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123

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Anisotropic Track Etching in Apatite

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The etching studies of fission tracks on various planes of apatite crystal are carried out in order to observe its anisotropic behaviour. The bulk etch rate (V_G), the track rate (V_T) and the track etching efficiency (η) are found to vary with crystallographic orientation. The value of η is maximum for a plane parallel to c-axis of the crystal. The fission track age is found to vary with the crystallographic orientation. A plane parallel to c-axis is found to be the best one for age determination.

1 Introduction

The anisotropic track etching and annealing studies in crystalline minerals have drawn considerable attention in recent years[1-3]. In most of the track etch studies, simple detectors like glasses and plastics are generally used which are isotropic. In fission track dating, however, there is no choice but to use the natural detectors available in the form of uranium bearing minerals[3]. Most of these minerals are anisotropic and are expected to have complicated track etching and annealing characteristics. This anisotropic behaviour can affect the fission track age of the mineral. In the present study, an attempt has been made to understand the characteristic differences existing in track etching along various crystallographic orientations in apatite. Apatite crystal, procured from Laval University Museum, Quebec and collected from Wakefield area in Canada has been used for such studies. This work for track etching and age determination is completed on different planes cut from the single crystal.

Apatite is a calcium phosphate mineral which occurs as six-sided columnar crystal (Fig. 1). This is the most widely investigated mineral for fission track age determination because of the ease with which it can be polished and etched for track revelation.

2 Experimental Procedure

The samples from 0001, $1\bar{1}01$ and $10\bar{1}0$ planes of apatite were separated and prepared after grinding and polishing as described elsewhere[4]. One sample from each plane was irradiated with ^{252}Cf fission fragments in 2π geometry for 10 min for standardisation of etching conditions. Samples from each plane were irradiated with a collimated beam of fission fragments in vacuum chamber at $15 \pm 2°$ and 90° angle of incidence for the measurement of track etch rate (V_T) and bulk etch rate (V_G) respectively. These samples were etched in 2% HNO_3 at 25°C for various time intervals ranging from 60 to 500 s. The track density, mean track length corrected for dip and track diameter were measured using an optical microscope at a magnification of 1500 × (+ oil immersion) and the variations of these parameters with etching time are presented in Figs 2-4.

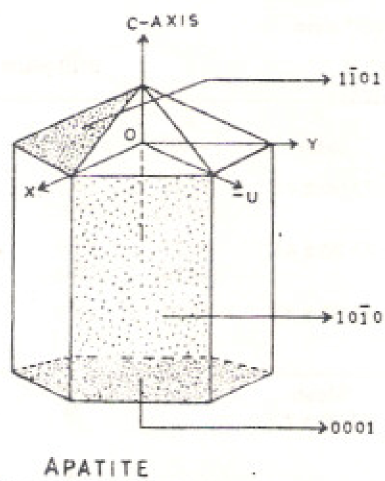

Fig. 1 — Apatite crystal showing various planes

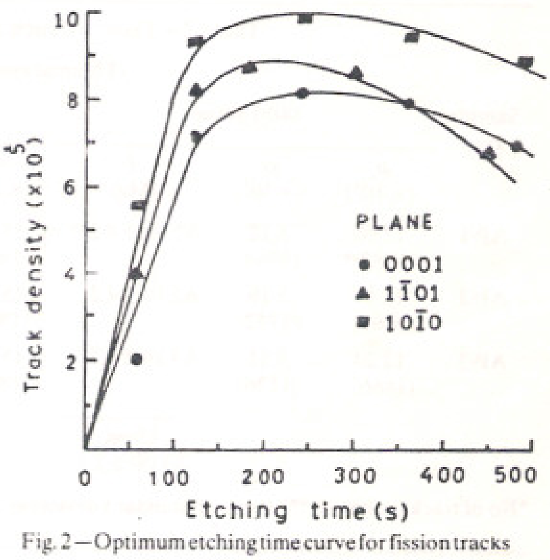

Fig. 2 — Optimum etching time curve for fission tracks

V_T was calculated from the slope of the linear portion of the plot of track length vs etching time[5,6], whereas V_G was calculated from one half of the slope of the curve track diameter vs etching time. The etching efficiency (η) was calculated from the relation:

$$\eta = 1 - \frac{V_G}{V_T} \quad \ldots(1)$$

The results are reported in Table 1.

