SUBSTANCE FLOW IN A DIAMOND ANVIL CELL CAUSED BY STRUCTURAL PHASE TRANSFORMATION PROCESS AND ADDITIONAL SHEAR DEFORMATION

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Introduction. Investigation of solids under high pressure and great shear deformations in Shear Diamond Anvil Cells [1–3,6] is very important because at these mixed conditions the simulations of natural processes (geology, geophysics) and technology (metallurgy, technology of semiconductors, cold welding, etc.) can be provided.

This method was successfully applied to optical, X-ray, electric and mechanical investigations of the pressure-induced phase transitions in solids [1–7].

Additional shear deformation of the sample under constant load on the chamber is performed by smooth rotation of one of the couple of anvils around axis of the load in a specially designed Shear high-pressure cells [1–4,6,7]. These additional deformations lead to the substance flow in the chamber and this motion affects the physical processes in the chamber. Otherwise, investigations of parameters of that substance flow at nonhydrostatical conditions can give information about the physical conditions in the chamber and physical properties of the substance being studied.

Methods and the experimental results. We have used the computer-based image processing system, manufactured by "Omega", Poland for the investigation of substance flow in the Shear DAC. In Fig. 1,*a–d* development of the structural phase transition in ZnSe sample under constant load and increasing angle of rotation of the anvil is shown. Fig. 1,*e,f* shows the images of the sample after decreasing of the load and further shearing. The diameter of the sample is equal to the diameter of the working surfaces of the anvils 0.4 mm. Thickness of the sample is $20-30 \cdot 10^{-3}$ mm. In the Fig. 2,*a,b* the images of KCl and NaCl samples of the same size, as ZnSe sample are presented.

ZnSe is initially transparent and it is known [8] that it undergoes phase transition at 13.7 GPa to the opaque phase. It differs from the initial phase by the type of structure (sphalerite → rocksalt), greater density and greater bulk elasticity coefficient.

KCl also undergoes structural phase transition, but at lower pressure ~ 2.3 GPa, with the type of the structures B1→B2.

Substance NaCl is chosen to observe the motion in the sample which has no phase transition in this pressure region.

Black points on the samples images (Figs. 1,2) — are the particles of ruby, indicating the value of pressure at the point through the change of fluorescence spectra. Black region in the central part of the ZnSe sample is the opaque high-pressure phase.

Fig. 2,*c,d* and Fig. 3 show the trajectories of the motion of ruby particles and, correspondingly, substance flow, caused by the rotation of anvil in the DAC. These pictures were obtained by subtracting images one from another, with every image corresponding to a shear by 3–5° step. If the particles were not displaced after shear deformation, then brightness of such points was equal to zero in the resulting image. But if the particles were displaced, then the brightness of the points was not a zero

