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FISSION-TRACK DATING OF A TEPHRA LAYER IN THE ALAT FORMATION OF THE DANDIERO GROUP (DANAKIL DEPRESSION, ERITREA)

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Abstract. Attempts to date a biotite separate from a tephra layer recognised near Buia (Danakil Depression, Eritrea) in the lower part of the *Homo* remains – bearing Dandiero Group (formerly attributed to the Danakil Formation) using the $^{39}Ar/^{40}Ar$ method failed because of xenocrystic contamination. For this reason it was applied the fission-track method on glass, since no other phases datable with this technique were present. The quality of glass was very poor for fission-track dating, because of the small size of grains. In addition, after polishing only few glass shards showed useful surfaces for track counting and only 25 spontaneous tracks were counted. The determined fission-track age – 0.75 ± 0.16 Ma – is a rejuvenated age due to the presence of a certain amount of annealing of spontaneous tracks. An attempt to apply the plateau method for correcting this apparent age failed. A corrected age of 1.3 ± 0.3 Ma was computed using the size-correction method. In spite of its low precision, this fission-track age represents a significant result, since it corroborates the attribution to Jaramillo Subchron of the normal magnetozone near the base of which the tephra is located.

Riassunto. Recentemente importanti resti di *Homo* sono stati scoperti nel Gruppo del Dandiero (precedentemente attribuito alla Formazione Dancala) presso Buia (Eritrea). Un primo tentativo di ottenere un dato cronologico datando con il metodo $^{39}Ar/^{40}Ar$ un campione di biotite separata da una cinerite rinvenuta presso la base della Formazione di Alat 85 metri sotto il livello contenente resti di *Homo* non ha avuto successo a causa della contaminazione con cristalli detritici. Per questo motivo un nuovo tentativo è stato intrapreso con il metodo delle tracce di fissione nel vetro, unica fase databile con questo metodo presente nella cinerite. Le caratteristiche del campione risultavano poco adeguate per l'analisi, soprattutto per le piccole dimensioni dei frammenti di vetro. Inoltre, dopo il montaggio, pulimentazione e l'attacco chimico del campione soltanto una piccola frazione dei frammenti di vetro presentavano una superficie utile per il conteggio delle tracce. Per questo motivo, nonostante siano stati preparati sei montaggi diversi, si sono potute contare soltanto 25 tracce fossili. L'età misurata – 0,75 ± 0,16 Ma – è una età ridotta a causa della presenza di un parziale tasso di accorciamento delle tracce fossili, fenomeno che nei vetri si verifica generalmente anche a temperatura ambiente. Il tentativo di ottenere l'età di formazione del vetro utilizzando il metodo del plateau non ha avuto successo a causa della riduzione ulteriore sia della densità di tracce che delle superfici utili per il conteggio. Non rimaneva altro che stimare l'età della cinerite applicando il metodo delle dimensioni delle tracce per la correzione delle età apparenti, che comporta un incremento dell'errore della misura. L'età ottenuta con questo metodo – 1,3 ± 0,3 Ma – ha una precisione modesta. Nonostante ciò, questa datazione rappresenta un risultato significativo dato che conferma l'attribuzione al subcrono Jaramillo della magnetozona diretta presso la base della quale è stato rinvenuto il livello cineritico.

Introduction

Tephra are synchronous layers of pyroclastic material which intercalate several continental as well as marine sedimentary series. If they have peculiar chemical, physical and/or geological characteristics, tephra can serve as marker horizons that can be used for stratigraphic correlation. In addition, many tephra include mineralogical phases which can be dated through geochronological methods. So they provide geologists a precious opportunity of fixing time constraints that may be very useful for stratigraphical studies.

