X-RAY DIAMOND CELL WITH THE ROTATING ANVIL FOR THE SHEAR DEFORMATION (DACS)

V. D. Blank, N. R. Serebryanaya, A. A. Vorontsov, A. Yu. Zerr*

Institute for High Pressure Physics of Russian Academy of Sciences,
142092, Troitsk, Moscow region, Russia

Powder diffraction has been applied to a diamond anvil cell for the shear deformation (DACS). The adjustment of DACS is carried out by using a laser beam. The shear deformation is caused by the rotation of the anvil support via a thrust bearing. The MoK_α-radiation and film technique were used. The pressure distribution is determined by the ruby-luminescence and the diffractional data of the sample compressibility. The method was tested by the investigation of PbTe-phase transitions.

Introduction. The DACS has been constructed earlier for the optical [1–3] and electric resistance [4] investigations. The support of one of anvils was attached to the chamber wall via a thrust bearing that made it possible to rotate the support by up 360°. This rotation causes a shear deformation. The sample is loaded without a pressure transmitting media. The main result of these unvestigations is a decrease of the phase transition pressure.

The present study is aimed at an application of the X-ray powder diffraction to DACS. For this purpose the diffractional slot (0.4×4.0 mm^2) has been cut into the cylinder tungsten-carbide rocker of DACS and a scattering angle2Θ is about 40°

The lead telluride (PbTe) was chosen for testing of methods. PbTe is crystallized in the NaCl-structure type at normal conditions. At high pressure there are two transitions of PbTe, which are accompanied by a sharp increase of the resistance and a change of the crystal structure.

I transition: cubic type NaCl → orthorhombic type GeS, $P = 5$ GPa [5], $P = 6$ GPa [6].

II transition: orthorhombic type GeS → cubic type CsCl, $P = 13.5$ GPa [6], $P = 16.0$ GPa [7].

At the first transition atoms of lead are displaced from the symmetry centre into the face of the cube, according to the relations of unit-cell dimensions of normal and high pressure phase [8]. The result of the displacement is a shift and goffering of NaCl-layers. At the second transition atoms displace into the face-diagonal direction, forming the primitive cubic structure of the CsCl-type.

X-ray diffraction methods in DACS. The adjustment of DACS is worked out by using a laser beam. Figure 1 shows the disposition of DACS and the laser on the X-ray source table (MoK_α, $W = 300$ Wt). The DACS adjustment includes the coincidence of the load axis with the X-ray beam. This procedure should ensure that the beam passes through the centre of the gasket hole and the series of steps described below is achieved.

Firstly, the helium-neon laser, disposed on the adjusting device *2*, is set parallel to a guide rail *3*, then we make the laser beam to coincide with the collimated X-ray beam by using the screw *4* and screws of the adjusting device *2* for the translation along *X*, *Y*, or *Z*-axes. Next the device *5* together with DACS *6* and the flat film

* Hohdruckgruppe, Max-Planck-Institute fur Chemie, Postfach 3060, 6500 Mainz-1, Federal Republic of Germany

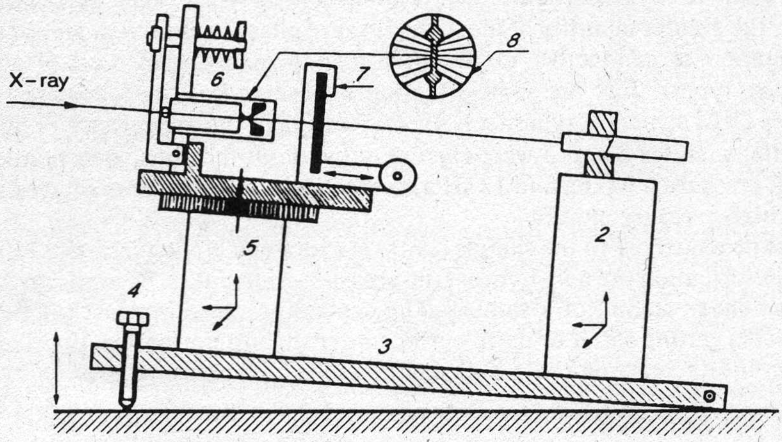

Fig. 1. The scheme of the disposition of DACS and the adjusting laser at the X-ray source-table

cassete *7* are set on the guide rail *3*. The diffractional slot is disposed verticaly as regards the direction of X-ray.

The cassete is transfered, permitting three patterns to be obtained on the same film. The DACS movement along *x*, *y*, *z*-coordinates and the rotation about the limb axis passing through the sample, are provided by the adjusting device *5*. The insertion *8* shows the disposition of diamond anvils with the sample.

Further, the gasket hole (without the sample), coincident with the anvil centre, is preliminarily set along the maximum laser beam. The most accurate adjustment is being carried out by X-ray beam maximum, when the sample is in the gasket hole. The laser adjustment is generated for the further development at the synchrotron.

The effect of the shear deformation on the crystal structure was studied by the following way. First the diffraction pattern is obtained at the definite pressure of the sample centre. Then the shear deformation is made by the smooth rotation (in 10 sec) of the movable support of the anvil through an angle $\varphi = 5°$ and the movable assembly is rotated back to the initial position. The pressure distribution is recorded and the pattern is produced once again. This procedure is repeated after each load. If it is necessary the additional rotation is made.

The fine-focus $(0.1 \times 0.1\ mm^2)$ tube and a collimator with the hole diameter 0.1 mm is used for the production of patterns. The diamond culet size is 400 μm. The beforehand compressed Nb-gasket is used. The hole diameter of the gasket is equal to 0.2 mm.

