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APPENDIX DR1

Analytical procedures

Sixteen samples from along the length of the HMCC footwall were selected for apatite (U-Th)/He analysis. After mineral separation, apatite aliquots were examined under a polarizing binocular microscope and immersed in ethanol to check for visible mineral inclusions. Only clear euhedral grains, with similar grain radii were selected for analysis and their grain geometry and dimensions were then measured and recorded. Due to the relatively low U and Th content of the grains studied some samples involved handpicking up to 40 grains. Samples were outgassed in a resistively-heated vacuum furnace at ~870 ˚C for 20 minutes, spiked with ^{3}He and their gas volume was determined using a Balzers quadrupole mass spectrometer. A hot blank was run after each sample to verify complete outgassing of the grains. The Durango apatite standard was run together with every batch of five samples analyzed and served as a check on analytical accuracy. Outgassed samples were removed from their capsules. Dissolution in HNO_3 and spiking was performed at CSIRO, North Ryde, Sydney. U and Th contents were determined at the University of Technology, Sydney, on a Perkin Elmer Sciex 5000a ICPMS using the isotope ratio application. Apparent (U-Th)/He ages were calculated and corrected for α-emission following the approach of Farley *et al*. (1996).

Some samples yielded irreproducible ages, suspected of being too old, suggesting that some of the age determinations are unreliable. The presence of microscopic U- and/or Th-bearing inclusions is thought to be the most common reason for irreproducible ages (Ehlers and Farley, 2003; Farley, 2002; Farley and Stockli, 2002) and a number of samples were therefore selected for an alternative dissolution protocol (Total Dissolution – TD). This procedure, rather than utilizing the standard HNO_3 procedure to dissolve apatites after outgassing, utilizes a range of acids, comprising a combination of concentrated HF, Perchloric acid, HCl and concentrated HNO_3. The TD procedure is designed to dissolve not only apatite, but also any U- and/or Th-bearing micro-inclusions present, thereby enabling all the parent isotopes to be measured. It should be noted that the presence of U- and/or Th-bearing inclusions may also produce a relatively severe α-ejection correction problem. The most likely scenario is that the inclusion will be located more than ~20 µm from the grain boundary, and therefore all of the ^{4}He produced by it will be retained in the grain. Consequently, the applied α-correction, which is made assuming homogeneous U and Th distribution, will be an overcorrection. However, the results suggest this effect is small in comparison with the direct age-related effect arising from the parentless ^{4}He (i.e. even the uncorrected ages for problematic aliquots are usually significantly greater than the expected uncorrected ages). An additional consideration is the effect of inclusions on closure temperature. Because all aliquots are made up of grains free from visible inclusions, it is assumed that any inclusions that are present in selected grains are extremely small, estimated to be <15 µm. This is smaller than the average α-stopping distance for phases likely to contain significant levels of U and Th (e.g. the average stopping distance for α-particles produced by ^{238}U-decay in zircon is 16.68 µm - Reiners *et al.*, 2004). Therefore most ^{4}He generated within such inclusions would be contained within the apatite matrix, and behave according to the diffusion characteristics of that mineral.

In each sample where there had been previously irreproducible age determinations, the TD protocol yielded an age either within error of the youngest previous determination (samples TC00-32, TC02-20 and TC02-7) or younger than all previous ages (sample TC00-34). This suggests that these samples had been affected by the presence of microscopic U- and/or Th-bearing inclusions in some previous analyses and highlights the success of the TD approach for such samples.

However, for other samples (TC02-15 and TC02-11), the TD protocol yielded ages within uncertainty limits of one or more previous age determinations which were considered too old (Table 1). This suggests that some other factor was influencing the ‘excess’ ages. Analysis of the apatite fission track mica solid state track detectors and results from a laser ablation ICPMS (LA-ICPMS) study for sample TC02-15 indicate that U and Th have a strongly zoned distribution within many of the grains. U and Th are generally zoned with one grain analyzed showing ~90 and ~40 times as much Th and U, respectively, in the core than the rim. In addition, the distribution of spontaneous fission tracks within the majority of grains suggests that the zoning is such that almost no U (and by comparison with the LA-ICPMS data, Th) is located in the outer ~20 µm of the grain. We have therefore assumed that the majority of dated grains also showed this type of zonation, which suggests that a more accurate age for this sample would be obtained if the alpha-correction was not applied. Therefore, Figure 2b contains uncorrected ages for TC02-15 instead of α-corrected ages. The ages for TC02-15 now become ~12.5-14.5 Ma, concordant with other ages in the structurally deeper part of the footwall.

