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New insights into the formation and annealing behavior of latent fission tracks

New insights into the formation and annealing behavior of latent fission tra

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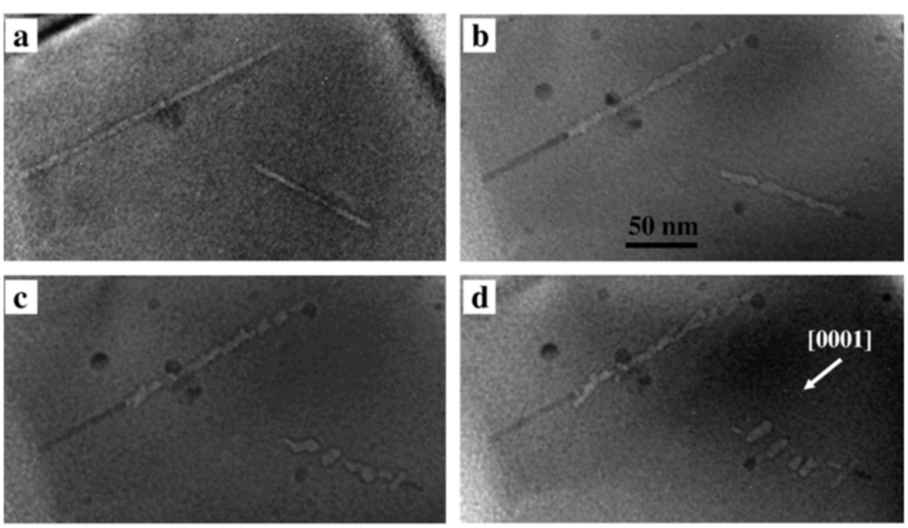
The fission track dating technique, as it is used in geological studies, is largely based on mathematical fits to data for etched track-lengths as a and composition. Observations of tracks in geological materials by techniques other than optical microscopy (e.g. Paul and Fitzgerald 1992; Jaskie less common. Consequently, the internal structure of latent (i.e., unetched) fission tracks is rather poorly known. In fact, the added value of using microscopy to study fission track annealing behavior - in order to improve the geological application of the technique - has been questioned (Paul behavior of latent fission tracks, as revealed by TEM investigations, was thought not to correlate with that of etched tracks.

During the past decade, important papers (e.g., Jonckheere 2003; Hendriks and Redfield 2005) have highlighted the need to investigate the struc tracks at the nano-scale. Most recently, researchers at the University of Michigan have led a multidisciplinary effort to understand the internal struc advanced high-resolution analytical techniques. Transmission Electron Microscopy (TEM) and synchrotron based Small Angle X-ray Scattering been carried out, and the authors of cited articles are from: University of Michigan (USA), Helmholtz Centre for Heavy Ion Research (GSI, G Undersøkelse (NGU, Norway), Rensselaer Polytechnic Institute (USA), Australian National University, Australian Synchrotron, Centre Interdiscip Ions, les Matériaux et la Photonique (CIMAP, France) and Pacific Northwest National Laboratory (USA). Exciting results offering new perspectives annealing have appeared in a series of papers: Lang *et al.* 2008a, 2008b, 2009a; Zhang *et al.* 2010; Li *et al.* 2010, 2011; Afra *et al.* 2011. These s heavy ions can be used to simulate fission tracks in minerals with and without pressure, that detailed thermal annealing studies can be comple observed *in situ* by high-resolution TEM, that very precise data on track size can be obtained on millions of tracks at a time and observed *in situ* du SAXS, and that molecular dynamics methods can be used to simulate the internal structure of tracks.



Li *et al.* (2010) studied the interr tracks in Durango apatite by TEM having an amorphous core, as is g tracks in apatite are actually por lower atomic density than the high-resolution TEM images sug fission tracks are not solid but ma The new data also provide dependence of the annealing ra track with respect to the crystallog is, of course, well-known to the O fission tracks, but a completely n nano-scale track-segments actu original orientation (which could be to become parallel to the c-axis. apparent inconsistency between t and unetched fission tracks impli tracks in minerals can survive to (track fragments are still be obser 130 minutes at 700°C).

