Fission-track investigations on sphene from the KTB Deep Drilling Project (Germany): Post-Permian cooling history and *in situ* annealing

David A. Coyle and Günther A. Wagner Max-Planck-Institut für Kernphysik, Saupfercheckweg 1 69117 Heidelberg

Abstract

Seventeen samples spanning the entire depth range of the KTB *Hauptbohrung* have had their sphene fission-track data analysed. The results presented here give important insights into the thermal stability of fission tracks in sphene, and the post-Permian, and particularly Cretaceous, tectonic history of the KTB site. Fission tracks in the KTB sphenes are found to be stable to higher temperatures than was previously inferred. The final temperature at which these tracks anneal completely is now thought to be at least 300±20°C. Two episodes of substantial crustal cooling—presumably related to tectonically driven uplift and denudation—occurred both in the Triassic at ca. 245 Ma, and in the upper Cretaceous at ca. 95 Ma. Furthermore, a minimum 3.5 kilometers Cretaceous net vertical displacement of the paleoisotherms is observed across the SE1 reflector-Franconian Line.

Introduction

Fission track analysis of minerals recovered by the German Continental Deep Drilling Program KTB provides unique information about the long-term retention properties of fission tracks under geological conditions. Whereas the *Vorbohrung* (final depth 4000 m) allowed to study the fission-track system of apatite, the *Hauptbohrung* (present depth 8730 m) now opens the possibility to investigate the fission-track (FT) systems in sphene and zircon. At the maximal temperature (118°C) of the *Vorbohrung* the FT-ages of apatite are nearly zero which agrees with previous observations in other bore-holes (Wagner & Van den haute 1992). Fission tracks in zircon and sphene are known to be more heat-resistant but exact information is still missing. The high temperatures within the *Hauptbohrung* enable for the first time to study the track annealing behaviour of these minerals under *in situ* p-T-conditions. Such basic data is essential for the interpretation of FT-analyses. However, for the KTB-site itself, the potential of fission tracks to unravel the thermal evolution of the rocks within the brittle temperature region is undoubtedly of wider interest. From the FT-age and length data ultimately the Postvariscan tectonic as well as the denudation history may be derived. In this contribution we report on the current state of our FT-studies of sphene from the KTB rocks.

Methodology

Fission tracks are linear damage trails formed in a crystal lattice as a result of the spontaneous fission of 238 U. They form at a constant rate over time, and the mechanics of calculating a fission-track age are similar to those for any other isotopic dating method. The tracks, having a diameter of ca. 40 Å, are normally unobservable, but can be made visible by chemically

etching a polished internal surface of the crystal. The measured density of tracks per unit area represents the *daughter* concentration of the system. The parent concentration is determined by placing a detector, a sheet of U-free muscovite, next to the polished crystal and irradiating the two together with thermal neutrons. The thermal neutrons induce ^{235}U in the sample to fission, with some of the fission fragments entering into the detector so that they may in turn be revealed. Because the isotopic ratio of 238U/235U is uniform, the calibrated ratio of spontaneous to induced tracks will give the fission-track age of the crystal.

The two calibration factors used in calculating the age are zeta and ρ_D . Zeta is a calibration against standards of known age, (Table 1), performed independently of the sample dating, and pp is a calibration against a standard (a homogeneous glass) of known U content, and is used for calibrating the thermal neutron fluence of the individual irradiations. This yields the fission-tack age equation:

$$\tau = (1/\lambda_d) \ln(1 + \lambda_d \zeta (\rho_s/\rho_i) \rho_D 0.5)$$

where λ_d is the total uranium decay constant and ρ_s, ρ_i are the densities of spontaneous and induced tracks, respectively (Fleischer and Hart, 1972).

