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X-Ray Diffraction Studies of Structures of Be, Al, LiF, Fe+3%Si, Si, SiO₂, KCl under Dynamic Pressures from 2 Gpa to 20 Gpa

L.A. Egorov, A.I. Barenboim, V.V. Mokhova, V.V. Dorohin and A.I. Samoilov

Russian Federal Nuclear Centre-VNIIEF, Nizhni Novgorod Region, 607190 Sarov, Russia

Abstract. Currently, the only direct method to study behaviour of solid crystal substance structures under dynamic compression is method to record X-rays diffraction pictures of crystal structures under shock compression. The paper presents results of X-rays diffraction measurements concerning structural parameters of shock compressed substances at pressures higher than Hugoniot elastic limit (Be, Al, LiF, Fe+3%Si), lower than Hugoniot elastic limit (Si, SiO₂, LiF) and in the area of pressures of phase transformation beginning (KCl, Si). Recorded states of shock-compressed substance structures demonstrate identity of structural deformations at pressures higher and lower than Hugoniot elastic limit as well as at pressures above the phase transformation point, which can be characterized as single-axial deformations.

Resume. La méthode d'enregistrement de la structure des cristaux comprimés par l'onde de choc est un seul procédé direct jusqu'à présent permettant d'étudier le comportement des structures des solides cristallins comprimés dynamiquement. Les paramètres de la structure des matières comprimées par le choc, mesurés par la méthode de diffraction aux rayons X dans le domaine des pressions au-dessus du limite élastique de Hugoniot (Be, Al, LiF, Fe + 3%Si), au-dessous du limite élastique de Hugoniot (Si, SiO₂, LiF) et dans le domaine des pressions correspondantes au début de la transformation de phase (KCl) sont présentés dans l'exposé. Les états déséquilibrés de la structure des matières comprimées montrent l'identité des déformations de structure dans le domaine des pressions au-dessus et au-dessous du limite élastique de Hugoniot, aussi bien que dans le domaine de la transition de phase, lesquelles peuvent être caractérisées comme monoaxiales.

1. INTRODUCTION

From 1981 to 1993 RFNC-VNIIEF implemented the program of dynamically compressed crystal studies using the technique of X-ray diffraction measurements of parameters realized behind the shock front of the structures. Polycrystalline samples of beryllium, silicon iron, aluminum, as well as monocrystalline samples of lithium fluoride and potassium chloride were studied at pressures higher than Hugoniot elastic limit. Monocrystalline samples of silicon, α -quartz and lithium fluoride were studied at pressures below Hugoniot elastic limit. The recorded diffraction patterns of samples with various initial packings (Be is of hexagonal close-packed structure, Fe + 3%Si of body-centered cubic structure, Al of face-centered cubic structure, Si of diamond type structure, SiO₂ of trigonal structure, LiF and KCl of NaCl type structure) demonstrate the universal process of structural material reorganization leading to realization of two structural components nonequilibrium and equilibrium, behind the shock front. The data of the photographs demonstrate, that the structural transformation proceed on shock front and outside front transformation process slows down. It allows to observe «frozen» structural states, in which the part of substance has equilibrium structure, and part - nonequilibrium. Formation nonequilibrium structure is possible to present as result of compression of initial structures of crystals cubic and trigonal syngonies along the crystallographic direction $\langle 111 \rangle$, hexagonal syngony - $[001]$.

2. RESULTS OF X-RAYS-DIFFRACTION MEASUREMENTS CONCERNING STRUCTURAL PARAMETERS OF CRYSTALS AT PRESSURES HIGHER THAN HUGONIOT ELASTIC LIMIT

Photographs are taken from [1]. There are presented geometry of the scheme of experiment, characteristic of the used hardware complex and procedure of measurement.

Fig. 1

Fig. 2

FIG. 1. Diffraction photograph of beryllium structure under pressure $P = 22.8$ GPa, $\sigma = 1.16$, and the geometry of the dynamical loading assembly: 1-striker (Al) with thickness $L = 6$ mm, velocity $W = 3.65$ km/sec; 2-Li layer ($L = 15$ mm); 3-sample; X- x-ray (the characteristic molybdenum emission)

FIG. 2. Diffraction photograph of the structure of silicon iron under pressure $P = 6.8$ GPa, $\sigma = 1.04$; 1-striker (Cu, $L = 10$ mm, $W = 0.62$ km/sec); 2-sample ($L = 0.3$ mm); 3-screen (Be, $L = 5$ mm); 4 - diffraction reference: RS-reference signal.

