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*RAPIDLY RUNNING TRANSITIONS AT HIGH PRESSURE**

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For the study of the phase changes in minerals and other substances a highly successful method is that involving the now well-known Griggs-Kennedy "squeezer."[1a] This device, consisting basically of carboloy anvils between which a thin layer of material is compressed to several tens of thousands of bars, is remarkably effective in many applications, especially that of determining phase equilibria in mineral systems under the combined effect of elevated temperatures and pressures. However, such methods usually depend upon the feasibility of "quenching" the equilibrium by rapid cooling. But some transitions take place promptly; the assemblage of phases existing at a given temperature and pressure cannot be preserved by sudden cooling.

Diamond "Squeezer."—In order to deal with such rapidly running transitions, we have modified the "squeezer" technique by using diamond, instead of carboloy, for one of the anvils. Pressure is applied by means of a small press of such size and shape as to permit mounting it on the spindle of a Norelco recording diffractometer. A beam of high intensity X-radiation is made to impinge on the sample, so that a chart record of the diffracted beam is obtained, and thus gives an indication of the extent of any phase transitions.

Originally, it was thought that two diamonds were necessary in order to avoid the confusion of extraneous lines in the diffraction pattern, but it was soon found that a lower anvil of carboloy, with or without a covering of a thin sheet of platinum foil, contributed only a few peaks, which were easily identified, labeled, and ignored. In order to get satisfying records with the recording diffractometer the anvil face should be 2 mm or more in diameter (5 mm is better). Pressure is applied by means of a Blackhawk "jack" (4 tons rated capacity). The first diamond anvils were made by sawing a natural octahedron in two so as to make two four-sided pyramids, and the sawn surfaces polished and edged to make circular faces. The pointed end was embedded in sintered metal for mounting in the steel piece bearing against the upper place of the press.

A better method was that shown in Figure 1. The diamond is fashioned with a second (larger) flat face, which bears against a flat surface of metal (hardened steel or carboloy).

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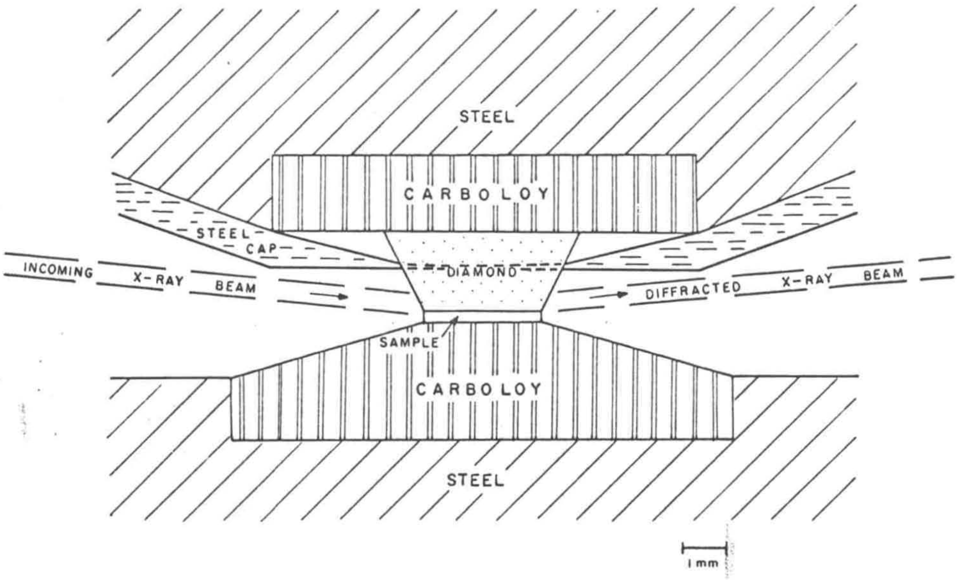

FIG. 1.—Diamond anvil. An X-ray beam is diffracted from the sample that is pressed between diamond and carboloy surfaces. The beryllium anvil was used in essentially the same way.

hard material is capable of withstanding very considerable pressures. Subsequent to the time (1958) that this research was started, Weir and others[1b] reported that two diamond anvils, carefully aligned by an optical interference method and compressed by steel blocks, withstood over 50 kb[1c] pressure and permitted the obtaining of excellent infrared spectrograms of substances under high pressure. About the same time Jamieson and others[2] described a "diamond piston" technique for X-ray diffraction patterns. One was successfully used up to 35 kb, although there were failures with various other diamonds at lower pressures, especially when the stone contained a flow or "knot."

