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# SUPERPLASTICITY OF METALS IN MODERN ENGINEERING AND TECHNOLOGY

Currently, studies of structural superplasticity (SP) are of great interest, since the use of this mode in metalworking technologies allows for the production of parts of various shapes in one operation (with high repetition accuracy of even very complex shapes), while requiring less energy and material resources (relatively low pressures and tool wear) compared to deformation in the 'normal plasticity' mode. Other advantages of using a structural SP are improved physical and mechanical characteristics of the finished product: better surface quality after deformation, high ductility at elevated temperatures, increased strength at temperatures close to room one without reducing ductility (most often there is an increase in ductility), increased cyclic strength, hardness, impact resistance, elevated corrosion-resistance durability, and absence of anisotropy of properties after superplastic deformation.

**Keywords:** superplasticity, superplastic deformation, mechanical characteristics, corrosion resistance, grain boundaries, dislocations, diffusion creep.

## 1. Introduction

Superplasticity is understood as the ability of materials (more often metal) to be uniformly deformed to high degrees of deformation at a speed sensitivity level of 0.3-0.5 at a low-stress level [1]. The basic work on superplasticity was performed during testing of materials under uniaxial tension conditions, therefore, in the future, unless otherwise specified, this particular deformation scheme will be implied.

Superplasticity is not limited only to high degrees of deformation and low-strain stress. Thus, in single crystals of high-purity metals, when tested in a vacuum, elongation during stretching can reach several hun-

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dred percent, and their flow voltage is 10-20 MPa. Thus, the main guideline in the superplastic deformation separation is the stability of the flow. In a state of superplasticity, the mechanisms of suppressing the development of necks work in the material [2–5].

Several possible mechanisms of superplasticity are known [6]:

• structural (observed in fine-grained metals under definite temperature and velocity conditions, and occurs due to grain boundary sliding);

• martensitic (observed in materials prone to martensitic transformation);

• recrystallization (observed during dynamic recrystallization).

Strictly speaking, recrystallization superplasticity is also structural since it depends on the initial structure of the material.

There are known three main factors of the material transition to the superplastic state. These factors are adduced below [7].

(i) Microstructure. In the vast majority of cases, superplasticity is manifested in materials with a fine-grained structure (up to 10 microns). Fine grain contributes to the occurrence of all three types of superplasticity mentioned above, although it is not a prerequisite for the last two.

(ii) Temperature. The temperature range of the manifestation of superplasticity lies in the range from the temperature of the beginning of recrystallization to the melting point.

(iii) The rate of deformation. The optimal deformation rate for most superplastic materials lies in the range of  $10^{-4}-10^{-2}$  s<sup>-1</sup>.

The main experimental characteristics of superplastic deformation of metals [6, 7]:

- low level of deformation hardening (0-0.1),
- high-speed sensitivity,
- high level of plasticity (300% or more),

• low level of flow stress (0.1-0.3 of the yield strength for this material).

It is worth noting that abnormally large elongations, orders of magnitude higher than the maximum elongations with 'normal' plasticity (the latter, as a rule, do not exceed 50%), have been detected under definite temperature and velocity conditions for several alloys in experiments on uniaxial stretching for a long time. For example, the obtained elongations are 163% (brass [8]), up to 400% (tin and lead alloy [9]), almost up to 2000% (tin alloy [9]) and others.

Based on the research results presented in the monographs [9, 10], there are two main types of SP: transformational and structural ones. Transformational SP is carried out due to phase transformations during deformation of metals and alloys capable of experiencing polymorphic transformations (for example, steels, iron, titanium and some alloys based on it, *etc.*) [9]: the implementation of this type of SP weakly depends on the initial grain size and processes acting at the boundaries of the crystallites of the matrix phase, significantly, from processes developing near

the interface; if, in the interval of phase transformations, the material is subjected to mechanical action, then it will elongate to large degrees under the action of a tiny force. It is complex to 'catch' such a moment in laboratory conditions (this is a very narrow temperature and speed range), accordingly, the use of a transformational SP for the manufacture of complex shaped parts is associated with great difficulties, therefore this type of SP is practically not used in practice. The structural SP, as the name implies, is determined mainly by the structure of the material: it significantly depends on its initial state, namely, it must be approximately homogeneous fine-grained or ultrafine-grained with an equiaxed grain shape (it is conventionally assumed that the average grain size does not exceed 10 microns [9]). Structural SP from the perspective of its application in technological processes is more promising since it can be implemented in many (several researchers believe that in almost all) alloys with appropriate preparation of the structure and under definite temperature and speed test conditions (relatively wide ranges). In Ref. [6], the following classification of superplastic deformation by species can be found: (1) structural (described above), (2) subcritical (due to the state of pre-transformation), (3) martensitic (due to martensitic transformation), (4) recrystallization (occurs during dynamic recrystallization). The authors of [7], along with the structural SP, propose the term 'dynamic SP', which essentially combines types 2-4 of classification [6] and emphasizes the possibility of implementing SP not only in polymorphic transformations. At the same time, it is noted that the presence of an initial equiaxed fine-grained structure is 'not necessary, but desirable since grain grinding makes it possible to increase the deformation rate by 2-3 orders of magnitude, other things being equal' [11, 12].

# 2. Analysis of the Results of Experimental Studies of Superplastic Deformation of Metals and Alloys

Currently, a large amount of experimental data has been accumulated on SP, its manifestations, and features for a wide range of materials [13-15]. Within the framework of various studies, estimates of the resource of the deformation ability of materials, the dependence of true stresses on deformations for various deformation rates and temperatures, the dependence of residual elongation, and the velocity sensitivity parameter on the deformation rate and temperature are given. The results of data analysis on changes in the material structure during deformation are presented: grain sizes and shapes, the proportion of high-angle boundaries (or histograms of the distribution of misorientation angles), and texture-orientation distribution functions (ODF) of grains. The researchers' assumptions about possible operating mechanisms and the assessment of their contributions to the overall deformation are also given. It should be noted that the

processes of superplastic deformation depend on a significant number of factors. In this regard, a review of modern works devoted to the study of structural SP in various materials, with the consolidation of information, is an essential stage of work that allows us to form a further complete picture of this phenomenon, which, in turn, will help in building a model more adequate to the actual physical process.

