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Mechanisms of Structure Formation under Severe Plastic Deformation: A Review

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Modern materials often have multicomponent alloys, whose properties are determined by their phase and structural composition formed as a result of previous thermomechanical processing. Therefore, the problem of managing the structural state occupies an important place in the overall strategy of developing new materials. Particular attention is drawn to phase transformations in ultrafine systems, in particular, in alloys subjected to severe plastic deformation. In this case, the sample size becomes an important parameter determining the physical properties of the substance and, in particular, its structural and phase compositions. Despite the considerable interest of researchers, until recently, it was not possible to achieve significant theoretical results in this area. Therefore, the development of modern models for describing structural transformations in heterogeneous systems and their application to ultrafine-grained materials seem promising and relevant.

Key words: severe plastic deformation, nanostructure, fragmentation mechanism, ultrafine-grained structure.

Сучасні матеріали часто являють собою багатокомпонентні стопи, властивості яких визначаються їхньою фазовою і структурною будовами, сформованими в результаті попереднього термомеханічного оброблення. Тому проблема управління структурним станом посідає важливе місце в загальній стратегії розроблення нових матеріалів. Особливу увагу привертають фазові перетворення в ультрадисперсних системах, зокрема, у стопах, що зазнали інтенсивної пластичної деформації. У цьому випадку розмір зразка стає важливим параметром, що визначає фізичні властивості

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речовини і, зокрема, її структурно-фазову будову. Незважаючи на значний інтерес дослідників, до останнього часу не вдавалося домогтися значущих теоретичних результатів у цій галузі. Тому розвиток сучасних моделей для опису структурних перетворень у неоднорідних системах і застосування їх до ультрадрібнозернистих матеріалів видається перспективним і актуальним.

Ключові слова: інтенсивна пластична деформація, наноструктура, механізм фрагментації, ультрадрібнозерниста структура.

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1. INTRODUCTION

The development of methods of severe plastic deformation (SPD) in recent years has expanded the process of evolution of metal systems up to the formation of nanostructured state. Many review papers [1–3], experimental [4–15] and theoretical papers [16–18] have appeared in this direction, including those using computer modelling, the authors of which, systematizing the results obtained, complementing and enriching each other, create a general picture of severe deformation of solids. Nevertheless, it should be recognized that today there is still no general physical model of matter evolution under plastic deformation.

Under the evolutionary model of a deformable solid, it is necessary to understand a set of laws and a description of the mechanisms that occur during plastic deformation. Such a model should make it possible to understand how a substance changes its structure under the influence of external mechanical influences at all stages of deformation and at all scale levels as well as how it degrades subsequently. However, in addition, such a model can become the basis for solving specific technological problems, in particular, the formation of nanoscale structures.

The evolutionary path of metal systems under deformation conditions largely depends on the nature of the material, the degree of interatomic bonding, elastic modules, the energy of packaging defects, the degree of alloying, the presence of second phases, the degree of metastability of the initial phases, temperature, the deformation degree and the stress state scheme.

At the same time, methods for obtaining materials with ultrafine-grained (UFG) and especially with nanocrystalline (NC) structure both in the past and now are characterized by high labour intensity and low productivity. Therefore, for their improvement, it is important to understand the process of formation of ultrafine grains during plastic deformation. Currently, there are many scientific ideas and corresponding models [19–21] devoted to this issue. In them, the formation of ultrafine grains is described at micro- or macrolevel. In the first case, grinding occurs due to the passage of the so-called ‘low tempera-

ture' dynamic recrystallization and/or phase transformations. In the second case, the grinding does not depend on the crystal structure of the workpiece but is the result of continuous shifts in the workpiece toward tangential stresses determined by the simple shear scheme. Note that there is no faultless evidence of the implementation of processes corresponding to the noted representations in these works and other works developing these representations.

In this paper, based on the analysis of literature data and own results, it is proposed to present, indicating the main points, the structure evolution of metallic materials under severe plastic deformation.

2. STRUCTURE FRAGMENTATION DURING DEFORMATION

The active development of methods of severe plastic deformation in recent years has been dictated primarily by the need to obtain a nanostructured state due to fragmentation of the initial grains. Most works in this direction discuss the fragmentation phenomenon as a process of discretionary boundary formation because of the dislocation interaction [22–24]. The fragmentation phenomenon is characterized for the first time as the dominant mode of defective structure evolution at the stage of advanced plastic deformation, leading to the organization of discretionary boundaries. To date, several models have been put forward to determine possible ways of fragment formation.

The fragmentation process begins with strain $\varepsilon \approx 0.2\text{--}0.3$. Further, as the strain increases, the transverse dimensions of the strips and cells in them decrease with respect to the direction of extraction. The longitudinal dimensions of the strip increase but not adequately to the elongation of the sample due to their splitting into parts due to local curvatures. Such curvatures are characteristic for many lamellar-type formations because of the influence of transverse disturbances on them [25]. The uneven distribution of strain over the cross-section of the material already at an early stage of deformation leads to the formation of various modifications of strip structures: deformation, transition and microstrips, differing in transverse dimensions. The largest transverse dimensions are deformation strips filled with several rows of enlarged cells; the smallest dimensions are microstrip in which there is one row of cells of minimal size. At the same time, the transverse dimensions of the microstrip coincide with the transverse dimensions of the cells contained in them; as a result, the longitudinal boundaries of the cells that coincide with the boundaries of the strips acquire, in contrast to the transverse boundaries of the cells, increased angular misorientation.

In Ref. [26], a slightly different view of the development of fragmentation is proposed, which is based on the regularities of the formation of band structures. The mechanisms of shear band formation,

the orientation of the boundaries of which differs from the orientations of the planes of light sliding, have not yet been fully elucidated. The fact of alternately changing the sign of the angle of disorientation of the crystal planes of neighbouring bands has not received a scientific explanation. The authors of [26] proposed a model for the formation of a nanostructure during deformation by equal-channel angular pressing (Fig. 1). However, the question of the mechanism of the reorientation band formation was not discussed in this work.

An interesting interpretation of the shear bands is given in [27], where, based on the consideration of the joint action of two sliding systems, the shear band begins at the intersection of the two sliding systems, *i.e.*, a stress mesoconcentrator arises, called ‘christon’, the carrier of lattice displacement quanta during shear deformation. Such a concentrator is able to polarize actively the medium at the mesoscale, creating an excessive dislocation charge of the opposite sign in the vicinity of the initial concentrator forming a dipole pair with the initial charge.