Having corrected the results for all the known factors that may have contributed to anomalous ages, the data were generally considered reliable only if they had been replicated within 2σ error, they were less than or equal to the coexisting AFT age and their gas level was above a pre-determined level (based on the minimum difference between the hot-blank gas level and sample gas level for quantitative analyses, as described by Potts (1992)).

Precision and accuracy

Analytical uncertainties for the University of Melbourne (U-Th)/He facility are assessed to be ~2.4% (1σ), which incorporates gas analysis and ICPMS uncertainties. Durango is routinely used as an internal standard and the weighted mean age of all Durango analyses conducted (31.5 ± 1.6; 1σ) is concordant with the reference age accepted by the apatite fission track community of 31.4 ± 0.6 Ma (within 1σ - Green, 1985; Wagner and Van den Haute, 1992). The weighted mean age is also in agreement with other published mean AHe ages for Durango: 32.0 ± 1.0 Ma (Farley, 2002), 32.1 Ma (Farley, 2000), 32.1 ± 1.7 Ma and 32.1 ± 1.3 Ma (House *et al*., 2000; all 1σ). The precision of Durango results is 5% (1σ), which incorporates analytical uncertainties as well as natural inhomogeneities within the Durango crystals themselves (Boyce and Hodges, 2001).

The age uncertainties in this manuscript are calculated from propagated analytical uncertainties, combined with an α-correction-related constituent, which takes into account an estimated 5 μm uncertainty in grain size measurements.

REFERENCES FOR DATA REPOSITORY

Boyce, J.W., and Hodges, K.V., 2001, Chemical variations in the Cerro de Mercado (Durango, Mexico) fluorapatite: Assessing the effect of heterogeneity on a geochronologic standard: EOS, Transactions, American Geophysical Union, v. 82, p. F1322.

Ehlers, T.A., and Farley, K.A., 2003, Apatite (U-Th)/He thermochronometry: methods and applications to problems in tectonic and surface processes: Earth and Planetary Science Letters, v. 206, p. 1-14.

Farley, K.A., 2000, Helium diffusion from apatite: General behavior as illustrated by Durango fluorapatite: Journal of Geophysical Research-Solid Earth, v. 105, p. 2903-2914.

Farley, K.A., 2002, (U-Th)/He dating: Techniques, calibrations, and applications, *in* Porcelli, D., et al., eds., Noble Gases in Geochemistry and Cosmochemistry: Washington, D.C., Geochemical Society and Mineralogical Society of America, Reviews in Mineralogy and Geochemistry, v. 47, p. 819–844.

Farley, K.A., and Stockli, D.F., 2002, (U-Th)/He dating of phosphates: Apatite, Monazite, and Xenotime, *in* Kohn, M.J., et al., eds., Phosphates – Geochemical, Geobiological, and Materials importance: Washington DC, Geochemical Society and Mineralogical Society of America, Reviews in mineralogy and geochemistry, v. 48: p. 559-577.

Farley, K.A., Wolf, R.A., and Silver, L.T., 1996, The effects of long alpha-stopping distances on (U-Th)/He ages: Geochimica et Cosmochimica Acta, v. 60, p. 4223-4229.

Green, P.F., 1985, Comparison of zeta calibration baselines for fission-track dating of apatite, zircon and sphene: Chemical Geology, v. 58, p. 1-22.

House, M.A., Farley, K.A., and Stockli, D.F., 2000, Helium chronometry of apatite and titanite using Nd-YAG laser heating: Earth and Planetary Science Letters, v. 183, p. 365-368.

Potts, P.J., 1992, A handbook of silicate rock analysis: Glasgow, Blackie academic and professional.

Reiners, P. W., Spell, T. L., Nicolescu, S., and Zanetti, K. A., 2004, Zircon (U-Th)/He thermochronometry: He diffusion and comparisons with $^{40}Ar/^{39}Ar$ dating: Geochimica et Cosmochimica Acta, v. 68, no. 8, p. 1857-1887.

Wagner, G.A., and Van den Haute, P., 1992, Fission track dating - Solid Earth Sciences library v. 6, Kluwer academic publishers, 296 p.

