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"PROPERTIES AND PROCESSING OF NANOCRYSTALLINE MATERIALS"

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GENERAL INTRODUCTION

The present Report completes the investigations in the frame of the project for the first year. It is important to estimate our achievements in the investigation of properties of nanocrystalline materials obtained by severe plastic deformation and their production. We think that the main results obtained can be summarized as follows:

1. We performed an improvement of the die-set for equal channel (ECA) pressing and torsion under high pressure with the aim to increase dimensions of the samples produced and to conduct processing of low ductile materials.

2. It was established that in pure metals severe plastic deformation led to the formation of an ultra fine-grained structure with a mean grain size of 100-200 nm, while in alloys due to severe plastic deformation and/or special methods of treatment (a decrease in the temperature of deformation, an increase of the pressure applied etc.) the grain size could be decreased down to a few tens of nanometers.

3. Samples produced by severe plastic deformation are characterized not only by the presence of ultra fine grains but also by a specific defect structure (internal stresses, density and distribution of dislocations, crystallographic texture etc.). A change in phase composition is also observed in UFG alloys.

4. UFG materials display a number of fairly attractive properties (high strength, ductility, superplasticity, etc.). The origin of these properties is associated with both the fine grain size and the other structural features of the UFG materials.

5. Rather interesting application areas and perspectives of UFG materials have been outlined. In our researches we focused on the application of a high strength state of titanium and its alloys for production of articles used in medical engineering and on the application of superplasticity for manufacturing of articles of complex shapes.

At the same time, the results of the investigations conducted indicate on the importance of further studies and the following questions are to be distinguished here:

- effective production of bulk samples and semi-products with an UFG structure for which an application of combined upsetting and torsion on a special die set seems to be most reasonable.

- quantitative description of the structure of samples and measurements of all basic parameters characterizing the UFG state, namely, the grain size distribution, the density of defects, the level of internal stresses and crystallographic texture. The solution of this task requires the use of different diffraction methods of analysis, in particular, X-ray, neutron scattering and electron diffraction methods.

- the development of structural engineering of UFG materials aimed to establish the processing guidelines for ultra fine-grained materials in order to enhance their properties by means of optimization of the structure and fabrication conditions.

- profitable commercial application of advanced properties of UFG materials.

We hope that the solution of all these tasks will be continued in the frame of the present Project.

PART I FUNDAMENTALS

In [1] we presented the results of investigations of the structure and mechanical behavior of pure metals and commercial alloys in the ultrafine grained (UFG) state. There we studied the histograms of grain size distribution in pure nickel, changes of lattice parameter and the value of static displacements of atoms from their equilibrium positions in lattice sites in pure copper, analysed the density of extrinsic grain boundary dislocations in UFG metals, investigated the structure and thermal stability of commercial alloy U12, the structure of intermetallic TiAl and considered the influence of elastic stresses on the mechanical behavior of pure copper. It was established that the structure of UFG pure metals was characterized by a lognormal distribution of grain sizes, high level of internal stresses and a specific defect structure which significantly influenced the mechanical behavior of samples. The investigation of commercial steel U12 showed that the samples prepared by torsion deformation were characterized by mean grain size of about 20 nm and high strength.

In IV quarter we have continued the structural characterization of pure metals and started a systematization of structural studies of commercial alloys. In this chapter we present the results of studies of the elastic strains distribution in UFG materials by means of TEM on an example of pure copper, new data of X-ray investigations of dislocation densities, stress fields and the mean grain size, data of structural studies of UFG iron produced by equal channel angular (ECA) pressing. Of a particular interest are the results of investigation of the structure and phase composition of UFG commercial alloys, demonstrated on the example of a titanium alloy VT22 and steel 20G4F. Studies of the structure of commercial alloys VT9, ShKh15 and 110G13 are in progress. Another important direction of this part is the investigation of mechanical behavior of pure UFG metals that is important from the view of understanding of the physical origin of high strength state. Defect structure of grain boundaries in deformed polycrystals have further been theoretically considered in order to work out a structural model of the grain boundaries in UFG materials.

1.1. Structural characterization

1.1.1. Pure metals

Study of stress fields by TEM. Details on the methods of determination of stress fields based on the study of dislocation line bow-out and bend extinction contours in TEM [2,3] were given in [1]. Below the results of a study of stress distribution in UFG pure copper are presented.

Detailed TEM investigations have shown that in UFG copper there exist several sources of long range internal stresses and correspondingly several types of distribution of internal stresses.

The first type of stresses are those arising between adjoining grains or groups of grains as a result of the intense plastic deformation. They lead to the appearance of bend extinction contours, expanding on the bodies of separate grains or grain groups. An example of such contours typical for this case is presented in Fig.1.1, where in grain A an elastic bending and in grain B an elasto-plastic bending is observed. The corresponding distribution of stress field is plotted in Fig.1.2 in terms of the change of lattice curvature χ along the bend contours. From this figure one can see, that on the boundary of grains A and B, where the elasto-plastic bending changes to elastic one, a four times increase of the lattice curvature is observed. Another example of elastic bending observed inside a separate grain is given in Fig.1.3. The corresponding graph of the curvature change from one grain boundary to the other is presented in Fig.1.4.

The elastic stress sources of second type are triple line disclinations. In this case the images of bending contours either commence from a triple line (Fig.1.5a), or are located on concentric circles around the junctions (Fig.1.5b). The change of lattice curvature with the distance from a triple junction depicted in Fig.1.6 for the case of a triple junction in Fig.1.5a shows that the characteristic feature of internal stresses of this type is the sharp decrease of the stresses with the distance from the junction.

The third type of internal stress sources are grain boundaries. In this case the extinction contours commence and end at the same grain boundary. An example of observation of such sources is given in Fig.1.7. Such stress fields are usually of pure elastic nature and have an almost constant value of curvature ($\chi = 3.3 \times 10^4 \text{ cm}^{-1}$).

To obtain an experimental relationship between the lattice curvature χ and the value of internal stress τ determined by dislocation bow out, instantaneous statistical measurements of χ and τ were carried out for UFG copper. The measurements have shown that the dependence between χ and τ is linear, but the experimentally measured coefficient between τ and χ is approximately 20-40 times less than the calculated one. This testifies on the presence of relaxation processes in UFG copper which screen the stress field sources and significantly decrease the level of elastic stresses.

X-ray studies of grain size, internal stresses and dislocation density. In this quarter X-ray studies of a series of pure metals with an UFG structure obtained by severe plastic deformation have also been carried out. In these studies for the determination of average crystallite size and internal stresses Warren-Averbach method [4] is used which is more accurate than the method employed in earlier studies.

In the previous X-ray study Scherrer and Wilson equation

$$\beta_{\tau} = \frac{\pi}{D_{hkl}} + \langle \epsilon_{hkl}^2 \rangle, \quad (1.1)$$

where β_{τ} is equal to $\beta_p \pi \cos \theta / \lambda$, β_{τ} is the integral broadening of the physical profile, scattering

Fig.1.1. An illustration of two neighbor grains in one of which there is an elastic (grain A) and in the other an elasto-plastic (grain B) bending

Fig.1.2. The change of lattice curvature on the boundary of grains A and B from Fig.1.1

Fig.1.3. An example of the elastic bending of a separate grain; the arrow indicates on the bend extinction contour

Fig.1.4. The change of lattice curvature from one boundary of the grain presented in Fig.1.3 to the other

Fig.1.5. Stress fields caused by disclinations at triple junctions; extinction contours are indicated by arrows

Fig.1.6. The change of disclination induced curvature with the distance from the triple junctions depicted in Fig.1.5; curve 1 corresponds to the junction in Fig.1.5a, curve 2 to the junction in Fig.1.5b

Fig.1.7. Stress fields from a grain boundary (bend contours are indicated by arrows)

vector $\tau = 4\pi \sin\theta/\lambda$, and D_{hkl} and $\langle \epsilon_{hkl}^2 \rangle^{1/2}$ represent the crystallite size and the root mean square (rms) strain averaged inside of the crystallites and in the grain boundary phase, was used to obtain the volume averaged crystallite size and the rms strain of the same UFG Cu sample.

It was supposed that the physical profile as well as the profiles describing size and distortion broadening effects could be described by a Lorentzian function. Thus, the values of the integral broadening of the physical β_p , size β_s , and distortion β_d profiles of the same Bragg reflection (hkl) were taken as additive ones.

$$\beta_p = \beta_s + \beta_d \quad (1.2)$$

This assumption makes the obtained (by the Scherrer and Wilson equation) data rather rough but we can use it to estimate the volume averaged crystallite size.

The volume average values of the crystallite size obtained by this procedure were equal to 95 nm and 88 nm for the directions perpendicular to the planes (111) and (200), respectively. In the present study a computer software was developed for a precise analysis of the X-ray data. Particularly, it became possible to calculate the crystallite size and internal elastic distortions by Warren-Averbach method. By the use of this method it has been revealed that the area averaged crystallite size of UFG copper is about 63 nm (111) and 35 nm (200). This means that they are smaller than the values obtained by Scherrer and Wilson equation.

