

## Data Repository Item 2003155

### Model Explanations, (U-Th)/He Dating Procedures, Data, and Sample Site Photographs

#### HELIUM DIFFUSION MODEL

We determine the fractional He loss from apatite and zircon grains using a two-part numerical model; the first part determines time-temperature paths for individual grains depending on their depth from the surface and the temperature and duration (T-t) of the heating pulse, while the second part determines the loss of He resulting from that T-t event.

We use a 1-D, space-centered, fully explicit (forward in time) finite difference model of the heat diffusion equation to generate time-temperature paths for crystals at different depths beneath the rock surface (Carslaw and Jaeger, 1959, Crank, 1956):

$$\frac{\partial^2 T}{\partial x^2} = \frac{1}{\rho c} \frac{\partial T}{\partial t} \quad (1)$$

where  $T$  is temperature,  $x$  is distance,  $t$  is time, and  $\rho$  is the thermal diffusivity of granite (1 mm sec<sup>-1</sup>, Carslaw and Jaeger, 1959). This finite difference model has a spatial domain of 500 mm, with T-t determined for every mm, with 5-second timesteps. The T-t of a given “wildfire” is forced via the uppermost mm, while the deepest mm is held constant at 20 °C. None of the T-t histories run here caused an increase in temperature at 500 mm depth.

The diffusion of He from spherical, isotropic domains (e.g. Farley, 2000) is described as:

$$\frac{\partial^4 He(r,t)}{\partial t^4} = \frac{D(t)}{a^2} \left( \frac{\partial^2 He(r,t)}{\partial r^2} - \frac{2}{r} \frac{\partial He(r,t)}{\partial r} \right) \quad (2)$$

where  ${}^4He(r,t)$  is the concentration of He at a particular time,  $r$  is the radial distance from the center of the sphere,  $t$  is time, and  $D$  is a time-dependent diffusion coefficient (Carslaw and Jaeger, 1959). This diffusion coefficient is related to a T-t history by the Arrhenius equation:

$$D = D_0 \exp\left(-\frac{E_a}{RT(t)}\right) \quad (3)$$

where  $\log(D_0)_{\text{apatite}} = 1.5$  cm<sup>2</sup>/s,  $E_{a(\text{apatite})} = 33$  kcal/mol (Farley, 2000), and  $\log(D_0)_{\text{zircon}} = -0.19$  cm<sup>2</sup>/s,  $E_{a(\text{zircon})} = 40.3$  kcal/mol (Reiners et al., in press).  $R$  is the gas constant (1.987 cal/Kmol), and  $T(t)$  is the time-dependent thermal history (Wolf et al., 1998; Farley, 2000), and  $D_0$  is the frequency factor and  $E_a$  is the activation energy. Because the temperature beneath the rock surface varies through time, we introduce a dimensionless variable:

$$\vartheta = \frac{D_0}{TMa^2} \int_0^t \exp\left(-\frac{E_a}{RT(\xi)}\right) d\xi \quad (4)$$

where  $a$  is 50 μm, the average radius of our measured apatite grains (e.g. Brandt, 1974; Dodson, 1975; Gillespie et al., 1982; McDougall and Harrison, 1999). We used our model to calculate  $\vartheta$

for a square pulse temperature history and compared it to the analytical solution for  $D$  and verified that both give the same answer. Finally, the overall fraction of He lost ( $f$ ) for the apatite grain as a function of  $t$  is given by:

$$f = 1 - \frac{4}{6} \frac{D}{\phi^2} \left( \frac{1}{n^2} \left( \exp\left(\frac{4}{n^2} \phi^2 t\right) - 1 \right) \right) \quad (5)$$

which we approximated for different fractional loss intervals using the following equations from McDougall and Harrison (1999):

$$0.85 \leq f < 1 \quad f = 1 - \frac{4}{6} \frac{D}{\phi^2} \exp\left(\frac{4}{n^2} \phi^2 t\right) \quad (6)$$

$$0 \leq f < 0.85 \quad f = \frac{8}{15} \frac{D}{\phi^{2/3}} \left( \frac{1}{\phi^2} t \right)^{3/2} - \frac{4}{3} \frac{D}{\phi^2} \left( \frac{1}{\phi^2} t \right) \quad (7)$$

