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Etching of recoil tracks in solids

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Abstract—Low-energy (keV/atomic mass) ions in solids displace atoms, producing damage sites that are often described as tracks. Common natural sources of such tracks are recoiling residual nuclei from natural alpha-particle decay. Different models of such tracks are noted and compared with existing experimental results. First, the drastically lower rates of etching along such tracks in muscovite mica, compared to those for continuous ionization-produced tracks (such as result from fission), imply that recoil tracks are discontinuous, with clumped damage that is interrupted by gaps that either are undamaged, or lightly damaged. Second, based on the size of recoil-tracks and the numbers that bring about total disorder in crystals such as zircon, the tracks are diffuse regions–containing only about 2% of misplaced atoms. A simple model describes the profound effect of the gaps in recoil tracks on etching efficiency, removal of recoil nuclei by leaching, track revelation, and their vastly different etching behavior from that of high-energy tracks. *Copyright © 2003 Elsevier Ltd*

INTRODUCTION

When a heavy atom spontaneously decays by emitting an alpha particle (with a typical energy of 5 MeV), conservation of momentum requires that the residual nucleus will recoil with an energy of around 90 keV. Within many natural crystals this recoil will move the nucleus 20 to 25 nm, these ranges being derived from the calculations of Lindhard and Scharff (1961)and Lindhard and Thomsen (1962); the result is a trail of atomic disorder that is referred to as an alpha-recoil track. (The alpha particles themselves do not make tracks in minerals.) These tracks are of importance to a number of geochemical phenomena and applications–including a method of radiometric dating, alpha recoil dating (Huang and Walker, 1967), disordering of crystals (metamictization) (Ewing, 1994), and understanding how in the U and Th decay chains isotopic disequilibrium comes about (reviewed briefly by Szabo ,1969). Alpha damage is also thought to provide a mechanism for radon release from the earth (Fleischer, 1982a, 1983). In addition, the nature of the damage itself is of fundamental interest.

In this work I consider information that comes primarily from existing results of chemical etching of recoil damage and of disordering. These data have much to convey about the nature of recoil tracks, and they lead to better understanding of how to use recoil etching. First, it is desirable to point out that there are striking differences between low-energy tracks, such as come from alpha recoils, and high-energy ones, such as are produced by fission fragments and heavy cosmic-ray nuclei (Fleischer et al., 1975).

Low-energy versus High-energy Track Production

Fission-fragment tracks are probably the most familiar natural tracks in minerals. They are an example of fast-moving atomic nuclei — fast usually meaning MeV/amu (atomic mass number) or more, which corresponds to high enough speeds that the particles have lost a good many of their orbital electrons and are thus multiply-charged. Such a projectile deposits energy in a solid mostly by knocking electrons loose, i.e., by ionization (Fleischer et al., 1965). Typically the resulting tracks are regions of continuous damage, and preferential etching along them can develop without interruption.

Less familiar, but more abundant in terrestrial minerals, are the tracks left by atoms that recoil from alpha particle emission – an example of low-energy tracks. At the typical keV/amu velocities the particles move more slowly than the orbital speeds of most of their bound electrons – which then can adjust rather than being removed from the projectile or from atoms of the solid. In this case ionization is minimal. However, because the particle retains all or most of its electrons, it is a massive object (of atomic, rather than nuclear size). It loses energy mostly by atomic, billiard-ball-like collisions, which are subject to wide statistical fluctuations in the damage that is created and the path followed by the projectile (Lindhard et al., 1963).

MODELS OF RECOIL TRACKS

The first of the models to be considered is that of Brinkman (1955), who considered atomic displacements by what he called "high-energy" particles. His term is different from high-energy as described above. Brinkman merely means that the particle has an energy well above that needed to displace an individual atom – a value that is typically around 25 eV in metals. For example, the value to displace an atom is 22 eV for copper in the earliest, clean experimental determination (Corbett et al., 1957). Brinkman concludes that the trajectory of what is called here a low-energy particle may bounce around, but it ends in what he calls a displacement spike. Such a spike is described in his Figure 3 as a dense, short, but continuous region of displaced atoms and vacant lattice sites. Such a defect would be expected to etch continuously, but only briefly since it is short, only 5 to 10 nm long as sketched by Brinkman.

