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Fission track dating of zircon crystal

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Abstract: The track etching and fission track (f.t) age studies are carried out on various natural planes of zircon crystal collected from Khammam District of Andhra Pradesh. The etching studies are carried out using two different sets of etchants viz. (a) 1 : 1 mixture of H_2SO_4 : HF and (b) 4 : 15 : 6 mixture of NaOH : KOH : LiOH $.H_2O$. It is observed that the track etching anisotropy is more in the basic medium (b) than that in acidic medium (a). This leads to the different values of track etching efficiency on various planes of zircon. Because of this anisotropic etching behaviour of the crystal, the fossil track density is found to vary, from plane to plane. However, the concordant f.t. ages are obtained on various planes of zircon, by applying the appropriate corrections for anisotropic etching.

Keywords: Fission track dating, track etching, zircon crystal, fossil track density, anisotropic etching.

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1. Introduction

Chemical etching makes the fission tracks visible because the rate of etching along the damaged region of the track (V_T) is greater than the general etching rate of the surface (V_G). The relationship between V_T and V_G controls the etching efficiency, η (Fleischer *et al* 1975). In minerals, where V_T is much higher than V_G, tracks have the form of cylindrical holes, and a high etching efficiency ($\sim$ 100%) is implied. In other materials, such as glasses, etched tracks have the appearance of broad cones because V_T is only a little greater V_G (Gleadow 1978). In these materials, tracks at shallow angles to the surface are not revealed by etching when the vertical component of V_T is less than or equal to V_G. The classic description of V_T, V_G and η has proved extremely useful in understanding track etching in isotropic materials, such as glasses and plastics, but the etching behaviour of natural minerals is more complicated (Gleadow 1981, Sandhu *et al* 1987). Most of the minerals occur in the form of crystals with variable atomic spacing along different crystallographic orientations. Hence their etching and annealing characteristics for radiation damage vary with the crystal orientation (Sandhu *et al* 1987, 1988).

In the present investigations, the track etching and fission track age studies are carried out on various natural planes of zircon crystal collected from Khammam district of Andhra Pradesh. Fission track dating has proved to be quite suitable for zircon due to its relatively high uranium content and low annealing rate of radiation damage compared to other minerals. Three planes of zircon crystal viz. (100), (101) and (001), are taken for study.

2. Experimental procedure

The polished samples from different planes of zircon were irradiated with ^{252}Cf fission fragment source in 2π geometry. Prior to irradiation the samples were heated at 700°C for 6 hrs to erase all fossil tracks. The etching of the samples is performed using two different etchants namely :

(i) NaOH : KOH : $LiOH.H_2O$ (Zaun and Wagner 1985)

(ii) HF : H_2SO_4 (Krishnaswami *et al* 1974)

A new track etchant for zircon is reported earlier by Zaun and Wagner (1985). This new etchant (NaOH : KOH : $LiOH.H_2O = 6 : 14 : 1$) reduces the etching time for track revelation compared with the eutectic melt etchant, proposed by Gleadow *et al* (1976). After considerable experimentation, a slight modification was done in this new etchant for getting good results. For this 4 gms of NaOH, 15 gms of KOH and 6 gms $LiOH.H_2O$ were mixed with 2 ml H_2O in a teflon lined steel capsule. The irradiated samples from each plane of zircon crystal were immersed in this improved etchant and the capsule was closed air tight and placed in the muffle furnace pre-heated to 200°C. The samples were step etched and removed periodically from the capsule for track length and diameter measurements. The mean track length (corrected for dip) and diameter were measured at each etching event using an optical microscope at a magnification of 1250×(+oil immersion). In order to study the etching behaviour of fission tracks in zircon using the acidic mixture (Krishnaswami *et al* 1974), 5 ml of 48% HF was mixed with 5 ml of 98% H_2SO_4 in the teflon capsule. Etching of the samples was carried out under pressure at 175°C. The track length and diameter were measured at regular intervals. All length measurements were made only of horizontal tracks, characterized by low dip angle. These tracks were sharply focussed along their entire length and have a very bright reflection in the incident light.