Sample	N	ρs (cm ⁻² 10 ⁶)	Ns	ρi (cm ⁻² 10 ⁶)	Ni	P(χ2) %	ρ _D (cm ⁻² 10	ND 1 ⁶)	zeta a cm ²	error (1σ)
FCT1	20	6.922	1231	15.36	2732	39	0.942	4747	128.4	4.8
FCT2	20	7.508	1279	15.78	2688	9.3	0.942	4747	124.4	4.2
FCT3	20	6.923	1277	16.27	3002	70.	0.992	4652	132.0	4.9
MTD1	10	55.53	2099	31.46	1189	0.1	0.942	4747	119.7	7.6
MTD2	11	52.14	1235	30.78	729	16.	0.942	4747	124.7	6.1
					~				105.05	

125.85

Table 1: Zeta calibration values (Hurford, 1990). FCT: Fish Canyon Tuff, 27.8 Ma; MTD: Mt. Dromedary banatite, 98.7 Ma. Nx are the actual numbers of tracks counted in each category

Age estimates are made for a collection of individual crystals. Because of the complex mechanics of track annealing, and the possible influence of composition on the annealing rate, it is possible to have *overdispersion* of the single-grain ages within a single sample. In such cases, a statistical test such as the χ^2 test will fail (Galbraith 1981). Graphical inspection of the single grain estimates on a radial plot (Galbraith, 1990) will often indicate the type of overdispersion. Occasionally it is the case that the grains fall into a number of discrete components: when this happens, the component ages can be recovered (Galbraith & Green, 1990).

The other information that is extracted from fission-track data comes from the length of tracks. To eliminate as far as possible any observational biases, only horizontal and confined tracks have their lengths measured (Bhandari et al, 1971). The annealing process by which tracks shorten with time begins even at ambient Figure 1: Induced tracks in Mt. Dromedary sphene. temperatures, so the length distribution of Left: plot of length versus the acute angle to the induced tracks is convenient as a maximum Mean length: 13.35 µm, Standard deviation: 0.95.µm reference (Figure 1). Note that the strongly



direction of fastest etching. Right: length histogram.

-	А	65	-
---	---	----	---

Sample	Ν	ρs	Ns	ρί	Ni	P(χ2)	ρ _D	ND	Age	error
		$(\text{cm}^{-2} 10^6)$		$(cm^{-2} 10^6)$)	%	(cm ⁻² 10 ⁶)		(Ma)	(lo)
HC510b	13	27.64	1279	6.981	323	88.	0.9919	4652	243.	16.
HDSP234	12	27.55	836	6.625	201	18.	0.9419	4747	242.	19.
HDSP237	13	40.25	1623	11.11	448	9.5	0.9419	4747	211.	12.
HC2650	16	33.54	2174	8.47	549	13.	0.9919	4652	243.	12.
HDSP238	14	37.43	1777	9.478	450	12.	0.9419	4747	230.	13.
HDSP241	13	19.84	786	4.998	198	51.	0.9419	4747	231.	19.
HF4350	13	2.027	1052	0.6761	351	6.7	0.954	4808.	177.	11.
HF4500	13	1.468	993	0.4066	275	38.	0.954	4808.	213.	15.
H010-B9	11	14.17	240	6.437	109	23.	0.9419	4747	129.	15.
HC5573	15	4.892	466	2.803	267	12.	0.9919	4652	108.	8.4
H027-D2	10	1.147	548	0.7011	335	86.	0.954	4808.	97.5	6.9
H029	8	11.01	152	5.793	80	55.	0.9419	4747	112.	16.
HC7368	18	15.27	1025	4.305	289	0.29	0.9919	4652	212.	20.
HC7454	17	14.71	762	5.597	290	29.	0.9919	4652	162.	11.
HC7688b	20	18.16	1071	7.275	429	39.	0.9919	4652	154.	9.1
HC8250	20	1.959	1635	0.6973	582	5.3	0.954	4808.	166.	8.4
HC8700	20	1.964	1241	1.343	849	0	0.954	4808.	111.	16.

Table 2: Fission-track data from the KTB-HB. ρ_X is the track density in number of tracks per cm². N_X is the number of tracks observed, where x = s: spontaneous, i: induced, D: dosimeter. Dosimeter is CN1 (39 ppm [U]). Samples are in order of increasing depth.

anisotropic etching of sphene is recognizable in the spatial distribution of the *observable* tracks in sphene. This effect means that one observes fewer confined tracks than are really there. This effect becomes more pronounced as annealing progresses.

