Scanning force microscopy of natural and heavy ion induced damage in phlogopite

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A new geological dating technique uses alpha-recoil tracks (ART), created by natural α-decay of U, Th, and their daughter products, to determine the formation age of Quaternary volcanic rocks (< 10^6 a). Visualization of etched ART by scanning force microscopy (SFM) enables the access to track densities beyond 10^8 cm^{-2} and thus the extension of the ART technique to an age range > 10^6 a. To simulate natural radiation damage, samples of phlogopite, originating from Quaternary and Tertiary volcanic rocks of the Eifel (Germany) and the Kerguelen Islands (Indian Ocean) were irradiated with U, Ni (11.4 MeV/u), Xe, Cr, and Ne (1.4 MeV/u) ions at GSI, and with Bi (200 keV) ions at JAERI (Takasaki, Japan) [1].

When cleaving natural phlogopite, latent ART near to the cleavage plane form small hillocks imageable by SFM [2]. There occurs also a second kind of hillocks, subsequently called H_N (N denoting 'natural'). Not observable with SFM 8 h after cleaving, the H_N were found when imaging again after 21 h (see Fig. 1). The areal density increase after cleaving can be described by a growth curve not saturating even after 147 h. Also the mean hillock diameter grew with time. The H_N whose origin could not be clarified yet, always appear, and therefore latent ART cannot be used for dating.

To better understand these findings, samples were first annealed at 500 °C for 3.5 h, secondly irradiated and then studied with SFM. A further type of hillocks (denoted by H_T) was found [3], which can be attributed to ion tracks. These hillocks were observed without time delay after irradiation with U and Xe, with areal density about equal to the fluence for U ions, and about 30 % for Xe ions. In the case of Ne, Ni, and Cr ions, no H_T were observed. Within the time period indicated above, the H_N always appeared also on the annealed and irradiated samples. Remarkably, H_N do not transform into etch pits, and thus ART and ion tracks can be separated from them by etching. It is interesting to note that muscovite (white mica) exhibits no H_N.

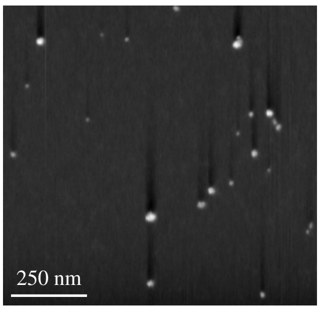

Fig. 1. SFM image of non-irradiated phlogopite 21 h after cleaving. Hillocks appear on an initially flat area.

Hexagonal etch pits are typical of U, Xe, and Cr ion tracks, while the pits of Ni, Ne, and Bi ion tracks are triangular (see Fig. 2). After etching for 900 s with 0.4 % HF, the pits show similar depths for U and Xe, whereas the depths scatter and shrink with decreasing electronic energy loss, *dE/dx*, for Cr, Ni, and Ne. This can be explained by a transition from a continuous to a discontinuous track with decreasing *dE/dx*. The etching rate v_H (parallel to the cleavage plane) depends on the chemical composition of the phlogopite. The etching rate $v_{T'}$ (along the track) becomes greater with increasing *dE/dx* (see Fig. 3) [4].

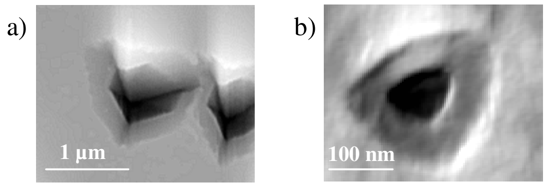

Fig. 2. SFM image of irradiated phlogopite samples after etching with HF. The pits are hexagonal for U (a) and triangular for Bi (b).

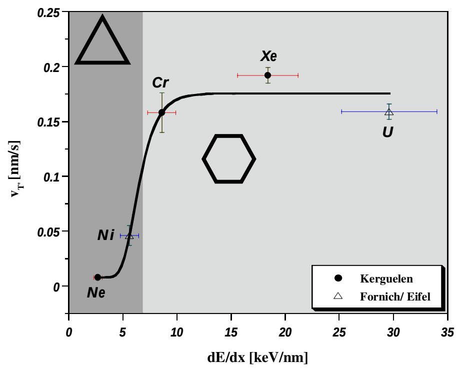

Fig. 3. Etch rate $v_{T'}$ as a function of the energy loss in phlogopite after etching with HF.

[1] Lang, M., *Rasterkraftmikroskopie an natürlichen und Ionen-induzierten Strahlenschäden in Phlogopit*. Diploma thesis, University of Heidelberg, 2001.

[2] von Grabczewski, N., *Untersuchung von Alpha-Rückstoß-Spuren in Biotit mittels Rasterkraft-mikroskopie*. Diploma thesis, University of Heidel-berg, 2000.

[3] Lang, M., Glasmacher, U.A., Moine, B., Müller, C., Neumann, R., Wagner, G.A., *Heavy-ion induced defects in phlogopite imaged by scanning force microscopy*. Surface and Coatings Technology, spec. vol., Elsevier, Amsterdam, accepted for publication.

[4] Lang, M., Glasmacher, U.A., Moine, B., Müller, C., Neumann, R., Wagner, G.A., *Artificial ion tracks in volcanic dark mica simulating natural radiation damage: A scanning force microscopy study*. Nucl. Instr. and Meth. B, in press.

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