The conclusions made in [28] are largely consistent with the ideas about the formation of boundaries with discrete misorientation of mating regions in terms of the formation of dipole dislocation charges. When the dislocation dipole has a configuration of two flat dislocation clusters of different signs, the displacement field around such a charge represents a cylindrical bend (similar to the curvature of a tile).

Consequently, it can be imagined that during deformation, a periodic elastic displacement field arises which, under certain conditions, can

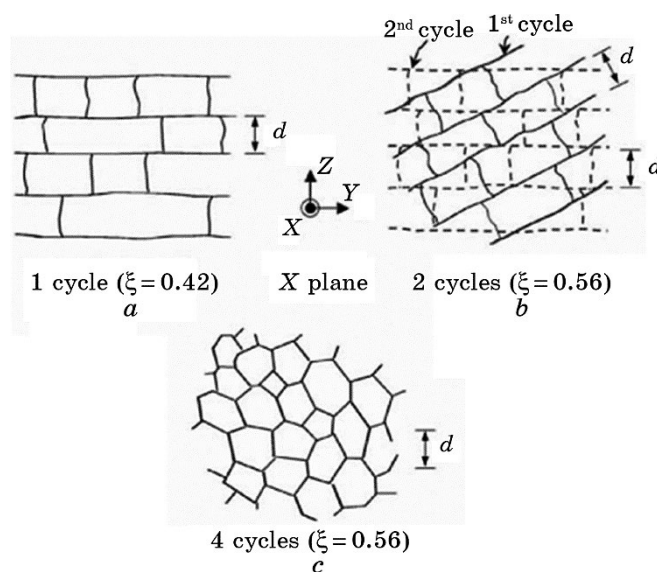


Fig. 1. Microstructural model showing grain refinement after: 1 (a), 2 (b) and 4 pressing cycles (c) [26].

relax into shear bands with alternating changes in orientation and non-crystallographic direction of the boundaries.

When the sample enters the matrix, it appears in the field of increasing gradient stresses. However, in addition, additional stresses arise in the material associated with the need to reorient the lattice to a favourable location of the planes of light sliding relative to the direction of external influence. This process causes the formation of dipole dislocation charges and the subsequent wave field of atom displacement, forming the bending of crystal planes.

In the new methods of severe plastic deformation, the stress state scheme assumes a change in the direction of external action and, in particular, the introduction of shear components. Changing the orientation of the planes of maximum shear stresses requires a new reorientation of the planes of light sliding in the material, thereby contributing to the grinding of grains.

The nature of fragmentation consists of the deformation grinding of the structure, which consists of splitting the initial grains into smaller disoriented areas separated by small- and medium-angle boundaries. At the same time, the misorientation angles θ increase with the growth of strain according to the linear law. In the works of V. V. Rybin and his followers determined that at temperatures below the recrystallization temperature, fragmentation patterns are universal and persist under any deformation methods for structural metals of any chemical composition, crystal structure and initial structure [29]. During fragmentation, the dislocation structure evolves during plastic deformation: increasing the density of uniformly distributed dislocations to 10^{12} m^{-2} ; the redistribution of dislocations (tangles, plexuses) and their annihilation and the density increases to 10^{14} m^{-2} ; the formation of a poorly oriented cellular structure separated by imperfect dislocation boundaries; the emergence of collective forms of movement of strong dislocations, the appearance of plastic reversals of one part of the crystal relative to the other.

In a deformed metal, with an increase in the degree of deformation, the density of defect increases. When the possibility of uncorrelated movement of individual dislocations is exhausted, the collective movement of interacting dislocations occurs due to the occurrence of rotational plasticity modes. At the first stages of fragmentation, fragments are formed in the structure, disoriented at small angles, with a developed substructure inside, as the deformation continues, the disorientation angles increase. The final stage of fragmentation is the formation of isotropic fragments, disoriented by large-angle boundaries and practically free from dislocations.

In essence, fragmentation is the result of plastic accommodation, the heterogeneity of structural defects evolves into a fragmented substructure consisting of dislocation subboundaries of deformation origin and

an internal space containing or not containing dislocations. To date, fragmentation processes have been well studied in austenitic steels, titanium, aluminium, and ferritic steels during cold deformation.

The article [30] is devoted to the theoretical and experimental study of grinding for various SPD processes (equal-channel angular pressing, high-pressure torsion, orthogonal cutting). The experimental results show that in the processes studied, there are some limits to grain grinding. An energy approach was used for theoretical research. It is assumed that the applied mechanical work is spent on increasing the energy of the defective microstructure (dislocations, vacancies; when constructing the model, this contribution is neglected), a change in the surface energy of the grains and an increase in temperature. It is assumed that grain sizes change because of two competing processes: grinding due to deformation (possible at room temperature) and growth due to thermally activated diffusion, a simple evolutionary relation is proposed to describe the change in grain size (an ordinary differential equation). When determining the temperature change, both adiabatic conditions and the possibility of heat energy runoff due to heat transfer to the environment are considered. The results of calculations of the change in the average grain size for aluminium, copper and nickel samples subjected to the specified SPD processes are presented.

Particular attention when considering grain-grinding processes is paid to the consideration of disclination and their interaction with dislocation ensembles [29]. The research results indicate the emergence of 'new' structures in the depths of the 'old' and the gradual absorption of the latter by the former. In particular, the grinding of the grain structure begins with the formation of dislocation and disclination walls, which cause the turns of parts of the crystallites relative to each other; the ongoing plastic deformation leads to an increase in the disorientation of crystallites, the formation of subgrains and new grains smaller than the original sizes. On the basis of the obtained data on the evolving microstructure, numerous so-called 'physically oriented' models are based, a significant part of which operates with continuous variables to describe the interacting dislocation and disclination substructures changing during deformation [31–34] of the process is the presence of a certain effect of metal hardening under the combined influence of plastic deformation and temperature. The changes in the structure of steel caused by riveting are usually stable and persist after double phase recrystallization $\alpha \rightarrow \gamma \rightarrow \alpha$, therefore, preliminary plastic deformation can play a significant role in the formation of the austenite structure during subsequent heat treatment. The acquisition of properties is influenced by the hereditary transmission of structural defects: the density of imperfections, fragmentation of the structure, as well as the grinding of austenitic grains.