The K-Ar as well as the $^{39}Ar/^{40}Ar$ method has been widely used in tephrochronology. Especially the $^{39}Ar/^{40}Ar$ method is nowadays preferred to other techniques for dating tephra beds for its high precision. Nevertheless, numerous volcanic ash layers do not include mineralogical phases datable with this method. For this reason since early seventies the fission-track

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method became an alternative dating technique frequently used in tephrochronological studies. In principle, fission-track dating seems to be an ideal tool for this purpose. Most tephra contain glass shards or mineral phases, such as zircon, apatite or sphene that are suitable for the application of this technique. However, fission-track dating has the disadvantage of its lower precision. Glass shards revealed to have unfavourable properties for the application of the K-Ar as well as ^{39}Ar/^{40}Ar method. But although some glasses yield only low-precision ages, this material is of great importance in fission-track dating because it is the only datable phase of many tephra (Walter 1989; Westgate 1989).

Recently, significant *Homo* remains were discovered near Buia (Eritrea) in the Dandiero Group, a ca. 1000 m thick succession of fluvio-deltaic and lacustrine deposits. The Alat Formation has been distinguished in the lower half of the Dandiero Group with a 1 m thick ash layer occurring ten meters from its base and 85 meters below the *Homo* layer. The latter is placed toward the top of a normal magnetozone which was identified as the Jaramillo Subchron (Abbate et al. 1998; Albianelli & Napoleone 2004). With the aim of establishing a chronological constraint, an attempt of dating a biotite separate using the ^{39}Ar/^{40}Ar method was made, but xenocrystic contamination prevented determination of a reasonable age. For this reason, a second attempt using the fission-track dating method on glass shards was carried out and the results are here presented.

Peculiarities of fission-track dating of glass

The spontaneous nuclear fission of ^{238}U occurs at a constant rate during time. Each fission event produces in the solid in which it takes place a damaged region – a trail ~ 10 ÷ 20 μm long – named 'latent track' that can be developed by chemical etching and observed under a microscope (Fig. 1). The number of 'fossil' fission tracks accumulated during geological times in a mineral or natural glass is proportional to its U-content as well as to the time elapsed since it formed. Therefore, the fission-track dating method consists in the determination of the fraction of ^{238}U atoms which experienced the spontaneous fission. An irradiation with thermal neutrons in a nuclear reactor produces the induced fission of the ^{235}U isotope. The number of induced tracks formed during irradiation is proportional to the unknown U-content. In practice, a fission-track age determination consists in the estimate of two track areal densities – the spontaneous and the induced track densities – through counting procedures (an exhaustive description of the fission-track method and of its applications is given by Wagner & Van den haute 1992).

Considering the description made of fission-track dating, one can easily understand the reason why this technique commonly produces lower precision ages compared to the ^{39}Ar/^{40}Ar method. Experimental evidences prove that in case of homogeneous U-content, track-counts follow Poisson distributions. Therefore, in this favourable case (most glasses show poissonally distributed tracks, whereas very rarely minerals do) the experimental error of an areal density determined through a certain number N of tracks is $N^{1/2}$, which means that if N is 100 or 1000, the corresponding relative error is 10% or ~ 3%, respectively. In most cases glasses or minerals from Plio-Pleistocene tephra do not show spontaneous track densities larger than few thousands of tracks cm^{-2}. For many samples to count 1000 tracks turns to be an utopia.

The fission-track dating method is based on the assumption that the 'fossil' tracks are stored undisturbed in a sample. Actually, thermal stability of tracks in some materials is rather poor: especially in glass a certain degree of annealing of the latent tracks frequently takes place also at ambient temperatures. For this reason, commonly other phases – such as zircon, apatite and sphene – are preferred in tephrochronological studies because of (1) their higher fossil track stability (heavy minerals from volcanics commonly contain virtually undisturbed tracks) and of (2) their higher U-content.