It was observed during the experimental work that the tracks were not revealed on the basal plane (001) of zircon crystal using NaOH : KOH : $LiOH.H_2O$ mixture. This may be due to very high bulk etch rate normal to 001 plane, which was characterized by the quick removal of polished layer containing externally implanted fission tracks. However, using acidic etchant, it was possible to observe the etched tracks on basal plane (001).

Fission track age on different planes of zircon crystal was determined by using the external detector method (Hurford and Green 1982). The measurements were repeated with the two different sets of etchants namely : with a 1 : 1 mixture of HF and H_2SO_4 and with the basic mixture viz. NaOH : KOH : $LiOH.H_2O$. After counting the fossil tracks, the samples from different planes of zircon were irradiated with thermal neutrons ($\phi = 4.5 \times 10^{14}$ $n.cm^{-2}$) together with external muscovite detectors (which were subsequently etched for 50 min in 48% HF to reveal the induced tracks).

3. Results and discussion

The track etch rate, V_T, is calculated from the slope of the linear part of the curve, etching time versus track length. The bulk etch rate, V_G, is calculated from the one half of the slope of the curve, etching time versus track diameter. The etching efficiency (η) and critical angle (θ_c) are calculated from the following relations (Fleischer et al 1975) :

$$\eta = 1 - \sin \theta_c \tag{1}$$

and

$$\theta_c = \sin^{-1} \frac{V_G}{V_T} \tag{2}$$

The plane 100 show low critical angle and higher etching efficiency (97.6%) compared to the other planes, using basic medium as the track etchant (Table 1).

Table I. The values of etching efficiency (η) and critical angle (θ_c) for different planes of zircon.

Plane	NaOH : KOH : $LiOH.H_2O$		$HF : H_2SO_4$	
	θ_c	$\eta(\%)$	θ_c	$\eta(\%)$
100	1.38°	97.6	4.5°	92.1
101	22.9°	61.1	5.1°	91.1
001	—	—	5.5°	90.4

Also, the etching efficiency is found to be higher for 100 plane using HF : H_2SO_4 as the track etchant. On comparing the results reported in Table 1, it appears that HF : H_2SO_4 shows more isotropic etching behaviour. However, for the planes parallel to c-axis (100 plane), the NaOH : KOH : LiOH mixture seems to be the best etchant, as this etchant yields high etching efficiency (97.6%), as compared to that (92.1%) obtained by HF : H_2SO_4 etchant.

The fission track age of different planes of zircon crystal is determined using the following relation (Price and Walker 1963) ;

$$T=6.45\times 10^{9}\ \ln\left(1+9.30\times 10^{-18}\times g\times \frac{\rho_s}{\rho_i}\times \phi\right) \qquad (3)$$

where, ρ_s and ρ_i are the fossil and induced track density ; and g is the geometry factor.

It is desirable in external detector method (EDM) to measure the fossil track density on surfaces with identical etching efficiency as that of muscovite detector. It is observed (Table 1) that only the prismatic plane of zircon possess a high etching efficiency (97.6%) closely resembling to that of the muscovite detector, which approaches 100% (Fleischer *et al* 1975). Moreover, in order to get more reliable f.t. ages on different planes of zircon, geometry factor (g) for each plane is determined using both types of etchants. The value of g is obtained (Table 2) by comparing the density of induced fission tracks on an internal surface (ρ_{in}, 4π geometry) of each plane with that induced simultaneously on an adjacent muscovite detector ($\rho_{e.d.}$, 2π geometry).

Table 2. Measured values of geometry factor for different planes of zircon.

