STRENGTH
AND PLASTICITY

Influence of Pressure on the Processes of Formation and Evolution of the Nanostructure in Plastically Deformed Metals and Alloys

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Abstract—The role of volume changes on the structural rearrangements of dislocation pileups accompanied by the formation of the intercrystallite boundaries, pores, and microcracks upon the plastic deformation of materials is analyzed. The results of this analysis are applied to the study of the microscopic mechanisms of the effect of pressure on processes of the formation and fracture of the nanostructure of metals and alloys.

1. INTRODUCTION

A considerable amount of attention has recently been given to the nanostructured materials obtained on the basis of metals and alloys by severe plastic deformation under pressure [1]. As a result of such treatment, various structural-phase transformations occur in the materials, which lead to the formation of nanostructured states [1, 2] in which the materials are characterized by an unusually high strength and plasticity. However, despite the considerable interest, the mechanisms of the formation of the structural-phase state remain the subject of discussions [3–6].

According to a widespread view, such behavior is explained by the accumulation of defects in the process of severe plastic deformation and also by the fact that the external compressive pressure prevents the opening of microcracks [7]. However, this approach does not cover the entire set of processes that occur in the nanomaterials during plastic deformation. Upon the deformation of materials, there is always a competition between the processes of accumulation of the internal stresses, which determine the kinetics of fracture, and the processes of the relaxation of these stresses. If the rate of relaxation of the internal stresses in some points of the material turn out to be lower than the rate of the stress increase, the fracture of the material can occur at these points. Otherwise, the material can be deformed to any degree of deformation [8].

Thus, upon the study of the behavior of nanomaterials during plastic deformation, it should be taken into account that the imposition of pressure can affect not only the behavior of microcracks in the field of mechanical stresses, but also affect the microscopic mechanisms of relaxation of the internal stresses associated with the possible processes of structural-phase transformations.

The aim of our work consists in the studies of the microscopic mechanisms of the pressure influence on the processes of formation and fracture of nanostructures associated with the volume changes introduced by the structural-phase transformations occurring in the process of plastic deformation of the metals and alloys.

2. RESULTS AND DISCUSSION

2.1. Theoretical Models of the Formation of Structural Elements

Any transformation of the structure of a substance under the action of external stresses σ will be considered a relaxation process, which adapts the structure of the system to the conditions of deformation, thereby reducing the thermodynamic Gibbs potential G under these conditions. It is known from the thermodynamics that the influence of pressure (p) on any processes is closely associated with the volume changes δV in the course of these processes. An additional thermodynamic stimulus controlling the relaxation processes under pressure becomes the change in the Gibbs potential determined by the volume effect of the transformation: $\delta G = p\delta V$.

The nanostructures of materials represent a rather dense and branched system of interacting crystal-lattice defects. Consequently, the influence of pressure on the processes of the structural-phase transformations occurring in the nanostructures during plastic deformation should be seen in the changes in the vol-
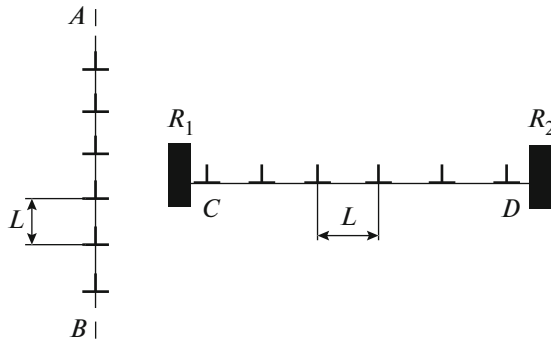
Fig. 1. Flat dislocation pileups [10].

Fig. 2. Dependence of the dilatation generated by the excess volume in the cores of separate dislocations on the distance L between the dislocations [10]. Curve *1* corresponds to the vertical dislocation wall of the *AB* type. Curves *2*, *3*, *4*, and *5* describe the dependence of the dilatation in the cores of the extreme dislocations in the horizontal pileups of the *CD* type with the numbers of dislocations n = 2, 5, 10, and 20, respectively. The dashed horizontal line specifies the amount of dilatation in the core of a single edge dislocation.

ume introduced by these defects, first of all, by dislocations, which are the main carriers of plastic deformation in crystals [1–3, 6–9].