Value of compression of a sample $\sigma = V_0/V = D/(D-u)$. D - speed of a shock wave, u - mass speed of material behind shock front, V_0 and V - specific volumes of initial and compressed states of a sample accordingly. The diffraction reflections from the (010) and (011) planes of the hcp structure of beryllium are recorded in the photograph (Fig. 1). It is sufficient to know the angular distance between the (010) and (011) peaks and the volume of the hexagonal cell in order to determine uniquely the cell parameters:

$a = 2.28 \pm 0.01 \text{ \AA}$ and $c = 3.1 \pm 0.01 \text{ \AA}$. The volume of the hexagonal cell was taken to be the cell volume of the initial state of the structure of the sample reduced by a factor of σ . Comparison with the parameters of the initial state of the beryllium structure - $a_0 = 2.286 \text{ \AA}$ and $c_0 = 3.584 \text{ \AA}$ - shows that, to within the measurement error, the cell is compressed along a single crystallographic direction

(the C axis is compressed) and it does not depend on the orientation of this direction with respect to the shock front.

The silicon iron (Fe + 3 wt.% Si) structure exhibits dynamical deformation of the same uniaxial character (Fig. 2). The photograph recorded three peaks with diffraction angles θ_i and the corresponding interplanar spacings d_i

$$\theta_1 = 10.1^\circ, \quad d_1 = 2.024 \text{ \AA},$$

$$\theta_2 = 10.23^\circ, \quad d_2 = 1.999 \text{ \AA},$$

$$\theta_3 = 10.37^\circ, \quad d_3 = 1.972 \text{ \AA}.$$

The relative error of measurement of value of interplanar spacing here and further everywhere is not worse than 0.5%. The recorded result can be understood as the realization of two states: One state, determined by the angle $\theta_2 = 10.23^\circ$, is an equilibrium state and has the bcc structure with edge length $a = a_0 \sigma^{-1/3}$, i.e., an isotropically compressed cell of the initial structure. The other is a nonequilibrium state, determined by the two remaining peaks and is a distorted bcc structure with the angle between the edges different from 90° and cell volume equal to the volume of the initial cell reduced by a factor of σ . Such a deformation of a bcc structure can be obtained, if one of the body diagonals of the cubic cell is compressed by a factor of σ . Then the primitive rhombohedron, on which the bcc structure is based, and for which the angle $\alpha = 109.46^\circ$ between the edges, transforms into a rhombohedron with $\alpha = 110.16^\circ$, and the (110) peak of the initial structure splits into two peaks, one of which ($\theta_1 = 10.1^\circ$) is not shifted while the other is shifted into a position determined by the angle $\theta_2 = 10.37^\circ$.

FIG. 3. Diffraction photograph of the sample $\langle 110 \rangle$ (a), the sample $\langle 100 \rangle$ (b) and the sample $\langle 111 \rangle$ (c) of lithium fluoride structure under pressure $P = 6.6$ GPa, $\sigma = 1.08$ (a,b) and $P = 13.5$ GPa, $\sigma = 1.16$ (c); 1-striker Al, $L=10$ mm, $W=1.6$ km/sec); 2-sample ($L = 3$ mm); 3-layer of plexiglas (a,b) or Be (c), $L = 5$ mm. In Fig. 3b the reflection from the (110) planes of the rhombohedron with diffraction angle $\theta = 10.16^\circ$ is superposed on the reflection from the (200) plane of the fcc structure with $\theta = 10.44^\circ$ and is seen as a shoulder of a stronger reflection. These same reflections, formed by K_β Mo anode radiation, can be seen on the left; further to the left the (100) peak of the structure of the rhombohedron can be seen.

The characteristic feature of the diffraction photographs of lithium fluoride and aluminum which are presented in Fig. 3 and Fig. 4 is the splitting of the peak of the initial structure in the dynamical photograph into two peaks. The peak with the

larger diffraction angle corresponds to an equilibrium state of the compressed structure. The relation between the interplanar spacing d , determined by the peak of the equilibrium structure of the compressed material, and the interplanar spacing d_0 , determined by the peak of the initial structure, is given by $d = d_0 \sigma^{-1/3}$, i.e., the cell of the structure of the dynamically compressed material is equal to the isotropically compressed material with the cell volume of the initial structure decreased by a factor of σ . The peak with the smaller diffraction angle coincides with the peak recorded in the preliminary photograph. Additional researches [1] have demonstrated, that this peak is formed by structure of compressed substance.

FIG. 4. Diffraction photograph of the structure of aluminum under pressure $P=23$ GPa, $\sigma = 1.21$. The loading assembly is similar to that shown in Fig. 1. Peaks with $\theta \approx 9.31^\circ$ and 10.77° were determined as reflections from (111) and (200) planes of the fcc lattice of the structure of the compressed material; peaks with $\theta \approx 8.73^\circ$ and 10.09° , coinciding with the (111) and (200) peaks of the initial state of the structure of the sample, are determined as reflections from $(\bar{1}10)$ planes of two rhombohedral structures of the compressed material, which have rhombohedra with the same volumes but different angles α . Reflections formed by K_β Mo anode radiation can be seen on the left.

