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Fibers of dumortierite in quartz

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Abstract

During recent experiments involving the hydrothermal etching of quartz samples obtained from different locations, scanning-electron microscopy of etched samples revealed that many of the geometric etch features developed around small, included-mineral fibers. The fibers were most abundant in samples of rose quartz from the Ruby Range, Montana. No fibers were observed in samples of optically pure quartz from Arkansas. The fibers, which appear to be randomly oriented in the samples, measure about 0.1–0.4 μm in width by 0.1–0.2 μm in thickness and possibly extend hundreds of micrometers or millimeters in length. Small masses of the fibers were isolated from rose quartz by digestion in concentrated HF followed by filtration; these fibers were identified by X-ray diffraction and transmission-electron microscopy as dumortierite.

Introduction

Although generally rare, dumortierite, $(Al,Fe^{3+})_7O_3(BO_3)(SiO_4)_3$, is reported to occur at numerous localities throughout the world. Most of the reported occurrences are in rocks such as pegmatites, dikes, and adjacent metamorphic rocks associated with late-stage pneumatolytic fluids. Grametbaur (1959) has compiled numerous references regarding these localities.

On a macroscopic scale, dumortierite appears as aggregates of fibrous or columnar crystals that display vivid color, commonly blue or violet and sometimes pink. Distinctive optical properties include strong pleochroism and negative elongation.

During recent studies involving the etching of quartz from a variety of locations, examination of etched surfaces by scanning-electron microscopy (SEM) revealed single, included fibers emanating from many of the etch features of some samples (Fig. 1). The fibers, which measure about 0.1–0.4 μm in width and 0.1–0.2 μm in thickness, are too small to be seen with a normal petrographic microscope and are exposed for viewing by SEM only following careful etching and sample treatment.

Sample selection and preparation

The fibers were discovered while performing experiments involving the hydrothermal etching of quartz (Hicks, 1985). Various quartz samples were crushed to a grain size of 60–80 mesh and etched in distilled water at 280°C and 1.2-kbar pressure for 12–35 h in a cold-seal pressure vessel. Some samples were also etched in dilute HF. Following the etching treatment, the samples were usually rinsed with distilled water, coated with gold, and examined by SEM.

The fibers became evident while studying the etching response of rose-colored quartz from the Ruby Range, Montana. We should emphasize that the samples were *not* subjected to ultrasonic treatment as part of the preparation for the SEM study. Although ultrasonic treatment is often recommended for cleaning samples prior to viewing by SEM, it is apparent that even brief treatment can dislodge the fibers from etched samples.

In an attempt to determine if the fibers occur in other varieties of quartz, we examined a few other samples that were immediately available to us. These included pegmatitic quartz from the Black Hills of South Dakota, optically pure vein quartz from Arkansas, and quartz from a granite of unknown origin.

SEM observations

The results of the etching experiments are shown in Figure 1. Figure 1a shows individual fibers emanating from the centers of etch pits developed in quartz samples from the Ruby Range. Fibers very similar in both morphology and abundance were also observed in samples of quartz from the Black Hills. The fibers appear to be randomly distributed throughout the samples. Whether they are related to any growth feature of the quartz or cross grain boundaries could not be established by SEM techniques. We have not examined other minerals for their presence.

Coiled fibers were rarely observed in samples of quartz from the Black Hills (Fig. 1b). Their occurrence suggests that the fibers predate the quartz crystallization, i.e., they were captured by growing quartz crystals.

The fibers were also present but appeared to be far less numerous in quartz grains from the granite sample. Although severe etching was induced in these samples, it is more than likely controlled by other types of dislocations and defects. Etching was also induced in samples of Arkansas quartz, but no fibers were observed.

