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Electron microscopic and optical studies of prism faces of synthetic quartz

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Spirals on quartz are very seldom seen. The centre of the spirals and screw ledges are studied in detail using electron and optical microscopic techniques. Unusual profiles of spirals have been found with overhanging kinks on one side of the centre of these spirals. It is suspected that the complicated structures might have been developed after the completion of the growth of the crystals. It is also conjuctured that such structures would arise when growth-inhibiting substances are adsorbed on the crystal faces and result in imperfect crystals.

1. Introduction

There are several publications describing the observations of growth spirals on many different crystal faces. These features are studied in detail to investigate the nature and history of the crystal.

The observations of growth spirals on natural and synthetic quartz habit faces are very rare [1–3]. One of us (A.S.V.) has observed such growth pattern on $(10\bar{1}0)$ faces of synthetic quartz [4]. Owing to special circumstances it was difficult to investigate them in detail when they were observed for the first time.

In the present paper the investigations of the growth spirals [4] have been advanced further, employing electron and optical microscopic techniques. The study reveals some unique characteristics of centres of the spirals which have not been reported earlier.

2. Experimental

Proper selection and execution of high resolution replication techniques were imperative in the electron microscopic investigation of the centres of the spirals. Several replication procedures were tried. Of these, the single step carbon–platinum technique was selected for its optimum resolution. Since carbon is evaporated directly onto the specimen, a release agent* had to be used prior to its deposition. The details of the procedure are as follows.

The sample was thoroughly cleaned in an ultrasonic generator in a 50/50 solution of water/ethyl alcohol for a period of 5 min and dried with warm air. The 3/16 in. long point of a carbon rod was dipped into a 30% solution of release agent in water and then air dried. This dipping and drying was repeated four times. By then a thin hazy layer of the release agent was visible on the thinned portion of the carbon rod. The sample was placed in the evaporator and positioned to a shadowing angle of 12°. After pumpdown to 10^{-5} Torr, release agent-coated carbon was heated slowly by raising the control to between 10 and 15 V (~2 A) and leaving it at this setting for approximately 5 min, by that time the release agent boiled off. The control was turned down and switched over to the evaporation of the platinum–carbon pellet for shadowing. Then the 3/16 in. length of the carbon rod was evaporated to form a 200 Å thick film on the crystal face. The specimen was removed from the evaporator and a few drops of a 4% solution of Parlodion in amyl acetate were put on the surface of the specimen and allowed to dry to strengthen the carbon film. The carbon–Parlodion film was separated from the surface of the crystal by holding it over boiling water and slowly teasing the

*"Victawet", a proprietary wetting agent, available from one or more suppliers of electron microscopy materials and accessories.

film off with forceps. The carbon film side was reinforced with melted wax. After solidification, a 1/8 x 1/8 in. square containing the area of interest was cut out. It was placed into a vessel filled with amyl acetate to dissolve off the Parlodion film. Then the carbon–wax film was lowered onto the surface of almost boiling water and left there until the wax melted, and washed repeatedly in toluene until all traces of wax were removed. The cleaned carbon replica was placed on an electron microscope grid and examined. A light-microscopy interferometric technique was also used to understand more about screw ledges and related features.

3. Observations and discussion

Fig. 1 is an optical micrograph of a region of a prism $(1\,0\,\bar{1}\,0)$ face of synthetic quartz; it has been taken out of focus in order to see the details of the region better. Fig. 2 is a corresponding light-microscopy interferogram taken above the same region shown in Fig. 1. The structure marked "*S*" in Fig. 1 is a rectangular spiral as shown in Fig. 3, which is a positive reflection phase-contrast micrograph. It is evident from Fig. 2, that the spiral is an elevated feature. This was found to be so for all the spirals observed on the prism faces of synthetic quartz [4]. It may be noted that the details of the spiraled pyramids and other related features observed were on such microscales that it was almost impossible to view them without the use of phase-contrast microscopy at higher magnifications. Consequently, multiple beam interferometric techniques were not used for quantitative measurements.

In Fig. 2, the step height of the screw ledge marked "XY" (Fig. 1) ranges from 0 to 1820 Å. Here all the vertical displacements of the points on the screw ledge were plotted and it was confirmed that the steps were straight. Thus the steps run to zero height at one end and to a maximum height at the other. It was also verified that the step was not due to optical reference flat, because the reference flat could be arranged in such a way that the fringes surrounded the end-point of the step, thereby concluding unambiguously that the step had ended. Therefore, the point where the step ends must be considered as the point where a screw dislocation emerges from the surface.