Figure 1 (from Li *et al*. 2011): TEM images showing the preferential motion of fission track segments along the crystallographic c-axis in fluorapatite and the slower fragmentation of tracks along this direction during thermal treatment at 700°C (a – before experiment; b – after 1 minute; c – after 17 minutes; d – after 1 hr).

zircon. Fission tracks in zircon sl core, and this difference in structu results in very different anneali apatite and zircon. Annealing of t thermo-emission of vacancies fr surrounding matrix, in contrast interstitials and vacancies within the amorphous zircon fission tracks. The amorphous core of zircon fission tracks is not significantly different surrounding solid, which results in a relatively simple annealing mechanism. Fission tracks in zircon fade in a continuous way and they do not s also explains the agreement between the total annealing temperatures of etched (800°C after 1 hr; Yamada et al. 1995) and unetched fission trac hrs as observed by TEM). The thermal annealing of the porous tracks in apatite is a much more complicated process involving several underlying tracks can segment into periodic droplets (controlled by Rayleigh instability) and random motions of atoms on the surface of track segments (voids these segments. Preferential motion of atoms along the c-axis results in a more rapid track segmentation and annealing of tracks perpendicular to fission tracks in apatite is due to the high surface energy and high diffusivity of atoms on the inside surface of the porous tracks.

The two studies by Li *et al.* referred to above employed TEM, which can image individual tracks. A disadvantage of this technique is that prolonged electron irradiation can actually anneal the damage structure. Li *et al.* circumvented this issue by using a low current density beam and by moving samples away from the beam during the thermal annealing by the heating stage inside the TEM. SAXS however, does not have this disadvantage, has rapid data acquisition times and does not require elaborate sample preparation. This technique is sensitive to subtle changes in electron density within latent tracks by measuring the scattering of x-rays due to density fluctuations on length scales comparable to the lateral track dimensions. A suitable model is used to describe the experimentally determined scattering intensities (Figure 3a). The change in electron density due to radiation damage is significant and the damage boundaries sharp, which makes ion tracks into excellent scattering objects. Thus, high-quality data on track dimension and morphology (discontinuous *vs.* continuous tracks) can be obtained with accuracy down to fractions of a nanometer. Since x-ray beams of 100 μm spot size are used in transmission mode, millions of tracks can be investigated along their entire length during a single measurement without any track modifications (x-rays are non-destructive). Afra *et al.* (2011) used SAXS to study fission tracks in the same set of Durango apatite samples used for TEM investigations (Li *et al.* 2010, 2011) and performed both isochronal *ex situ* and isothermal *in situ* annealing experiments (Figure 3b). They demonstrated that this technique can determine the detailed structure of the latent tracks as function of temperature in apatite and found that annealing initially occurred by structural relaxation which was then followed by recrystallization.

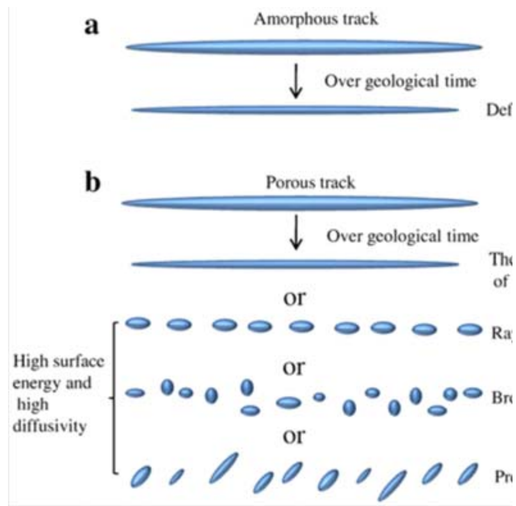


Figure 2 (from Li *et al*. 2011): Comparison of the ann amorphous core (a) to that of a porous core (b). The is controlled by the gradual shrinkage and the discon

The initial paper to come out of our collaborative efforts (Lang *et al.* 2008a; also presented at the Fission Track meeting in Anchorage) documented the first report of fission tracks simulated in a laboratory under geologically relevant pre studies of pressure effects done previously were conducted on tracks created at surface conditions, only to be put under pressure and temperature of our experiments was to create tracks in zircon at 250°C and 7.5 kbar (typical of conditions described for subduction zones) by irradia diamond-anvil cell (DAC) with very energetic ions (Figure 4). Subsequently, track diameters were measured by TEM after pressure release and co tracks induced at ambient conditions. A statistically significant difference was observed (with the high P/T tracks slightly larger), which was relevance for zircon fission track studies employing chemical etching. Other materials have since been studied by the same technique by Lang *e* different material's response at more extreme conditions. The newest development in this direction combines irradiation at ion accelerators a synchrotrons. This will not only allow to create the tracks directly under pressure, but also to study their annealing kinetics at high temperatur pressure.