The X-ray investigations of a similar UFG Cu sample [5] by the Warren-Averbach analysis revealed comparative crystallite sizes about 33 nm and 23 nm for the directions perpendicular to the planes (111) and (200). These values are smaller than the data obtained

in the present study. At the same time, the values of the internal elastic strains obtained in [5] were approximately twice larger than in this investigation. It was revealed [5], that the crystallite size calculated using Scherrer formula was close and even less compared to the data of the Warren-Averbach analysis.

The possibility to calculate the volume averaged crystallite size D_{hkl} from size integral broadening β_s obtained from the size coefficients $A_s(n)$ in frames of the Warren-Averbach method was realized by the equation [6]

$$D_{hkl} = \frac{\lambda}{\beta_s \cos \Theta} \quad (1.3)$$

It was found that the values of the volume averaged crystallite size obtained in this way are equal to 80 nm and 45 nm for the directions perpendicular to the planes (111) and (200), respectively. Thus, one can see that the values obtained using the Scherrer and Wilson equation are much closer to these values than to the area averaged ones. At the same time it should be pointed out that the approximation concerning the additivity of size and distortion broadening makes the method employing Scherrer and Wilson equation less reliable than the Warren-Averbach method.

The curves of dependencies of the internal elastic rms strain $\langle \varepsilon^2 \rangle^{1/2}$ averaged over the distance L_n perpendicular to (hkl) planes plotted in logarithmic and linear versions of the Warren-Averbach method possess maxima at small values of the distance L_n (Fig.1.8). At smaller values of L_n the internal elastic rms strain $\langle \varepsilon^2 \rangle^{1/2}$ decreases very rapidly and at larger values of L_n very slowly. A similar behaviour of these curves was observed before in [7].

Usually the step of the change of distance in the Warren-Averbach analysis is taken fairly large. Therefore, one can have simply missed some details of the graphs near small values of L_n . On the other hand, this decrease could be caused by some experimental errors in the determination of the background intensity. The decrease of the internal elastic rms strain $\langle \varepsilon^2 \rangle^{1/2}$ at large values of L_n can be caused by an inhomogeneity of the strain and a reduction of the contribution of smaller crystallites which may have larger values of strain [7].

It was found that the values of internal elastic rms strain $\langle \varepsilon^2 \rangle^{1/2}$ averaged to distances $L_n = 0.1 D_{hkl}$ (D_{hkl} is the crystallite size in direction (hkl)) are equal to $6.5 \times 10^{-2} \%$ and $13.8 \times 10^{-2} \%$ for the directions perpendicular to the scattering planes (111) and (200), respectively. These values are close to those obtained using Scherrer and Wilson equation which are equal to $4.8 \times 10^{-2} \%$ and $14.3 \times 10^{-2} \%$ respectively.

An analysis of the X-ray data obtained from a UFG copper sample processed by severe plastic deformation using uniaxial torsion straining under a pressure of a few GPa at room temperature showed that to obtain reliable values of the crystallite size Warren-

Fig.1.8. Elastic strain distribution in the direction (200) in UFG copper prepared by torsion straining

Averbach method should be used. Thus, a conclusion can be made that the Warren-Averbach method gives more reliable values of crystallite size than the method using Scherrer and Wilson equation.

On having established that the Warren-Averbach method provides a reliable information on the crystallite size and value of internal stresses, it has been applied to analyse a series of pure metals with an UFG structure obtained by severe plastic deformation.

The results of investigations have shown that in all the samples obtained by torsion straining an UFG structure with the crystallite size of a few tens of nanometers and high level of internal stresses forms (Table 1.1).

The investigated structural characteristics of copper and nickel both having a f.c.c. lattice are similar. Meanwhile, UFG iron having a b.c.c. lattice is characterized by the smallest crystallite size and highest internal stresses.

In the case of UFG copper one can see different influences of the character of intense deformation on the structure of material. Thus, internal stresses in samples prepared by ECA pressing are considerably higher than in those prepared by torsion under quasihydrostatic pressure for both the directions (111) and (200).

It is well known that the grain size determined by TEM methods is significantly larger than the size of crystallites (coherently scattering areas) determined by X-ray diffraction methods. Considering that in nanocrystalline materials each grain is a coherently scattering

Table 1.1

Crystallite size, root mean square (rms) strain and average dislocation density in pure metals with an UFG structure; QHP-torsion under quasihydrostatic pressure; ECA- equal channel angular pressing

Material	Preparation method	Crystallographic direction	Crystallite size, nm	Rms strain, $\times 0.01\%$	Average dislocation density, $\times 10^{14} \text{ m}^{-2}$
Cu	QHP	111	63	6.5	1.4
		200	35	14	5.4
Cu	ECA	111	64	18	3.8
		200	39	22	7.6
Ni	QHP	111	56	7.9	2.0
		200	41	11	3.7
Fe	QHP	110	25	28	15.6

crystallite, one should take into account that some areas near GBs are strongly distorted due to the presence of extrinsic grain boundary dislocations (EGBDs). Thus, X-ray diffraction allows to measure only the size of perfect parts of the grains.

Dislocation density is one of the most important structural characteristics of material. In UFG materials produced by severe plastic deformation, besides the lattice dislocations present inside grains, at certain conditions there are a large number of (EGBDs). Unfortunately, so far there is no reliable method for separate measurements of the densities of lattice dislocations and EGBDs by X-ray diffraction. Therefore, in the present study average density of lattice dislocations and EGBDs has been determined by the use of the following equation [7]:

$$\rho = \frac{2\sqrt{3} \langle \varepsilon^2 \rangle^{1/2}}{Db}, \quad (1.4)$$

where $\langle \varepsilon^2 \rangle^{1/2}$ is the rms strain, D the measured crystallite size and b the Burgers vector of dislocations.

The dislocation densities calculated by this equation are also presented in Table 1.1.

Thus, on the basis of experiments by the use of X-ray diffraction methods a structural characterization of a series of UFG metals has been performed which includes the determination of such structural characteristics as crystallite size, residual internal stresses and average dislocation density.

Structural characterization by TEM. In IV quarter we have also continued the structural characterization by TEM of pure metals subjected to severe plastic deformation.

Previous results of our investigations on a series of pure metals showed that the samples were characterized by fine grains, high internal stresses and a significant crystallographic texture. It was established that the samples of UFG iron had the finest granular structure and highest level of internal stresses. Below we present new results of TEM studies of the microstructure of bulk sample of iron subjected to ECA pressing.

Bright field TEM images show that UFG iron processed by ECA pressing has a mean grain size of 150 nm and mean dislocation density higher than 10^{15} m^{-2} (see Fig.1.9a). An important structural parameter of UFG materials is the density of EGBDs. In [1] dislocation densities in UFG metals have been theoretically analysed and it has been shown that the density of tangential dislocations at which microcrack formation at triple junctions can occur is of the order of $(1-3) \times 10^8 \text{ m}^{-1}$. A dark field image of UFG iron subjected to ECA pressing demonstrates an example of a grain boundary on which a density of normal EGBDs of the order of $8 \times 10^7 \text{ m}^{-1}$ is observed (Fig.1.9b).

Another feature of the TEM images of UFG Fe is the presence of bright-dark contrast between adjoining grains that indicates on a high-angle misorientation between the grains, and the presence of a large number of bend extinction contours near GBs and triple junctions that is characteristic of crystallites with high internal stresses. A diffraction contrast having thickness extinction contours is observed, as a rule, only on a few number of crystallites that is also caused by a high value of elastic stresses near GBs [8]. An electron diffraction pattern obtained in by microdiffraction from a foil area $0.5 \mu\text{m}^2$ consists of separated point spots located on circles. Such a pattern is typical for UFG metals with mean grain size of the order of 100 nm and have been observed in UFG Cu and Ni in a similar microdiffraction conditions.

1.1.2. Commercial Alloys

TEM studies have shown that after torsion straining the samples of steel 20G4F are characterized by equiaxed granular structure with very fine grains. The mean grain size observed in dark field images amounts about 20 nm (Fig.1.10). Such diffraction contrast features as thickness and bending extinction contours, lattice and grain boundary dislocations practically are not visible in both the bright and dark field images. Microdiffraction pattern taken from a foil area of $0.5 \mu\text{m}^2$ consists of separated spots located on circles.

Another commercial alloy, titanium based one VT22 has also been investigated in this

a

b

Fig. 1.9. Micrographs of the structure of iron subjected to ECA pressing: a- a bright field image, b- a dark field image; A grain boundary is indicated by an arrow; dislocations having a density $8 \times 10^7 \text{ m}^{-1}$ are seen in the boundary

a

b

Fig. 1.10. Micrographs of the structure of commercial steel 20G4F: a- a bright field image, b- diffraction pattern

α

β

Fig. 1.11. The microstructure of UFG titanium alloys subjected to torsion straining in dark field image: a- VT22 (β),

b- VT22 ($\alpha + \beta$)

a

b

Fig.1.12. The microstructure of UFG titanium alloys subjected to torsion straining followed by annealing at 400°C in dark field image: a- VT22 (β), b- VT22 ($\alpha+\beta$)

quarter. Initial samples were subjected to two kinds of thermal treatment before straining. The first set of samples were quenched from the temperature 950°C. After this treatment an X-ray analysis of the samples showed the presence of only β -phase. The other set was quenched from temperature 820°C that led to a structure containing α and β phases. Below the samples of these two sets will be denoted VT22 (β) and VT22 ($\alpha+\beta$), respectively. After torsion straining an X-ray analysis indicated on the precipitation of an α -like phase in VT22 (β), though the standard thermomechanical treatment of alloy VT22 (β) leading to the precipitation of α -like phases is ageing at 400°C. TEM images of UFG samples of VT22 (β) and VT22 ($\alpha+\beta$) show that they have a highly dispersed structure with mean grain size of 10 nm (Fig.1.11). Microdiffraction patterns from an area of 0.5 μm^2 consist of continuous circles which are the superposition of spots from α and β phases due to the coherency of these phases. Annealing at 400°C results in an increase of the mean grain size up to 20 nm (Fig.1.12) and in the appearance of additional rings of the α -phase in the diffraction pattern. Dark field images and diffraction patterns of VT22 (β) and VT22 ($\alpha+\beta$) samples annealed at 400°C are practically the same and may differ only by the volume fractions of α and β phases.

