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Revelation of stepped dislocations in amethyst crystals by hydrothermal etching

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Abstract

Triangular etch pits on rhombohedral surfaces due to hydrothermal etching are reported. Our experiments show that the hydrothermal etch pits on rhombohedral surfaces of amethyst have the same dislocation origin they have for quartz. We demonstrate that for a good number of cases successive etching results in the development of another pit away from the geometrical center of the original point-bottomed pits and this tiny pit grows bigger and bigger as the etching is carried further. Resulting terracing of the pits on prolonged etching is illustrated. These results of prolonged etching are attributed to the presence of stepped configuration of dislocations in amethyst crystals.

Introduction

One of the means for direct observation of dislocations is to study etched surfaces of a crystal. Several investigators [Tsinzerling and Mironova (1963, 1965), Joshi and Vagh (1967), Joshi *et al.* (1970), and Joshi and Kotru (1969)] have reported studies of dislocations in quartz and amethyst crystals using selective etch methods. In this paper we discuss some typical observations made on rhombohedral surfaces of natural amethyst crystals etched by a hydrothermal method.

Experimental

Crystals cleaved along rhombohedral planes were etched in a leak-proof steel bomb, which after having been partially filled with distilled water was heated to 260°C in a muffle furnace. After the required time of etching the crystals were thoroughly cleaned. The cleaved surfaces were coated with thin films of silver in a vacuum-coating plant and then examined under a metallurgical microscope.

Etching of amethyst

Figures 1a and 1b show the etch patterns produced by the hydrothermal method (etching time 8 hours) on a pair of matched rhombohedral cleavages of an amethyst crystal. On each of these two photomicrographs one finds both shallow flat-bottomed and deep point-bottomed etch pits. Corresponding to every point-bottomed pit on one cleavage face there is a point-bottomed etch pit on each matched cleavage, whereas no such correspondence is observed for the flat-bottomed pits. On prolonged etching the point-bottomed pits were observed to get deeper and deeper whereas the flat-bottomed pits vanished. These observations suggest a dislocation origin for the formation of point-bottomed etch pits and zero-dimensional defects responsible for the formation of shallow point-bottomed pits.

Stepped dislocations

Etching techniques have been successfully employed by several investigators to work out the configuration of dislocations within the body of a given crystal [Amelinckx (1956), Bontinck (1957), Sagar and Faust (1967), Bhagavan Raju *et al.* (1969), and Hari Babu and Bansigir (1969)]. Etch pits produced by the hydrothermal method are ideal for such type of study on amethyst crystals and were utilized in the present study.

Figure 2a shows triangular etch pits on a rhombohedral cleavage of amethyst etched at 260°C for 8 hours. Further etching of additional 8 hours made the pits grow in lateral dimensions and in depth, as shown in Figure 2b. The pits marked *X* and *Y* have

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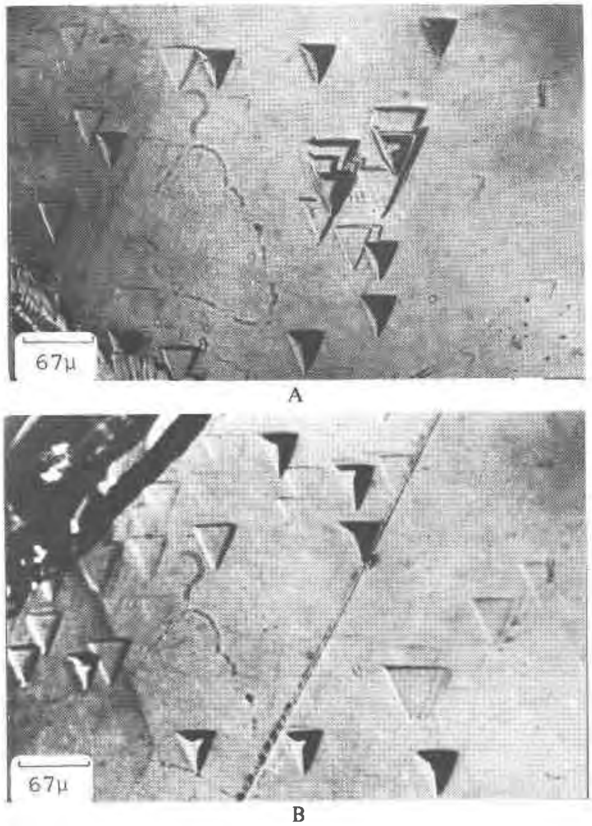

Fig. 1. Exact correspondence of point-bottomed etch pits and non-correspondence of flat-bottomed pits produced by hydrothermal etching on matched rhombohedral cleavages of amethyst.

