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X-Ray Diffraction and Optical Observations on Crystalline Solids up to 300 kbar

WILLIAM A. BASSETT, TARO TAKAHASHI, AND PHILIP W. STOOK
Department of Geological Sciences and Space Science Center, University of Rochester, Rochester, New York 14627
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Diamond anvils driven by a simple piston and screw device have been used to subject solid samples to pressures up to 300 kbar at ambient room temperature. Samples can be examined optically and by x-ray diffraction while under pressure. Pressure is measured by means of the lattice parameter of sodium chloride mixed with the sample. Reflectivity and volume data on iron, as well as crystallographic data on PbS, PbSe, and PbTe, are presented.

INTRODUCTION

DIAMONDS have been used as anvils in high pressure studies of various substances by means of infrared,[1] visible light,[2] and x-ray diffraction.[3–6] Various other designs for investigating substances at high pressure by x-ray diffraction have been used as well.[7–10] The instrument described in this paper is based on many of the design features employed by VanValkenburg, Lippincott, Deucker, Weir, and Piermarini.

It has become apparent that a combination of visual and x-ray observations of a sample is extremely valuable, especially for the study of polymorphs. For this reason we have designed our instrument so that it can be transferred between x-ray diffraction assembly and microscope without disturbing the sample, the pressure, or any of the alignments. In addition, it is small, light, and simple, allowing freedom for rotation and focusing on the microscope stage, as well as reproducible mounting on the x-ray diffraction assembly. Other design features described later make possible a high degree of precision in measurements of lattice parameters, as well as pressures up to 300 kbar.

We have used this instrument in three principal areas of investigation, (a) for crystallographic identification of reversible high pressure polymorphs, (b) for study of the effect of pressure on lattice parameters and volume under isothermal conditions, and (c) for study of the effect of pressure on optical properties of metals.

DESCRIPTION OF INSTRUMENT

A sample in polycrystalline form is placed between two opposing flat diamond anvil faces. As the anvils are driven together, the sample extrudes until friction prevents further extrusion. This results in a pressure distribution with maximum pressure at the center and essentially atmospheric pressure at the perimeter.

A cross section of the instrument is shown in Fig. 1. The diamond anvils A are 30 mg brilliant cut single crystals which have had their culet faces enlarged. One anvil has a face that is approximately 1.8 mm in diam, and the other anvil has a smaller face (0.2 to 0.6 mm diam have been used). A maximum of 300 kbar can be achieved with a face of 0.3 mm in diameter, and 125 kbar with a face 0.5 mm in diameter. These seem to be safe limits, beyond which the diamond anvils may fail. The larger the diameter of the anvil face, the thicker the resulting sample. Therefore, an x-ray diffraction pattern can be obtained more quickly when a larger diameter is used.

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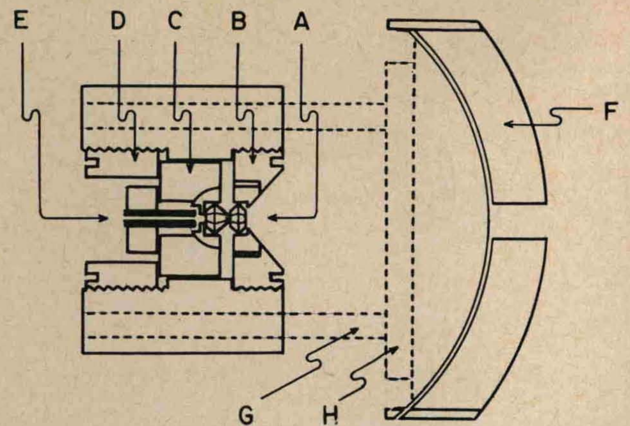

FIG. 1. Cross section of the diamond anvil press; A—diamond anvils; B—stationary piston; C—sliding piston; D—driver screw; E—collimator; F—film cassette; G—cassette mounting rods; H—cassette translating bars.

The diamond anvils are fastened to rockers, which in turn are mounted in troughs at the ends of two pistons (Fig. 2). These rockers are arranged in such a way as to permit both angular and translational adjustment, so that the anvil faces can be matched and made parallel to each other. Piston B (Fig. 1) is stationary when screwed into place, while piston C slides inside a cylindrical hole in the squeezer body. Piston C is keyed in order to prevent rotation. Piston C is driven against piston B by a driving screw (part D).

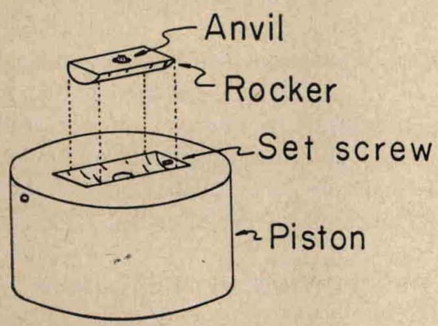

FIG. 2. Rocker mounting of the diamond anvils. The rocker permits translational as well as angular adjustment. In the opposing piston, the rocker's axis is turned 90° from this one.

Passage for light and x rays is provided by axial holes in parts D, C, and B. The opening in part B is conical to allow divergence of rays scattering from the sample. A Pyrex glass capillary tube (50 μ i.d. 7 mm o.d.) (part E) which serves as a collimator for x rays is placed in the hole in part C. This type of collimator produces a beam of x rays approximately 50 μ in diameter, but allows entrance of sufficient illumination for visual observation of the sample by transmitted light. The x-ray beam, thus collimated, passes perpendicular to the anvil faces, impinging on a small area of sample at the center of the anvil faces. Scattered x rays pass out through the diamond and hole in piston B. The conical hole in piston B permits a range in 2θ up to 45°. When K_α radiation from molybdenum is used, this makes it possible to measure d spacings greater than approximately 1 Å.

A film cassette F having the shape of a quadrant of a cylinder with a 50.00 mm radius is fastened to the squeezer body by four rods G and two translation bars H. This mounting is adjustable, so that the axis of the cylinder can be made to pass through the sample.

Although other x-ray sources have been used successfully, the most satisfactory source has been found to be a combination of a Jarrell-Ash Microfocus x-ray generator with molybdenum target and a curved quartz crystal monochromator. This generator provides a very high energy (50 kV and 2.5 mA) per unit area (100 by 1600 μ) of target. In addition, it serves as a point source in the x-ray diffraction geometry, thus yielding photographs with good definition. The use of monochromatized radiation eliminates Laue spots produced by the single crystal diamond anvils and greatly improves the signal-to-noise ratio.

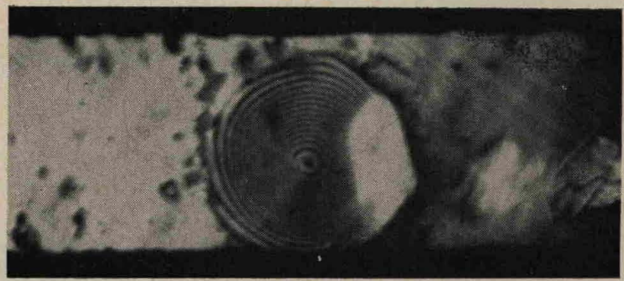

FIG. 3. Interference bands produced by reflections of monochromatic light from upper and lower surfaces of NaCl under pressure between the diamond anvil faces. The pressure at the center of the area is about 100 kbar.

FIG. 4. The position of the x-ray beam within the sample area. This photograph was produced by placing film between the anvils, applying enough pressure to cause indentation, and then passing the x-ray beam through it. The diameter of the diamond face is 0.5 mm and the diameter of the x-ray beam is 0.05 mm.