Identification of the fibers

The etching experiments revealed that the fibers were insoluble in HF. Thus, small quantities of the fibers were easily extracted from quartz samples by digesting samples of crushed quartz in HF. A 15-g sample of rose-colored

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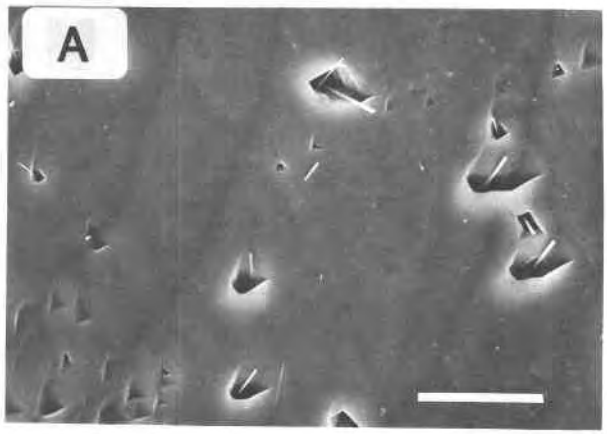

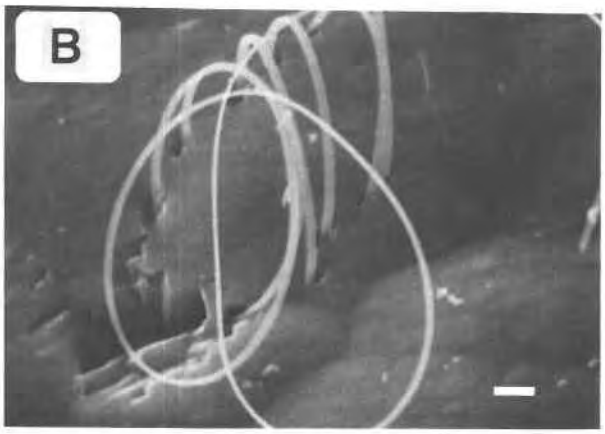

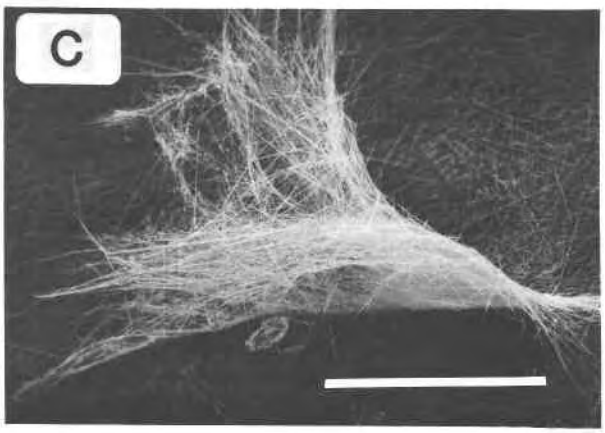

Fig. 1. (a) Fractured surface of rose quartz from the Ruby Range, Montana, etched for 12 h in distilled water at 280°C and 1.2-kbar pressure. Well-developed etch pits surround fibrous inclusions of dumortierite. Bar scale is 10 μm. (b) Coiled dumortierite fiber exposed at the surface of quartz from the Black Hills, South Dakota. The sample has been etched for 35 h in distilled water at 280°C and 1.2-kbar pressure. Bar scale is 1 μm. (c) Fibers collected following the digestion of rose quartz from the Ruby Range, Montana, in concentrated HF at room temperature for 4 d. Bar scale is 100 μm.

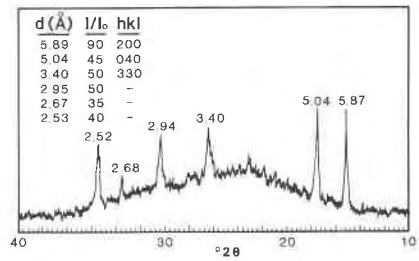

Fig. 2. X-ray diffractogram of fibers collected from rose quartz from the Ruby Range, Montana. Selected ASTM X-ray data for dumortierite are given in the upper left corner.

quartz from the Ruby Range was treated in concentrated HF for a few days at room temperature, leaving a residue of rose-colored material. The residue was collected by first diluting the HF with distilled water and then filtering the suspension through a filter of 0.1-μm pore size. This process yielded a small mass of fibers (Fig. 1c) that was later analyzed by X-ray diffraction and transmission-electron microscopy (TEM).

X-ray diffraction analysis of the matted sample yielded a diffractogram compatible with dumortierite (see Fig. 2). The *d* spacings and intensities obtained correspond very well to the X-ray powder data for gem-quality dumortierite reported by Claringbull and Hey (1958).