In the present study, we have used the population method[7] for age determination on different planes of the apatite crystal. Fossil and induced samples were etched in 2% HNO_3 for 3 min at 25°C. The thermal neutron irradiation was carried out from the CIRUS Reactor, at BARC, Trombay. The fission track (f.t) age was calculated using the equation[8,9]:

$$T = 6.57 \times 10^9 \ln\left(1 + 9.25 \times 10^{-18} \times \frac{\rho_s}{\rho_i} \times \phi\right) \quad \ldots(2)$$

where ρ_s and ρ_i are the fossil and induced track densities respectively. The total thermal neutron dose (ϕ) is related with the track density (ρ_d) in the dosimeter by the relation[10]:

$$\phi = k\,\rho_d \quad \ldots(3)$$

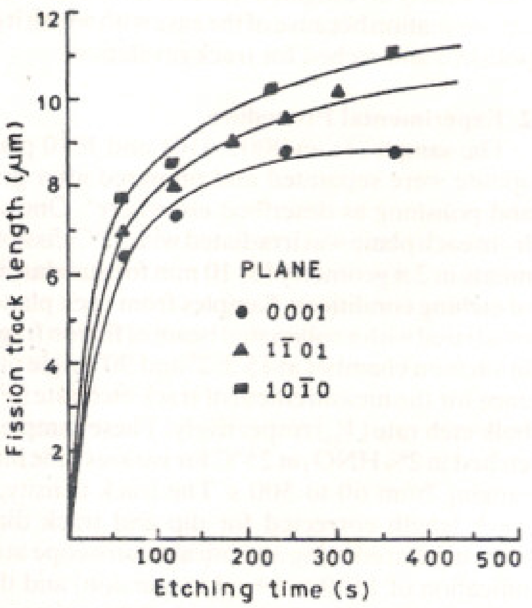

Fig. 3—Variation of track length with etching time for different planes of apatite

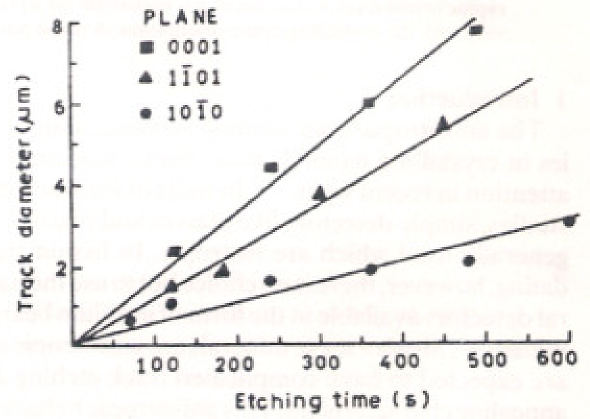

Fig. 4—Variation of track diameter with etching time for different planes

Table 1—Values of V_T, V_G and η for Different Planes of Apatite Crystal

Plane	V_T μm/s	V_G μm/s	η %
0001	0.107	0.0085	92.1
1$\bar{1}$01	0.115	0.0060	94.8
10$\bar{1}$0	0.128	0.0035	97.3

Table 2—Fission Track Age on Different Faces of Apatite Crystal

(Thermal neutron dose $\phi = 4.5 \times 10^{14}$ n cm^{-2})

Sample No.	0001 plane			1$\bar{1}$01 plane			10$\bar{1}$0 plane		
	ρ_s ($\times 10^5$)	ρ_i ($\times 10^5$)	T (Ma)	ρ_s ($\times 10^5$)	ρ_i ($\times 10^5$)	T (Ma)	ρ_s ($\times 10^5$)	ρ_i ($\times 10^5$)	T (Ma)
AP-1	12.00 (1852)*	5.12 (1764)	62.90 ± 4.2**	15.15 (1860)	5.57 (1774)	73.00 ± 4.8	18.16 (1874)	5.83 (1781)	83.50 ± 5.5
AP-2	12.19 (1861)	5.19 (1772)	63.10 ± 4.2	15.26 (1863)	5.59 (1778)	73.20 ± 4.8	18.21 (1878)	5.88 (1788)	83.10 ± 5.5
AP-3	12.24 (1866)	5.21 (1776)	63.10 ± 4.2	15.29 (1870)	5.61 (1782)	73.20 ± 4.8	18.31 (1881)	5.93 (1793)	82.80 ± 5.5
			Mean 63.00 ± 2.4			Mean 73.10 ± 2.7			Mean 83.10 ± 3.8

*No of tracks counted; **Estimated standard deviation (σ) = 1 where k is calibration constant (k for GEC glass[11] is 0.46×10^{10}). The age results are summarized in Table 2.

