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Fission Track Dating

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PART I

INTRODUCTION  
TO  
FISSION TRACK  
DATING

### History and Theory:

Techniques used for dating geologic and archaeologic materials using fission-fragment tracks have evolved over the last decade. Fission-track dating is just one facet of the rapidly expanding field of Solid State Track Recorders (SSTR) (Fleischer and others, 1975). The early developmental work on SSTR was done by three physicists, Robert L. Fleischer,

## Introduction

The purpose of this report is to outline the basics of the fission track dating method. It is divided into two parts. The first part deals with the theory, annealing, and a few geologic examples of fission-track dating. The second part is a laboratory cook book. I have tried to give step by step instructions for dating most materials. No doubt there are a number of different and possibly better ways to proceed, but I have found these to be useful and successful.

This report is assembled from a number of different sources. It combines lecture notes, and a listing of laboratory procedures made for visitors and students.

Fission-track dating is not a do-it-yourself, start from scratch type of a project. There are a number of possible pitfalls and blind alleys to which the unsuspecting can stray. It is also very possible to get the "right" age for very wrong reasons. "Right" is not always "correct." I think that this is a problem every fission track geochronologist has had at least once. I therefore strongly recommend that anyone starting out in fission-track dating visit an operating laboratory. You will save months and possibly years with a 2-week visit.

Why make the same mistakes everyone else has, when you can be original.

P. Buford Price, and Robert M. Walker, working at the General Electric Companies Research Laboratory in Schenectady, New York. Although they did not discover that charged particles leave a damage zone in a solid, they are the ones largely responsible for SSTR's current state of development.

The first charged-particle tracks observed were those produced by fission fragments. Initial discoveries of tracks were reported by Young (1958) and Silk and Barnes (1959). Young observed shallow pits in lithium fluoride crystals which had been etched after being irradiated with fission fragments. Silk and Barnes, using an electron microscope, observed fission fragment tracks in a piece of mica; but the tracks they observed faded quickly in the electron beam. Price and Walker (1962) discovered that the damage zone created by the passage of a fission fragment could be made visible in an optical microscope by chemical etching. This enlarged the tracks and made them visible with the aid of an optical microscope. Early studies dealt with the dating of micas (Price and Walker, 1962) and natural glasses (Fleischer and Price, 1963). Their early studies have led to the use of SSTR in many diverse fields: geology, biology, archaeology, high-energy physics, nuclear physics, and the study of extra-terrestrial materials such as meteorites and returned lunar samples.

A charged-particle track is the damage zone formed as a charged particle passes through a solid. A number of

different types of tracks exist (Fleischer and others, 1975). The length and shape of the different tracks are dependent on the type of particle that formed the track. In terrestrial samples there is only one common, naturally occurring track: the fission track. Most of the other types of natural tracks are found in meteorites and in lunar minerals and glasses. These tracks are caused by cosmic-ray interactions within the extraterrestrial materials. Because the earth's atmosphere absorbs most of the cosmic rays capable of forming a track, these tracks are not seen in terrestrial materials. The remainder of this paper will be devoted to the fission track, the track formed by the two nuclei of a fissioning heavy element such as uranium; the original nucleus breaks up into two lighter nuclei of approximately equal mass and liberates about 200 MEV of energy. The two nuclei recoil from each other in opposite directions. Figure 1 shows what is generally thought to be the track-forming mechanism (Fleischer and others, 1965a). The fragments formed at the time of fission are highly charged nuclei that disrupt the electron balance of the atoms in the mineral lattice along their path. As the fission fragment passes, it leaves a zone of positive charge in its wake. This causes the positively charged ions in the lattice to repulse each other and force themselves into the crystal structure, forming the track or damage zone. The new track is only angstroms wide and about 10-20 micrometers long.

The track is stable in all insulating solids, but conducting and semi-conducting solids do not retain tracks as movement of electrons rapidly neutralizes the ions produced.

Three naturally occurring isotopes spontaneously fission:  $^{232}\text{Th}$ ,  $^{235}\text{U}$ , and  $^{238}\text{U}$ . Of these, only  $^{238}\text{U}$  produces a significant number of fission events. The other two have such long half-lives for spontaneous fission that, for all practical purposes, all fission tracks can be assumed to have come from  $^{238}\text{U}$ .  $^{238}\text{U}$  decays by alpha emission as well as by spontaneous fission. Millions of uranium atoms decay by alpha emission for each atom that decays by spontaneous fission.

Once a track is formed it is stable in most insulating materials at temperatures less than  $100^{\circ}\text{C}$  (Fleischer and others, 1975). With increasing temperature, the atomic movement in a solid increases, and the displaced ions along this damage zone will diffuse back into the track and cause the damage zone to fade until it can no longer be seen by etching. Track fading, annealing, and its usefulness will be covered in a later section of this paper. Assuming a track formed at a time when the temperature of a solid was below the temperature at which fading begins, it will be stable over a long period of time.

Tracks in their natural state are too small to be seen except with an electron microscope. By choosing the proper chemical etchant, it is possible to dissolve out the damage zone and not dissolve its crystal (Price and Walker, 1962).

Once the track is etched, it can be observed in an optical microscope at moderate magnifications (200-500X). Common etchants used include nitric acid (apatite), hydrofluoric acid (micas, glass), and concentrated basic solutions and fluxes (sphene, zircon) (Fleischer and others, 1975). Figures 2 through 4 show tracks in two minerals (apatite and zircon) and in a piece of teklite glass (moldovite). Other common minerals in which fission tracks have been seen include sphene, garnet, epidote, muscovite, and biotite. Natural glasses such as tektite, obsidian, and glass shards also contain fission tracks. Fleischer, Price, and Walker (1975) present an extensive list of minerals which may contain fission-tracks, along with the proper etchants and etching conditions to develop the tracks.

A given mineral can be dated if enough time has elapsed since its formation to accumulate a significant number of tracks. The track density or number of tracks per unit of surface area is a function of the age of the material and its uranium concentration. In very young rocks, minerals having a high concentration of uranium must be found; in old Precambrian rocks, the problem is to find minerals that have a low concentration of uranium. Zircons are generally very good minerals for dating Cenozoic rocks, but they usually have too high a track density to be useful in dating Precambrian rocks. In order to calculate the age of a mineral or glass, we need the spontaneous track density and the uranium concentration.

The fission-track-age equation is as follows (Price and Walker, 1963):

$$A = \ln \left[ 1 + \frac{\rho_s}{\rho_i} \cdot \frac{\lambda_D \phi \sigma I}{\lambda_F} \right] \frac{1}{\lambda_D}$$

Describing the various terms in this equation will show how a fission track age is determined. The fossil track density " $\rho_s$ " is dependent on the age and uranium concentration of the mineral. The Cenozoic zircon could have the same track density as a Precambrian epidote. So if the uranium concentration can be determined, we can calculate the age. The best way to determine the uranium concentration is to induce a new set of fission tracks. To do this we utilize the neutron fission reaction of  $^{235}\text{U}$ . If a sample is sent to a nuclear reactor and exposed to thermal neutrons, a new set of tracks is made. The terms  $\rho_i$ ,  $\phi$ ,  $\sigma$ , and  $I$  are related to the irradiation process:  $\rho_i$  is the density of neutron induced tracks,  $\phi$  is the number of neutrons per square centimeter that pass through the sample,  $\sigma$  is the cross-section for neutron fission reaction of  $^{235}\text{U}$ ;  $I$  is the atomic ratio  $^{235}\text{U}/^{238}\text{U}$ .  $\lambda_D$  is the total decay constant for  $^{238}\text{U}$  decay and  $\lambda_F$  is the decay constant for spontaneous fission. The values for the constants are listed below:

$$\begin{aligned} I &= 7.252 \times 10^{-3} \\ \lambda_D &= 1.551 \times 10^{-10} \text{ yr}^{-1} \\ \lambda_F &= \begin{array}{l} 6.85 \times 10^{-17} \text{ yr}^{-1} \\ 7.03 \times 10^{-17} \text{ yr}^{-1} \end{array} \begin{array}{l} \text{(Fleischer and Price, 1965a)} \\ \text{(Roberts, Gold, and Armani, 1968)} \end{array} \end{aligned}$$

or  $8.42 \times 10^{-17} \text{ yr}^{-1}$  (Spadavacchia and Hahn, 1967; Wagner and others, 1975)

$$\sigma = 580 \times 10^{-24} \text{ cm}^2$$

Three methods are commonly used to determine  $\rho_i$  independent of  $\rho_s$ .

The first is to divide the mineral grains into two groups (Naeser, 1967). The first group is mounted in epoxy and polished. These grains will be used to determine the fossil-track density. The second group is heated in a furnace to a temperature high enough to cause complete annealing of the fossil tracks. These grains are then irradiated. Following the irradiation they are mounted, polished, and etched along with the first mount. The induced track density is then determined from the grains in this group. This method can be used only if the uranium concentration of all the grains is uniform. Apatite usually can be dated this way. This method requires a minimum of several hundred grains.

A second method is to use an external detector arrangement (Fleischer and others, 1964; Naeser and Dodge, 1969). For this, a mineral is mounted in epoxy, polished, and etched in order to reveal the fossil tracks. The grain mount is then covered with a piece of clean, low-uranium muscovite or lexan (a polycarbonate plastic), and irradiated with a neutron dose monitor. The uranium that fissions near the surface of the grain will send fission fragments into the adjacent muscovite detector. After irradiation, the muscovite is etched and the

induced tracks are counted. The advantage of this method is that the fossil tracks are counted on the grain and the induced tracks are counted on the image of the grain in the detector; therefore, variations in uranium concentrations can be dealt with. It is possible, but not recommended, to date only one grain using this method; normally five or more grains would be dated. This method is used for most minerals other than apatite. An assumption made is that the etching efficiencies for the mineral and the muscovite are similar, or that if they are not, the proper correction has been made. Figures 5, 6, 7, and 8 show a sphene grain and induced tracks from it in a muscovite detector.

A third method, which is primarily used for dating glass (Fleischer and Price, 1963), involves mounting, polishing, and etching a sample prior to irradiation. The sample is irradiated, and after irradiation the fossil track density is determined. The sample is then repolished (to remove about 20-30 micrometers), etched, and counted again. The second count contains both the fossil and induced tracks. The induced-track density is obtained by subtracting the fossil-track density from the combined track density. A variation of this method is to split the sample and do a population type of procedure (see preceding discussion for apatite), except to omit the annealing prior to irradiation. Annealing of glass can alter its properties, which could change the etching characteristic of the glass. This probably would cause a

spurious age. This procedure is used for glass because it has a very much lower etching efficiency than muscovite, which prevents the use of an external detector.

Terrestrial samples as young as 8,000 yr (Storzer and others, 1971) and as old as  $2.7 \times 10^9$  yr (Naeser, unpublished data, 1971), have been dated using fission-track procedures. Figure 9 shows some examples of fission-track ages plotted against K-Ar or Rb-Sr ages. Generally, fission-track ages of minerals such as apatite, zircon, and sphene from young, Cenozoic, volcanic deposits are concordant with each other and with the K-Ar ages of those deposits (Naeser and McKee, 1970; Naeser, 1971; and Izett and Naeser, 1976). Natural glasses are both concordant and discordant (Fleischer and Price, 1964b; Storzer and Wagner, 1969; Storzer, 1970). Tests for annealing should be made before accepting a fission-track age on glass (Storzer and Poupeau, 1973; Storzer and Wagner, 1969). Minerals such as apatite, zircon, and/or sphene dated by fission track from plutonic and metamorphic rocks can show discordant ages (Naeser and Dodge, 1969; Wagner, 1968; Wagner and Storzer, 1970, 1975; Naeser and Ross, 1976). These discordant ages are caused by track fading under geologic conditions. Track fading is one of the most useful aspects of fission-track dating.

Early work in fission track dating showed that sometimes a sample would have a fission track age which was younger than the K-Ar or Rb-Sr age on other minerals from

the same rock. This early work showed that heating of the mineral was responsible for the loss of fossil tracks which resulted in a younger age. If the mineral or glass was heated above a certain temperature, tracks will begin to disappear. If the temperature is increased more, the tracks will completely disappear. The process is that the ions ejected during the track formation gradually diffuse back into the track and seal it. Once the track is cut, the etchant cannot proceed past the annealed part. As the annealing process proceeds, the etchable track becomes shorter and shorter until it can no longer be seen.

Each mineral species has its own annealing characteristics. Figure 10 shows the annealing characteristics of 16 minerals and glasses. This shows the effect of one hour heating at different temperatures. Autunite will lose all its tracks in one hour at 60°C, while quartz must be heated to over 1050°C for an hour before it will lose all its tracks. Unfortunately, the uranium concentration in quartz is so low, less than a part per trillion, that it does not contain any fossil tracks. The minerals commonly dated by fission tracks are in the middle of the figure: apatite, sphene, and zircon.

Recently it has been found that the apparent annealing characteristics of a mineral may depend on the type of etchant used. Sphene is the first mineral in which this effect has been observed. Sphene is shown twice in Figure 10. One curve

shows its annealing characteristics if hydrochloric acid is used and the other the characteristics if sodium hydroxide is used. They differ by about 150<sup>o</sup>C.

The annealing properties of several minerals and how they are determined will be covered in a later session.

#### Annealing:

In fission-track dating, the term annealing is used for the partial to complete erasure of tracks (Fleischer and others, 1965b). The fading occurs when some of the ions that were displaced during formation of the track diffuse back into the track and heal some of the broken bonds. This diffusion is temperature-dependent. As annealing progresses, the average etchable length of the track gets progressively shorter until it can no longer be etched and observed in an optical microscope. The annealing process appears to slow the etching rate, and so the etchant takes longer and longer to etch out the track. After a certain amount of annealing, the track has been broken by diffusion so many times that it etches at the same rate as the crystal and therefore cannot be seen.

Annealing (Fleischer and others, 1965b) results largely from heating a sample rather than from pressure. The effect of an elevated temperature is very dependent on time. The same degree of annealing can be obtained by heating a sample to a high temperature for a short time or to a low temperature for a long time. For example, apatite will lose all of its tracks in one hour at 350<sup>o</sup>C, whereas it will take over a

million years to achieve the same effect if the sample is held at 100°C (Naeser and Faul, 1969). Annealing can prevent the dating of the primary age of a mineral, but it can be helpful in understanding the thermal history of an area. In some cases, such as that of glass, a sample showing reduced track diameters can be corrected for partial annealing (Storzer and Wagner, 1969, figure 11; Storzer and Poupeau, 1973) to give the primary cooling age. Fortunately, each mineral species has a characteristic temperature at which annealing takes place. For example, it takes one hour at 750°C to remove all the tracks in epidote (Naeser and others, 1970), whereas an hour at 350°C is sufficient to remove the tracks in apatite (Naeser and Faul, 1969). Fleischer and others (1975) have summarized the available annealing data for a number of minerals and glasses. In order to determine the annealing characteristics of a mineral, it is necessary to heat it in the laboratory at known temperatures and times and then determine the reduction of the track density (figure 12). This laboratory data must then be extrapolated to geologically meaningful times and temperatures. Figure 13 is such an extrapolation. Apatite is one of the few minerals that has been studied to determine its annealing characteristics in more than one laboratory. The results presented by Wagner (1968) and Naeser and Faul (1969) are in good agreement for apatite.

Using extrapolated annealing data such as shown in figure 13, it is possible to reconstruct the thermal history of a rock by dating various minerals present in the rock. For example, consider the mineral pair apatite and sphene. If they have the same age, it indicates that the rock has cooled very rapidly from temperatures in excess of 500°C to temperatures less than 100°C within a short period of time. If the apatite is younger than the sphene, there was either: 1) a very slow cooling of the rock, as in cooling following a regional metamorphic event, or 2) a younger thermal event that caused the apatite to lose some or all of its tracks.

Figure 14 illustrates how a younger thermal event can cause a resetting of one or both minerals. Apatite and sphene present a granitic pluton in California, intruded by a younger basalt, were dated by the fission-track technique (Calk and Naeser, 1973). Within a few meters of the contact, both the apatite and sphene in this granite were reset to the age of the intrusive basalt; at distances of a kilometer or more, the two minerals are concordant with the age of the primary intrusion. But, in between, the ages are discordant.