The nature of the physical processes occurring at the high-pressure

torsion (HPT) in AA7075 alloy varies depending on the deformation mode. The predominant mechanism of relaxation of internal stresses in various true strains $e \leq 6.4$ is fragmentation, while the structure is crushed to nanoscale. The average grain size at $e = 6.4$ is 55 nm. At $e \geq 6.9$, another channel of elastic energy dissipation begins to operate in the alloy—low-temperature dynamic recrystallization. Unlike static recrystallization, in dynamic recrystallization, the nucleation and growth of new grains occurs during deformation and not after it as part of a separate heat treatment. Reducing the grain size increases the risk of grain boundary sliding at elevated temperature and reduces the mobility of dislocations in the material. The new grains are less deformed, which reduces the hardness of the material. Dynamic recrystallization allows obtaining new grain sizes and orientations, which can prevent the spread of cracks. Instead of deformation causing the destruction of the material, the deformation can initiate the growth of a new grain by absorbing atoms from neighbouring pre-existing grains. In this case, less defective grains with clear boundaries appear in a mixed structure consisting mainly of fragmented grains [35].

As described before, applying high levels of equivalent plastic strains at low temperatures via various SPD routes can drastically increase the density of statically stored and geometrically necessary dislocations. At first, during primary SPD passes caused by low strain levels, the structure of these dislocations is in the form of a tangle. However, by continuously repeating the SPD process and increasing the strain level, dislocations can interact and rearrange to form cellular structures in ultrafine or nanoscale ranges. In the following, more generations of dislocations *via* further straining can increase the density of dislocations through the cellular walls. This can increase the misorientation angle between the cells by gliding dislocations from the cells inside toward the relating walls and accordingly shifts these cellular structures from low-angle grain boundaries (LABs) toward more high-angle grain boundaries (HABs) [36]. In this case, the structural restorations for the severely plastic-deformed structures before and after post-annealing treatment are schematically demonstrated in Fig. 2, *a* and *b*, respectively.

3. GRAIN STRUCTURE REFINEMENT MODELS

In the article [37], it is assumed that the process of grain refinement in a polycrystal can be conditionally divided into three stages. At the first stage, the boundaries of the disoriented regions of the crystal lattice or blocks are formed. At the second stage, these boundaries turn into small-angle subboundaries of fragments. At the third stage, subboundaries develop into large-angle boundaries. With further deformation of the formed grains, the process is repeated until the for-

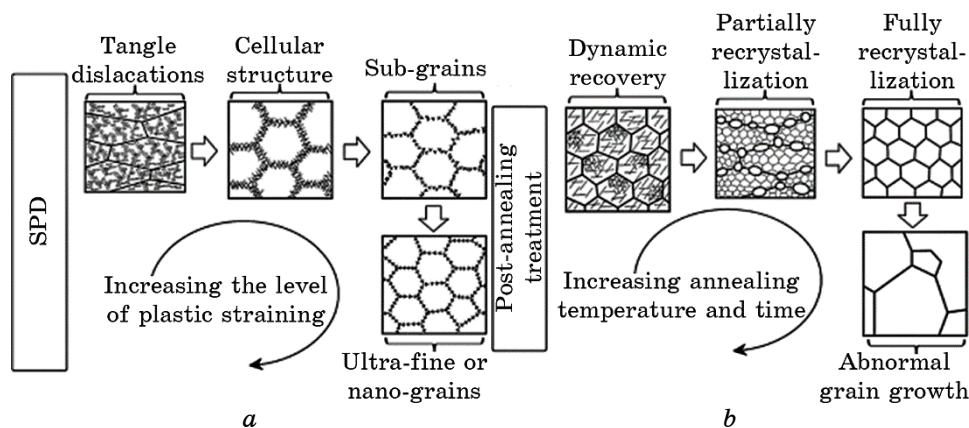


Fig. 2. Schematic plots illustrating the mechanisms for grain structural modification during: SPD treatment (a), post-annealing modification (b) [36].

mation of a nanostructured state with equiaxed grains. To describe the first two stages of grain fragmentation, the authors suggest the use of dislocation and declination models. Regarding the third stage, the development of large-angle boundaries, the authors propose the following analogy for consideration. The large-angle grain boundaries consist of regions with a structure characteristic of the crystal lattice and regions with a disordered structure characteristic of a supercooled liquid. The origin and development of large-angle grain boundaries in SPD is considered as a structural transformation, in which the volume fraction of the structure characteristic of a supercooled liquid becomes significant at the macrolevel. The distribution of atoms in the inter-grain space is described by two order parameters. In the local approximation of nonequilibrium thermodynamics, the evolution equations for the order parameters are obtained. Solutions describing the stratification of a homogeneous state with the formation of a spatially inhomogeneous distribution of order parameters at the grain boundary characteristic of fragmentation are considered.

The description of the above mechanisms, it seems, must necessarily be included in multilevel models for studying the deformation of polycrystalline metals and alloys. Of course, depending on the experimental data for a particular material and the SPD process, the set of grinding mechanisms considered can be supplemented.

The article [38] presents the results of a study of the SPD process of samples from materials with a lattice of b.c.c. and h.c.p. by equal channel angular pressing (ECAP) method and describes the optimal processing schemes for obtaining a fine-grained structure. The relationship between the change in the deformation trajectory and the grain-grinding process is investigated. It is concluded that it is neces-

sary to analyse the deformation at different levels to determine the most effective loading scheme.

The paper [39] analyses the results of a series of experiments to study the efficiency of the process of obtaining an ultrafine-grained structure in steel wire by the method of deformation nanostructuring according to the ‘drawing–torsion’ scheme. Because of metallographic analysis of the microstructure of the wire after the application of this method, a change in the main structural components (crushing, *i.e.*, grinding due to brittle destruction of cementite plates, reduction of the interplate distance in perlite) indicates the development of grinding processes of structural components of steel with an increase in the degree of deformation by torsion and the formation of an ultrafine-grained structure.

The articles [40–43] systematize experimental data on the properties of ultrafine-grained and nanostructured materials and discuss the current state of the problem of the theoretical description of the processes of obtaining a fine-grained structure. The authors emphasize that a comprehensive theory of this process has yet to be developed, since various modifications of traditional SPD methods can lead to effects that cannot be predicted within the framework of classical theories of plasticity.

The results of an experimental study and determination of the optimal (from the point of view of grain grinding) mode of ECAP of annealed copper samples are presented in [44]. It is based on the requirement to preserve in each passage the equality of the angle between the shear plane and the grain elongation plane to the angle between adjacent sliding planes of the h.c.p. lattice {111}. The angle of the channel break is set to 120°. The angles of rotation of the workpiece between 8 consecutive approaches are determined. As a result, a polycrystal with an average grain size of 55.5 nm, a high tensile strength and a large elongation to fracture was obtained.