SP is observed in the temperature range from 0.4 up to 0.9 homologous temperature [16, 17], at deformation rates in the range of  $10^{-5}$ - $10^{-1}$  s<sup>-1</sup> (sometimes up to 100 s<sup>-1</sup>), with an average grain size of approximately 0.2 microns and up to 10 microns, in rare cases – and more (e.g., [18] shows the results obtained with a grain size of 17 microns, and in Ref. [19], 20 microns). In Ref. [20], attention is focused on the fact that in the material prepared for structural SP, the most important thing is not so much the presence of highly crushed grains as the predominance of large-angle nonequilibrium grain boundaries (with a high-energy defective structure). As one example, aluminium alloy 1420 can be cited, for which the value of the critical grain size is in the range of 0.1-0.3 microns, with smaller sizes, SP deteriorates with a possible change in deformation mechanisms [20]. The grain size restriction from below, equal to 0.1 microns, occurs for almost all metallic materials since with a smaller grain size, the structure becomes nanocrystalline with a large number of triple joints in the structure and, accordingly, the predominant influence of mechanisms associated with triple joints rather than facets of boundaries [21–23].

Diffusion creep occurs in metallic materials at low deformation rates and temperatures above  $0.5T_{\rm m}$ . Under such conditions, deformation may not depend on dislocation displacement. Under the conditions of the formation of a selected stress direction, the diffusion flux of vacancies becomes ordered, which leads to macroscopic changes in the shape of the sample.

Grain boundary sliding (GBS) is accompanied by shifting and individual grain rotation relative to each other without increasing grain size.

A model of the micromechanism of the GBS is described in Ref. [24]. A separate emphasis is placed on the ratio of the grain size d in the material and the size of the substructure element  $\lambda$  (Fig. 1). It is assumed that dislocations move along the grain boundary between two neighbouring grains and accumulate at the triple grain junction designated as A. That creates a stress concentration so that intragrain sliding begins in the neighbouring grain, which leads to the formation of an accumulation at the opposite grain boundary (fragment) at point B and subsequently accommodation of the shift at the opposite grain boundary.

Experimental observation of sliding along the boundary of two grains of a magnesium alloy was carried out in (Fig. 2) [25].

It should be noted that all the structure characteristics depend on the method of preparing the material for the joint venture and on the initial

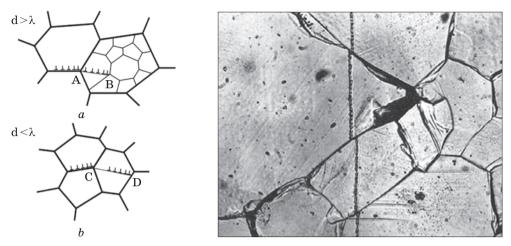


Fig. 1. Diagram of the GBS mechanism in a material with different grain sizes, viz, where (a) grain size is significantly larger than the substructure element, and (b) grain size is smaller or comparable to the substructure element [24]

Fig. 2. The GBS observation: scratch shear in the Al–Mg alloy, when stretched horizontally at 200  $^{\circ}\mathrm{C}$  [25]

temperature and speed test conditions. Two main methods of thermomechanical processing (TMP) can be distinguished, as a result of which the internal structure of the sample is prepared for the superplastic deformation test: (1) using cold or warm deformation (up to approximately 0.4homologous temperatures) with usually subsequent recrystallization annealing [26-32]; (2) due to deformation at high temperatures (more than 0.5 homologous, e.g., [17, 33-38] and others). In the first method, a finegrained structure is formed mainly by fragmentation and crushing during deformation, with subsequent annealing and ongoing static recrystallization playing an important role, which contributes to the transition to a fine-grained equiaxed structure; the intensity of this process is related to the degree of grinding of the structure [39]. As an example, grain crushing in titanium alloys, which actively occurs as a result of twin shifts, leads to an increase in the number of static recrystallization centres during annealing and contributes to an increase in SP under definite temperature and velocity conditions. In the second method, a fine-grained structure is formed mainly by dynamic recrystallization [35, 36, 40]. The use of the first method for preparing the structure of the material is restricted due to the need to implement large deformations at low temperatures before annealing, which is technologically unprofitable (due to significant energy consumption and rapid wear of equipment), therefore, the second method of preparing the structure is the most commonly used (however, special attention is required here to select the temperature to achieve optimal phase and structural state of the material).

Recently, due to the advent of severe plastic deformation (SPD) methods, the preparation of the structure of samples for joint ventures often occurs by equal-channel angular pressing or extrusion (ECAP or ECAE) [41-45], torsion under high pressure at elevated temperatures [46-48]. In such SPD processes, some relaxation of internal stresses occurs, the grains are crushed, and the boundaries move to a more equilibrium state (i.e., they become less defective or, in other words, 'smoothed out', since they gradually evolve from a nonequilibrium high-energy structure to a more equilibrium low-energy one [49, 50]) in comparison with the boundaries formed due to SPD at temperatures close to room temperature [51]. It can be noted that the existing SPD methods have their advantages and allow the production of bulk and sheet SP materials (in the latter case, in combination with rolling [52-54]). It should also be added that the improvement of SPD methods in combination with various types of TMP makes it possible to expand significantly the list of materials, in which SP is observed by creating an optimal structure in them by modifying the phase and chemical composition. The latter, in turn, leads to an expansion of the ranges of temperature and velocity conditions of the superplastic deformation.

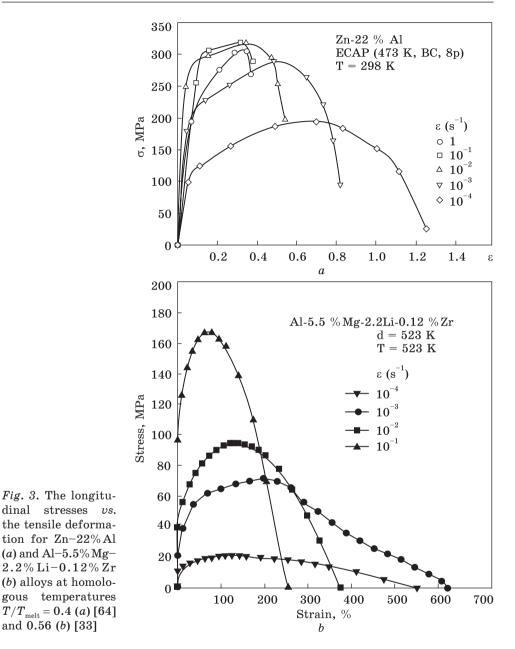
The structural joint venture is observed mainly in multicomponent close to single-phase and two-phase alloys with different atomic phase structures. In the first case, the structure of alloys after sample preparation is usually a limited solid solution with a matrix structure of the metal of the principal phase with particles of intermetallic phases distributed by volume and grain boundaries (the volume fraction of additional metal components in such alloys does not exceed 10%, but particles play an important role, primarily by limiting grain growth and ensuring the stabilization of the grain structure for the implementation of the superplastic deformation).