It was shown in our previous article [10] that the edge dislocations can have an excess (free) volume and that the value of this volume depends on the defect structure of the whole material. In particular, for the flat dislocation pileups presented in Fig. 1, dependences were obtained for the dilatation ε_0, proportional to the excess volume δV per unit length of separate dislocations of the pileup, on the distance between the dislocations L (Fig. 2).

In the theory of the intercrystallite boundaries, walls of the AB (Fig. 1) type of a pileup are used to model the low-angle and special tilt boundaries (Fig. 3) [9, 11, 12]. The pileups of the CD type (Fig. 1) arise at the interfaces between the phases F_1 and F_2 with different lattice parameters in the boundary plane (Fig. 4) [12] and also at the intercrystallite boundaries of the common (random) type [8, 9, 11].

In these cases, the dislocations of the *CD* pileup are called misfit dislocations; and and the joints (that we labelled R_1 and R_2) of neighboring grains serve as obstacles (see Fig. 1). In the equilibrium intercrystallite and interphase boundaries, the long-range stresses of the *CD* pileup are compensated by the stresses caused by the coherent fragments of the boundary located between the dislocations; in the non-equilibrium boundaries, the stresses in the *CD* pileup are compensated only partially.

The calculation carried out using the model formulated in [10] shows that the excess volume of each of the dislocations of the AB type of pileup decreases with an increase in their linear density in the wall (curve *1* in Fig.2), which is due to the partial compensation of the elastic fields of the neighboring dislocations. The curves *2–5* in Fig. 2 show that the value of the excess volume of the extreme ("lateral") dislocations in the pileup *CD* increases not only with a decrease in the distance L, but also with an increase in the number of dislocations in the pileup at a fixed distance between them. The same conclusion refers to other dislocations of the pileup *CD*.
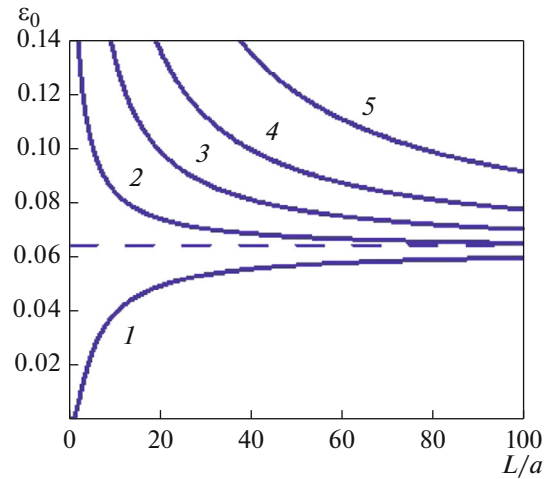
2.2. Mechanisms of the Pressure Influence on the Processes of Nanostructure Formation

It is known that the process of the formation of a nanostructure during plastic deformation of metals and alloys consists of several stages [1, 2]. The main stages are the processes of preliminary accumulation of dislocations, of polygonization, and of the formation of special intercrystallite boundaries and of com-
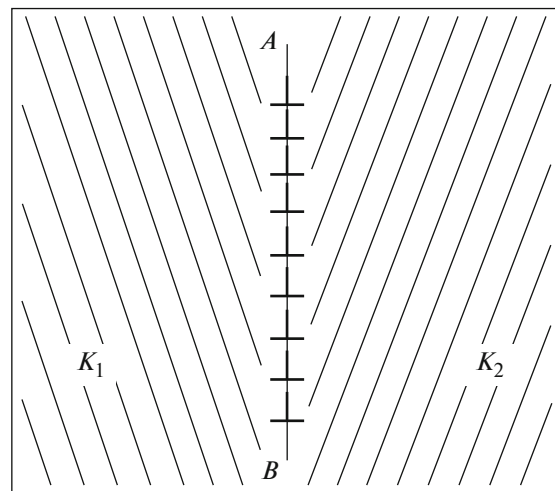

