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Annealing Properties of Natural Glass Fission Tracks at the Controlled Temperature and its Implication on the Isothermal Plateau Dating Technique

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(with 10 figures and 2 tables)

Abstract

In fission track dating, size-correction and plateau-correction techniques are generally applied account for track loss. The Isothermal Plateau Technique (ITP) combines the size- and plateau-correction methods, wherein the sample are subjected to one-step annealing at a constant temperature. Westgate (1980) applied the ITP technique on samples that were heated at 150°C for 30 days, and plateau phase determined on the coincidence of both induced and spontaneous mean-track diameters. In an attempt to reduce the annealing time, we carried out experiments by annealing samples at temperature to 165°C. The results of these experiment are reported in this paper. When changing experimental conditions for the ITP technique, it is necessary to illustrate the process by which samples achieve plateau phase and its stability under the modified experimental conditions. Furthermore, in order to apply the ITP dating method confidently, detailed analysis of the track properties such as size and density ratios under the plateau conditions are required. In the present experiment, two samples were heated over a period of several days and their annealing behavior from the point of view of both size and density ratio were observed. The results indicate that induced track density decrease dramatically with initial increase in annealing time and subsequently remains constant. On the other hand, spontaneous track density and diameter do not show distinctive changes, even in initial stages of annealing. In the present experiment, the distribution of mean track diameter of induced and spontaneous are observed to coincide after five days of annealing suggesting attainment of the plateau phase. The results indicate that the plateau phase identified by the ratio of induced and spontaneous track density appear before the plateau phase indicated by the coincidence mean track diameter. It is therefore, suggested that the coincidence of mean track diameters defines a prominent and strict plateau phase, as compared to the spontaneous and induced track density ratio. The ages of glass samples that were subjected to annealing at 165°C for 5–10 days using the ITP method are reported using the above mentioned experimental results are reported and the validity of the results are discussed.

Key Words: ITP-FT dating, JAS-G1, glass

Introduction

Natural glasses are formed during short, intense geological events, such as volcanism, meteoritic impact and tectonic frictional fusion, when rock-melts solidify rapidly without crystallization. Volcanic glasses are frequently used for fission track dating. Earlier studies on glass fission-track, dating were attempted using tektite (SASAJIMA and NISHIMURA, 1971), basalt glass of mid-ocean ridge (FLEICHER *et al.*, 1968), obsidian arrowhead (SUZUKI, 1970) and volcanic glass (STORZER, 1970). A serious problem with glass fission-track dating is that of track fading. The length of fission track become

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shorter when samples are subjected to a thermal affect related to natural spatio-temporal and temperature conditions. Generally, the spontaneous fission tracks in natural glasses have undergone some degree of fading in the normal ambient temperatures (LAKATOS and MILLER, 1972). Fading phenomena of spontaneous tracks has also been verified based studies on track diameter (NAESER *et al.*, 1980; WAGNER, 1979). Glass samples are rather sensitive thermal annealing characteristics (WAGNER and HAUTE, 1992). In order to resolve the thermal affect on the fission track, several annealing experiments have been carried out (FLEISCHER *et al.*, 1964; STORZER and WAGNER, 1971). These experiments suggest that the tracks become fewer in number and shorter with increasing temperature and duration of annealing.

Correcting FT ages of glass samples

Several methods have been suggested in order to obtain correct ages of glass sample. These techniques can categorized as size correction method and plateau method. The method of size correction requires the relation between the mean induced track diameter (D_i) and the induced track density (ρ_i) at various stages of annealing. The results of experiments are plotted on a D_i/D_0–ρ_i/ρ_0 graph and the solid curve presents the smoothed variation through the data spots (D_0 and ρ_0 are values of the non-heated induced track). These curves can be used to determine the ρ_s/ρ_0 rate and the correct apparent age using ρ_i' which represents a point where ratios D_s/D_0 and $D_{i'}/D_0$ are equal.