The characteristics of the structure depend on the temperature and speed conditions of the implementation of a particular SPD method and the number of passes. For example, in alloy 1420, in addition to prepared grains and a large number of high-angle boundaries (after 8–10 passes of ECAP and ECAE), enlarged grains with a developed substructure are found, characterized by the presence of separate dislocations, piles (clusters) of dislocations and dislocation sub-boundaries, the density of dislocations is  $10^9$  cm<sup>-2</sup> [55–57]. For alloy 1421, it is noted that after 12 passes of the ECAE [40], the proportion of recrystallized grains is approximately 95%, the dislocation density is relatively low and is cm<sup>-2</sup>; after 16 passes of the ECAE [35], the recrystallized structure is almost completely formed. In Ref. [26] for the alloy Al–3% Mg–0.2% Sc, the authors also note that, after 8 passes of the ECAP, there is still a significant proportion of subgrains (about 10% of the total volume) and, accordingly, sub-borders in the sample; after 12 passes, there are practically no sub-

grains and sub-borders in the sample. In all the sources found data on SP testing of aluminium alloys are given for samples obtained using 8 (in some cases up to 10) SPD passes, *i.e.*, containing a certain proportion of non-crystallized material. It can be assumed that with a larger number of passes, additional changes occur in the grain and boundary structure, in particular, related to the redistribution or change in the number of particles in the volume of the material, which does not lead to an improvement in SP properties [58–60].

In the magnesium-based Mg-9% Al alloy close to single-phase, the structure of the extruded material is heterogeneous and contains discrete regions of large (average size of about 12 microns) and small (with an average size of about 0.5 microns) grains; after high-pressure torsion loading at temperatures of 298 K and 423 K, the structure becomes more homogeneous with a small size grain amounting to approximately 0.15 microns and 0.37 microns, respectively (the latter means that the preparation of the material at higher temperatures is accompanied by grain growth) [61]. In tensile tests of samples made of rolled Mg-3% Al-1% Zn magnesium alloy sheets (with a grain size of about 12 microns), the structure is crushed, which, under optimal conditions, leads to an 85% homogeneous structure with an equiaxed grain shape with an average size of about 6 microns [36]. The grain structure of the Mg-5.88% Al-0.74% Zn alloy is equiaxed, but not fine-grained (the average grain size is 17 microns), while the structure practically does not change after deformation [18]. In Mg-1.0% Zn-0.26% Zr alloy, the structure is bimodal, *i.e.*, it consists of small and bigger grains (it is noted that the latter is quite often observed in magnesium alloys), with an increase in the number of passes of the ECAP, this trend persists, while the average grain size decreases [62].

In two-phase alloys, the material internal structure after sample preparation consists of differently distributed grains of each phase. For instance, in the works [36, 63, 64] for the Zn-22% Al alloy, the structure is characterized by an approximately uniform distribution of equiaxed grains of the Zn and Al phases. In Ref. [65] for the Zn-15% Al alloy, it is noted that with a decrease in the extrusion temperature, the volume fraction of the Zn phase increases. The microstructure of the Al-30% Zn alloy consists of small equiaxed grains of the aluminium phase with a size of about 0.4 microns and smaller grains of the zinc phase located in triple joints. There is a small number of very small (5-15 nm in size) Zn grains located inside Al grains [66]. The structure of the Pb-62% Sn alloy contains two interpenetrating phases based on Pb and Sn [67, 68], after one passage of the ECAP, part of the eutectic remains in the structure, and the rest disintegrates, *i.e.*, smaller grains of one phase are distributed in another phase, with an increase in the number of passes, the structure becomes more homogeneous [69]. Cu-40% Zn alloy is another classic example



of two-phase materials containing grains of the Cu and Zn phases crushed after ECAP. In Ref. [70], the results of a study of the behaviour of the same alloy, but with various additives, with which solid solutions based on Cu and Zn are formed, which leads to a change in the characteristics of the phases (in particular, diffusion ones) and can improve the joint venture. The microstructure of Ti-6Al-4V two-phase titanium alloys prepared for

joint venture consists of small equiaxed grains and phases [71, 72], the proportion of which increases with an increase in the number of ECAP passes [73-75].

Based on some of the presented examples of alloys, it can be concluded that the range of materials for which a structural SP can be observed is extensive, and there are many modified ('improved' for SP) alloys. In the following sections, the results of experimental studies of the SPD of some materials with a description of the changes in their structure occurring during deformation will be considered.

An analysis of the results of studies on uniaxial stretching of samples from several alloys with access to the SP mode showed that stress-strain dependences exhibit stages for various materials (close to single-phase: aluminium alloys [17, 27, 33, 55], magnesium alloys [62, 76]; two-phase alloys based on zinc and copper [64, 70, 77], titanium alloys [72, 78]). In Fig. 3 for examples illustrating this trend, the tensile test curves for samples from different materials (two-phase alloy and solid solution) at various temperatures are presented.

The emphasis on the gradation of curves in superplastic deformation tests is made in many works. For example, in Ref. [78], it is noted that the stretching curves for fine-grained materials in the ranges of temperature and velocity influence characteristic of SP demonstrate a sufficiently extended hardening stage, followed by an at least extended softening section, 'as a result of which the curves acquire a characteristic bell shape'. In many works, for example, in Refs. [20, 35, 79, 80], it is noted that the deformation proceeds uniformly and there are no necks within the measuring base of the sample; therefore, it can be assumed that the presence of a falling section of the curve is due precisely to the properties of the material, and not to inaccuracies in the interpretation of experimental results. In Ref. [42], three stages were defined for aluminium alloy 1420: hardening (immediately after the elasticity section), a short stage with constant flow stress, and a softening stage. It is shown that there is a continuous decrease in the real deformation rate with an increase in the degree of real deformation, that dependence is correlated with the tensile curve and it is obtained that the first stage of deformation corresponds to the real deformation rate of  $10^{-2}$  s<sup>-1</sup>, the second stage corresponds to  $10^{-3}$  s<sup>-1</sup>. In Refs. [42, 55], for two stages of hardening and softening, the activation energy of plastic deformation was determined using a standard method by the angle of inclination of the temperature dependence of the real deformation rate obtained based on experimental data and a well-known phenomenological relationship linking the deformation rate, stress, and temperature. For the hardening stage, a value of 1.4 eV was obtained, corresponding to the self-diffusion energy in the grain volume, for the softening stage, it is approximately of 1 eV, characteristic of the self-diffusion energy in the boundaries. Based on the obtained values, the authors conclude that at the first stage, the deformation is controlled by intragrain dislocation sliding (IDS), and at the second stage, by GBS.

