

Dating Single Zircon by Fission-Track and U-Pb Methods-A Case Study on a Granite at the Cooper Basin HFR Site, Australia-

Hisatoshi Ito

Central Research Institute of Electric Power Industry, 1646 Abiko, Abiko city, Chiba 270-1194, Japan

ito_hisa@criepi.denken.or.jp

Keywords: dating method, zircon, fission-track, U-Pb, Cooper Basin

ABSTRACT

Both fission-track (FT) and U-Pb dating methods have contributed to determining the timing of volcanic activity and the thermal history of granitic plutons. Zircon FT dating provides information at a temperature of ~250-300 °C and zircon U-Pb determines the timing of zircon crystallization. If both dating techniques are applied to the same zircon crystal, a more clear understanding is possible for the thermal history of the dated zircon crystal and its host rock, while to date few attempts have been done in this regard.

Here, these two dating methods were applied to a granitic host rock at the Cooper Basin Hot Fractured Rock (HFR) site, Australia. Firstly, a zircon FT age of ~170 Ma was obtained. Together with zircon FT length data, it was assumed that the granite has experienced much higher temperature (280-320°C) than the present one (220-250°C) during a later stage of its subsidence history. Secondly, analyzing the same zircons used for FT dating, a reliable LA-ICP-MS U-Pb age of ~330 Ma was obtained. Therefore it was revealed that both FT and U-Pb dating are applicable using the same zircon grains.

1. INTRODUCTION

Radiometric dating techniques have contributed not only to determine timing of geologic events but also to provide geothermal information. Among them, FT dating using zircon has been widely applied to date volcanic activity by determining mainly acidic volcanic products such as tuffs and lavas and to constrain the thermal history of granitic rocks. Compared to the FT method, whose dating range is ~0.1 – 100 million years, U-Pb dating has been far less applied for geothermal field, probably because its suitable dating range had been older (>100 million years) and availability of dating equipments had been limited.

Recently, U-Pb dating method has gained much more popularity mainly because of the advent of Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS). U-Pb dating by LA-ICP-MS is much easier than the other techniques (TIMS and SIMS/SHRIMP) and is applicable to dating of zircons as young as 1 Ma.

Both FT and LA-ICP-MS U-Pb methods can date using small areas on zircon surface, which means it is possible to obtain ages by both methods using the same zircon grain. In this case, the age information is much more valuable than that obtained by different grains. For example, non-essential zircons may be included in cuttings samples from a wellbore. If a single zircon grain has both FT and U-Pb age data, it is easier to discriminate whether it is essential or

non-essential. Moreover, both dating methods have significantly different thermal properties: zircon FT dating system provides information at a temperature around ~250-300 °C and zircon U-Pb determines the timing of zircon crystallization. Revealing thermal history of individual grains should contribute to better understand thermal and tectonic history in the study area.

2. GEOLOGICAL BACKGROUND IN THE COOPER BASIN

The Cooper Basin Hot Fractured Rock (HFR) site is situated in southwestern part of the Cooper Basin and also in northeastern part of South Australia (Figure 1). The Cooper Basin is the place where geothermal gradient is the highest in Australia. Thousands of wells were drilled in the Cooper Basin by oil industry. The HFR site was selected at a place where granite resides at a relatively shallow depth.

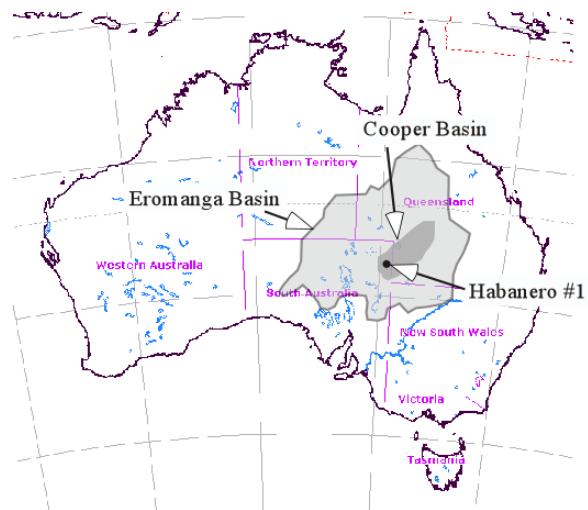


Figure 1: Position of Cooper Basin HFR site (well Habanero #1) and related basins

Seismic reflection survey shows that ridge and trough structure extends ENE-WSW (Figure 2). This ridge and trough structure is believed to have been formed mainly by glaciations and hence the granite has a weathered zone up to 50 m (Gravestock and Jensen-Schmidt, 1998). The HFR site is situated at a small ridge in a larger trough (Figure 2).