Local Geology

The KTB location is situated in the Bohemian Massif near its western border. The surrounding area consists of three main tectono-metamorphic units: the Saxothuringikum, the Moldanubikum and the Ebendorf-Vohenstrauss-Zone (ZEV) in which the KTB actually is located. Each of them is characterized by an individual metamorphic history. During the Variscan orogeny the Moldanubikum was thrusted northwards onto the Saxothuringikum. After their collision both units were affected by a regional low-pressure metamorphism about 320 Ma. The ZEV complex—not affected by the latter metamorphic event—underwent a medium-pressure metamorphism of Devonian age. This complex covers the Saxothuringian-Moldanubian suture and is predominantly composed of metapelites and amphibolites. After the low-pressure metamorphism, post-tectonic granites intruded the region between 320 and 290 Ma.

About 5 km west of the KTB location the crystalline basement of the Bohemian Massif is separated from the South German sedimentary basin by the Franconian Line. This NW-SE striking fault system dips towards the NE with ca. 45° underneath the basement. It appears as seismic reflector SE1 and intersects the KTB drill hole as a thick cataclastic zone at ca. 7000 m depth . The Permo-Mesozoic sediments west of the Franconian Line are between 1000 and 3000 m thick. Since the Latest Cretaceous the sedimentary area has undergone denudation. The regional distribution and polymict character of coarse sized clastic sediments reveal

strong uplift and denudation of the western Bohemian Massif during the Early Permian, the Triassic and the Upper-Cretaceous (Schröder 1987, 1990).

The temperature profile of the borehole is well documented by temperature logs and by several long-term measurements at different depths. The temperature increases from an annual mean of 8°C at the surface to ca. 250°C at the bottom of the hole in 8730 m depth. This corresponds to an average thermal gradient of 27.7 K/km.

Results

Seventeen uniformly-spaced samples from the KTB-HB were analysed for their sphene fission-track data. The age data are summarized in Table 2, depths for samples numbered

"HCxxxx" are the integer portion, in metres. Other sample depths are: HDSP234: 1391, HDSP237: 2470, HDSP238: 2700, HDSP241: 3900, HO10-B9: 4820, HO27-D2: 6357, and HO29: 6540 metres. All samples are metabasite (amphibolite). Confined track length measurements were performed on most samples, but counts were often low and no meaningful trends downhole were observed, so this data is omitted. Note that all of the samples in Table 2 passed the χ^2 test, with the exception of samples HC7368 and HC8700. For sample HC7368, no identifiable components or structure to the single-grain ages were observed, nor could components be meaningfully extracted,

Component	Age	95%
	(Ma)	Confiden
		ce
		Interval
		82.2
Young	94.2	
		107.9
		142.1
Old	185.9	
		243.1

Table 3: Individual age components for sample HC8700. Note that the 95% confidence intervals do not overlap.

and so the data is simply overdispersed (Galbraith & Laslett, 1993), which may have a variety of causes.

In comparison, sample HC8700 shows clear evidence of two components upon visual inspection of the radial plot (Fig. 2) and a numeric two-component analysis yielded a good separation with a young component at 94.2 Ma. The older component is interpreted as being a partially annealed component, with the age being of no geological significance. If one imagines that the sample had been sitting very low in the partial annealing zone in the Cretaceous, with the ages of *most* of the grains reset to zero, those zero-age grains will record the timing of uplift, while the grains that had retained a portion of their original age now have an age which is some combination of that partially annealed component, and the time since cooling. Such distributions of single-grain ages in highly annealed samples are most often related to chemical variation between the grains, which can stongly affect their annealing characteristics. The temperature to which this sample had been subjected prior to uplift at ca. 95 Ma can only be guessed at (see below), but it was likely around $300^{\circ}C$ (±20).

All other ages in Table 2 can be inferred to be true cooling ages, recording the time when the sample cooled through the sphene fission-track partial annealing zone. Without accurate length information and given the strong biases in measuring the lengths using present techniques, it is not possible to estimate the relative rate of the cooling, nor can a length "correction" to the age, compensating for the effect of slow cooling, be applied. Experiments on enhancing the measurement of confined track lengths in sphene are being carried out.