### 3. Mechanisms of Superplasticity in Alloys

Superplastic flow is usually associated with three mechanisms that are also characteristic of creep:

- grain boundary sliding;
- intragrain dislocation sliding/crawling;
- diffusion creep (DC).

Currently, the phenomenological description of the mechanisms of superplastic deformation is well developed. However, there is no complete fundamental generally accepted understanding of the mechanisms of superplasticity and the theory of their accommodation. Many studies of the phenomenon of superplasticity have been conducted, but so far in the literature [81, 82], there are very different views on the dominant mechanism of superplastic deformation in various alloys.

The ratio of the operating mechanisms of superplasticity, as can be assumed, should be determined by microstructural features and temperature-velocity deformation conditions.

The superplastic flow is accompanied by processes such as grain rotation [81], migration of grain boundaries and dynamic grain growth [81], dynamic recrystallization and polygonization [82, 83]. Note that the term intragrain dislocation sliding should be understood as both conservative and non-conservative dislocation movement, *i.e.*, creeping.

The change of grain neighbours is the most essential function of the GBS, since, when stretched, it leads to an increase in the number of grains in the longitudinal direction and a decrease in their number in the direction transverse to stretching. That is the result of a massive mutual displacement of neighbouring grains, which may explain the possibility of giant elongations of superplastic alloys. It is characteristic only of SPD and rarely manifests itself in the processes of high-temperature creep or hot deformation. Most works [84] define grain boundary sliding as the dominant mechanism of superplasticity, which can occur due to the migration of grain boundaries and grain displacement relative to each other.

However, the question arises about the accommodative mechanisms of deformation since many discontinuities should have formed during grain boundary sliding, and pore formation prevented large elongations.

The principal process accompanying the superplastic flow is the dynamic growth of grains, which depends on their initial size and the kinetics of growth over time and affects the flow stress. The dynamic grain growth under superplasticity is anisotropic in a (quasi-)single-phase material; this unique characteristic was first considered by several studies back in the 1990th [81], but today attracts the attention of only a limited num-

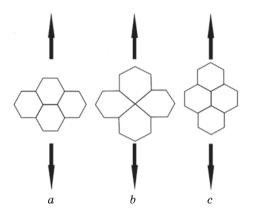


Fig. 4. The Ashby-Verrall model of diffusion-accommodated flow [88]

ber of researchers [29], although its mechanism is still unclear.

Rabinovich and Trifonov [85], as well as Shin and colleagues [86] explain grain elongation by anisotropic migration of grain boundaries, whereas Li et al. [87] consider it as a joint effect of dislocation creep and coalescence of grains during rotation.

In 1973, Ashby and Verrall [88] proposed a model (Fig. 4) explaining superplasticity as a transition region between diffusion creep acting at low strain rates and dislocation creep at high strain rates. The Ashby–Verrall model assumes the movement of grains using grain boundary sliding while maintaining the shape of the grains. Moreover, Ashby and Verrall explained that the stress difference between the beginning and the end of deformation is accommodated by grain boundary sliding and bulk diffusion. The Ashby–Verrall model has many attractive features and combines essential typological features inherent in superplasticity.

There are several works criticizing this model [89]. Spingarn and Nix [90] believe that the movement of grains cannot occur exclusively through diffusion flows and assume that such a process is physically impossible.

Currently, there are three models for the theory of grain boundary sliding, accommodated by dislocation sliding: the Ball-Hutchinson model [91], the Mukherjee model [92, 93], and the Gifkins model [94, 95].

Ball and Hutchinson in 1969 [91] proposed a model for the joint movement of a group of grains (Fig. 5) as long as such movement is possible. When deformed, the grain boundaries line up correctly and slide in one direction as a group. When sliding is blocked by other grains, an increase in stress leads to dislocation activity dislocations form and accumulate at the

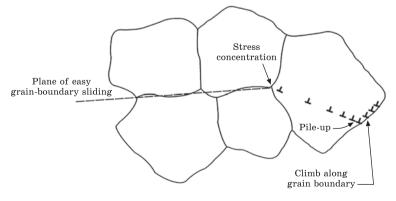


Fig. 5. The Ball– Hutchinson model of grain boundary sliding accommodated by dislocation motion [91] opposite grain boundary until applied stresses block them. Dislocations can creep along the grain boundary or the grain body. Thus, grain boundary sliding, which is regulated by the creeping of dislocations along grain boundaries, is possible due to the constant replacement of boundaries.

However, Mukherjee and colleagues [92] modified the model, assuming that initially, the grains move separately and only then combine into groups. The model supposes the presence of a dislocation accommodating the grain boundary sliding of one grain. Dislocations are generated at grain boundaries the mechanism of generated dislocations is the same as the mechanism according to the Ball-Hutchinson model.

Among the most famous works are Mukherjee and Arieli [93] as well as Langdon [94]. According to these authors, the GBS includes the movement of dislocations along grain boundaries, and due to excessive stress in the triple joints, the movement of dislocations spreads over the grain body.

Superplastic flow is often associated with Rachinger sliding, which consists of changing grain neighbours by overgrowth, grain boundary sliding relative to each other, and relatively small changes in grain shape due to diffusive mass transfer. Dislocation structures, for example, subgrain boundaries, are rarely present during deformation, and single dislocations along the grain body are usually observed. A sign of diffusion creep is zones free from secretions (ZFS), which are formed in alloys containing particles of the second phases [95].

Diffusion creep is a natural and necessary mechanism in any hightemperature deformation, in particular, in superplastic flow. Studies of creep mechanisms for most alloys show that under conditions of low mechanical stresses and temperature with small grain size (parameters related to superplasticity), deformation is controlled by diffusion along grain boundaries (Coble creep). The classical diffusion creep can be refuted only because calculations do not confirm the possibility that the diffusion rate is commensurate with the rate of superplastic deformation.

It is essential to understand the reason why diffusion creep may not be observed. That may be because, under experimental conditions, either an alternative, faster mechanism works, in which diffusion creep does not have time to pass, or one of the other processes necessary for creep and controlled by the rate of deformation, for example, the occurrence or runoff of vacancies, as well as grain boundary sliding, which is geometrically necessary for creep.