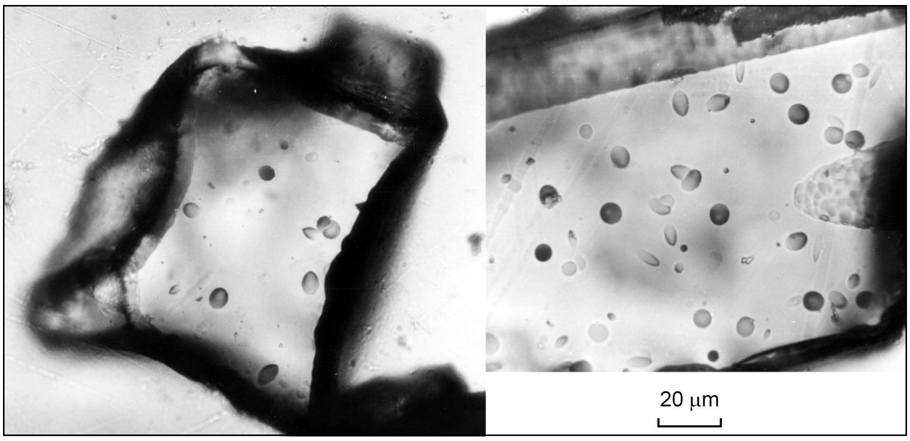

Fig. 1 - Induced tracks developed on glass shards from tephra beds (etching: 120 s in 20% HF at 20°C).

Partially annealed tracks on glass are developed with reduced efficiency in comparison with the ^{235}U induced tracks, which are 'fresh' tracks artificially produced. Therefore, etched fossil tracks commonly show a certain reduction of the mean size D_S (the mean major axis of the etch-pit). A D_S/D_I mean size ratio < 1 (where subscript I denotes induced tracks, assumed as reference undisturbed tracks) indicates reduced etching efficiency of spontaneous tracks and a corresponding decrease of the areal spontaneous track density and, consequently, of the age which is determined through the spontaneous to induced track density ratio. The less the D_S/D_I ratio is, the more the fission-track age is reduced.

Therefore, a fission-track age on glass is commonly a 'minimum' age (referred to as 'apparent' age), unless a technique for correcting thermally lowered ages is applied. Storzer & Wagner (1969) and Storzer & Poupeau (1973) proposed the 'size-correction method' and the 'plateau method', respectively.

The first technique is based on estimate of track density loss by track-size measurements. Thermal treatments of varying intensity, obtained changing duration and temperature, are imposed to several splits of an irradiated sample in order to produce variable amounts of track-annealing. For each split the D/D_0 (ratio between the mean size of partially annealed and undisturbed tracks) and ϱ/ϱ_0 (ratio between areal density of partially annealed and undisturbed tracks) ratios are measured. The D/D_0, ϱ/ϱ_0 points obtained in this way allow to draw out an experimental curve named 'correction curve' which represents the relationship between track-size reduction and corresponding track areal density decreasing (Fig. 2). Using this curve, the value in the ϱ/ϱ_0 axis corresponding to the D_S/D_I ratio determined in the sample represents an estimate of the age reduction due to the spontaneous tracks partial annealing.

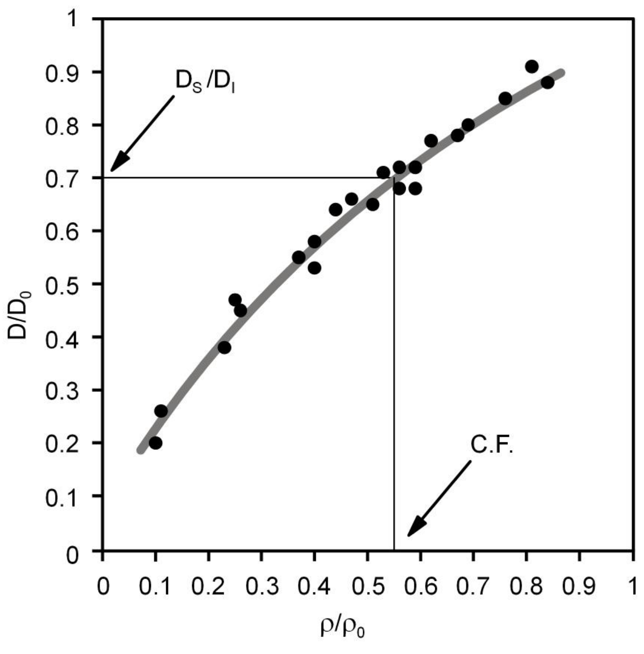

Fig. 2 - The experimental curve that relates reduction of areal track density (ϱ/ρ_0) with corresponding reduction of mean track size (D/D_0) is drawn through annealing experiments of induced tracks. Using this curve, from the D_S/D_I ratio determined on a sample one can deduce a correction factor (C.F.) for obtaining the glass formation age (Corr. Age = App. Age / C.F.).