1.2. Modeling of the Grain Boundary Structure in Ultrafine Grained Materials

1.2.1. Introduction

In [1] it has been pointed out that in UFG materials prepared by severe plastic deformation three main types of nonequilibrium extrinsic grain boundary dislocation (EGBD) arrays can exist: 1) disordered networks of EGBDs having Burgers vector normal to the GB plane (sessile EGBDs); 2) excess density of sessile EGBDs described in terms of junction disclinations; and 3) arrays of EGBDs with Burgers vectors tangential to the GB plane. Earlier it has been shown that random EGBD arrays are stable in copper, nickel and iron polycrystals for sufficiently long time for the average dislocation densities up to $\rho_{\text{max}} = 10^8\text{-}10^9 \text{ m}^{-1}$ [9]. From the criterium of microcrack formation at a junction of grains, Rybin and co-workers obtained an estimate for the maximum strength of triple line disclinations, $\Omega_{\text{max}} = 1\text{-}2^\circ$ [10]. By the use of three criteria, namely, microcrack formation at and dislocation emission from a triple junction and stimulation of secondary slip in a grain, we found in [1] that the limiting density of tangential arrays of EGBDs might lie in between $\rho_{\text{max}}^{\parallel} \approx 8 \times 10^7\text{-}3 \times 10^8 \text{ m}^{-1}$ assuming their Burgers vector to be equal 0.1 nm (this is equivalent to the Burgers vector density $8 \times 10^{-3}\text{-}3 \times 10^{-2}$). In our previous works [11,12] the contributions of the disorder and disclination system to the root mean square elastic strain, excess energy and volume expansion in UFG materials were calculated. For a complete analysis of the applicability of dislocation-disclination modeling to the structure of GBs in UFG materials, one needs such calculations for the tangential EGBD arrays too.

The present paragraph aims to evaluate the elastic energy of tangential EGBD arrays in a heavily deformed material. First we consider a single square grain whose GBs contain

such arrays. Then the simplest statistics will be applied to estimate the average strain energy in a model two-dimensional polycrystal consisting of square grains. It will be shown that this model can predict the upper limit for the energy of tangential dislocation arrays in a deformed polycrystal. Finally, these results will be combined with the results on disordered EGBD arrays and disclinations to find the total excess energy caused by GB defects.

1.2.2. Energy of a Single Grain with Tangential EGBDs

Consider a square grain of size $d=2a$ the GBs of which contain EGBDs as shown in Fig. 1.13. Such a configuration of the tangential EGBDs is characteristic for a deformation induced GB defect structure and can form due to slip in grain on slip planes inclined to the GB planes. Let the total Burgers vectors accumulated on horizontal and vertical boundaries be equal to B_1 and B_2 , respectively. Assume the EGBDs to be distributed continuously. Then Burgers vector densities in each type of boundaries can be defined as $\rho_1=B_1/2a$ and $\rho_2=B_2/2a$. These are related to the linear densities of dislocations ρ'_1 and ρ'_2 by $\rho_1=\rho'_1 b_1$ and $\rho_2=\rho'_2 b_2$, where b_1 and b_2 are the Burgers vectors of single EGBDs on the horizontal and vertical GBs, respectively.

The total elastic energy per unit length along the z -axis associated with this system of dislocations can be calculated as the sum

$$W_{\text{tot}} = W_2(\rho_1) + W_2(\rho_2) + W_i(\rho_1, \rho_2) \quad (1.5)$$

of energies of pairs of opposite horizontal and vertical arrays, $W_2(\rho_1)$ and $W_2(\rho_2)$, and of the interaction energy of these pairs, $W_i(\rho_1, \rho_2)$.

The energy of a pair of opposite EGBD arrays, $W_2(\rho_i)$, is equal to the work of creating this pair, that is to the work done during moving the walls so that their spacing changes from 0 to $2a$. Consider the creating by this way of a pair of horizontal walls. The stress component σ_{xx} induced by wall 1 (Fig. 1.14) at any point (x, y) is given by [13]

$$\begin{aligned} \sigma_{xx}(x, y) &= -D\rho y \int_{-a}^a \frac{3(x-x')^2 + y^2}{[(x-x')^2 + y^2]^2} dx' = \\ &= D\rho y \left[\frac{x+a}{(x+a)^2 + y^2} - \frac{x-a}{(x-a)^2 + y^2} \right] - 2D\rho \left[\arctan \frac{x+a}{y} - \arctan \frac{x-a}{y} \right], \end{aligned} \quad (1.6)$$

where $D=G/2\pi(1-\nu)$, G and ν being the shear modulus and Poisson's ratio, respectively. For brevity, we omit the index 1 at ρ , unless we need to distinguish between the walls of two types.

The y component of force acting on dislocations belonging to an element dx of wall 2 is equal to $df_y = \sigma_{xx}(x, y)\rho dx$. The work done by interaction forces during the moving wall 2 from the distance y to a distance R is then given by

Fig.1.13. Tangential EGBD array induced by shear deformation in a square grain

$$A(y, R) = \int_{-a}^a dx \int_y^R \rho \sigma_{xx}(x, y) dy = 2D\rho^2 \left[\frac{3R^2 - 4a^2}{4} \ln(4a^2 + R^2) - \frac{3}{4} R^2 \ln R^2 + \dots \right. \\ \left. + \frac{4a^2 - 3y^2}{4} \ln(4a^2 + R^2) + \frac{3y^2}{4} \ln y^2 + 4ay \arctan \frac{2a}{y} - 4aR \arctan \frac{2a}{R} \right]. \quad (1.7)$$

Using (1.3) one can find the self energies of each single wall equal to $W_1(\rho) = -\frac{1}{2} A(y=0, R=R_s)$ (R_s is the characteristic size of sample) and the total energy of pair of walls $W_2(\rho) = -\frac{1}{2} A(y=0, R=2a)$. Thus, one has

$$W_1(\rho) = \frac{G(2a\rho)^2}{4\pi(1-\nu)} \left[\ln \frac{R}{2a} + \frac{5}{2} \right] \quad (1.8)$$

and

$$W_2(\rho) = \frac{G(2a\rho)^2}{2\pi(1-\nu)} (\pi - \ln 2). \quad (1.9)$$

To calculate the interaction energy of the pairs of horizontal and vertical walls, consider the work done during moving a vertical wall from its position in the grain to a distance of the order of $x=R$ in the stress field of the pair of horizontal walls (Fig.1.15). The force acting on an element dy of the vertical wall along x -axis is equal to $df_x = -\sigma_{yy}\rho_2 dy$, where σ_{yy} is the stress component induced by the pair of horizontal walls. Substituting of the expression for σ_{yy} leads to the following equation for f_x :

Fig.1.14. To the calculation of elastic energy of a pair of opposite walls

$$f_x = D\rho_1\rho_2 \left\{ (x+a) \ln[(x+a)^2 + 4a^2] - (x+a) \ln(x+a)^2 - \right. \\ \left. - (x-a) \ln[(x-a)^2 + 4a^2] + (x-a) \ln(x-a)^2 \right\}. \quad (1.10)$$

Integrating Eqn.(1.6) over x from a to R for $R \gg a$ yields the following simple equation for the interaction energy of a single vertical wall with the pair of horizontal walls:

$$W_i^\perp = -D\rho_1\rho_2(2a)^2 \ln 2. \quad (1.11)$$

Since the interaction energies of both vertical walls with the pair of horizontal walls are equal, the energy given by Eqn.(1.7) is doubled in the total energy of EGBD ensemble under consideration, that is $W_i(\rho_1\rho_2) = 2W_i^\perp$. Hence, one finally has for the energy of EGBD ensemble depicted in Fig.1.13

$$W_{\text{tot}} = \frac{(2a)^2 G}{2\pi(1-\nu)} \left[\pi(\rho_1^2 + \rho_2^2) - \ln 2 (\rho_1 + \rho_2)^2 \right]. \quad (1.12)$$

An analysis of Eqn.(1.8) shows that W_{tot} has minimum value, when the densities of EGBDs on horizontal and vertical GBs are equal:

$$W_{\text{tot}}^{\min}(\rho_1 = \rho_2 = \rho) = \frac{(2a\rho)^2 G}{\pi(1-\nu)} [\pi - 2 \ln 2]. \quad (1.13)$$

1.2.3. Energy of an Ensemble of Tangential EGBDs in a Polycrystal

In each grain of a deformed polycrystal there exists a tangential array of EGBDs similar to that presented in Fig.1.13. For simplicity, we will suggest that the densities of tangential EGBDs formed on horizontal and vertical boundaries of grain i are the same and equal to ρ_i . The density of EGBDs come to the GBs from each grain depends on strain of the

Fig.1.15. To the calculation of interaction energy of orthogonal EGBD arrays

grain and, therefore, on its orientation with respect to the applied stress. Hence, there is always a distribution of EGBD densities ρ_i come from different grains. Since dislocation ensembles having opposite signs of dislocations are equally probable for a non-textured material, we suggest that the average EGBD density corresponding to this distribution is equal to zero: $\langle \rho \rangle = 0$. Denote the variance of this distribution $\langle \rho^2 \rangle$.