Until recently, annealing data for track fading in minerals were acquired under laboratory conditions. The laboratory data were extrapolated to geologically reasonable times and temperatures. Samples from two holes in the crust have recently been used to study the effect of increased temperature with depth on the K-Ar and fission-track age systems

in minerals recovered from cores (Turner and Forbes, 1976; Naeser and Forbes, 1976). One hole penetrated almost 3,000 meters of a Mesozoic metamorphic complex near Eielson Air Force Base, Alaska. The bottom-hole temperature in the Eielson, Alaska, deep test hole was 96°C. The second deep drill hole is in New Mexico, where a geothermal test hole was drilled on the western flank of the Valles caldera, a Pleistocene volcanic center. The hole is about 2,900 meters deep and the bottom-hole temperature is 197°C. The lower 2,200 meters of this hole was drilled in a Precambrian igneous and metamorphic complex. By dating the various minerals present in core recovered from these holes, it is possible to get a better understanding of how fission-track age systems behave under long-term geologic heating and how these ages compare to ages determined by other dating methods.

Twenty apatite concentrates and one zircon concentrate have been dated from the Eielson deep hole and its related exploratory holes. The fission-track ages of these minerals are shown in figure 15, along with the K-Ar trends for biotite and hornblende. The fission-track ages for the apatite range from about 100 m.y. near the surface to about 14 m.y. at the bottom of the hole. Only one zircon concentrate was obtained, and it had an age that is older than the coexisting apatite and younger than the K-Ar age of the biotite from the same depth. The apatite data show a large scatter, because these

apatites contain less than 0.5 ppm U; fossil-track counts are therefore very low, resulting in a high analytical uncertainty for the apatite ages. If the apatite data are extrapolated to a depth at which apatite would give a zero age, the temperature at that depth would be about 105°C. This is a region where Mesozoic metamorphism has been followed by a period of slow uplift. This apatite at the bottom of the hole has therefore been at a temperature of greater than 100°C for at least 100 m.y.

A second example of long-term natural annealing is shown in figures 16 and 17. Figure 16 shows the ages of apatite in the Precambrian rocks intersected in the Los Alamos Scientific Laboratories geothermal test wells 1 and 2. The apatite has a zero apparent age at a temperature of about 135°C. Figure 17 shows both the sphene and apatite fission-track age data. The sphene does not show any age reduction until temperatures are in excess of 177°C. The rocks in this hole have been heated only very recently (within a few million years). From the above two studies, geological evidence that supports the laboratory data is available of the time-temperature dependency for annealing. A slow-cooling regional metamorphic complex gives a zero age at about 105°C, whereas a recent thermal pulse, on the order of 1 m.y. ago, requires the higher temperature of 135°C for complete annealing of the apatite.

The extrapolated laboratory annealing data, shown in figure 13 for apatite, are within about 30°C of the values determined in the deep holes. Although the apatite data are in good agreement with the laboratory results, the sphene data are not. The laboratory data suggest that a temperature of about 250°C for a million years would be necessary to begin annealing in sphene. This is over 50°C higher than the temperature observed in the Los Alamos geothermal hole.

This natural data for the apparent zero age of apatite can be used to help understand the tectonic history of a region. Figure 18 shows the age of apatite on Mt. Evans (4,346 meters), just west of Denver, Colorado, as a function of elevation. Mt. Evans is made up of Precambrian igneous and metamorphic rocks. Apatite below 3,000 meters reflects the rapid uplift of the Rocky Mountains during the Laramide orogeny starting about 65 m.y. ago. The apatite above 3,000 meters was only partially annealed during the Cretaceous burial prior to the uplift.

Prior to the Laramide uplift in latest Cretaceous time there was about 3,000 meters of upper Paleozoic and Mesozoic sedimentary rocks over the Front Range of Colorado (Tweto, 1975). The following quote from Tweto (1975, p. 1) is based on the geologic record of the basins surrounding the Front Range, and is instructive as to the timing of the Laramide uplift.

"The Front, Park-Sierra Madre, and Medicine Bow Ranges on this site of the late Paleozoic Front Range Highland, rose after the marine Fox Hill Sandstone was deposited over their sites 67.5 m.y. ago. Uplift and erosion were rapid. By 66 to 65 m.y., and before the close of Cretaceous time, 3,000 meters of sedimentary rocks had been eroded from at least parts of these ranges, and streams were carrying detritus from Precambrian rock to bordering basins."

The inflection point at about 3,000 meters represents the position of the 105°C isotherm (based on the Eielson data) prior to the Laramide uplift. The present geotherm in the Front Range is 29°C/km (Roy and others, 1968). If the gradient was the same in Cretaceous time, the point which is now 3.3 km above sea level was about 3 km below sea level 66 m.y. ago. Thus the total uplift since latest Cretaceous time has been about 6 km. Wagner and Reimer (1972) have presented similar data for apatites in the Swiss Alps.

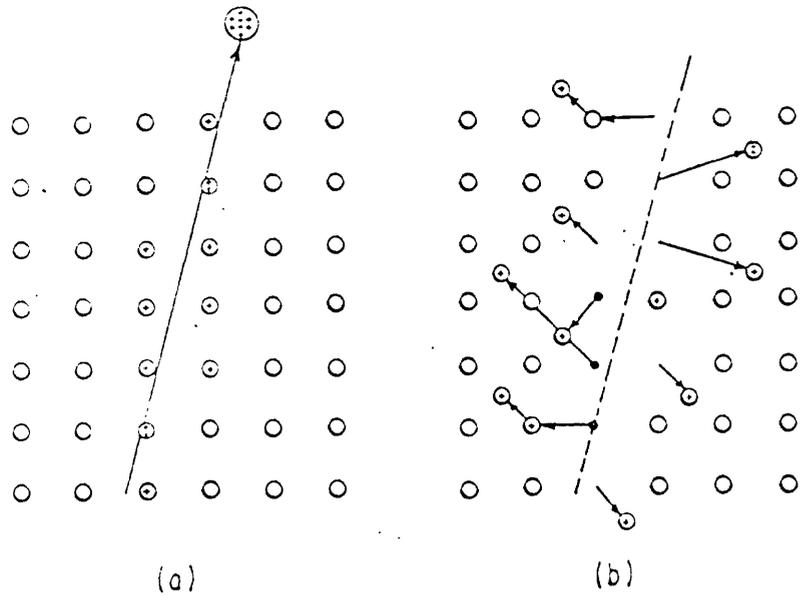


Figure 1. Track formation in a simple crystalline solid: (a) the atoms have been ionized by the massive charged particle which has just passed; (b) the mutual repulsion of the ions has separated them and forced them into the lattice (figure courtesy of R.L. Fleischer).



Figure 2. Fission tracks in tektite glass, the length of the bar is 10 micro meters in this and the following figures.



Figure 3. Fission tracks in an apatite crystal.



Figure 4. Fission tracks in a zircon crystal.

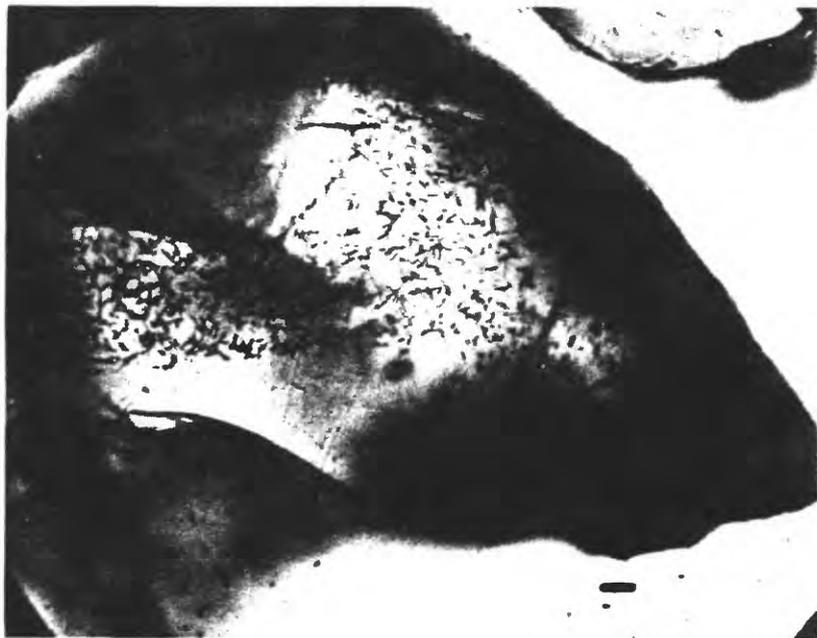


Figure 5. Sphenite grain etched for fossil fission tracks, note the zoning of the uranium within the crystal.

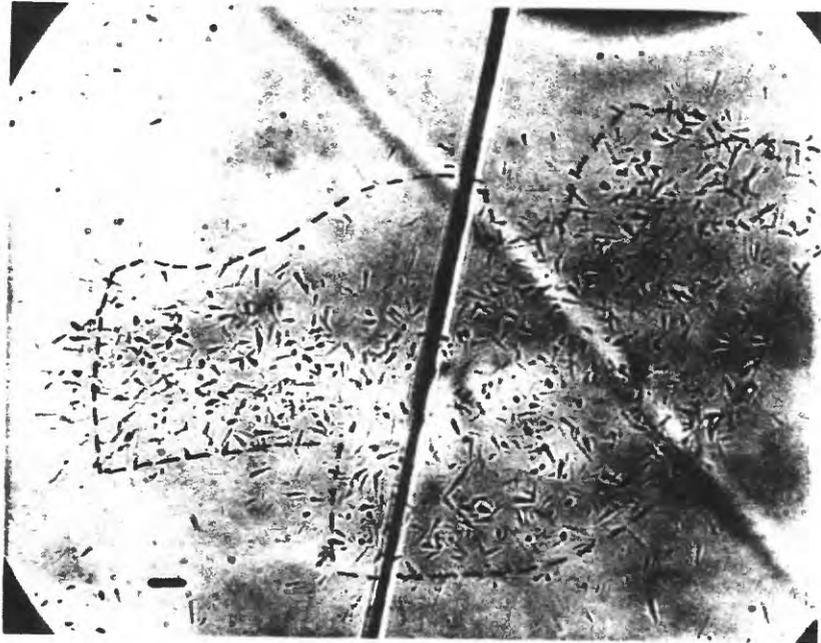


Figure 6. Induced tracks from sphene grain in figure 5 recorded in a muscovite detector. Edge of sphene grain has been outlined.

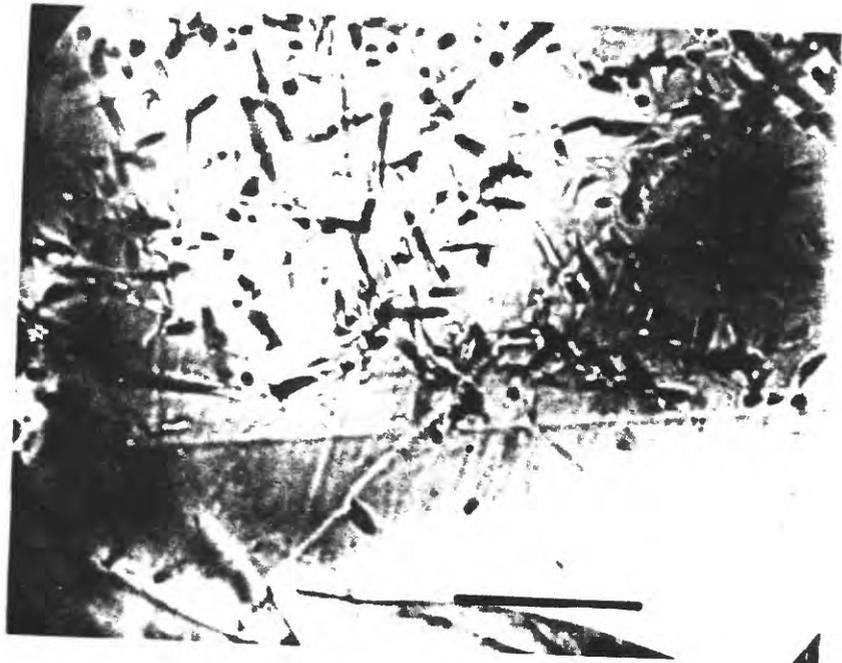


Figure 7. Higher magnification of sphene grain in figure 5, showing fossil fission tracks.

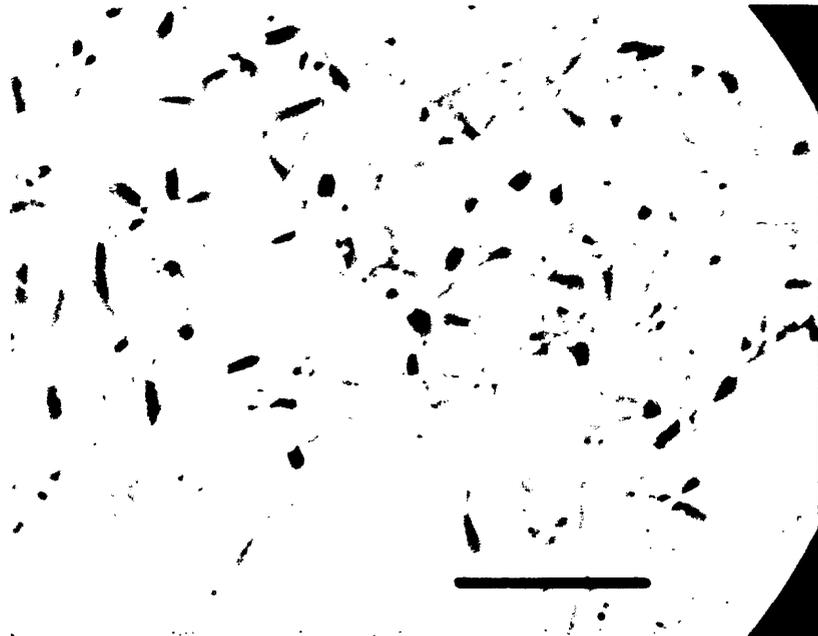


Figure 8. Higher magnification of muscovite shown in figure 6.

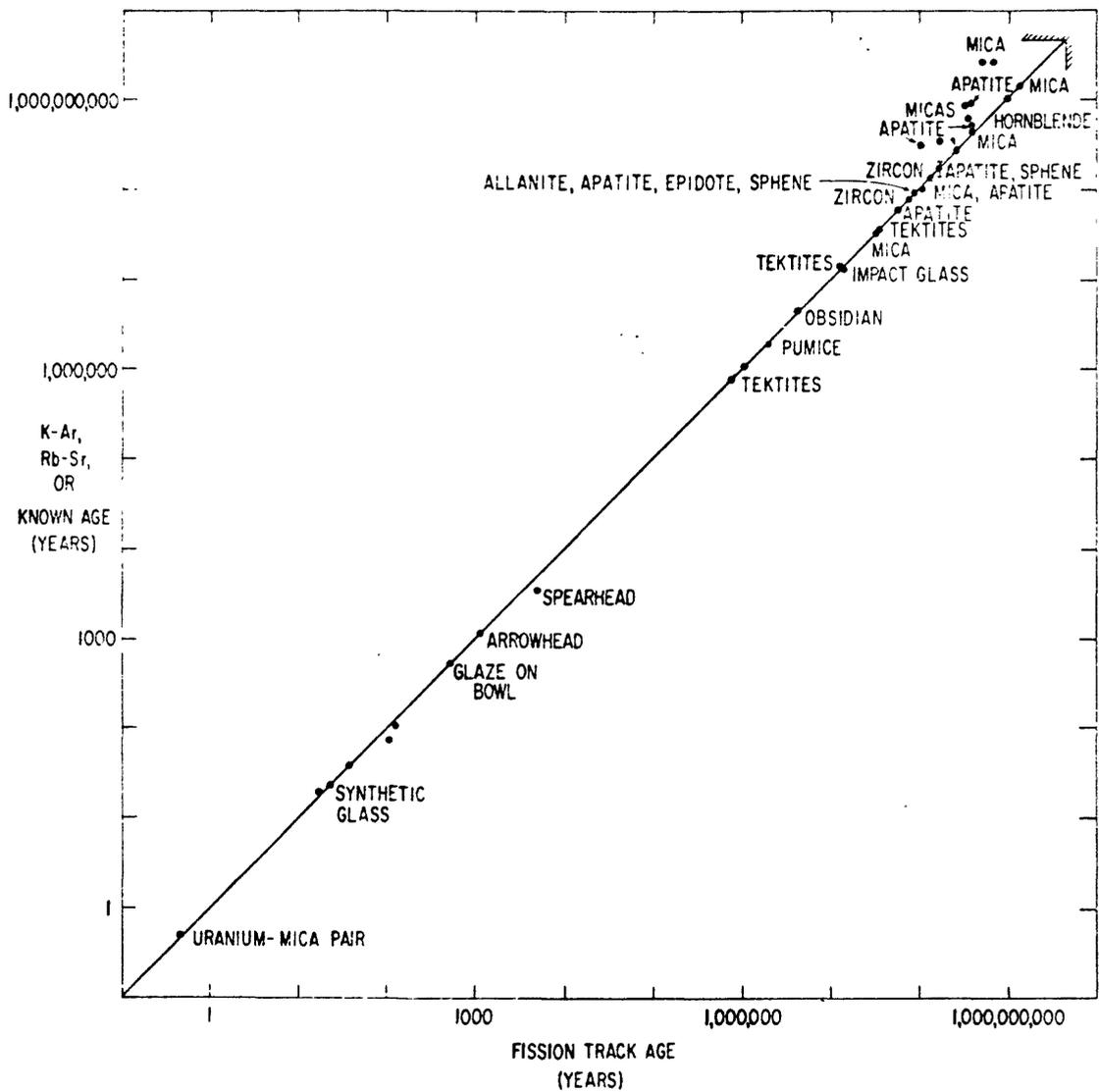


Figure 9. Plot of some fission track ages versus other ages for some object.