Later, Vineyard (1961) showed (Figure 1) the results of an atomistic calculation that yields a rather different looking result. Here we see disconnected damage, with groups of defects off to the side of the path of the projectile; and the damage-producing nucleus is repeatedly deflected from side-to-side.

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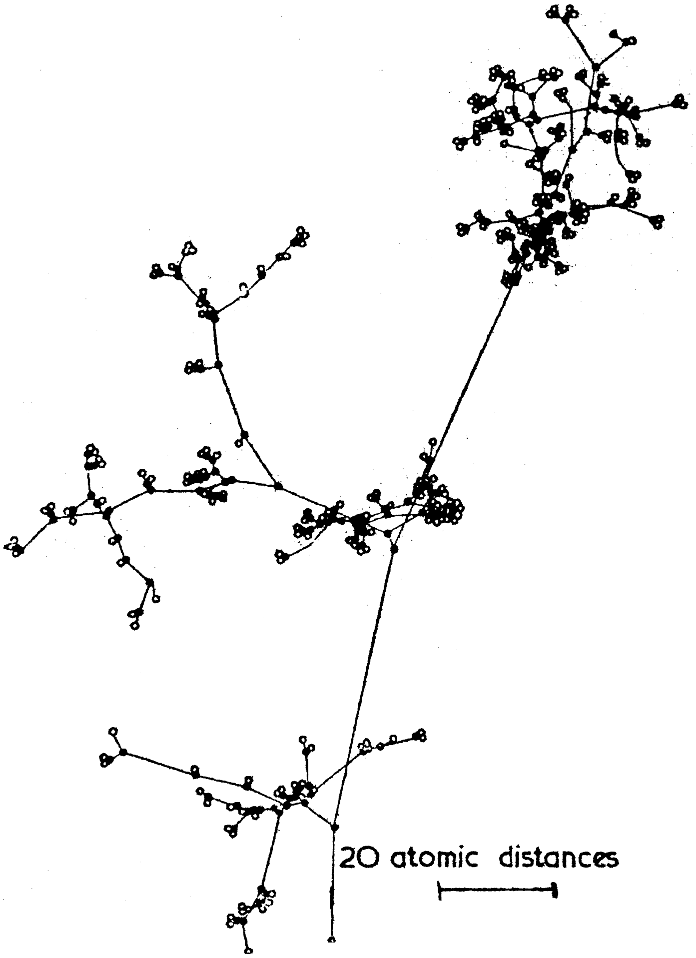

Fig. 1. Calculated collision-cascade, modeled for a 10 keV Ge atom in Ge. Dots represent interstitials and circles are for vacancies. From Vineyard (1961), reproduced by permission of The Royal Society of Chemistry.

The expected etching behavior for such a defect is quite different, since the density of damage (number of atomic defects per unit volume) is far less than in a displacement spike, and since the damage is irregular along the "track." It is likely that Brinkman would describe each of the regions of dense atomic disorder in Figure 1 as a displacement spike. Although Vineyard's result was obtained long ago, modern calculations have given a qualitatively very similar result for a collision cascade, as given by Figure 1 in a report by Matzke and Wiss (2000).

Snowden-ifft and Chan (1995) studied intermediate-energy O-, Si-, and K-ion damage in muscovite (i.e., energy loss that was a roughly even mixture of ionization and atomic collisions). However, for all three ions the ionization rate was below the threshold for track formation in muscovite; so damage was derived only from collisions. The authors found short tracks and quantitatively interpreted their observations as due to damage being disconnected, consisting of occasional short damage globs and mostly undamaged intervals. For alpha-recoil damage (because it is from slower, heavier particles) the energy-loss rate is more than 10 times higher than for Snowden-Ifft and Chan's ions, and we will conclude that damage is nearly continuous, but interrupted by undamaged gaps – in short a dashed line, rather than a line of dots.