In the case of the Gifkins model [95] and others related to the movement of dislocations along grain boundaries, the theory assumes that there are no reasons preventing the movement of dislocations line up in walls, forming subgrain boundaries.

The simplest model of superplastic deformation, which is consistent with all experimental data, quite naturally arises from the basic geometric theory of grain boundary structure. This theory proposed by Bollmann

and supported by some scientists provides a set of basic rules, according to which certain types of grain boundaries and, in particular, grain boundary dislocations should arise. The principal assumption of the theory is that in a well-annealed sample, most grain boundaries have a quite perfect low-energy structure and are limited by internal secondary grain boundary dislocations, which show a deviation from the perfect structure. Secondary grain boundary dislocations are not mobile, and their movement is controlled by crawling. Their movement, as a rule, leads to simultaneous grain boundary sliding and migration of grain boundaries. The movement of dislocations also depends on the rate of deformation. Depending on the strain rate and temperature, some mechanisms may be strengthened and others weakened, and vice versa [96].

The analysis of various experimental data shows that GBS, which is the displacement of one crystallite relative to another along the common boundary, is an essential mechanism for the deformation of metals at temperatures above  $0.4T_{melt}$  (where  $T_{melt}$  is a melting point) [6]; at low temperatures (less than  $0.3T_{melt}$  grain boundaries act as barriers [97, 98], which leads to hardening (the stronger the smaller the grain size by the well-known Hall-Petch effect [99, 100]). Deformation rates, stresses, the chemical and phase composition of the material, the internal structure (the shape and size of the grains, the structure of the boundaries, and the relative misorientation of the grains) also have a significant effect on the GBS. There is a close relationship between GBS and the development of sliding in grains (IDS): grain boundaries can act as active sources and sinks for lattice (intragrain) dislocations and vacancies, which leads to a softening of the boundary regions (an increase in the return rate and GBS), the more intense the more intensive the extent of grain boundary areas at high temperatures (*i.e.*, with a decrease in grain size). Deformation rates are essential for registry office processes. At very low deformation rates, IDS and GBS are poorly developed, and diffusion processes play an essential role (especially at elevated temperatures), with a decrease in grain size, the role of grain boundary diffusion increases. At relatively high deformation rates, the role of the IDS is significant, the GBS is small (the boundaries do not have time to prepare), and the influence of diffusion processes is less meaningful the lower the test temperature.

Under optimal temperature and speed conditions for SP and grain size, the GBS acts as the leading mechanism (in the joint venture mode), which is noted in many modern works for both alloys close to single-phase and two-phase alloys. For example, in Ref. [101] for the Zn-0.3% Al alloy, using marker lines applied to the sample surface, it was found that GBS is active, the role of which decreases with an increase in the deformation rate. For other close to single-phase aluminium and magnesium alloys Al-0.2% Zr [102], 1420 (Al-2.1% Li-5.5% Mg-0.1% Zr) [6], 1421 (Al-5.1% Mg-2.1% Li-0.17% Sc-0.08% Zr) [35], 5083 (Al-4.7% Mg-

0.7% Mn) [54], 6061 (Al-1.1% Mg-0.45% Si) and its modifications [103], 7055 [104], 8090 (Al-2.4% Li-1.15% Cu-0.67% Mg-0.11% Zr) [105], AZ31 (Mg-3% Al-1% Zn) [36], AZ61 (Mg-5.88% Al-0.74% Zn) [18], AZ91 (Mg-9% Al-1% Zn-0.2% Mn) [106], ZK60 (Mg-6% Zn-0.5% Zr) [76], it is also indicated that the GBS is the prevailing mechanism. For a two-phase Zn-15% Al alloy, it is noted [65] that, with a decrease in the extrusion temperature during the preparation of the material structure, the volume fraction of the Zn phase increases, the number of grain boundaries becomes larger and, accordingly, the GBS is more significant.

The analysis of the information leads to the conclusion that in the SP mode, the GBS is the leading mechanism, however, for its activation and implementation, the action of accommodation mechanisms (IDS and grain boundary diffusion) is necessary to ensure the necessary state of the material structure (grains and boundaries).

In the course of several studies, it has been shown that IDS is the principal accommodative mechanism of GBS in SP (influx of lattice dislocations into the boundaries). For example, studies on Al-Zn-Mg allow samples using internal markers have shown that IDS occurs with SPD (the latter is confirmed by electron microscopic studies) [107]. The importance of the role of IDS is also noted for the Cu–Zn alloy system: its effect is observed on the surface of deformed samples in the form of wide folded zones located in the border regions [70]. In the classical Pb-62% Sn SP alloy, no sliding lines were found inside the grains, and the contribution of IDS to the overall deformation is small, however, its role as an accommodative mechanism of GBS is great (the influx of lattice dislocations into the boundary contributes to GBS due to the constant formation of external mobile grain boundary dislocations in the boundary [19]. During the study of the microstructure of the SP sample made of Zn-0.3% Al alloy [101], significant dislocation activity was found near grain boundaries. For Al-3% Mg-0.2% Sc alloy, it was also noted that the IDS is an accommodative mechanism of the GBS. Many more examples can be given describing a similar effect of the IDS mechanism in the GBS of various alloys. It is necessary to note the main difference for the considered classes of materials [26]: in classical two-phase SP alloys, some examples of which are given above, lattice dislocations pass quickly and unhindered through the grain into the boundary (since no particles are acting as barriers); in solid solution alloys close to single-phase ones, the movement of lattice dislocations can be inhibited due to the presence of solid barrier particles: dislocation creeping processes are complex, however, if GBS is actively occurring, dislocations do not accumulate in front of the boundary and good SP performance can be achieved as in conventional two-phase alloys.

It is important to note once again that the above information is related to the SP mode. Differences in the action of mechanisms at each stage are also associated with the presence of stages of the stretching curves. For

example, the enlarged noncrystallized grains and unprepared boundaries contained in the initial (prepared for SP) structure of many materials lead to dominance at the initial stage of IDS, which plays a significant role in activating the process of dynamic recrystallization. At the same time, the GBS is poorly developed and anisotropic; there is a notable difference in the intensity of sliding along different boundaries, depending on their orientation concerning the characteristic loading axes. In the mode of stable structural SP, when the internal structure is fully prepared, GBS is carried out along almost all boundaries together with the accommodation mechanism of the IDS, while grain boundary diffusion, the processes of changing neighbouring grains and rotations of their lattices play an essential role, as a result of which the deformation becomes close to isotropic [54] with a predominance of the contribution of GBS to inelastic deformation.