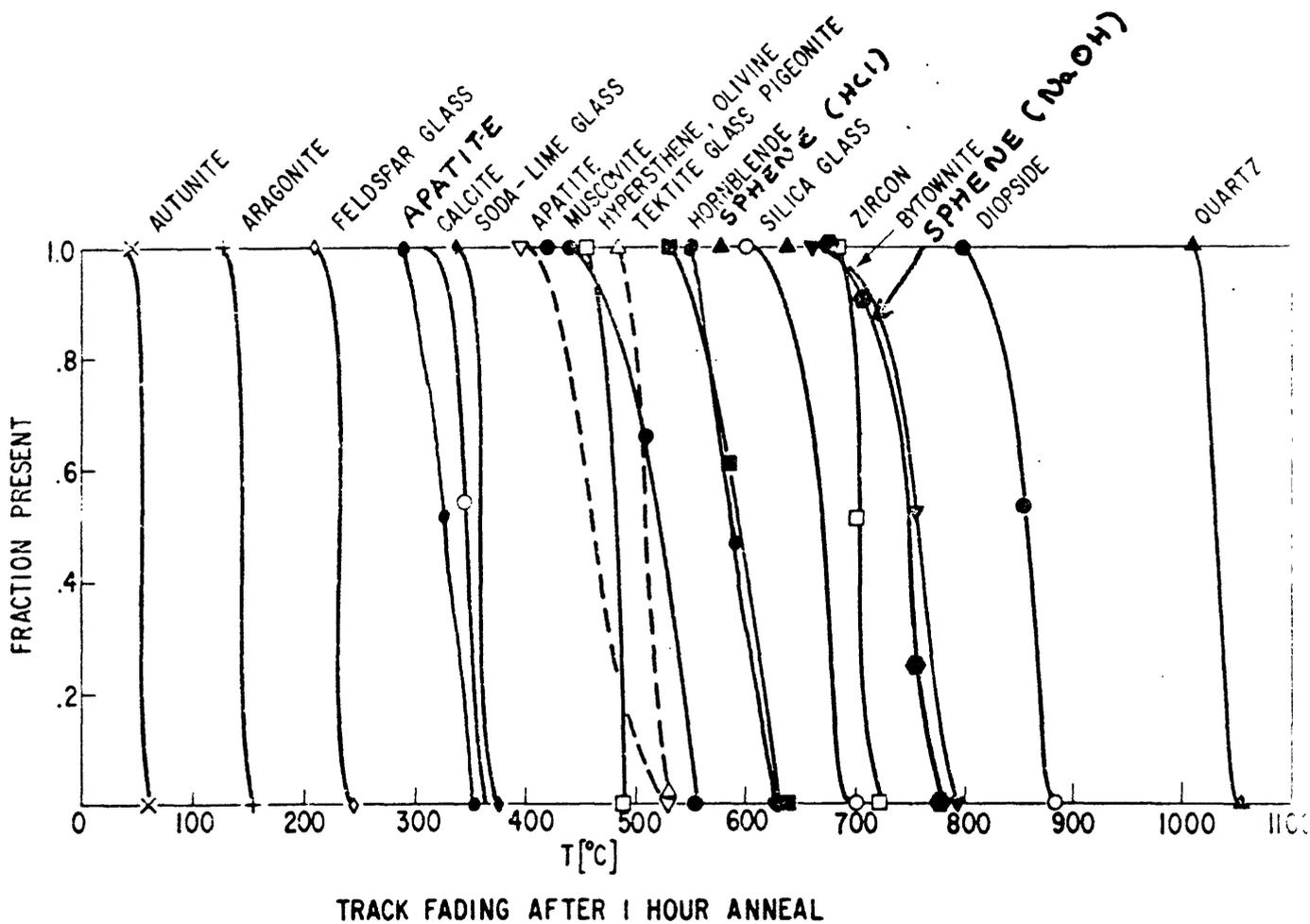


Figure 10. One hour track fading for various minerals and glasses.

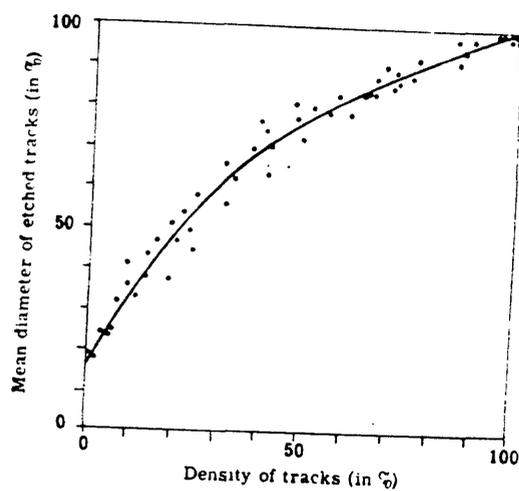


Figure 11. Diameter reduction of tracks in glass (Storzer and Wagner).

# Heating of Apatite

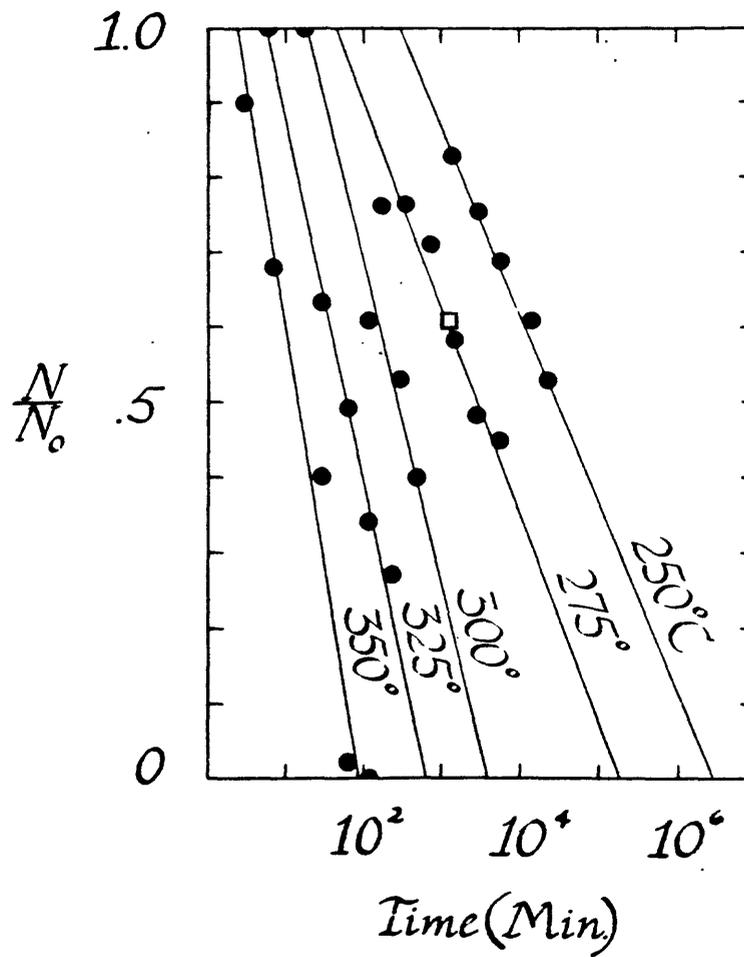


Figure 12. Annealing data for apatite.

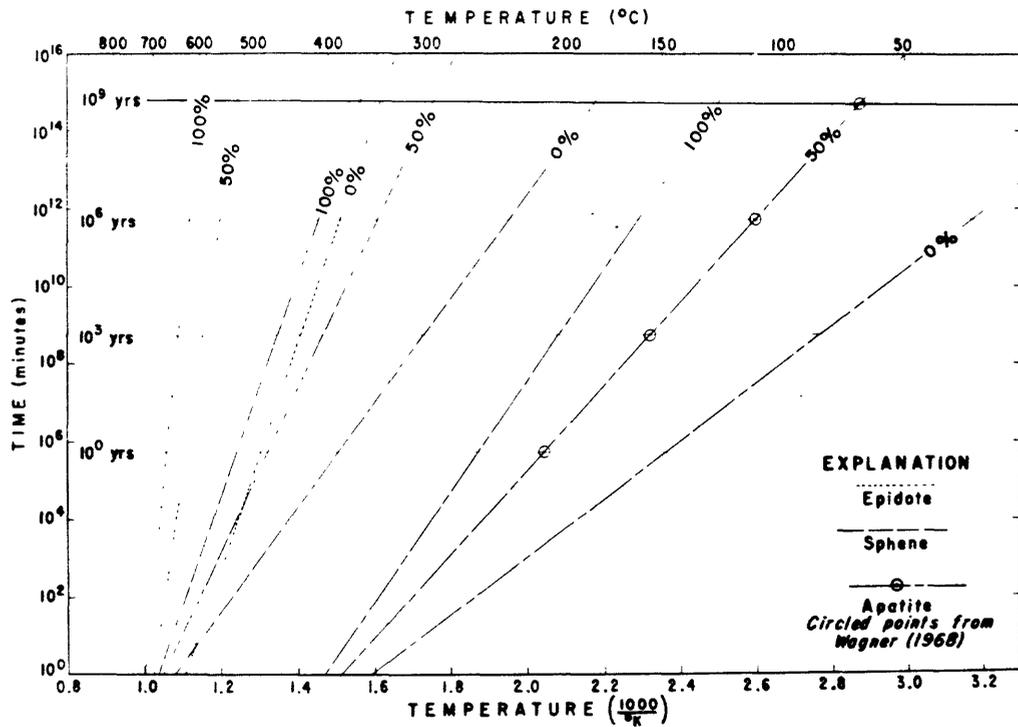


Figure 13. Extrapolated annealing data for sphene and apatite.

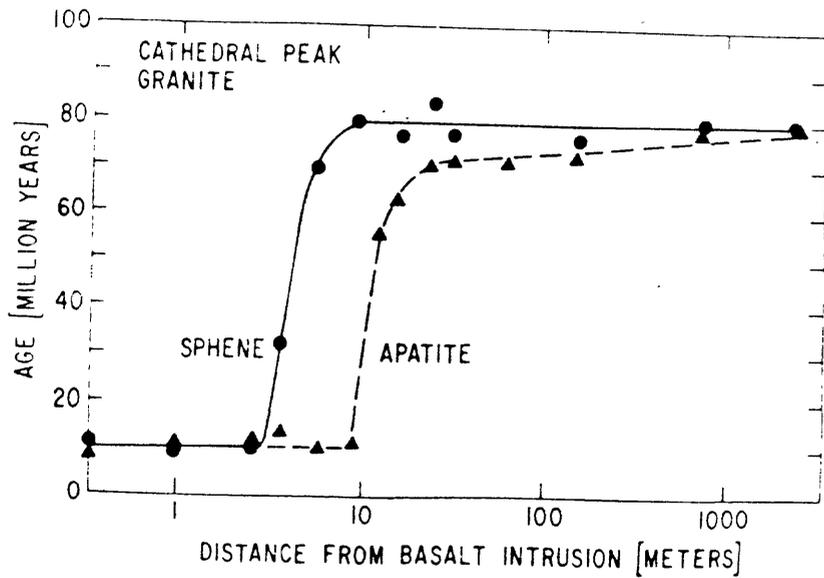


Figure 14. Fission-track ages of sphene and apatite in quartz monzoite as a function of distance from a basaltic intrusion (Calk and Naeser, 1973).

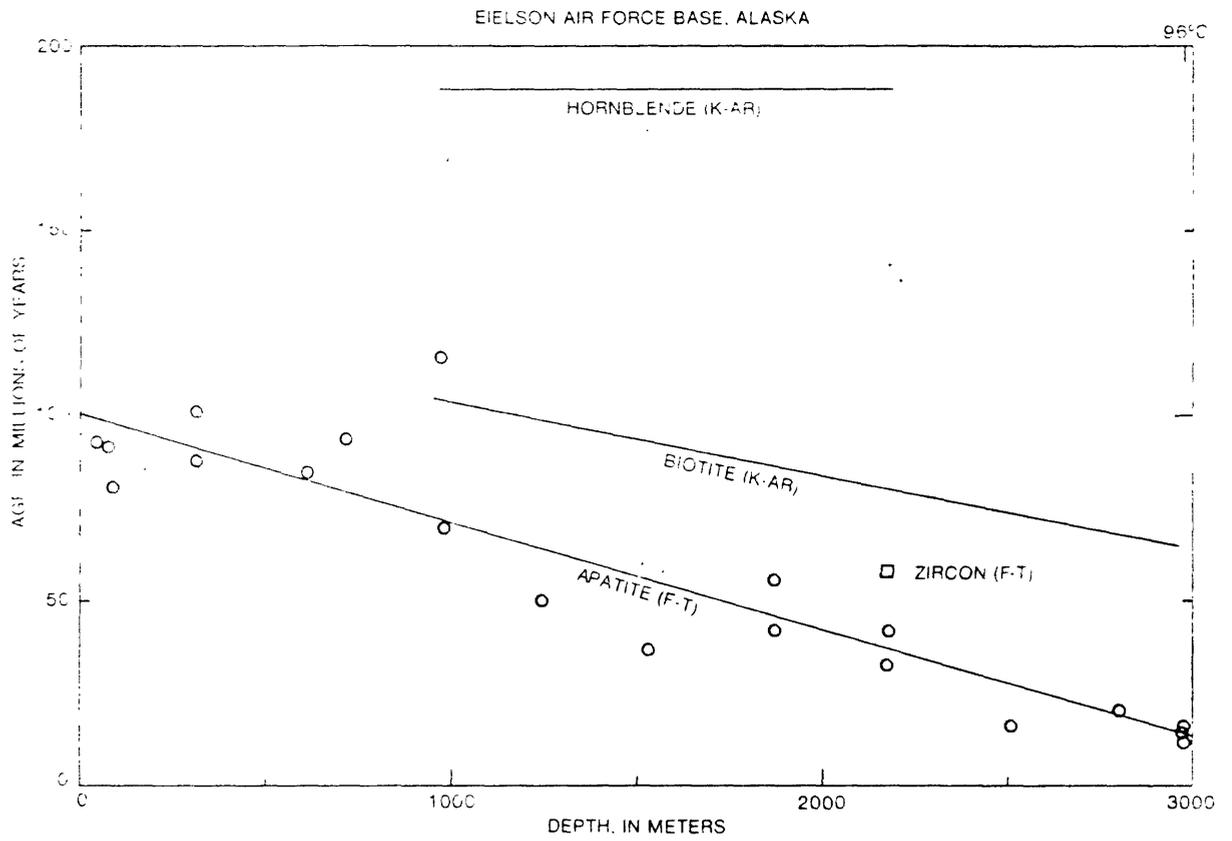


Figure 15. Fission-track ages of apatite and zircon in the Eielson Air Force Base, Alaska, deep drill hole.

Age Equation:

The fission track age equation breaks down to two parts: the production of the spontaneous fission tracks, and the production of the induced tracks. The number of fossil tracks on a square centimeter of material can be calculated from the following equation:

$$\rho_S = A \lambda_F N_V C^{238} R^{238} n^{238} \quad (1)$$

where

A = age in years

$\lambda_F$  = decay constant for spontaneous fission of  $^{238}\text{U}$

$N_V$  = number of atoms per unit volume of material

$C^{238}$  = atom fraction of  $^{238}\text{U}$

$R^{238}$  = range of the fission fragments

$n^{238}$  = etching efficiency of  $^{238}\text{U}$  track

The number of induced tracks per square centimeter can be calculated from a similar equation:

$$\rho_I = (\sigma\phi) N_V C^{235} R^{235} n^{235} \quad (2)$$

where

$\sigma$  = thermal neutron fission cross section for  $^{235}\text{U}$

$\phi$  = integrated neutron flux (neutrons/cm<sup>2</sup>) and C, R, and A are similar to same constants in the fossil equation, except are related to  $^{235}\text{U}$ .