### (U-Th)/He CHRONOMETRY

All apatite and zircon ages are single-grain analyses, performed at Yale. The procedure for apatite generally follows that of House et al. (2000). Crystals were hand-picked from separates with high power (160x) stereo-zoom microscopes with cross-polarization for screening inclusions (all apatites were free of visible inclusions using this procedure). Selected crystals were measured and digitally photographed in at least 2 different orientations, and loaded into 1-mm Pt foil tubes, which were then loaded into stainless steel sample planchets with 32 sample slots. Planchets were loaded into a ~10 cm laser cell with sapphire window, connected by high-vacuum flexhose to the He extraction/measurement line. Once in the laser cell and pumped to  $<10^{-7}$ - $10^{-8}$  torr, crystal-bearing foil tubes were individually heated by lasing with about 1-5 W on the Nd-YAG laser, for three minutes for apatite or 20 minutes for zircon. Temperatures of heated foil packets were not measured, but from experiments relating luminosity and step-wise degassing of both apatite and zircon, we estimate typical heating temperatures of 900-1000 °C for apatite, and 1250-1400 °C for zircon.  $^4\text{He}$  blanks (0.05-0.01 femtomol  $^4\text{He}$ , after correction for  $^4\text{He}$  in the spike) were determined by heating empty foil packets using the same procedure. Crystals were checked for quantitative degassing of He by sequential reheating. In this study, neither apatites nor zircons exhibited residual gas after the first extraction. Gas liberated from samples was processed by: 1) spiking with ~0.4 pmol of  $^3\text{He}$ , 2) cryogenic concentration at 16K on a charcoal trap (condensation time calibrated for no significant  $^4\text{He}/^3\text{He}$  fractionation), and purification by release at 37K, and 3) measurement of  $^4\text{He}/^3\text{He}$  ratios (corrected for HD and  $\text{H}_3$  by monitoring  $\text{H}^+$ ) on a quadrupole mass spectrometer next to a cold Zr-alloy getter. All  $^4\text{He}/^3\text{He}$  sample ratios were referenced to multiple same-day measured  $^4\text{He}/^3\text{He}$  ratios of  $^3\text{He}$ -spiked  $^4\text{He}$  standards of known volume processed in the same way. Linearity of the  $^4\text{He}/^3\text{He}$  ratio with  $^4\text{He}$  pressure over the range of observed unknown  $^4\text{He}/^3\text{He}$  ratios has been confirmed by spiked  $^4\text{He}$ -standard gas-splitting, with varying  $^4\text{He}$  pressure over more than three orders of magnitude, and age determinations of Durango apatite aliquots with varying  $^4\text{He}$  intensities over more than four orders of magnitude.  $^4\text{He}$  standard reproducibility averages 0.2% on a daily and long-term (tank-depletion corrected) basis. Estimated 2 $\sigma$  analytical uncertainty on sample He determinations, including precision and accuracy from original manometric  $^4\text{He}$  standard calibrations, is 1-2%.

Following degassing, samples were retrieved from the laser cell, spiked with a calibrated  $^{229}\text{Th}$  and  $^{233}\text{U}$  solution, and dissolved. Apatites were dissolved *in-situ* from Pt tubes in ~30%  $\text{HNO}_3$  in teflon vials. Zircons were removed from foil and then dissolved in teflon microvials in Parr bombs with  $\text{HF}$  and  $\text{HNO}_3$ , followed by  $\text{HCl}$  and/or  $\text{H}_3\text{BO}_3$  to remove fluoride salts, and a final dissolution in  $\text{HNO}_3$ . Each sample batch was prepared with a series of procedural blanks (including Pt foil blanks for apatite) and spiked normals to check the purity and calibration of the reagents and spikes. Spiked solutions were analyzed as 0.5 ml of ~1-5 ppb U-Th solutions by isotope dilution on a Finnigan Element2 ICP-MS with a teflon micro-flow nebulizer and double-pass spray chamber. Procedural U and Th blanks by this method are  $1 \pm 0.5$  pg and  $2 \pm 1$  pg, respectively. Routine in-run precisions and long-term reproducibilities of standard  $^{232}\text{Th}/^{229}\text{Th}$  and  $^{238}\text{U}/^{233}\text{U}$  are 0.1-0.4%, and estimated uncertainty on sample U-Th contents are estimated to be 1-2% ( $2\sigma$ ).

Alpha ejection was corrected using the method of Farley (2002). Replicate analyses of Durango apatite and Fish Canyon Tuff zircon during the period of these analyses yielded mean ages of  $32.5 \pm 1.6$  Ma ( $2\sigma$ ) and  $27.8 \pm 2.4$  Ma ( $2\sigma$ ), respectively. On the basis of reproducibility of these and other intralab standards, we estimate an analytical uncertainty of 6% ( $2\sigma$ ) for apatite and zircon age determinations in this study.

