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An X-ray powder camera for taking photographs at low temperatures

MARTIN J. BUERGER AND GERALD L. SHOEMAKER

Institute of Materials Science, University of Connecticut
Storrs, Connecticut 06268

Abstract

In volume 28 of this journal, a high-temperature X-ray powder camera was described which had been designed so that the cassette could be removed for development of the film without disturbing the temperature of the specimen. A modification of that design which permits taking powder photographs at low temperatures is given here. The modification consists essentially of replacing the electrically-heated furnace assembly by an insulated cooling chamber. The chamber proper is a copper capsule which is cooled by circulating a cold gas, such as nitrogen or helium, through a helical channel. The gas is cooled by leading it through a heat exchanger consisting of a copper coil immersed in liquid nitrogen, the desired temperature being achieved by regulating the rate of flow of the gas. The cooling capsule is insulated by a hollow cylinder of foamed plastic supported within a thin-walled nylon tube. This tube, and also the concentric cassette and shaft for rotating the specimen, fit neatly into machined concentric grooves in a nylon disk which replaces the machined metal disk of the high-temperature camera. The temperature of the specimen is monitored by a thermocouple inserted into the cooling capsule on a line coaxial with the rotation axis of the specimen.

Background

In our study of opal (Buerger and Shoemaker, 1972) we discovered thermal effects in this mineral below room temperature. It seemed reasonable to expect that this might be caused by a downward displacement of the transformation temperature of the high cristobalite. To investigate this we needed a suitable low-temperature powder camera in which the cassette could be removed for development of the film without changing the environment of the specimen, a common requirement for most X-ray diffraction research at either high or low temperatures. Since this feature had been designed into a high-temperature powder camera (Buerger *et al.*, 1943), the base and cassette of that powder camera were carried over into the low-temperature camera to be described here.

Diffraction experiments at low temperatures have a feature not encountered in experiments at high temperatures. At low temperatures the ambient air contributes heat to the cooling chamber, which tends to limit its cooling effect, and the air also contributes frost along the path of the X-ray beam unless precautions are taken to avoid this. To reduce the heating effect the material near the cooling chamber may be plastic instead of metal. Elimination of the frost is discussed later.

Description

A general view of the assembled camera is shown in Figure 1. The base and cassette are the same as in the heating camera (Buerger *et al.*, 1943). The cassette is held to the base in the same way as in the heating camera, except that the connection is through a machined nylon (instead of metal) disk, seen in Figure 2. The cooling chamber is shown at the right of the illustration. It consists of a copper cylinder in which a helical groove has been machined; this acts as a pathway for conducting a cooling gas around the cylinder. The groove is closed to the outside by a cylindrical copper sleeve. The resulting sealed groove is connected to two pieces of 0.019-inch copper tubing which serve to lead the gas to and from the cooling chamber. The chamber is insulated from the exterior by a covering of foamed plastic in the form of a cylinder made in two parts, seen in Figure 2 (between the capsule and the base of the camera). The assembled cooling chamber and its insulation are held together by a thin-walled nylon tube which can be seen at the lower left of Figure 2. The assembled

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unit is fitted into the machined disk as shown in Figure 3. The wires entering the left side of the long cylinder in Figure 3 are leads from the thermocouple whose sensitive end is positioned near the specimen. A cross-section of the assembled camera without the cassette is shown in Figure 4.

Figure 5 is an "exploded" diagram of the assembly. The specimen can be retracted into the hollow central hub of the camera base by pulling the shaft and its pulley to the right. Most of the features of this low-temperature camera are the same as those of the high-temperature camera (Buerger *et al.*, 1943, p. 293–294).

Operation

In our original experiments on opal, the refrigerant was cooled nitrogen gas. The cooling was effected by passing the gas through a coil of copper tubing immersed in liquid nitrogen. The temperature of the specimen, as monitored by the thermocouple, was controlled by the rate of flow of the nitrogen through this heat exchanger.

While this arrangement was adequate for our experiments on opal, we did encounter condensation of the moisture of the ambient air. In order to avoid this situation we advise leading the dry cooling gas, after it leaves the cooling chamber of the camera, into a plastic bag which surrounds the entire camera unit, allowing it to exhaust into a simple trap. It is also preferable to use helium gas as a coolant, since the scattering by this gas along the path of the direct X-ray beam is minimal, thus leading to the production of cleaner X-ray photographs.