Discussion

i) in situ annealing

Prior estimates of the temperatures at which fission tracks anneal in sphene have been based upon either extrapolations from laboratory experiments, or interpolations within a suite of different mineral ages from a single sample (ie: a sphene fission-track age usually lies between Rb/Sr biotite, and apatite fission-track ages). These latter estimates realistically have ranged between ca. 180°C and ca. 300°C, with the most widely held value that of Harrison *et al.* (1979), who estimated that fission tracks in sphene anneal at ca. 240°C.

That tracks are still retained at a present-day temperature of ca. 250°C allows us to state unequivocally that fission tracks in sphene in the KTB do not anneal at Harrison *et al*'s 240°C. The question remains: at what temperature *do* the tracks begin to anneal? There is good evidence that even at this temperature, the top of the partial annealing zone has not been intersected by the KTB. Consider the two data types: the lengths of the tracks at 8700 metres, and the distribution of single grain ages.

There is no significant difference between the lengths distributions for the sample at 510

m and that from 8700 m (Figure 2). If the sample at 8700 m were presently within the annealing zone, no long tracks would be observed. In fact, the longest tracks are proportionally more abundant than those from sample HC510. The evidence based upon the single-grain age distribution is more subtle. Note that the radial plot for sample HC8700 has one distinct component at 94 Ma (also Table 3). This component represents a "minimum age": a component that had been fully annealed at some point in the past and was subsequently cooled. Cooling took place in the Cretaceous, and this event corresponds to the cooling profile seen immediately above the Franconian Line, discussed below. If the sample were presently annealing, there would be no discrete young component, nor would



Figure 2: Comparison of single-grain age plots (radial plots) and confined length data for samples HC510 and HC8700

the younger single-grain ages be likely to correspond to a known event. Thus it is likely that at 8700 m and a temperature of ca. 250°C, the partial annealing zone of sphene has not yet been entered into.

The only mineral system for which the fission-track partial annealing zone is known is apatite. The partial annealing zone for apatite is ca. 60°C "wide". Naively applying this figure to sphene suggests that the temperature at which fission tracks will totally anneal in

sphene will not be less than 300°C. It is unlikely that the partial annealing zone for sphene will be exactly 60°C wide, and this will be checked by a more detailed examination of the uplifted partial annealing zone in the region 4000–5000 m. There are as yet too few data from this zone to determine if it is intact, or has been in any way disturbed by faulting, which would cause it to appear either thicker (reverse faulted) or thinner (normal faulted).

Mineral chemistry is known to have an effect on the annealing of fission tracks in apatite (Gleadow and Duddy, 1981). The corresponding effect of sphene composition on track stability has yet to be investigated, so the above preliminary temperature estimates must be regarded as valid for the KTB sphenes only.

ii) Post-Permian uplift

The plot of sphene fission-track age versus depth shows several well-defined features related to the uplift and cooling of the KTB site.

1). There is a near vertical age gradient between the surface and ca. 4000 m. This section has an early-middle Triassic age of ca. 245 Ma. This indicates that there was at least 3km and possibly up to 4 km of denudation at this time. Apatite fission-track data from the *Vorbohrung* indicates that there is the possiblility of a measurable amount of post-Cretaceous stacking of the section here (Wagner et al, 1994), so the lower figure (3 km) is the more reasonable one. Overall, assuming a gradient the same as today's, the total post-Permian denudation of the KTB site amounts to 10-11 km.

2) There is a rapid decrease in the apparent ages between approximately 4000 and 5000 m, followed by a second steep gradient of Cretaceous ages. In form this is very similar to uplifted apatite partial annealing zones, and is interpreted to be the first such uplifted partial annealing zone ever observed for sphene.

3) Below the uplifted partial annealing ages, depending upon whether the sample has zone, there is a steep age gradient down to passed or failed the χ^2 test, respectively



Figure 3: Plot of sample age against depth. Note that the ages plotted are the pooled or central ages, depending upon whether the sample has passed or failed the χ^2 test, respectively

the position of the Franconian Line (SE1 reflector), giving Cretacous ages of ca. 95 Ma. This is interpreted to be a second phase of rapid uplift, in the Cretaceous, an event also observed in the apatite fission-track data from the upper section of the *Vorbohrung* (Wagner et al, 1994).