The granite is named the Big Lake Suite whose emplacement age was determined at 300 – 320 Ma by SHRIMP U-Pb method (Gatehouse et al., 1995). Immediately after its emplacement, the granite experienced rapid uplifting and erosion followed by two major stages of subsidence: the Permian-Triassic Cooper and the Jurassic-Cretaceous Eromanga subsidence. After the Eromanga subsidence the depth of the top of the granite remained relatively stable (Figure 3).

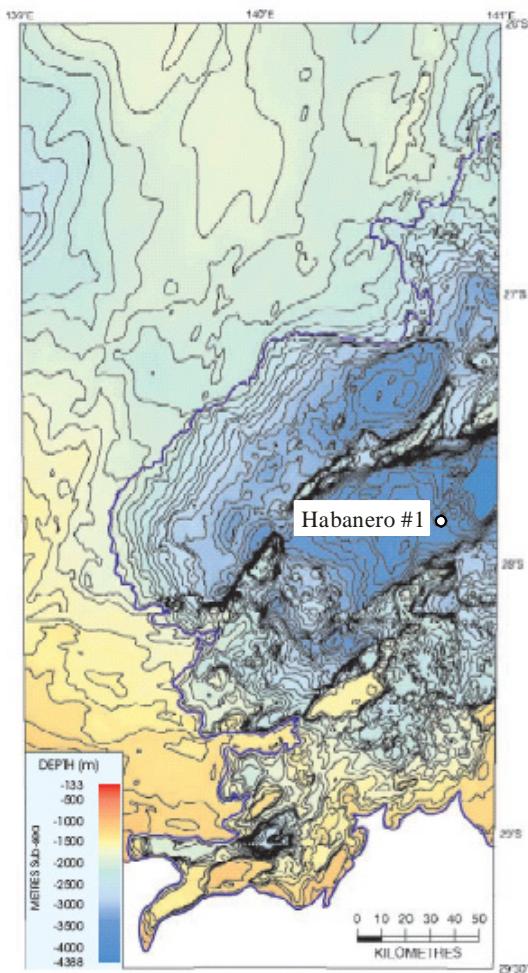


Figure 2: Base of the Cooper Basin depth structure contour map (after Gravestock and Jensen-Schmidt, 1998)

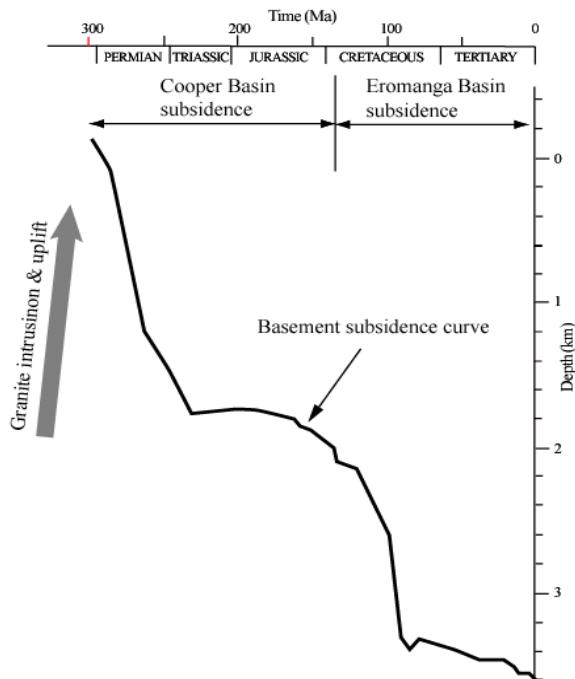


Figure 3: Simplified subsidence history of the Cooper Basin HFR site (modified from Deighton and Hill, 1998)

3. METHODS

Cuttings of ~1.5 kg in weight were obtained at 5 different depths from Habanero #1 wellbore (Table 1). The uppermost sample (HB3650: the number corresponds with sampling depth in meter) is from near the bottom of sedimentary rock and the others are from granite. The lowermost sample is from near the bottom hole.

