

### A Simple X-Ray Camera for High Pressure Uses

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An extremely simple camera has been developed which enables X-ray powder diffraction under pressures up to 100 kbar at room temperature. A thick cylinder of a pressure vessel acts as a body of a Debye-Scherrer camera. A pressure-clamping method is used. To test the camera, the lattice parameter of NaCl has been measured at the Bi III-V transition point.

During the last ten years various types of X-ray apparatus have been developed<sup>1)</sup> to study the state of solids under high pressures. We also have attempted to design an X-ray camera which can be easily handled for high pressure uses. As shown in Fig. 1, this camera consists of Bridgman anvil, collimator, beam trap, camera body, and upper and

lower screws. A method of "conservation" of pressure originally developed by Chester and Jones<sup>2)</sup> is employed. The present camera has a thick body which can also act as a cylinder of a pressure vessel. Since the X-film is simply stuck outside the body, a sample can be automatically located at the center of the camera only by placing the

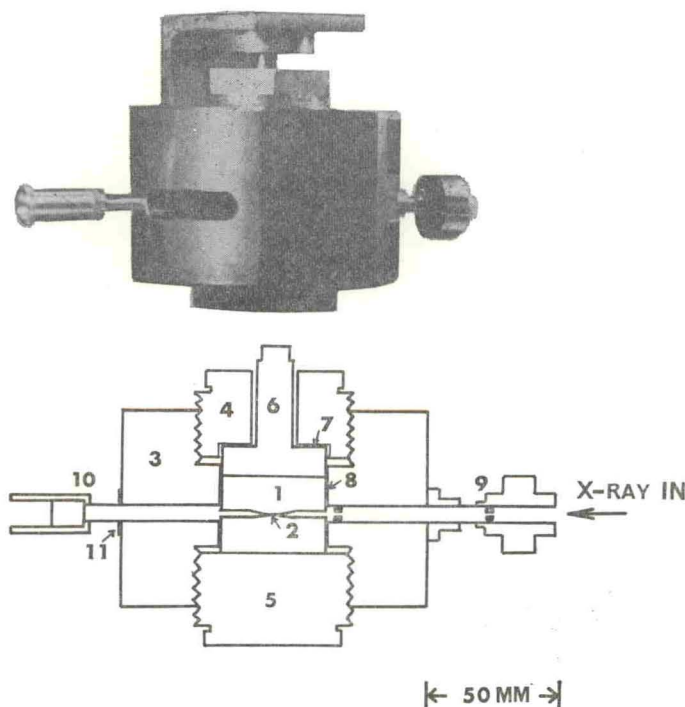


Fig. 1. Photograph and cross section of the X-ray camera.

- |                 |                                      |
|-----------------|--------------------------------------|
| 1: anvil        | 2: sample                            |
| 3: body         | 4: upper screw                       |
| 5: lower screw  | 6: washer                            |
| 7: teflon sheet | 8: bakelite ring                     |
| 9: collimator   | 10: beamtrap with fluorescent screen |
| 11: film        |                                      |

sample at the center of the anvil, the height of which is already adjusted by rotating the lower screw. After the camera is held in a vise, pressure is loaded by tightening the upper screw with a spanner. To prevent the upper anvil from kneading the sample by rotation, an L-shaped fixture holding the head of the washer is held together with the camera in the vise. For a rough estimation of load, a resistance strain-gauge is attached to the inclined surface of the anvil. The body and screws are made of mild steel. The anvil consists of a 3% Co-bearing tungsten-carbide core with a 160° truncated cone and a 3 mm face surrounded by a stainless steel binding ring. As the pressure medium with low absorption coefficient, a disk of amorphous boron powder mixed with epoxy resin (30 wt%) is used. A sample is packed into a 0.3 mm hole at the center of the disk which is 3 mm in diameter and 0.5 mm in thickness. A rotating-anode type X-ray generator with molybdenum target is used. At a maximum output of 60 kV and 100 mA, an exposure of about 0.3 hour is sufficient to obtain an X-ray diffraction pattern from NaCl for pressures around 100 kbar. Such an exposure time is considerably short compared with that for other high pressure cameras which mostly require 10-20 hour exposure. The diameter of the present camera is 114.6 mm. Diffraction patterns of  $10^\circ < 2\theta < 50^\circ$  are obtained.