Based on the results given in various experimental studies [108–112] and theoretical [113] studies, it can be concluded that there is some ambiguity in the estimates of the contributions of the mechanisms: on the one hand, the proportion of GBS is of about 50-70%, then, the remaining contribution of 30-50% should be carried out at the expense of IDS (taking into account the negligibly small contribution to deformation from diffusion); on the other hand, direct measurements of IDS lead to values of its contribution of only a few percent, which means that the predominant part of the deformation is realized at the expense of GBS. Within the framework of a given work, it is assumed that, when determining the shares of IDS and GBS, it is necessary to specify what is meant: the ratio of small increments (velocities) of contributions at the current time or the total estimate for the entire deformation process. If the process goes on without a significant change in the shape of the grains, which is typical for the SPD stage, then, there is practically no deformation of the grains relative to the initial configuration as a result of the process; therefore, there should be no (significant) contribution to the final macro-deformation from the IDS. During the process, at any given time, the IDS in the grains can be significant, as well as the rotation of the grains; however, due to the simultaneous implementation of these mechanisms, the total deformation of the grains at the stage of implementation of the SP regime is close to zero (the grains constantly 'turn up' which prevents them from acquiring an elongated shape as a result of the IDS). In addition, differences in estimates of the contribution of mechanisms may also be related to the presence of stages in the stretching curves. Often, the determination of the contributions of various mechanisms is established using experiments on stretching or compression of samples at small elongations of the order of 20-50%. With the SPD of materials demonstrating staging with such small elongations, as a rule, the stable SP mode (with the leading GBS mechanism) does not yet occur, which means that the contributions of the GBS mechanism at small and large elongations may be different.

# 4. Mechanisms of Superplasticity in Alloys with Different Structures or Compositions

Superplastic alloys are structurally separated into two main types: duplex and matrix. A structure is called a duplex if it consists of two phases of similar shape and size that are distinguishable from each other. Matrix alloys are divided into single-phase and multiphase. Single-phase alloys consist only of a solid solution. The multiphase ones include a solid solution and particles of the second phase. According to the structure, matrix alloys are divided into: (1) alloys with initially recrystallized grain structure before superplastic deformation; (2) partially or completely noncrystallized structure before the onset of superplastic deformation.

A striking representative of alloys with a matrix type of structure is alloy AA5083 based on the Al-Mg-Mn-Cr system, in this alloy a superplastic state can be realized due to significant deformation during cold rolling [114]. It has been shown that the presence of chromium in alloys of the Al-Mg-Mn system ensures the formation of small grains and superplasticity due to the release of more compact dispersoids [95]. At the same time, with an increase in the magnesium content in chromium alloys, superplasticity indicators are improved, deformation rates and the m index increase, flow stress decreases, and elongation increases.

Currently, there are contradictory conclusions in the literature about the mechanism of accommodating GBS during superplastic deformation in various alloys. Many studies (see, e.g., [88, 92]) emphasize the importance of directional diffusion mass transfer as an accommodative process of GBS (Lifshitz's GBS). In many cases, zones free from secretions that appear during deformation have been observed, usually, such zones are associated with diffusion creep [81, 115]. In ones of the recent papers, Rust, Todd [116], Sotoudeh and Bate [81] confirmed that diffusion creep without any dislocation activity is inherent in aluminium alloys of the AA5000 type. However, some studies [117, 118] have suggested the importance of dislocation sliding. Bate and colleagues [119] showed that dislocation sliding is the principal deformation mechanism in some Al-based alloys. Accommodation due to the movement of dislocations is the basis of the theory of grain boundary sliding, according to Rachinger [120]. Langdon [24] concluded that GBS, according to Rachinger and Lifshitz, leads to the same displacements of surface marker lines. At the same time, the Gifkins deformation in the 'mantle' of the grain should be the cause of the curvature of the surface markers near the grain boundaries, in contrast to the typical precise shifts of the marker lines, according to Rachinger.

In several works on the study of superplastic deformation in twophase alloys, it is noted that recrystallization does not occur (for example, for Pb-62% Sn alloy); in other works [67-69] with data for the same alloy, grain growth is not mentioned. For the Zn-22% Al alloy, the reviewed

papers [63, 64] also say nothing about grain size changes. In another experimental work [121] for the Zn-22% Al alloy, it was indicated that grain growth occurs at very high test temperatures (of about 493 K ( $0.75T_{melt}$ )), while it is insignificant. Some grain growth was detected for Cu-40% Zn alloy and its modifications at a temperature of about  $0.6T_{melt}$  and a deformation rate of  $10^{-3}$  s<sup>-1</sup>. Grain growth is observed in the A1-33% Cu alloy at high temperatures and different deformation rates.

In alloys close to single-phase alloys, dynamic recrystallization is often realized to varying degrees at the initial stages of superplastic deformation tests. This issue was considered in more detail using the example of some aluminium alloys. Data on static annealing in Al-3% Mg and Al-3% Mg-0.2% Sc allows show that the structure is more or less stable up to about 450 K [26]. For Al-3% Mg-0.2% Sc alloy at a temperature of 673 K and a deformation rate of  $3.3 \cdot 10^{-2}$  s<sup>-1</sup>, a rather significant grain growth from 0.2 to 4 mµ is observed. For the same alloy, but deformed at a temperature of 403 K  $(0.43T_{melt})$  [50], dynamic return and recrystallization are realized, leading to rearrangement of dislocations, limited grain growth, and the formation of high-energy nonequilibrium boundaries, which then gradually transform into more equilibrium ones. It is also noted that, unlike conventional dynamic recrystallization, which occurs, for example, during hot metal processing and consists of the constant formation of multiple localized recrystallization centres (nuclei) throughout the entire volume of the material, which germinate into more defective grains (absorb the latter), in the considered fine-grained alloy, recrystallization is carried out without nucleation in one cycle, it is controlled by the stored energy of the material accumulated during its preparation by the SPD methods (and at the initial stage of the superplastic deformation test).

The principal mechanism of superplastic deformation in aluminium alloy AA7475 or its analogue AA7075 is grain boundary sliding, which involves the change of neighbouring grains and their rotation. GBS in an alloy is always closely related and manifests itself together with one of the accommodation processes, such as diffusion creep or intragrain dislocation sliding; or is consistent with the theory of Gifkins, as suggested in Ref. [122]. It should be noted that the operating mechanisms may change with an increase in the degree of deformation, as noted in Ref. [123]. The evolution of GBS with an increase in the degree of deformation has not been sufficiently studied so far. Based on the available data, it can be assumed that the mechanisms of superplastic deformation depend on the characteristics of the alloy and the test parameters. The inconsistency of the researchers' data is possible due to different test conditions, sample geometry, grain size, etc.