Recently more sophisticated models have been used to deal with recoil damage. For example Trachenko et al. (2001) have used molecular dynamics to represent recoil damage in zircon. They show that the damage from a single event is mostly within a region that is about 2 × 2 × 3 nm in dimensions — this presumably being a single knock-on. Their model was limited to a crystal that was only slightly larger than the calculated damage diameter.

We will compare observations of track etching with these models and with what is observed for high-energy particles. It is appropriate to note that Lindhard and co-workers calculations and surveys of observations (Lindhard and Scharff, 1961, Lindhard and Thomsen, 1962, Lindhard et al., 1963) describe a smooth average slowing down of low-energy particles, but with unusual variability in the trajectories. As an example, Lindhard et al. (1963) in their Table 3 calculated that the typical fluctuations in the projected range R_P (the distance from the start to the final position of the particle) are about $0.5R_P$. This result is in striking contrast to fluctuations in the ranges of fast, ionizing particles, which Rossi (1952), page 37, gives as only about 0.05 R_P. We shall look at etching results to find signs of the irregularities to be expected in low-energy tracks. The exact chemical composition enters into the range versus energy calculation, but the ranges for most silicate minerals vary by less than a factor of two, and the most common ones being in the interval 15 to 30 nm.

IN-SITU OBSERVATIONS

There are few direct observations of recoil tracks. One of the more useful studies that describes the character of the damage is that of Narayan et al. (1985). They studied Si bombarded with 100 keV Bi ions, which are good surrogates in mass and energy for typical alpha-recoil nuclei. Disordered blobs of disorder that range around 5 nm in diameter appear in transmission electron micrographs (TEM's) – often as groups that are more or less in line. Atomic-resolution TEM photos of cross sections of the damage show (1) totally disordered circular regions of diameter 8 and 9 nm and (2) regions of partial disorder across which crystal planes can be clearly traced (their Fig. 2a). Presumably the globs are displacement spikes, and the chains of spikes delineate the repeated scatterings in the trajectory of the low-energy ion – as in Figure 1. The regions of partial order imply non-uniform damage. Similar observations were made by Lumpkin and Ewing (1988) in microlite (their Fig. 12c) and by Hobbs et al. (1994) in zircon (their Fig. 16a,b).

OBSERVATIONS BY ETCHING

Chemical etching is sensitive to non-uniformities. Thus it will give information about the damage distribution; and conversely, knowing its behavior will help in developing optimal etching procedures for different uses of recoil tracks. Alpha-recoil tracks were observed first by Huang and Walker (1967), using chemical etching of various micas (they reported results on muscovite and phlogopite). Those authors were concerned primarily with the possible use of these tracks to date minerals, and they tacitly assumed that etching was uniform along recoil tracks.

Gögen and Wagner (2000) and Glassmacher and Wagner (2002) observed that the density of etched recoil tracks in phlogopite mica increased linearly with etching time, a result that is to be expected if more and more tracks are reached as surface layers are removed by the (slow) etching rate v_G (perpendicular) and by uncovering deeper layers by the lateral enlargement of etch pits.