Being satisfied of the existence of screw dislocations and spirals on the prism faces of synthetic quartz, an effort was made to study the centres of the spirals. One can see from Fig. 3 that the centres of two unlike spirals are square in nature. The details of one of the centres is shown in Fig. 4, which is an electron micrograph. The centre is anti-clockwise with a flat region and the steps varies in width depending on the *E*, *W*, *N* and *S*

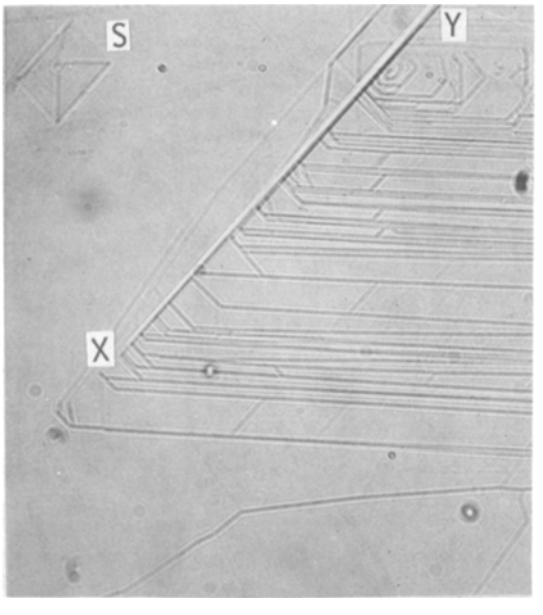

Figure 1 Prism face of a synthetic quartz showing a spiraled pyramid marked "S" and a screw dislocation ledge marked 'XY', × 100.

Figure 2 A two beam reflection interferogram revealing the topography of the corresponding regions of Fig. 1, × 100.

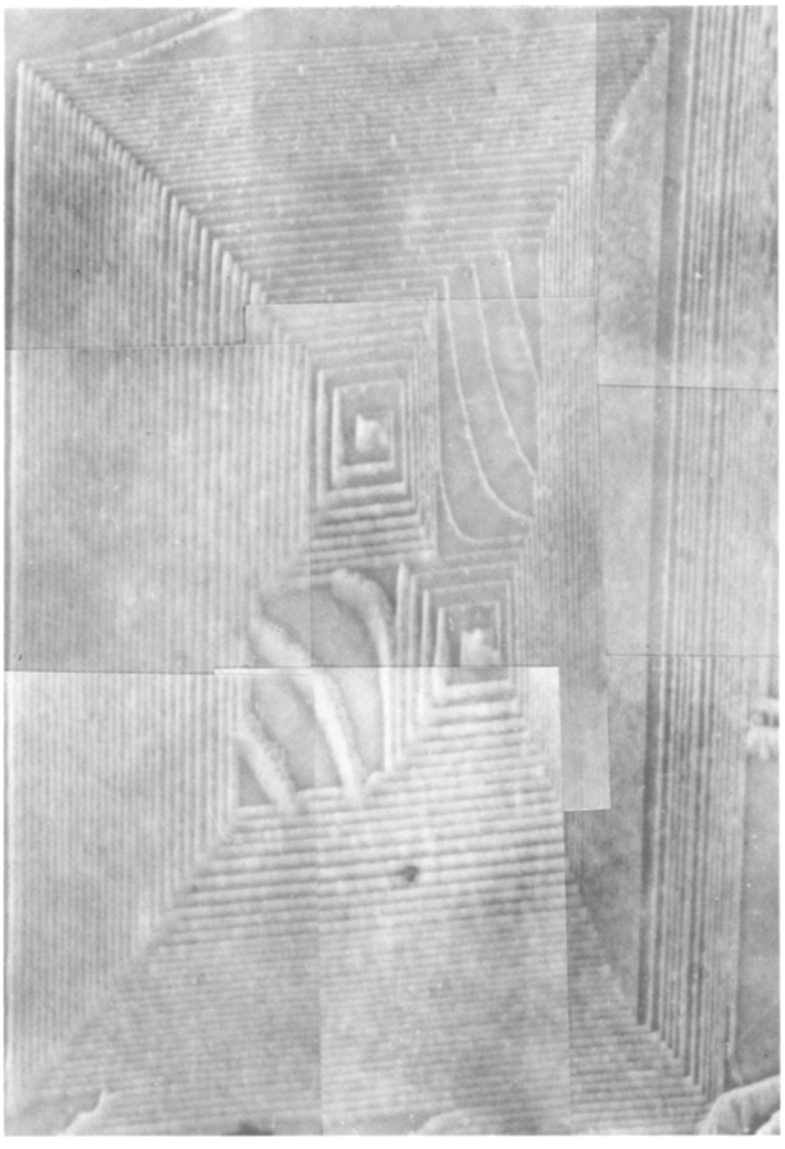

Figure 3 Positive phase-contrast reflection photomicrograph showing details of structure "S" from Fig. 1, × 1000.

directions. The steps are narrower in the N direction, and in between them are depressions which become progressively narrower away from the centre. The width of the steps in different directions are $E = \sim 2.1\,\mu m$, $W = \sim 2.2\,\mu m$, $N = 0.5$ to $0.6\,\mu m$ and $S = \sim 1.5\,\mu m$. The height of the steps measured (shadowing angle 5:1) is equal to 80 Å. Fig. 5 represents an electron micrograph of steps at a higher magnification. These steps are from the N direction of Fig. 4. The steps are kinked, and this is in agreement with the theoretical prediction [5].