The EGBD density on a GB connecting two neighbor grains i and $i+1$ is equal to $(\Delta\rho)_{i,i+1} = \rho_{i+1} - \rho_i$. Further one can assume that ρ_i are random variables independent of each other. Then the variance of total EGBD densities are equal to the sum $\langle (\Delta\rho)_{i,i+1}^2 \rangle = \langle \rho_{i+1}^2 \rangle + \langle \rho_i^2 \rangle = 2 \langle \rho^2 \rangle$.

Estimates made for the limiting density of tangential EGBD arrays [1] concerned the total density $(\Delta\rho)_{i,i+1}$. Therefore, the root mean square EGBD Burgers vector density, $[(\Delta\rho)^2]^{1/2}$, may have values in an interval $(1-3) \times 10^{-2}$.

The total energy of the dislocations in the whole polycrystal consists of the self energies of constituting elements (square arrays like in Fig.1.13) and the interaction energy of these elements. For the independent EGBD densities ρ_i the interaction energy averaged over the polycrystal is equal to zero, since it is proportional to $\langle \rho_i \rho_j \rangle = \langle \rho_i \rangle \langle \rho_j \rangle \equiv 0$. Hence, the total elastic energy due to tangential EGBDs in this case is simply the sum of self energies of single square EGBD arrays. Per one grain, this is equal to the average energy of one square array, which, according to Eqn.(1.9), is written as follows:

$$\langle W \rangle = \frac{G(2a)^2 \langle \rho^2 \rangle}{\pi(1-\nu)} (\pi - 2 \ln 2) = \frac{G(2a)^2 \langle (\Delta\rho)^2 \rangle}{2\pi(1-\nu)} (\pi - 2 \ln 2). \quad (1.14)$$

In real polycrystals EGBD densities ρ_i are not independent, since the strains of adjacent grains are correlated. Hence, an interaction term appears in the elastic energy which usually tends to decrease the energy given by Eqn.(1.10). Thus, Eqn.(1.10) can be used to

estimate the upper limit of the contribution of tangential EGBDs to the stored energy of a polycrystal.

It should be noted that the assumptions on the distribution of parameters of tangential EGBD arrays made here are the same as those used during the study of junction disclination ensembles [12].

1.2.4. A Comparison of the Contributions of Three Components to the Elastic Energy

Now we can summarize the results obtained for the elastic energies of three components of nonequilibrium, that is the disorder, disclinations and tangential EGBD arrays. For the simplicity of comparing with the data of differential scanning calorimetry experiments, expressions for the energy per unit mass will be given, and energy for copper nanocrystals with grain size $d=100$ nm will be calculated ($G=4.5 \times 10^4$ MPa, mass density $P=9 \times 10^6$ g/m³).

i) Disorder effect [11]:

$$E_{dr} = \frac{Gb^2\rho_0}{2\pi(1-\nu)dP} \ln \frac{d}{b}, \quad (1.15)$$

where ρ_0 is the average linear density of EGBDs. Earlier we have shown that in copper disordered EGBD arrays with densities up to 10^8 m⁻¹ are stable at room temperature [9]. Then for dissociated EGBDs ($b=0.1$ nm) $E_{dr} \approx 0.07$ J/g and for non-dissociated dislocations having the lattice Burgers vector magnitude $b=0.25$ nm $E_{dr} \approx 0.44$ J/g.

ii) Disclination quadrupole lattice [12]:

$$E_q = \frac{G \langle \Omega^2 \rangle \ln 2}{8\pi(1-\nu)P}. \quad (1.16)$$

Taking $\langle \Omega^2 \rangle^{1/2} \approx 3 \times 10^{-2}$, that is the root mean square disclination strength approximately equal to 2° , one finds $E_q \approx 0.2$ J/g.

iii) Tangential EGBD arrays:

$$E_t = \frac{G \langle (\Delta\rho)^2 \rangle (\pi - 2 \ln 2)}{2\pi(1-\nu)P}. \quad (1.17)$$

If $\langle (\Delta\rho)^2 \rangle^{1/2} \approx (1-3) \times 10^{-2}$, then $E_t \approx 0.2-2.0$ J/g.

The present estimates show that all the three components of nonequilibrium GB structure can make comparable contributions to the energy of UFG materials. Partial components are very sensitive to the parameters of defect structure, that is to the Burgers vectors of EGBDs, densities of EGBDs and the strengths of disclinations. These characteristics depend on many factors: temperature, grain size, geometry of GBs etc. and for UFG materials can vary as a function of deformation route, ageing etc. For reasonable values

of these parameters the total effect of the three components can account for the experimentally measured stored energy in UFG copper which is of the order of 1 J/g [14].

1.2.5. Summary

Thus, we have calculated the elastic energy due to tangential EGBD arrays accumulated on GBs during plastic deformation and compared it to the energy of other components of nonequilibrium GB structure which are believed to exist in UFG materials produced by severe plastic deformation: disordered EGBD networks and junction disclinations. The analysis has shown that all the three components may have comparable contributions to the stored energy in these materials. It seems that these contributions can account for the experimentally observed energies of UFG materials, thus giving a support to the validity of dislocation-disclination modeling of the GB structure in UFG materials.

A possible further development of the model is an interpretation of the annealing behavior of UFG materials in terms of the relaxation of nonequilibrium dislocation ensembles in GBs. Considering the relaxation kinetics and characteristic relaxation times for the three types of dislocation arrays and comparing them with the kinetics of recovery of macroscopic properties of UFG metals, one will be able to decide further on the comparative roles of these components in the structure of UFG materials. As we have already studied the relaxation of random EGBD arrays [9], in the next quarters we will focus on the study of relaxation in the ensembles of disclinations and tangential EGBDs.

1.3. Mechanical behavior of iron subjected to ECA pressing

The samples of UFG iron were subjected to tensile, compression tests and Vicker's hardness measurements with a load 300 N. The samples for tests were cut by spark machining. The surfaces of cut samples were polished to eliminate melted and softened layer to a depth of 50 μm . The sizes of samples were controlled by a micrometer with an accuracy of 0.01 mm.

Compression tests of samples sized 4x4x4 mm³ were conducted between dies of hardness 64 HRC on a testing machine IR5057. The load was detected by a tensometre with the maximum load 5x10⁴ H. The strain rate was kept equal to 5x10⁻⁴ s⁻¹.

Tensile tests were carried out on 2x1.5x7 mm³ sized samples schematically represented in [9]. The load was detected by a tensometre having a maximum load 5,000 H, and the strain rate was 1x10⁻³ s⁻¹.

The intervals of structural transformations were found by an analysis of DSC curves obtained at heating rates 5 and 200 deg/min (Fig.1.16). The change of heating rate did not affect the temperature interval of structural transformations. Two peaks have been found, one in interval 250-325°C and the other in between 400-600°C. According to this, the samples for compression tests were annealed for one hour at 200, 300, 400, 500, 650°C and for 2 hours at 500°C. The samples for tensile tests were annealed at 200 and 300°C for one hour.

Fig.1.16. A DSC curve of UFG iron

The results of compression tests are presented in Fig.1.17. One can see from the figure that the annealing at 200°C only slightly influences the strain hardening rate but decreases the yield stress of material. After the first peak of DSC curve (300°C) a decrease of the yield stress occurs at a certain increase of the strain hardening rate. Annealing at 400°C (starting point of the second peak) evidently caused the beginning of recrystallization, since a yield plateau on the deformation curve appears. The increase of annealing temperature to 500°C leads to a further decrease of yield stress and more pronounced yield plateau. The increase of annealing time at this temperature results in the appearance of a yield point practically with no changes of the strength. The annealing at 650°C, evidently, leads to a grain growth and, as a consequence, the decrease of strength. Metallographically, in this sample a granular structure with mean grain size of 2-3 μm is observed.

The results of tensile tests (Fig.1.18) are in good agreement with the above data. One can see that low temperature annealing around the first peak of DSC curve results in a decrease of yield stress at an increase of tensile strength and strain hardening rate.

A specific feature of the deformation behavior of UFG Fe prepared by ECA pressing is the increase of ultimate strength during tensile tests of samples annealed at 200 and 300°C at unaltered values of microhardness that may be related to the rearrangement of lattice and grain boundary dislocations during low temperature annealing. This point requires further detailed structural studies.