Fig. 3. Dislocation model of low-angle and special tilt boundaries between the crystallites K_1 and K_2. The inclined straight lines show the atomic rows in the corresponding crystallites.
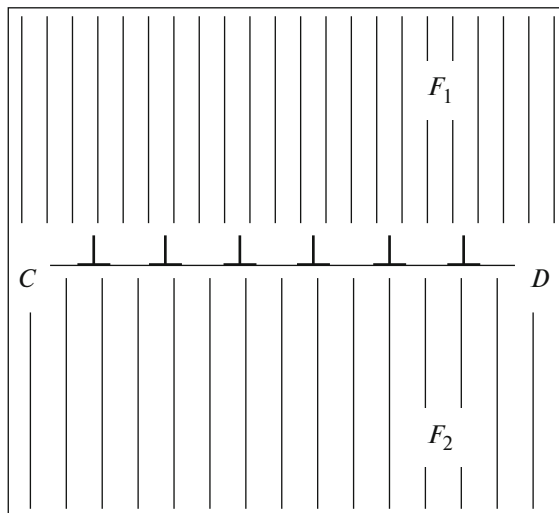
Fig. 4. Dislocation model of the misfit boundaries between the different phases F_1 and F_2. The straight vertical lines show the atomic rows in the corresponding phases.

mon-type high-angle boundaries. The latter arise upon the dynamic recrystallization, deformation twinning, and upon deformation-induced polymorphic transformations of martensitic type [2, 4]. It is clear that an external compressive pressure should promote the development of those processes that lead to a decrease in the total volume of the material.

It can be seen from Fig. 2 (curve *1*) that the processes of the formation of low-angle tilt boundaries from the disordered dislocation pileups should occur with a decrease in the total volume of the material. Usually, the low-angle boundaries form a substructure of the deformed grains [9, 11, 12]. It is also known that the special high-angle tilt boundaries can arise when the low-angle tilt boundaries trap lattice dislocations with a Burgers vector orthogonal to the plane of the boundary [9, 11]. This means that the application of an external compressive pressure upon plastic deformation of metals will lead to the development of a substructure in the bulk of grains by means of polygonization and to a further transformation of the substructure into the system of high-angle tilt boundaries of special type.

The special high-angle tilt boundaries can also arise in the processes of the deformation twinning [2, 4, 9, 11, 12]. If the formation of a twin is not accompanied by the absorption of defects or by a transformation of the initial defect structure, the total volume of the material will increase upon deformation by twinning. Consequently, the external pressure will prevent the formation of twins in the defect-free volumes of an ideal lattice.

At the same time, the boundaries of nanocrystallites can serve as catalysts for the nucleation of deformation twins. This can easily be shown. If a fragment of an appropriately oriented intercrystallite boundary

is replaced by a thin nucleus of a twin, then, for the mechanical equilibrium to be retained in the system, it is necessary that the total surface tension of the two twin boundaries be less than or equal to the surface tension of the remaining part of the initial intercrystallite boundary [13]. This condition is feasible if we consider that the surface energy of the intercrystallite boundaries of common type is usually several times greater than the energy of twin boundaries [9]. It is also known that the energy of the intercrystallite boundaries is proportional to their excess volume [14]. This means that no increase in the volume of the material due to the formation of a twin nuclei at the boundary will occur. Therefore, the pressure will not prevent the catalytic mechanism of deformation twinning at the boundaries of nanograins.

A similar effect will be exerted by the external pressure on the formation of the boundaries of a special and common types arising upon polymorphic transformations under the influence of shear deformation. Indeed, it is known from the theory of the first-order phase transformations that the increment of the Gibbs thermodynamic potential upon the formation of a nucleus of a new phase under isothermal-isobaric conditions is about one third of the surface energy of the nucleus [15]. Consequently, the boundaries of nanograins, which have a sufficiently large specific free energy and excess volume, will retain the role of catalysts of the processes of nucleation of new phases upon plastic deformation under pressure.