A significant part of researchers considers the accumulation of excess internal energy in the material due to the generation of crystal lattice defects as the main reason for the change in the grain structure. In the mechanics of a deformable solid, accumulated inelastic deformations are usually used to assess the level of ‘defect’, the measure of which is the integral over the process time of the intensity of the plastic (viscoplastic) component of the velocity strain tensor. It should be noted that this measure does not have a clear physical meaning (expressed through the characteristics of the main mechanisms of inelastic deformation at the meso- and microlevels) [94]; the ‘transparent’ mechanical (geometric) meaning of this measure is only for the simplest loading of a macroform by uniaxial tension—compression, in this case it is equal to the longitudinal logarithmic deformation.

In addition, it is known that the behaviour of mono- and polycrystal-

line metals and alloys, the evolution of their defective structure at both macro- and meso- and microlevels is significantly influenced by the type of stress-strain state and the complexity of the loading process.

The internal energy accumulated during inelastic deformation relaxes because of activation of physical mechanisms of various structural-scale levels, the intensity of which depends on the ranges of parameter values describing the thermomechanical effects acting on the material under study. The relaxation of internal energy is accompanied, as a rule, by a significant restructuring of the micro- and mesostructure (including grain and dislocation structures). At relatively low deformation temperatures, the restructuring of the structure is carried out mainly due to mechanical influences. Under these conditions, intensive multiplication of dislocations, their interaction with each other, and self-organization lead to the formation of cellular, block-cellular, fragmented structures, reversals of fragments and subgrains relative to each other [45, 46] and eventually to a crushed 'new' grain structure.

From a geometric (kinematic) point of view, the change in the grain structure is associated with the reversals of individual parts of the grains relative to each other; when a certain value (usually 15°) is reached, the disorientation of the grain subdomains relative to the orientation of the 'mother' phase and neighbouring parts, these subdomains are considered separate grains. Models, in which along with translational degrees of freedom for material particles, rotational modes of motion are also introduced, appeared in continuum mechanics (primarily for elastic materials) in the early twentieth century [47].

Two different scenarios of the evolution of the grain structure in the SPD processes, which can be superimposed on the mechanism of crystallite refinement, are considered from the energy standpoint in [48, 49]. A common feature of the considered scenarios is their cyclicity. According to the first of them, the process of crushing (primary fragmentation) is replaced by primary recrystallization followed by secondary fragmentation, after which comes the stage of dynamic recrystallization. In the second scenario, after the primary fragmentation stage, alternating processes of amorphization and nanocrystallization follow. The authors note that the 'trajectory' of structural rearrangements is determined by factors such as temperature, the size of the dislocation Peierls barrier and their ability to diffuse rearrangements, and the energy difference of the crystalline and amorphous states. The evolution of the defect structure (point defects, dislocations), their interaction, restructuring, and the formation of new boundaries are considered as the driving force of the processes of crystallite change.

The transition to fragmentation and amorphization (and for some materials and/or deformation modes—to a solid-state phase transition) occurs after the possibility of relaxation of the supplied mechani-

cal energy is exhausted due to dislocation mechanisms and point defect flows. Grain sizes do not explicitly appear in the proposed model; another characteristic is introduced to describe the grinding process: the density of the boundaries.

To describe these mechanisms within the framework of phenomenological theories, models developed in terms of mechanical variables (stresses, inelastic deformations, strain gradients) are used, sometimes with the introduction of internal variables. With an increase in the processing temperature, the mechanisms caused by diffusion (atoms, vacancies, etc.) are activated, due to which return and recrystallization are realized, also leading to a change in the subgrain and grain structure.

A brief overview of the results of experimental studies and the main physical mechanisms of grinding is given in [50], which mainly considers studies of SPD at low temperatures, obviously lower temperatures, at which the recrystallization process becomes significant and solid-state phase transitions can be realized. Our article is devoted to the review of existing continuum models focused mainly on the description of changes in the grain structure under these conditions [51].

At the same time, the mentioned transformations can occur in metals and alloys in the SPD processes even at temperatures significantly different from the characteristic ranges of their realization under the action of only thermal factors. Due to the complexity of separating the various mechanisms, changes in the grain structure of the work, in which the latter are the result of the implementation of several mechanisms, will also be reflected in the review.

It is proposed to use a qualitative model based on the application of the first principle of thermodynamics (the law of conservation of energy) and the Landau theory of phase transitions to describe the SPD processes. In accordance with this, the terms responsible for the energy of various types of defects are introduced into the expression of internal energy along with elastic energy dissipated on plastic deformations and thermal (coming from outside the system) energies. At the same time, the main type of defects is considered high-angle grain boundaries, the formation of which determines the grinding of grains; the density of boundaries is taken as a quantitative measure of this type of defects.

The article [52] presents the viewpoint of Yu. M. Weinblat on the processes occurring in the alloys of the Al–Mg system. The formation of deformation textures in aluminium alloys is described as follows. At both high and low temperatures, the elementary mechanism of deformation of aluminium and its alloys is a shift along the plane $\{111\}$ in the direction $\langle 110 \rangle$. Sliding begins in the most favourably oriented grains, and then, as the stress increases, it spreads to all other grains. Shear deformation is accompanied by a regular rotation of the crystal lattice relative to external forces. In addition, each grain is affected by neighbouring grains, forcing it to change its shape in accordance with

the deformation scheme of the entire product or part of it. Because of coordinated turns with a degree of deformation of 30–50%, the grains acquire final orientations that do not change or change slightly with further deformation. The nature of the final orientations depends on the deformation scheme. Due to the symmetry of the deformation process, grains can take with equal probability one of several orientations symmetrical with respect to the main directions of deformation. The texture is usually described using these preferential orientations, otherwise called texture components.

Further in the article [53], mainly devoted to the review of the results of experimental studies of the mechanical behaviour of single-phase polycrystals at creep at elevated (in the vicinity of 0.5 homologous) temperatures, the issues of the formation and evolution of the subgrain structure are also considered. The subgrains begin to form at a transitional stage, and at the stage of steady creep, they form a stable and homogeneous structure. It is pointed out that the determining role for the formation of the subgrain structure of reproduction and self-organization of dislocations forming flat clusters and walls, which are further transformed into the boundaries of the subgrains, the latter have high mobility, which determines the growth processes of the characteristic sizes of the subgrains.