By dividing equation 1 by 2 it is then possible to solve for the age (A):

$$A = \frac{\rho_S \sigma \phi I}{\rho_I \lambda_F} \quad (3)$$

The terms  $N_V$ ,  $R$ , and  $n$  are equal and cancel out. The term  $I$  corrects for the fact that the fossil tracks come from  $^{238}\text{U}$  and the induced tracks come from  $^{235}\text{U}$ .

Because a substantial amount of the uranium decays by  $\alpha$  emission, it is necessary to take this decay into account. This gives the following equation:

$$A = \frac{1}{\lambda_D} \ln \left[ 1 + \frac{\rho_S \lambda_D \sigma I t}{\rho \lambda F} \right] \quad (4)$$

The values for the known in this equation were listed in the previous section.

The neutron-induced fission of  $^{235}\text{U}$  is used for the uranium analysis in the age equation for several reasons. The first and most important is that by making a new set of fission tracks the terms  $R$ ,  $n$ , and  $N_V$  can cancel out in the equation. A precise value for  $R$  and  $n$  is very hard to determine. The second reason is that the uranium content of a mineral can vary from grain to grain and within a grain. It is very important that the induced tracks which are counted came from the same part of the same grain that was counted for the fossil track count. Lastly, a normal uranium analysis is usually done on a number of grains and the resulting number is an average whose relationship to the individual grain counted isn't known.

One last factor which must be taken into consideration is the geometry of the surface being counted when it received the tracks. Figure 19 shows a plot of track density various distances from an external surface in a mineral.

If the track length in the material is  $10\ \mu\text{m}$ , it will be necessary to go at least  $10\ \mu\text{m}$  into the crystal before the track density doesn't change. This internal surface at least  $10\ \mu\text{m}$  inside the crystal was able to receive the same number of tracks from both above and below. This is called  $4\pi$  geometry. The external surface received tracks from U located only below the surface, assuming there was no U in the mineral in which it was in contact. This arrangement is called  $2\pi$  geometry. Most of the time the surface being counted was in  $4\pi$  geometry at the time it received its tracks. In the case where an external detector is used for the induced track count it was irradiated in  $2\pi$  geometry. Therefore the induced count must be doubled before being placed in equation 4. If the mineral or glass isn't ground or etched down into a zone of  $4\pi$  geometry, the resulting surface was in an unknown geometry at the time of irradiation and will give an erroneous result if substituted into equation 4 without some correction.

Some fission track dating laboratories prefer to re-etch the surface after irradiation to count the induced tracks. If this is done, the new etching removes some of the surface. The resulting surface with tracks was in an unknown geometry during irradiation. It is therefore necessary to experimentally determine for the etching conditions chosen the geometric correction necessary. This geometric correction can be avoided if enough mineral is ground and polished away after irradiation to produce a new surface which was in  $4\pi$  geometry.

The neutron dose to which the sample was subjected must be determined as accurately as possible. It is not enough to rely on what the reactor operator says you were given. The neutron dose can be off by 20 percent or more.

The best way to determine the neutron dose is to place a standard in the irradiation package. The standard should have a uniform uranium concentration and should produce an easily counted track density. That is, you should be able to count about 1000 tracks with a minimum amount of effort and time. If the uranium concentration is too low, it might be necessary to scan several square centimeters to get enough tracks. The standards are counted at 400-500 magnification. At this magnification there are about 4000 fields of view per square centimeter. On the other hand, too much uranium in the standard could produce too many tracks which can also make counting difficult. A standard containing between 0.3 and 30 ppm U will be useful as a monitor for most geologic material.

Glass standards suitable for neutron dose measurements are available from the U. S. National Bureau of Standards. There are four glasses available, and they contain the following uranium concentrations: 461 ppm U, 37.4 ppm U, 0.82 ppm U, and 0.072 ppm U. The two intermediate glasses make ideal standards for use when dating geologic materials. The standards must be calibrated in order to give an accurate determination of the neutron dose. There are two ways to calibrate a standard:

- (1) Run a series of minerals of known age and calculate the flux by solving for  $\phi$  in equation 4. Once the neutron dose is known, it can be related to the track density in the glass or muscovite detector covering the glass.

(2) This method is to compare the track density produced by your standard with the track density in another piece of the same glass which had been irradiated under known conditions. This method is far better than the previous method. The neutron dose your standard received can be calculated as follows:

$$\Phi_U = \frac{\rho_U}{\rho_K} \cdot \Phi_K$$

where

$\Phi_U$  = unknown neutron dose

$\Phi_K$  = known neutron dose

$\rho_U$  = track density in unknown

$\rho_K$  = track density in known

The U. S. National Bureau of Standards has for sale the glasses discussed earlier, which have been irradiated under known conditions. With each concentration, two irradiated and four unirradiated pieces of glass are sent.

The track density in glass is hard to determine with good reproducibility. The etching time and temperature are very critical. Because of this problem, it is better not to rely on counting tracks in glass after the initial calibration, unless both the known and unknown are always counted together. This includes repolishing and etching of the known every time. It is better to place a piece of low-uranium muscovite on top of the glass and etch it and count it each time. The tracks in the muscovite are easier to etch and count than tracks in the glass.

The muscovite detector must then be calibrated too. If it is placed on the glass in the original calibration runs, it can be calibrated at the same time. The equation for this is as follows:

$$f = \frac{\phi}{\rho_m} \quad (6)$$

where

$\phi$  = neutron dose (neutrons/cm<sup>2</sup>)

$\rho_n$  = induced track density in muscovite (tracks/cm<sup>2</sup>)

f = muscovite calibration factor

Once "f" has been determined, the neutron dose can be calculated without having to count the glass as follows:

$$\phi_v = f \cdot \rho_m \quad (7)$$

The glass standards can be used many times without using up the uranium in the glass. For a neutron dose of  $10^{15}$  neutrons/cm<sup>2</sup> about  $6 \times 10^{-7}$  of the uranium present is used up.

Statistics:

The basic assumption in fission-track statistics is that the fission-track count is part of a  $\rho$  Poisson distribution. In Poissonian statistics the mean is equal to the variance and if the mean of a number of determinations is made the theoretical distribution can be calculated from the binomial theorem:

$$f_a(n) = \frac{a^n e^{-a}}{n!} \quad (1)$$

where  $n$  = integer  
 $a$  = mean  
 $f_a(n)$  = probability of  $n$  occurring

Also the standard deviation is equal to the square route of the mean.

There are two methods used to calculate the error associated with a fission track age. One procedure is used if the age was determined by the external detector method, and another is used if the population method is used.

The statistics used in calculating the error of an age determined by the external detector method are as follows. This procedure was developed by V.R. McGee and N.M. Johnson of Dartmouth College. In this method the

fossil count is made on a crystal, or part, and the induced count is counted on a piece of muscovite which covered the crystal during the irradiation. Both counts are dependent upon the uranium concentration of the crystal, and are therefore dependent variables. Since they are dependent variables linear statistics can be applied. The calculation of the error can be broken down into four parts. Four or more grains should be dated from the sample.

1. Determine the counting error by combining counts for all grains.

$$\sigma_c = \sqrt{\alpha^2 + \beta^2} \quad (2)$$

where

$$\alpha = \sqrt{\frac{1}{\Sigma f}}$$

$\Sigma f$  = total number fossil tracks

$$\beta = \sqrt{\frac{1}{\Sigma i}}$$

$\Sigma i$  = total number induced tracks

The error on a single grain can be calculated with this formula.

2. Calculate the correlation coefficient for all the pairs of fossil and induced tracks.
3. Using figure 20 determine the correction for  $\sigma_c$  (equation 2). For example, if  $r$  ( $\sigma$  correlation coef) is equal to 0.9 the correction for  $\sigma_c$  is 0.3.

$$\sigma_c \times 0.3 = \sigma_r$$

4.  $\sigma_r$  must now be combined with the counting error in the neutron dose determination as follows:

$$\sigma_a = \sqrt{(\sigma_r)^2 + (\sigma_g)^2}$$

$$\text{where } \sigma_g = \frac{1}{\sqrt{g}}$$

$g$  = number of tracks counted in standard

$\sigma_\alpha$  = standard deviation of age

This method does not take into account any errors in geometry, etching, or track identification.

As of the present there is not an entirely satisfactory method for calculating the age and error of a mineral determined by the population method. This is because the population method assumes that the two sub sets counted have the same uranium concentration. This is not always true. In most cases the age is calculated from the mean of each count. The uranium in an apatite concentrate (usually dated by population) is variable. This variation can be as much as an order of magnitude or greater. Repeated analyses of apatite show that the spread in ages is usually larger than Poissonian statistics would permit. It is better to calculate a conventional standard deviation for each count.

$$\sigma = \left\{ \frac{\sum x^2 - \frac{(\sum x)^2}{N}}{N-1} \right\}^{1/2}$$

This reflects only the spread in the data points. In order to get the standard error of the mean it is necessary to divide the standard deviation by the square route of the number of grains counted:

$$\bar{\sigma} = \frac{\sigma}{\sqrt{n}}$$

This calculation can be made for both the fossil and induced counts and the results combined:

$$\sigma = \sqrt{(\bar{\sigma}_f)^2 + (\bar{\sigma}_i)^2}$$

This will give the uncertainty of the apatite age.

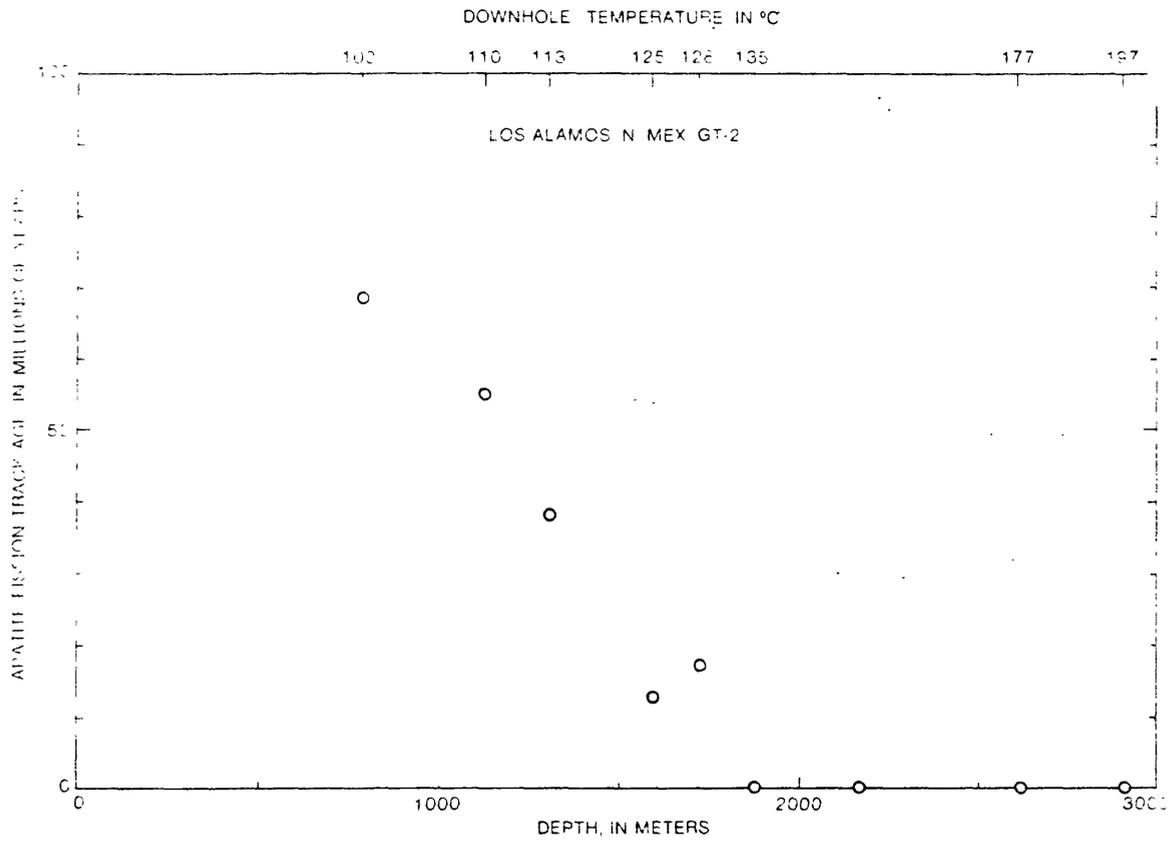


Figure 16. Fission-track ages of apatite in the Los Alamos, New Mexico, deep drill holes GT-1, GT-2.



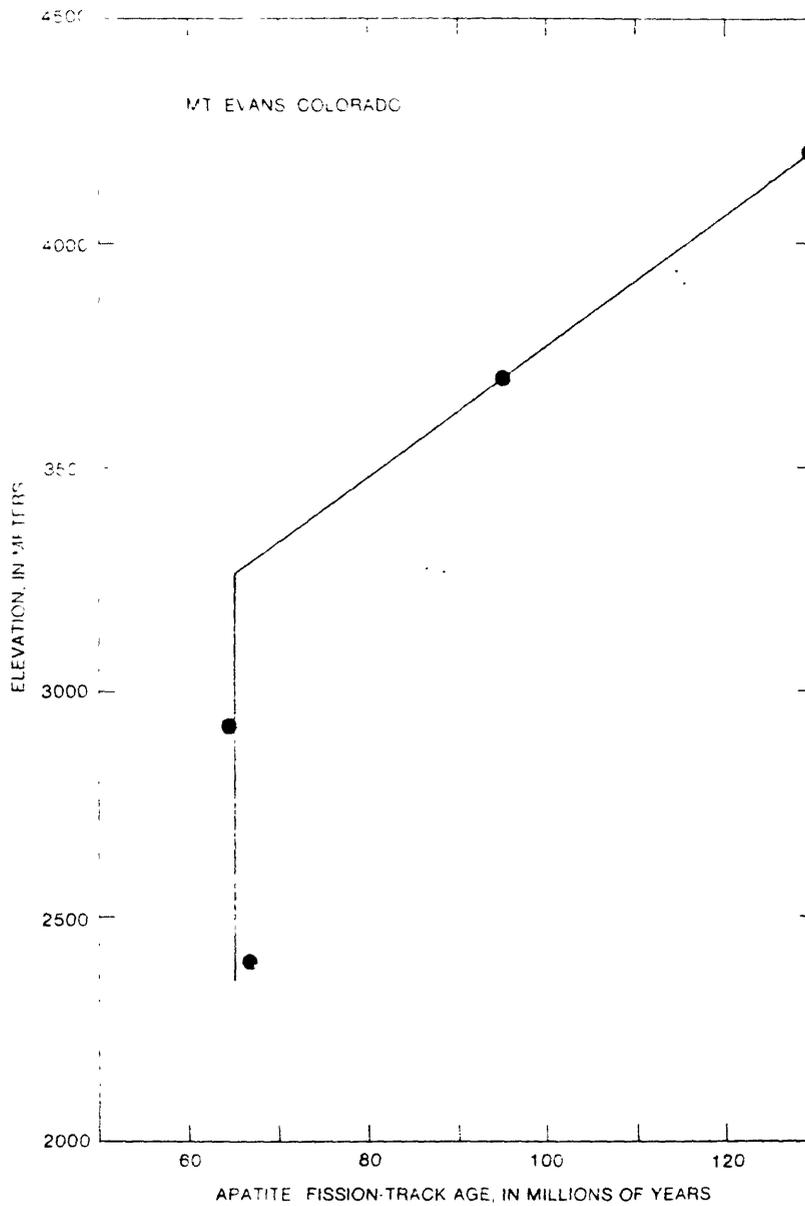


Figure 18. Fission track ages of apatite in the Precambrian rocks exposed on Mt. Evans, Colorado.