Table 1. List of Habanero #1 Samples for Zircon FT Dating Method.

Sample code	Sampling depth (ft)	Temp. (°C)	Description
HB3650	11950-11980	221	Bottom of sedimentary rock
HB3675	12050-12080	222	Top of granite (weathered part?)
HB4000	13120-13150	233	Granite
HB4255	13940-13970	242	Granite. Near a major fracture.
HB4400	14430-14460	248	Granite. Bottom of wellbore.

Zircons were obtained by standard heavy mineral separation techniques. Cuttings were ground by disc mill and grains of 74 – 250 μm were collected after magnetic and heavy liquid separations. A large quantity of heavy minerals (1 – 2 g) were obtained after CH_2I_2 (density: 3.3) separation. They were identified by XRD as barite (density: 4.5), which was used for drilling operation. After dissolving barite by NaOH/KOH eutectic liquid, ~200 zircon grains were obtained for each sample.

FT dating was performed by the external detector method using polished zircon surface (Gleadow, 1981). Ages were obtained by adopting the zeta approach (Hurford, 1990). Zeta values were determined using 3 age standards and 3 different irradiation runs as shown in Table 2. Zeta value of 126.7 ± 5.0 (2σ) was obtained using CN-1 standard glass and DAP detector (Yoshioka et al., 2003). FTs in zircon suitable for dating were revealed after etching in NaOH/KOH eutectic liquid for 9 -10 hours at 225 °C (Figure 4). Thermal neutron irradiation was performed using JRR-3 reactor at Japan Atomic Energy Agency. All of the 5 samples were dated by FT method and horizontally-confined spontaneous track lengths were also measured in order to constrain thermal history of the dated samples.

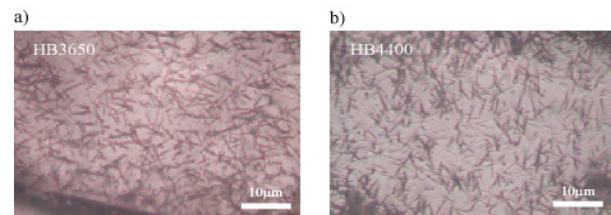


Figure 4: Spontaneous tracks in zircon from Habanero #1. a) Zircon from sedimentary rock at a depth of 3650 m (Temp. ~220°C). b) Zircon from granite at a depth of 4400 m (Temp. ~250°C)