The plateau methods are classified into ITP (Isothermal Plateau) and ICP (Isochronal Plateau) method. In the ITP method, sets of samples are annealed at constant temperature for different period of heating (BARCHERT *et al.*, 1979) and the induced and spontaneous tracks are counted. On the contrary, in the ICP method, each set of samples was annealed at different temperatures for the same time duration (STORZER and WAGNER, 1977). Both the ITP and the ICP methods involve dating of samples when "Plateau phase" is attained. This plateau phase is recognized when spontaneous and induced track-density ratios are constant (STORZER and POUPEAU, 1973). In the plateau method, it is necessary to heat the samples to different time duration, observe the spontaneous and induced track-density ratios, and determine the plateau phase when the track densities are constant. Therefore, this method requires many samples and also involves considerable experiment time. However, these difficulties can be overcomes by a one-step pre-annealing technique (MILLER and WAGNER, 1981; WESTGATE, 1982). In the one-step pre-annealing method, the samples are heated at constant temperature for a continuous time duration and the spontaneous and induced mean track diameters are observed. In the non-heated sample, the spontaneous fission tracks which are faded show smaller mean diameter than the induced tracks. However, the spontaneous and induced mean track diameters tend to become same after certain stage of heating when the samples are said to have reached the "plateau phase". WESTGATE *et al.* (1987) used this one-step pre-annealing ITP method by heat treatment at 100°C for 90 days; later, the heating condition was changed to 150°C during 30 days (WESTGATE *et al.*, 1989). Both above mentioned experimental conditions show good coincidence of mean track diameters. This one-step pre-annealing method is now referred to as "ITP method". However, the critical data on the annealing process concerning attainment of the plateau have not been reported so far.

In this study, we attempted to change the heating conditions for the purpose of reducing annealing time. Annealing experiments were carried out at 165°C. At present there are no established approach for determining the plateau phase at 165°C. In the present study, we illustrated the process of attaining the plateau phase and its stability when annealing temperature is maintained at a constant temperature of 165°C. Further, the relation between the two approaches for identifying the plateau phase (i.e. constant of track density ratio and coincidence of mean track diameters) has also been discussed.

Description of Samples

Natural glass are generally of two types. One is block glass, such as obsidian and the others the glass shard, such as volcanic glass in tephra. In the present experiment we used both types of samples, namely the JAS-G1 (Wada pass obsidian) and Pink-tuff glass shards. JAS-G1 is a block glass occurring in the vicinity of small rhyolite lava dome (about 45 m wide) of Quaternary age. The rhyolitic dome consists partly of a massive aphyric rock (15 m) with strongly banded obsidian lined with spherulite on either sides (+18 m; 9 m). The sample shows an almost perfect glass form and has no inclusion. This obsidian is recommended as a putative age standard for glass fission track (WADATSUMI *et al.*, 1994). Pink tuff glass is a kind of volcanic ash belonging to Osaka Group of Quaternary age. This glass occurs as flakes and fine-grained shards.

Experimental Procedure

In the ITP method, the samples are subjected to relatively low temperature in a series of heating steps at progressively longer time. WESTGATE (1989) indicated that partial track fading could be achieved by heat treatment at a constant temperature of 150°C over a period of 30 days. On the other hand, NEASER *et al.* (1980) found that when glasses were heated above 200°C, they became badly fractured, making it difficult to count tracks. In this experiment, in order to set the annealing temperature, arrhenious plot of the Clearlake water glass (FLEICHER *et al.*, 1968) was used. The arrhenious plot indicates that about five days annealing at 165°C yields almost similar results as 30 days annealing at 150°C (Figure 1). Therefore, in our experiment, the annealing temperature was maintained at 165°C. The present experiment was carried out to investigate the optimal heating requirement for fission track dating of glass. Based on the experiment, we have verified the arrival of plateau phase at 165°C annealing and confirmed the arrival time of plateau phase. Since the samples used in the present study have homogeneous uranium content and show isotopic etched tracks, the population-subtraction method can be applied for age determination. Population-subtraction method is based