Diamonds have been used in other ways for high-pressure X-ray diffraction studies. We may note, for example, the "split-diamond" bomb (Lawson and Tang[3]) and the single-crystal diamond apparatus by Jamieson.[4, 5] Quite recently C. E. Weir and G. J. Piermarini (personal communication) at the National Bureau of Standards have successfully used the diamond-anvil technique for obtaining photographic X-ray patterns of materials at pressures up to 30 kb, passing a narrow pencil of X-radiation through small holes normal to the touching diamond faces. Of all these devices only the present one and the "diamond piston" apparatus of Jamieson, Lawson, and Nachtrieb[2] can be readily adapted for studies of transformation rates.

As yet we have not been able to realize the pressures that we had expected to attain. Our apparatus, in comparison with that of Weir and others[1b] for infrared spectroscopy encounters two difficulties. First, with the anvils that are used with a recording X-ray diffractometer there is no readily apparent way of utilizing an optical method for putting the loading surfaces in perfect alignment. In the second place, the diameter of the sample needs to be at least 2 or 3 mm in order to obtain usable diffraction patterns under all conditions, and this sample size compared to the 0.2 to 0.3 mm, adequate for other purposes, renders the existence of slight misalignment much more serious. Then, too, the limitation imposed by even very small flaws or "knots" has not been fully appreciated. The diamonds we have used do not withstand much over 10 kb, but in this modest pressure range we have obtained some interesting measurements on rates of transformation and some precise results for the volume-change of various materials at the transition pressure.

As for the X-ray wavelength for the diamond anvils the best choice is molybdenum Kα. It is not feasible with diamond to use the more popular copper Kα, because for this wavelength (1.54 Å) the linear absorption coefficient in diamond is about 19, which means that the transmission through a thickness of (for example) 5 mm is only 2×10^{-4}. With Mo Kα ($\lambda = 0.71$), on the other hand, the linear absorption coefficient in diamond is only about 2.5 and the transmission through a 5-mm thickness is 0.29. Despite the somewhat inferior accuracy and resolution with Mo Kα, for diamond anvils, there is little choice other than molybdenum.

The materials were used in the form of circular films or disks, either 2.6 or 4.6 mm in diameter. One method of preparation consisted of making a thin pellet in a pellet press of appropriate internal diameter. It has become evident that it is important for the pellet to be quite thin, but it is difficult to make and handle a pellet less than 0.1 mm thick. A thickness of only a few microns (depending on the material) gives patterns with Mo Kα radiation having a considerable fraction of maximum peak height. For example, an AgI film having an effective thickness of 10 microns gives a good pattern; and, even with materials of much lower absorptive power, good records were obtained with films 5 or 6 microns thick. Various methods for making thin films were tried. Among the successful ones were: (1) the smearing of a small amount of a dry powder on the anvil face; (2) the placing of the desired amount of fine powder on the anvil and spreading it with the aid of a liquid such as acetone; and (3) allowing a fine suspension to settle on the desired surface. In our work with the diamond "squeezer" we have used these various methods and we are increasingly convinced that it is advantageous to use very thin films.

Silver Iodide.—It seems established that at room temperature and atmospheric pressure the stable form (II) of AgI is wurzite-type hexagonal. A metastable form, sphalerite-type cubic, also with closest packing and almost identical density, transforms at a little above 100°C to the stable hexagonal form, which in turn undergoes a reversible transition at 146° to CsCl-type modification. Bridgman[6] showed that another form, halite type, exists above 3 kb at room temperature, the transition pressure II $\leftrightharpoons$ III being nearly independent of temperature. Originally we had expected to study the transition of the hexagonal (type II) form to the high-pressure form, but it was found that grinding, and particularly pressing the AgI into a pellet, readily converted the material to the metastable cubic form. Although Bridgman indicated the transition at 3 kb to be from the ice-type (wurzite-type) to the halite type, it now seems quite certain that even moderate nonhydrostatic stress would have immediately converted his starting material to the cubic form and that his transition boundary was probably between the cubic sphalerite-type and the cubic halite-type forms. Jacobs,[7] writing in 1938, of Bridgman's work, refers to type II of his diagram as being cubic sphalerite type, but Jacobs' own measurements essentially confirm Bridgman's position of the II $\leftrightharpoons$ III boundary.