The external pressure can also exert an effect on the formation of high-angle boundaries of common type. As a rule, the common-type boundaries are not symmetrical. Their dislocation structure is presented schematically in Fig. 5. Along with the dislocation wall *AB*, which makes the main contribution to the determination of the angle of relative turn of the crystallites K_1 and K_2, the structure of the boundary includes the pileup *CD* consisting of grain-boundary edge dislocations, which compensate for the discrepancy between the interatomic distances of the neighboring crystallites in the boundary plane. Such a boundary can be obtained in three different ways: as a result of the processes of dynamic recrystallization; upon grain-boundary sliding; and via the absorption of lattice dislocations with a random direction of the Burgers vector by a special tilt boundary.

It can be seen from Fig. 2 that a high-angle tilt boundary of the common type always has a larger free volume than a special tilt boundary with the same angle of misorientation of crystallites. However, in the dislocation wall *AB* entering into the boundary, the ratio of the volume of each dislocation to the volume of a single dislocation outside the pileup is less than unity; and in the pileup *CD,* this parameter exceeds unity. Hence, the total volume effect upon the formation of a tilt boundary of common type from disordered dislocation pileups depends on the proportion

between the volume effect upon the formation of the dislocation wall *AB* and that upon the formation of the pileup *CD*. Therefore, the external compressive pressure can influence the processes of formation of the common-type tilt boundaries in different ways. It is obvious that it will activate any processes of deformation and recrystallization that lead to a decrease in the total volume of the material.

For example, the pressure can promote the development of processes of grain-boundary sliding, which transform the common-type tilt boundary into a special tilt boundary.

This process can be carried out by shear deformation of the crystallite K_2 (fixing the position of one of its boundaries, Fig. 5) until the restoration of the mirror symmetry in the arrangement of its atomic planes with respect to planes of the crystallite K_1. In this case, the pileup *CD* disappears and the density of dislocations in the wall *AB* will decrease slightly. As a result of this deformation, the total volume of the material may decrease. In this case, the pressure will promote the process of the transformation of the asymmetric tilt boundary into a special tilt boundary. On the contrary, the pressure should counteract to the processes of grain-boundary sliding, which transforms the special boundaries into asymmetric boundaries of common type, since the total volume of the nanomaterial will increase in this case.

On the other hand, under certain conditions of plastic deformation, a compressive pressure can promote the transformation of special tilt boundaries into the boundaries of common type. The structural transformations of this kind are associated with the absorption of lattice dislocations by the intercrystallite boundaries [9, 11, 12].

Indeed, when being absorbed by an intercrystallite tilt boundary, the lattice edge dislocation can dissociate into two grain-boundary dislocations, one of which, with the Burgers vector parallel to the boundary, will replenish the pileup *CD*, and the other, with the Burgers vector orthogonal to the boundary, will be embedded into the wall *AB*. The total volume of the material may decrease if the orthogonal component of the Burgers vector significantly exceeds its parallel component. In this case, the external pressure will promote transformation processes of the special boundaries into the common-type boundaries. Similarly, the pressure will activate the processes of dynamic recrystallization, which form common-type intercrystallite boundaries from the dislocation pileups; as a result, the total volume of the deformed nanomaterial will decrease.

Considering the above, it can be concluded that the mechanisms of pressure influence on the processes of the formation of the metal nanostructure at each stage of deformation are different.

At the initial stage of deformation, when the dislocation density in disordered dislocation pileups is
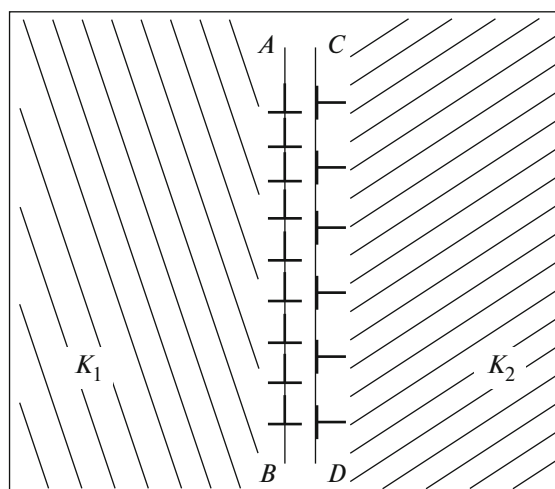

Fig. 5. Dislocation model of a high-angle non-symmetrical tilt boundary of common type between the crystallites K_1 and K_2. For the explanations, see the main text.

small and the temperature conditions are insufficient for the occurrence of the processes of dynamic recrystallization, it is the pressure that plays the decisive role in the development of the processes of polygonization and of the formation of the substructure in the bulk of crystallites of deformed polycrystals.