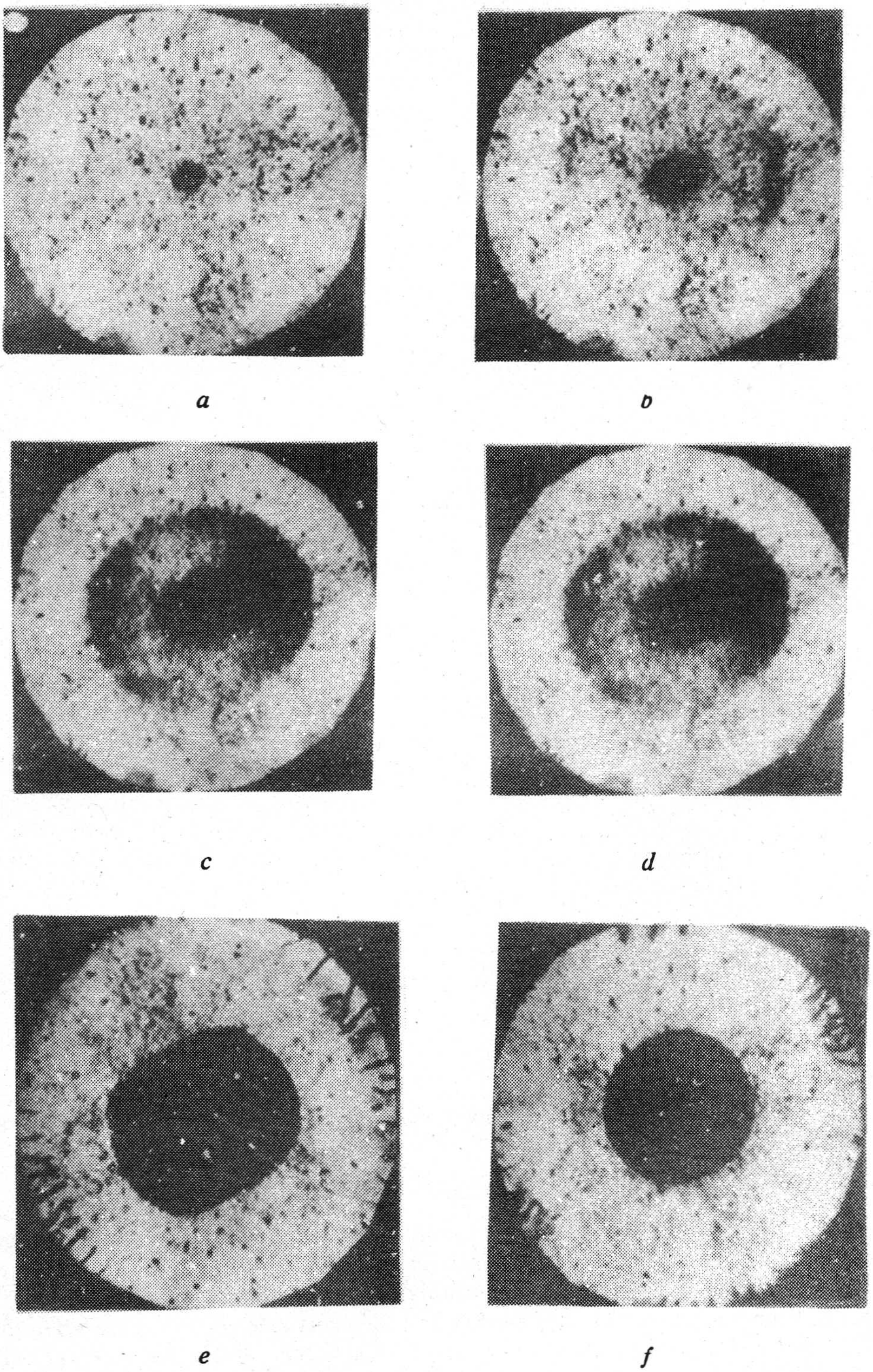

Fig. 1. Development of the phase transition in ZnSe under constant load and shear deformation angle φ : *a)* $\varphi = 0°$, direct transition; *b)* $\varphi = 3°$; *c)* $\varphi = 6°$; *d)* $\varphi = 11°$; *e)* $\varphi = 0°$, reverse transition; *f)* $\varphi = 25°$

one, thus the motion of substance was registered. Figs. 2, 3 show the pictures of subtraction with inverted brightness for better presentation.

One can observe, that all pictures of the motion presented have some similar and some peculiar features.