The microstructure of the deformed semi-finished product is formed by means of two processes: the gradual transformation of the original microstructure and the creation of new microstructure elements of deformation origin. The first process consists of changing the shape of the grains in accordance with the deformation scheme in a given cross-section zone and the second consists of the formation of new grain boundaries. There is a definition of grain as an area surrounded by a high-angle boundary (*i.e.*, a boundary with a misorientation angle of more than 10–15°) and containing no such boundaries within itself. In cast metal, dendrites meet this definition. The proof that new boundaries arise during deformation is the experiments on pressing and rolling single crystals, because of which they turned into polycrystals. The degree of hereditary influence of the original structure is the weaker, the more the process of formation of new boundaries is developed. New boundaries are formed in cases, where it is difficult to transform the initial shape of the grains into the final one and when there is a distinct texture in the workpiece that is very different from the texture of semi-finished products. The intensive formation of new boundaries is observed, for example, during the deformation of cast grains having a complex shape and with longitudinal sedimentation of the pressed workpiece. In the latter case, both factors are at work, since the texture of the pressing is very different from the texture of the precipitation.

If the grain elongation directions coincide during the first and second deformation (double pressing, rolling without edging), then, new

boundaries almost do not arise and new grain sizes are easy to calculate, knowing the old dimensions, scheme and degree of deformation. The formation of new boundaries is also unlikely when the grains in the workpiece are equiaxed, and there is no distinct texture [54].

4. MECHANISMS OF GRAIN REFINEMENT AT SPD

It is possible to distinguish several main features of structures obtained using SPD methods, which have a significant impact on the strength characteristics of the material.

The first feature is an increase in the density of defects in the structure and a decrease in the size of structural elements because of SPD. At the first stages of the formation of the UFG structure, with an increase in the density of dislocations in metals, a cellular structure is formed and fragmentation of the structure is observed. The structure formation occurs in the same way in other single-phase h.c.p. materials [55].

At low degrees of deformation, dislocations accumulate and tangles and plexuses of dislocations form; fragmentation of the structure is observed. The angles of misorientation of grain boundaries between fragments are less than 1° . As the degree of deformation increases, the size of the fragments decreases and a cellular structure is formed. With a further increase in the degree of deformation, dislocations are built into dislocation 'walls' and the formation of 'knife boundaries'. These boundaries have disorientations of the order of several degrees and are very long. Against the background of the developing cellular structure, microstrips and shear bands are formed, which contribute to the formation of the grain structure. Shear bands, as a rule, are formed inside clearly defined mesofields. The formation of microstrips leads to the fact that the initial grains are divided into separate sections where various sliding systems operate [56–58]. With an increase in the degree of deformation, the angles of misorientation of grain boundaries increase and an UFG structure with a smaller grain size and predominantly high-angle misorientation of grain boundaries is formed in the material (Fig. 3).

The second feature of the UFG structure of h.c.p. materials with low energy of packaging defects, which in particular include austenitic steels when deformed at relatively low temperatures or to high degrees of deformation, is the presence of deformation twins [58]. As the authors show [59], the presence of twins, their shape and size can also influence the hardening of the material. The size of the twins depends on the energy of the packaging defects, with its decrease, the size of the twins also decreases, reaching several nanometers [58]. As shown in [60], deformation twinning is also accompanied by the appearance of multiple secondary twins, which also contributes to the formation of UFG structures.

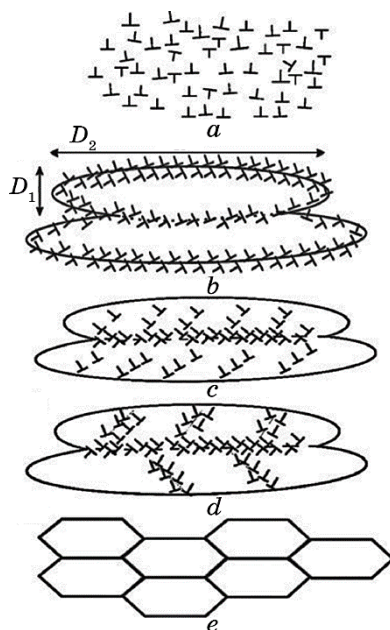


Fig. 3. Schematic illustration of microstructural evolution during severe plastic deformation: uniform distribution of dislocations (*a*), formation of elongated cells (*b*), blocking dislocations by subgrain boundaries (*c*), destruction of elongated subgrains (*d*), reorientation of subgrain boundaries and formation of ultrasmall grains (*e*) [57].

In Ref. [61], the effect of grain size on the process of deformation twinning was investigated. It is shown that, with a decrease in the size of the initial grains, deformation twinning can be suppressed. Accordingly, deformation twinning may be absent or present to a lesser extent in UFG states [62, 63].

During the formation of UFG structures, the dislocation density increases significantly, and dislocation hardening undergoes significant changes too. When the dislocation density in the metal exceeds 10^6 – 10^8 cm $^{-2}$, their elastic interaction with each other begins to affect, causing braking of sliding dislocations and, as a consequence, an increase in shear stresses. The main process of inhibition of mobile dislocations is considered to be their intersection with dislocations of the ‘forest’, *i.e.*, with dislocations that do not lie in the primary sliding plane of the moving dislocation. As a result, dislocation clusters are formed, creating elastic stress fields that gradually lock up the sources of dislocations.

Potential barriers caused by the interaction of dislocations can be divided into long- and short-range stresses. Long-range stresses are caused by elastic stress fields of dislocations and their groups. Short-

range stresses are associated with short-range forces acting at a distance of several lattice periods and arising at the intersection of dislocations, their splitting, the formation of thresholds, kinks, dislocation dipoles.

Adjacent to the theory of hardening by short-range stress fields are theories linking deformation hardening with braking of screw or mixed dislocations with the formation of thresholds on them as a result of mutual intersection. According to Gilman's theory, dislocation dipoles are formed when dislocations with thresholds move—stable pairs of closely spaced dislocations of the opposite sign, chains of vacancies and interstitial atoms that hinder the movement of other dislocations.

In Ref. [64], the main mechanisms of meso- and microstructure changes during hot deformation of samples from AA5052 and AA7050 alloys are considered, which include: generation of dislocations; formation of subgrains due to the formation of dislocation walls; migration of small-angle boundaries, accompanied by rotation of subgrains and transformation of boundaries into large-angle boundaries; migration of large-angle boundaries with absorption of dislocations, small-angle and parts of the large-angle boundaries, the formation of new grains (continuous dynamic recrystallization). A system of equations is given to describe the action of these mechanisms, most of which are modifications of previously known phenomenological relations. To identify and verify the model as a whole, the results of macroexperiments (for torsion and precipitation at different temperatures and deformation rates) were used. A comparison of theoretical and experimental data on several parameters (the dependence of the flow stress on deformation, the proportion of large-angle boundaries, the evolution of the average grain size) demonstrates a satisfactory correspondence.

