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Thermal transformation of anomalously biaxial dimetric crystals

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Abstract

Certain dimetric crystals exhibit anomalous biaxiality (optic axial angle). Changes in biaxiality as a function of temperature for several dimetric crystals—elbaite tourmaline, jeremejevite, $Al_6B_5O_{15}(F,OH)_3$, and synthetic emerald (beryl)—have been examined and documented. Measurements of the orientation of the optical axial plane and $2V$ were made in crystal slices cut normal or nearly normal to the c axis of each specimen. Measurements at room temperature were followed by measurements at temperatures between −145° and 800°C. In some instances, crystals which are anomalously biaxial can have the internal stress removed by heating or cooling. The temperature needed to produce uniaxiality when the material is cooled back to 25°C varies from mineral to mineral and even within individual minerals, depending on localized stress conditions. Chilling may also remove stress in an anomalously biaxial mineral, which lowers the annealing temperature.

Introduction

Certain isometric and dimetric crystals exhibit anomalous biaxiality (optic axial angle). Possible origins for this phenomenon have been discussed by Foord and Mills (1978). This paper documents changes in biaxiality as a function of temperature in several dimetric crystals.

A summary of temperature effects was given in Berek (1953). Applications of hot- and cold-stage microscopy, and some of the stages available, have been discussed by Hartshorne and Stuart (1970). Optical interference figures at high or low temperatures, particularly of anomalously biaxial minerals, appear not to have been investigated.

Three minerals studied by Foord and Mills (1978) were used as samples for this report. They are elbaite tourmaline from the San Diego Mine, Mesa Grande district, San Diego County, California; two samples of jeremejevite, one from Mt. Soktuj, Dauria, USSR (NMNH B12207), the other from Southwest Africa (NMNH 128518); and a synthetic emerald.

Experimental procedure

The orientation of the optic axial plane and the value of $2V$ obtained using Tobi's (1956) method were mapped at various points in doubly-polished crystal slices ranging from 0.04 mm to 1.1 mm in thickness and cut normal or nearly normal to the c axis. The measurements at room temperature were followed by measurements at temperatures between −145° and 550°C with a modified Chaix-Meca[1] heating/freezing stage. The lower temperatures were attained by passing dry nitrogen through a Dewar flask filled with liquid nitrogen. Heating rates were about 2°/minute for the elbaite slices and about 8°/minute for the other specimens. After the desired temperature was reached and maintained, $2V$ of the sample was photographed and measured. Temperatures above 550° and to 800°C were achieved with a muffle furnace, but in each case the samples had to be removed and cooled to room temperature for observation.

Results

Jeremejevite

For a prismatic crystal of jeremejevite, $Al_6B_5O_{15}(F,OH)_3$, from Swakopmund, Southwest Africa, $2V_x$ ranged initially from 0° to 28° (±2°) at various points in the slice (0.3 mm thick). The portions with the largest $2V$ were sectors outside of a central uniaxial core. A polished plate from this crystal was heated and successive measurements taken

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[1] The use of brand names in this report is for descriptive purposes only and in no way constitutes endorsement by the U.S. Geological Survey.

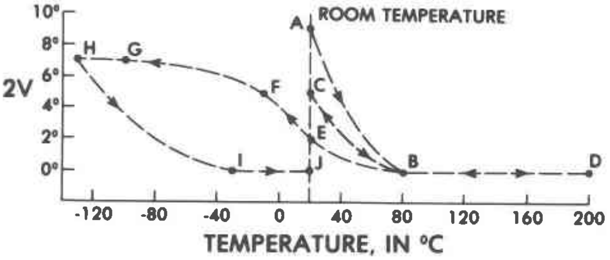

Fig. 1. Change in optic axial angle as a function of temperature for tourmaline #1. Heating and cooling rate approximately 2°C/min. Point A is $2V$ at room temperature, point B is $2V$ when heated to 80°C, point C is $2V$ when cooled to room temperature, point D when sample heated to 200°C, point E when cooled to room temperature, points F, G, and H when cooled down to −130°C, and points I and J when taken to room temperature.

between 24° and 550°C, with no observable increase or decrease in the size of the $2V$ upon either heating or cooling back to room temperature. However, the color of the rim material deepened from an initial cornflower blue to an indigo-navy blue. Microprobe analyses showed iron in the rim portion, which presumably became oxidized. A second plate (0.75 mm thick) from the crystal, after heating in a muffle furnace at 800°C (±25°) for 16 hours, was almost completely bleached of the blue color and was an opaque white after treatment.

A 0.4 mm thick slice of jeremejevite from the USSR had a maximum $2V_x$ of 33±2° prior to heating. During heating to 400°C $2V$ decreased slightly, with no further change to 550°C. When this plate was cooled (at about 2°/min) to room temperature, $2V$ returned to its original size. The sample was then heated in a muffle furnace at 800°C for 8 hours and air-quenched to room temperature. The sample remained transparent, but $2V$ decreased from 33±2° to 6°±2°.