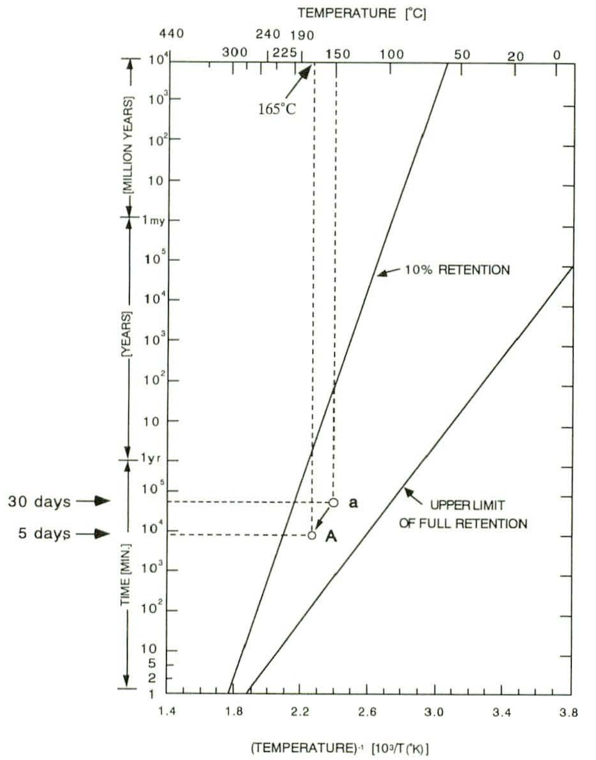

Fig. 1. Arrhenious diagram of fission-track annealing experiments for Clearlake water glass (after FLEISCHER *et al.*, 1968). (a) indicate 30 days annealing at 150°C (WESTGATE, 1989). Five days annealing at 165°C shown by point (A) yield almost similar results as 30 days of annealing represented by pinnt (a). The line joining points (A) and (a) bisects the angle between the two lines .

on the combined counting of both spontaneous and induced tracks (induced sample) and separate counting of spontaneous tracks alone (spontaneous sample) using duplicate samples (Figure 2). At the onset of the experiment, the Tephra samples were washed using isotonic washer and sieved to obtain grains of 75–250 μm (60–200 mesh). The clay minerals were removed from the glass shards and the samples were dried at room temperature. Then, the samples were separated, using heavy liquid. Obsidian samples were sliced using cutter and the surfaces were polished. Each sample was divided into two portions, one portion was irradiated by thermal neutrons for induced track count, and the other portion was used for counting the spontaneous. Irradiation by thermal neutrons was carried out at the Research Reactor Institute of Kyoto University.

The Pink tuff glass shards were heated for 1, 3, 5, 7, 10, 14, 21 and 28 days, and the JAS-G1 samples were heated for 1, 2, 3, 5, 7, 10, 15, 20, 25 and 30 days, respectively at 165°C. After the annealing experiment, the glass samples were mounted on glass

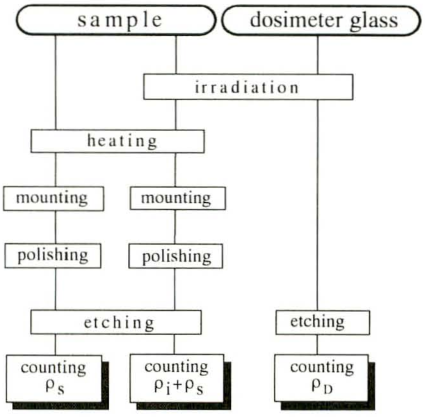

Fig. 2. Method of population-subtraction. Sample is divided into two, one is for spontaneous track counting and the other portion is for combined counting of induced and spontaneous track. The heating and etching for the two portion were carried out together under same experimental conditions. Induced track densities are calculated using the difference between (a) and (b). Dosimeter glass determine the thermal neutron fluence.

slide using epoxy resin, ground and polished. Both induced and spontaneous samples were etched and annealed together under the same conditions (time, temperature and etching conditions). The density and diameter represented by the longest dimension of the ellipse were determined for each set of samples. Track density in Pink tuff glass were measured using the point-counting technique (NEASER, 1982) because the glass shards were of small dimension. In order to count tracks and diameters, the CIPS-FTD (Computer Image Processing System for Fission Track Dating; WADATSUMI and MASUMOTO, 1989) and automatic stage (MASUMOTO and WADATSUMI, 1990) were used.