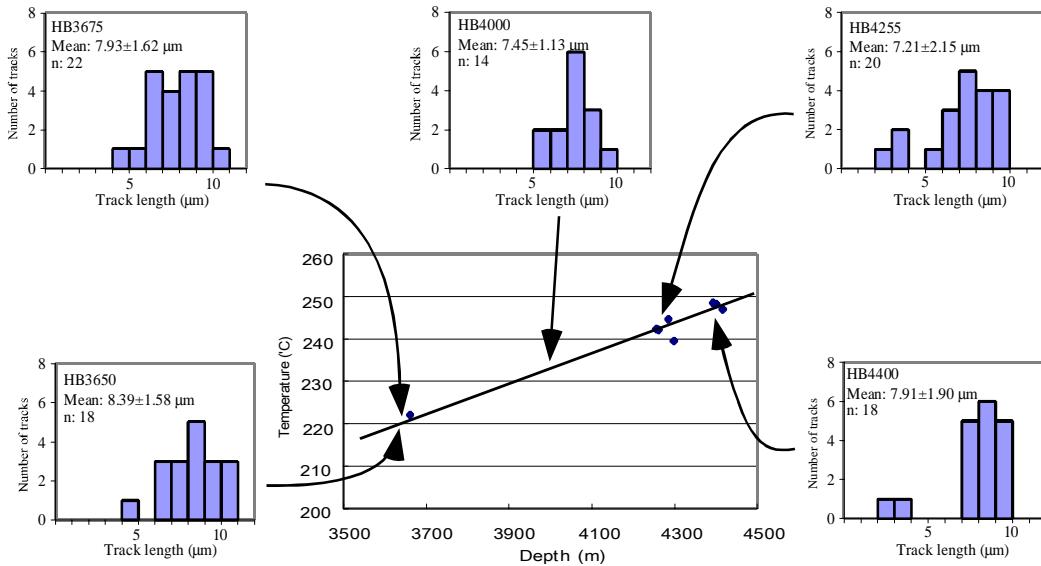
LA-ICP-MS U-Pb dating was performed using New Wave Research UP-213 laser coupled to a Thermo Fisher Scientific X series II ICP-MS that was installed at Central Research Institute of Electric Power Industry (CRIEPI). Ablation spot size was 40 μm in diameter and the laser energy was set to 40%. Ablation was done three times successively on the same spot and these three data were treated independently. Laser induced elemental fractionation and instrumental mass discrimination were corrected by normalization to reference zircons (Tardree Rhyolite), which were analyzed under exactly the same conditions as the age-unknown zircons.

Table 2. Zeta Values of Zircon Age Standards Determined Using JRR-3 at Japan Atomic Energy Agency.

Age standard	Sample code	Irradiated date	Number of grains	Dosimeter number	Density $\times 10^5 \text{ cm}^{-2}$	Spontaneous number	Density $\times 10^6 \text{ cm}^{-2}$	Induced number	Density $\times 10^6 \text{ cm}^{-2}$	Observer: H. Ito	
										$P(\chi^2)$ %	$\zeta \pm 1\sigma$ Ma
Fish Canyon Tuff	FCT04-1	2004/11/25	20	1455	3.737	2476	5.781	2316	5.407	77	140.0 \pm 6.0
Fish Canyon Tuff	FCT05-1	2006/7/7	9	2577	3.922	1100	5.78	865	4.545	95	112.1 \pm 5.9
Fish Canyon Tuff	FCTZ-2	2009/2/20	15	1503	4.117	2359	5.359	2243	5.096	0.5	129.2 \pm 5.6
Fish Canyon Tuff	FCT08-2	2009/2/20	15	1503	4.117	1825	5.448	1591	4.749	0.5	118.4 \pm 5.5
Bukuk Member 4 Tuff	BM05-1	2006/7/7	12	2577	3.922	610	1.013	891	1.48	6	122.3 \pm 7.0
Tardree Rhyolite	TR05-1	2006/7/7	11	2577	3.922	1303	5.625	651	2.81	6	150.2 \pm 8.3
Weighted mean											126.7 \pm 2.5

Table 3. Zircon FT Dating Results of Habanero #1.

Sample code	Number of grains	Dosimeter number	Density $\times 10^5 \text{ cm}^{-2}$	Spontaneous number	Density $\times 10^7 \text{ cm}^{-2}$	Induced number	Density $\times 10^6 \text{ cm}^{-2}$	$P(\chi^2)$ %	Observer: H. Ito	
									U-content ppm	$T \pm 2\sigma$ Ma
HB3650	9	1455	3.737	1453	1.99	145	1.99	27	173	233 \pm 44
HB3675	9	1455	3.737	1278	1.84	115	1.66	2	184	276 \pm 60
HB4000	7	1455	3.737	749	2.80	118	4.41	20	455	149 \pm 31
HB4255	8	1455	3.737	892	2.82	115	3.64	91	408	181 \pm 38
HB4400	9	1455	3.737	872	2.70	143	4.43	53	461	143 \pm 27

**Figure 5: Spontaneous track length distributions in zircon from Habanero #1**

One sample (HB4255) was dated by U-Pb method. The sample was the same one that was used for FT dating, that is, etched and irradiated zircons for FT were re-used for U-Pb dating.