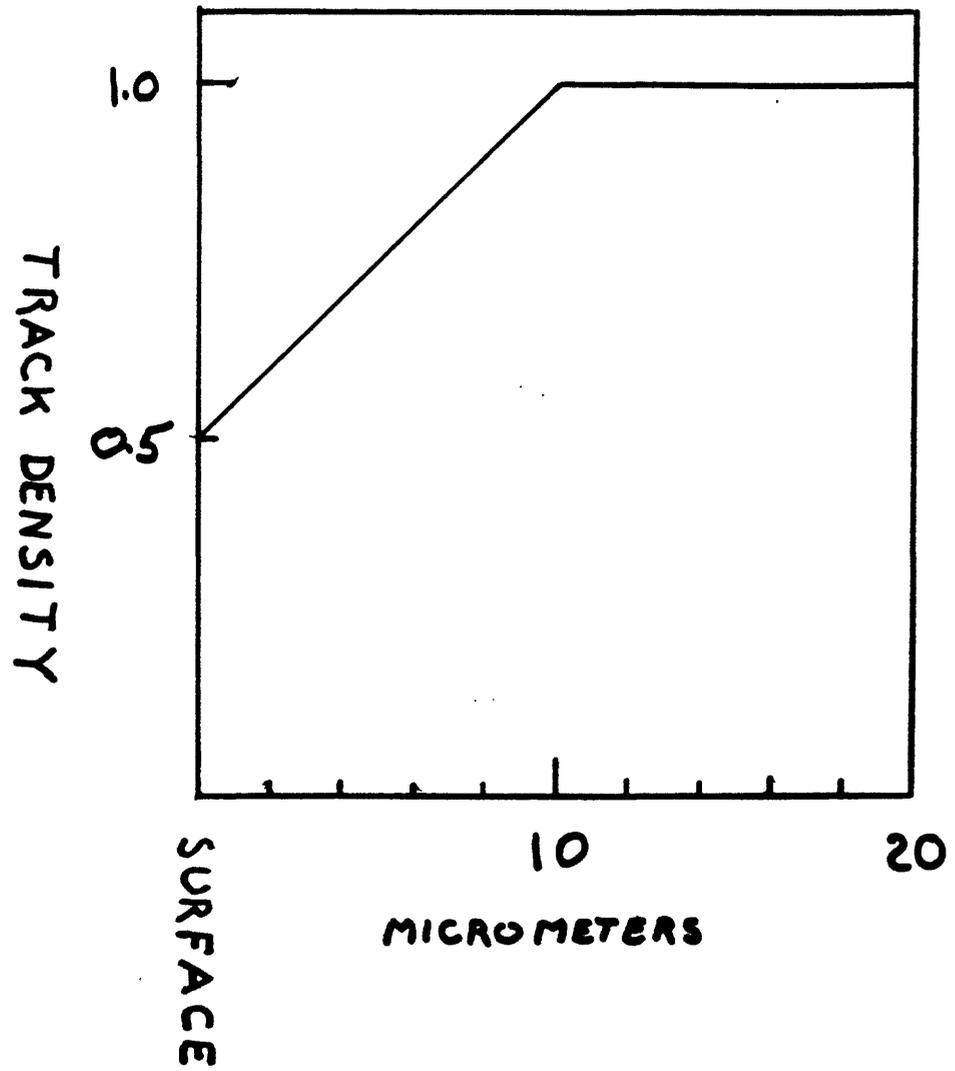


Figure 19. Track density versus depth in grain or glass.

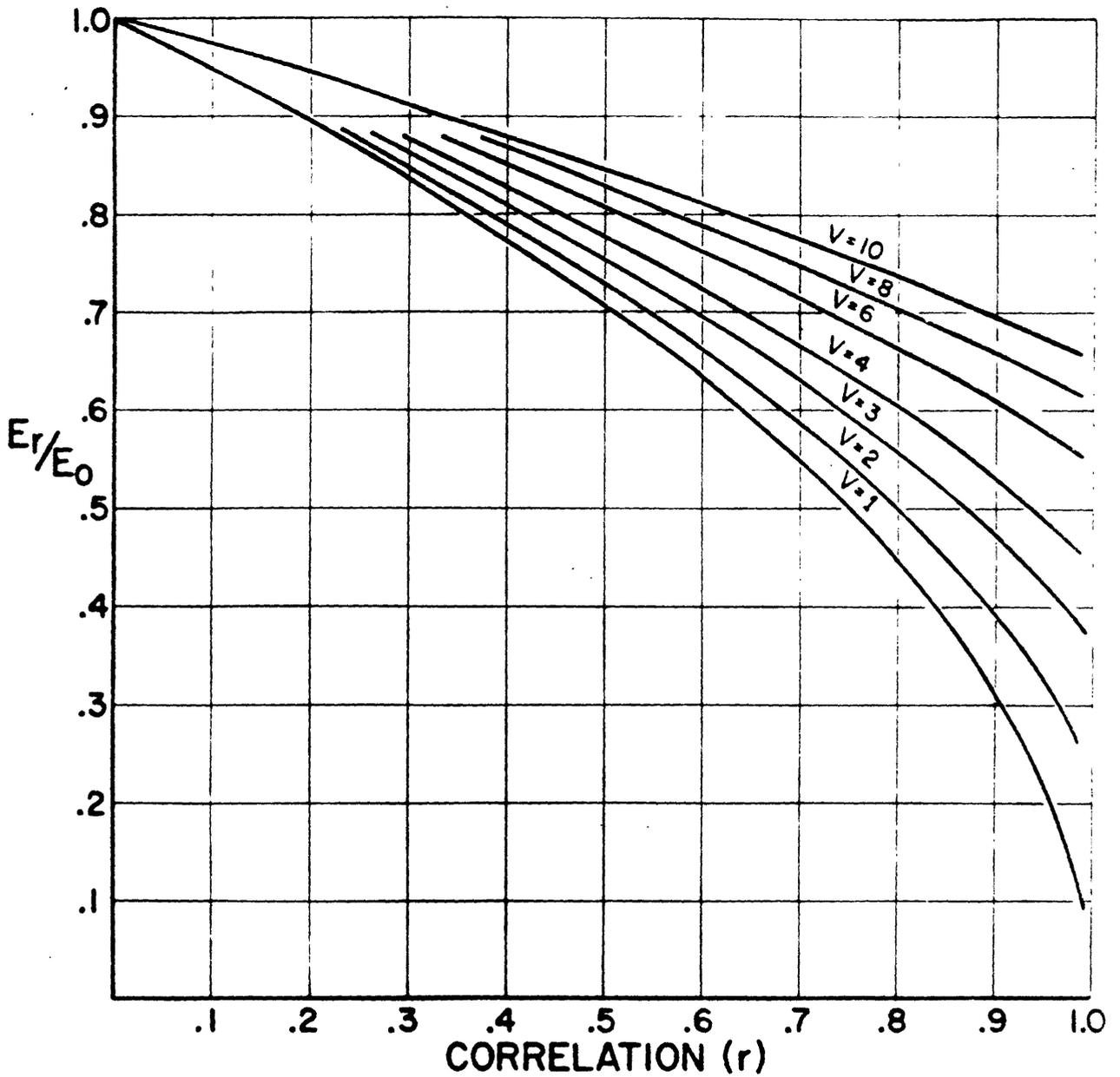


Figure 20. "Magic Ratio Graph", used for correcting basic counting statistics for external detection method of dating. V is fossil counts/induced counts or induced counts/fossil counts, whichever is greater.

PART II

LABORATORY PROCEDURES

## Mounting Minerals in Epoxy

### Equipment needed

1. Hot plate
2. Plastic block or sheet (Teflon is best) or a teflon coated surface
3. Box of microscope slides
4. A small spatula
5. Epoxy resin and hardner in graduated syringes
6. Something to mix the epoxy in a small evaporating dish or watch glass (Teflon)
7. Diamond scribe
8. Scotch magic tape or another tape which can be written on
9. Thin strips of plastic (about 0.2 mm thick)
10. Small square of aluminum foil 3 cm x 3 cm

### Procedure

1. Set hot plate to low (about 100°C)
2. Place dish or watch glass for mixing epoxy on the hot plate
3. Clean surface of mounting block
4. Put a strip of tape down along the edge of the block (see figure 1).
5. Write sample numbers on tape; about 3 cm apart
6. With diamond scribe write sample numbers on glass slides, and then clean its slides to remove finger prints and oils.
7. Pour several hundred grains from the sample bottle onto the mounting block. They should form a small circle (about 1 cm in diameter), and the grains should form a layer 1 grain thick. Do this with each sample to be mounted.
8. If apatite is being done, by the population method, now is the time to prepare the split to be irradiated. Number a square of aluminum foil (figure 2). Start with 1 and go up. In your record book note which apatite is being given number 1,2,3,..... and so on. When pouring the apatite grains on the mounting block, pour a similar amount in the center of the numbered aluminum square. Fold the aluminum square to form an envelope. This envelope is then put in an oven at 500°C for 3 or more hours.
9. Spacers should now be placed along 2 edges of the row of grains. These are to insure that the grain mounts are of a uniform thickness and paralleled to the microscope slide used as backing of the grains are to be etched and irradiated in the epoxy mount (sphene, epidote, garnet, etc.) use a thick spacer (about 1 mm thick). A glass microscope slide is about right. If the grains are to be etched only a thinner spacer can be used (thinner mounts save epoxy, and are easier to polish).

10. If the epoxy is hard to mix cold, and is very viscous it is best to mix it on a hot plate. Squeeze the proper amounts of each part out onto the watch glass (which is on the hot plate). Stir the epoxy until it is well mixed, clear, and nearly free of bubbles.
11. Pour several drops of epoxy on each group of grains. If thick mounts are being made, use 6 to 8 drops. If the mounts are to be thin, use 4 to 6 drops.
12. Take the numbered slides and place them over the epoxy and press down until it touches the spacers at each end.
13. If the epoxy has turned cloudy, place the mounting block under a heat lamp for about 15 minutes. The clouding of the epoxy is because it was not mixed well enough.
14. Allow the epoxy to cure. Most epoxies require about 18 hours at 25°C to be hard enough for polishing. The curing can be speeded up by heating the block under a heat lamp. It is best to wait until the epoxy is stiff before heating. If it's not, a lot of small bubbles will form in the epoxy at the surface where it is in contact with the teflon mounting block.
15. After the epoxy is hard, the slide mounts can be taken off by flexing the mounting block, or by using a knife to gently pry them off.

## Polishing Mounts for Fission-track Dating

### Equipment:

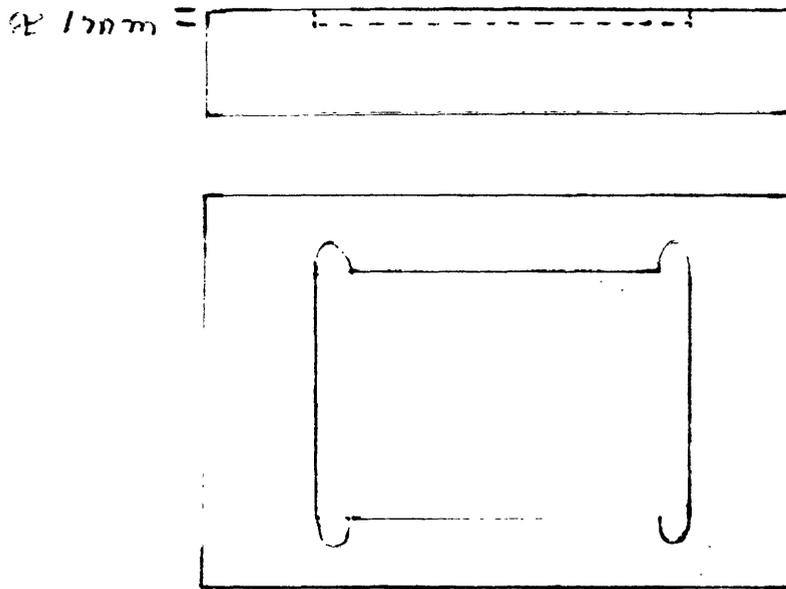
1. #400 silicon carbide abrasive paper with adhesive backing.
2. #600 silicon carbide abrasive paper with adhesive backing.
3. 6 micron diamond polishing compound.
4. 1 micron diamond polishing compound.
5. Extender fluid for diamond polishing compounds.
6. One piece of plate glass 30x30 cm, or Handimet grinder.
7. Polishing machine with at least two lap wheels.
8. Small plastic block milled out to hold a microscope slide (Figure 3).
9. Micro cloth disks with adhesive backing.

### Procedure

1. Place a slide with epoxy wafer attached in the slide holder. A drop of water on the back of the slide will hold it in place.
2. Place 400 and 600 silicon carbide papers on handimet grinder or glass plate. If possible use a flow of water to flush the surface during grinding.
3. Place slide holder on #400 paper and grind. Push away from yourself. After each pass rotate the slide holder 180°. This will help prevent the wafer from becoming wedge shaped. Grind only until the grains are exposed. It doesn't take too much grinding at this step and the next one to remove all the grains. Five or six passes are usually enough. About ten samples can be ground before it is necessary to replace the abrasive paper.
4. Now proceed to the #600 silicon carbide paper. Before grinding here rotate the holder 90°. The scratches from this step should be perpendicular to those from the first step. Grind until the scratches from the 400 paper are gone. Be sure to rotate holder 180° before each pass on the paper.
5. Place a piece of microcloth on a lap wheel and put it on the polishing machine.
6. Put a small amount of diamond on the microcloth and a bead of diamond compound about 1 cm long is enough. Add some extender fluid and smear the diamond out on the cloth.
7. Hold the sample holder so that the diamonds will pass under the epoxy perpendicular to the scratches from the previous step. Using a moderate amount of pressure polish the sample until the scratches from the 600 silicon carbide are gone. After this a round and round movement of the holder is possible if desired.

8. Put a piece of micro cloth on the second lap wheel, and add 1 micron diamond compound just as was done in the previous step.
9. Proceed with the polishing, making sure that the scratches from this step are going only in one direction. This is done by keeping the holder on a radius and moving it in and out along that radius. Do not go around and around on the lap. The fission tracks are easier to observe if the polishing scratches are going in one direction rather than crossing.
10. After proper polishing the minerals grains should be fairly flat and smooth. They should look very bright if light is reflected off of them.
11. It is not necessary to replace the cloth very often. It can be used many times. The lap wheels with the cloth attached should be stored in a clean area after polishing.

Figure 3  
Polishing Block  
milled from Lucite



The depression should be milled out so that the microscope slide will just fit inside. Specific dimensions are not given, because the size of the petrographic slides changes from vendor to vendor.

## Etching

With the exception of apatite, all mineral and glass mounts should be removed from the glass slide after polishing and before etching.

1. Place a drop of water on the glass slide at the edge of the epoxy wafer.
2. Using a sharp knife, gently work the edge of the blade under the epoxy wafer at the drop of water. When the wafer has started to separate from the glass, carefully work the blade under until the wafer comes free. The water helps to prevent the wafer from breaking.
3. Save the numbered glass slide; it will be used again after irradiation.
4. Put the sample number on the back of the wafer.

### Apatite:

Solution: 7%  $\text{HNO}_3$  1)  
Temp. 25°C (or room temperature)  
Time: 20-40 seconds

The etching time will vary from sample to sample. It is necessary to etch the sample long enough to insure complete track development. This is when no faint tracks can be seen.

- 1) Concentrated  $\text{HNO}_3$  is 70%  
use 1 part  $\text{HNO}_3$  to 9 parts  $\text{H}_2\text{O}$

### Sphene

Solution: 50 M NaOH  
(40 gm NaOH; 20 gm  $\text{H}_2\text{O}$ )  
Temp. 130°C  
Time: 10-60 minutes  
Vessel: Stainless Steel Beaker 100-150 ml.

The etching time for sphene is dependent upon the track density. Sphene with a very high fossil track density ( $10^6$  to  $10^7$ ) will etch much faster than those with lower track densities. The mount will float, so be sure the mount is placed in the solution grain side down.

### Garnet:

Solution: Same as sphene  
Temp: " " "  
Time: Usually greater than 1 hour  
Vessel: Stainless steel

Garnet is highly variable in uranium content. Some garnets have enough uranium for dating, but most do not.

Epidote:

Solution: Same as sphene  
Temp. " " "  
Time: Usually greater than 1 hour  
Vessel: Stainless steel

Epidote like garnet is very variable in uranium content. The probability of being able to date an epidote concentrate is low.

Most other silicates:

The sodium hydroxide solution used for sphene is the best one to try if an exact etching solution has not been described for the mineral you wish to etch.

Zircon

See attached instructions

Micas

Solution: 48% HF  
Temp. 25°C (or room temperature)  
Time: 5 seconds to 15 minutes  
Vessel: Plastic cup or beaker

Tracks in biotite may be etched in as short a time as 5 seconds, while muscovite may take as long as 15 minutes.

After etching a mica it is necessary to heat the mica on a hot plate (about 100 to 150°C). This will drive off any HF which is absorbed between the cleavage planes. Otherwise it is possible that you will etch the microscope objective.

Natural Glass

Solution: HF either 48% or 24%  
Temp. Room temperatures or 25°C  
Time: 5 seconds to 1 minute  
Vessel: Plastic cup or beaker

The etching conditions for glasses are very dependent upon the chemistry of the glass. It is usually necessary to make a few trial runs of the glass before it is etched for dating. It is also best if the mounts for the fossil and induced tracks are etched at the same time. This can be done by holding the two mounts back to back and then holding them in the solution. After etching let warm water run over the mounts for 5 minutes this will remove most HF, and prevent etching of the microscope objective.