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Our measurements were made with sphalerite-type cubic formed by grinding ordinary precipitated AgI in a mortar. This was more convenient and effective than any of the precipitation methods. At a pressure of about 3 kb in the "squeezer" AgI inverts to a new form which, in agreement with Jacobs, is readily identified as halite-type cubic. The spacings given in Table 1 were obtained from the measured 2 θ's of the high-pressure lines, the angles (with Mo Kα radiation and the diamond squeezer) being corrected by calculating, from the known compressibility, what the angles for the remanent low-pressure lines should read at the transition pressure. From these spacings it may be readily calculated that the edge of the unit cell (with 4 molecules of AgI) is 6.068 Å, from which the specific volume of the high-pressure form is 0.1431 at the inversion pressure. Comparing this with 0.1737, the specific volume of the low-pressure form at 3 kb, we obtain 0.0306 for Δv, the volume change in cm^3 per gram at the transition. This agrees not too well with Bridgman's value 0.023 and somewhat better with that of Jacobs, 0.0286.

TABLE 1

PARAMETERS OF LOW- AND HIGH-PRESSURE FORMS

Compound	AgI	RbI	NH_4I	RbCI	Ce
Low-pressure data					
Structure type	$F\bar{4}3m$	$F\frac{4}{m}\bar{3}\frac{2}{m}$	$F\frac{4}{m}\bar{3}\frac{2}{m}$	$F\frac{4}{m}\bar{3}\frac{2}{m}$	$F\frac{4}{m}\bar{3}\frac{2}{m}$
Mols (or atoms)/cell	4	4	4	4	4
Cell const. (Å)	6.495	7.342	7.261	6.582	5.150
Sp vol (cm^3/gm) $P = 0$	0.1759	0.2806	0.3978	0.3549	0.1468
Sp vol (cm^3/gm) $P = P_{tr}$	0.1737	0.2720	0.3968	0.3439	0.1408
High-pressure data					
Trans pressure (kb)	2.9	3.97	.49	5.41	7.0
Structure type	$F\frac{4}{m}\bar{3}\frac{2}{m}$	$P\frac{4}{m}\bar{3}\frac{2}{m}$	$P\frac{4}{m}\bar{3}\frac{2}{m}$	$P\frac{4}{m}\bar{3}\frac{2}{m}$	$F\frac{4}{m}\bar{3}\frac{2}{m}$
Mols (or atoms)/cell	4	1	1	1	4
Obs'd spacings (Å)	3.024 (200) 2.152 (220) 1.751 (222)	3.040 (110) 2.163 (200)	4.328 (100) 3.062 (110) 2.502 (111)	3.876 (100) 2.744 (110) 2.243 (111) 1.946 (200)	2.791 (111) 2.411 (200) 1.704 (220) 1.391 (222)
Cell const (A) $P = P_{tr}$	6.068	4.310	4.331	3.884	4.824
Sp. vol (cm^3/gm) $P = P_{tr}$	0.1431	0.2268	0.3375	0.2933	0.1205
Δv (cm^3/gm)	0.030_6	0.045_2	0.059_3	0.050_6	0.020_3
Apparatus	Diamond	Beryllium	Diamond	Beryllium	Beryllium
Radiation	Mo Kα	Mo Kα, Cu Kα	Mo Kα	Mo Kα, Cu Kα	Mo Kα, Cu Kα

From Figure 2 (taken with Mo K radiation at a nominal pressure of 2.6 kb) it may be seen that the high-pressure lines become evident at (nominal) pressures below the expected transition pressure. Even at a considerable excess pressure the lines of the low-pressure form are found to be still evident. This is the result of pressure inhomogeneity in the material between the anvils, an effect that is probably always present in the squeezer-type apparatus at low pressures.