In order to give some useful information when

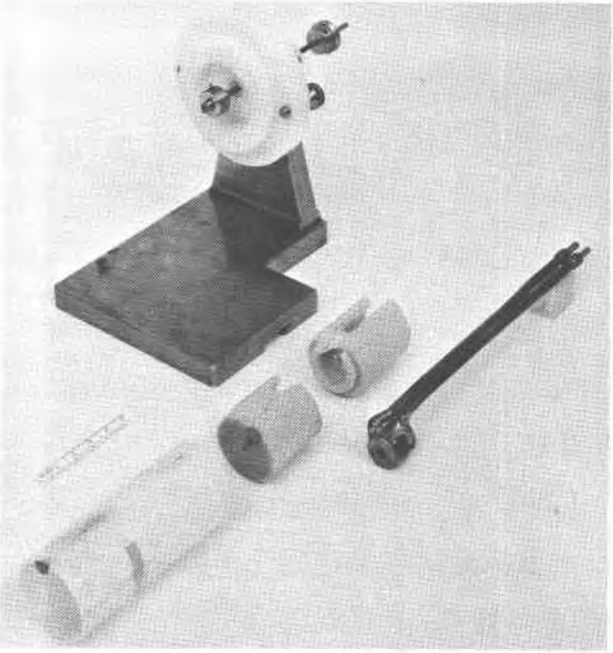

Fig. 2. Individual parts of the cooling unit and its mounting, showing (from upper left to lower right): the machined nylon mounting disk with its retractable specimen holder, attached to the camera base; the two parts of the foam plastic insulation, with its tubular nylon support at its lower left; the copper cooling capsule (photograph by John Hall).

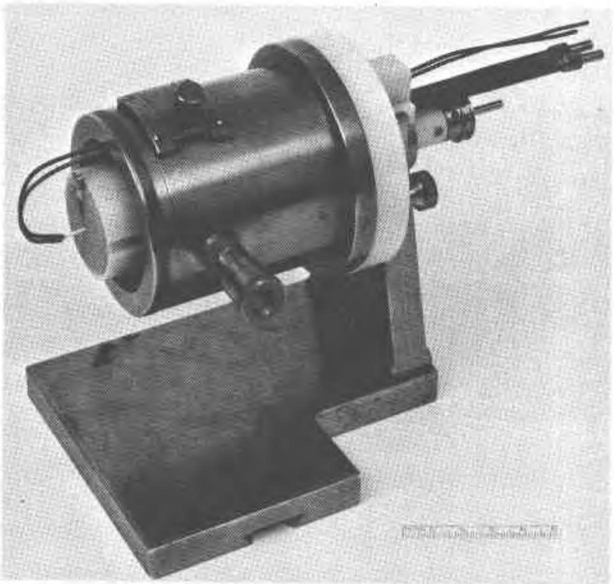

Fig. 1. The assembled low-temperature powder camera with film cassette in position (photograph by John Hall).

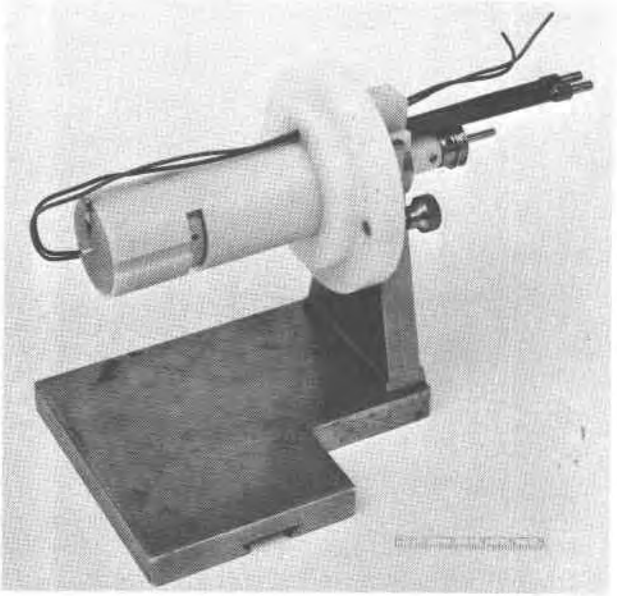

Fig. 3. The assembled low-temperature apparatus with thermocouple in position (photograph by John Hall).