### Mounting zircons in FEP teflon

1. Heat a metal block on a hot plate to about 310°C. Also have a glass slide heating on the hot plate.
2. On a second, cold, glass slide pour out about 100 zircons in a small area--approximately 1 cm<sup>2</sup> or less.
3. Place this slide with the zircons on the hot metal block and allow it to come to temperature.
4. Take two small pieces of 10 mil FEP teflon and place them together so that the concave sides face each other.
4. *Use one piece of 20 mil FEP teflon*
5. Place the pieces of teflon on top of the zircon grains.
6. Take the first glass slide and put on top of the teflon.
7. Using a roller of some kind, press the top of the slide down on the teflon and the zircons. The teflon will become water clear when it starts to melt. Continue to press or roll for a few seconds after the teflon clears.
8. Remove both glass slides from the hot plate and allow the entire package to cool to room temperature.
9. When it is cool remove the teflon wafer and scribe in the sample number on the backside away from zircons. Otherwise the number may be lost during grinding and polishing.
10. Proceed with your normal polishing procedure.

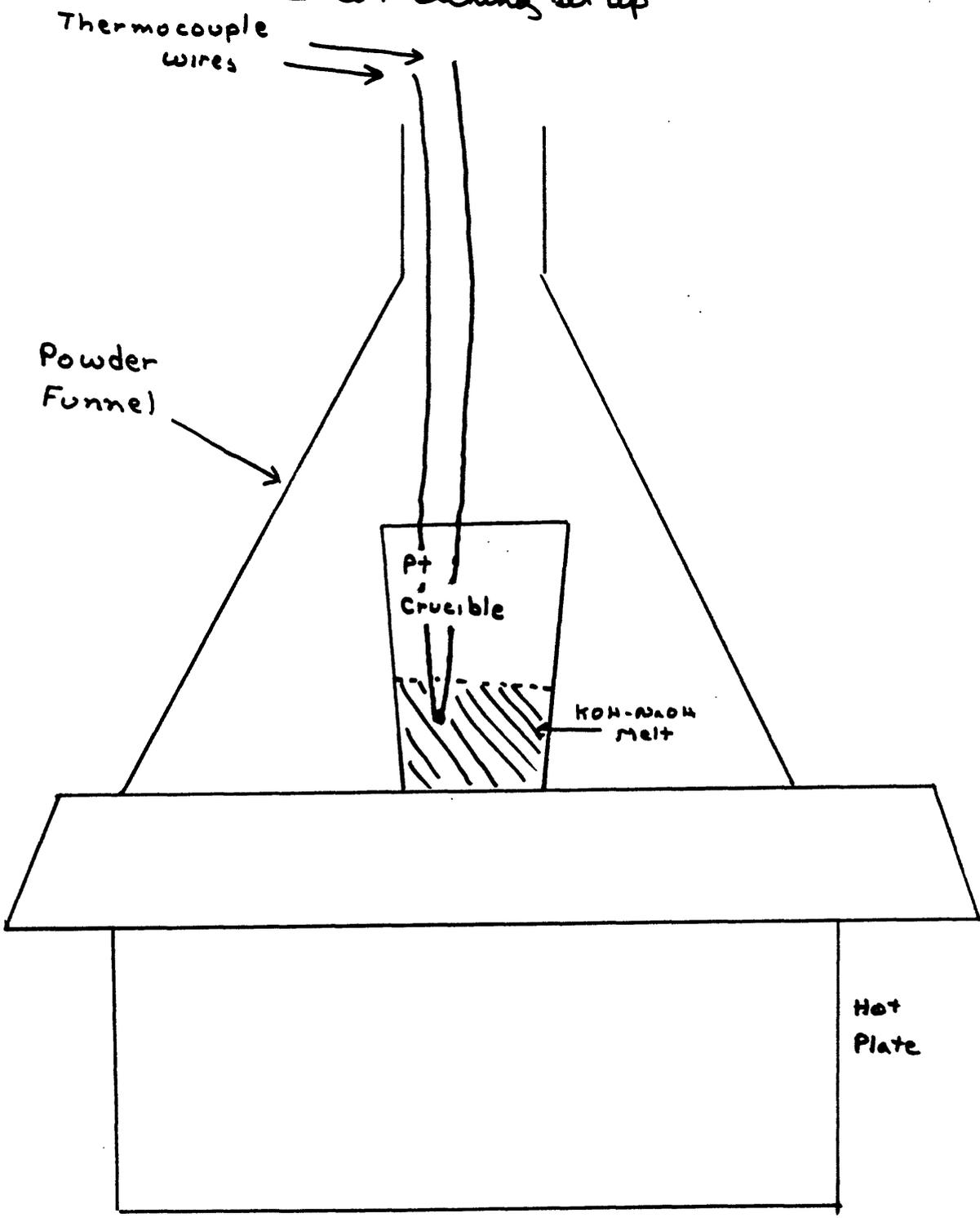
## ETCHING OR ZIRCONS MOUNTED IN FEP TEFLON

1. See figure 1 for experimental set up; or use a well controlled oven  $\pm 2^{\circ}\text{C}$ .
2. Place 7.2g NaOH pellets and 10g KOH pellets (Gleadow and others, 1976) in platinum crucible or evaporating dish.
3. Place dish in oven or on hot plate and heat to  $230^{\circ}\text{C}$  (it may be necessary to cover dish to prevent crust from forming around the top of the dish).
4. When the flux is at temperature place the polished teflon wafers in with the zircon side down. The wafers will float.
5. The etching time will vary depending upon the track density. Those with a high track density ( $>10^7\text{t}/\text{cm}^2$ ) will etch in 4-6 hours. Etching times of 18-24 hours are required for most zircons of average track density ( $10^6$ - $10^7\text{t}/\text{cm}^2$ ). Zircons with  $<10^6\text{t}/\text{cm}^2$  will require much longer times. When in doubt check etching progress after the first four hours.
6. After a satisfactory etch is obtained cover the mount with muscovite and irradiate.

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Gleadow, A.J.W., Hurford, A.J., and Quaife, R.D., 1976, Fission track dating of zircon; improved etching technique: Earth and Planetary Science Letters, v. 33, no. 2, p. 273-276.

Fig 1.  
Zircon Etching set up



### Etching Zircons Mounted in Epoxy

- (1) Mount a minimum of 100 grains in epoxy.
  - (2) Polish this mount as you would any mineral mount. This is to expose an interior surface ( $4\pi$ ).
  - (3) Place epoxy mount in hot etching solution. I use 11.2 g KOH, 8 g NaOH in a covered 50-ml platinum crucible. The etch solution should be at a temperature of 250°C. The standard zircons take about 6 hours to etch in this solution.
  - (4) Remove crucible from hot plate and allow to cool (this is very important for safety).
  - (5) Fill a 600-ml beaker about half full with warm water, place the cool crucible in it, and let the NaOH dissolve.
  - (6) After the NaOH-KOH has dissolved, wash the crucible out with a wash bottle (filled with alcohol) so that any zircons left in the crucible will go into the beaker.
- (Now comes the black magic.)
- (7) Stir the water in the beaker such that a vortex forms in the center.
  - (8) Allow the vortex to die away. The zircons should now be in a small pile on the bottom in the center of the beaker.
  - (9) With an eye dropper, pick up the zircons and transfer them to a small test tube.
  - (10) Repeat 7, 8, and 9 several times until there are no more zircons left in the beaker.
  - (11) Wash the zircons in the test tube with ethanol at least 4 times. This should remove the NaOH solution.
  - (12) For this step I have an aluminum block (2.5 x 3 x 10 cm) on a hot plate. I have a hole drilled into the center of the block, along the long axis. Into this hole a thermometer is placed. Heat the block to about 190°C. Place a glass microscope slide on the hot plate next to the block.

- (13) With another eye dropper, transfer the zircons in the test tube to a second (cool) glass slide. Spread them out to a single layer, and allow the ethanol to evaporate. There should be no film present after evaporation if the NaOH was completely washed out.
- (14) Place the slide with the zircons on top of the aluminum block, and give it time to heat up.
- (15) Take a small square of lexan <sup>2c</sup> ~~10~~ mil thick and about 1-2 cm on a side. Place this on top of the zircons.
- (16) Now take the other glass slide (hot) and place it on top of the lexan and press down. This will embed the zircons in the soft lexan. Take the slides off the hot plate and allow them to cool.
- (17) The lexan containing the zircons will come off the glass slides. Now look at the zircons in the microscope to see if tracks have been etched. There will be both polished surfaces and natural faces present. Check for the degree of etch on the polished surface only. If more etching is necessary, the lexan chip can be placed in a fresh NaOH solution for further etching. Then repeat all steps again (3-17).
- (18) For the induced tracks I place a piece of muscovite over the zircon and irradiate the sandwich. After irradiation I etch the muscovite and count the fossil tracks in the zircon and the induced tracks in the muscovite. I only count the polished surfaces. These are easily identified by the polishing scratches.

## Preparing samples for Irradiation

### Equipment

1. Liquid soap or detergent
2. Alcohol in wash bottle
3. Scotch magic tape
4. Muscovite cleavage flakes
5. Sharp knife
6. Pin vise with sharp pin or a dissecting needle
7. Micrometer or ruler
8. Irradiation container

### Procedure

1. Cleave muscovite into sheets between 0.05 and 0.1 mm thick.
2. Cut muscovites to a size that will cover the grains on the mount. It is best if it is not cut into a rectangle or square. Two right angle corners and two at an angle are best (figure 4). Cut enough pieces to cover all mounts.
3. Lay out all mineral mounts in some sort of order.
4. Clean the mounts one at a time. It is best to clean one mount, then cover it with muscovite before going to the second mount. The mount should be washed in a mild soap or detergent solution, then rinsed with distilled water and alcohol. With compressed air gently blow off the alcohol, or let the alcohol evaporate.
5. Take a long strip of magic tape (about 20 to 30 cm) and stick one end to the edge of the lab. bench. Now take a piece of the trimmed muscovite and stick it to the tape. Using tweezers gently remove the muscovite from the tape. A thin layer of muscovite will stay on the tape leaving a clean-fresh cleavage surface in the tweezers. Put the clean side of the muscovite down on the grains in the mount.
6. Take a short piece of magic tape (2-3cm) and place it over the muscovite and mount. Press down to hold muscovite in place. With a sharp knife cut around the edge of the mount to trim off excess tape. DO NOT FOLD TAPE AROUND EDGES OF MOUNT.
7. Repeat steps 4,5, and 6 until all mounts are covered by a detector and tape.
8. In a similar way clean and cover two pieces of glass standard with a muscovite detector. It may be necessary to wrap the tape around the glass to be sure the detector is held in place. Put a "T" on one and a "B" on another.

9. Number the muscovite mount packages. If some apatite packages are to be included in the reactor package start with the next number after the last apatite package.
10. In your sample book record the irradiation number and sample number of each sample (fig. 5).
11. With the dissecting needle or pin vise, punch 6 holes through the muscovite and into the plastic mount (figure 6). These holes will serve as reference marks which will aid in finding the proper area to count in the detector. Do this with each grain mount. It is not necessary to do it to the glass standards.
12. Measure the thickness of each mount and record in book along with the sample numbers.
13. Place the samples and standards in the irradiation tube in the order they are numbered. The "B" glass should be placed in the bottom of the tube muscovite side up. Then the samples are placed in again muscovite side up. The glass "T" is then placed on top of the last sample, but this time the muscovite side faces down (Fig. 7).
14. Add enough packing material so that when the top of the irradiation tube is screwed on the samples will be pressed together so that there will be no gaps between the mount and muscovite.
15. Decide on the proper neutron dose. It is best in most cases to have the induced track density within a factor or two of the fossil track density. The exception to this would be when the fossil track density is very low or very high. In the former case it is desirable to make enough induced tracks so that they will be easy to count. In the latter case it is desirable to make fewer tracks to count as  $10^7$  tracks/cm<sup>2</sup> is a very hard density to accurately count in muscovite. The following formula can be used to estimate the neutron dose:

$$\emptyset = 1.63 \times 10^7 \times T \times f$$

Where  $\emptyset$  = neutron dose

T = expected age in years

f = 1 for internal tracks (apatite)

f = 2 for external track (muscovite detector)

Figure 4  
MUSCOVITE DETECTOR  
Shape

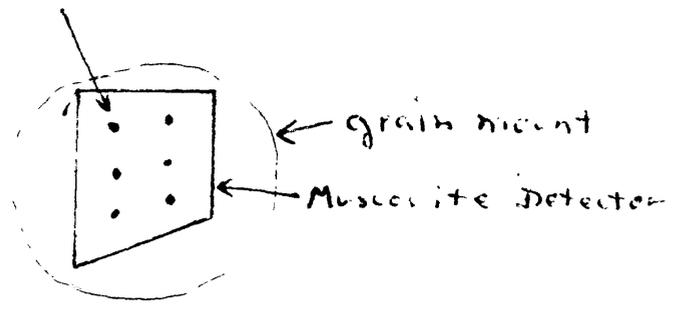


Figure 5

Sample Listing of Irradiation Package  
Reactor Run 11-13-74 in Al tube  
500 kw for 30 min. (Lazy Susan)

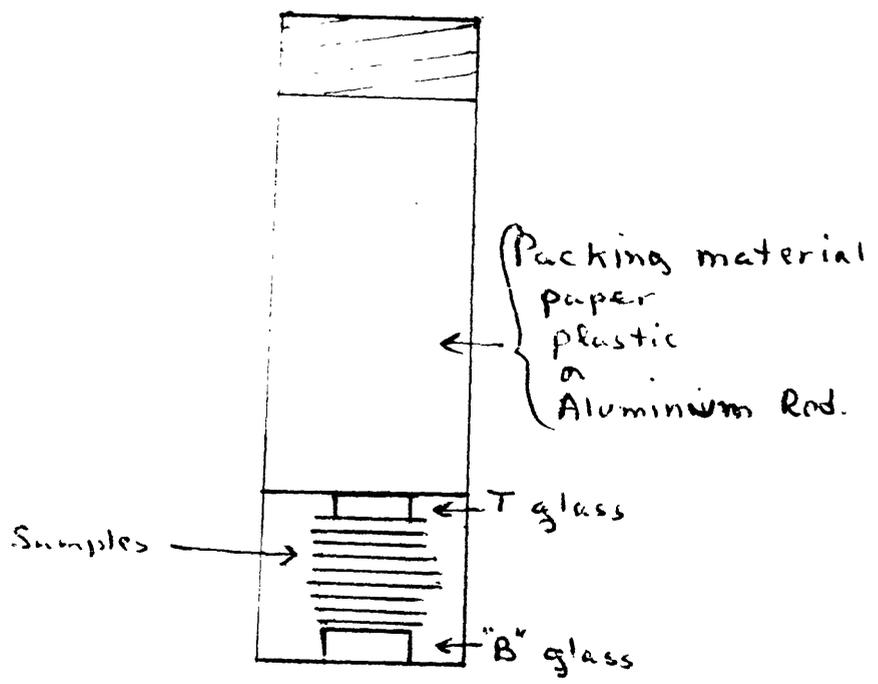
Bottom		mm Thickness
"B"	USGS glass standard with muscovite	
1	70L126 (apatite - Al package)	0.4
2	B-75 (apatite in Al package)	0.5
3	KR-1 sphene in epoxy	1.5
4	70L126 zircon in lexan	1.0
5	B-24 " " "	0.5
6	#9 " " "	0.9
7	22052 " " "	1.0
8	22052 " in teflon	0.5
9	B-24 " " "	0.3
T	USGS glass standard with muscovite	

Figure 6  
Punch Marks



Punching done after Muscovite taped in place

Fig 7  
Irradiation tube



### Post Irradiation Procedures

1. The irradiation package will be very radioactive after the irradiation. It is necessary to wait for the activity to reach a safe level before proceeding. The health physics personnel at the reactor can help with this.
2. Prepare your laboratory bench for emptying the package. Spread several paper towels out on the top. Wearing plastic gloves, or holding the irradiation tube with paper towels open the package and pour contents out onto the paper towels. The outside of the irradiation tube picks up a lot of contamination during the irradiation, so it is best not to handle it with bare skin.
3. Separate the standards, mineral mounts, and the apatite packages. They will each be processed differently.
4. With a sharp knife cut a "T" through the tape into the back of the muscovite covering the top glass standard, and a "B" through the back of the muscovite detector over the bottom standard care should be taken not to cut through the muscovite. These marks serve two purposes. First it identifies "T" and "B", and second it identifies the front and back of the muscovite. The back has the scratched letter, and the front has the induced tracks. Using a sharp knife carefully cut around the muscovite and gently lift it off of the glass standard. It is not necessary to remove the tape from the back of the muscovite as it will come off during the etching. Repeat with the other glass and detector.
5. Place the two muscovite detectors in a small plastic cup. Put them in so that the tape side is down and the track side is up. Pour in 48% HF and etch for one hour at 25°C.
6. Rinse the muscovite well with distilled water and alcohol, then put it on a hot plate, track side up, to boil off any HF left.
7. Take a clean glass slide and number it with the type of standard used, reactor run number, and put a "T" and a "B" along one side (Figure 8). Put a small amount of epoxy or laceside cement on the slide and stick the muscovite detector to the slide. Be sure they are put track side up and the "T" and "B" are in the proper place.
8. Take a plastic grain mount and with a sharp knife cut along the edge of the muscovite through the tape and into the plastic. This will leave an outline of the muscovite in the plastic. Gently lift the muscovite off the mount and put it in a plastic cup for etching; again tape side down and track side up. Remove any excess tape from the edges of the plastic mount.
9. Etch the muscovite detector in 48% HF for 11 minutes at about 25°C, if it is hotter a little shorter.
10. Rinse the muscovite as in step 6, then place next to mount.