4. RESULTS AND DISCUSSION

4.1 FT Dating

Results of track length measurements and FT dating are shown in Figure 5 and Table 3, respectively.

Mean track length ranges 7.2 – 8.4 μm , and no correlation between track length and depth (or temperature) exists (Figure 5). There is a peak at 7 – 9 μm in track length distribution. Because un-annealed tracks in zircon have their mean length and peak in distribution at 10 – 11 μm (e.g., Ito et al., 1989; Hasebe et al., 1994), tracks in zircon

from Habanero #1 should have experienced thermal annealing.

Zircon FT ages of 143 – 276 Ma were obtained (Table 3). The age of HB3675 failed the χ^2 test at 5% level, meaning the dispersion of individual grain ages is larger than is statistically expected. However, the other four samples passed the χ^2 test, indicating that, as a whole, grain ages cluster within the ranges statistically allowed. A weak tendency exists that the deeper samples have younger ages in that ages from 4000 – 4400 m are 143 – 181 Ma and those from ~3660 m are 233 – 276 Ma. This is not the case for the samples HB4000 and HB4255.

In general, a correlation for FT age and mean track length is expected for partially annealed granite, that is, the younger samples should have shorter tracks, whereas no such

correlation exists in that the sample with shortest track length (HB4255) does not have the youngest age.

From these discussions it seems the FT data cannot bear rigorous analyses by comparing individual sample results. Therefore all the FT data are grouped together and only general features are discussed hereafter. Before that, it should be mentioned that zircons in HB3650 taken from the vicinity of the bottom of sedimentary rocks are also from the same granite origin because of the similarity of crystal morphology and FT data with the other zircons from granite origin. The overall weighted mean track length and FT ages are $7.8 \pm 1.4 \mu\text{m}$, $173 \pm 16 \text{ Ma}$ (2σ error). The age is younger than the emplacement age of the granite ($300 - 320 \text{ Ma}$ by U-Pb method; Gatehouse et al., 1995). Taking into account that the granite reached near the ground by $\sim 280 \text{ Ma}$ by rapid uplifting (Figure 3), the original FT age should be $\sim 300 \text{ Ma}$. This means thermal annealing reduced FT age by 42%. Likewise, it reduced track length by 26% assuming the original track length of $10.6 \mu\text{m}$ (Hasebe et al., 1994). This reduction ratio is comparable with that obtained by laboratory thermal annealing experiment (Tagami et al., 1990), which means the FT age and track length data are reliable.

The track length data is plotted on an Arrhenius diagram (Figure 6) that shows track length reduction in terms of annealing temperature and duration. The data was plotted in the range including 280°C for 10^8 years annealing and 320°C for 10^6 years annealing. Taking into account that thermally un-annealed tracks ($> 10 \mu\text{m}$) are rare, the granite may have experienced higher temperature than the present-day temperature ($220 - 250^\circ\text{C}$) during a later stage of its subsidence history.

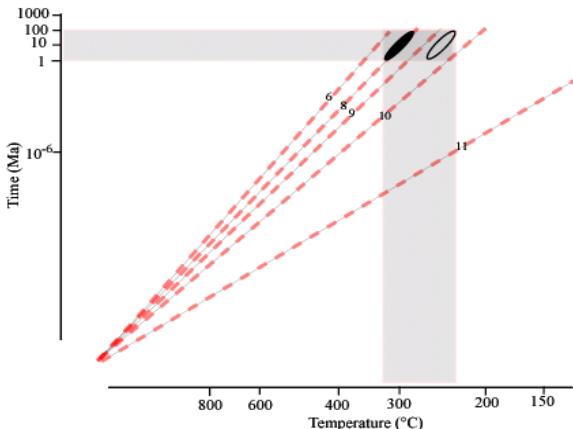


Figure 6: Thermal annealing property of FT length in zircon shown in the Arrhenius diagram (modified from Tagami et al., 1998). Weighted mean track-length of $7.8 \mu\text{m}$ from Habanero #1 zircons and the present temperature ($220 - 250^\circ\text{C}$) range are shown in filled and open circles, respectively