The interpretation of results of registration diffraction photographs lithium fluoride and aluminum is similar to interpretation of registration photographs of silicon iron; a mix of two structural states was realized. One of them, equilibrium, is identical to isotropically compressed initial structure of a sample, other, nonequilibrium, is characterized by preservation of interplanar spacing of system of crystallographic planes, parallel shock front.

The formation of a nonequilibrium structural component can be understood if, by analogy with the results obtained for the silicon iron structure, the dynamical deformation of the lithium fluoride and aluminum structures is interpreted

assuming compression along the (111) axis of the initial structure. In a deformation of this kind this structure loses some symmetry elements of the initial packing and turns into a trigonal crystalline system. The unit cell of the Bravais lattice of the trigonal system - a rhombohedron - is determined by two parameters: the edge length l and the angle α between the edges. By varying the angle α of the rhombohedron it is possible to describe a wide class of structural transformations accompanying deformation of the rhombohedron along its only axis $[111]$ without leaving the trigonal system. The following particular cases are obtained: 1) fcc structure, $\alpha = 60^\circ$; 2) simple cubic structure with $\alpha = 90^\circ$; 3) bcc structure with $\alpha = 109.46^\circ$.

A characteristic feature of the deformation of the rhombohedron along the $[111]$ axis is that the interplanar spacing $d_{(110)}$ (the Miller indices are written in a

coordinate system whose basis vectors are determined by the edges of the rhombohedron) are preserved. The reason is that the deformation direction lies in the $(\bar{1}10)$ plane. This assertion is equally applicable to all planes of the zone whose axis is the $[111]$ direction. The interplanar spacing $d_{(220)}$ does not change

under deformation along the $[111]$ axis - the results of the experiment (Fig. 3a) where a $\langle 110 \rangle$ lithium fluoride sample was investigated can be interpreted in this manner; the shock front in this sample was parallel to one of the $\{220\}$ planes according to the conditions of the measurements. Hence it follows that the deformation axis lies in the gas dynamic fracture plane.

For $\langle 100 \rangle$ and $\langle 111 \rangle$ samples the interpretation of the measurements is more complicated, since the compression of the initial structure along the $[111]$ direction does not preserve the interplanar spaces $d_{(200)}$ and $d_{(111)}$ - the body diagonals of the cubic cell do not lie in any plane of the form $\{200\}$ and $\{111\}$.

The indexing problem can be solved by taking into account the weak peak with the diffraction angle $\theta = 8.65^\circ$, recorded in Fig. 3b. If the peak with $\theta = 10.16^\circ$ is indexed as a reflection from the (110) planes and the peak with $\theta = 8.65^\circ$ is indexed as a reflection from the (100) planes of the rhombohedron, then the

rhombohedral parameters are $\alpha = 103.6^\circ$ and $l = 2.56 \text{ \AA}$ and the ratio of the volume of the rhombohedron of the initial structure to the volume of the rhombohedron found is equal to the bulk compression of the sample. The diffraction data for the $\langle 100 \rangle$ sample indicate that the initial structure is reoriented in the shock front to a position with respect to the plane of the front in which deformation conditions similar to those for the $\langle 110 \rangle$ sample are satisfied with the deformation remaining uniaxial. To underscore this fact, the microscopic process of deformation of a $\langle 100 \rangle$ sample can be conventionally divided into two stages: a) reorientation of the initial structure with preservation of cell volume and the condition $d_{(110)} = d_{(110)}$ with a possible change in the parameters of the

rhombohedral; b) compression of the reoriented cell in the $\langle 111 \rangle$ direction with a reduction of its volume by a factor of σ . The appearance of a (100) reflection in the photograph can be explained by the fact that a small part of the layer of material studied is realized in an orientation when the (100) plane is in a reflecting position. The condition that the deformation axis be parallel to the gasdynamic fracture plane is not preserved for the structure of this part of the layer of the material.

Variant of a microscopic picture of real process of reorganization of structure on shock front, taking into account above stated facts, is possible to present consisting from two stages: a) initial uniaxial compression of initial structure of a sample under action of one dimension flow of substance (the axis of the deformation coincides with a vector of a shock wave); b) the reorientation of structure of compressed substance as transition (catastrophe) from a nonequilibrium structural state in a direction to equilibrium, orientation and parameters of structure after such transition, when it appear outside shock front, is possible to determine on date of x-rays photographs.

In conclusion we note that the recorded diffraction patterns cannot be understood on the basis of existing ideas physical processes occurring with rapid deformations of a material. The concepts of dislocation kinetics[3,4], which are the basis for these ideas, do not incorporate the uniaxial structural compression process which changes the packing symmetry of the atoms. Apparently, the representations of restructuring of chemical bonds in the condensed state of the material under dynamical loads with high deformations rates $> 10^4 \text{ sec}^{-1}$ must be modified

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