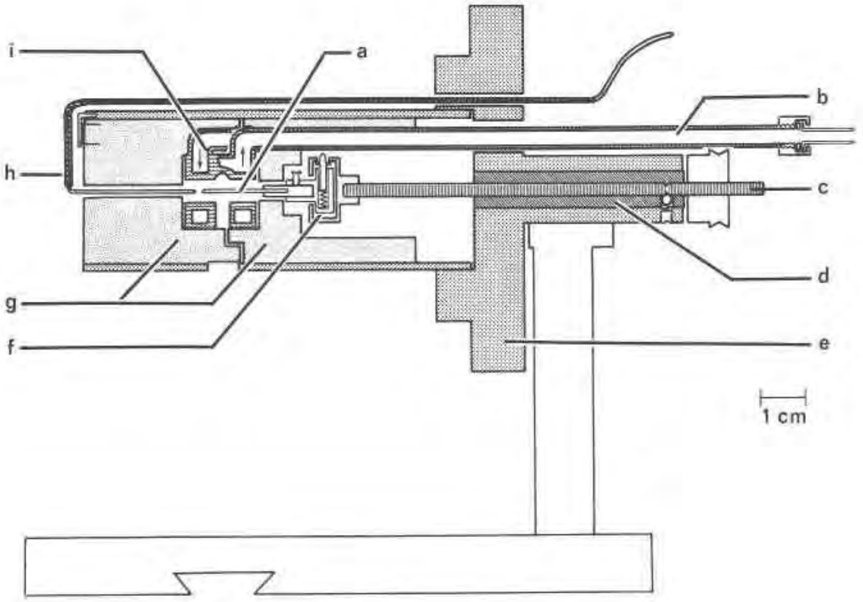

Fig. 4. Cross-section of the assembled camera without the cassette: (a) specimen; (b) copper tubing for refrigerant; (c) retractable specimen shaft; (d) brass bushing; (e) machined nylon disk; (f) sample-centering chuck; (g) two-part machined foam insulation: (h) thermocouple wire; (i) copper cooling capsule.

helium gas is used as refrigerant, we cooled the helium by passing it through the coil of copper tubing immersed in liquid nitrogen. The cold helium gas was delivered to the camera through rubber tubing insulated by a surrounding tube of soft foam plastic with a wall thickness of about 0.5 inch. After leaving the cooling unit the helium was exhausted into a plastic bag surrounding the camera. The temperature at the specimen proved to be very sensitive to the rate of flow of the coolant, but could be readily controlled to within 1°C for runs of about one hour. The lowest temperatures readily attained were in the neighborhood of −65°C. In order to achieve this temperature a gas flow rate of about 60 ft^3/ hr was necessary.

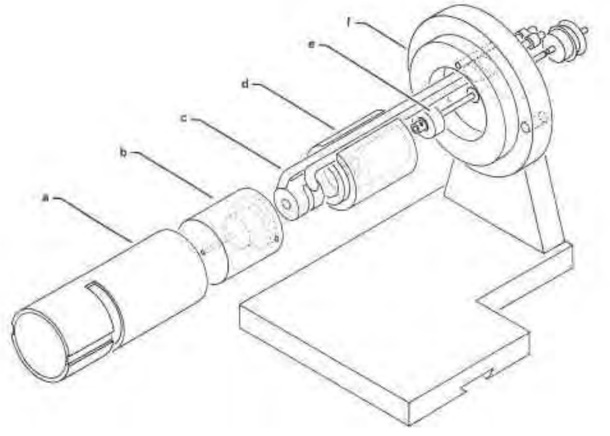

Fig. 5. Exploded view of the low-temperature assembly and its mounting: (a) nylon retaining shell; (b) distal part of foam insulation; (c) copper cooling capsule; (d) proximal part of foam insulation; (e) sample-centering chuck; (f) machined nylon disk for mounting assembled cooling unit and cassette.

While we did not explore the temperature gradient throughout the cooling chamber, we did make a test on the difference in readings between the monitoring thermocouple and a second thermocouple placed as nearly as possible at the position where the specimen would intersect the X-ray beam. We obtained readings indicating a temperature difference of 3 to 6°C for the separation between the two thermocouples. Time was not available for testing whether the two thermocouples differed in their calibration.

References

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