter plots show double-dated zircon grains (solid dots with 2σ error bars) that were selected following the criteria outlined in Farley (2002) among those zircon grains not derived from Periadriatic magmatic rocks, as indicated by their U-Pb age. Note that no grains plot on the ZUPb =

ZHe line, which would ensure that (U-Th)/He ages of these double-dated zircon grains constrain exhumation, not magmatic crystallization (e.g., Jourdan et al. 2013; Bootes et al. 2019; Lu et al. 2020). **e:** Double-dated zircon crystals from a single country-rock cobble (sample S5). **f:** Zircon (U-Th)/He age (ZHe) vs. effective Uranium concentration (e[U]) in double-dated zircon grains from samples S1 to S5. ZHe ages overlapping Bergell and Novate magmatism are indicated in dark grey (these ZHe ages characterize the zircon grains derived from erosion of country rock affected by the Bergell and Novate contact aureole).

Figure 5

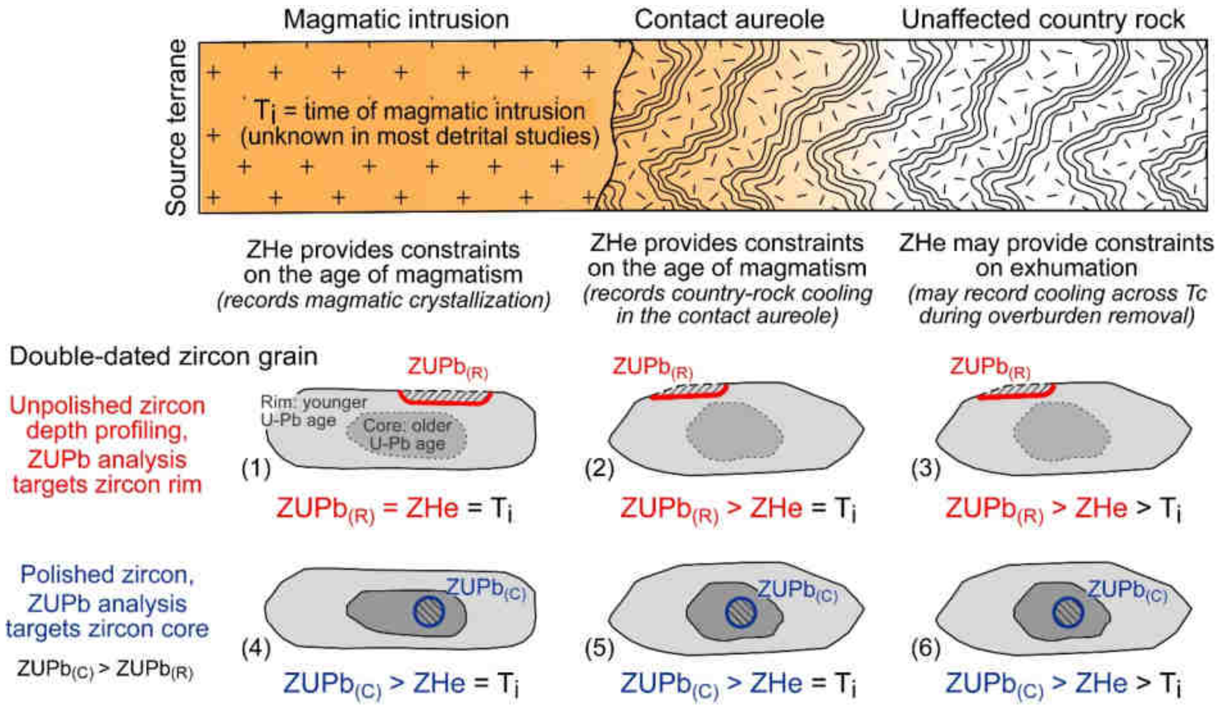

Figure 5: Potential misidentification of exhumation ages by double dating. The cartoon shows the ages expected for high-temperature (e.g. ZUPb) and low-temperature (e.g., ZHe) thermochronologic systems when zircon grains eroded from a shallow-level volcanic-plutonic complex and its country rocks are correctly analyzed by a double-dating approach with ZUPb analysis that targets the zircon rim (Cases 1 to 3, upper row in red). Results expected when ZUPb analysis targets an older xenocrystic core is shown for comparison (Cases 4 to 6, lower row in blue). *Case (1)* - Zircon grains derived from shallow-level magmatic rocks: they yield ZHe ages that record magmatic crystallization at time T_i and that are indistinguishable within error from the $ZUPb_{(R)}$ ages yielded by depth profiling of the grain rim. *Case (2)* - Zircon grains derived from the contact aureole: they yield ZHe ages = T_i that reflect country-rock cooling immediately after intrusion and that are younger than the pre-intrusion $ZUPb_{(R)}$ ages of the grain rim. *Case (3)* - Zircon grains derived from country rocks not affected by the intrusion: they yield ZHe ages > T_i that are younger than the $ZUPb_{(R)}$ ages of the grain rim. Note that the ZHe system provides constraints to the age of magmatism both for case (1) and case (2). However, only for case (1) do zircon grains show $ZUPb_{(R)}$ = ZHe. Because only part of the grains yielding magmatic ZHe ages can be identified by double dating (i.e., $ZUPb_{(R)}$ = ZHe), the assumption that all the remaining grains provide constraints on exhumation is prone to lead to

incorrect interpretations. *Cases (4) to (6)* - When ZUPb analysis targets a xenocrystic core, the $ZUPb_{(C)}$ age is invariably older than the corresponding ZHe age.