Figure 4 shows the above mechanisms contributing to the evolution of the microstructure and viscoplastic flow of aluminium alloy during hot stamping: 1—accumulation of dislocations during moulding (Fig. 4, *a*), which lead to the formation of new subgrains with low-angle grain boundaries (Fig. 4, *b*); 2—migration and accumulation of dislocations of the same sign in the grains with low-angle boundaries and subsequent shifting of grain boundaries, which leads to rotation of the subgrains, for example, subgrains 1, 2, 4, 11 and 14 in Fig. 4, *c*; 3—rotation of the subgrains leads to an increase in the misorientation angle and eventually transforms part of the low-angle boundaries into high-angle ones, for example, subgrain 11 in Fig. 4, *b, c, d*; 4—migration of low-angle boundaries absorbs dislocations of grains and leads to an increase in misorientation, gradually turning part of the low-angle boundaries into high-angle ones, for example, low-angle subgrains 21 in Fig. 4, *b, c* in high-angle grains 21 in Fig. 4, *d*; 5—migration of high-angle boundaries sweeps away part of the higher and low-angle boundaries, which leads to a decrease in the corresponding ar-

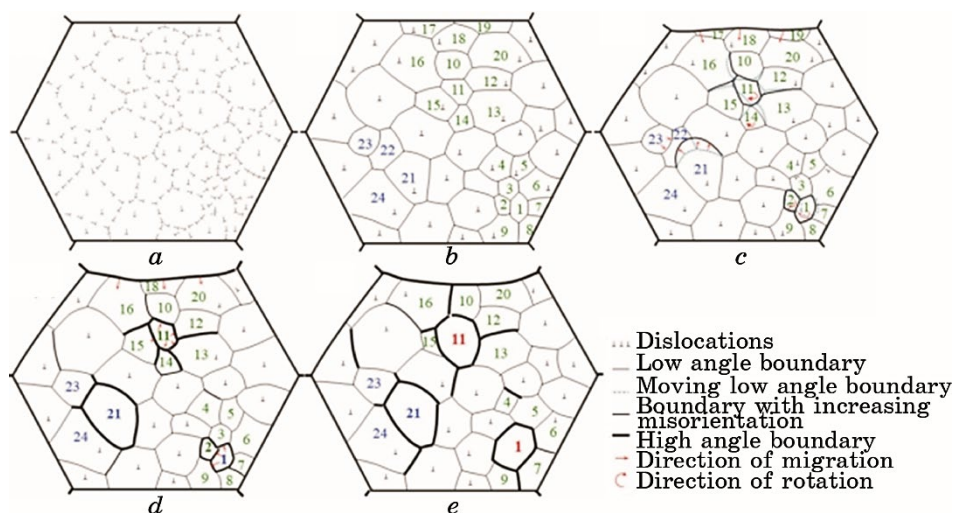


Fig. 4. Schematic illustration of microstructural evolution during deformation: accumulation of dislocations (*a*), formation of LABs and subgrains (*b*), increase in disorientation of LABs due to rotation of subgrains and migration of LABs and decrease of LABs due to migration of HABs and LABs (*c*), formation of HABs from LABs; *e*—decrease of LABs and HABs due to HABs migration (*d*) [64].

eas. For example, migration of the initial high-angle grain boundary leads to annihilation of the low-angle boundary surrounding the subgrains 17 and 19 from Fig. 4, *c* to Fig. 4, *d*; migration of newly formed high-angle boundaries leads to annihilation of the low-angle ones surrounding the subgrain 3 from Fig. 4, *d* to Fig. 4, *e*. Migration of grains with high-angle boundaries sweeps away part of the grains with low and high-angle boundaries that surround grain 11 from Fig. 4, *d* to Fig. 4, *e* [64].

The conclusions made in [65] are largely consonant with the ideas about the formation of boundaries with discrete misorientation of mating regions in terms of the formation of dipole dislocation charges (described earlier in this section, when discussing the bending mode of deformation). When the dislocation dipole has a configuration of two flat dislocation clusters of different signs, the displacement field around such a charge represents a cylindrical bend (similar to the curvature of a tile).

Consequently, it can be imagined that during deformation, a periodic elastic displacement field arises, which, under certain conditions, can relax into shear bands with alternating changes in orientation and non-crystallographic direction of the boundaries.

When the sample enters the matrix, it appears in the field of increasing gradient stresses. However, in addition, additional stresses

arise in the material associated with the need to reorient the lattice to a favourable location of the planes of light sliding relative to the direction of external influence. This process causes the formation of dipole dislocation charges and the subsequent wave field of displacements of atoms, forming the bending of crystal planes.

There are two main models explaining the effect of grain size on the polycrystal flow stress. The classical theory of Hall and Petch is based on the formation of dislocation clusters at the grain boundaries. This concept explains the increase in the strength of metals with a fine-grained structure by an increased concentration of dislocations in clusters and the activation of several sources of dislocations. The strain-hardening model relates an increase in the flow stress during grain grinding to an increase in the dislocation density generated by the boundaries, which is inversely proportional to the grain diameter in accordance with a decrease in the free path of the dislocation. Both models give a qualitatively identical dependence of the polycrystal yield strength gain on the grain diameter, expressed by the Hall–Petch ratio [66, 67].

It is shown in Ref. [68] that three stages of structure formation can be clearly distinguished depending on the interval of realized degrees of deformation, which are shown in Fig. 5. The first stage of structure