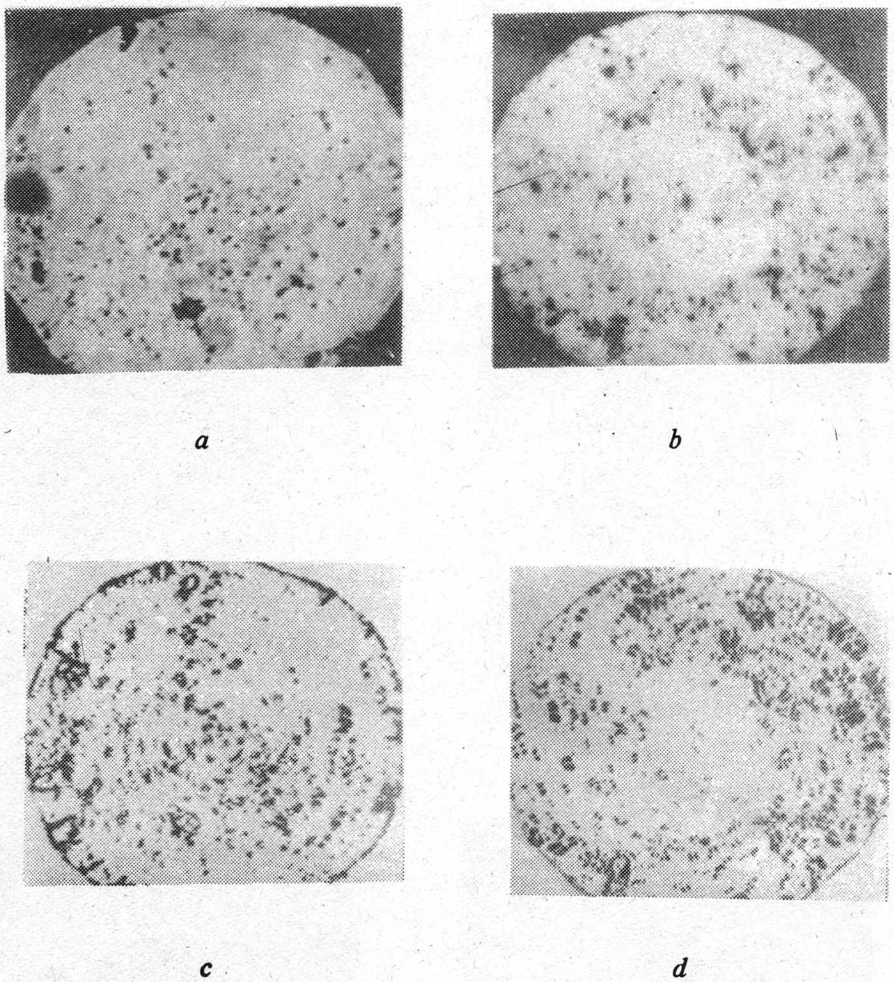

Fig. 2. Images of the NaCl (*a*) and KCl (*b*) samples under pressure, and trajectories of ruby particles under shear deformation in NaCl (*c*) and KCl (*d*)

The shape of the trajectories of the motion greatly varies in different substances. In NaCl the trajectories of the particles look almost like circles with a slight spirality (Fig. 2,*c*). Trajectories at KCl look spirals (Fig. 2,*d*). Character of the motion in ZnSe is more complicated. It is hard to find the exact rule of the motion when the first steps of the rotation was performed (Fig. 3,*a,b*), but when the load was decreased, and reverse transition started, shear deformation caused the spiral-type trajectories (Fig. 3,*d*). During the first step of the deformation at the reverse transition conditions (Fig. 3,*c*), the shape of the trajectories is somewhere similar to the shape of the phase boundary.

Another interesting phenomenon of the shear deformation of substance under condition of direct structural transition (Fig. 2,*d*; 3,*a*) is that the motion in the region of high-pressure phase is less intensive, than in the peripheral area, occupied by the initial phase. It is easy to see also the evidences of the 2nd-order symmetry type in the picture of the motion in the ZnSe sapmle (Fig. 3,*d*). This 2nd-order symmetry emerges also in the shape of the high-pressure phase (Fig. 1,*b*–*e*).