For the angular distribution studies of etched tracks in apatite crystal, two samples from 10$\bar{1}$0 plane, one containing fossil tracks and other containing fresh induced (^{235}U) tracks were etched simultaneously for 3 min in 2% HNO_3 at 25°C. The angular distribution of fossil (Fig. 5) and induced tracks was obtained by measuring the inclinations of the ends of the tracks relative to a fixed but arbitrary direction[12] in the horizontal plane. These measurements were taken with the help of protractor eyepiece provided with the microscope. The results along with the reference lines are shown in Figs 6 and 7.

Another sample of same plane containing induced tracks (^{235}U) was heated at 350°C for 1 hr and etched at the standard etching conditions. The angular distribution of these tracks is shown in Fig. 8.

For the optical studies, samples from different planes of apatite crystal were ground to slices of about the same thickness (~ 215 μm), and the surface polished carefully. The samples taken for this study contained only fossil tracks. The pairs of slices which had the same thickness were inserted into an ultraviolet (UV-visible-240 Shimadzu) double-beam recording spectrophotometer, one piece (plane perpendicular to c-axis) serving as a comparison blank and the other two pieces (with one plane parallel and the other inclined to c-axis) were placed on the test probe of the spectrophotometer[13]. The absorption difference spectrum was recorded at room temperature as a function of wavelength from 315 to 500 nm (Fig. 9).

3 Results and Discussion

The optimum etch time for revealing maximum

Fig. 5—Microphotograph of fossil fission tracks in 10$\bar{1}$0 plane of apatite

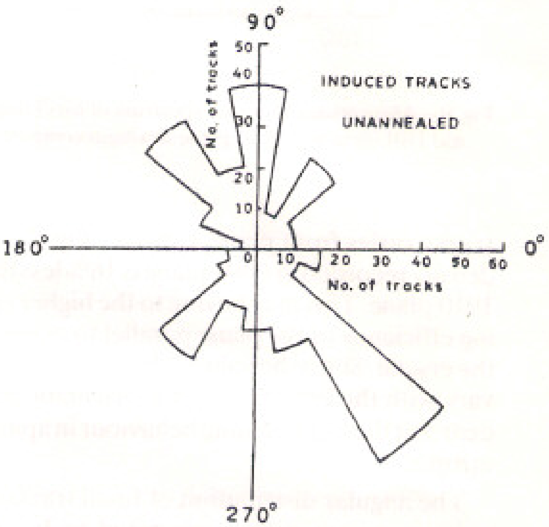

Fig. 7—Angular distribution of induced tracks in 1010 plane

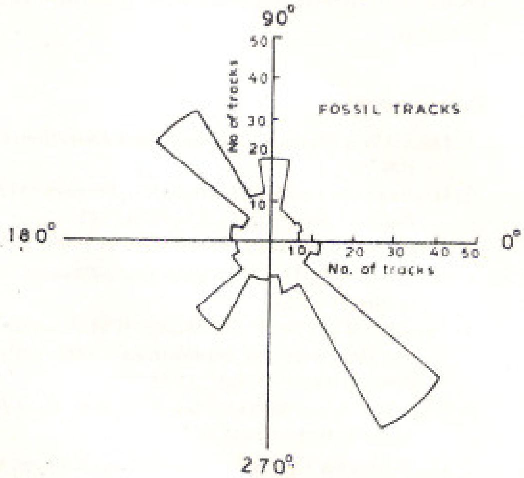

Fig. 6—Angular distribution of fossil tracks in 10$\bar{1}$0 plane

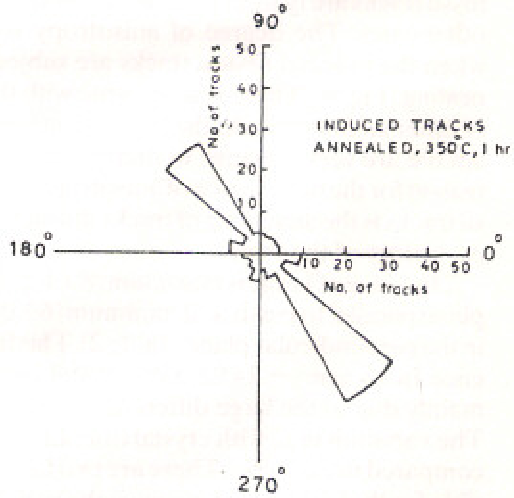

Fig. 8—Angular distribution of induced tracks in 1010 plane annealed at 375°C for 1 hr