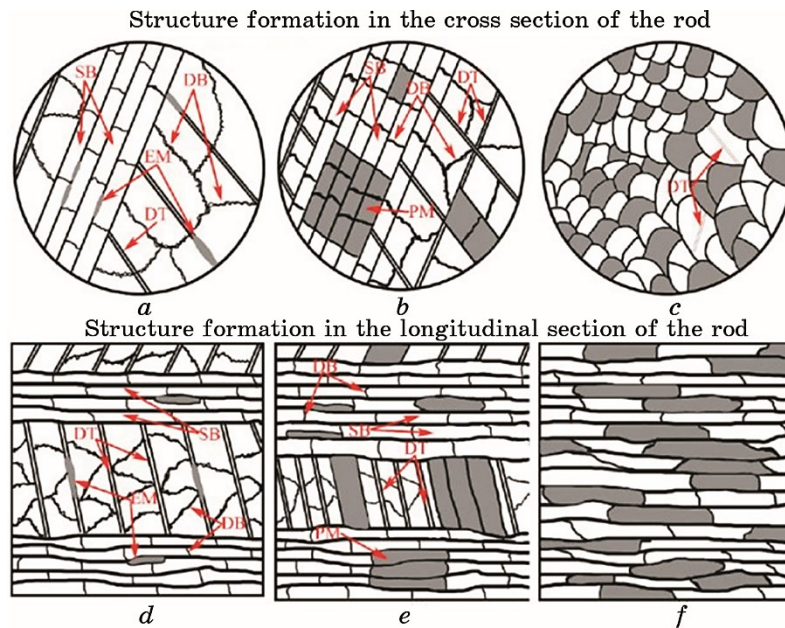


Fig. 5. The scheme of forming the rod structure: the first stage (*a, d*), the second stage (*b, e*), the third stage (*c, f*). Note, that white colour—*austenite*, grey colour—*martensite*, DB—*dislocation boundary*, DT—*deformation twin*, SB—*shear band*, EM—*martensite embryo*, PM—*martensite packages* [68].

formation (Fig. 5, *a*) is characterized by deformation (with degrees up to $e=0.56$) in accordance with the twinning mechanism—the TWIP effect, which forms a barrier for sliding dislocations with the formation of dislocation cells. Deformation twins cross the boundaries of previously formed dislocation cells and further act as barriers to dislocation movement, which causes the formation of cells inside microvolumes bounded by deformation twins. Inside austenitic grains, twinning occurs mainly in one system. Twins in steels with a face-centred cubic lattice are formed as a result of radiation from a partial Shockley dislocation in a system of planes $\{111\}$. In addition to the deformation twins, the structure includes formed shear bands that split austenitic grains into microvolumes of austenite with a lamellar structure oriented in a predictable way along the axis of the rod (Fig. 5, *d*). In addition, at such degrees of deformation, martensitic nuclei are formed in the structure caused by deformation. Thus, at the first stage of deformation, a lamellar austenitic structure with deformation twins and shear bands is formed.

At the second stage of structure formation (the degree of deformation in the range from $e=1.00$ – 1.71), the process of deformation-induced twinning on secondary systems increases, which leads to the transformation of the lamellar structure obtained at the first stage into a block trapezoidal structure in cross-section. However, this provides the basis for martensitic transformation caused by deformation because of the formation and growth of embryos with further formation of martensitic packages.

Thus, at this stage, a block trapezoidal austenitic-martensitic structure is mainly formed (Fig. 5, *b*) in the transverse direction along with obtaining a ribbon structure in the longitudinal section (Fig. 5, *e*).

At the third stage, in most of the studied fields of the microstructure of the cross-section of the rod, the block trapezoidal structure is transformed into an equiaxially granular one (Fig. 5, *c*), which is probably caused by the rotation of each part until an equilibrium orientation is reached. In addition, deformation twins are not found in most grains, for the largest grains, which is due to the so-called restriction effect. In the longitudinal section at this stage, a ribbon austenitic-martensitic structure is formed (Fig. 5, *f*), which is further fragmented by dislocation and interfacial boundaries in the transverse direction.

In Ref. [69], experimental data are presented on the significant effect on the process of changing the microstructure (grain size, texture, grain disorientation) of the processes of dynamic return and dynamic recrystallization in samples from the intermetallic Ni–Al compound obtained by thermostatic pressing from a powder composition. Experiments on the compression of samples were carried out in the temperature ranges of 1100 – 1300°C and strain rates of 10^{-3} – 10^{-1} s^{-1} . At high temperatures and medium strain rate, intermittent dynamic recrystal-

lization prevails, whereas at medium temperatures and high strain rate the role of continuous dynamic recrystallization increases. It is shown that, with an increase in temperature and deformation rate, the proportion of large-angle boundaries increases; a decrease in temperature and an increase in the deformation rate in these ranges lead to greater grain refinement.

The methodology and results of an experimental study of the effect of heat treatment (annealing) of pre-subjected plastic deformation of nickel superalloy samples are presented in [19, 70]. After pressure treatment, the samples have an inhomogeneous grain structure, which can be significantly changed due to annealing. In the first article [19], various variants of single-stage annealing at different temperatures (from 900 to 1100°C) and holding times (from 5 to 180 min) are considered. Because of static recrystallization, grinding and homogenization by grain size occurs, while the δ -phase falling out along their boundaries plays an important role in restraining grain growth; with increasing temperature and annealing time, the proportion of large-angle boundaries increases sharply. The annealing mode at a temperature of 980°C for 10 min is considered optimal from the viewpoint of grinding the grain structure. However, with the increase of temperature, a certain number of large grains remain in the microstructure, which reduces the strength characteristics of the material. In this regard, a two-stage mode was proposed [70]: at the first stage, annealing at a temperature of 900°C for 9–12 hours, and at the second stage, annealing at a temperature of 980°C for 60 min. This mode makes it possible to obtain a homogeneous fine-grained structure with high strength characteristics.

Severe plastic deformations accompanied by a significant change in the grain structure, are also inherent in many (especially high-speed) machining processes. [71] presents the methodology and results of a thorough experimental study of the formation of a nickel (containing 12.3% Cr and several percent Co, Nb, Ta, Al, Ti, Fe) superalloy of the so-called white layer—a surface layer 2–4 microns' thick with a nanocrystalline grain (average size 200 nm) in a milled sample. The grinding of grains, according to the authors, is due to both mechanical influences proper and intensive recrystallization and return occurring in the near-surface layer due to its strong heating (up to 1000–1200°C). An important effect on the inhibition of grain growth during the recrystallization process of particles of γ' inclusions is noted.