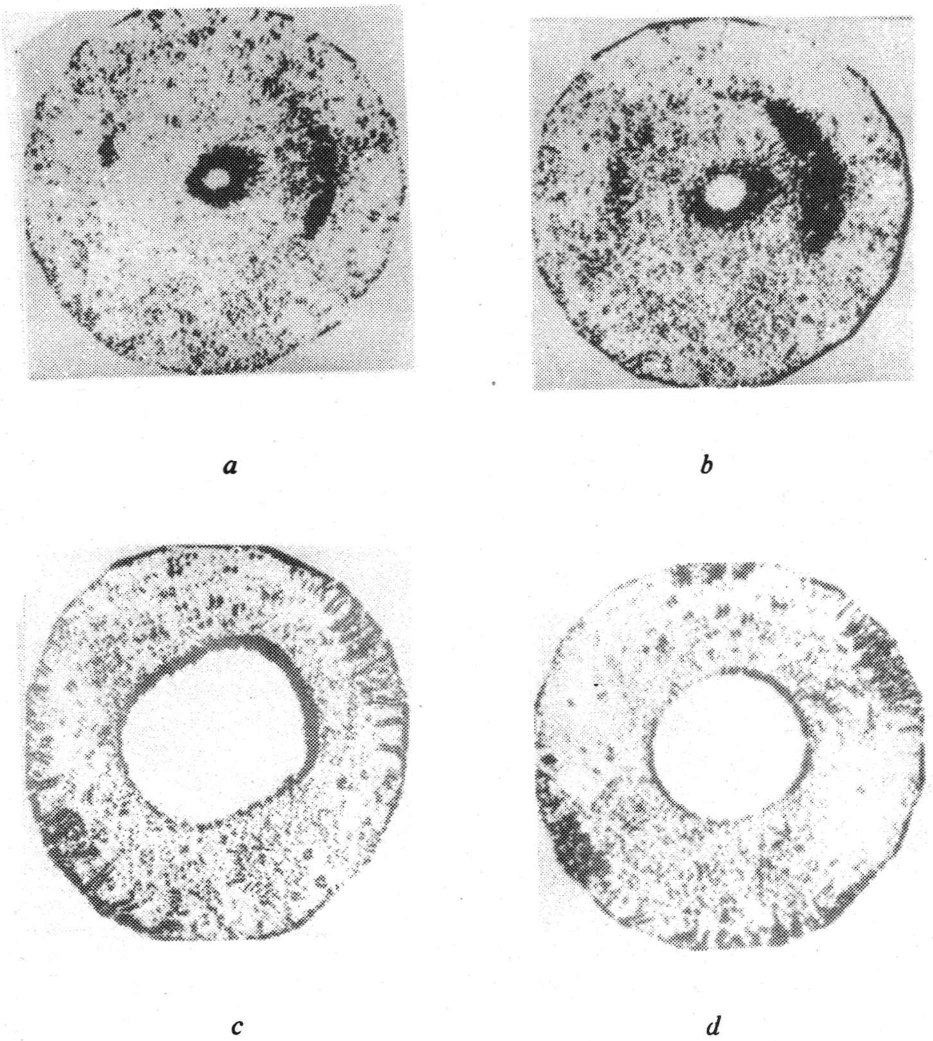
a *b* *c* *d*

Fig. 3. Trajectories of ruby particles in ZnSe sample under pressure and shear deformations: *a*) the 1st shear of $\varphi = 3°$ under direct transition; *b*) the 2nd shear; *c*) shear of $\varphi = 5°$ under reverse transition; *d*) the 4th shear of 5° under reverse transition

When trying to understand the reason for thase peculiarities of the substance flow in chosen samples, it is important to know the pressure distributions along the surface of the anvils in the chamber.

Distributions of pressure in KCl and NaCl before and after shear deformation were investigated in [1].

In Fig. 4 the 3-D distributions of pressure in ZnSe sample are shown.

3–Dimentional distributions of pressure were obtained with the use of a specially designed computer-based system for scanning of the sample with a step of 20 microns, datd acquisition and processing. Technique of pressure measurements in the point, similar to the described in [9], was used.