Fig. 1.17. Stress-strain curves during compression tests of UFG iron

Fig. 1.18. Stress-strain curves during tensile tests of UFG iron

1.4. Conclusions

1. On the basis of TEM studies of bending extinction contours in UFG copper it has been established that there exist different distributions of stress fields in dependence on the types of stress sources (triple junctions, grain boundaries and stresses between grains) and on a character of bending (elastic or elasto-plastic).
2. By the use of X-ray diffraction technique a structural characterization has been carried out for a series of pure UFG metals produced by torsion straining. Such structural characteristics as mean crystallite size, residual internal stresses and average dislocation density have been measured.
3. Detailed characterization of the structure and phase composition has been fulfilled for several commercial titanium and iron based alloys. It has been shown that in commercial alloys severe plastic deformation results in the formation of considerably finer grains and to the change of phase composition as well.
4. Elastic energy of UFG materials due to an ensemble of tangential (gliding) EGBD components has been calculated for a two dimensional model polycrystal and compared to the energy caused by other components of nonequilibrium grain boundary structure. It has been demonstrated that disordered EGBD arrays, junction disclinations and tangential EGBD arrays have comparable contributions to the grain boundary energy the sum of which can account for the excess energy observed in UFG materials.
5. Deformation behavior of UFG iron prepared by ECA pressing has been studied during tension and compression tests and the dependence of strength characteristics on the annealing temperature has been established.

1.5. Plans for the next quarter

1. To complete the set of programs for X-ray studies and finish the detail X-ray structural characterization of UFG copper prepared by severe plastic deformation (grain size, internal stresses, atomic displacements and texture).
2. Study of the grain growth kinetics in UFG copper having different structural parameters (internal stresses, dislocation densities etc.).
3. A structural characterization of superplastic alloys.
4. A structural characterization of commercial UFG alloys produced by ECA pressing.
5. TEM and X-ray studies of germanium and silicon subjected to torsion straining under pressure.
6. A theoretical study of the kinetics of grain boundary recovery due to the relaxation of triple line disclinations and application to UFG materials.

PART II ADVANCED PROPERTIES OF UFG MATERIALS

2.1. Introduction.

Investigations of advanced properties of UFG metallic materials processed by severe plastic deformation such as the high strength state, high ductility, superplasticity and magnetic properties were continued in the 4th quarter. The main attention was focused on commercial titanium alloys and some steels in which severe plastic deformation resulted in the formation of a granular structure with mean grain size of less than 100 nm and also in some considerable changes of phase composition (for details, see Part III). Studies of both the deformed state and states after annealing at different temperatures allowed to determine the role of different structural parameters in the origin of the properties of UFG materials.

Also we have worked out a program of further investigations of the wear resistance and fatigue strength of UFG metals and started to study these properties.

2.2. High Strength State.

Pure titanium (VT1-0, 99.99%), commercial high concentrated alloy VT22 (Al-5%, Mo-5%, V-1%, Fe-1%, Si-1%), steels ShKh15, G13 and steel 20G4F were chosen as the objects of investigations. As noted in [1], the UFG state in these materials was produced by the use of severe plastic deformation in torsion under high pressure. Methodical aspects of the measurements of strength characteristics for these materials were also reported in detail in [9].

2.2.1. *Titanium and its alloys.*

VT1-0. The results of the measurements of microhardness H_v , yield stress $\sigma_{0.2}$ and ultimate strength σ_s during bending tests and TEM data on the grain size d as a function of annealing temperature for titanium samples processed by severe plastic deformation are given in Fig.2.1. It is seen that a noticeable grain growth starts at a temperature above 300°C. However, already below this temperature a change of strength characteristics (H_v , $\sigma_{0.2}$, σ_s) is observed. Some features are to be noted here. First of all, some increase of microhardness and bending strength occurs after annealing at 250°C, but at the same time the yield stress decreases considerably. The increase of microhardness after low temperature annealing has already been mentioned in investigations of a number of ultra fine-grained (nano- [15] and submicrocrystalline [16]) materials. At the same time, a decrease of yield stress was revealed in UFG copper after annealing of the samples prior to the grain growth onset. The structural investigations testify that in UFG materials at low temperature annealing prior to the grain growth onset a structure recovery occurs. This recovery is attributed to the rearrangement and annihilation of extrinsic grain boundary dislocations. From this point of view the revealed changes of strength properties are caused by the influence of the defect structure of grain boundaries on mechanical behaviour of UFG materials. On the basis of the data

Fig. 2.1. The effect of annealing temperature on the mechanical properties and mean grain size of UFG titanium; 1- the grain size, 2- H_v , 3- $\sigma_{0.2}$, 4- σ_s .

given in Fig. 2.1 one can make two conclusions. Firstly, the established difference in the dependence of H_v and $\sigma_{0.2}$ on annealing temperature indicates that measurements of microhardness can be applied for study of the Hall-Petch relationship with some precaution, since this relationship is used for the yield strength dependence on grain size. Secondly, an increase of the difference between σ_s and $\sigma_{0.2}$ after annealing at 250°C as compared to that in the initial UFG state testifies to a considerable strain hardening of the samples and indicates on the change of the deformation mechanism. An analogous conclusion was made on the basis of investigations of the deformation behaviour of UFG copper conducted in the 3rd quarter [1].

VT22. A possibility of obtaining a high strength state due to thermo-mechanical processing was investigated in the alloy VT22. This processing includes pre-heat treatment, severe plastic deformation by torsion and final heat treatment. Considerable hardening of this alloy can be obtained during conventional rolling [17]. The aim of the present study was to determine the limit level of high strength state achievable in the alloy under consideration by severe plastic deformation and ageing. Two types of the alloy, $(\alpha+\beta)$ and (β) , obtained by means of different pre-heat treatments were subsequently subjected to severe plastic deformation. Microhardness was measured after ageing at different regimes within the temperature interval 200-550°C. The results are given in Table 2.1.

Immediately after torsion the value of hardness for the two-phase alloy is higher than

Table 2.1

Microhardness of titanium alloy VT22 after different treatments

No.	Phase composition	Treatment	Microhardness				
			Initial state	After annealing			
				200°C-2h.	450°C-2h.	550°C-2h.	650°C-2h.
1	β	i. Quenching from β -phase	3000	3100	4200	4500	4300
		ii. Quenching + deformation	4000	4000	5500	5450	5300
2	$\alpha + \beta$	i. Quenching from $\alpha + \alpha$ phase	3300	3300	4250	4300	4300
		ii. Quenching + deformation	4900	4900	5550	5550	5400

that for the one-phase one. Probably, this is caused by the fact that in addition to the martensite α' and α'' phases formed during severe plastic deformation and revealed by phase analysis, precipitations of the α -phase are also present in this alloy. Ageing of the alloy in both states increases the microhardness, but the effect of hardening is larger in the alloy in β -state. This can be explained by an additional precipitation of α -phase during ageing. At the same time, in the alloy, regardless of its initial structure, a percentage of phases close to an equilibrium one for the given alloy at the given temperature must be established. This is confirmed by equal values of microhardness for both alloys after ageing. One should note that the effect of hardening in the alloy VT-22 after heat treatment is higher than in pure titanium (VT1-0). This is evidently due to additional mechanisms attributed to phase transformations. With respect of obtaining a high strength state the optimal temperature of ageing in the alloy VT22 is 450-500°C.

2.2.2. Steels.

In steels G13 (C-1.18%, Mn-13.3%) ShKh-15 (C-1.05%, Cr-1.5%) and 20G4F (C-0.2%, Mn-4%, V-0.15%) the high strength state was obtained by a method similar to that

described for alloy VT-22. Having in the initial state austenite (G13) and dominantly ferrite (20G4F) structures and using severe plastic deformation, which also changes the phase composition, one can try to obtain an enhanced level of mechanical properties by subsequent ageing and tempering.

As noted in [1], the presence of microcracks in the samples did not allow to conduct bending tests. Thus, in Table 1.2 only the microhardness values of the above indicated steels in the as-deformed and annealed states are presented.

From the table one can see that heat treatment in the temperature interval 100-200°C after severe plastic deformation allows to increase the microhardness of steels ShKh15, G13 and 20G4F by 10-15% and 30%, respectively, as compared to the deformed state. As already mentioned, the maximum hardening effect is achieved in the steel 20G4F which has the least value of microhardness in the initial state [1]. This occurs due to both the plastic deformation and the heat treatment. Evidently, the hardening effects in steels are of a complex nature and require more comprehensive structural studies. Nevertheless, data on phase composition (Part 1) testify that one of the main reasons of hardening is the formation of metastable phases due to deformation (85% of martensite¹ in the steel G13 and carbon oversaturated ferrite in the steel 20G4F) and their decomposition during further heat treatment.

2.3. Superplasticity

In [1] it was shown that in UFG Al-3%Mg alloy with a structure of solid solution an elevated ductility could be achieved at relatively low testing temperatures as compared to an alloy with a coarse-grained structure. Investigations of superplasticity in UFG materials are of considerable importance, since this effect can be realized in these materials at lower temperatures (less than $0.4T_m$) or at higher strain rates that is very important for practical applications [18]. In this connection, we initiated investigations of the plastic behavior of typical superplastic alloys having an eutectoid (Zn-22%Al) and eutectic (Pb-62%Sn) composition. In these alloys a decrease of the grain size down to 100 nm can be achieved by severe plastic deformation.