4.2 U-Pb Dating

Ten zircons were dated by LA-ICP-MS U-Pb methods both for Tardree Rhyolite and Big Lake Suite granite (sample name: HB4255). In order to check and delete data that were affected by radiogenic Pb loss and common Pb gain (Jackson et al., 2004), Tera-Wasserburg plot (Tera and Wasserburg, 1972) was employed using Isoplot v. 2.49 (Ludwig, 2001). After this treatment, $48.4 \pm 3.6 \text{ Ma}$ was obtained for Tardree Rhyolite (Figure 7) and $260 \pm 16 \text{ Ma}$ was obtained for Big Lake Suite granite (Figure 8). These

are ages affected by laser induced elemental fractionation and instrumental mass discrimination. Recently, an age of $61.23 \pm 0.11 \text{ Ma}$ (2σ) was obtained by TIMS U-Pb method and is recommended as the crystallization age of Tardree Rhyolite (Chew et al., 2008). Using this age as the reference age, the corrected U-Pb age of the Big Lake Suite granite is calculated as $329 \pm 32 \text{ Ma}$ (2σ). The obtained U-Pb age is in agreement with the previously reported SHRIMP U-Pb age of $300 - 320 \text{ Ma}$ (Gatehouse et al., 1995).

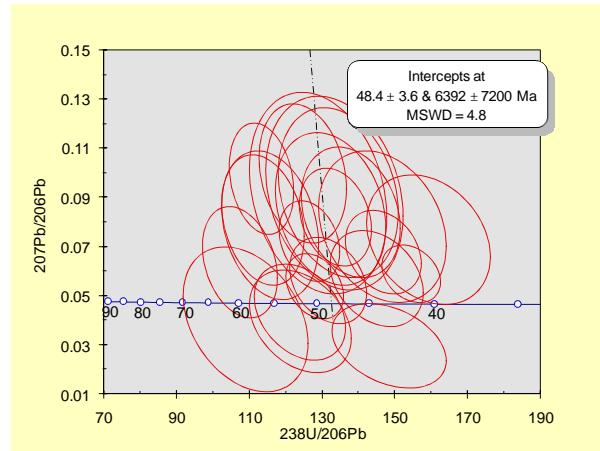


Figure 7: Tera-Wasserburg plot for Tardree Rhyolite. Seven data out of 30 were deleted because they are extremely away from clusters that lead to an age of $48.4 \pm 3.6 \text{ Ma}$

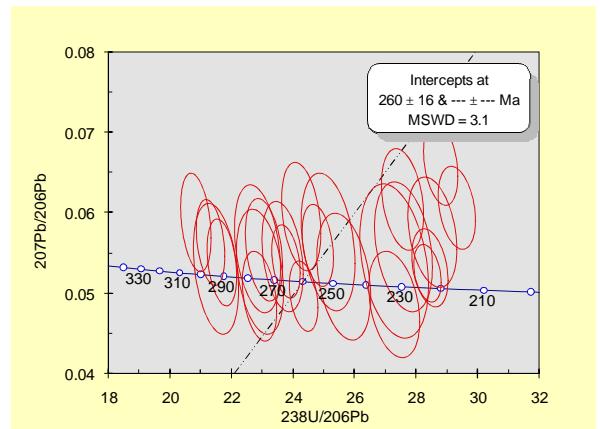


Figure 8: Tera-Wasserburg plot for Big Lake Suite granite (sample name: HB4255). Six data out of 30 were deleted because they are extremely far from the clusters that lead to an age of $260 \pm 16 \text{ Ma}$.

CONCLUSIONS

A thermally reduced zircon FT age of $173 \pm 16 \text{ Ma}$ (2σ) was obtained from a granite at depths of $\sim 3650 - 4400 \text{ m}$ from Habanero #1 well. From track length and age analyses, the granite may have experienced higher temperature (280°C for 10^8 years duration - 320°C for 10^6 years duration) than the present-day temperature ($220 - 250^\circ\text{C}$) during a later stage of its subsidence history. A reliable LA-ICP-MS U-Pb age of $\sim 330 \text{ Ma}$ was obtained using the same zircons that were used for FT dating. Therefore it was revealed that both

FT and U-Pb dating are applicable using the same zircon grains.