In the course of testing the camera, the lattice parameter of NaCl has been observed at the end of the Bi III-V transition and has been compared with a previous datum. Bi wire of 0.1 mm in diameter is mounted vertically in the NaCl powder hardened with epoxy resin. In order to measure the electrical resistance of Bi under pressures, the upper anvil and washer are insulated from the body using bakelite ring and teflon sheet. The upper screw of the camera is tightened and stopped just at the point where the electrical resistance of Bi across the anvils ceases to drop due to the Bi III-V transition. Then it is set for the X-ray beam by observing the fluorescent screen of the beam trap. The shade of the Bi wire is clearly distinguished on the screen. The diffraction patterns obtained for NaCl at 1 bar and at the end of the Bi III-V transition are shown

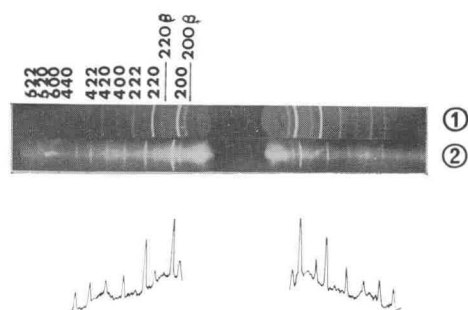


Fig. 2. X-ray patterns of NaCl at different pressures (unfiltered Mo radiation): (1)-1 bar, (2)-about 80 kbar. The lower figure is a chart obtained by means of a microphotometer from the above pattern (2).

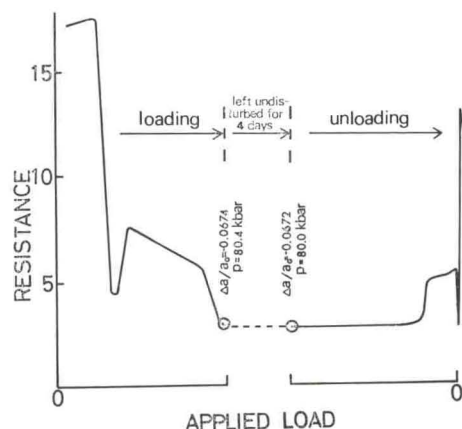


Fig. 3. The resistance of Bi vs. applied load. Electrical resistance is shown in arbitrary unit. Absolute amount of applied load is unknown.

in Fig. 2-(1) and (2) respectively. The lattice parameter of NaCl is determined by taking the arithmetic average of the values obtained for the six reflection lines: 200, 220, 222, 400, 420 and 422. Calibration of the camera is made in each series of runs using the same sample by plotting the correction curve for NaCl at 1 bar before and after loading. Figure 3 shows the resistance of Bi vs. applied load which is represented by relative deflection of a strain-meter. Values of  $\Delta a/a_0$  of NaCl and the corresponding pressure calculated from Decker's equation<sup>3)</sup> at the end of the Bi III-V transition with increasing load and those after leaving the camera undisturbed for four days are entered in Fig. 3. Therefore it is proved that the pressure remained almost constant, although the applied load might have been decreased by an

unknown amount. A fear that the pressure might slowly decrease with the passage of time due to an expansion of the body material has been found to be utterly groundless. The value of  $\Delta a/a_0$  at the end of Bi III-V transition obtained in the present experiment is in satisfactory agreement with McWhan's value:  $\Delta a/a_0 = -0.0645 \sim -0.0686$ .<sup>4)</sup> X-ray diffraction patterns have been obtained also for the three high pressure phases of InSb reported by Banus and Lavine.<sup>5)</sup>

This camera shows a reasonably high degree of precision for the measurement of lattice parameters, and can easily be operated under pressures up to 100 kbar. A maximum of 150 kbar may be a safe limit beyond

which the tungsten-carbide anvil face may fail owing to an inhomogeneous strain.

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#### References

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