Tourmaline (elbaite)

Four samples of tourmaline from the San Diego Mine were prepared as doubly-polished plates, cut normal or nearly normal to *c*.

The first tourmaline, a 'pencil' 0.15 mm thick and twinned on $\{10\bar{1}1\}$, had three prominent arms of pink material within a colorless core. Surrounding the central twinned core was a concentric rim of pale green elbaite (Foord and Mills, 1978, Fig. 2b). Prior to heating or freezing, $2V_x$ at various points ranged from 0° to a maximum of 14 (±2°). A point was selected where the $2V_x$ was uniform and equal to 9±2°. The sample was heated to 80°C and $2V$ (Fig. 1, point A) decreased to 0° (point B). When the sample had cooled (at about 2°/min) to 20°C (point C), $2V$ had increased, but only to 5°, indicating that when the mineral was heated to 80°C, the process was only partially reversible at that rate of cooling. The sample was reheated and $2V$ measured at 20° increments between 20° and 200°C (point D). By 80°C the crystal was uniaxial, and when it was cooled (at about 2°C/min) to 20° from 200°C, $2V$ opened up only to 2° (point E). Upon cooling the sample to −10°C, $2V$ opened up to 5°, and thence to 7° by −100°C (points F and G). No further change was noted upon cooling to −130°C (point H). The sample was again heated, to observe the temperature at which $2V$ returned to 0°. At approximately −30°C (point I), $2V$ was essentially 0° and remained there until room temperature was reached (point J). The sample was not moved or disturbed during this treatment. All lighting conditions were standardized. Following this treatment, the crystal of tourmaline remained uniaxial throughout, except near the sharp twin boundaries, indicating the internal stress had been annealed.

The second elbaite, a first-generation pocket crystal 0.2 mm thick, contained an achroite core grading outward to pink and then green rim material. A point was selected within the achroite portion of the crystal where $2V_x$ was 3°. Upon heating to 150°C, $2V$ became almost 0°, and at 300°C the material became truly uniaxial. Cooling (at about 2°/min) to −100°C caused $2V$ to increase to 7°. When the sample was returned to room remperature (again at about 2°/min), $2V$ became nearly identical to that at the start. This 'primary' crystal thus behaved generally like the first crystal but over a different temperature range.

A third sample of 'primary' achroite tourmaline behaved identically to the 'pencil' tourmaline. Upon heating, $2V$ decreased perceptibly at 40°C from 7° to 5° (±2°), and at 80°C the material became uniaxial. The sample was cooled (2°/min) to room temperature and $2V$ increased to 6° but did not regain its initial magnitude of 7°, indicating that a slight amount of stress had been removed. The sample was then cooled to −80°C and $2V$ increased to 9°. Upon return to room temperature the sample became uniaxial.

A fourth sample of tourmaline, a 'primary' crystal of achroite (as was no. 2), was examined in an area of maximum birefringence. In areas of uniform $2V$, the crystal behaved as did the previously-studied tourmalines and became uniaxial at 80°C; however, upon heating to 550°C, the crystal developed areas of localized strain, as represented by a large $2V$ along linear domain boundaries and fractures. There was no appreciable change up to and beyond 550°C or after subsequent cooling to 25°C (at 2°/min).

Beryl (emerald)

No changes in $2V$ as a function of temperature were observed in a synthetic emerald grown by flux melt techniques. At room temperature, the crystal contained areas with $2V_x$ up to 7° (±2°). An area within the central portion of the zoned crystal (Foord and Mills, 1978, Fig. 4b), adjacent to an obvious strain center, was chosen for examination. The sample was heated to 550°C and returned to room temperature, with no resulting change in $2V$. The sample was then heated in a muffle furnace at 800°C for 16 hours and no changes were noted upon cooling.

Conclusions

An anomalously biaxial crystal in certain instances can have the internal stress removed by heating or cooling. If it is heated only by the amount needed to reduce $2V$ to 0°, the elastic energy will be such that the crystal will again be strained upon cooling but not as much as it was initially. If, however, the crystal is heated well above the temperature needed to make it appear uniaxial, the stress may be removed, in which case the mineral will remain uniaxial down to room temperature. The minimum temperature needed for this varies from mineral to mineral and even within individual minerals, depending on localized stress conditions.

An anomalously biaxial mineral can also have the strain removed by chilling, that is, stress is placed on the mineral in an opposite sense when freezing (contraction) than when heating (expansion). Upon lowering the temperature, additional stress builds up in the crystal, resulting in an increase in $2V$. When the crystal is returned to room temperature, it may become uniaxial, indicating that the annealing temperature has been lowered.

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