Alloy Pb-62%Sn. In [19] more than 1000% total elongation to failure during tension at temperature 150°C was observed in Pb-62%Sn alloy with mean grain size of about 100 nm. It was interesting to estimate the same parameter in the alloy with even smaller grains and to study the nature and mechanisms of superplastic deformation in this alloy.

The aim of the first stage of investigations was to produce bulk samples by severe plastic deformation and to conduct preliminary tests of their state by measuring the microhardness. Disk shaped samples with diameter 200 mm and thickness 0.15 mm and rods

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¹⁾ During conventional cold working in this steel only 60% of martensite is formed

Table 2.2

Microhardness of the steels investigated

Material	State	Microhardness H_v , MPa	Phase Composition
G13	Initial	2500	Austenite
	As-deformed	9500	Martensite + austenite (15%)
	annealed at:		
	200°C-2h.	11000	Martensite + austenite
	300°C-2h.	10000	Martensite + austenite + (FeMn)3C
	450°C-2h.	6000	
	600°C-2h.	2500	Ferrite + Carbide
	700°C-2h.	2300	Ferrite + Carbide
20G4F	Initial	2700	Ferrite + Cementite (5%)
	As-deformed	6700	
	annealed at:		
	100°C-10h.	7900	
	100°C-20h.	8850	
	100°C-70h.	6500	
ShKh15	Initial		
	As-deformed	10500	
	annealed at:		
	200°C-2h.	11500	
	300°C-2h.	11000	
	400°C-2h.	6000	
	600°C-2h.	3000	
	700°C-2h.	2300	

having a diameter 20 mm and length 100 mm were produced of cast alloy by methods of torsion straining and equal channel angular pressing, respectively. To prevent the heat of samples during ECA pressing samples were cooled in liquid nitrogen. The results of measurements of microhardness and grain size in the initial cast state and in states deformed

Table 2.3

Microhardness and grain size of alloys Pb-62%Sn, Zn-22%Al.

Alloy	State	H_v , MPa	Load, g	Grain size, μm
Pb-Sn	Cast	124	10	5
	ECA-pressing	47	10	0.3
	Torsion straining	20	1	0.15
Zn-Al	Cast	700	50	2
	Torsion straining	340	50	0.10

by the both methods are given in Table 2.3.

From the data given in the table one can see that severe plastic deformation leads to a decrease of the grain size and a considerable reduction of the value of H_v in the alloy Pb-62%Sn. More than twice decrease of H_v is observed after ECA pressing and 6 times in the samples subjected to torsion. This strong softening effect observed in the samples of the alloy subjected to severe plastic deformation testifies to its transformation to a superplastic state already at room temperature.

At the next stage we are going to conduct tensile tests of the obtained samples at different temperatures and strain rates.

Alloy Zn-22%Al. In the initial state the eutectoid cast alloy had a diameter of 30 mm and a structure with grain size of 2 μm . Rods, 20mm in diameter and 100 mm in length, were cut from this ingot and subjected to ECA pressing at room temperature to the true logarithmic strain $\epsilon=4$. Due to severe plastic deformation the structure is refined and the grain size becomes about 200 nm. The data on microhardness from Table 2.3 show that severe plastic deformation leads to a considerable plasticification of the alloy. For example, a twice decrease of H_v occurs in the state after torsion when compared to the initial cast state.

The samples obtained by ECA pressing were cut into specimens which were tested by tension at different temperatures and strain rates. The results of preliminary tests are given in Table 2.4.

Elongation at temperature 473 K and strain rate $3.3 \times 10^{-1} \text{ s}^{-1}$ is considerably higher in the UFG alloy than in the same alloy with a grain size of 10 μm , where elongation to failure amounts 1150% in the same testing conditions [20]. This experimental fact proves that superplastic state is achieved in the UFG alloy at higher strain rates, this being rather attractive from a practical point of view.

Thus, the conducted tests show that the obtaining of UFG structure in Zn-22%Al and

Table 2.4

Elongation to failure (in %) at different testing conditions in the alloy Zn-22%Al subjected to ECA pressing

T, K	Strain rate, s ⁻¹			
	3.3x10 ⁻⁴	3.3x10 ⁻³	3.3x10 ⁻²	3.3x10 ⁻¹
373	450			
423	700	940		
473			1900	1540

Pb-62%Sn alloys considerably increases parameters of superplasticity. In the alloy Zn-22%Al an increase of elongation to failure was revealed at high strain rates $\dot{\epsilon} = 3.3 \times 10^{-1} \text{ s}^{-1}$. In the alloy Pb-62%Sn the superplastic state is achieved already at room temperature.

2.4. Fatigue.

Among a number of mechanical properties characterizing the material resistance to failure the resistance to fatigue failure is one of the most important ones, since it determines the durability of components under alternative stress conditions.

The fatigue failure can be especially dangerous, because it occurs under stresses which are much less than the yield stress and ultimate strength. Fatigue properties like other mechanical properties depend on a number of factors characterizing the material structure. For example, it is known that in steels at a given tensile strength grain size influences the fatigue resistance. Materials with finer grains have higher fatigue limit than materials with coarse grains [21]. However, in the literature there are almost no data on the fatigue behaviour of UFG materials. One of the first works on this subject is the paper by A.B.Witney, P.G.Sanders, J.R.Weertman and J.A.Eastman [22]. The paper deals with investigations of the structure stability and deformation mechanisms during repeated loading of nanocrystalline copper produced by condensation in inert gas and subsequent consolidation at the Argonne National Laboratory. However, because of the small dimensions of samples produced (3x2x0.2 mm) and their limited quantity it is difficult to make conclusions about the complex fatigue behaviour of nanocrystalline copper on the basis of these results.

In this connection, we plan to conduct low cyclic fatigue tests of bulk UFG copper samples produced by ECA pressing in the first quarter of 1996. At present we are dealing

with the preparation of these samples. The preparation includes pressing, machining, cutting and development of a testing procedure. We plan to use a hydraulic machine "SHENK" with a pulser for testing. The loading will be conducted by a scheme of tension. The main aim of the investigations is to determine fatigue limits and fatigue crack growth rates for smooth and notch samples with UFG and coarse-grained structure.

2.5. Enhanced ductility

In the previous report we noted that the considerable decrease of the temperature of creep acceleration effect can be connected with an increase of grain boundary diffusion coefficients (D_b) in submicrocrystalline materials. The measurements of D_b values for copper diffusion in submicrocrystalline nickel at 423 and 573 K showed that the diffusion coefficients exceeded the corresponding values for coarse-grained nickel by 5-6 orders of magnitude. It was assumed that this phenomenon was associated with a sharp reduction of the activation energy for grain boundary diffusion in submicrocrystalline materials. To check this assumption, we conducted an additional measurement of D_b at 398 K. The result is $D_b = 4.7 \times 10^{-15} \text{ m}^2/\text{s}$.

On the basis of the data available the temperature dependence of diffusion coefficients of copper in UFG nickel was plotted and the activation energy Q_b was calculated from the slope of this dependence in logarithmic coordinates. From Fig.2.2 one finds $Q_b = 66.3 \text{ kJ/mol}$. The obtained value of Q_b is approximately twice lower than the activation energy for GB diffusion in coarse-grained nickel ($Q_b = 131 \text{ kJ/mol}$).

Thus, the state of grain boundaries in UFG materials significantly differs from the GB state in coarse-grained materials. This difference is as essential as is the difference between activation energies for volume and GB diffusions in coarse grained materials. On the basis of the data presented one can assume that grain boundary sliding provides a dominant contribution to the total deformation during creep of UFG nickel under conditions of grain boundary activation by diffusion flows of copper.

2.6. Wear Resistance

An effect of very high wear resistance characterized by the absence of any noticeable wear at the contacting surfaces of a series of materials at certain values of temperature-rate parameters of contact sliding is known for hardly loaded friction units [23,24].

The investigations of friction and wear of materials with different degrees of structure dispersity (at micro-, meso- and nanostructural levels) show a considerable importance of the structure in the formation of conditions providing abnormally low values of friction and wear parameters (friction coefficient of about 0.01, and linear intensity of wear 10^{-10} - 10^{-11}) [25-27].

For instance, the results of investigations of friction surfaces of materials "steel-copper alloy" in the conditions of high wear resistance show that the wear resistant structure

Fig.2.2. Temperature dependence of grain boundary diffusion coefficient of copper in UFG nickel.

consists of a finely dispersed layer (5-20 μm) with equiaxial grains sized from 0.2 to 0.5 μm covered by a very fine barrier layer of thickness 0.2-0.5 μm . This barrier layer provides a low friction coefficient due to the absence of microrelief on a friction surface and a carrier ability due to high hardness. This layer also protects materials of friction pairs from chemical and adhesion interaction with each other and the environment.

Some authors [28,29] believe that the formation of a layer with a submicron particle size determines the mechanism of wearless friction since the realization of an effect of superplasticity in a finely dispersed layer near the surface becomes possible.

This opinion is proved by the results of investigations of friction of metallic materials with as-prepared finely dispersed structure in the temperature-rate interval of superplasticity [30] which indicates on a considerable decrease of the absolute values of friction stress and wear parameters.

In this connection, the investigation of the role of ultra fine grained structure in the realization of high wear resistance presents a particular interest. Thus, we plan to solve the following tasks in the next quarter.