Ammonium Iodide.—Here the transition under pressure is from halite-type cubic

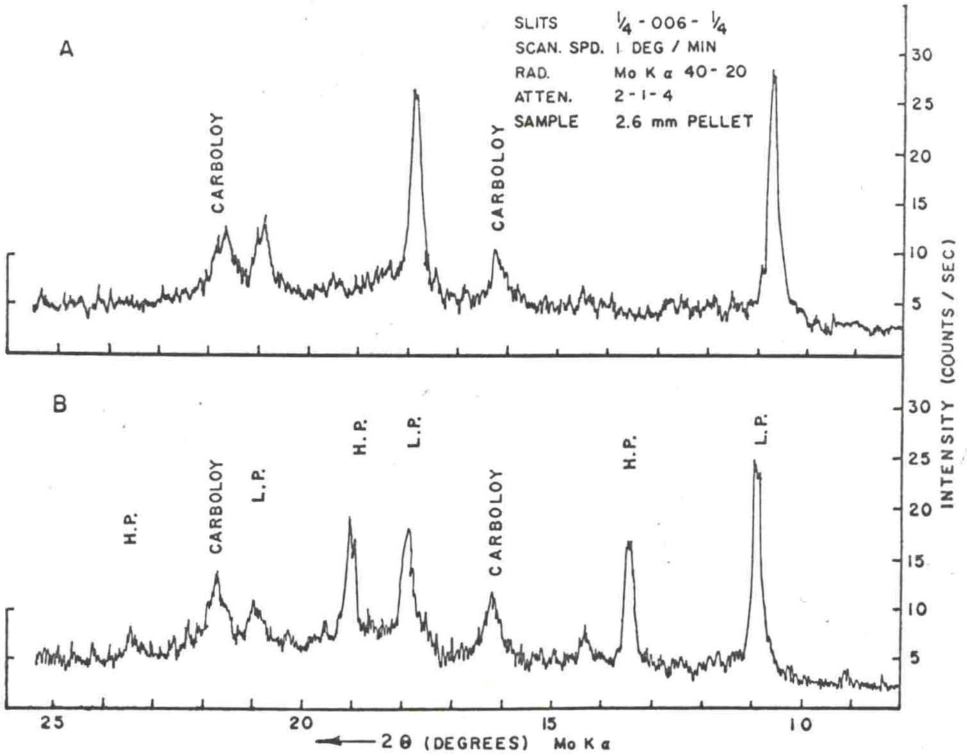
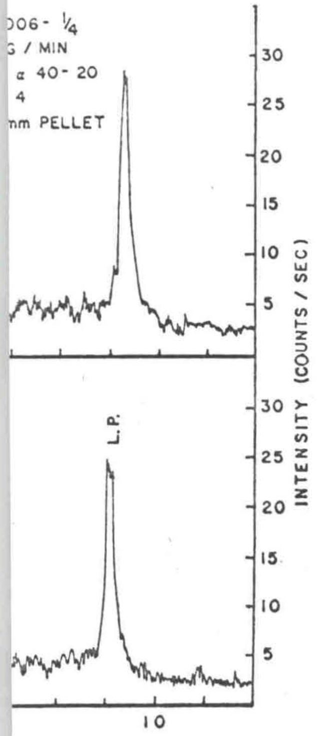


FIG. 2.—Diffraction pattern of AgI under a *nominal* pressure of 2.6 kb (lower section). The upper section is for atmospheric pressure. Molybdenum radiation and diamond anvil. Pressure inhomogeneity accounts for the presence of both high- and low-pressure peaks in the high-pressure pattern.

to cesium chloride-type cubic. According to Bridgman,[17] the transition pressure at room temperature is 0.5 kb. Good values for the (100), (110), and (111) peaks were obtained. The best value (from d_{110}) gives a cell edge of 4.331 Å which corresponds to a specific volume of 0.3375. That of the low-pressure form, corrected for compressibility is 0.3968. The change in volume at the transition is therefore 0.0593, which may be compared with 0.054 obtained by Jacobs.