As the substructure is refined and the density of disordered lattice dislocations increases in it, processes of dynamic recrystallization will participate in the process of formation of the nanostructure. At this stage, the role of the pressure consists in the activation of processes of recrystallization and absorption of lattice dislocations by the intercrystallite boundaries, which lead to a decrease of the volume of the material.

At low temperatures of deformation, the nanostructure will be formed mainly under the influence of pressure; and at elevated temperatures, the properties of the nanostructure on the whole will be determined by the processes of dynamic recrystallization. However, at a sufficiently small average size of nanocrystallites ($20 \geq D \geq 3$ nm) their bulk regions become free from lattice dislocations [1]. As a result, the processes of the dynamic recrystallization at the dislocation pileups will be suppressed. In this case, the mechanisms of plastic deformation of the nanomaterial can occur only on the basis of grain-boundary sliding, deformation twinning, and polymorphic transformations of martensitic type [2, 4]. Therefore, the pressure again becomes the main regulator of the formation of the nanostructure at this stage.

Since the number of dislocations in nanocrystallites of small size becomes insignificant, the rate of absorption of lattice dislocations by the intercrystallite boundaries at this stage decreases and the intensity of the formation of new boundaries of common type will be weakened. Under these conditions, the structure of
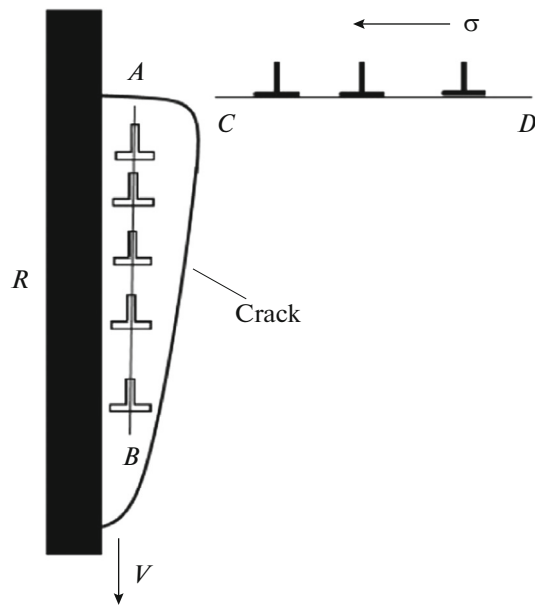
Fig. 6. Scheme of the microscopic mechanism of fracture of metals. For the explanations, see the main text.

the common-type boundaries under influence of pressure and of the processes of grain-boundary sliding will gradually approach the structure of special boundaries. The total density of high-angle boundaries upon the deformation can increase only due to the processes of twinning and polymorphic transformations [2, 4], but the rate of an increase in the free volume of the nanomaterial will gradually tends to zero.

When the nanomaterial structure approaches the X-ray-amorphous state ($D \leq 3$ nm), the density of the intercrystallite boundaries becomes so high that the occurrence of the processes of dynamic recrystallization at the dense pileups of intercrystallite boundaries becomes possible. As a result, separate inclusions of larger nanocrystallites (with $D > 3$ nm) will be formed in the X-ray-amorphous matrix [2]. Experimentally, this process was observed in the work [16] and was called the process of nanocrystallization. Since the total area of boundaries in this process decreases and the rate of growth of the excess volume of the nanomaterial becomes negative, the pressure will promote the emergence of the processes of deformation nanocrystallization of the X-ray-amorphous metals and of cycling transformations: "X-ray-amorphous state" $\Leftrightarrow$ "nanocrystallization" [2, 16].