Results

The experimental results of Pink tuff glass shards are shown in Table. 1. Because of their low uranium content, the Pink tuff shards show very low spontaneous track densities, although large areas were examined. Change in track density is shown in Figure 3. At the beginning of heating, the induced track density decreases rapidly, but after a certain stage the track density remains constant. In the case of spontaneous track density, stability is observed after three days of annealing. During the first three days of heating, both induced and spontaneous track density decrease, but induced track density decrease more dramatically than the spontaneous. This difference in rate of decrease can be attributed to the fading affect. In the Pink tuff glass shards, natural fading phenomena of the spontaneous tracks is observed even before heating. The distribution of track density ratio is shown in Figure 4. The ratio for non-heated sample lower when compared to the heated samples. Hence, after one day of heating, the sample remain stable and reaches the plateau phase. Figure 5 shows the effect on mean

Table 1. The result of annealing of Pink-tuff glass shards.
Induced samples were irradiated using thermal neutron fluence at Research Institute of Kyoto University. Thermal neutron fluence that were irradiated is 2.14×10^{15} cm^{-2}. The data showing number of induced track included some spontaneous tracks, in order to using population-subtraction method. Therefore, the number of induced track, Induced+spontaneous track density are given. Induce track density are estimated as the difference between induced+spontaneous to spontaneous density. D_s/D_i show the ratio of spontaneous mean track diameter (D_s) and induced track diameter (D_i). Coincidence of both diameter, D_s/D_i shown 1.

Heating Time (day)	Number of induced tracks (t)	Counting Area ($10^{-3}cm^2$)	Induced track density ($10^4 \cdot t/cm^2$)	Number of spontaneous tracks (t)	Counting Area ($10^{-3}cm^2$)	Spontaneous track density ($10^2 \cdot t/cm^2$)	Mean track diameter of induced (μm)	Mean track diameter of spontaneous(μm)	Ds/Di ratio	Age (Ma ± 1σ)
0	1999	17.42	11.39	34	39.74	8.56	9.55 ± 2.58	5.84 ± 1.27	0.61	0.98 ± 0.17
1	1323	25.77	5.07	24	40.86	5.88	6.84 ± 2.09	5.85 ± 0.82	0.86	1.52 ± 0.20
3	538	16.62	3.20	10	27.29	3.67	5.56 ± 1.52	5.50 ± 0.76	0.99	1.50 ± 0.32
5	453	13.97	3.21	6	17.50	3.43	6.34 ± 1.94	6.27 ± 1.32	0.99	1.40 ± 0.41
7	710	21.75	3.23	8	21.92	3.65	5.71 ± 1.67	5.70 ± 1.04	1.00	1.48 ± 0.36
10	497	14.61	3.36	8	19.83	4.04	5.42 ± 1.53	5.52 ± 0.36	1.02	1.57 ± 0.36
14	589	19.88	2.93	7	21.60	3.24	4.85 ± 1.21	4.68 ± 0.45	0.96	1.45 ± 0.38
21	362	12.96	2.76	7	20.71	3.38	4.53 ± 1.10	4.31 ± 0.39	0.95	1.60 ± 0.38
28	420	15.97	2.63	8	22.16	3.61	5.24 ± 1.59	4.97 ± 1.09	0.95	1.79 ± 0.36

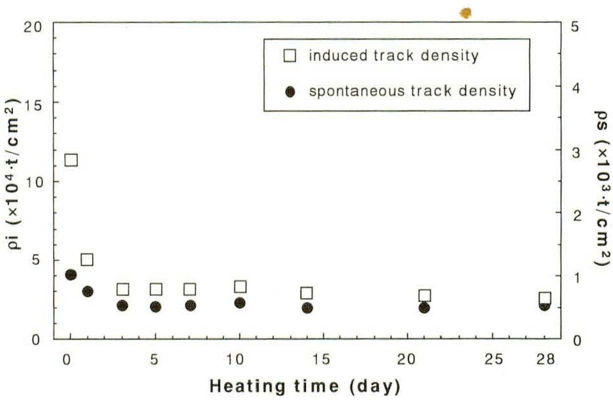

Fig. 3. Changing of induced and spontaneous track density in Pink-tuff glass shards at constant temperature heating. Induced track density decrease rapidly at the beginning, but spontaneous track density does not change so much. After three days of annealing both densities are stable.

