Optimized etching of swift heavy ion tracks in calcite*

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Carbonate minerals are of interest for thermochronology (fission-track dating). By irradiating calcite ($CaCO_3$) with swift heavy ions, we perform systematic track etching studies to develop and optimize the track etching process.

For optimal etching several properties need to be fulfilled: (i) Each ion track should lead to a single etch pit, i.e., the etching efficiency should be 100%. (ii) The etching solution should be selective, i.e., the track etch rate should be larger than the bulk etch rate. (iii) The overall etching process should be slow enough to handle the samples in a controllable manner. (iv) The etch pits should be as homogeneous allowing automated analysis by optical microscopy (standard method in geology).

Earlier investigations revealed etched tracks in calcite crystals using HNO_3 [2, 3] or a 1:1 mixture of ethylenediaminetetraacetic acid (EDTA) and 0-5% acetic acid as etchant [1], but the results were far from being optimal.

Natural calcite crystals were irradiated with 11.1 MeV/u ^{197}Au ions at the UNILAC accelerator applying a fluence of 10^6 ions/cm^2. During irradiation, the crystals were covered with a hexagonal mask to provide neighboring irradiated and non-irradiated surfaces.

Knowing that calcite crystals are very sensitive to nitric acid, etching series were performed at two temperatures (15 and 21°C) in highly diluted HNO_3 of concentrations of 0.91, 0.091, and 0.0091%. Regarding shape and size of the etch pits, best results are obtained for 4 s etching in 0.091% HNO_3 at 15°C. As expected [3], the etch pit geometry depends on the solution: tracks etched in EDTA + 5% acetic acid have an elongated hexagonal shape while 0.091% HNO_3 produces pentagon-shaped pits (Fig.1).

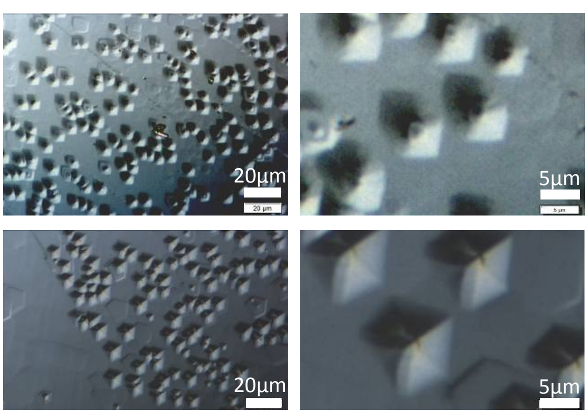

Fig.1: Optical images of etched tracks in calcite crystals at different magnifications: (top) 20 s etching in EDTA + 5% acetic acid; (bottom) 4 s etching in 0.091% HNO_3. Note the different shape of the etch pits.

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Figure 2 compares the size of the etch pits as a function of etching time for HNO_3 and EDTA. Compared to EDTA, the etch rate of HNO_3 is by a factor of about five higher. Besides that, the development of the etch pits regarding length and width is the same, both show linear growth with length > width.

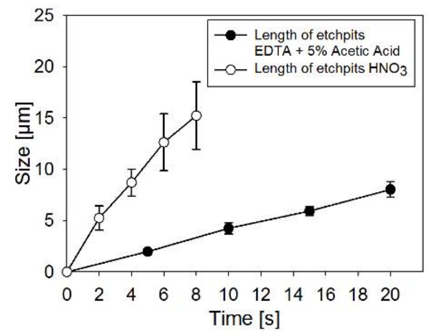

Fig. 2: Length of etch pits as a function of etching time for two etching solutions at 21°C: (open symbol) EDTA + 5% acetic acid and (full symbol) 0.091% HNO_3

Another promising aspect of HNO_3 as etching solution is given by the fact that the contrast of the etch pits in calcite under the optical microscope is excellent. This allows automated counting by image analyzing software (Fig. 3), an important issue for fission track dating.

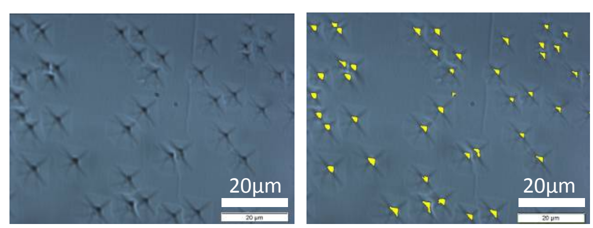

Fig.3: Optical image of HNO_3-etched tracks recorded in transmission mode. The excellent contrast allows automated counting indicated by the white detection marks (right image) produced by the analyzing software.

In conclusion, HNO_3 is a very promising option for etching heavy ion tracks in calcite crystals. The solution is easier to produce and it fulfils the above mentioned requirements for thermochronological track application.

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