Among the various aluminium alloys, the AA7XXX series of the Al-Zn-Mg system with Sc and Zr additives stands out for its excellent strength [71] and high formability combined with superplasticity. Such al-

loys are used for the production of various parts in the aerospace industry, since their superplastic forming rate is much higher than that of most aluminium alloys and can reach  $1 \times 10^{-2}$  s<sup>-1</sup> [124] due to particles that control grain growth and restrain recrystallization. The microstructure of alloys of this type ('supral' type [125]) is fibrous at the beginning of superplastic deformation; recrystallization does not occur or occurs only partially, when samples are heated to the temperature of superplastic deformation, even in the subsolidus region. It is known that Al<sub>3</sub> (Sc, Zr) particles inhibit grain growth and provide high resistance to recrystallization. Many studies [126, 127] have shown that the resistance to recrystallization for alloys with Al<sub>3</sub>(Sc, Zr) particles is higher compared to alloys with Al<sub>3</sub>Sc particles.

Based on the above, it can be concluded that dynamic recrystallization during SPD tests can be carried out mainly in alloys close to single-phase, less often in two-phase alloys; probably, interphase boundaries inhibit grain growth to a greater extent than phase particles in solid solutions (the initial structure of the material always plays an important role and temperature-speed test conditions).

At moderate temperatures (of the order of 0.4-0.5 homologous) and low deformation rates  $(10^{-5}-10^{-4} \text{ s}^{-1})$  or at elevated homologous temperatures (up to 0.5-0.7; the upper-temperature value may vary depending on the degree of dissolution of the particles restraining growth) and stable flow stress, or with its gradual decrease). It is assumed that, at not very high temperatures, the principal role of recrystallization is to complete the preparation of the internal structure of the material for the structural SP regime by reducing the density of dislocations in grains accumulated after sample preparation by IPD methods (grain growth occurs), approaching an almost completely recrystallized low-defect grain structure with an equiaxed shape (initially elongated or changed shape as a result of the action of the IDS, the grains return to an equiaxed shape).

The mode of structural SP (a stage on a curve with stable flow stress or with its gradual decrease) can be characterized by the following signs: the predominance of the GBS mechanism, accompanied by accommodative mechanisms of IDS and grain-boundary diffusion, rotations, and active change of neighbouring grains; the structure is stable (remains equiaxial fine-grained). The stability of the structure can be understood in different ways: either all grains remain practically unchanged (due to the simultaneous implementation of accommodative IDS and grain rotations when grain shaping occurs at each moment, but due to constant rotations, the resulting grain deformation is close to zero), or continuous dynamic recrystallization continues to operate (as noted above, for example, at high test temperatures) with a complete restructuring of the structure, but the latter remains fine-grained equiaxial.

The exit from the structural SP mode is carried out when the GBS ceases to be a leading mechanism. The latter may occur for geometric

reasons (strong thinning of the sample, as a result of which there is no possibility of intergranular sliding in the volume of the sample material), as well as when the deformation rate exceeds the range required for superplastic deformation (due to the peculiarities of the experiment itself, leading to a constant decrease in the deformation rate), as a result of which the consistency of the interaction of mechanisms (*i.e.*, the GBS weakens, the IDS increases and the activation of dynamic recrystallization is possible).

The structural SP regime can be rather long, so, as a result, good SP indicators (large elongations) can be obtained; if the SP regime is implemented for a short time; then, high elongation rates may not be achieved, however, in this case, the material may experience superplastic properties.

The mechanisms of high-speed deformation are not fully established. High-speed deformation is possible in several aluminium alloys with an initial noncrystallized structure before the SPD. Wang et al. [128] proposed that the presence of a liquid phase works during high-speed deformation, which leads to large elongations in alloys because at low stresses the resulting porosity is 'healed' by the presence of a small amount of liquid during deformation. Authors of Ref. [129] also supported the idea that a small amount of liquid phase during superplastic deformation can properly enhance the superplasticity of aluminium alloys. Meanwhile, Johannes and Mishra [130] concluded that the evidence for the existence of liquid phases along grain boundaries is the presence of fibres in the structure after deformation. Moreover, Duan et al. [124] believe that the existence of fibres is indirect evidence of grain-boundary sliding.

In some works, the dominant mechanism of superplastic deformation in alloys with a fibrous structure is discussed [81], but experimental studies give contradictory results.

Recently, a new high-strength superplastic alloy of the Al–Zn–Mg system containing large Al<sub>3</sub>Ni [131] particles has been developed [70]. The alloy has a partially noncrystallized structure before the onset of superplastic deformation, and a fine grain is formed as a result of dynamic recrystallization during superplastic deformation. As a result, the alloy sheets, after simple thermomechanical processing, are capable of superplastic deformation at a high rate (up to  $10^{-1}$  s<sup>-1</sup>). The mechanisms of high-speed superplastic deformation in this alloy have not been studied.

### 5. Shaping under Conditions of Superplasticity

One of the promising directions in the practical implementation of the superplasticity effect is associated with volumetric isothermal shaping.

The basic principle of isothermal deformation is that the work piece and the tool are heated to the same temperature, corresponding to the optimal shaping mode. In other words, the advantages of isothermal deformation consist in the possibility of reducing the deformation force due to wide regulation of temperature and speed modes of processing, ensuring a more uniform flow of metal with the highest plasticity, allowing for the implementation of precise volumetric shaping schemes and, consequently, to obtain a higher metal utilization factor, accuracy, etc., creating favourable conditions for the operation of the deforming tool (to increase its durability) and regulated changes in the structure and properties of the metal (the quality of products increases, the production culture improves and the technological discipline of labour increases).

The advantages of using the superplasticity effect in volumetric shaping include almost all the advantages of isothermal deformation, with the only difference being that an even more sizeable margin of plasticity and even lower deformation resistance make it possible to obtain more complex and precise stamped forgings, and pressed products with thin web, high ribs, etc. on less powerful equipment.

Since in most traditional metal forming processes, it is difficult to realize all the advantages of the superplastic flow of the processed metal, the creation of such specific superplastic deformation processes as filterless drawing and pneumatic or vacuum forming, which has several varieties and modifications, is of the noble interest. The disadvantage of such processes is the limited range of products: wire, rods, small profiles, and capillary tubes, as well as products of sheet stamping and stamping.