Beryllium Squeezer.—It was found that beryllium, used in place of the upper diamond anvil of Figure 1, gave good diffraction patterns of various materials, and was usable at surprisingly high pressures. The beryllium (obtained from Brush Beryllium Co., Los Angeles, Calif.) was used in the form of cylindrical pellets cut from a rod, 4.85 mm in diameter, of well-compacted beryllium (density 1.85, and reported purity 98.3 per cent). The usual length was about 2 mm. These pellets, or anvils, bore against a lower carboloy anvil (as in the case of the diamond anvils) and withstood 20 kb without troublesome spreading. Even more interesting and important was the fact that here, unlike the case for diamond, copper X-radiation gives clear, usable, patterns. This is because the linear absorption coefficient of beryllium with copper $K\alpha$ X-radiation is practically identical with that of diamond for the molybdenum radiation, so that in either case the transmission through a thickness of 5mm is 29 per cent.



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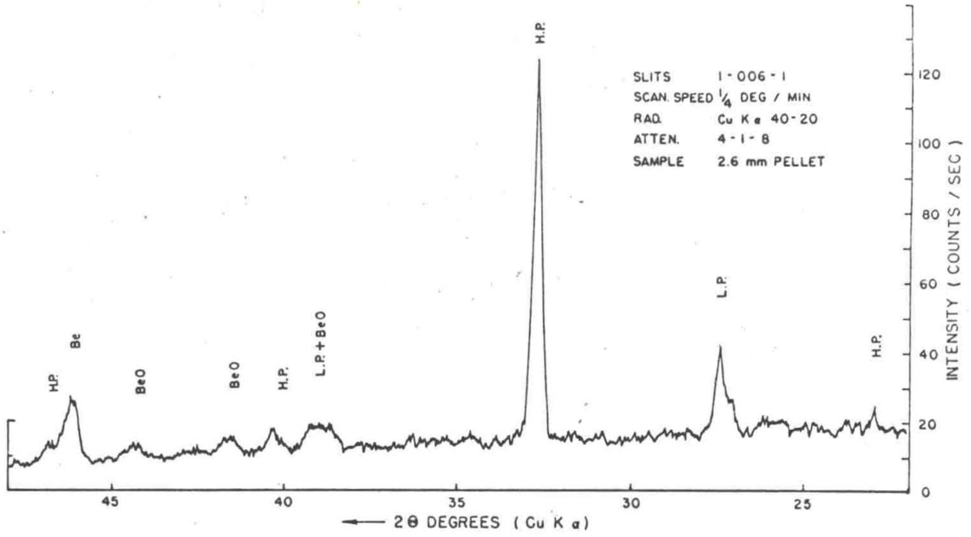

FIG. 3.—Diffraction pattern of RbCl under a *nominal* pressure of 5.8 kb. Copper radiation and beryllium anvil.

Rubidium Chloride.—In Figure 3 there is shown a tracing of the diffraction pattern for rubidium chloride with the beryllium squeezer and Cu $K\alpha$ radiation at a nominal pressure of 5.8 kb, which is somewhat above 5.41, the transition pressure as measured by Bridgman.[11, 17, 18] The low-pressure lines shown in the pattern are evidence of the fact that near the circumference of the sample the pressure is lower than the average.

Our starting material was from a sample labeled "spectroscopic purity." From four good peaks of the Cu $K\alpha$ record the spacings shown in Table 1 were obtained. These are easily indexed as pertaining to CsCl-type cubic and lead to a cell constant of 3.884Å for the high-pressure form (when corrected to the transition pressure by using the known compressibility of the salt). The corresponding specific volume 0.2933 and the volume change at the transition pressure is therefore 0.0506 cm^3/gm— almost identical with Bridgman's value 0.0505. Nearly the same value for cell constant was obtained with Mo $K\alpha$ and the beryllium squeezer.

Rubidium Iodide.—With the beryllium squeezer this salt gave good records with both Mo $K\alpha$ and Cu $K\alpha$ radiation at a (nominal) pressure of 4.9 kb. Although the transition pressure according to Bridgman[11, 18, 19] is 3.97 kb, both the lines of the low-pressure and those of the high-pressure form are visible at a nominal pressure 1 kb higher than the transition pressure.

