Teschemacherite, ammonium bicarbonate, was deposited inside the wellhead of the Broadlands geothermal drillhole BR 9 after the bore had been shut for several weeks.

The crystals, white and up to 4 mm long, were identified by X-ray diffraction and infra-red spectroscopy. The deposit, which filled the 198 mm diameter pipe to an estimated depth of about 0.5 m, was ejected in a block, "like a champagne cork," when the bore was opened. Gas pressure at the wellhead prior to discharge was 51.0 bars, the temperature was ambient, and the water level in the bore was about 490 m below ground surface.

The teschemacherite apparently formed from reaction between CO_2 and NH_3, which collect at the wellhead after separating from deeper waters, as represented by: $NH_3 + CO_2 + H_2O \rightleftarrows NH_4HCO_3$. The gas content of water discharged when BR 9 was opened has been calculated as 0.22 mole percent of which 93.5 mole percent was CO_2 (Browne and Ellis, 1970) and the total NH_3 content of the discharged waters was 5.5 ppm (Mahon and Finlayson, 1972).

Teschemacherite has not been reported from other geothermal fields but occurs in some guano deposits (Palache *et al.*, 1951). Cores from drillhole BR 9 were examined for the ammonium feldspar, buddingtonite (Erd *et al.*, 1964) but none was detected.

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American Mineralogist
Vol. 57, pp. 1305–1309 (1972)

A SIMPLE HEATING STAGE FOR SINGLE-CRYSTAL DIFFRACTION STUDIES UP TO 1000°C

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ABSTRACT

An inexpensive heating stage for single-crystal X-ray diffraction studies up to 1000° has been developed. The furnace is capable of maintaining temperatures from 20° to 1000°C to within ± 10°C indefinitely.

DESCRIPTION

Much information about the crystal chemistry of silicates can be obtained by refinement of the crystal structures from high–temperature single–crystal diffraction data. The development of automated single–crystal diffractometers and high speed data processing equipment now allows such refinements to be rapid, routine, and precise. A simple heater has been designed for use with a Picker four–circle goniostat. This device can easily be reproduced from the plans given in Figure 1 for a fraction of the cost of any commercially–available heater.

Several high temperature stages for various single–crystal diffraction geometries have been descirbed in the literature. Reviews of those developed before 1964 are presented by Goldschmidt (1964) and Buerger (1964). More recently, Foit and Peacor (1967) have described a furnace for Weissenberg geometry, and Viswamitra and Jayalakshmi (1970) have described a heater for a Hilger and Watts diffractometer. Within the last year, a small furnace suitable for use with precession and four–circle geometries has become commercially available through Blake Industries. The latter two devices heat crystals primarily by radiative transfer, which may cause uncertainties in temperature measurement if the crystal and thermocouple have different thermal absorption properties. In addition, if the crystal or its X–ray transparent capsule is exposed directly to incandescent platinum, there is a possibility of evaporating an opaque platinum film onto the crystal.

Another furnace capable of maintaining crystal temperatures to ±0.1°C has been described by Lynch and Morosin (1971). This device, however, is rather expensive to build because it requires machining small hemispheres of beryllium. If, however, a temperature precision of ±10°C can be tolerated a much less expensive furnace can be constructed.

To measure diffracted X–ray intensities from a crystal at high temperatures, an extremely localized heat source must be employed which is stable over a period of several days and has a geometry which does not block the incident or diffracted X–ray beams. An instrument has been designed and constructed to meet these requirements. Mounting techniques which allow temperature measurement to within 10°C, do not obstruct the X–ray beams, and prevent oxidation of ferrous iron have been developed for use with this instrument. A dimensioned shop drawing of the device as adapted for use with a Picker four–circle goniostat is given in Figure 1. A diagram of the high temperature goniometer head appears in Figure 2. The device mounts in the χ–

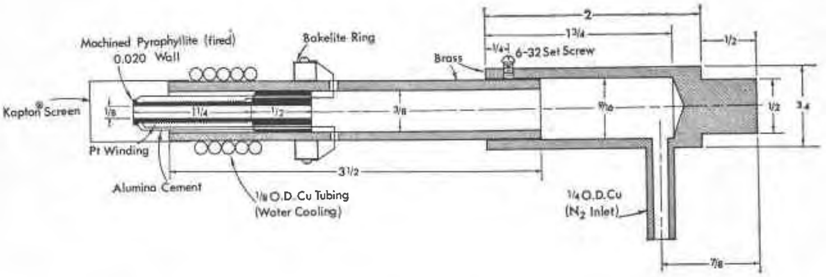

Fig. 1. A longitudinal section through the heater. All dimensions are in inches. A small twist of Pt foil is placed in the cool end of the pyrophyllite tube to cause turbulent flow of nitrogen through the furnace and eliminate temperature gradients across the orifice. The thin wall of the pyrophyllite tube is threaded at 44 turns per inch to separate the windings of 0.015 inch diameter Pt wire. A current of 6.0 amperes at 20 volts AC was required to maintain the crystal at 900°C at a distance of 3 mm from the orifice.

rotation circle opposite the goniometer head and parallel to the ϕ–rotation axis. With modification of the base of the brass tube, the heater and high temperature goniometer head are also suitable for use with precession and Weissenberg cameras.