For the most detailed discussion of the problem of using superplasticity in volumetric technologies, we will draw on the results presented in Ref. [132].

In the practice of pressure treatment of most metals, processes using a volumetric stress state scheme with a predominance of compressive stresses (rolling, forging, pressing, etc.) are used.

Only a relatively small part of the processes uses schemes with a predominance of tensile stresses (sheet stamping, deep drawing, and drawing). Therefore, from the point of view of the practical application of the superplasticity effect, the possibility of achieving very high elongation rates is of less importance than the issue of a sharp decrease in deformation resistance. On the other hand, the possibility of obtaining significant deformation is vital, especially in the practice of processing low-plastic metals and alloys.

The methodology of metal pressure treatment based on the combination of deformation processes and the formation of little grains, according to Ref. [26], has not yet found sufficient application. Almost all technological advances in pressure treatment are related to structural (micrograin) superplasticity. This phenomenon is due to several theoretical and technological reasons, which should be discussed in more detail.

(i) The technological embodiment of superplasticity is usually limited by sheet stamping methods. In them, the implementation of the grain

boundary sliding mechanism, characteristic of superplasticity, is significantly facilitated compared to the processes of volumetric shaping. Note that the initial superplastic fine-grained semi-finished product is manufactured at metallurgical enterprises using a technology that is more complicated than usual. The processing of this semi-finished product to obtain an appropriate technical and economic effect due to the use of superplasticity takes place at a machine-building enterprise. This circumstance objectively hinders the growth of the production of superplastic semi-finished products.

(ii) The known model representations in the form of equations of state [133] correspond mainly to sheet stamping processes. They are based on the hypothesis of the existence of sigmoid-type dependence between the stress of plastic flow and the rate of deformation. As follows from Ref. [133], the constant parameters of the model are determined directly from the technological experiment.

(iii) It is believed that in combination with diffusion welding, superplastic sheet forming creates conditions for a qualitative leap in the production technology of monolithic large-size thin-walled structures of complex shape, which can replace heavier and less durable structures. At the same time, the material science tasks of improving the physical, mechanical, and operational properties of alloys in structures, products, and systems remain out of attention.

(iv) It is also stated that the advantages of using superplasticity for volumetric isothermal shaping processes are achieved due to the complexity of the technology due to the need to regulate the structure of the work piece, temperature, and deformation rate. At the same time, it is believed that the structure preparation by bringing it to fine-grained is not always necessary, when solving specific tasks. One can certainly agree with this opinion. Indeed, the formation of little grains should be carried out under the combined influence of heating and deformation. Nevertheless, most importantly, it should be implemented in the final product or semifinished product with an optimal combination of energy, kinematic, and thermal parameters of the process.

The mathematical formulation and rigorous solution of the problems of volumetric shaping of metals and alloys are fraught with serious difficulties. The classical definition of the metal forming process implies giving the metal the required shape with the achievement of the required level of physical and mechanical parameters. It should be noted that the principal attention was usually focused on the first part of the process. The formation of optimal structural parameters (in the equilibrium state [134–137]) corresponding to the complex of required physical and mechanical characteristics with a definite (stoichiometric or nonstoichiometric [138–142]) chemical composition was given secondary importance and is currently being completed by mechanical grinding of the cast structure, and the transition to a rational microstructure and reducing the grain size to the little level remains for final heat treatment [60, 143].

## 6. Conclusions

An important and urgent task being solved at the junction of modern materials science, solid-state physics, and metal pressure treatment is to increase the service and functional properties of industrial structural metal materials by optimizing/changing their composition, as well as, increasing the efficiency of their production and processing. It can be seen from the review that, to ensure widespread industrial use of the phenomenon of superplasticity, it is necessary to look for easier ways to transfer used and promising metals and alloys to this state, based on the use of the phenomenon of dynamic superplasticity in the broadest sense of the word, *i.e.*, in the process of deformation of the initial work piece with a conventional structure. One of these ways is to find the temperature-velocity parameters of the superplastic state of the material in the field of intensive development of the dynamic recrystallization process.

The advantages of superplastic deformation of metals include:

• reduction of deforming forces by 2-10 times, which makes it possible to use equipment of lower power, saving production space and the number of maintenance personnel, simplicity of design, and low cost of stamps;

• a sharp increase (by 2-3 orders of magnitude) in the plasticity of the work pieces being processed, which ensures accurate filling of the stamp engravings, the possibility of obtaining products of a very complex shape without welding, and, as a result, an increase in the metal utilization factor;

• isotropy of properties, increased corrosion resistance and toughness, high surface quality of parts associated with the presence of fine equiaxed grain;

• the absence of impact loads on the dies and relatively low deformation rates of the work pieces, which lead to the use of cheap die materials and the possibility of robotization and realization of the process.

• The disadvantages of using the superplasticity effect include:

• low deformation rate necessary for the manifestation of the effect of superplastic flow (this speed is almost two orders of magnitude lower than the speeds used in metalworking by pressure);

• the need to maintain with a high degree of accuracy the specified temperature range of deformation and, consequently, the heating of the tool, which requires the use of heat-resistant materials;

• the need to use new technological equipment and special equipment;

• the need either to obtain the initial fine-grained structure or to create conditions for grinding grain during deformation.

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#### НАДПЛАСТИЧНІСТЬ МЕТАЛІВ У СУЧАСНІЙ ТЕХНІЦІ ТА ТЕХНОЛОГІЇ

На цей час великий інтерес становлять дослідження структурної надпластичності, оскільки використання цього режиму в технологіях оброблення металів тиском уможливлює за одну операцію одержувати деталі різної форми (з високою точністю повторення навіть дуже складної форми); водночас потрібні менші енергетичні та матеріальні ресурси (відносно малі тиски та зношування інструментів) порівняно з деформуванням у режимі «звичайної пластичності». Іншими перевагами використання структурної надпластичності є поліпшені фізико-механічні характеристики готового виробу: ліпша якість поверхні після деформування, високі показники пластичності за підвищених температур, підвищена міцність за близьких до кімнатної температур без зниження пластичності (найчастіше відбувається зростання показників пластичності), підвищена циклічна міцність, твердість, опір ударній в'язкості, підвищена корозійна стійкість, а також відсутність анізотропії властивостей після надпластичного деформування.

Ключові слова: надпластичність, надпластична деформація, механічні характеристики, корозійна стійкість, межі зерен, дислокації, дифузійна повзучість.