4) Across the region of the Franconian Line, there is a marked jump in ages, from the Cretaceous back up to the Triassic. This is clear evidence of reverse motion along the

Franconian Line, causing the profile to repeat itself. The vertical displacement across the fault is some 3.5-4 km.

5) Below the Franconian Line down to the total depth at 8700 m, the ages decrease again, in what is also most likely an uplifted partial annealing zone. The young component of age of the deepest sample indicates that this section too was uplifted in the Cretaceous, by an amount equal to or greater than the thickness of the partial annealing zone.

The timing of the most recent movement along the Franconian is well constrained by the fission-track data. There was significant uplift of the lower block at the same time as reverse motion and large displacement along the Franconian Line, all happening in the upper Cretaceous, beginning at ca. 95 Ma. This is corroborated by the apatite fission-track data from the *Vorbohrung*, which also indicates at least 2 km of denudation in the upper Cretaceous.

Conclusions

The sphene fission-track data from the KTB-HB is proving to be of great value in reconstruting the thermal, and therefor tectonic, history of the KTB site. The great vertical distance over which the samples are available allows us to peer as far back as the Triassic, and when coupled with the fission-track data from apatite, almost up to the present time. Several issues still need to be resolved, however; most notable among them being the smaller-scale effects of minor faults on the age-depth profile, and also resolving as accurately as possible the true temperatures at which fission-tracks in sphene anneal, information vital to the final thermal interpretation of the KTB site, and all subsequent projects that make use of sphene fission-track data.

References

- N. Bhandari, S.G. Bhat, D. Lal, G. Rajagopalan, A.S.J. Tamhane & V. Venkatavaradan, 1971: Fission fragment tracks in apatite: recordable track lengths. Earth and Planetary Science Letters, 13., pp 191–199.
- R.L. Fleischer and H.R. Hart, 1972: Fission track dating: techniques and problems. in W.W. Bishop, D.A. Miller and S. Cole (eds), *Proc. Burg Wartenstein Conf. on Calib. of Hominid Evolution*, Scott. Acad. Press, Edinburgh, pp 135–170.
- R.F. Galbraith, 1981: On statistical models for fission track counts. Mathematical Geology, 13, pp 471–438.
- R.F. Galbraith, 1990: The radial plot: Graphical assessment of spread in ages. Nuclear Tracks and Radiation Measurements, 17, no. 3, pp 207–214.
- R.F. Galbraith and P.F. Green, 1990: Estimating the component ages in a finite mixture. Nuclear Tracks and Radiation Measurements, **17**, no. 3, pp 197–206.
- R.F. Galbraith and G.M. Laslett, 1993: Statistical models for mixed fission track ages. Nuclear Tracks and Radiation Measurements, **21**, no. 4, pp 459–470.
- A.J.W. Gleadow and I.A. Duddy, 1981: A natural long-term annealing experiment for apatite. Nuclear Tracks and Radiation Measurements, **5**, pp 169–174.
- T.M. Harrison, R.L. Armstrong, C.W. Naeser & J.E. Harakal, 1979: Geochronology and thermal history of the Coast plutonic complex, near Prince Rupert, British Columbia, Canadian Journal of Earth Sciences, 16, pp 400–410.

- A.J. Hurford (secretary), 1990: International Union of Geololgical Sciences Subcommission on Geochronology: Recommendation for the standardization of fission track dating calibration and data reporting. Nuclear Tracks and Radiation Measurements, 17, no. 3, pp 233–236.
- B. Schröder, 1987: Inversion tectonics along the western margin of the Bohemian Massif. -Tectonophysics, 137, pp 93-100, Elsevier (Amsterdam).
- B. Schröder, 1990: Spät- und postvarikische Schollentektonik des KTB-Umfeldes. KTB Report, 90-4, pp 293–299.
- G.A. Wagner, E. Hejl and P. Van den haute, 1994: The KTB fission-track project: methodical aspects and geological implications. Radiation Measurements, **22**, *in press*.
- G.A. Wagner and P. Van den haute, 1992: Fission-track Dating. Kluwer Academic Publishers (Dordrecht), 285 pp.