Figure 6

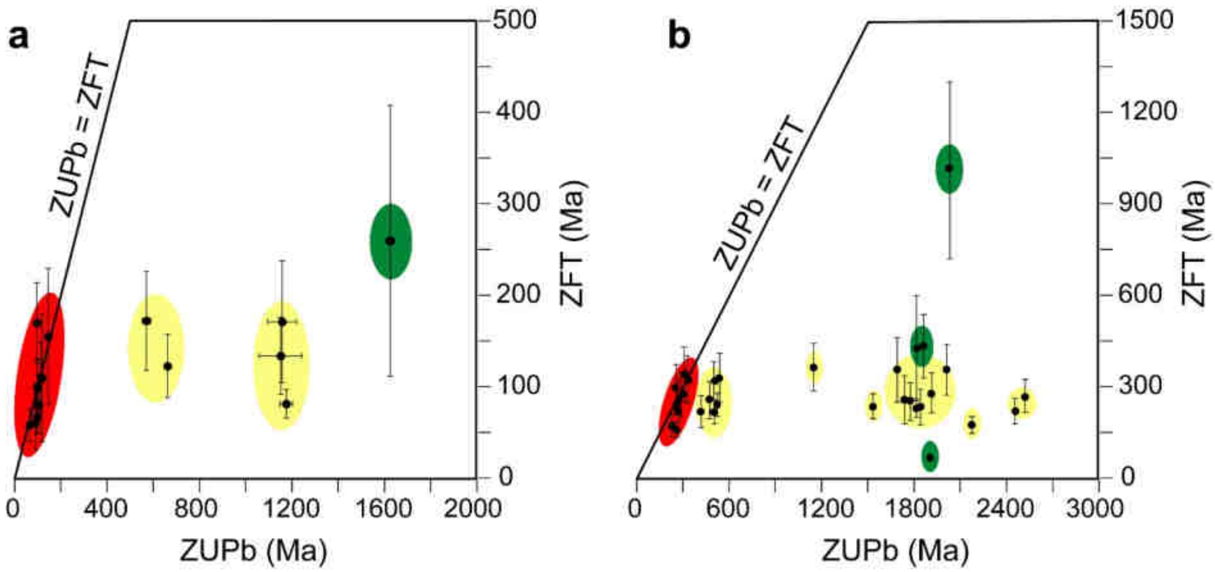

Figure 6: Application to previous studies. a: Double-dated zircon grains from the Zhaguo Member, Tethyan Himalaya (Najman et al. 2010). **b:** Double-dated zircon grains from modern sands of the Mu Us desert, Ordos Basin, north central China (Stevens et al. 2013). Both zircon U-Pb (ZUPb) and zircon fission-track (ZFT) ages were quoted at 2 sigma errors. ZUPb analysis targeted the grain rim in (a) and the grain core in (b). Keys: Red = ZFT ages that record magmatic crystallization of shallow-level magmatic rocks; Yellow = ZFT ages that may record country-rock cooling in a contact aureole or magmatic crystallization (due to ZUPb dating of old xenocrystic cores); Green = ZFT ages that may provide constraints on exhumation (see Malusà and Fitzgerald 2020 for further details). Both red and yellow marked grains should be excluded from exhumation rate calculations based on lag-time analysis (see text for explanation). Note the increasing proportion of double-dated zircon grains marked yellow when ZUPb analysis targets the zircon core (b).

Figure 7

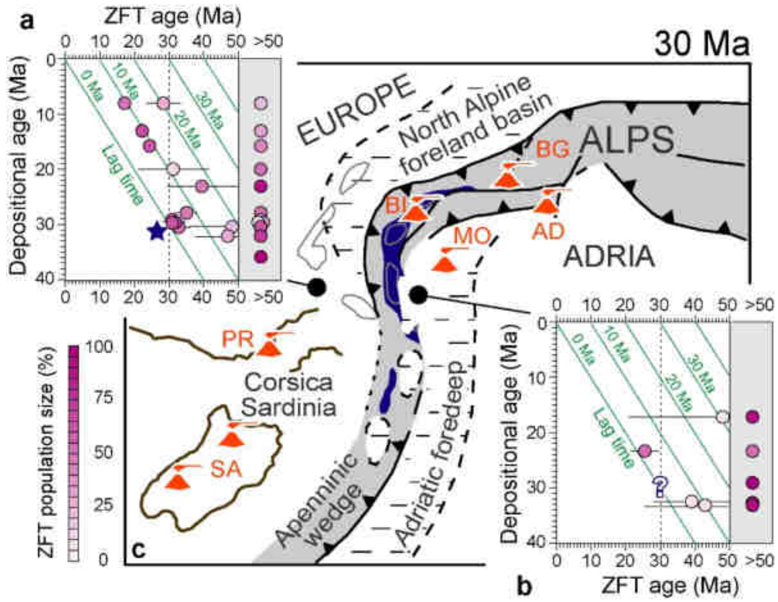