Like RbCl the rubidium salt inverts under pressure from halite-type cubic to cesium chloride-type cubic. Good high-pressure patterns were obtained with Cu $K\alpha$. The d-spacings for (110) and (200), as given in Table 1, give 4.310Å for the edge of the unit cell (containing one molecule RbI). The corresponding specific volume of the high-pressure form at the transition pressure is 0.2268 cm^3/gm, while the specific volume of the low-pressure form at atmospheric pressure is 0.2806 and at the transition pressure 0.2720; the volume change, Δv, is therefore 0.0452 cm^3/gm, which may be compared with that from Bridgman,[11, 17] 0.029, and that from Jacobs,[7] 0.037.

Cerium.—This element inverts at 6.9 kb according to Bridgman[15] to a high-pressure modification. An earlier, and presumably less accurate, result by Bridgman[20] is appreciably higher. Lawson and Tang[16] have shown that the transition is to another face-centered ("collapsed") cubic form. With the beryllium "squeezer" good records were obtained of the high-pressure form with Cu Kα radiation and also with Mo Kα. Figure 4 shows the chart obtained at 8.1 kb nominal pressure with Mo Kα radiation, which in this case gives a somewhat better chart than the one for copper radiation. For the transition pressure at room temperature we take 7.0 kb, which is the average of the later result by Bridgman and that obtained by A. Jayaraman (personal communication). In Figure 4 the chart shows both low-pressure and high-pressure peaks, indicating incomplete conversion at 8.1 kb "nominal" pressure. As shown in Table 1, the measured d-spacings yield the value 4.824 Å for the cell constant of the high-pressure form. This corresponds to a specific volume of 0.1205, so that Δv the volume change at the transition pressures is 0.0203.

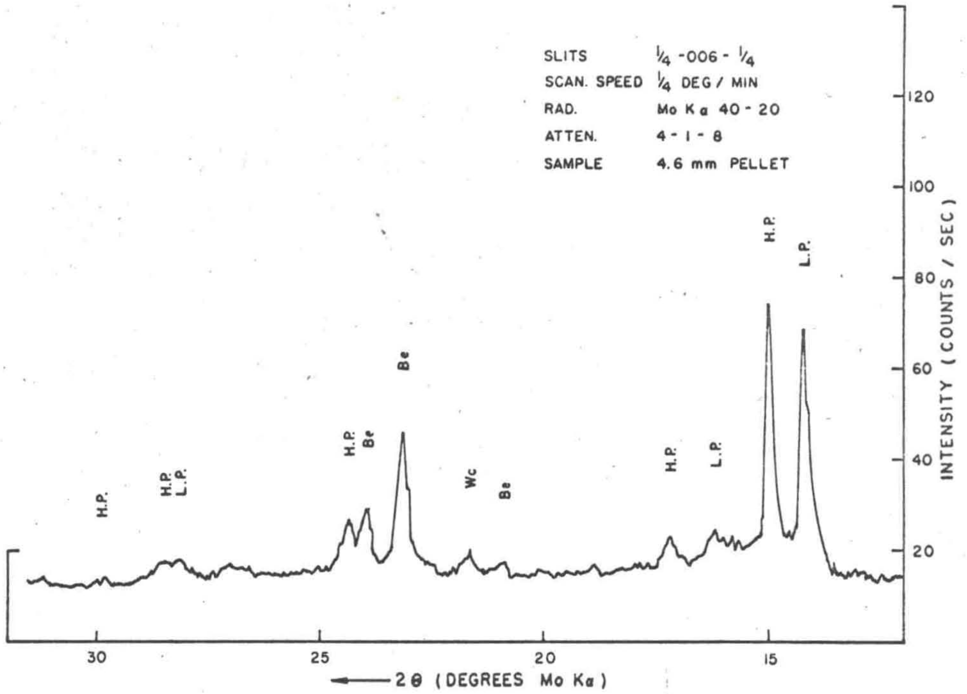

FIG. 4.—Diffraction pattern of Ce under a *nominal* pressure of 8.1 kb. Molybdenum radiation beryllium anvil. The peaks of both the low-pressure form and the high-pressure ("collapsed") form are present in the pattern.