TABLE DR1. HARCUVAR MOUNTAINS - Apatite (U-Th)/He DATA

Sample No.	Elevation (m)	Location (Lat., Long.)	Dist. in slip dir. (km)	No. of grains	Av. grain radius (μm)	^{4}He (ncc)	U (ppm)	Th (ppm)	Ft[*]	Initial Age (Ma)	Corrected Age (Ma ± 2σ)	Screened? (Y/N)[†]
89AZ09-1	640	33°44.73',	4.1	10	80.4	5.815	13.48	22.89	0.83	15.9	**19.2 ± 1.1**	N
89AZ09-2		113°40.42'	4.1	10	58.7	2.032	12.86	20.63	0.77	16.0	**20.7 ± 1.4**	N
89AZ09-3			4.1	38	45.4	2.497	12.45	19.23	0.52	14.0	**26.8 ± 4.5**	Y
89AZ09-4			4.1	22	77.8	7.996	10.87	16.73	0.81	17.9	**22.1 ± 1.3**	N
89AZ12-1	720	33°53.08',	17.1	11	58	0.449	3.49	1.62	0.76	18.0	**23.7 ± 1.6**	Y
89AZ12-2		113°37.88'	17.1	20	45.6	0.3	2.58	1.04	0.71	15.9	**22.5 ± 1.8**	Y
89AZ12-3			17.1	13	96.7	1.689	3.66	1.30	0.85	13.9	**16.3 ± 0.9**	N
89AZ14-1	810	33°55.32',	20.6	16	43.5	0.216	4.25	7.48	0.67	8.4	**12.5 ± 1.2**	N
89AZ14-2		113°37.17'	20.6	22	48.9	0.669	5.76	9.36	0.7	11.5	**16.4 ± 1.4**	Y
89AZ14-3			20.6	29	77.1	2.179	3.96	6.22	0.81	11.4	**14.1 ± 0.8**	N
89AZ39-1	735	33°58.77',	29.5	10	86.8	0.318	1.86	0.89	0.83	8.6	**10.3 ± 0.6**	Y
89AZ39-2		113°33.08'	29.5	17	87.9	0.631	1.52	0.36	0.85	11.6	**13.7 ± 0.7**	N
89AZ39-3			29.5	27	88.3	1.146	1.70	0.54	0.85	11.4	**13.5 ± 0.7**	N
DF90 212-1	775	34°05.17',	58.0	15	37.3	0.072	2.98	7.17	0.59	7.0	**11.8 ± 1.5**	N
DF90 212-2		113°15.32'	58.0	10	40.3	0.151	7.40	6.10	0.65	9.6	**14.7 ± 1.5**	N
TC00-32-1	524	33°43.55',	1.9	34	45.2	1.179	5.72	12.61	0.67	17.4	**26.2 ± 2.5**	Y
TC00-32-2		113°41.11'	1.9	34	50.7	1.176	4.96	15.08	0.69	13.1	**19.0 ± 1.7**	N
TC00-32-3			1.9	33	45.1	0.858	4.76	11.52	0.65	13.8	**21.1 ± 2.1**	N
TC00-33-1	582	33°44.88',	4.5	30	45.5	1.632	10.58	17.84	0.67	12.2	**18.3 ± 1.7**	N
TC00-33-2		113°40.29'	4.5	30	48.7	1.883	9.36	17.06	0.69	15.3	**22.0 ± 2.0**	N
TC00-34-1	692	33°51.95',	16.1	26	59.3	0.964	2.86	2.17	0.76	18.5	**24.4 ± 1.7**	Y
TC00-34-2		113°37.54'	16.1	25	55.1	0.863	2.45	5.93	0.73	20.8	**28.5 ± 2.2**	Y
TC00-34-3			16.1	30	47.3	0.466	3.49	6.08	0.68	10.3	**15.0 ± 1.3**	N
TC00-35-1	778	33°57.28',	27.5	26	69.9	0.634	2.16	1.78	0.79	11.0	**14.0 ± 0.9**	N
TC00-35-2		113°33.26'	27.5	28	60.7	0.375	1.93	3.19	0.74	9.7	**13.0 ± 0.9**	N
TC00-35a-1	775	33°57.40',	27.5	33	46.6	0.901	4.52	1.37	0.7	20.0	**28.5 ± 2.4**	Y
TC02-21-1	803	33°51.75',	10.9	18	53.2	0.38	2.75	4.92	0.71	13.8	**19.3 ± 1.6**	N
TC02-21-2		113°41.76'	10.9	29	50.8	0.66	3.03	5.40	0.71	14.0	**19.7 ± 1.6**	N
TC02-20-1	798	33°55.35',	23.0	19	59.1	0.67	3.76	6.52	0.74	11.9	**16.0 ± 1.2**	N
TC02-20-2		113°35.13'	23.0	26	53.3	0.801	4.14	6.43	0.71	13.7	**19.2 ± 1.6**	Y
TC02-20-3			23.0	19	61.6	0.675	3.20	5.80	0.76	10.9	**14.3 ± 1.0**	N
TC02-7-1	820	34°05.21',	57.8	20	57.6	0.17	1.45	1.17	0.75	8.9	**11.9 ± 0.9**	N
TC02-7-2		113°15.57'	57.8	22	74.3	0.62	2.31	0.57	0.81	10.4	**12.8 ± 0.8**	N
TC02-7-3			57.8	39	52.2	0.54	4.06	2.89	0.67	11.8	**17.7 ± 1.7**	Y
TC02-7-4			57.8	37	57.1	0.733	2.80	3.28	0.74	9.9	**13.2 ± 1.0**	N
TC02-15-1[§]	782	33°58.48',	35.7	21	51.7	2.553	20.58	15.96	0.71	**13.7**	19.3 ± 1.6	N
TC02-15-2[§]		113°27.50'	35.7	13	51.8	1.287	17.98	15.53	0.71	**12.5**	17.5 ± 1.4	N
TC02-15-3[§]			35.7	20	52.1	2.4	17.46	15.44	0.71	**14.4**	20.3 ± 1.6	N
TC02-11-1	1115	34°04.77',	54.6	20	58.8	0.16	1.25	0.42	0.76	10.4	**13.7 ± 1.0**	N
TC02-11-2		113°17.87'	54.6	46	50.9	0.4	2.42	1.87	0.67	11.0	**16.3 ± 1.6**	N
TC02-11-3			54.6	47	53.1	0.527	1.58	1.25	0.73	13.3	**18.3 ± 1.4**	Y
TC02-12-1	1314	34°04.40',	52.5	25	54.6	0.596	5.17	1.43	0.73	9.6	**13.1 ± 1.0**	N