The first tests of wear resistance will be carried out on the basis of UFG Cu processed by ECA-pressing in the following structural states:

- I - as-prepared;
- II - after annealing (before grain growth);
- III- as-prepared + protective coating by Cr;
- IV - as-prepared + annealing + protective coating by Cr;

V - coarse-grained state;

VI - coarse-grained state + coating by Cr.

Such an approach will allow to evaluate the influence of both the grain size and the coating on copper wear resistance.

2.7. Magnetic Properties

It is known that a high coercitivity state in magnetic hard alloys of RFeB system (R is a rare-earth metal) can be obtained by using traditional methods of powder metallurgy [31], rapid quenching [32], methods of mechanical alloying [33] and hot deformation [34]. As a rule, all these methods are based on the formation of ultra fine grained structures which lead to a sharp increase in coercitivity.

In the present work to obtain a high coercitivity state in alloys RFeB severe plastic deformation method has been used. The goal of the work was to study the influence of torsion deformation and further heat treatment on the magnetic properties of alloy $\text{Pr}_{20}\text{Fe}_{73.5}\text{B}_5\text{Cu}_{1.5}$. The annealing was conducted in a furnace with vacuum 10^{-2} Pa within a temperature interval from 773 to 973 K. Samples pre-magnetized in an impulse field of $H=5600$ kA/m were used to measure hysteresis characteristics. These measurements were made by a vibromagnetometer in a magnetic field of 1600 kA/m. The curves of temperature - magnetization dependences were obtained on the vibromagnetometer in a field of 720 kA/m.

The magnetic parameters of samples subjected to severe plastic deformation by torsion considerably differ from initial values in cast alloy.

Fig.2.3 represents the dependence of coercitivity H_c on the angle of anvil rotation for the Pr-Fe-B-Cu alloy. Whereas in the initial cast state the coercitivity of the alloy is 20 kA/m, due to deformation the coercitivity increases with increasing the rotation angle and achieves maximum at $\phi=5\pi$. With a further increase of the number of rotations and, consequently, with an increase of the deformation degree the coercitivity drops to a value of 47 kA/m at $\phi=20\pi$.

The curves of hysteresis for the samples subjected to torsion at different ϕ and for heat treated samples are given in Fig.2.4. At strains corresponding to $\phi=5\pi$ and higher ones typical bends are observed on hysteresis loops. These bends indicate on the presence of hard magnetic and soft magnetic phases (Fig.2.4, curves 2,3), the volume fraction of the second phase increasing with the strain.

Annealing of the deformed sample in temperature interval 773-973 K for 0.5 h. sharply increases its coercitivity. For example, the coercitivity of the sample deformed to $\phi=5\pi$ (Fig.2.4, curve 5) increases from 350 to 1500 kA/m, residual induction B_r amounting 0.56 T. A bend on a hysteresis loop typical of samples after deformation is not observed in this case. This can be explained by the disappearance of a low coercitivity phase due to the annealing.

Fig.2.3. Dependence of coercivity jH_c on an angle of rotation ϕ for deformed samples

Fig.2.4. Hysteresis curves of samples deformed at different angles of rotation (1- $\phi=2\pi$, 2- $\phi=5\pi$, 3- $\phi=10\pi$, 4- $\phi=20\pi$) and heat treated (5- $\phi=5\pi$, $T=873$ K)

Investigation of the temperature dependence of magnetization for Pr-Fe-B-Cu alloy in the initial cast state shows the presence of two ferromagnetic phases with Curie temperatures 520 and 1070 K, which correspond to the Curie temperatures of $\text{Pr}_2\text{Fe}_{14}\text{B}$ and $\alpha\text{-Fe}$ phase, respectively. The temperature dependence of magnetization of the deformed

alloy are shown in Fig.2.5. During heating within the temperature interval from room temperature to 520 K (curve 1) the magnetization of the alloy subjected to torsion of $\phi = 10\pi$ decreases more sharply as compared to the magnetization of the cast alloy. Such a $\sigma(T)$ dependence of the severely deformed alloy may be connected with the destruction of magnetic exchange coupling in the phase $\text{Pr}_2\text{Fe}_{14}\text{B}$ due to a high density of lattice defects, disordering and partial amorphization of this compound. The $\sigma(T)$ curve during cooling for the deformed alloy after heating to 1070 K (Fig.2.5, curve 2) shows the disappearance of an interval of a slight decrease of magnetization in the range of temperatures 300-550 K.

Fig.2.5. Temperature dependence of the magnetization $\sigma(T)$ for the sample of Pr-Fe-B-Cu alloy deformed to $\phi = 10\pi$; 1- a heating curve, 2- a cooling curve

The initial alloy in the cast state had a structure with non-equiaxed grains of the basic $\text{Pr}_2\text{Fe}_{14}\text{B}$ phase, 14 μm in length and 3 μm in thick. TEM investigation of the alloy samples subjected to torsion straining show that during deformation the structure is strongly refined: the grain size decreases down to 0.1-0.3 μm , and inside the grains a high density of dislocations is observed.

The rise of coercivity of deformed samples of Pr-Fe-B-Cu alloy after heat treatment can be explained by the relaxation of internal stresses and the rearrangement of lattice and grain boundary dislocations. Such an effect of thermal treatment is similar to an effect observed in melt-spun Nd-Fe-B alloys [35].

Thus, it has been shown that severe plastic deformation under quasihydrostatic

pressure is an effective method for obtaining a high coercitivity state in the $\text{Pr}_{20}\text{Fe}_{73.5}\text{B}_5\text{Cu}_{1.5}$ alloy. After deformation and subsequent heat treatment in certain chosen regimes the coercitivity of the alloy reaches 1500 kA/m, that is not lower than the coercitivity of rapidly quenched magnets from this alloy. However, the samples obtained by the present way are bulk ones whereas the rapid quenching allows to produce only thin ribbons.

2.8. Conclusions

The results reported in the present part can be summarized as follows.

1. The dependence of such mechanical properties as microhardness, yield stress and ultimate strength on the annealing temperature has been studied in UFG commercial titanium produced by severe plastic deformation. It has been established that like to the behavior of UFG copper studied earlier H_v and $\sigma_{0.2}$ behave differently during annealing near the temperature of grain growth onset: H_v slightly increases, whereas $\sigma_{0.2}$ considerably decreases.
2. It has been established that in commercial titanium alloy VT22 and steels G13, ShKh15 and 20G4F the highest level of strength characteristics is achieved after intense plastic deformation and subsequent low temperature annealing that is caused by the occurrence of ageing processes.
3. In alloys Zn-22%Al and Pb-62%Sn after severe plastic deformation a significant softening is observed. Preliminary tests show that superplastic deformation occurs in Zn-22%Al at a strain rate approximately equal to 10^{-1} s^{-1} and in Pb-62%Sn at already room temperatures.
4. Severe plastic treatment allowed to achieve record values of coercitivity in magnetic alloys of R-Fe-B system.
5. Investigations of the perspective properties during fatigue and wear have been initiated.

PART III PROCESSING

While developing the processing routes of UFG materials by the use of severe plastic deformation we focused in the following directions:

1. Production of large samples of Zn-22at%Al alloy and Cu having a diameter of 20 mm and thickness 2 mm by torsion under high pressure.
2. Equal channel angular pressing of Cu at cryogenic temperatures.
3. Carrying out of experiments with the combined torsion and upsetting on a specialized rolling mill.
4. Development of new methods of UFG structure processing.

3.1. Torsion under High Pressure

This method has been developed to obtain bulk samples of Cu and Zn-22at%Al with a diameter of 20 mm and thickness 2 mm under high pressure. For this a special equipment for torsion straining having a lateral thrust has been designed. This equipment is schematically represented in Fig.3.1.

Fig.3.1. A die-set for torsion under pressure with a side support: 1- upper die; 2- bottom die; 3- specimen; 4- limiting rings

The measurements of microhardness of Cu samples subjected to torsion under pressure in this equipment showed that microhardness increased from 300 MPa in the initial state to 1100 MPa after deformation.

increases during the first three steps after what only a slight increase of load occurs with further increase of strain.

Fig.3.3. Strain diagrams of copper during ECA pressing; 1- for the pre-cooled sample deformed in cooled equipment; 2- deformation at room temperature

3.3. Experiments on Torsion with Upsetting on a Specialized Rolling Mill

In this quarter we planned to fabricate a special device for the treatment of low ductile materials by combined torsion and upsetting on a specialized rolling mill. The drawing of this device was given in Fig.3.6 of [1]. Unfortunately, because of financial reasons its manufacture has not been finished yet.

3.4. Development of New Methods of Processing of UFG Structure

One of new methods of obtaining an UFG structure which has been mastered this quarter is multilateral forging which consists in the repeated upsetting with the turn of samples. To use this method a special equipment has been fabricated. The equipment consists of an isothermal die-set with flat and V-like dies. A press with a load of 250 tons was used for forging.

Commercially pure titanium VT1-0 was used for investigations. The dependence of hardness on temperature of deformation for the alloy VT1-0 is given in Fig.3.4.

TEM studies of the structure of samples have shown that after deformation in the temperature interval 200-300°C a structure with grain size of about 300 nm was formed in VT1-0.