diameters of induced and spontaneous tracks. With increased annealing, not only the track density is reduced, but the diameters of the induced samples are also decreases and approaches the diameter of spontaneous mean track. On the other hand, the spontaneous tracks always remains constant. It can be observed from the D_s/D_i ratio shown in Table 1 that after three days heating, the mean track diameters of induced and spontaneous tracks are coincident. This coincidence of mean track diameter indicates

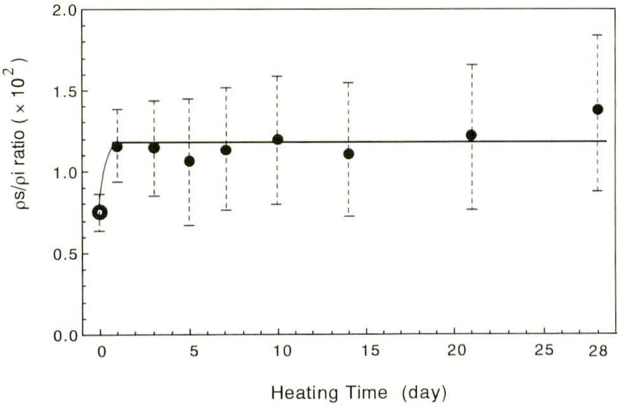

Fig. 4. Spontaneous and induced track density ratio of Pink-tuff glass shards. After one day of annealing, track density ratio almost constant. The line show approximate trend of this distribution.

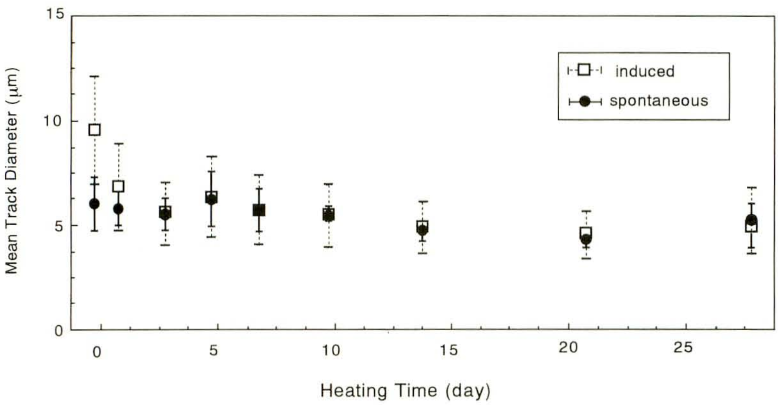

Fig. 5. Circles represent spontaneous mean track diameters and squares show induced mean track diameters of Pink-tuff glass shards. Each bar shows its one-sigma error. Spontaneous samples show wide spread distribution, but by increasing annealing time, each sample becomes concentrated and mean diameters of induced and spontaneous tracks samples coincide.

the attainment of the plateau phase.

The results of JAS-G1 are shown in Table 2. Since the obsidian sample offers a wider counting area and have more uranium content than the Pink-tuff glass, several spontaneous tracks were observed. The Figure 6 indicates change track density. The induced track density decreases after three days of heating and subsequently the track density remained stable. Regarding the spontaneous track density, there is a slight decrease until three days, but after that it remains constant. Figure 7 shows the ratio of spontaneous and induced track density. The plateau phase appeared after heating. Distribution of induced and spontaneous mean track diameter is shown in

Table 2. The annealing results of JAS-G1.
Induced sample were irradiated thermal neutron fluence at Research Institute of Kyoto University. Thermal neutron fluence that were irradiated is 1.14×10^{15} cm^{-2}. The data showing number of induced track included some spontaneous tracks, in order to using population-subtraction method. Therefore, the number of induced track, Induced+spontaneous track density are given. Induce track density are estimated as the difference between induced + spontaneous to spontaneous density. D_s/D_i show the ratio of spontaneous mean track diameter (D_s) and induced track diameter (D_i). Coincidence of both diameter, D_s/D_i shown 1.