High-magnification viewing of low-energy tracks in muscovite mica gave strikingly distinct results that show recoil tracks to be vastly different from most high-energy, ionization-produced tracks. The taper, or cone angle, of a track is diagnostic of the damage along the track. From the profile of the etched recoil tracks Snowden-Ifft et al. (1993) showed the track depth to be 30 nm and the width to be 3500 nm, leading to the accelerated etching v_T being only 0.15% elevated over the general attack rate v_G. It is well established that for high-energy, ionization-produced tracks (Fleischer et al., 1965) the etching rate v_T increases with ionization rate relative to v_G, allowing individual track-forming particles to be identified (Price et al., 1967). Table 1 shows a comparison for muscovite mica of the ratio of the fractional increase in etching rates (v_T - v_G)/v_G first for alpha particles (which form no tracks) and then for fission and recoil tracks — each of which deposit nearly the same amounts of energy per unit distance moved. As compared to 0.0015 for recoils, for fission tracks the etching acceleration is at least by 3,000 times! This limit is derived from Bean et al.'s (1970) sensitive electrical-conduction measurements of etching through a thin 4.2-micron-thick mica sheet used as a membrane in an electrochemical cell. From Figure 8 of Bean et al. v_T is greater than 4.2 micro-meters (the thickness of the muscovite sheet) per 5 seconds (the shortest time interval measured) and v_G is 0.25 nm/s (from the long-term slope of the hole radius versus time) The simplest interpretation is that the recoil tracks resemble the calculated results shown in Figure 1 in having gaps in the damage – gaps that slow the preferential etching rate markedly.

Table 1. Energy loss rates and etching ratios in muscovite mica.

Projectile	Energy (eV)	Range (μm)	dE/dx (eV/cm)	Preferential Etching Ratio ($v_T - v_G$)/v_G
Alpha particle	5×10^6	17	0.3×10^{10}	0
Alpha − recoil nucleus	8.5×10^4	0.02	4.3×10^{10}	0.0015
Fission fragment	8×10^7	12	6.7×10^{10}	$>3 \times 10^3$

It should be noted that muscovite is special in its high etching anisotropy, which can be used to understand the immense ratio $>2 \times 10^6$ ($\rightarrow 3 \times 10^3 / 1.5 \times 10^{-3}$). Fission tracks are continuous and etch at a vastly higher rate than the rate of general attack v_G (parallel) on the sides of tracks along the layer planes of the muscovite mica (as noted, by at least a factor of 3×10^3). Further, general etching in the orthogonal direction, normal to the layer planes is nearly, but not quite, zero; and it is less rapid than etching along the layers. According to Snowden-Ifft et al. (1993) v_G (perpendicular) is about 20 times lower than v_G (parallel). If fission tracks are heated until tracks begin to fade, they develop gaps in a small fraction of the originally continuous lines of damage. When etching reaches such a gap, it is held up until that very slow etching penetrates the gap. For short etching times, only the partial length of the track is revealed – giving rise to etched tracks still being displayed in nearly the true, original abundance, but being shortened (Fleischer et al., 1964).

Further Information on the Nature of Recoil Tracks

In addition to the insight given by Table 1, which compares the etching rates of recoil tracks with those of high-energy tracks, a second important bit of information on the nature of recoil tracks comes from considering the doses of recoil events needed to disorder crystals.

Consider the size of recoil tracks, preparatory to noting how many of them are needed to fill a sample with damage. Lindhard and Scharff (1961) and Lindhard and Thomsen (1962) showed that the particles lose their energy primarily by billiard-ball-like collisions with other atoms, and they produce damaged regions that do not have a simple, clean geometry. The damage consists of localized groups of atomic defects. The defects have been only occasionally seen directly, but can be inferred by general disordering of crystals and by accelerated localized preferential leaching of many materials. Lindhard's method gives ranges in common minerals of 16 to 26 nm, as calculated by Fleischer (2004). Since the atomic collisions that are involved have wide statistical fluctuations in location and direction, the diameters of regions that include most of the displaced atoms are expected to be larger than those measured for fast-ion tracks – 6.6 nm in mica (Bean et al., 1970) and 13 nm in plastic (DeSorbo 1979) — probably between 10 and 15 nm. (As noted, Narayan et al. (1985) observed 8 to 9 nm for the displacement spikes in Si, and the Vineyard (1961) calculation shows a similar value.) Recoil lengths observed by chemical etching are 30 nm in mica (Huang and Walker, 1967) and 20 nm in albite, a feldspar mineral (Turkowsky, 1969). Thus recoil tracks are nearly equiaxed in shape, perhaps resembling prolate ellipsoids in their outer boundaries. The overall size is about 20 nm long by 10 nm in diameter ($= 1.6 \times 10^{-18}$ cm^3). Figure 1 makes clear their irregular structure.