11. Repeat process until all detectors are etched. Once you are familiar with this process it is possible to etch more than one using separate cups. The detectors can be put in at 2 minute intervals. Two minutes is enough time to take care of the various steps.
12. Find the labelled glass slide used to hold the grain mount during polishing. Along one side add the reactor run number (Fig. 9).
13. Mix up enough epoxy to give all the mounts and detectors to their respective slides. Quick setting epoxy can be used for this step, if desired; it is not necessary to heat the epoxy for this step. Take the numbered glass slide and smear out a thin film of epoxy on the side opposite the numbers. Take the mount and the muscovite detector and put it on the epoxy. Be sure the track side of the muscovite is up. To tell this the detector should form a mirror image of the knife marks in the mount. This is why a rectangle or square shape for the detector is not good. Try to put them on so that the sides are parallel (Figure 10). Allow the epoxy to set. If too much epoxy was used it will flow up through the punch hole in the detector. This will prevent the counting of tracks near the holes. Do not do anything about it. That will only make it worse, only the next time use less epoxy under the detector.
14. Take an apatite package and carefully open it over a piece of smooth paper. Empty all of the grains onto the paper.
15. Pour the grains from the paper onto the mounting block and proceed with the mounting procedures outlined in the section on mounting minerals. Be sure the block is properly marked so that it is known which apatite went where. When numbering the glass slide for the mounting be sure to add the reactor run number as well as the sample number. Repeat for all apatite packages.
16. Allow the epoxy to set, polish and then etch the apatite grains for the same length of time as the fossil tracks. It is best if the two are etched together at this time.

Figure 8  
Detector Slide for Standard

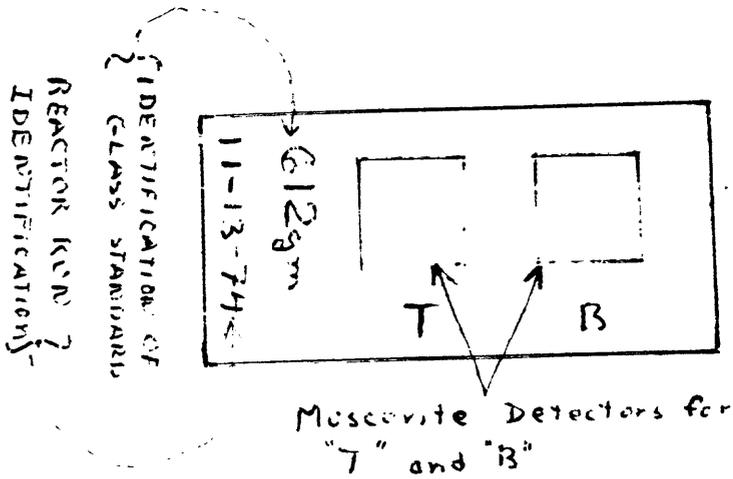


Figure 9  
Marking of glass slide for Mount

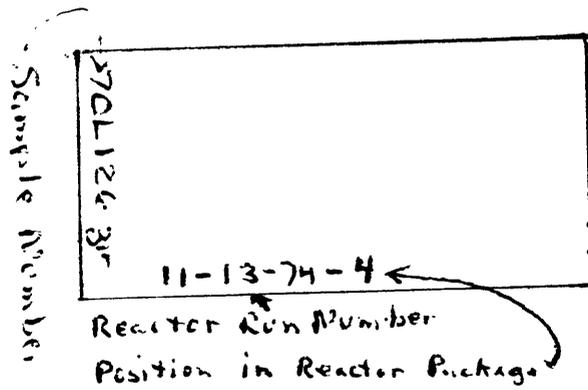
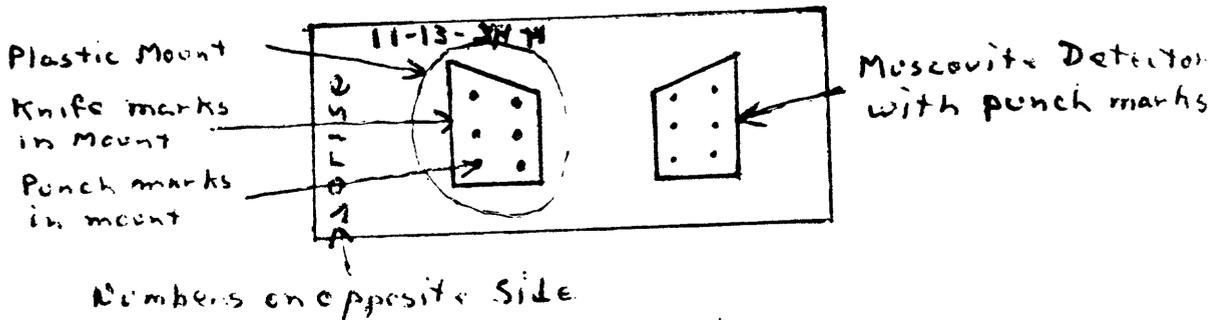


Fig 10  
Mounting detector and mount on slide



## Counting of Fission Tracks

When counting fission tracks it is necessary to know the area being counted. Track counts are usually reported in tracks per  $\text{cm}^2$ . Usually there are too many tracks in the entire field of view of the microscope to count accurately. The counting is done with the aid of a grid reticle in the eyepiece of the microscope. Most microscope manufacturers make an eyepiece that will take interchangeable reticles. Leitz makes three reticles which are excellent and cover the entire range needed: one is a 1 cm square divided into 25 2 mm squares; the second is a 1 cm square divided into 100 1 mm squares; and the third is a 1 cm square divided into 400 0.5 mm squares. The choice of the reticle depends on the number of tracks present in the small square. I have found that when there are more than 20 tracks in a square it is best to change reticles and go to the next smaller square.

The counting of tracks in minerals, and the muscovite detector over the minerals should be done at magnification of greater than 800 x. I have found that 1000x is an ideal magnification. I do not recommend magnification of greater than 1250x because the depth of focus is so short it becomes difficult to see the track in any detail. These magnifications can be easily achieved with a 100x oil immersion objective (be sure to put the oil on the slide when using this objective) and a 10x eyepiece.

The counting of tracks in glass and the muscovite detector over the glass standard is best done at 400 to 600x. I think that a reflected light objective used with transmitted light is best for this counting. It is designed for use without a coverglass. Most 40 to 50x objectives on petrographic microscope are designed to be used with a cover glass. If one of these objectives is to be used put a small cover glass on top of the glass or mica, this will greatly improve the image. With a very dark glass it may be necessary to count the tracks in reflected light.

It is necessary to calibrate the squares in the grid, so it is known how much area they cover on the sample being counted. The calibration can be done with a stage micrometer. If a binocular microscope is being used the calibration changes with the interocular spacing. If two more more persons are using the same microscope it will be necessary for each to determine the calibration for their own interocular distance.

In the determination of the neutron dose it is best to count a minimum of 1000 tracks in each detector. Some reactors have a gradient in its neutron flux. This is the reason for a standard at the top and the bottom of the irradiation tube. The neutron dose may be different along the length of the package the dose for an individual sample can be estimated by constructing a graph. The "Y" axis should be the dose and the "X" axis should be distance along the package between "T" and "B", with the dose at "T" at one end and the dose at "B" at the other (Fig. 11). A straight line is drawn connecting "T" and "B". Then

the sample dose can be read off the graph for its position in the stack. The best set of standards available now is the National Bureau of Standards glasses; these are SRM 961, 962, 963, 964. These are excellent glasses for calibrating a reactor and using as standards in each run.

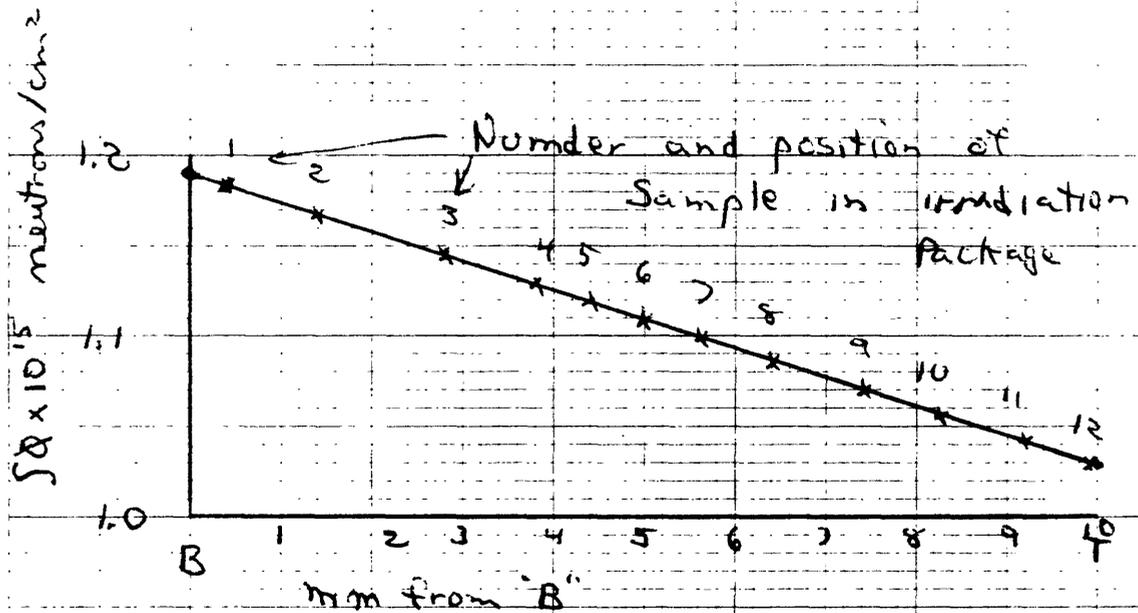
When counting apatite it is best to always count the same number of squares on each grain. The number of squares used will depend on the grain size, the track density and distribution within the grain. The number of squares counted should be shown on the page where the counts are recorded. If you chose to count 9 squares on the 5x5 grid it could be recorded as 9/25, or 8 squares on the 10x10 would be recorded as 8/100. A minimum of fifty grains should be counted for both the fossil and induced count.

The procedure for counting minerals in plastic mounts where an external detector is used is quite different. Here only 6 or so crystals will be counted and only parts of them. The number of squares counted may vary from grain to grain but count the same number of squares on the detector as were counted on the grain. Extreme care should be taken to count the exact area on the detector that was over the part counted on the grain. This is because the uranium distribution is not constant through out most crystals. It should be remembered that the image in the detector is reversed along a vertical axis. Thus if the upper left corner of a grain was counted the upper right corner of the image in the detector should be counted. With a little practice it becomes quite easy to locate the image in the detector and to count the proper part. Figures 12 to 14 will illustrate this. If the grain is embedded in epoxy or teflon the plastic comes right to the top edge of the grain (Figure 15), and a sharp image of the grain is found in the detector. If the zircon is embedded in lexan after etching the melted lexan does not completely come up to the top edge of the grain (figure 16). In this case the image in the detector will not be sharp, a number of tracks can be observed radiating away from the grain. This is because fission fragments originating along the side of the zircon are not absorbed and then register in the detector. When locating the area to count in the detector it is necessary to look for the last tracks which are vertical, or appear to point inward. These will then mark the edge of the crystal. Do not count any tracks outside of that imaginary line.

In figures 17, 18 and 19 are examples of data pages for the counting of minerals.