Heating Time (day)	Number of induced tracks (t)	Counting Area ($10^{-3}cm^2$)	Induced track density ($10^4 \cdot t/cm^2$)	Number of spontaneous tracks (t)	Counting Area ($10^{-3}cm^2$)	Spontaneous track density ($10^2 \cdot t/cm^2$)	Mean track diameter of induced (μm)	Mean track diameter of spontaneous(μm)	Ds/Di ratio	Age (Ma ± 1σ)
0	1585	8.28	19.16	130	51.72	25.1	11.03 ± 3.75	8.21 ± 2.43	0.74	0.91 ± 0.09
0.25	1048	6.90	15.21	103	44.82	23.0	6.66 ± 1.42	5.86 ± 1.28	0.88	1.06 ± 0.11
0.5	1170	9.38	13.52	112	72.40	22.2	6.49 ± 1.32	5.25 ± 0.82	0.81	1.15 ± 0.10
1	1523	13.79	11.04	114	67.10	17.0	5.68 ± 1.28	5.90 ± 1.42	1.04	1.07 ± 0.10
2	1178	11.03	10.68	123	72.40	17.0	5.46 ± 1.15	5.37 ± 0.84	0.98	1.11 ± 0.10
3	1269	12.41	9.34	101	72.40	14.0	5.86 ± 1.46	5.04 ± 1.13	0.86	1.05 ± 0.11
5	940	10.00	9.40	109	72.27	15.1	5.37 ± 0.81	5.75 ± 1.76	1.07	1.12 ± 0.10
7	1140	12.41	9.19	103	70.68	14.6	5.43 ± 0.98	5.10 ± 0.76	0.94	1.11 ± 0.11
10	1304	13.72	9.50	123	75.71	16.2	5.25 ± 1.13	5.05 ± 0.81	0.96	1.12 ± 0.10
15	1316	15.10	8.71	124	86.20	14.4	4.45 ± 0.72	5.01 ± 0.86	1.12	1.15 ± 0.10
20	1170	12.41	9.43	123	84.47	14.6	4.79 ± 1.01	4.29 ± 0.56	0.90	1.08 ± 0.10
25	1618	17.86	9.06	98	65.23	15.0	5.72 ± 1.44	5.73 ± 1.45	1.00	1.16 ± 0.11
30	1050	10.00	9.24	107	72.40	14.8	5.32 ± 1.35	5.36 ± 1.34	1.01	1.12 ± 0.10

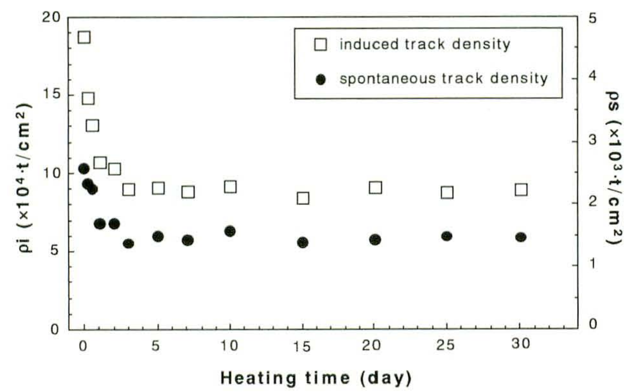

Fig. 6. Annealing of induced and spontaneous track density of JAS-G1. Both track density decrease rapidly at the beginning of annealing. Both densities remain constant after three days of annealing.