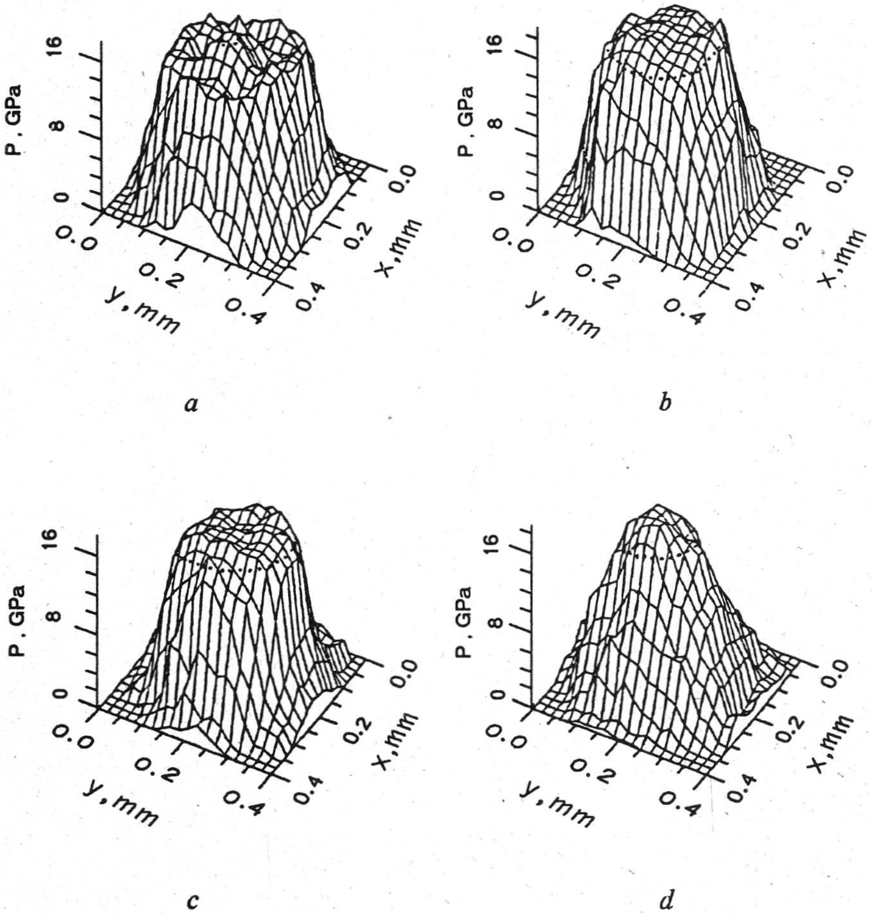

Fig. 4. 3-Dimensional distributions of pressure along the surfaces of the anvils in the working zone of the Shear DAC with ZnSe sample: *a)* $\varphi = 0°$, direct transiton; *b)* $\varphi = 11°$; *c)* $\varphi = 0°$, reverse transition; *d)* $\varphi = 25°$. Dotted lines show the position of a visible phase boundary

Discussion. The simplest picture of the substance flow takes place in Fig. 2,*c*, which corresponds to the NaCl sample. Maximum pressure value in the central part of the sample was ~ 7 GPa. Circular shape of the trajectories permitted conclude, that under these experimental conditions (diameter of the sample, value of the load, thickness of the sample), shear deformation occurs in a form of almost pure rotation, with a small radial component of the motion in the edges of the sample.

Maximum pressure value in the central part of the KCl sample was ~ 6 GPa. Great difference in the intensity of the motion in the central part of the sample and in the peripheral area probably can be explained by the existence of the structural phase transition in this sample. High-pressure phase B2 has greater bulk elastic coefficient, than the initial B1 phase. Thus the central part of the sample (phase B2) is compressed stronger than the peripheral part (phase B1) [1]. Under these conditions the value of deformation depends nonlinearly on the radius of the rotation, though the stresses of the additional deformation is the linear function of the radius. Furthermore, due to the great pressure gradient, which takes place at the pressure self-multiplication conditions [1], the radial component of the flow is observed. That is the reason for the spiral-like trajectories of the motion (Fig. 2,*c*; 3,*f*).