Another route to the size of a recoil track is this: Think of metamictization as an accumulation of many disordered regions until the original crystal is completely disordered. The number of recoils per unit volume then tells the effective size of the individual regions of disorder. As an example, for the most studied mineral, zircon ($ZrSiO_4$), disorder saturates at 10^{19} recoils/gm in young samples that have not had geological times for track annealing (Ewing, 1994). This dose is equivalent to 4.7×10^{19} recoils/cm^3. Allowing for tracks that overlap tracks, this number implies that about 3×10^{19}/cm^{3}would suffice to fill the volume with disorder if there were no overlap. Since there are about 6×10^{22} atoms/cm^3 in zircon, about 2,000 atoms are disordered per recoil event. A check on this number is to take twice the head-on displacement energy, which is about 22 eV for a typical material such as copper (Corbett et al., 1957), and divide it into a typical recoil energy 85,000 eV, giving nearly the same number — 1,930. 2,000 atoms are sufficient to fill a sphere 4 nm in diameter. This size is about 2% of the volume inferred in the preceding paragraph, and this fact is of profound importance: It tells us that the displaced atoms are not in a single, dense clump. Rather, as Figure 1 suggests, it is a diffuse region with mostly undamaged space. On the average about 50 recoil tracks (100%/2%) must overlap everywhere in order to fully disorder the crystal.

Model of track etching with gaps

A simple picture that describes the profound effect of gaps comes from recognizing that the time to etch at an average etching rate v is a weighted result of the time to etch across a fraction f of gaps and that for etching damaged regions that occupy the remaining 1-f of the length.

$$\text{Thus } 1/v = f/v_G + (1 - f)/v_T \qquad (1)$$

It is helpful to re-write this equation as $v = v_T/[(f\, v_T/v_G) + (1\text{-}f)]$. In one limiting case, for $f = 0$, $v = v_T$ –as it must where there no gaps; but when v_T/v_G is much larger than f, v is slowed dramatically and approaches v_G/f. For muscovite, where v_T/v_G is $>10^6$, even a tiny f slows the etching effectively. No other model of which I am aware explains the results in Table 1.

In another example, silica glass and many obsidians have cone angles around 19° and therefore v_T/v_G values around 3. From the equation it follows that $v = v_T/(1+2f)$, so that for 2% of gaps $v = v_T/1.04$. 2% is chosen as a fraction that would not alter the track density very much, but for most micas would have a major effect in reducing track length. The lesson is that for larger cone angles etching can bypass the gaps in damage without major delays, and thus v is little affected.

RELEVANCE TO USES OF RECOIL TRACKS

Huang and Walker (1967) proposed that alpha-recoil tracks would provide a method of age determination for minerals: The accumulation of tracks should be proportional to geological time, and the alpha-decay rate is such that their track numbers should be far higher than for fission tracks, such are used in fission track dating (Price and Walker, 1963; Fleischer and Price, 1964). However, as noted earlier, alpha-recoil track densities increase with time of etching, so that there needs to be a logical choice made as to what etching time to use for dating. Glassmacher and Wagner (2002) proposed to overcome this problem by deciding on a time by requiring a fit with a known age. This procedure is likely to give a different result for each type of mica, and within a given type it may be needed separately for different major element compositions, and possibly could also be altered by variations in minor elements. A proper choice might also depend on the absolute track densities – which may alter the value of v_G (perpendicular) by increasing the rate at which below-the-surface recoil tracks are uncovered. Thus knowledge of the behavior and nature of recoil tracks gives a cautionary message. Some type of calibration of recoil-track registration is desirable for individual samples.