Figure 8. Non-heated samples are different from each other and obviously not in plateau phase. Starting from 6 hours up to five days heating, samples show almost similar time of arrival for the plateau phase. However, with three days of annealing, D_s/D_i (Table 2) do not coincide. Therefore, it seems these samples have not attained the plateau

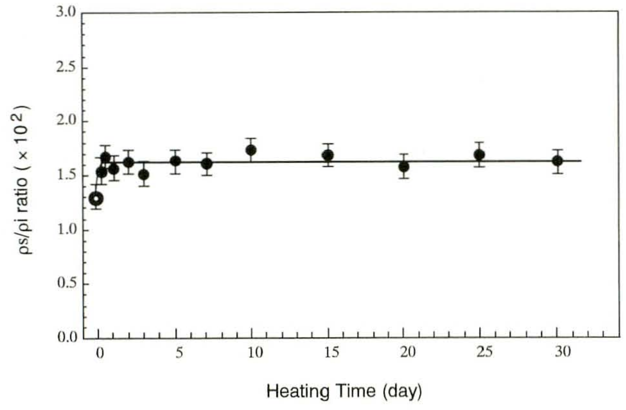

Fig. 7. Heating time vs. spontaneous/induced track-density ratio. After heating, spontaneous and induced track density ratios remain constant and show the plateau. The line shows approximate line of this distribution.

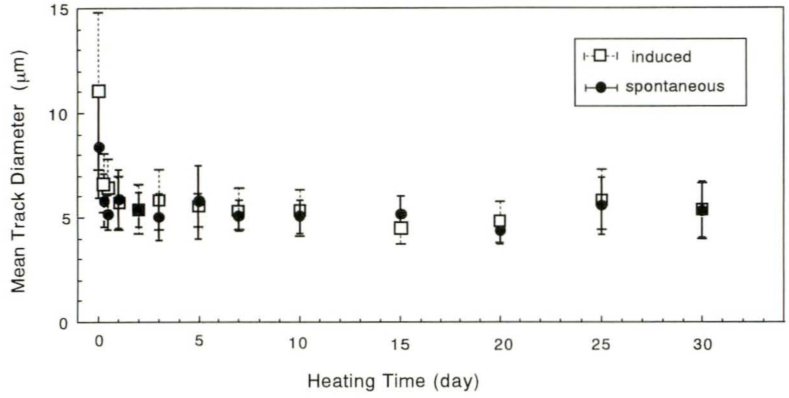

Fig. 8. Distribution of induced and spontaneous track diameters of JAS-G1. Both induced and spontaneous samples show widespreaded error at non-heated samples. After one day heating, both mean track diameters coincide, although these are unstable until five days.

phase as yet. After five days of heating, the plateau phase is recognized by the coincidence of the mean diameters of induced and spontaneous tracks. Based on the results for Wada pass obsidian it is observed that the coincidence of mean diameters of induced and spontaneous tracks appears after track density ratio becomes constant. The plateau phase for JAS-G1 occurs later when compared to the Pink-tuff sample. Figure 9 and 10 show the age determination for both samples at different stages of heating. The ages calculated before the plateau phase is attained are unstable and show younger ages when compared to ages determined after the plateau phase.

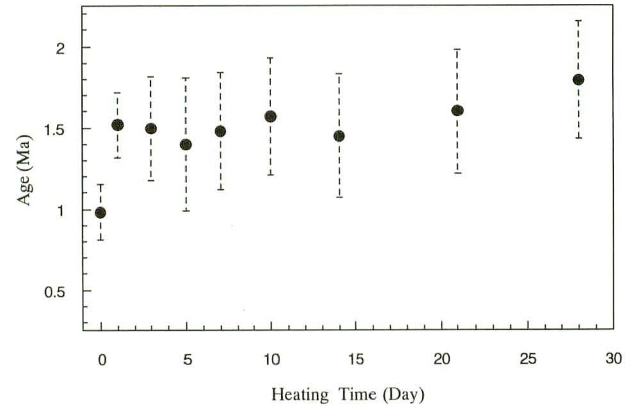

Fig. 9. Age distribution of pink-tuff. Ages caliculated from the age equation $t=1/\lambda_D \cdot \ln[1+\lambda_D \Phi \sigma I \rho_s/\lambda_f \rho_i]$, using following values: $\lambda_D=1.551\times10^{-10}$; $\lambda_f=7.03\times10^{-17}$; $\sigma=580\times10^{-24}$; $I=7.252\times10^{-3}$; $\Phi=2.14\times10^{15}$.