The microstructural mechanism of grain grinding during SPD can be schematically depicted in five stages, formed sequentially during microstructural evolution (Fig. 6). In this Figure, the symbol 'T' indicates dislocations. The boundaries of large-angle grains and dense dislocation walls are represented by solid lines. At the first stage, large dislocation cells are formed, which contain numerous dislocations. In the second stage, microstrips are formed and some early dislocation

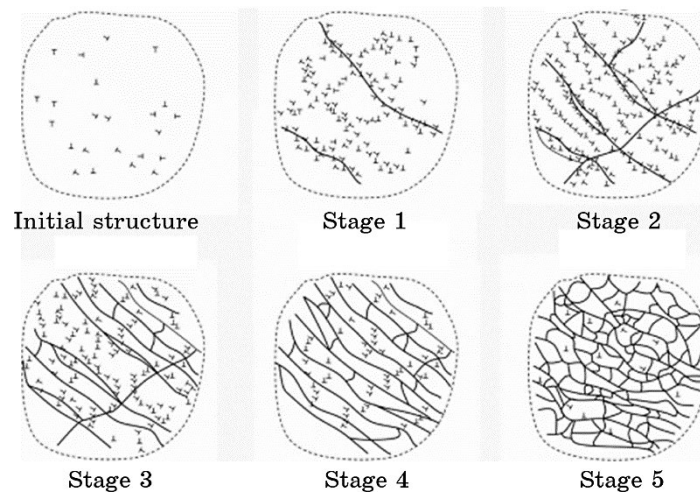


Fig. 6. Diagram of the microstructural mechanism of grain refinement during SPD [72].

cells are transformed into cell blocks. At the third stage, the formation of subgrains occurs, which contain numerous dislocations. At the fourth stage, well developed deformed and equiaxed subgrains are formed. At the fifth stage, there is a homogeneous distribution of equiaxed ultrafine grains or nanograins.

For example, in Ref. [73], the process of grinding grain from ultrafine-grained to nanocrystalline state in Cu–30% Zn alloy during deformation by the HPT method is also schematically described in five stages (Fig. 7). At stage 1, equiaxed ultrafine-grained grains are divided into double lamellae. With an increase in the degree of defor-

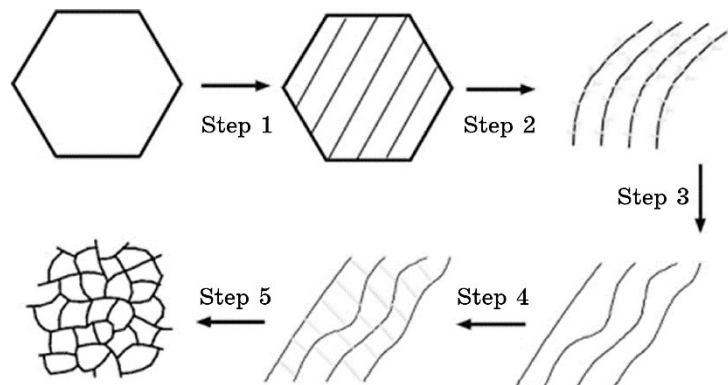


Fig. 7. The mechanism of grain refinement from ultrafine-grained to nanocrystalline state [73].

mation, the density of dislocations, packaging defects and twins increases. Some of these dislocations accumulate in the twins because the twins are obstacles for dislocations to slip [74–76]. At stage 2, there is a continuous accumulation of dislocations on the double, which leads to a gradual bending of the double and turns it into a semi-coherent double. At stage 3, with further deformation, the semi-coherent twins completely lose coherence and turn into a large-angle boundary. As a result, long parallel lamellar grains are formed. At stage 4, secondary grain boundaries and twins divide lamellar grains into rhombic domains. At stage 5, the twin boundaries are transformed into incoherent large-angle boundaries. Such a mechanism is characteristic of metals with low EPD.

In the article [77], the austenite grain refinement process in stainless steel at HPT is described in four stages (Fig. 8). The initial austenite had a coarse-grained structure with a low density of randomly distributed dislocations (Fig. 8, *a*).

At stage 1, deformation twins are formed in coarse-grained austenite. Twinning systems are activated in each grain depending on the relative orientation of the individual grains (Fig. 8, *b*). At stage 2 (this stage covers the deformation stages 2 and 3), a detwisting process occurs, which increases the distance between the twin boundaries. Also at this stage, the remaining twins turn into ordinary large-angle boundaries due to the interaction of dislocations with the twins (Fig. 8, *c*). During the deformation at stage 3, the resulting high-double ultrafine-grained structure goes through a second detwisting process. The decrease in the density of twins caused by the second de-twisting

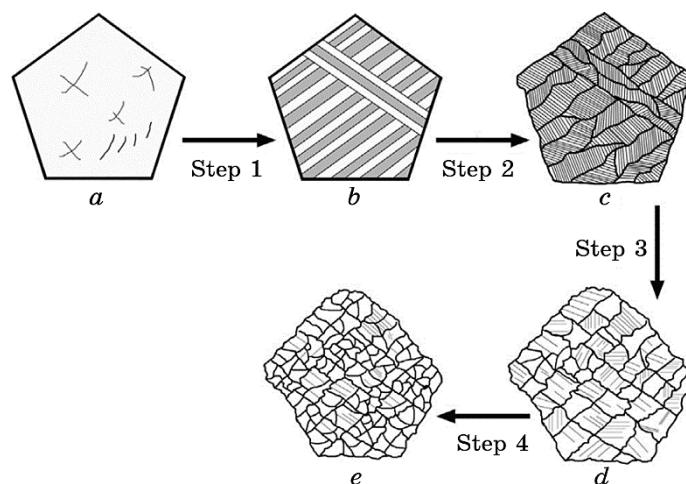


Fig. 8. The scheme of the grain regeneration process in austenite caused by HPT [77].

process increases dislocation activity in the planes $\{111\}$, which leads to a significant interaction of dislocations with twins and consequently to the transformation of the remaining twins into large-angle boundaries for further grain separation (Fig. 8, *d*). Further deformation occurs through the interaction between the newly formed dislocations and nanoparticles, which leads to further refinement of the grains (Fig. 8, *e*).

The microstructural evolution of austenite is rich in interesting features; so, it is worth studying the details of the austenite microstructure at each stage of deformation in order to clarify the deformation mechanisms involved at different stages and how these mechanisms affect the microstructure and properties of the material.

There is still a large amount of work on the evolution of the microstructure and mechanical properties of various metals and alloys in SPD, but the mechanisms of structure formation themselves are very rarely painted and at the moment there is no consensus on the mechanism of formation of UFG and nanostructure.

5. CONCLUSION

The review given in this article showed that the most important factor responsible for the formation of unusual physical and mechanical properties in materials with UFG and NC structure are the features of their highly non-equilibrium structure, in particular the defective substructure of grain boundaries, characterized by high defect density, structural and thermodynamic disequilibrium, the presence of significant fields of local internal stresses, changes in atomic density in border zones, *etc.*

To date, a fairly large amount of experimental papers on the structural features of UFG and NC materials obtained using SPD methods has been published. However, it is not yet possible to link fully these features with the formation of special physical and mechanical properties. In addition, the mechanisms of the formation of UFG and NC states under various SPD conditions have not yet been identified. The latter is largely due to the extremely complex (cooperative) nature of the plastic flow and the reorientation of the crystal lattice under conditions of large plastic deformations.

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