

Supplementary: Apatite (U-Th)/He and fission-track methods

AHe analysis was performed at the University Paris Saclay (France). Two to ten single grains were selected based on size, morphology and lack of inclusions. Grain dimensions were measured after which each crystal was placed into a platinum (Pt) capsule and degassed under vacuum twice at $1050\pm 50^\circ\text{C}$; the ^4He content was determined by comparison with a ^3He spike using a Prima quadrupole mass spectrometer (Gautheron et al., 2021). The Pt baskets were then placed into single-use polypropylene vials. Apatite grains were dissolved for one hour at 90°C in a $50\ \mu\text{l}$ HNO_3 solution containing a known concentration of ^{235}U , ^{230}Th and ^{149}Sm , and then filled with 1 ml of ultrapure MQ water. U, Th and Sm concentrations were analysed with a quadrupole ICP-MS (series II CCT Thermo-Electron) at LSCE, Gif-sur-Yvette, France. Analytical accuracy and reproducibility for U-Th isotope measurements by using ICP-QMS were controlled with the Durango apatite standard. Individual ages are corrected for ejection using the factor F_T (Ketcham et al., 2011); the equivalent-sphere radius (R_s) was calculated using the procedure of Gautheron and Tassan-Got (2010). The 1σ error of the AHe age is considered at 8%, reflecting the sum of errors in the ejection-factor correction and age dispersion of the standards. Table 4 summarizes the AHe data.

AFT analysis was performed using the external detector method at the ISTERre laboratory (France). Apatite grains were mounted in epoxy resin, polished, and etched for 20 seconds at 21°C with 5.5 molar HNO_3 (Donelick et al., 2005). Each sample mount was covered with a muscovite sheet and irradiated with a nominal fluence of 8.0×10^{15} neutrons at the FRMII research reactor of the Technical University Munich (Germany). The samples were irradiated together with IRMM540R dosimeter glasses and Fish Canyon Tuff and Durango apatite age standards. After irradiation, the mica detectors were etched for 18 min in 48% HF at 21°C . Fission tracks were counted dry at 1250x magnification using an Olympus BX51 microscope and the FT Stage 4.04 system. AFT ages, calculated with the BINOMFIT program (Ehlers et al., 2005), are reported as central ages in Table 3, together with D_{par} values and track-length measurements. The apatite grains are characterized by low to moderate effective uranium contents ($eU = U + 0.24\times\text{Th}$) ranging from 2 to 38 ppm (Table 4), except for sample 12-04 (38 to 237 ppm). We followed the recommendation of Gautheron et al. (2022) that proposes to give single ages and not mean ages for He dates. The best thermal history QTQt is predicting ages for 100,000 different thermal histories (100,000 preburn and 100,000 post burn).

References in supplementary

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