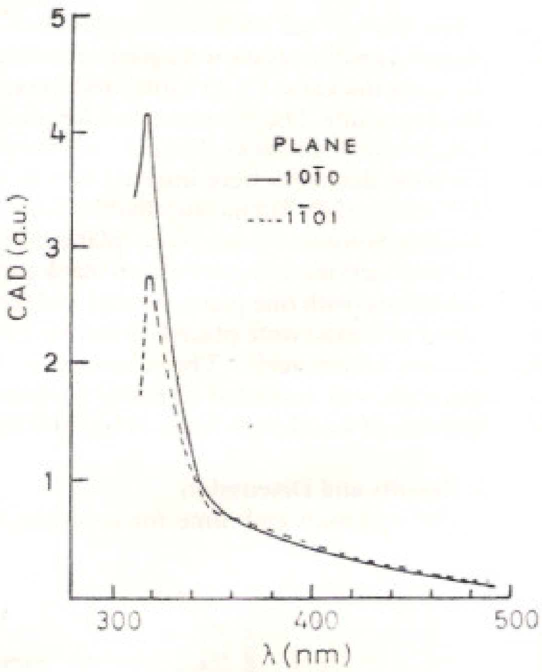

Fig. 9—Absorption difference spectrum of fossil tracks for $10\bar{1}0$ and $1\bar{1}01$ plane with 0001 plane serving as comparison blank

tracks varies from plane to plane (Fig. 2). The track density recorded in 0001 plane is 16% less than that in $10\bar{1}0$ plane. This may be due to the higher track etching efficiency of the plane parallel to c-axis ($10\bar{1}0$) of the crystal. Since the values of V_G, V_T and η (Table 1) vary with the crystallographic orientations, it is evident that the track etching behaviour in apatite is anisotropic.

The angular distribution of fossil tracks (Fig. 6) is quite non-isotropic as compared to fresh induced tracks (Fig. 7). Also it is clear from Fig. 5 that more fossil tracks are lying in a particular direction than the other ones. The degree of anisotropy is increased when the induced fission tracks are subjected to annealing (Fig. 8). These results agree with the findings of other authors[14,15]. As the fission fragment tracks in apatite are very sensitive to thermal effects, the only reason for the high degree of anisotropy in case of fossil tracks is the annealing of tracks during the geological history of the sample.

The value of f.t age is maximum (83.1 ± 3.8 Ma) in a plane parallel to c-axis and minimum (63.0 ± 2.4 Ma) in the perpendicular plane (Table 2). This large difference in f.t age (~ 24%) with crystal orientation is mainly due to the large difference in the value of ρ_s. The variation in ρ_i with crystal orientation is small as compared to that of ρ_s. There are two factors responsible for the variation of ρ_s, one is the variable etching efficiency (Table 1) as discussed earlier and the other is the annealing effect. The latter effect has a greater contribution as the annealing increases the degree of anisotropy.

It is well known now that the radiation damage is the source of characteristic absorption difference, CAD[16]. The peak height of CAD for a sample parallel to c-axis is higher than that for a sample inclined to c-axis (Fig. 9). No CAD was observed when the samples from the same plane were inserted in the test and comparison probes. The absorption difference spectra of $10\bar{1}0$ and $1\bar{1}01$ planes containing fossil tracks show a characteristic sharp absorption peak at about 320 nm. Since the peak height of CAD is proportional to the defect concentration, it is evident (Fig. 9) that the samples from $10\bar{1}0$ plane have higher damage density as compared to $1\bar{1}01$ plane. This fact is further supported by the large fossil track density for $10\bar{1}0$ plane (Table 2).

Conclusions

1. The etching of latent damage trails is quite anisotropic in case of apatite.

2. The annealing augments anisotropy of track revelation in the crystal.

3. The f.t age should be determined on a plane with high etching efficiency and low track loss due to annealing.

4. In case of apatite, a plane parallel to c-axis (i.e. $10\bar{1}0$) is most suitable for f.t dating.

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