The ZnSe substance flow caused by shear under direct transition (Fig. 3,*a,b*) is more complicated for interpretation, than the results of the experiment with NaCl, KCl or ZnSe under load decrease. Fig. 3,*a* shows, that the intensity of motion was not homogeneous along the square of the sample after the first step of deformation, when the load was increased. The explanation for this fact is probably the same as in the case with the KCl sample, because strong pressure gradient takes place (Fig. 4). The response to the next step of the rotation of the anvil is different as compared to the first one (Fig. 3,*b*). The intensity of the motion in the central and the peripheral parts is not so different, with the only exception in the top part of the picture. Redistribution of the pressure along the anvils after the first rotation may be the reason for the different behaviour of the substance flow in these cases.

It is very interesting that the annulus fall of pressure is present in the initial pressure distribution and absent after shear in ZnSe sample (Fig. 4). It is important, that the pressure maximum in the chamber has grown from 17.5 GPa to 18.5 GPa in the case of the direct transition (load increase) and from 17.0 to 18.5 GPa in the case of the reverse transition (load decrease). It should be noted, that such growth of the maximum pressure corresponds to the phase transition development, activated by shear stress at constant external load.

This effect occurs because there exists the annulus area of metastable (so-called two-phase state) besides pure initial and high-pressure phase areas. The existence of this region is explained by the pressure hysteresis of the transition. Shear deformations lead to a smaller hysteresis, resulting in a modification of elastic properties in this annulus region after shear [1,10].

The character of the ZnSe substance flow is different in the cases of direct and reverse transition, as seen from Fig. 3. If sample has been deformed under the condition of a reverse transition, then the trajectories of particles are circle-like with small radial component (Fig. 3,*d*). The shape of the trajectories resembles the one of the NaCl and KCl samples under the load increase (Fig. 2, *c,d*). But after the first step of the rotation in the case of the load decrease the rule of the motion is not clear (Fig. 3,*c*).

We suppose, that the difference between the character of the motion in ZnSe in the cases of direct and reverse transitions can be explained by the effect of the volume jump. Therefore, two tendencies of radial motion take place in the case of direct transition–motion to the edges of the sample caused by the pressure gradient and motion to the center, caused by volume decrease of high pressure phase. Since local maximum of pressure occurs in a certain annulus area (Fig. 4,*a*), radial motion must be absent there.

When load was reduced and reverse transition started, volume of substance in the central part began to increase. That led to a radial motion of the substance. The trajectories of the particles look like spirals (Fig. 3, *d*).

The appearance of the 2-nd order symmetry in the picture of the ZnSe flow (Fig. 3, *d*) is of great interest. This phenomenon of the 2-nd order symmetry in the pictures of the phase transition and substance flow may be the consequence of the crystal properties of the diamond anvils, which are cut off the diamond monocrystals, thus having anisotropic elastic properties. That must be essential at high pressure region (above 10 GPa probably).

Conclusion. Investigations of the different substances flow performed in the Shear DAC drive us to a conclusion, that in one-phase system (like NaCl at chosen pressure region) additional shear stresses obtained by rotation of the anvil around the axis of the load, cause simple circle-like substance flow with a small radial component. The dependence of deformation on the shear stress is close to linear.

Substance flow in a two-phases systems (KCl, ZnSe) is more complicated. Pictures of the flow provide additional information about the kinetics and other parameters of phase transitions. The obtained results can be described by the earlier performed theoretical investigations [1,10].

Pictures of the substance flow in ZnSe reveal the 2-nd order symmetry of the motion, which is characteristic for the picture of the phase transition as well. This is probably the consequence of the monocrystalline properties of the diamond anvils.

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