Extensive accumulation of recoil tracks in a solid can progressively destroy the order of a crystal – producing ultimately a glass that is in what geoscientists call the metamict state, reviewed recently by Ewing (1994). Some types of crystals spontaneously anneal such damage at ambient temperatures, making those materials candidates for storage of nuclear waste (Eyal and Fleischer, 1985). One particular mineral that undergoes metamictization, zircon ($ZrSiO_4$), is abundant and unusually durable in nature. For these reasons it is used extensively in geochronology. For zircon, increasing the disorder makes the fission-track dating technique progressively more difficult, because, first, tracks from fission (which are formed at higher energies by ionization (Fleischer et al., 1965) rather than by atomic collisions (which produce recoil tracks) are so numerous, and therefore difficult to count. Second, etching rates strikingly increase with progressive disorder (making it difficult (Garver et al., 2000) to etch properly a mixture of polished grains of assorted densities of radiation damage).

Furthermore, information on alpha recoils can increase our understanding the effects of other low-energy ions – two examples are ions from ion implanters used widely in the electronics industry and solar-wind ions which bombard the surfaces of materials in space producing a flood of damage that leaves thin veneers of glass on the topmost grains in the lunar soil (Bibring et al., 1972). The fluence of recoil-like particles to bring about various degrees of disorder will depend on the atomic distribution of displacements from individual events.

The irregular and clumped character deduced for recoil tracks can help to understand some of the results in studies of how alpha recoils can bring about natural isotopic disequilibrium in radionuclides in the U and Th decay chains. As an example, ^{234}U is often enriched relative to ^{238}U in water that has percolated through a formation – and reduced in the formation (see Szabo, 1969). Two mechanisms that have been shown to operate are direct recoil into the water (Kigoshi, 1971) and preferential leaching of the recoil damage from the ^{234}Th that precedes ^{234}U in the ^{238}U decay chain (Fleischer, 1980). The results of extensive experiments on leaching of recoil tracks (Fleischer, 1980,1982a,b) can now be better understood.

Those studies showed that recoils entering a solid from a surface can be removed later by exposure to aqueous solutions due to preferential solubility of the damage created by the recoiling atom. In a series of experiments with a variety of materials, recoiling ^{235}U from alpha decay of thin ^{239}Pu sources was injected into several materials. After separate 24-hour treatments with multiple solutions, the samples were tested for the fraction of the injected uranium that remained – by using neutrons to induce fission and recording the resulting fission tracks. For the ten different materials that were tested, fractions of 0.2 to 0.8 (average = 0.5) were removed by 24 hour treatments, and for the mica as much as 85% by a week-long treatment. In short, although a considerable fraction was leached out for all the materials, removal was not total. Therefore, a continuous path for etching was not provided to many of the recoil atoms – the same conclusion as was just inferred from the etching behavior of muscovite mica. (In the other 9 materials besides mica, general etching is not vastly less than etching of damage. For this reason longer etching can etch fairly rapidly across gaps, and thus for longer treatment times the more active of the reagents can reach the track-producing particle and release it into the solution.) For weak solutions and neutral pH water it is likely that full extraction of the near-surface recoils will not happen, or will require far longer times than have been used up to the present.

In a straightforward extension of the leaching work, embedded ^{222}Rn from ^{226}Ra decay can be released by solutions – providing a mechanism for radon release from permeable rocks and from soil (Fleischer, 1982a,1983).

SUMMARY

The damage distribution of atomic displacements from alpha recoils has an envelope whose volume is about 50 times that of the number of atoms that are displaced by one such event; so damage is diffuse, and it includes of separated clumps that resemble Brinkman's (1955) displacement spikes. The special etching behavior for fission tracks in annealed muscovite mica, established that gaps could be formed in those tracks (Fleischer et al., 1964). The similar behavior of fresh alpha-recoil tracks implies that they too contain gaps, as some theories predict. This behavior may make recoil dating of most micas difficult (because of their unique anisotropy of etching), but it should affect biotite and other common minerals less (with their larger general etching rates that can bypass the gaps). The gaps help to explain the observed slow, partial leaching-out of recoils in common substances (Fleischer 1982a,b).

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