The profile of the steps in Fig. 4 is shown schematically in Fig. 6c where overhanging kinks in the N direction are seen while the rest of the directions have smooth steps. Usually steps of multiple nature have been observed on various crystals. The literature cites three possibilities for the nature of multimolecular steps, namely (1) the step is sharply defined without kinks (Fig. 6a), (2) the step is kinked and kinks appear to be homogeneous as Fig. 6b indicates, and (3) the step is kinked and shows overhangs such as those on the right-hand side of Fig. 6c. The overhangs are

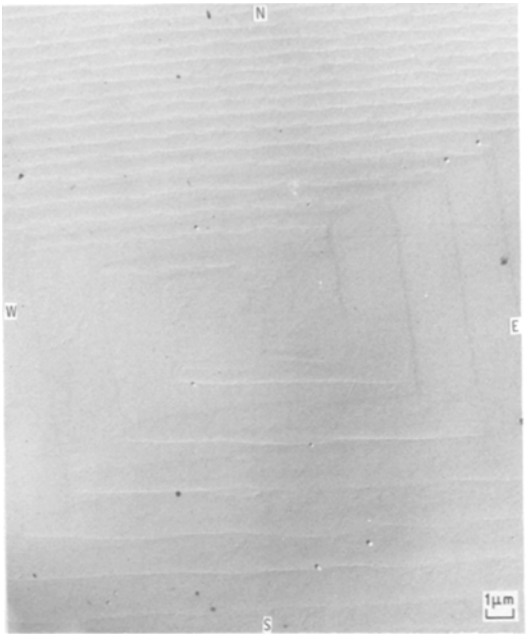

Figure 4 Electron micrograph revealing the details of the centre of the anticlockwise spiral from Fig. 3.

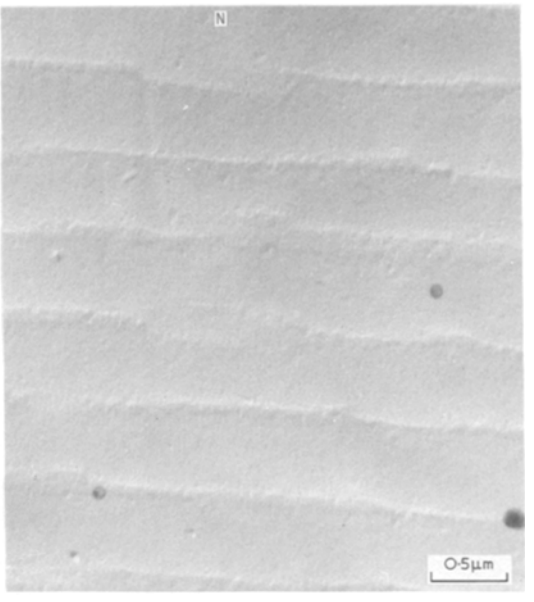

Figure 5 Electron micrograph showing steps from *N* direction of Fig. 4.

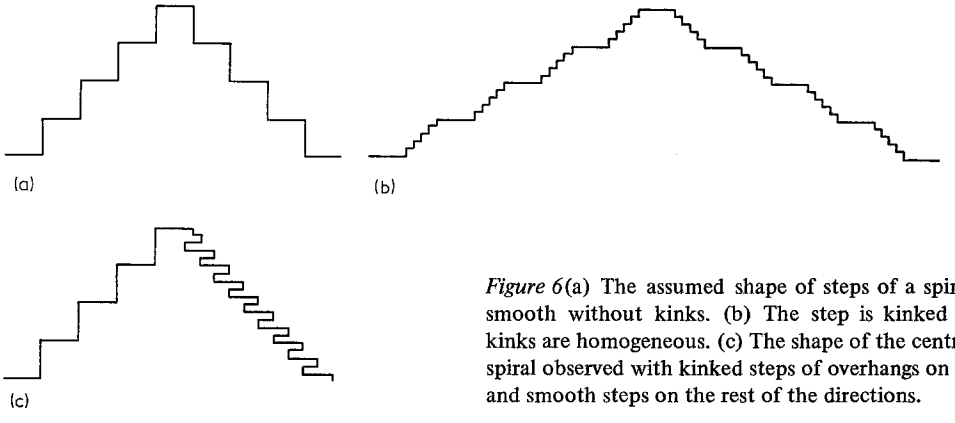

Figure 6(a) The assumed shape of steps of a spiral being smooth without kinks. (b) The step is kinked and the kinks are homogeneous. (c) The shape of the centre of the spiral observed with kinked steps of overhangs on one side and smooth steps on the rest of the directions.

highly improbable because they have a substantially greater surface area than other profiles, and therefore the surface free energy may be greatly reduced by a small displacement of matter. Profiles like those in Fig. 6c could only result when a strongly growth-inhibiting substance is adsorbed on the crystal surface, and continuing growth with such a step profile would produce a very imperfect crystal. In the present case it is conjectured that the growth-inhibiting impurities have been adsorbed at the centre of the spiral and in the *N* direction. It is also suspected that the complications of such structures may develop at and after the cessation of growth, rather than represent the profile during steady continuing growth.

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