The beryllium used for these measurements contained over one per cent of beryllium oxide. Evidence of this may be seen from the fact the stronger peaks of BeO are found on some of the records. Fortunately the strong beryllium oxide peaks (e.g., at 45.80°, 50.93°, and 52.80°, 2θ, for Cu Kα) are relatively few in number, and like those of diamond and of beryllium metal, are easily identified.

Previously, beryllium has been used in various ways for obtaining powder diffraction patterns under pressure, especially by Jacobs,[7-10] Lawson and Riley,[12] Vere-

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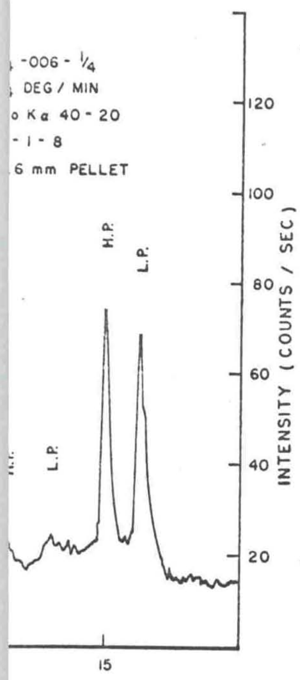

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schagin *et al.*,[13] and Guengant and Vodar;[14] but it is the combination of beryllium squeezer and copper radiation, as now described, that give the especially useful chart records.

Transition Rates.—A principal objective of the present investigation was to learn something as to the rates at which polymorphic transitions take place at high pressures. Somewhat to our surprise, we have found that with the "squeezer" and with the materials here reported on, the transition rates, either from low-pressure form to high-pressure form, or from high to low, proceed quite rapidly even at ordinary temperatures. In each case the transition with moderate overstepping (or understepping) of pressure was essentially complete in a matter of seconds rather than minutes. It is quite likely that the special conditions of stress in the "squeezer" (i.e., deformation and shear) are largely responsible for the fast transformation. This matter will be considered further in connection with some results, soon to be published, on transition rates of materials under pressure in a beryllium cylinder.

Summary.—With diamond pistons, and also with those of beryllium, operating on the "squeezer" principle, X-ray patterns have been obtained on the Norelco diffractometer for various materials at pressure above and below the transition pressures. Both molybdenum Kα radiation (with diamond and with beryllium) and copper Kα radiation (with beryllium) were used. At present, the maximum pressures withstood by diamond are much less than had been anticipated. Beryllium squeezers, on the other hand, were usable to gratifyingly high pressures. The recorded angles, corrected by means of the remanent low-pressure peaks, yield values of satisfying precision for the parameters of the high-pressure forms.

Although it was easy to observe the change in height of a given peak as it took place during a transition, it turned out that in the squeezer, and with the materials here investigated, the transitions (contrary to our expectations) took place very rapidly, the bulk of the change occurring in a few seconds.

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*MAGNETOHYDRODYNAMIC WAVES IN A CONSTANT DIPOLE MAGNETIC FIELD**

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1. *Introduction.*—Dungey[1] in his remarkable report of 1954 has discussed in some detail the electrodynamic behavior of the outer atmosphere in the presence of a constant dipole magnetic field. We shall here approach the problem from a different point of view, however, concentrating our attention (as we did in our previous studies[2, 3]) on the vorticity field and the current density.

Consider an infinite mass of an electrically conducting fluid at rest, embedded in a constant dipole magnetic field $\mathbf{H}$. To simplify the discussion, take the conductivity as infinite (an approximation justified for a large-scale disturbance) and assume the fluid to be a homogeneous incompressible material. Assume that as a result of a perturbation, a velocity field $\mathbf{v}$ is produced in a certain region and that the magnetic field becomes $\mathbf{H} + \mathbf{h}$. The amplitude is assumed to be small enough for nonlinear terms to be neglected. We propose to investigate the magnetohydrodynamic behavior of the fluid in terms of generalized Alfvén waves by means of vorticity and current density. The equations obtained are complicated, however, and solutions will be discussed only for large distances from the center of the dipole. We shall conclude this note with an appendix where the geometry of lines of force is briefly discussed; the results obtained there are not all original, but are essential to an understanding of the subject.