The pressure distribution is determined by the rubi-luminescence [9]. At the precise adjusting of anvils, the bell-mouthed pressure distribution with the maximum of the anvil centre is produced. The application of the ruby as a nonhydrostatic pressure indicator is proved correct by the luminescence determination before and after the shear deformation [1].

Results. Table shows results of the PbTe-investigation at different pressures of the sample centre and different rotation-angles φ. In the range far from the transition-pressure (up to 4.0 GPa) a shear strain of a different φ had no practically effect on the crystal structure (the series 1).

At the higher pressure (4.6 GPa, ser. 2) the rise of the central pressure up to 6.5 GPa is observed after the shear deformation. The traces of a GeS-type phase are found at the X-ray patterns after the rotation of the $\varphi = 5°$ angle, at the larger angle (40°), the greater quantity of the high pressure phase is observed. Hence the shear deformation is conductive to the first high pressure transition (NaCl → GeS structure types). Just the same situation is observed at the following transition (GeS → CsCl structure types, ser. 4). For testing of the appearance of both phase transitions, series 3 and 5 were carried out without the shear deformation at the central pressure 6.6 GPa and 12 GPa, respectively. The patterns showed no traces of the high pressure phases.

The pressure rise in the sample centre is explained by Boguslavskii [10]. At the shear deformation the non-hydrostatic pressure distribution depends on the elastic and the shear moduli of a sample. The central pressure increases if the sample material is getting softer or more plastic under shear deformation [9]. Taking into account the decrease of PbTe-bulk moduli at the high pressure (B_0 = 38.9 GPa for NaCl-type, B_0 = 36.8 GPa for GeS-type and B_0 = 38.1 GPa for CsCl-type [5]), under the shear deformation the increase of the central pressure is getting more clear.

Table shows the transition pressure, obtained from X-ray patterns and valued by the volume compressibility of the unit cell [7] and bulk moduli [5]. The decrease of X-ray pattern pressure of transitions is possible to be explained by the peculiarity of the pressure distribution. It is experimentally determined that the non-hydrostatic conditions lead to the displacement of the centre of gravity of diffractional lines to smaller angles Θ°, because of the increase of the sample-material quantity

Table. The pressures of PbTe transitions determined by the X-ray (P_x) and the ruby-luminescence (P_c) methods

Series N	Before shear def.			After shear def.		
	P_c, GPa	P_x, GPa	φ°	P_c, GPa	P_x, GPa	structure of phasees
1	4.0	2.0	5.4			NaCl
2	4.6	1.7	0			NaCl
	4.6		5	6.6	3.0	NaCl+3%GeS
	4.6		40	6.6	3.4	NaCl+50%GeS
3	6.6	1.23	0			NaCl
4	9.6	3.8	0			NaCl+15%GeS
	9.6		40	12.0	10.2	GeS+3%CsCl
5	12.3		0			NaCl+20%GeS

at the sample-edge. Owing to the bell-mouthed-pressure distribution, the pressure of the sample-edge is by far less than the central pressure. For example, in the series 2: the central pressure is equal to 4.6 GPa before the shearr deformation, the edge pressure is equal to 1.7 GPa. The greater part of the intensity is contributed by the peripherical part of the sample because of its larger volume. Therefore the X-ray-determined pressure is to be less than the pressure of the central part determined by the ruby-luminescence, and if the X-ray pattern shows the appearance of the pressure-induced phase, the X-ray-determined pressure is smallest for the pressure transition.

Thus, the shear deformation initiates the phase transitions at the pressure lower , than the transition pressure under hydrostatic conditions. The first transition of PbTe (NaCl → CsCl types) begins at 3.0 GPa, and the second transition (GeS → CsCl types) begins at $P < 10.2$ GPa. These transitions have never been observed at these pressures under hydrostatic conditions, even at the unloading.

In conclusion, the authors would like to notice the lack of the X-ray determination of pressure from the impossibility of the pressure-determination at each point of the sample as such as by the ruby-luminescence method.

1. *I. A. Barabanov, V. D. Blank, Yu. S. Konyaev*, Pribory i Technika Experimenta 2, 176 (1987).
2. *V. D. Blank, S. G. Buga*, Pribory i Technika Experimenta 4, 180 (1992).
3. *V. D. Blank and A. Yu. Zerr*, High Pressure Research 8, 567 (1992).
4. *V. D. Blank, V. G. Danilov, I. A. Lastenko*, Pribory i Technika Experimenta 2, 186 (1990).
5. *N. R. Serebryanaya*, Izvestia Academii Nauk USSR, ser. Neorganicheskie materialy 27, 1611 (1991).
6. *T. Chattopadhyay, A. Werner, H. G. von Schnering, J. Pannetier*, Rev.Phys.Appl. 19, 807 (1984).
7. *Y. Fujii, K. Kitamura, A. Onodera, Y. Yamada*, Solid State Comm. 49, 135 (1984).
8. *S. S. Kabalkina, N. R. Serebryanaya, L. F. Vereschagin*, Fizika Tverdogo Tela 10, 734 (1968).
9. *V. D. Blank, Yu. Ya. Boguslavskii, M. I. Eremets et al.*, Sov. Phys. JETP 60, 525 (1984).
10. *Yu. Ya. Boguslavskii*, Fizika Tverdogo Tela 33, 2689 (1991).