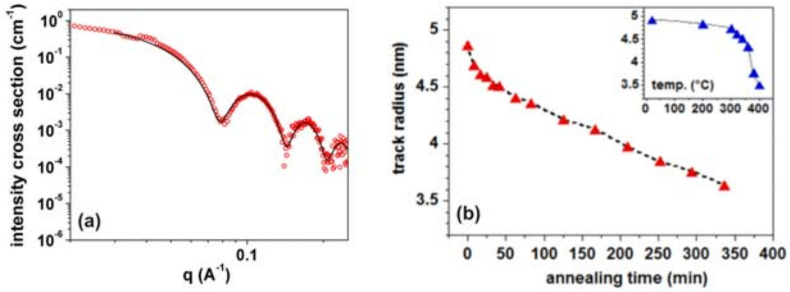


Figure 3 (from Afra *et al*. 2011): Synchrotron SAXS measure Durango apatite after irradiation with 5×10^{10} gold ions/cm^2 o 2.2 GeV. (a) A suitable fit (solid line) to experimental scatte (open circles) provide the radius of millions of latent ion trac single measurement (4.85 ± 0.1 nm). (b) *In situ* annealing a leads to a decrease of track size with increasing time; track-size reduction is also observed with increasing tempera fixed annealing time (*ex situ* annealing).

Using TEM it is not possible to measure track lengths as is commonly done on etched fission tracks. However, the morphology of a fission trac etching because the core of the track as well as some of the surrounding material is removed. The contrasting nature and annealing of latent fissio as documented by Li *et al.* (2011) is a striking example of how the internal structure of a track controls its annealing behavior, and this informatio studies of chemically-etched tracks. Etching can reveal the final, etchable lengths of individual tracks, but details about their annealing behavior changes in morphology during annealing depend on many factors and only some of these are considered in traditional etching based studies (e.g. orientation, etc.). Others, such as track radius, radiation damage in the surrounding material, potential gas content and the uniformity of the track al taken into account. The fission track technique as it is used in geological studies will ultimately benefit from these new insights, just as the consi and the crystallographic orientation of tracks has already improved the fission-track technique in the 80s and 90s.

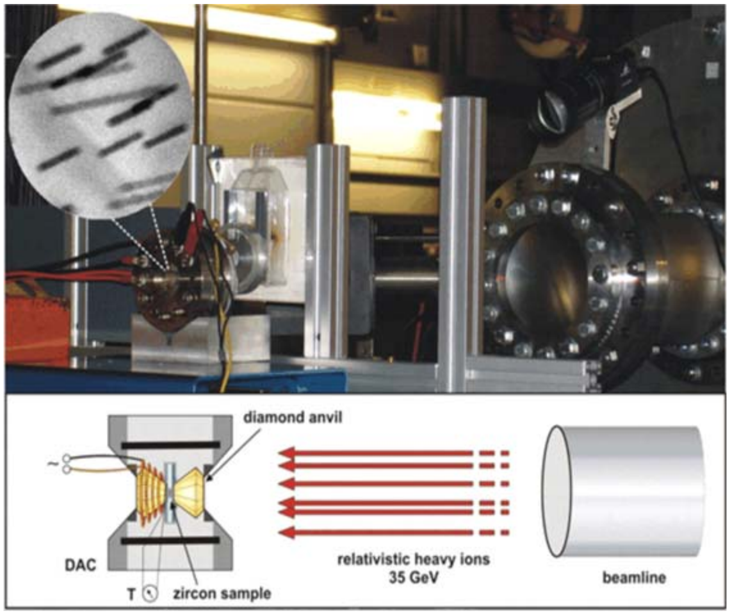


Figure 4 (from Lang *et al.* 2008a): Photograph (top) and schematic illustration (bottom) of the irradiation experiment for exposing pre to a beam of relativistic heavy ions. The very small sample is enclosed in the DAC. The inset is a TEM-image and displays simulated 7.5 kbar and 250 °C. For the irradiation, the DAC is placed in air, 45 cm behind the beamline window. The schematic (not to scale) sho including the resistance heating coil (only shown for one anvil).

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