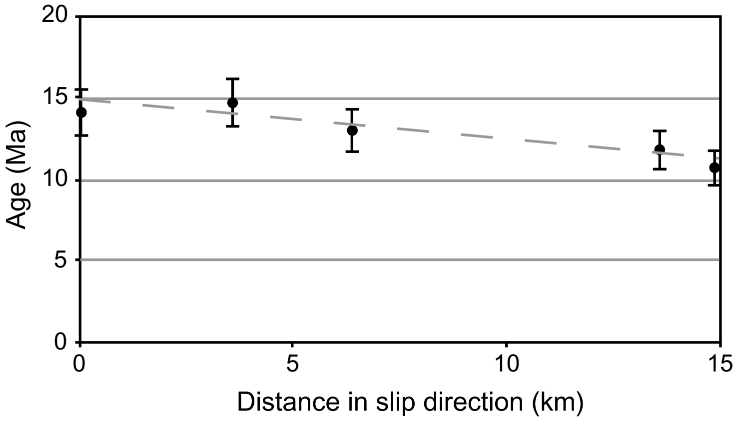

Figure DR1a. Plot of distance in the slip direction against apatite (U-Th)/He age (± 2σ) for footwall rocks in the Buckskin-Rawhide core complex. Error bars represent total estimated errors for weighted average data, according to Brady (2002). Weighted average data reproduced from figure in Brady (2002).

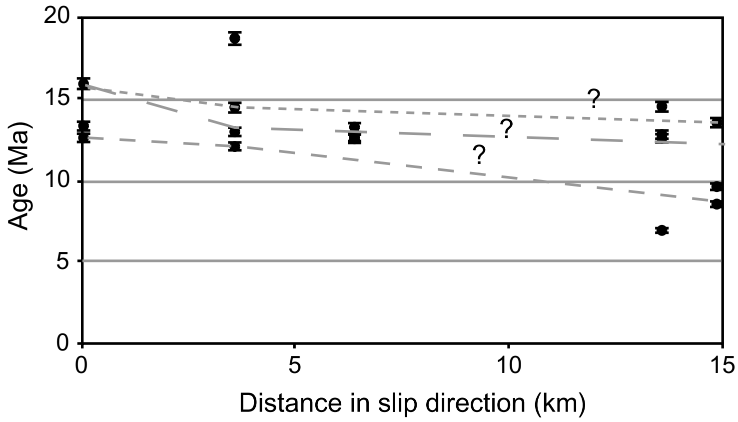

Figure DR1b. Plot of distance in the slip direction against apatite (U-Th)/He age (± 2σ) for footwall rocks in the Buckskin-Rawhide core complex. Error bars represent analytical error for each age determination. Data are individual age determinations reported by Brady (2002). Dashed lines show some of possible interpretations allowed by scatter in data.