The results of the TEM analysis are also compatible with a dumortierite composition for the fibers. The intensity ratios of elements relative to Al determined in seven fibers were calculated from TEM X-ray energy-dispersive spectra (EDS) and are given in Table 1. The intensity ratios show Al and Si as the major elements in the fibers. The only additional elements found in the fibers were trace amounts of Fe and Ti. Although B also occurs as a major element in dumortierite, it is too light to be detected by TEM-EDS analysis. As shown, the average Si/Al ratio for the fibers is 0.46. Assuming an ideal chemical formula of $(Al,Fe^{3+})_7O_3(BO_3)(SiO_4)_3$ for dumortierite gives a Si/Al ratio of 0.43, which is in reasonable agreement with the analytical value.

The electron-diffraction patterns obtained from various fibers are not inconsistent with an orthorhombic symmetry, but a large number of superlattice reflections made it difficult to determine exact lattice parameters. The periodicity along the fiber axis, however, was measured to be 4.71 Å. Moore and Araki (1978) reported the cell constants of dumortierite as $a = 11.828$ Å, $b = 20.243$ Å, and $c = 4.7001$ Å. Similar values have been reported by Claringbull and Hey (1958).

Although we did not specifically analyze the fibers in samples other than those from the Ruby Range, the similarity in morphology indicates that they also consist of dumortierite. Other possible candidates that commonly occur as inclusions in quartz, such as rutile and tourmaline, can be ruled out. Rutile is extremely soluble in acid and would not survive the HF treatment. Although tourmaline is only slightly soluble in strong acid, a limited

Table 1. Intensity ratios of elements relative to Al

Fiber no.	Al	Si	Ti	Fe
1	1.00	0.40	0.04	0.01
2	1.00	0.45	0.04	0.01
3	1.00	0.47	0.04	0.02
4	1.00	0.46	0.05	0.02
5	1.00	0.45	0.01	0.01
6	1.00	0.45	0.02	0.01
7	1.00	0.53	0.05	0.01
Average	1.00	0.46	0.04	0.01

Note: Determined from TEM X-ray energy-dispersive spectra of selected fibers extracted from rose quartz from the Ruby Range, Montana.

number of SEM-EDS analyses performed on fibers in other samples failed to detect the presence of any elements other than Al and Si that might be associated with tourmaline.

Conclusions

It is obvious that the fibers, which apparently penetrate tens of micrometers or more into the sample, exert a major control on the etching process. We suspect that in samples which contain abundant fibers, many of the etch pits that appeared to contain no fibers probably developed around fibers that were dislodged during sample handling. Hence, the presence of abundant fibers could significantly alter the chemical and physical properties of quartz.

The results of this study suggest that the abundance of dumortierite fibers in quartz varies with the mode of origin of the quartz. Although we did not collect the samples ourselves, the Ruby Range and Black Hills samples are believed to be pegmatitic in origin. Macroscopic dumortierite and other B-bearing minerals such as tourmaline have been noted to occur in the Ruby Range (Graham and Robertson, 1951). Thus, it is not surprising that these samples exhibited the greatest density of microscopic dumortierite fibers.

In contrast, no visible evidence was obtained for the existence of dumortierite fibers in the samples of vein quartz from Arkansas. The vein quartz is thought to originate by precipitation from hydrothermal fluids seemingly unrelated to magmatic sources. To our knowledge, no other B-bearing phases are associated with these veins.

Although the quartz from the granite sample contained a modest amount of the fibers, this does not suggest that all granites might be likely to contain dumortierite. Rather, the abundance of dumortierite is more likely controlled by the bulk composition of the fluid or melt and the phase relations that exist. At the same time, however, we suspect that the occurrence of dumortierite fibers in quartz may be more prevalent than previously thought. Chemical analyses of igneous rocks that show no optical evidence for the presence of B-bearing minerals, for example, sometimes show anomalous B contents that might possibly reflect the presence of the fibers.

Because the density of dumortierite fibers in quartz may be associated with the mode of origin of the quartz, etching studies such as those described above may be of practical use in determining the provenance of sedimentary quartz. For example, one could compare the abundance of dumortierite in sedimentary quartz grains with that in quartz collected from suspected source regions.

Finally, we should note again that the dumortierite fibers extracted from the Ruby Range samples by HF digestion were the same rose color as the bulk quartz. Although dumortierite may not be the major coloring agent of many rose quartz specimens, we strongly suspect that it is the cause of coloration in the Ruby Range samples.

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