Supplementary material SF2 :

Sample preparation and fission-track age measurements of apatite grains

Apatite samples were mounted in epoxy resin and then polished to expose an internal 4 surface. Apatite samples were etched in 5% HNO₃ for 20 seconds at $20\pm1^{\circ}$ C to reveal spontaneous tracks. Irradiations of apatite samples have been carried out at the Orphée reactor (CEA-Saclay, France) and at the Forschungsneutronenquelle Heinz Maier-Leibnitz (FRM II) research reactor at Garching (Germany). Thermal neutron fluence was monitored using Corning CN-5 glasses and is equivalent in both irradiation conditions and is 5.10^{15} neutrons/cm². Apatite grains were dated using the external detector method (Gleadow, 1981) with muscovite sheets as external detector. Muscovite detectors were etched after irradiation in a 40% HF solution for 20 minutes at $21\pm1^{\circ}$ C. Spontaneous and induced FTs were counted on an optical Leica DM LM microscope. Central ages (Galbraith and Laslett, 1993) have been calculated with the zeta calibration method (Hurford and Green, 1983) by using the age standards of Durango (31.3\pm0.3 Ma, Naeser & Fleischer, 1975) and Fish Canyon Tuff (27.8\pm0.2 Ma, Hurford & Hammerschmidt, 1985).

For each dated apatite crystal, etch-pit length parallel to *c*-axis (Dpar) was measured under a 1000X dry objective as they provide good assessment of annealing rate in individual apatite grains (Donelick, 1993; Barbarand et al., 2003). Grain-age distributions were decomposed following the binomial peak-fitting method (Galbraith and Green, 1990) and incorporated in the Binomfit software (Brandon, 2002). The best-fit solution is determined by directly comparing the distribution of the grain data to a predicted mixed binomial distribution. Peak-fitting analysis for detrital samples with mixed ages and a low number of dated crystals may provide unreliable results. In order to obtain more information about cooling ages, combined grain-age distributions of detrital samples have been analyzed where necessary, assuming that the samples did not record different degrees of partial annealing.

References :

- Barbarand, J., Carter, A., Wood, I., and Hurford, T., 2003, Compositional and structural control of fission-track annealing in apatite: Chemical Geology, v. 198, no. 1-2, p. 107-137.
- Brandon, M. T., 2002, Decomposition of mixed age distributions using Binomfit, On Track, Newsl. Int. Fission Track Community 24, Volume 1, p. 13-18.
- Donelick, R.A., 1993, A method of fission track analysis utilizing bulk chemical etching of apatite. U.S. Patent Number 5,267,274.
- Galbraith, R. F., and Green, P. F., 1990, Estimating the component ages in a finite mixture: Nucl. Tracks Radiat. Meas., v. 17, no. 197-206.

- Galbraith, R. F., and Laslett, G. M., 1993, Statistical models for mixed fission track ages: International Journal of Radiation Applications and Instrumentation. Part D. Nuclear Tracks and Radiation Measurements, v. 21, no. 4, p. 459-470.
- Gleadow, A. J. W., 1981, Fission-track dating methods: What are the real alternatives?: Nuclear Tracks, v. 5, no. 1-2, p. 3-14.
- Hurford, A. J., and Green, P. F., 1983, The zeta age calibration of fission-track dating: Chemical Geology, v. 41, p. 285-317.
- Hurford, A.J., and Hammerschmidt, K., 1985. ⁴⁰Ar/³⁹Ar dating of the Bischop and Fish Canyon Tufs: calibration ages for fssion-track dating standards: Chem.Geol., v. 58, p. 23-32.
- Naeser, C.W., and Fleischer, R.L., 1975. Age of the apatite at Cerro de Mercado, Mexico: a problem for fission-track annealing corrections: Geophys. Res. Lett., v. 2, p. 67-70.