Crystal plane	$\rho_{e.d.}$ ($\times 10^4$)	HF : H_2SO_4		NaOH : KOH : $LiOH.H_2O$	
		ρ_{in} ($\times 10^4$)	$g=\frac{\rho_{e.d.}}{\rho_{in}}$	ρ_{in} ($\times 10^4$)	$g=\frac{\rho_{c.d.}}{\rho_{in}}$
100	16.38	28.24	0.58	32.12	0.51
101	16.72	27.87	0.60	18.91	0.88
001	16.42	25.26	0.65	—	—

Table 3. Fission track age for different planes of zircon using NaOH : KOH : LiOH as the track etchant.
Thermal neutron fluence (ϕ) = 4.5×10^{14} n cm^{-2}

Sample No.	100 plane (g=0.51)			101 plane (g=0.88)		
	ρ_s ($\times 10^6$)	$\rho_{i\ (c.d.)}$ ($\times 10^4$)	$T\pm 1\sigma$ (m.y.)	ρ_s ($\times 10^6$)	$\rho_{i\ (c.d.)}$ ($\times 10^4$)	$T\pm 1\sigma$ (m.y.)
1	8.41	16.41	669.5 ± 7.8	4.89	16.50	668.2 ± 8.2
2	8.72	16.74	679.9 ± 6.3	4.88	16.60	663.1 ± 7.8
3	8.61	16.45	683.1 ± 8.2	5.13	16.91	683.2 ± 5.9
4	8.62	16.48	682.6 ± 6.8	4.85	16.15	676.7 ± 8.3
			Mean 678.8 ± 3.7			Mean 672.8 ± 3.8

Table 4. Fission track age for different planes of zircon using HF : H_2SO_4 as the track etchant. Thermal neutron fluence (ϕ) $=4.5\times10^{14}$ n cm^{-2}.

Sample No.	100 plane ($g=0.58$)			101 plane ($g=0.60$)			001 plane ($g=0.65$)		
	ρ_s ($\times10^6$)	$\rho_{i\,(e.d.)}$ ($\times10^4$)	$T\pm1\sigma$ (m.y.)	ρ_s ($\times10^6$)	$\rho_{i\,(e.d.)}$ ($\times10^4$)	$T\pm1\sigma$ (m.y.)	ρ_s ($\times10^6$)	$\rho_{i\,(e.d.)}$ ($\times10^4$)	$T\pm1\sigma$ (m.y.)
1.	7.39	16.42	668.7 ± 8 7	7.18	16.48	669.7 ± 10.2	6.74	16.44	681.9 ± 8.9
2.	7.62	16.65	679.4 ± 8.4	7.38	16.75	676.8 ± 8.9	6.62	16.49	670.4 ± 7.6
3.	7.63	16.51	685.8 ± 9.3	7 39	16.62	682.7 ± 7.8	6.73	16.63	673.6 ± 10.1
4.	7.48	16.56	671.1 ± 8.5	7.34	16.53	681.8 ± 8.7	6.69	16.46	676.4 ± 11.2
			Mean			Mean			Mean
			676.3 ± 4.4			677.8 ± 4.5			675.6 ± 4.8

It is observed (Table 2) that only the plane parallel to c-axis (100) of zircon shows the value of g very close to the ideal value (i.e. $g = 2\pi/4\pi = 0.5$). For other planes the value of g deviates considerably from the ideal value using both types of the etchants. Since ρ_{in} and $\rho_{e.d.}$ are measured in different materials, the observed track density ratio ($g = \rho_{e.d.}/\rho_{in}$) is affected due to difference in their etching efficiency.

It is observed that (Tables 3 and 4) the fossil track density varies significantly for different planes of zircon using both types of etchants. This is due to the anisotropic track etching along various crystallographic orientations in zircon. No remarkable variation of induced track density has been observed for various planes of zircon, since the same has been determined on the external muscovite detector. However, f.t. age obtained for each crystal plane is nearly the same (Tables 3, 4), irrespective of the etchant used. Thus the external detector method can successfully be applied for zircon, provided the appropriate geometry factor, has been evaluated. The f.t. age obtained in the present study is higher than that reported by Virk and Koul (1984) for zircon samples belonging to the same area. The lower value of f.t. age reported by Virk and Koul (1984) is due to the reason that the authors have measured the f.t. age by population method and have ignored the possible crystallographic orientation effects.

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