Fig.3.4. The dependence of microhardness of titanium VT1-0 on the temperature of deformation

3.5. Conclusions

1. Discs having a thickness 2 mm and a similar structure in the whole cross-section have been produced by torsion under pressure. Bulk samples of Cu and Zn-22at%Al alloy were used for this.

2. Taking Cu as an example it has been shown that the use of samples pre-cooled down to cryogenic temperatures and equipment cooled to -50°C leads to the formation of an UFG structure with a grain size of about 100 nm and a hardness of 1320 MPa, that is to better characteristics than those obtained by the deformation at room temperature.

3. The optimal regimes of processing by the use of multilateral forging have been determined for the production of bulk preforms having the diameter 15 mm, length 100 mm and grain size of 300 nm from commercially pure titanium VT1-0.

4. Works on fabrication of a die-set for the treatment by combined torsion and upsetting on a specialized rolling mill have been continued.

3.6. Plan for I Quarter, 1996

1. To produce sets of bulk semi-products with an UFG structure of titanium based alloys VT-9 and VT1-0 by manifold repeated upsetting.
2. To produce bulk semi-products (diameter 10mm and length 500 mm) with an UFG structure of low carbon steels by combination of torsion with upsetting on the specialized rolling mill.
3. To develop the method of ECA pressing and to obtain by this method an UFG structure in Al based alloys (system Al-Cu-Zr) and iron.

PART IV APPLICATIONS

The analysis of advanced properties of ultra fine-grained materials testifies that their application is possible in several directions:

1. As constructional materials for which of a very importance is the high level of a series of mechanical properties such as:

- short time properties during compression and tension;
- shock toughness and fatigue;
- damping parameters, hot strength, heat resistance and corrosion resistance.

2. As materials with a high technological ductility (superplasticity) for the development of forming processes.

3. As materials for electrical engineering, for example:

- hard magnetic and soft magnetic materials.

3.1. Application in Medicine

As it was noted earlier [1], the use of UFG titanium in medical engineering allows to increase the mechanical properties of articles by a factor of 1.5. The solution of this task includes two stages:

- production of bulk semi-products of titanium with UFG structure;
- manufacturing of articles and their testing.

In [1] we reported that we were going to produce orthopaedic articles of pure titanium.

As it was also noted in [1], one of the requirements for a successful application of materials in the medicine, in particular in orthopaedy, is the combination of high strength and biological inertness of the material. Commercially pure titanium VT1-0 meets these requirements. However, strength characteristics of the delivered titanium VT1-0 were rather low. In this connection, we carried out investigations to increase the mechanical properties of this material by means of strain-heat treatment at various temperatures and strains.

According to technical requirements, the articles of such a type should have the yield stress $\sigma_{0.2} > 800$ MPa. We hope that UFG structure will allow to achieve such a high yield stress. To solve this task, samples with different structural states were produced of pure titanium VT1-0 (see Part III). Their dimensions are the following: 100 mm in length, 20 mm in width and 5 mm in thick. This allows to determine a series of their mechanical properties for further optimization of the regime of thermo-mechanical treatment.

In the next quarter we plan to conduct mechanical tests of these samples, to produce articles of a "membrane" type and make their approbation.

3.2. Production of Complex Shape Semi-Products and Articles

Another direction of practical application of UFG materials is the production of articles of a complex shape. It is known that die forging is one of the most widely used

modes of forming of such articles. It is based on the principle that an ingot acquires the shape of a die. There are different types of forging and one of the most progressive ones is isothermic forging.

While comparing methods of forging and choosing the best one it should be taken into account that two different approaches to the process of forming of complex shaped articles are possible. The aim of the first one is to obtain an ingot of the given dimensions and shape. The least labour-consuming methods of plastic metal forming are usually applied in this case and at the final stage the article is subjected to heat treatment, the latter being a determining operation in obtaining of the service properties of the article. The aim of the second approach is not only to get the necessary configuration but also to obtain an optimal microstructure and mechanical properties. Since the requirements to mechanical properties of materials used in machine building and engine building increase, the processes applying to the second approach are being more widely developed.

However, the practical application of these processes is connected with the creation of stable explicitly specified conditions of deformation and the control of real thermomechanical parameters of the process. Though in a number of cases deformation under superplastic conditions may be referred to processes of the second group, during forging the superplastic deformation provides only a formation of a homogeneous micrograined structure and a set of mechanical properties corresponding to this structure.

One of the most promising methods of realizing the advantages of submicrocrystalline and nanocrystalline structures is the production of complex shaped preforms by forging in a superplastic regime.

Recently in a number of aluminium alloys it has been shown that in alloys with an UFG structure the optimal temperature of superplasticity decreases by 150-200°C [36]. It has been also shown that the strain rate of superplasticity can be increased by two orders of magnitude (see Part II).

The use of subsequent ageing of preforms with an UFG structure allows to increase the strength characteristics of aluminium alloys while maintaining the UFG structure [16]. In spite of a large number of investigations on SPD of aluminium alloys, their practical application for production of complex shaped articles was connected with technical difficulties and significant expenses needed for obtaining of large semi-products with homogeneous UFG structure and production of articles of these materials.

Thus, the production of large articles of a complex shape (like discs of motor-car wheels) consisting in forging of preforms with an UFG structure in the regime of low temperature superplasticity at high strain rates will not only decrease the cost of final products but also create prerequisites for development of technological processes and manufacture of a wide range of large articles of an intricate shape from titanium and aluminium based alloys.

Technical approach. In the technology suggested a decrease of deformation load and

an increase of the strain rate are achieved due to the combination of two phenomena, namely, transformation of the preform material to a submicrocrystalline state and the forming of preform in the regime of "low temperature" superplasticity. Therefore, the development of a technological process for manufacture of intricately shaped components requires the following research stages:

1. The determination of regimes of submicrocrystalline structure formation in bulk preforms for the particular material used for the production of a given component.
2. Establishing of the regimes of superplastic flow of the material. These regimes are revealed by laboratory tests in tension and compression of standard samples cut from this material.

Producing of car wheel discs by forging of ingots with an UFG structure in the regime of superplasticity allows to realize:

1. High quality and precise dimensions of the ingots that includes the maintenance of a uniform structure and required fibrous structure which provide a high level of mechanical properties.
2. The decrease of the load in a die-set by 1.5-2 times and more.
3. The decrease of the total labor expenses by a factor of 2 owing to the reduction of labor expenses for machining of discs and tooling and for the maintenance of die sets and other facilities.
4. The saving of metal by a factor more than 2 owing to the increase of the metal utilization factor and the decrease in consumption of materials.

The fabrication of intricately shaped large components of a disc type by casting, plastic working and machining requires much labour, material and power expenses.

In 1996 we plan to develop scientific and technological bases for the production of massive semi-products in the form of a forged wheel disc, 500 mm in diameter, for a motor car LADA. This task will be solved in some stages:

1. In the first stage (I and II quarters) we will conduct works on modification of the die-set and produce a preform, 200 mm in diameter, 250 mm in height, of the aluminium alloy Al-2.2wt. %Cu-0.6wt. %Mg-0.6wt. %Mn-0.9wt. %Si with an UFG structure.
2. In the second stage (III quarter) we will test the structure and a set of mechanical properties of the preforms produced.
3. In the third stage (IV quarter) we plan to fabricate a final preform-component of a disc in the regime of low temperature superplasticity.

3.3. Materials for Electrical Engineering: Permanent Magnets

One of the possible fields of the application of UFG materials is the production of hard magnetic materials with a high energetic capacity. As known, materials with the grain size less than the size of domains ($d = 1\mu\text{m}$ for Nd-Fe-B magnets, for instance) possess the maximum coercitivity H_c . Such a structure can be obtained by severe plastic deformation.

At present the most energetically capable magnets are produced from the alloys of R-Fe-B system by the use of methods of powder metallurgy, rapid quenching and hot deformation. Up to now severe plastic deformation has not been applied for production of a high coercivity state of R-Fe-B alloys.

The investigations conducted in the frame of the present Contract showed that due to severe deformation by torsion and further heat treatment a submicro-grained high coercivity state was formed in the alloy Pr-Fe-B-Cu. The first experiments showed that the coercivity H_c of Pr-Fe-B-Cu samples subjected to torsion and subsequent heat treatment achieved a value of 1600kA/m. This value is the highest one obtainable in R-Fe-B samples produced by other methods. We suppose that an optimization of severe plastic deformation and further heat treatment will allow to obtain record magnetic properties in magnets of R-Fe-B system.

The method of severe plastic deformation has some definite advantages as compared to rapid quenching and powder metallurgy. The latter, as known, are traditional methods of production of hard magnetic materials of R-Fe-B system. Severe plastic deformation allows to produce monolithic samples of R-Fe-B magnets having high coercivity and record parameters in the form of a disc, $d=20$ mm, $h=0.2$ mm, without using any complex multi-step operations of sintering, rapid quenching, high temperature pressing in vacuum. The development of an industrial severe plastic deformation technology of R-Fe-B magnets production will considerably reduce the price of magnets with high mechanical and magnetic properties of definite configurations, namely, discs, thin plates. Magnetic materials of such configurations with high magnetic properties will be used in microelectronics for production of small computers, microelectric engines and in other branches.

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