Figure 7: Insights from thermochronologic age trends through a stratigraphic succession. a-b: Detrital ZFT data from the western Alpine foreland basin (a) (Bernet et al., 2009; Jourdan et al., 2013) and the Monferrato (b) (Jourdan et al., 2013). Different color intensities in the lag-time diagrams indicate the relative percentages of each grain-age population (Malusà and Fitzgerald 2020). Grains older than 50 Ma are shown as a single population for the sake of simplicity. The vertical dashed line at 30 Ma marks the end of the Alpine magmatic climax. The dark blue star in (a) indicates prominent populations of double-dated zircon grains interpreted by Jourdan et al. (2013) as evidence of short-lived, fast erosional exhumation of the Western Alps at ~30 Ma. However, the question mark in (b) shows that similar ZFT ages are not found in basins located to the east of the Western Alps, which were demonstrably fed by detritus shed from the Alpine Internal Zone. **c:** Palinspastic reconstruction of the Alps-Apennines system at 30 Ma showing the exhumed eclogitic units of the Internal Zone (dark blue), active magmatic complexes (in orange), and the locations of the samples shown in (a) and (b) (solid black dots). Acronyms: AD, Adamello; BI, Biella and Traversella; BG, Bergell; MO, Mortara; PR, Provence; SA, Sardinia (Anfinson et al. 2016).

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