Zeta calibration values for fission track dating with a diallyl phthalate detector

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Key words: diallyl phthalate (DAP), fission-track dating, zeta value, zircon, apatite

Introduction

The fission-track dating adopts the zeta calibration approach according to the recommendation by the I.U.G.S. Subcommission on Geochronology (Hurford, 1990). The calibration factor zeta is subject to a whole measurement system, including, e.g., a nuclear reactor for sample irradiation, a microscope to observe fission tracks, and an external detector to record induced tracks.

Diallyl phthalate (DAP) resin plates are recently developed as a fission-track detector (Tsuruta, 1999, 2000). Compared to widely-used mica sheet, they have following advantages: less radioactivity after neutron irradiation, higher detection efficiencies, shorter etch time, and suitability for automatic counting. Fission-track registration and etching properties of DAP for the use as an external detectors have been already investigated (Yoshioka et al., 2002, 2003). In this short note, we report zeta calibration values of zircon and apatite for the measurement system using the plastic external detector.

Experimental procedures

Three zircons and two apatite samples were analyzed with the conventional external detector method (Gleadow, 1981) for fission track dating. For zircon, both internal (4π geometry) and external (2π geometry) surfaces were analyzed for comparison. For apatite, internal surfaces were only used. All samples are the age-standard materials recommended by the I.U.G.S. Subcommission on Geochronology (Hurford, 1990) : zircons from the Buluk Member Tuff with a reference age of 16.3 ± 0.2 Ma (Hurford and Watkins, 1987), from the Fish Canyon Tuff with a reference age of 27.8 ± 0.2 Ma (Hurford and Hammerschmidt, 1985), and from the Tardree Rhyolite with a reference age of 58.7 ± 1.1 Ma (Hurford and Green, 1983), and apatites from the Fish Canyon Tuff, and from the Durango Apatite with a reference age of 31.4 ± 0.5 Ma (Green, 1985).

Zircon grains were mounted in a Teflon PFA sheet (Danhara et al., 1993) with crystal surfaces exposed and two mounts were prepared for each sample. One of the mounts for internal surfaces (ED1) were polished with 15 μm, 3 μm and 1 μm diamond pastes stepwise. Another mount was not polished to use external surfaces (ED2). Each mount was etched in an eutectic mixture of NaOH and KOH at 225 °C for 18-40 h. Apatite samples were mounted in epoxy resin (Struers SpeciFix) with prismatic faces exposed. The mounts were polished with diamond pastes and etched in 0.7% HNO3 at 20 °C for 4-6 min.

Before irradiation, counting of spontaneous tracks in samples was carried out. Track counting was done using two types of observation systems: the direct observation system using a Nikon Biophot microscope with oculars and a 100 × dry objective (Danhara et al., 1991; Danhara and Iwano, 2001) and the monitor observation system newly set-up using a Nikon Eclipse E1000 microscope with a 100 × dry objective, a Tokyo Electronic Industry HVC7110 TV camera, and a Chuomusen THM-14A video monitor. Zircon and apatite grains with well-etched and isotropically distributed tracks were carefully selected for counting.

A DAP detector was then attached to each sample mount and NIST-SRM612, which was used as a dosimeter standard glass, and irradiated with a thermal neutron fluence of ~ 5 × 10¹⁴ n·cm⁻² and ~ 2.5 × 10¹⁵ n·cm⁻² for zircon and apatite, respectively. Seven irradiation runs were made in the pneumatic tube of JRR-4 reactor (thermal neutron flux: 3.2 × 10¹⁸ n·cm⁻²·sec⁻¹) unit of the Japan Atomic Energy Research Institute (JAERI). The cadmium ratio for Au at this position is 3.6.

After irradiation, the DAP detectors were detached and induced tracks were etched in a solution of 15% KOH, 65% ethanol and 20% H2O at 60 °C for 2 min (Tsuruta, 2001; Yoshioka et al., 2002, 2003). Induced-track counting for
### Table 1. Fission track data of zeta calibration for internal and external surfaces of zircon using the direct observation system (A) and the monitor observation system (B).

<table>
<thead>
<tr>
<th>Sample Mount</th>
<th>n</th>
<th>ρs (Nf, N0)</th>
<th>ρi (Nf, N0)</th>
<th>Doimeter F ((x^2))</th>
<th>(\zeta_{41})</th>
<th>(\phi_{41})</th>
<th>Irradiation No.</th>
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<tr>
<td>Sample Mount</td>
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<td>ρi (Nf, N0)</td>
<td>Doimeter F ((x^2))</td>
<td>(\zeta_{41})</td>
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#### A. Data for direct observation system

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<th>(\phi_{41})</th>
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#### B. Data for monitor observation system

<table>
<thead>
<tr>
<th>Sample Mount</th>
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<th>ρs (Nf, N0)</th>
<th>ρi (Nf, N0)</th>
<th>Doimeter F ((x^2))</th>
<th>(\zeta_{41})</th>
<th>(\phi_{41})</th>
<th>Irradiation No.</th>
</tr>
</thead>
<tbody>
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<td>Sample Mount</td>
<td>n</td>
<td>ρs (Nf, N0)</td>
<td>ρi (Nf, N0)</td>
<td>Doimeter F ((x^2))</td>
<td>(\zeta_{41})</td>
<td>(\phi_{41})</td>
<td>Irradiation No.</td>
</tr>
</tbody>
</table>