ACKNOWLEDGEMENTS

Dr. Doone Wyborn kindly gave me the Habanero #1 cuttings samples. Dr. Hideshi Kaieda and Dr. Fiona Holgate helped the sampling. Mr. Tomonori Taniguchi helped FT dating and Mr. Shigeju Ichimura arranged for the irradiation. Dr. Taiji Chida and Mr. Katsuaki Kurahashi helped LA-ICP-MS measurements. IsoPlot/Ex 2.49 by Dr. Kenneth Ludwig was used for Tera-Wasserburg plot.

REFERENCES

Chew, D., Ganerød, M., Troll, V., Corfu, F., and Meade, F.: U-Pb TIMS zircon age constraints on the Tardree Rhyolite zircon fission track standard, *On Track*, **16**, (2008).

Deighton, I., and Hill, A.J.: Thermal and burial history. in Petroleum Geology of South Australia Volume 4 - Cooper Basin-, Chapter 9., 143-155, edited by D.I.Gravestock, J.Hibbert, and J.F.Drexel, PIRSA, (1998).

Gatehouse, C.G., Fanning, C.M., and Flint, R.B.: Geochronology of the Big Lake Granite Suite, Warburton Basin, northeastern South Australia, South Australia, *Geological Survey. Quarterly Geological Notes*, **128**, (1995), 8-16.

Gleadow, A.J.W.: Fission-track dating methods: What are the real alternatives?, *Nucl. Tracks*, **5**, (1981), 3-14.

Gravestock, D.I., and Jensen-Schmidt, B.: Structural setting. in Petroleum Geology of South Australia Volume 4 -Cooper Basin-, Chapter 5., 47-67, edited by D.I.Gravestock, J.Hibbert, and J.F.Drexel, PIRSA, (1998).

Hasebe, N., Tagami, T., and Nishimura, S.: Towards zircon fission-track thermochronology: Reference framework for confined track length measurements, *Chem. Geol. (Isot. Geosci. Sect.)*, **112**, (1994), 169-178.

Hurford, A.J.: Standardization of fission track dating calibration: Recommendation by the Fission Track Working Group of the I.U.G.S. Subcommission on Geochronology, *Chem. Geol. (Isot. Geosci. Sect.)*, **80**, (1990), 171-178.

Ito, H., Sorkhabi, R.B., Tagami, T., and Susumu, N.: Tectonic history of granitic bodies in the South Fossa Magna region, central Japan: new evidence from fission-track analysis of zircon, *Tectonophysics*, **166**, (1989), 331-344.

Jackson, S.E., Pearson, N.J., Griffin, W.L., and Belousova, E.A.: The application of laser ablation-inductively coupled plasma-mass spectrometry to in situ U-Pb zircon geochronology, *Chem. Geol.*, **211**, (2004), 47-69.

Ludwig, R.K.: Users manual for Isoplot/Ex rev.2.49, Berkeley Geochronology Center Special Publication, No. 1a, (2001), 55p.

Tagami, T., Ito, H., and Nisimura, S.: Thermal annealing characteristics of spontaneous fission tracks in zircon, *Chem. Geol. (Isop. Geosci. Sect)*, **80**, (1990), 159-169.

Tagami, T., Galbraith, R.F., Yamada, R., and Laslett, G.M.: Revised annealing kinetics of fission tracks in zircon and geological implications, P. Van den haute and F. De Corte (eds.), *Advances in Fission-Track Geochronology*, Kluwer Academic Publishers, Dordrecht, (1998), 99-112.

Tera, F., and Wasserburg, G.J.: U-Th-Pb systematics in three Apollo 14 basalts and the problem of initial Pb in lunar rocks, *Earth Planet. Sci. Lett.*, **14**, (1972), 281-304.

Yoshioka, T., Tsuruta, T., Iwano, H., Danhara, T. and Koguchi, Y.: Fission-fragment registration and etching properties of dially phthalate with reference to its use as an external detector in fission-track dating, *Nucl. Instr. Meth. Phys. Res.*, **B207**, (2003), 323-332.