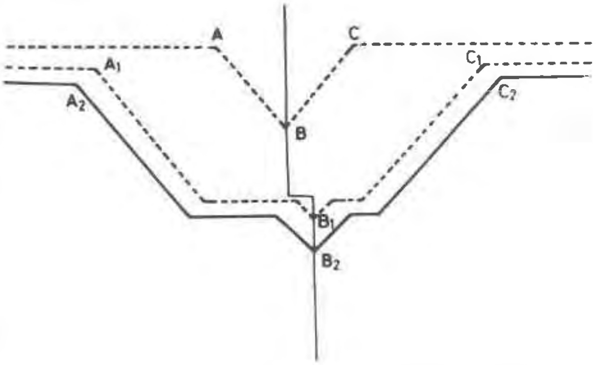

Fig. 3. Schematic diagram illustrating the profile of a pit due to stepped configuration of a dislocation.

become flat-bottomed in the second stage of etching. Close examination of Figure 2b shows that a small pit has nucleated inside each of the pits marked *X* and *Y*. All the pits, including the two small pits that originated during the second stage, have again grown further after another stage of etching of 8 hours, as shown in Figure 2c. The development of the small pits inside the flat-bottomed ones may be attributed to the presence of stepped dislocations inside the crystal, as explained in the schematic diagram in Figure 3. The thin solid line in the schematic diagram in Figure 3 represents the dislocation line, which runs perpendicular to the surface for some distance in the body of the crystal, turns parallel to the surface and

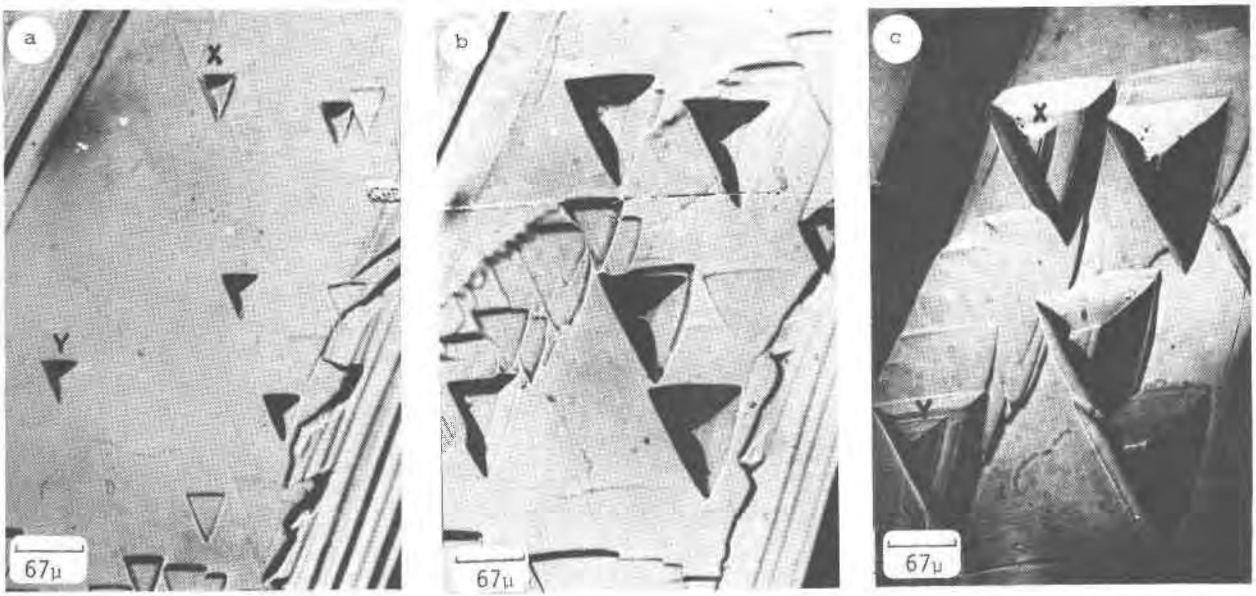

Fig. 2. (a) Point- and flat-bottomed pits produced by hydrothermal etching on a rhombohedral cleavage of amethyst (etching time 8 hours). (b) Etch pits of (a) after the second stage of etching, demonstrating nucleation of a small pit inside the pits marked *X* and *Y* (etching time 16 hours). (c) Same region of (a) and (b) after prolonged etching of 24 hours, illustrating further development of newly nucleated pits of (b) and further flattening of bigger ones *X* and *Y*.

again changes its direction to emerge in the original direction. When such a dislocation is etched, the profile of the pit first obtained will be shown by ABC in Figure 3. $A_1B_1C_1$ represents the pit profile at the end of the second stage of etching. Here the original pit becomes flat-bottomed, and a new tiny pit originates at a point slightly displaced from the geometrical center of the flat-bottomed pit. If further etching is allowed, one finally finds $A_2B_2C_2$ as the profile of the pit. Since the dislocation line runs deep into the body of the crystal, this newly formed pit will grow wider and deeper till the proposed configuration repeats. This type of configuration of dislocations in crystals as delineated by selective etching is here reported for the first time.

Conclusions

(1) Hydrothermal etch pits on the rhombohedral surfaces of amethyst crystals are very well defined in shape and structure. The point-bottomed etch pits formed on their rhombohedral surfaces have a dislocation origin.

(2) The variation in etch-pit morphologies with continued etching suggests that there are jogs in the dislocations and that some dislocations have branches.

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