X-ray analysis of substances under high pressure



FIG. 3. Upper and lower supports and beryllium chamber.

as created. It was held at a temperature of 1050°C by 3 hr and cooled smoothly down to room temperatute in the course of 16 hr.

An X-ray diffraction pattern of the beryllium chamber after it had been aged under pressure is shown in Fig. 4 (upper picture). The pattern taken after heat treatment is shown in the same figure. A chamber produced in this way was repeatedly ared at pressures up to 12,000 kg/cm², with only trivial deformation. The beryllium grain remained virtually unaltered up to pressure of 6-7,000 kg/cm².

Pressure calibration of the apparatus. The following measures were adopted to reduce friction in the exparatus. The inner surface of the beryllium chamber and the channels of the supports were very well surface-finished. Pistons, 7 and 8, of the lower support and the corresponding pistons of the experimentary of 0.06-0.09 mm in the channels. Paraffin wax was used as the packing material. The packings here and in the tapers of the supports (beryllium-support) worked very well bring tests up to 18,000 kg/cm².

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As can be seen from Fig. 1, as regards the stem of transmitting force to the centre of the hamber, the apparatus is symmetrical around a lane perpendicular to the drawing and passing trough the centre of the beryllium chamber and te collimator. It therefore made no difference to shich piston, 6, or the corresponding upper one, the tree which was used to create the pressure in the sparatus was applied. This was checked experirentally and it was found that the pressure differtree at the top and bottom of the beryllium chamber as the same with any force applied to piston 6 or the corresponding upper one.

The apparatus was calibrated in the usual kind f machine with compression and tension using a ference dynamometer type DS-3. From the forces termined on reference dynamometer DS-3, which was at the top of the apparatus, and dynamometer, 3, the pressure round the top and bottom of the beryllium chamber was calculated. To calculate the pressure the areas had to be calculated from precise measurements of the diameters of the corresponding openings in the supports. The pressure in the centre of the beryllium chamber was determined as half the sum of these pressures. Graduation was carried out separately for each chamber and for each substance tested.

The accuracy of the pressure measurement in the apparatus was determined from a considerable number of experiments in calibration, and it was found to be 2 per cent of the measured pressure value (up to $10,000 \text{ kg/cm}^2$). Pressure changes of 0.06 mm³ could be observed on the indicator of dynamometer 3 as the pressure rose.

X-ray diffraction camera. The X-ray diffraction camera was very carefully adjusted both in the course of manufacture and in assembly. The following requirements had to be very carefully satisfied.

a) The interior surface of magazine, 10, should have the exact shape of a cylinder and the taper of the cassette should precisely correspond with that of support, 9. The axes of the cylindical surface and the taper of the magazine must coincide with the axis of both supports and the beryllium chamber, 24, (maximum permissible deviation is 0.015 mm).

b) The collimator axis must form an angle of 90° to the axis of the cylindrical surface of the magazine and it must be in the centre of the split in ring, 20.

An ordinary microscope with magnification 25 was used to adjust the X-ray camera and to check its accuracy. Its focal length was 50 mm and the eyepiece was fitted with a micrometer (graduations of 0.05 mm). Adjustment was made by means of a

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