**TABLE DR-1. U, Th, He DATA, He AGES, AND FRACTIONAL He LOSS**

Sample ID	Average dist. from surface (cm)	He (ncc)	U (ppm)	Th (ppm)	Grain radius ( $\sigma\text{m}$ )	Corrected (U-Th)/He age (Ma)	Fractional He loss
<u>Apatite</u>							
SGM01aC	0.5	0.237	76.2	196	52.5	5.31 $\pm$ 0.32	0.72 $\pm$ 0.06
SGM01aD	0.5	0.056	72.2	115	34.3	1.91 $\pm$ 0.11	0.90 $\pm$ 0.06
SGM01aE	0.5	0.085	74.7	135	59.3	1.98 $\pm$ 0.12	0.90 $\pm$ 0.06
SGM01aF	0.5	0.482	80.7	152	51.3	10.5 $\pm$ 0.63	0.46 $\pm$ 0.07
SGM12aA	1.5	0.625	50.6	90.0	51.8	15.8 $\pm$ 0.95	0.19 $\pm$ 0.08
SGM12aB	1.5	0.422	70.9	125	43.5	16.3 $\pm$ 0.98	0.17 $\pm$ 0.08
SGM12aC	1.5	1.570	76.7	108	70.3	17.6 $\pm$ 1.1	0.10 $\pm$ 0.08
SGM23aC	2.5	0.320	77.6	138	41.5	18.2 $\pm$ 1.1	0.07 $\pm$ 0.08
SGM23aD	2.5	0.164	93.7	159	32.8	17.0 $\pm$ 1.0	0.13 $\pm$ 0.08
SGM23aE	2.5	1.310	84.6	150	65.8	18.0 $\pm$ 1.1	0.08 $\pm$ 0.08
SGM03aA	> 3	0.885	83.1	152	50.0	19.5 $\pm$ 1.2	0
SGM03aB	> 3	0.401	38.5	53.5	48.3	18.4 $\pm$ 1.1	0.06 $\pm$ 0.08
SGM03aC	> 3	0.828	88.5	160	45.3	18.8 $\pm$ 1.1	0.04 $\pm$ 0.08
SGMchipaA	N.A.	0.233	74.9	142	37.3	17.2 $\pm$ 1.0	N.A.
SGMchipaB	N.A.	0.246	85.6	138	40.3	11.9 $\pm$ 0.71	N.A.
SGMchipaD	N.A.	0.108	109	155	31.5	9.67 $\pm$ 0.58	N.A.
<u>Zircon</u>							
SGM01zA	0.5	4.54	126	36.5	47.5	54.7 $\pm$ 3.3	0.15 $\pm$ 0.08
SGM12zA	1.5	3.79	114	34.2	42.8	60.1 $\pm$ 3.6	0.07 $\pm$ 0.08
SGM23zA	2.5	19.6	89.5	24.7	82.5	64.0 $\pm$ 3.8	0.01 $\pm$ 0.09
SGM3zA	> 3	13.3	293	61.6	49.0	64.5 $\pm$ 3.9	0

## REFERENCES CITED IN REPOSITORY

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- Carslaw, H.S. and Jaeger, J.C., 1959, *Conduction of Heat in Solids*, Second Edition: Oxford, Clarendon Press, 510 p.
- Crank, J., 1956, *The Mathematics of Diffusion*: Oxford, Clarendon Press, 347 p.
- Dodson, M.H., 1975, Kinetic processes and thermal history of rocks: *Carnegie Institute of Washington Year Book*, v. 74, p. 210-217.
- House, M.A., Farley, K.A., and Stockli, D., 2000, Helium chronometry of apatite and titanite using Nd-YAG laser heating: *Earth and Planetary Science Letters*, v. 183, no. 3-4, p. 365-368.

## DR FIGURE CAPTIONS

Figure DR-1. Bedrock sample site. Arrow shows location of sample. One cm-thick subsamples were cut parallel to the vertical surface.

Figure DR-2. Spalled chips sample site. Arrows point to chips on the ground, distances from boulder are  $< 1$  m. These chips were highly angular with sharp edges, suggesting a very localized source (i.e. the boulder). In addition, the chips lay on top of the sooty soil layer of the previous year (2001), yet beneath the most recent (2002) layer of pine needles, suggesting that the chips spalled from the main boulder sometime shortly following the fire of 2001.

**SAMPLE COLLECTION PHOTOGRAPHS**



Figure DR-1

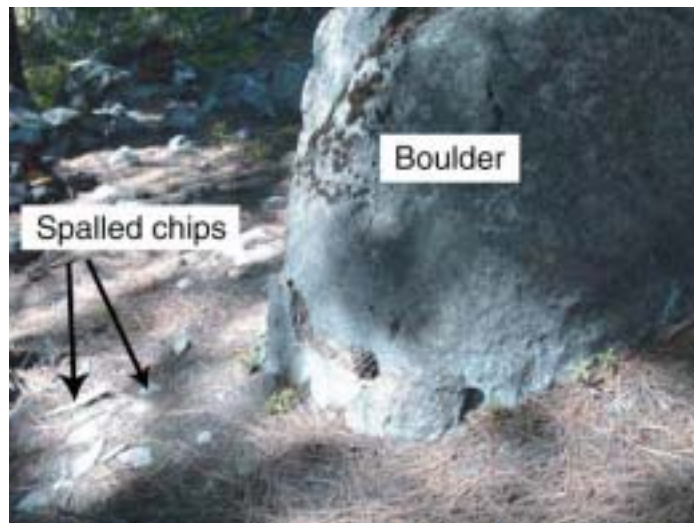


Figure DR-2