The plateau method consists in re-establishing by laboratory thermal treatments an identical etching efficiency of spontaneous and induced tracks. This technique is based on the experimental evidence that partially annealed tracks are progressively more resistant to further annealing. If increasing intensity heating steps (changing duration and/or temperature) are applied to two aliquots (one of them irradiated with neutrons) of a sample affected by partial annealing of spontaneous tracks, its age progressively increases up to a plateau. The plateau is reached when the amount of natural plus artificial annealing of spontaneous tracks = the amount of artificial annealing of induced tracks.

Experimental evidence indicates that these techniques produce equivalent results and that corrected fission-track ages on glass are commonly reliable formation ages (Arias et al. 1981; Naeser et al. 1981; Storzer & Wagner 1982; Westgate 1989). Nevertheless, the plateau method is commonly preferred, especially for its higher precision.

Experimental

Standard magnetic separation applied to a sample – sample DR 3L – from the tephra subject of this study did not yield mineral phases suitable for dating with the fission-track method. Therefore, the only opportunity of determining an age was provided by an abundant amount of glass, although it did not appear very promising, as it consisted of a population of very small glass shards. In addition, transparent grains were very rare.

Glass DR 3L was processed using methodologies developed by the fission-track group of Pisa (see, for instance, Bigazzi et al. 2000, Espizua et al. 2002). An aliquot of glass DR 3L was separated for irradiation with thermal neutrons. Irradiation was performed in the Lazy Susan (Cd ratio 6.5 for Au and 48 for Co) Triga Mark II nuclear reactor of the University of Pavia. The neutron fluence was determined using the standard glass NRM IRMM – 540 recently prepared by the Institute for Reference Materials and Measurements, on behalf of the European Commission (De Corte et al. 1998).

After irradiation, two aliquots from glass DR 3L (for spontaneous and induced track counting – the irradiated one) were mounted in epoxy resin and polished with diamond paste or spray with decreasing granulometry (down to 0.25 μm). Tracks were developed by chemical etching for 120 s in 20% HF at 20°C. Tracks were counted under a Jena – Jenaval microscope at a magnification of 500x using a grid. Most glass shards were pomiceous and did not showed surfaces useful for counting. In addition, only rare good quality transparent grains were present, but their size was very small (diameter less than 40 μm). These characteristics prevented application of the 'point-counting technique' (see Westgate, 1989, and references therein), a counting strategy that in common experimental conditions allows to count significantly larger numbers of tracks in shorter times. To use a traditional counting procedure – estimate of the area useful for counting of each glass shard using an eye-piece equipped with a grid – was the only thing we could do. Track-sizes were measured with Leica Microvid equipment at 1000x.

Due to the difficult experimental conditions, although six specimens had been prepared for spontaneous track counting, only few spontaneous tracks (25) were found after a time consuming counting procedure, in spite of a relatively large track density for a Pleistocene glass (Table 1). Also the number of the induced tracks we were able to count is relatively small, compared to other glasses from tephra beds. Commonly at least 1000 induced tracks are counted. The spontaneous to induced track-size ratio value shown in Table 1, D_S/D_I = 0.72 (see also Fig. 3), indicates a rather sensible degree of partial annealing for glass DR 3L. An attempt to apply the plateau method (Storzer & Poupeau 1973), failed because of a very significant reduction of surface useful for counting after the thermal treatment imposed for the attainment of the plateau condition. Therefore, the size-correction method (which implies an increase of experimental error, especially when a restricted number of sizes can be measured) was the only alternative for obtaining the formation age of the ash layer. Application of this technique (see also Arias et al. 1981) for correcting the apparent age of 0.75 ± 0.16 Ma produced an age of 1.3 ± 0.3 Ma.