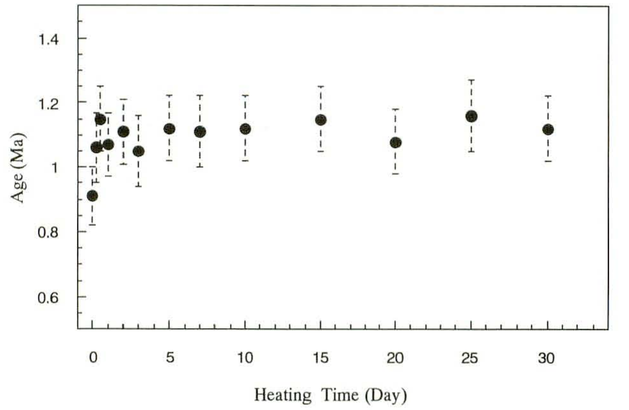

Fig. 10. Age distribution of JAS-G1. Ages caliculated from the age equation $t=1/\lambda_D \cdot \ln[1+\lambda_D \Phi \sigma I \rho_s/\lambda_f \rho_i]$, using following values: $\lambda_D=1.551\times10^{-10}$; $\lambda_f=7.03\times10^{-17}$; $\sigma=580\times10^{-24}$; $I=7.252\times10^{-3}$; $\Phi=1.14\times10^{15}$.

Discussion

The results of the present experiment suggest that the plateau phase of both pink-tuff glass shards and JAS-G1 is confirmed at 165°C. Based on the age determination, it is observed that samples show younger ages before the plateau phase is attained. This can be atributed to the prevalence of fading effect. The ages determined when plateau phase is attained are considered to represent the correct ages for the samples. In relation to the track density ratio and the mean track diameter distribution, coincidence of mean track diameter is observed after the track density ratio remains constant. Using these results from both samples, three stages of the annealing process are distinguished. The first is the non-heated stage wherein the samples are not the plateau phase. The second stage is the unstable plateau phase. This occurs from the beginning of heating up to five days. In the second stage the track densities decrease and no coincidence of the mean diameter of induced and spontaneous track is observed. Although the spontaneous and induced track density ratios tend towards the plateau phase in this stage, the coincidence of mean track diameters is not stable. In this unstable plateau phase, it seems that each sample traces its own process until it attains the plateau phase depending on individual sample characteristics. The third stage represents the real plateau phase which was observed from five to 30 days of annealing. The plateaus were defined on the basis of both track density ratio and coincidence of mean track diameters. Further, in this stage, the plateaus are very stable. Hence, the coincidence of mean track diameters defines the more prominent and stable plateau phase than the track-density ratio. Based on this observation, it is suggested that the coincidence of mean track diameter is a more reliable criteria for identifying the stable plateau phase.

The plateau phase defined by density ratio occurs at several points of the annealing period. On the contrary, the plateau defined by the coincidence of the mean track diameter's occurs only once during the entire period on annealing. Therefore, the one-step pre-annealing ITP method is convenient and reliable. WESTGATE (1989) reported results of annealing over a period of 30 days heating at 150°C. It is considered this annealing time was necessary to established in the stable plateau. The results of the present study have that the stable plateau can be identified by coincidence of mean track diameter for every analyzed sample. It is therefore suggested that heating can be maintained at 165°C, and that five to 10 days annealing is the optimal for confirming the plateau phase. Beyond 10 days it is difficult to maintain the heating temperature in the present experimental set-up and also dating is time-consuming.

Conclusion

In this investigation, an annealing experiment on glass fission tracks was carried out for the purpose of resolving the relationship between mean track diameter and track density ratio. The experimental results suggest new annealing temperature of 165°C can be used ITP technique for dating glass samples. Recognition of stable plateau is better defined by the coincidence of mean diameters of induced and spontaneous tracks. Further, the study also demonstrates that 5 to 10 days of annealing is the adequate when the annealing temperature of 165°C is used in the ITP technique. Prior to five days of annealing, coincidence of the mean track diameter is not so is not observed. On the other hand, heating beyond 10 days heating is time-consuming and makes counting of tracks difficult. Based on the above results it is suggested that five to 10 days annealing at 165°C is adequate to produce plateau stability, and that mean track diameter coincidence is the most suitable criteria for identifying the plateau phase in ITP method.

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