- ρ and N, density and total number of counted tracks; Analyses were made by the external detector method (ED, internal surface; ED2, external surface; GITG, 1981); Zeta (ζ) values were determined against a dosimeter glass NST-15486 (\(P^2(x^2)\)), probability of obtaining the \(x^2\) value for degrees of freedom (ν = number of crystals - 1) (Galbraith, 1981); Thermal neutrons were irradiated in the pneumatic tube of JRR-4 reactor unit of Japan Atomic Energy Research Institute. Irradiation time was 18 sec at an output of 3.5 MW. Age standards were measured using Fish Canyon Tuff (FC3, BTKN), Tulal Member Tuff (BMT9), and Tardree Rhyolite (TDR9). Track counting was done by TD using the direct observation system and by HI using the monitor observation system.

- The analytical data of zeta determinations for zircon standard samples are summarized in Table 1. For the direct observation system, the weighted mean zeta values measured by an observer TD are 390 ± 3 and 350 ± 3 for ED1 and ED2 data, respectively. For the monitor observation system, the weighted mean zeta values measured by another observer HI are 380 ± 3 and 347 ± 3 for internal and external surface data, respectively. There is no significant
Table 2. Fission track data of zeta calibration for internal surfaces of apatite using the monitor ionization system.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Mount</th>
<th>n Spontaneous</th>
<th>Induced</th>
<th>Dosimeter $P(x)^2$ (%)</th>
<th>$\varepsilon_{121}\pm1.0$</th>
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<td>49.68</td>
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<td>1906</td>
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<td>2646</td>
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<td>2560</td>
<td>47.29</td>
<td>3027</td>
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$\rho$ and $N$, density and total number of counted tracks, respectively; Analysts were made by the external detector method (ED1, internal surface) (Gleadow, 1981); Zeta ($\varepsilon$) values were determined against a dosimeter glass NIST-SRM612. ($P(x)^2$), probability of obtaining the $x^2$ value for $v$ degrees of freedom ($v$ = number of crystals - 1) (Galbrith, 1981); Thermal neutrons were irradiated in the pneumatic tube of JRR-4 reactor unit of Japan Atomic Energy Research Institute. Irradiation time was 90 sec at an output of 3.5 MW. Age standard samples used were Fish Canyon Tuff (FC3), and Durango Apatite (DA3). Track counting was done by HI using the monitor observation system.

Table 3. Summary of published zeta values and their experimental conditions.

<table>
<thead>
<tr>
<th>Citation</th>
<th>Danbara et al. (1991)</th>
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<th>This Study</th>
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Abbreviation: ED1 and ED2, external detector method applying to internal and external surfaces of a mineral, respectively; T.E.L., Tokyo Electric Industry; TD, Tohoku Database; HI, Hioki Iwaste; BMT, Bulok Member Tauff, FC3, Fish Canyon Tauff, TR, Tauff Rhyolite; MDC, Mount Domeyama Complex; BBT, Bulok Tauff; DA, Durango Apatite; MIT, Matsukai Institute of Technology; SPU, St. Paul's University; JAEI, Japan Atomic Energy Research Institute; RSR, Rockkep Apatite; FEW-65, a solution of 15% KOH, 65% ethanol and 20% water.
difference in weighted means for each method between the two systems. Failure of $z$-test (Galbraith, 1981) found mainly in ED1 data is explained by a non-Poisson variation of the vertical inhomogeneity below and above the observed internal surfaces of zircon grains (Danbara et al., 1991). The lower values for ED2 are due to the higher track counting efficiency on $2\pi$ geometry surfaces (Iwano and Danbara, 1997).

The analytical data of zeta determinations for apatite standard samples are summarized in Table 2. The weighted mean zeta value for the monitor observation system is $335 \pm 5$ that is $12\%$ lower than that for zircon using the same system and method. The difference is due to different track registration efficiencies among minerals, as found in previous results using the external detector method (Iwano and Danbara, 1998).

All these values differ a little from previous zeta values determined for NIST-SRM612 using mica sheet as an external detector (Danbara et al., 1991; Iwano and Danbara, 1997, 1998: see Table 3). The difference is probably due to change of microscope, thermalization in reactor, and unknown factors by adopting DAP detectors. Zeta values and their experimental conditions that we published before and in this study are summarized in Table 3 for users of fission track data.

Acknowledged: The authors wish to express their appreciation to Mr. K. Kobayashi of the Yamamoto Kogaku Co., Ltd. for his assistance on DAP plate preparation, and to Mr. S. Ichimura of the JAERI for his help with the thermal neutron irradiation. The authors thank Dr. A. J. Hurford for the supply of age standards (Buluk Member Tuff and Tardree Rhyolite zircons), two reviewers (Dr. N. Hasebe and an anonymous person) for helpful comments, and Drs. Y. Ganzawa and T. Nakajima for their encouragement.

References


* in Japanese with English abstract