Figure 11

Neutron dose determination  
for reactors with neutron  
gradient



$$\bar{S} \text{ at "B"} = 1.19 \times 10^{15}$$

$$\bar{S} \text{ at "T"} = 1.03 \times 10^{15}$$

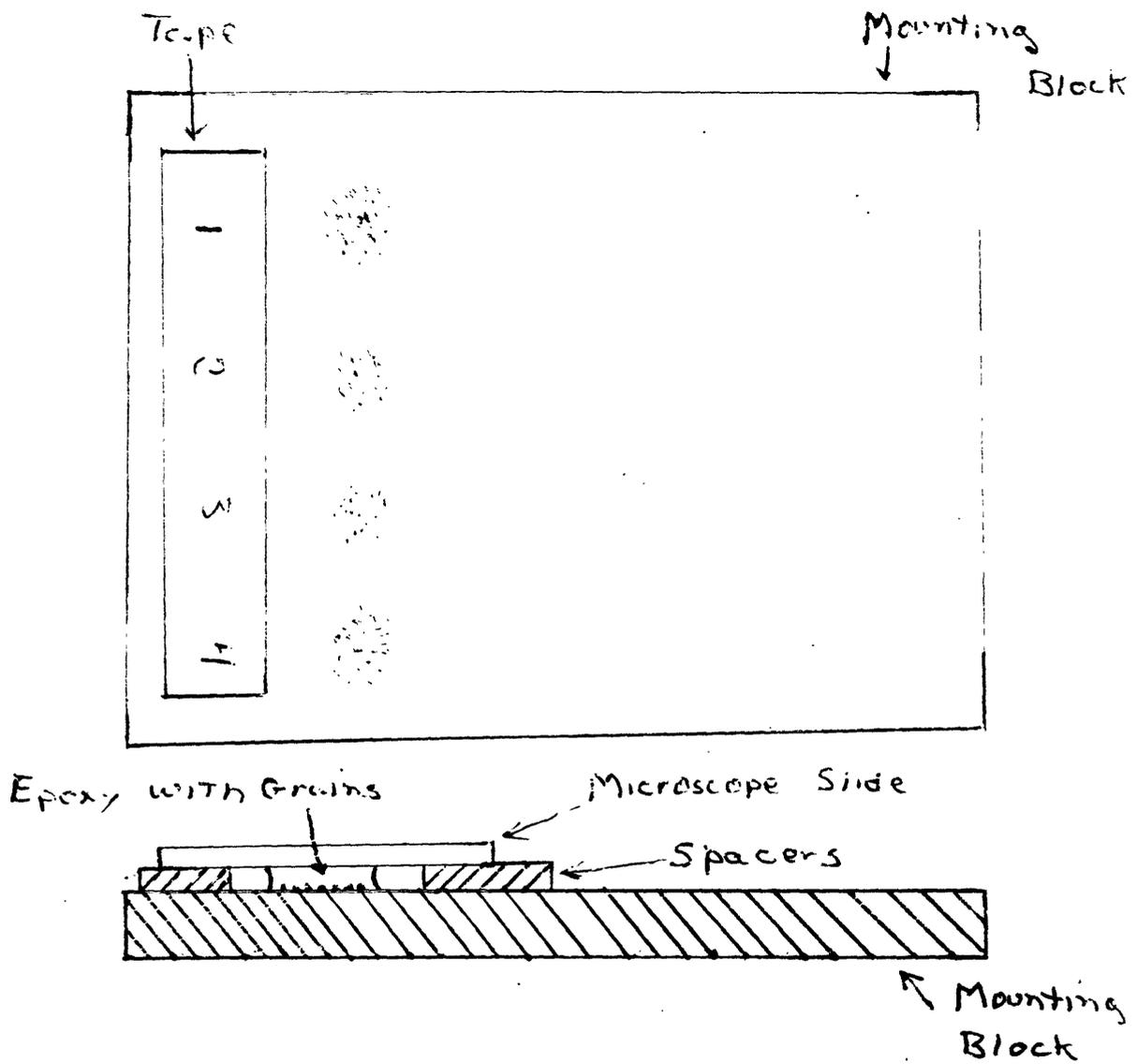
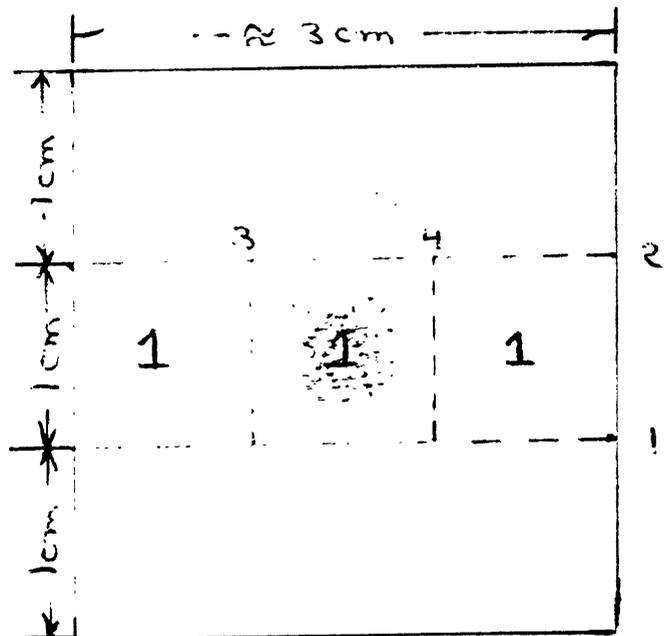
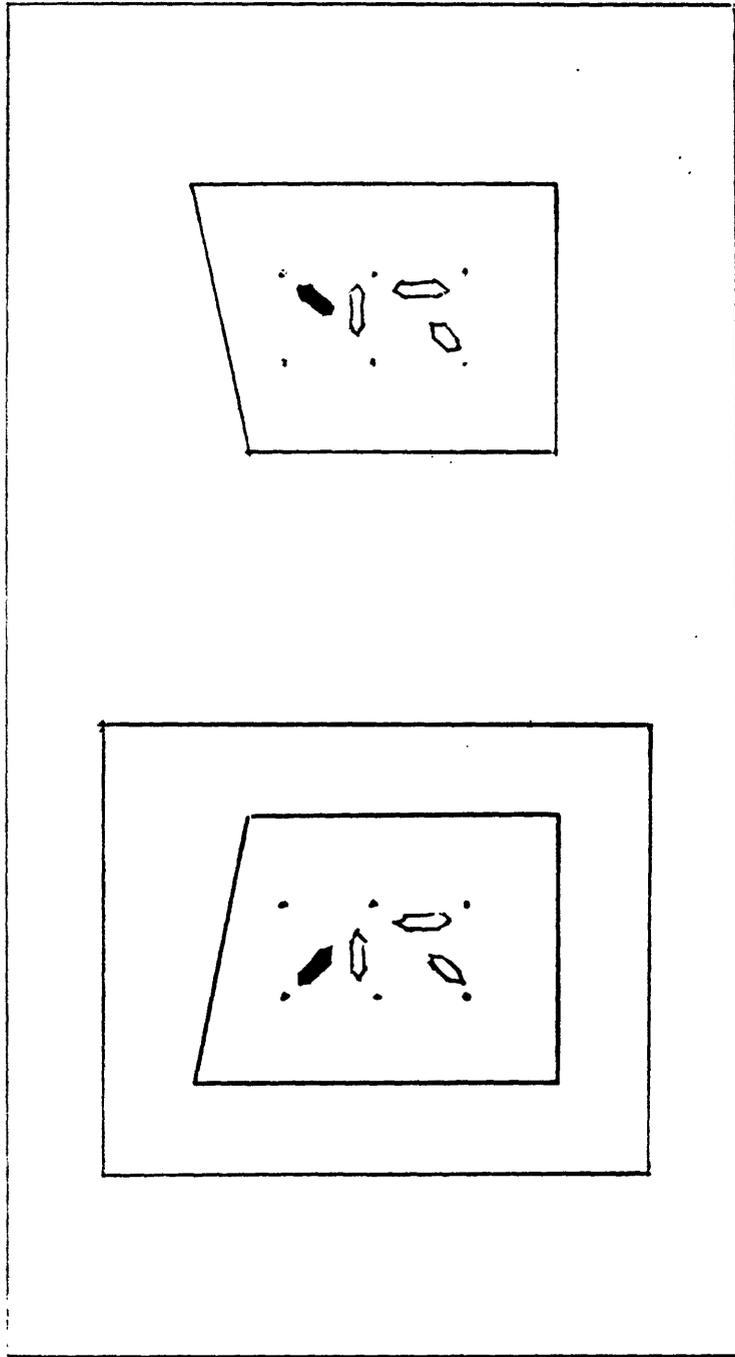


Figure 1

Figure 2. Apetit annealing,  
and irradiation package



1. Number Al foil in three places.
2. Turn Al foil over
3. Pour several hundred grains on center
4. Fold into thirds twice, ending with an envelope about 1cm on a side



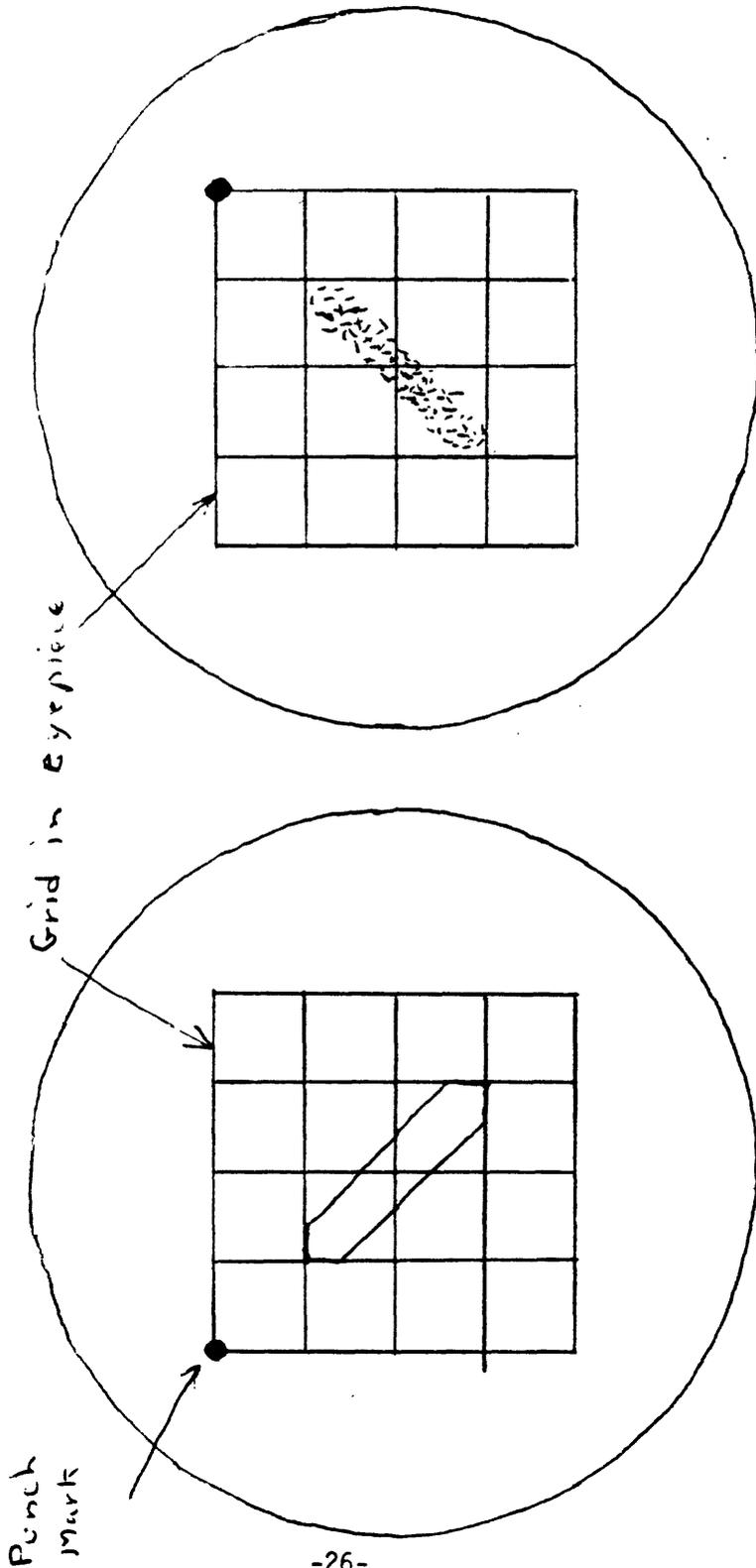
Etched Muscovite  
Detector with  
track image of grain.

Mount with etched  
crystals

Figure 12  
Slide with Mount and detector

Figure 13

Locating Image in detector



- ① at lowest magnification find punch mark nearest to grain to be counted. In this case the grain is down 2 squares and over two to the right from the punch mark.
- ② Go to detector and locate punch mark. In this case the image will be down 2 squares and over two to the left from the punch mark.

Figure 14

Locating area to count  
in detector; done at  
counting magnification

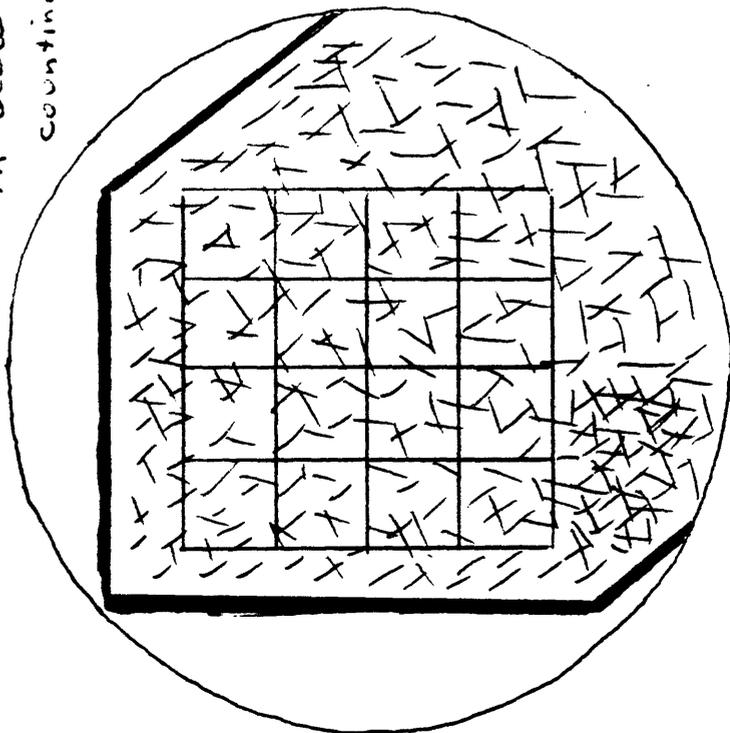
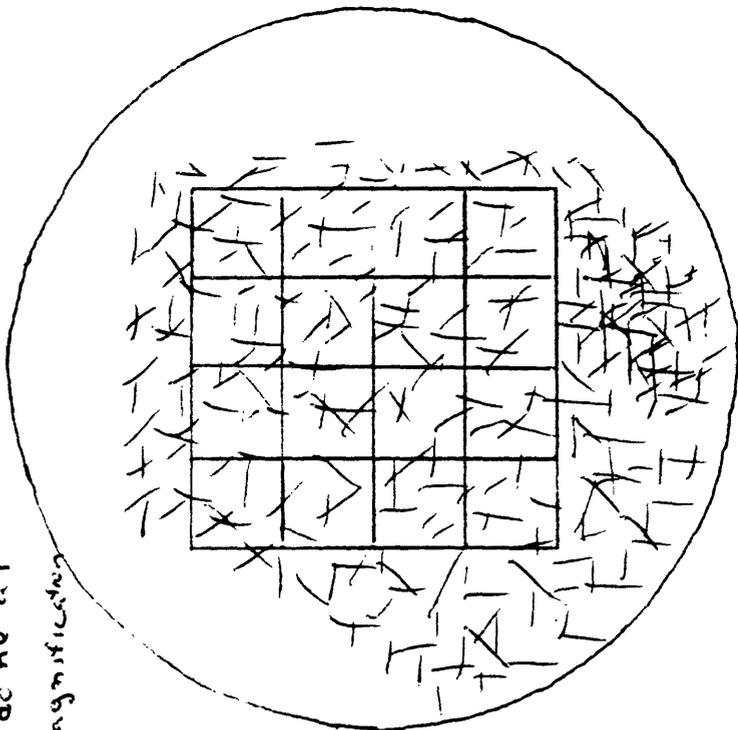


Figure 15

Cross section of grain in epoxy or teflon mount

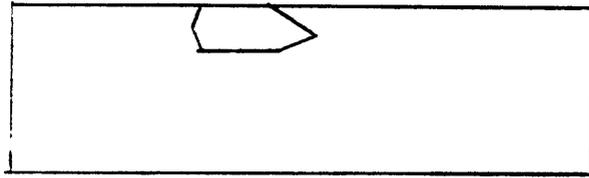


Figure 16

Cross section of grain in Lexan mount

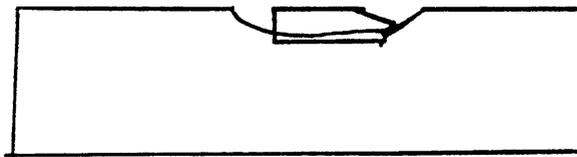


Fig 12.

Data Page for grains with external detector

DF-686 <sup>1)</sup> zircon <sup>2)</sup> 1/25 <sup>3)</sup> 1250x <sup>4)</sup> 21.7 x 51.0 <sup>5)</sup>

grain #1

fossil 3 4 3 6 10

$$P_3 = 4.89$$

1 8 3 6

$$\frac{1}{2} P_1 = 20.22$$

44/9

$$P_1 = 40.44$$

RR-10-4-74-16 <sup>6)</sup>  $\lambda = 1.15 \times 10^{15} \text{ m/cm}^2$

$$P_3/P_1 = 0.121$$

ind. in Musc 10 19 22 19 30

$$1.00131^{2)}$$

20 20 24 18

$$T = 8.5 \pm 1.4$$

182/9

- 1) Sample number
- 2) mineral
- 3) grid size and size of area being counted
- 4) microscope magnification
- 5) location of grain on ~~stage~~ mechanical stage
- 6) Reactor run date and position in package (16)
- 7)  $L_n$  term in age equation

Fig 18  
Combining Single Grain Data

DF-686 zircon

1/25 1250x RR-10-4-74-16

$$\lambda = 1.15 \times 10^{15}$$

grain	$P_3$	$\frac{1}{2} P_2$	$T$
1	44/9 <sup>1)</sup>	182/9 <sup>2)</sup>	8.5 ± 1.4
2	65/9	287/9	8.0 ± 1.1
3	<u>67/9</u>	<u>247/9</u>	9.6 ± 1.3
	176/27	716/27	

$$P_3 = 176/27 = 6.52$$

$$\frac{1}{2} P_2 = 716/27 = 26.52$$

$$P_1 = 53.04$$

$$\sigma = 8.4\%$$

$$P_3/P_1 = 0.123$$

$$2\sigma = 16.8\%$$

$$1.00136$$

$$T = 8.7 \pm 1.5$$

- 1) 44 tracks counted in 9 squares of the 25 grid  
 2) 716 " " " 9 " " " " "

Figure 19  
 Cephalite dates

DF-426 apatite 9/25 1250x

1	5	8	4	0	1
2	3	3	6	3	6
3	11	16	13	1	7
4	3	3	2	6	3
5	7	6	2	13	6
6	4	9	14	6	11
7	3	1	3	0	3
8	0	2	16	1	0
9	1	12	0	2	0
10	<u>4</u>	<u>5</u>	<u>0</u>	<u>8</u>	<u>8</u>

nine squares on the  
 25 grid (5x5) were counted  
 on 50 grains

41 65 60 40 45 = 251/50 = 5.02

$B = 5.02$

$f = 3.88$

$B/f = 1.294$

1.0255

$T = 164 \pm 33$  m.y.

RR-12-13-74-4  $\lambda = 2.09 \times 10^{15}$

1	3	2	9	3	0
2	5	3	10	3	7
3	4	1	2	7	0
4	7	3	2	1	3
5	1	1	3	6	3
6	5	10	3	3	9
7	3	4	0	6	5
8	10	9	2	7	9
9	4	2	2	2	6
10	<u>0</u>	<u>0</u>	<u>3</u>	<u>0</u>	<u>1</u>

42 35 36 38 43 = 194/50 = 3.88

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