2.3. Influence of Pressure on the Fracture Mechanisms of Nanomaterials

It is known that the maximum number density of dislocations at the maximum degree of plastic deformation $\varepsilon_{pl,0} \approx 0.2$–$0.3$ preceding the fracture of metals under normal conditions does not exceed $\rho_d \approx 10^{15}$–10^{16} m^{-2}

[17]. Using ideas on the dislocation structure of intercrystallite boundaries (Fig. 3–5) and assuming that the volume-average value of the crystallite misorientation angle in the nanostructure is close to $\theta \approx 30°$, it can easily be calculated that even at $D \approx 5$ nm the metal nanostructure is equivalent in the conditional number of dislocations per unit volume to an ordered dislocation pileup with a density $\rho_n \approx 10^{17}$–10^{18} m^{-2}. This means that, to create such a nanostructure, the degree of plastic deformation ε_n must be much higher than $\varepsilon_{pl,0}$. It is important that no fracture of the nanomaterials occurs in this case. In this case, it becomes relevant to study the mechanisms of pressure influence on the processes of the fracture of nanomaterials.

The scheme that explains the possible microscopic mechanisms of fracture upon deformation of metals is presented in Fig. 6 [18]. Under the action of a shear stress σ, the pileup CD is pressed to some obstacle R. At a sufficiently large number of dislocations in the pileup, the stresses near the obstacle can exceed the critical force necessary for the breaking of atomic bonds and will lead to the formation of a narrow cleavage crack expanding along the obstacle with a velocity V.

Usually, the description of the deformation fields created by such a crack is based on the model in which the cracks are replaced by a corresponding vertical dislocation pileup of the AB type. These "imaginary" dislocations (in Fig. 6 they are represented by hollow symbols) were called vacuum dislocations. It is assumed that they have the same deformation fields as the real dislocations [18].

Based on this model and graphs shown in Fig. 2, we can give a rather complete description of the influence of the external pressure on the micromechanisms of the fracture of nanomaterials.

An analysis of the curves *2–5* in Fig. 2 shows that the external pressure will prevent the formation of pileups of the CD type retarded at obstacles. There are several mechanisms of such retardation. First, the pressure can suppress the operation of the Franck–Read dislocation sources that increase the number of dislocations in the pileups CD [7, 9]. Second, the pressure can promote the withdrawal of dislocations from the CD pileups and their arrangement into real dislocation walls of the AB type. Third, the pressure can increase the absorption of dislocations from the pileup by the boundaries of obstacles. As a result, the concentration of stresses near the obstacles will be reduced and will be insufficient to break the atomic bonds.

From curve *1* in Fig. 2, it follows that a compressive pressure should prevent the increase in the distance between the dislocations in the pileup AB and the growth of the crack length. This means that a compressive pressure will suppress the spread of microcracks.

At the same time, it can be seen from Fig. 2 that any transformation of the CD-type pileup into an AB-type

pileup leads to a decrease in the excess volume of a nanomaterial. Consequently, the compressive pressure will always promote the transformation of the *CD* pileup into an *AB* pileup. But since the increase in the length of a pileup of vacuum dislocations is also not advantageous, the pressure will maximally reduce the length of the *AB* pileup, rounding the crack profile and transforming it to a pore (Fig. 7).

Thus, an external pressure exerts a complex effect on the processes of fracture. On the one hand, it can accelerate rather than block the processes of nucleation of the microcracks. But, at the same time, it always suppresses the growth and spread of cleavage microcracks, promoting the development of processes of pore formation. The conditions advantageous for the pore formation are observed upon the deformation of metals with a sufficiently small grain size by grain-boundary sliding [19].

Upon deformation under pressure at high temperatures, these pores can be healed via diffusion. However, at reduced temperatures, when the diffusion processes are inhibited, a residual porosity can be retained in the materials. The development of porosity in grain junctions was observed experimentally in many alloys with ultrafine grains deformed under conditions of superplasticity [19].

In addition to the above about the role of a compressive pressure upon deformation of metals, it should be noted that the negative values of pressure realized upon tension of materials are expected to inevitably promote an increase in the density of dislocations in the pileups of the *CD* type, as well as the decrease in the density of dislocations in the *AB*-type pileups (Fig. 6). This explains why the tensile mechanical stresses upon deformation can accelerate the processes of nucleation and propagation of microcracks observed experimentally [7, 18].