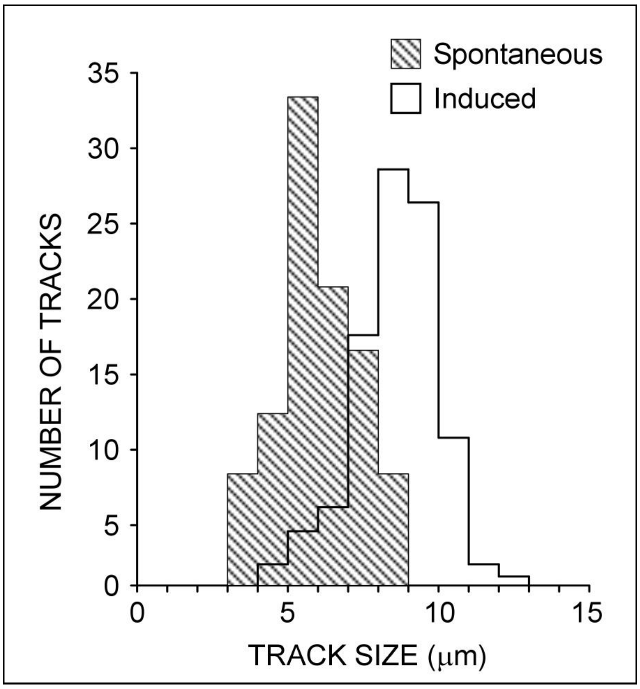

Fig. 3 - Spontaneous and induced track size distributions (normalised to 100) on glass DR 3L. A D_S/D_I ratio of 0.72 was determined.

Conclusions

After an unsuccessful attempt with $^{39}Ar/^{40}Ar$ method, fission-track dating method was applied to date a tephra 85 meters below a *Homo* bearing layer in the Dandiero Group near Buia (Eritrea). Glass turned to be the only datable phase of a sample from this tephra (sample DR 3L). The small sizes of the glass shards, the presence of rare surfaces useful for track counting after mounting, polishing and etching made very difficult the experimental procedure. Nevertheless, although the apparent (0.75 ± 0.16 Ma) and the size-corrected (1.3 ± 0.3 Ma) ages have large experimental errors (> 20%), they are the only radiometric datings at the moment available. The size-corrected age of glass DR 3L is consistent with the Jaramillo Subchron recognised in the studied succession and with paleontological evidence (Abbate et al. 1998; Martínez-Navarro et al. 2004).

Φ [cm^{-2}]	ρ_S [cm^{-2}]	N_S	ρ_I [cm^{-2}]	N_I	$p(\chi^2)$ (%)	D_S/D_I	App. Age (±1σ) [Ma]	Corr. Age (±1σ) [Ma]
1.7 x 10^{15}	1420	25	161,000	345	79	0.72	0.75 ± 0.16	1.3 ± 0.3

Table 1 - Fission-track dating of glass DR 3L

Φ: neutron fluence; ρ_S (ρ_I): spontaneous (induced) track density; N_S (N_I): spontaneous (induced) track counted; $p(\chi^2)$: probability of obtaining χ^2 value testing induced track counts against a Poisson distribution; D_S/D_I: spontaneous to induced track-size ratio. Parameters used for age calculation: $\lambda = 1.55125 \times 10^{-10} a^{-1}$; $\lambda_F = 8.46 \times 10^{-17} a^{-1}$; $\sigma = 5.802 \times 10^{-22} cm^2$; $^{238}U/^{235}U$ = 137.88. Errors of ages are propagation of Poisson track counting errors and of neutron fluence determination uncertainty (9877 tracks were counted on mica external detectors juxtaposed to glass NRM IRMM – 540 during irradiation). For the size-corrected age, the error added by the correction itself was also considered.

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