The device illustrated consists of a telescoping brass tube with a small platinum–wound, fired pyrophyllite furnace in one end. The furnace is positioned so that the crystal is three to eight mm beyond the opening. A small flow of nitrogen is maintained through the furnace to transfer heat to the crystal. The furnace end of the brass tube is water-cooled using a coil of ⅛ inch O.D. copper tubing. Temperature fluctuation due to air currents in the room are prevented by a cylindrical screen of 0.003 inch thick Dupont Kapton[1] high–temperature plastic film.

The 0.020 inch wall thickness of the threaded pyrophyllite tube is impermeable enough to prevent vaporized platinum from reaching the crystal and thin enough to allow good thermal conductivity. The heating element is 0.015 inch diameter platinum wire wound on the pyrophyllite wall threaded at 44 turns per inch. A small spiral of Pt–40 percent Rh foil is placed in the cool end of the furnace tube to break up lamellar flow and eliminate temperature gradients across the orifice. Relatively low flows of nitrogen through the furnace (one to two liters per minute) are used.

As shown in Figure 2, the crystal is mounted in an evacuated silica glass capillary, and held in place by means of silica glass rods sealed

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[1] Registered trade mark of E. I. Dupont de Nemours Co.

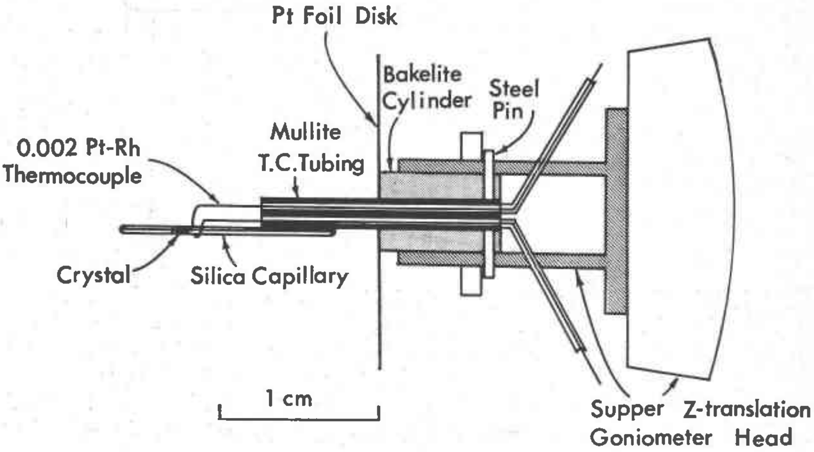

FIG. 2. A section through the top of a Z–translation goniometer head (Charles Supper Co.) modified for high temperature studies. The standard Z–translation block has been replaced by one made of Bakelite plastic open at the rear for emergence of the thermocouple wires. Very fine thermocouple wire must be used to prevent heat conduction down the wire. Alumina cement is used to attach the silica capillary to the 0.075 inch diameter thermocouple tubing.

in each end of the capillary. This type of mount was found to produce negligible scattering and absorption of X–rays and to be stable at temperatures as high as 1150°C. Oxidation of ferrous iron can be prevented by flushing out the capillary with hydrogen before evacuation and sealing. The temperature is measured using a 0.002 inch diameter Pt–Pt10 percent Rh thermocouple placed around the outside of the capillary. A 0.75 inch diameter platinum foil disk is used as shown to prevent excessive heating of the goniometer head. This device has been used successfully for routine automatic measurement of X–ray intensity data from ferrous-iron pyroxene minerals at temperatures as high as 1000°C.

ACKNOWLEDGEMENT

The author wishes to thank Professor Charles W. Burnham for help in preparing the manuscript and the National Science Foundation for Grant No. GA-12852.

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American Mineralogist
Vol. 57, pp. 1309–1310 (1972)

ANTIPHASE DOMAIN STRUCTURE OF THE INTERMEDIATE COMPOSITION PLAGIOCLASE FELDSPARS: ADDENDUM

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We wish to thank Dr. M. G. Bown and Dr. P. Gay for pointing out to us that certain statements in our recent article (Cinnamon and Bailey, 1971) are open to misinterpretation. The statements of concern are (p. 1185) . . . "The reciprocal $1/|S|$ has been cited as a measure of domain size by Smith and Ribbe (1969). This usage of equation (2) is based on the assumption that the same split-"*b*" reflections are being viewed in projections along the x, y, and z axes, however, and reduces the antiphase structure of the plagioclases to a one-dimensional system. This is an unproven assumption that has become accepted as a basic "truth" in the literature concerning the structure of the intermediate composition plagioclases. It seems much more probable from the general nature of the results discussed below that entirely different reflections with different splitting vectors are being viewed along each axis." . . .

The original intent of these statements was to question the interpretation of the intermediate plagioclase domain system as a one-dimensional system. We continue to do so. We no longer consider it probable, however, that entirely different reflections with different splitting vectors are being viewed along each axis. We have investigated the nature of splitting of the "*b*" satellites in detail, and confirm that the satellites occur only as pairs around the missing class (b) reciprocal points. We