2.4. Evaluation of the Magnitude of the Excess Volume

The adequacy of the dislocation models used above to describe the atomic structure of the intercrystallite and interphase boundaries can be verified by the comparison of the results of simulation with the known experimental data. It is sufficient to evaluate with their help the amount of the excess volume δV_n in nanocrystalline metals and alloys obtained by severe plastic deformation under pressure.

The above estimations have shown that, within the framework of the ideas on the dislocation structure of all the crystal-lattice boundaries, the nanostructure of materials with an average size of nanocrystallites $D \approx 5$ nm and the average angle $\theta \approx 30°$, of misorientation at the tilt boundaries is equivalent to an ordered pileup of dislocations with a density $\rho_n \approx 10^{17}–10^{18}$ m^{-2}.
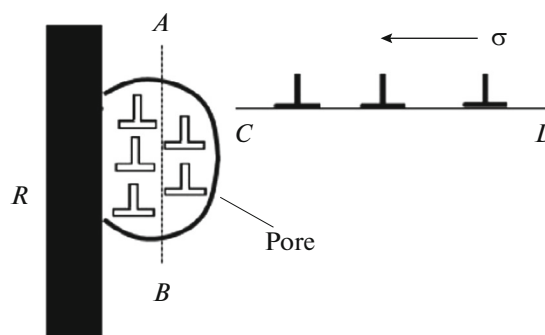

Fig. 7. Scheme of the microscopic mechanism of the formation of pores. For the explanations, see the main text.

It is also known that the nanostructures of plastically deformed metals contain the mainly-high-angle boundaries of common type [1, 20]. Since, the high-angle boundaries can be constructed from the pileups of the *AB* type with a reduced excess volume of dislocations and from the pileups of the *CD* type with an increased excess volume of dislocations, it can be assumed that the average value of the excess volume per unit length of each of the dislocations simulating the nanostructure is only insignificantly different from the excess volume of separate dislocations in the disordered pileups. Hence, we believe that the total excess volume of a nanomaterial should be proportional to the number density of conditional (virtual) dislocations imitating the nanostructure and to the excess volume of a single dislocation (Fig. 2). For the density $\rho_n \approx 10^{17}–10^{18}$ m^{-2}, we obtain $\delta V_n \approx 1\%$. Despite some rough assumptions about the structure of the grain boundaries used in the evaluations, the obtained value is in good agreement with the data presented in the works [14, 20−22]. This means that the dislocation models of grain boundaries in combination with the nonlinear theory of irreversible deformations [10, 22] used in this work can serve as a useful tool in the study of the volume effects arising upon plastic deformation of nanostructured metals under pressure.

3. CONCLUSIONS

(1) The influence of an external pressure on the processes of the formation and fracture of a nanostructure in plastically deformed metals and alloys is due to the presence of an excess volume in the defects of the crystalline structure.

(2) At the initial stages of the formation of the nanostructure of metals and at low temperatures of deformation, the external pressure is the main factor determining the rate of processes of the formation of the substructure and its transformation into a system of high-angle boundaries of the common type.

(3) At the elevated temperatures of deformation, the pressure promotes the development of dynamic

recrystallization processes, which form the main elements of the nanostructure of a material.

(4) At sufficiently small sizes of the nanocrystallites, the pressure is an important factor in the evolution of the nanostructure of metals and alloys. Its influence on the deformation processes can lead to the transformation of the structure of the high-angle boundaries of common type into a structure of special grain boundaries.

(5) The compressive pressure should promote the development of dynamic recrystallization processes upon plastic deformation of metals and alloys in the X-ray-amorphous structural state, since these processes are always directed toward reducing the total volume of the material.

(6) Upon the deformation of preliminarily annealed metals, the compressive pressure prevents the formation of large dislocation pileups that can promote the nucleation and growth of microcracks.

(7) The compressive pressure may accelerate the processes of nucleation of microcracks. However, it efficiently suppresses the processes of propagation and growth of the cleavage microcracks present in the metal and, under certain conditions, can promote the formation of pores at the grain junctions in fine-grained and nanostructured materials.

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Translated by G. Salnikov