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Abstract

Full Text

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OPTICAL STUDY OF PHASE TRANSITIONS UNDER PRESSURE

(Presented by Academician A. N. Terenin on 2 January 1962)

Optical studies of phase transitions under pressure were first carried out by Drickamer (1). He observed a sharp clouding of specimens at the moment of change in their crystal structure during the transition. We studied first-order phase transitions in polycrystals of RbCl, KBr, and KCl, transparent over a wide spectral region. For this purpose a method was developed for optical detection of transitions, which can also be used for calibrating high-pressure chambers intended for spectral studies.

For the static compression of solid objects, a chamber with rock-salt windows was constructed (Fig. 1). It withstood up to 80 experiments at pressures not exceeding 30,000 kg/cm². The substance under study was placed in a medium of the same salt and compressed by pistons 2, 4 in a cylindrical volume 1 of 5 mm diameter. The design of the chamber and the manufacturing technology of its windows differed from those proposed by Drickamer (2). Conical windows 8 were used, within whose thickness the negative pressure gradient changed gradually, owing to which their strength increased. To "enlighten" the salt windows, they were repeatedly subjected to all-round compression at a pressure of not less than 25,000 kg/cm²; this was repeated after loading the substance under study into the chamber. The height of the salt column containing the object was varied by the thickness of the steel gasket 9. Matrix 3 was pressed into cone 6 and fixed by nut 5. The windows were secured on the outside by obturators 7. The matrix and pistons were made of ShKh-15 steel and hardened to a hardness of 58-62 on the Rockwell C scale; the remaining parts were made of 45KhNMFA steel with hardening to 44-48 units.

Fig. 1. Chamber for optical studies under pressure up to 30,000 kg/cm²

Measurements of the transparency of the specimens were carried out on an apparatus consisting of a DATS-50 point light source, a high-pressure chamber placed between the pistons of a hydraulic press, and a photoreceiving device. The light beams were focused by means of a combination of spherical and plane

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mirrors. The amplified signal was recorded on the chart of an EPP-09 electronic potentiometer. The magnitude of the light flux passing through the specimen was continuously recorded during cyclic variation of the pressure. The cycle consisted of a direct run, in which the pressure was increased until completion of the transition of the substance into the new phase, and a reverse run, in which the substance returned to its initial state as compression was reduced. The pressure values were marked on the chart by a special reference device.

In the salts RbCl, KBr, and KCl, phase transformations of the crystal lattice from the NaCl type to the CsCl type occur at pressures of 5000, 18400, and 20060 kg/cm², respectively; they are accompanied by a strong change in volume (³). These salts are convenient in that they do not react irreversibly with NaCl, which was used as a solvent in those cases where the samples were not sufficiently transparent. The salts KBr and KCl were studied both in pure form and at 10% concentrations; the salt RbCl was studied at concentrations below 20%.

Fig. 2. Change in transparency of a KCl 10% sample with changing pressure

Fig. 3. Change in transparency of an RbCl 13% sample with changing pressure

The changes in transparency on the diagram appeared in the form of a peak, since both phases of each salt are isotropic, and after completion of the rearrangement of the crystal lattice the samples again restored their transparency. In KBr and KCl samples, at the moment of the phase transition it decreased (Fig. 2), while in RbCl samples it increased (Fig. 3).

For each salt there are characteristic half-widths of the peaks and the magnitude of the lag of the transformation processes (⁴), equal to the difference between the pressures corresponding to the peaks in direct and reverse experiments. The lag is determined by the elastic properties of the substance, while the peak half-width is determined by the rate of formation of the new phase in the parent phase. The lag did not depend on the concentration of the salt under study, while the peak half-width increased as the concentration decreased. For RbCl the lag was 8350 kg/cm², for KBr 16500 kg/cm², and for KCl 17 000 kg/cm². In direct experiments the peak half-width was, for RbCl 13%, 500 kg/cm², KBr 20%, 2400 kg/cm², and KCl 20%, 2800 kg/cm². In reverse experiments the half-width increased in different ways.

The decrease in the transparency of samples during phase transitions, also ob-

Fig. 4. Calibration curve of the chamber (F –load, P –pressure)

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served earlier ⁽¹⁾, was explained by an increase in the scattering of the incident light on nuclei with a CsCl-type crystal lattice arising among crystals with a NaCl-type lattice ⁽⁵⁾. We observed for the first time an increase in the transparency of RbCl during the transition. The previous hypothesis does not explain this phenomenon. Apparently, its cause should be sought in the change in the sizes of the crystallites of both phases of the salt during the transition ⁽⁶⁾, which may lead to a decrease in scattering at suitable crystallite sizes.

The results obtained were applied to the calibration of this high-pressure chamber, i.e., to establishing the relationship between the magnitude of the force developed by the press and the pressure that was genera-

were placed in the chamber. The loads corresponding to the phase-transition pressures were calculated as the mean of the results obtained on increasing and releasing the pressure (4). The points RbCl, KBr, and KCl on the calibration curve (Fig. 4) were determined by means of the method developed. The initial point at zero pressure was established directly from the magnitude of the load needed to overcome the frictional forces in the press. The Bi point was obtained by measuring the jump in electrical resistance at the transitions BiII \rightarrow BiIII \rightarrow BiIII (7) at an average pressure of 26,000 kg/cm², which are not resolved under quasihydrostatic conditions in our apparatus. The resistance measurements were made according to a circuit with a low-resistance PPTN-1 potentiometer; the stationary piston 2 in the chamber was insulated from the body by a paper gasket and by nut 5 made of ebonite.

Fig. 4. Calibration curve of the chamber (F –load, P –pressure)

The calibration curve, at loads greater than 4.4 tons, gradually changes its slope, which is caused by elastic deformation of the movable piston and, consequently, by an increase in the frictional forces in the chamber. In the future, chambers used in optical investigations of solids under pressure can more simply be calibrated by the method described, using the phase transitions of the three salts indicated.

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