

¹⁰Be quartz purification details

Based on Kohl and Nishiizumi (1992). Sediment collected from the Mexican Hat quarry was sieved to 250 to 500-micron size fraction. Samples were soaked in 1:1 hydrochloric acid with trace amounts of hydrogen peroxide to remove iron oxide and minor carbonate then rinsed with deionized water. Samples were leached in 2.5% hydrofluoric acid and 3% nitric acid to eliminate meteoric ¹⁰Be and accessory minerals. We used a commercial grade hot dog roller to heat and rotate the samples for 8-hour intervals. We removed the leachate and rinsed samples with deionized water between each run.

We mixed approximately 1 mg of ⁹Be carrier with each sample of clean quartz. We then dissolved samples using concentrated hydrofluoric and nitric acids to dissolve the samples in Teflon beakers. Once dissolved, we dried the samples via evaporation then treated each three times with aqua regia. Dried samples were redissolved in concentrated hydrochloric acid and run through 10 mL anion exchange columns to separate Be and remove Fe, Mn, Mg, and Co. We then evaporated the Be fraction before treating with hydrogen peroxide and redissolving in milliQ water plus trace amounts of sulfuric acid. We then separated the Ti, Be, and Al using 10 mL cation exchange columns. The Be fraction was dissolved in a solution of 6 N nitric acid and beryllium hydroxide precipitate was subsequently loaded into quartz crucibles and fired in a tube furnace to produce beryllium oxide. We mixed the beryllium oxide with niobium and loaded the powder into accelerated mass spectrometer (AMS) targets.

p-IR IRSL analysis details

No in situ gamma dose-rate measurements were available for these samples. However, the total beta (β) dose-rate contribution was estimated from U, Th, and K concentrations using the conversion factors of Liritzis et al. (2013). U and Th concentrations are measured by inductively coupled plasma mass spectrometry (ICP-MS), and K concentration is measured by inductively coupled plasma optical emission spectrometry (ICP-OES). A value of 12.5 ± 0.12 wt. % K content was used in calculating the internal dose-rate (Huntley and Baril, 1997). The cosmic ray contribution was derived from the burial depth and the latitude and altitude of the samples (Prescott and Hutton, 1994; Table 2). We determined the water content for each sample from their weights before and after drying. The total geologic dose rate was calculated using the DRAC online calculator (v.1.2; Durcan et al., 2015).

A post-IR IRSL protocol (Buylaert et al., 2009) was used to measure the equivalent dose (De) values for individual grains. Each grain was stimulated first at 50° C for 3 s, and then at 225° C for 3 s to measure the more time-stable post-IR IRSL signal. A preheat of 250° C for 60 s was used before natural and regenerative measurements. A hot bleach with IR diodes at 290° C for 40 s was added to the end of each SAR cycle (Wintle and Murray, 2006). Samples were given a beta dose of 21–26 Gy, preheated at 250° C for 60 s and left at room temperature for timescales ranging from about 300 s to 30,000 s to test for the presence of athermal fading (Huntley and Lamothe, 2001). None of the samples show significant fading during the laboratory timescale.

Supplemental information references

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