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Correlation of Plastic Forming and Structure Formation Parameters in Powder Materials

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Summary. A theoretical analysis of the kinetics of softening processes occurring in the powder porous body during deformation at high temperatures has been conducted. Constitutive equations relating the deformation parameters and structure formation parameters that are characterizing dynamic softening processes during deformation. The linear dependence of the axial stress logarithm and accumulated deformation of hard phase as a function of the reciprocal of the temperature. The activation energy of dynamic softening at uniaxial compression estimated. It has shown that at low deformation temperatures the softening mechanism is dynamic recovery and polygonization while dynamic recrystallization is taking place at elevated temperatures. Porosity decreases the activation energy of dynamic softening. The dynamic softening mechanism in powder material was confirmed by metallographic analysis.

Key words: mathematical model, recovery, polygonization, recrystallization, activation energy.

INTRODUCTION

A wide variety of working conditions of machine parts require the development of materials with special properties that ensure high wear resistance [1, 2]. This problem may be solved by implementation of new materials produced by a combination of heat treatment and plastic forming processes. The power and kinematic parameters of plastic deformation during hot and semi-hot deformation take an influence on structure of powder materials and changing of their operational properties. Consequently, investigation of the deformation of powder materials is important for solving problem of interrelation of deformation parameters and structure formation. This question has deeply studied for deformation of compact materials [3, 4]. It has shown clearly that temperature, strain rate and intensity of deformation take an influence on structure formation parameters that are determining the mechanism of softening process [5]. It has established theoretically and experimentally that increment of material's plasticity at small degrees of deformation and low temperatures is stipulated by dynamic recovery and polygonization, while it is connected with the kinetics of dynamic recrystallization at increasing of the temperature and degree of deformation [6, 7, 8]. Increasing of strain rate leads to growing resistance of deformation, decrease in plasticity due to intensification of hardening processes [9]. Obviously, the same dynamic softening processes may be observed into the solid phase of powder material [10]. However, the kinetics of softening processes is under the influence of pore phase presented in powder materials.

This paper is aimed in theoretical analysis of the kinetics of softening processes taking place in the powder material during plastic deformation at elevated temperatures and determination of dependences between deformation and structure formation parameters.

MATERIALS AND METHODS

Plastic forming at any conditions, on a basis of dislocation representations, may be characterized by changing of dislocation structure in hard phase of powder material, which causes the devel-

opment of two mutually balanced processes - hardening and softening. The expression of changing the dislocations' density during plastic deformation may be written as [11]:

$$\dot{\rho} = \eta\dot{\varepsilon} - k\rho, \tag{1}$$

where: ρ – is the dislocation density; k – is the Boltzmann function; $\dot{\varepsilon}$ – is the strain rate; η – is the coefficient that characterises the ability of hard phase to accumulate dislocations at particular strain rate.

The first term on the right side of the equation (1) describes the hard phase hardening and increment of dislocation density per unit of time that is growing with the strain rate $\dot{\varepsilon}$. The second term describes decreasing of strength through decrementing of dislocation density due to thermally activated processes expressed by the Boltzmann function:

$$k = k_0 \exp(-E / kT), \tag{2}$$

where: k_0 – is the frequency factor that is independent of temperature; E – is the activation energy of softening processes; T – is the absolute temperature.

The equation (1) has a solution [12] at the constant strain rate $\dot{\varepsilon}(t) = \varsigma = const$ and boundary conditions $\rho(0) = \rho_0$:

$$\rho = \left(\rho_0 - \frac{\eta\varsigma}{k}\right)\exp\left(-k\frac{\omega}{\varsigma}\right) + \frac{\eta\varsigma}{k}, \tag{3}$$

where: ω – is the accumulated deformation with taking into account the influence of porosity.

The accumulated deformation of hard phase is determined by the following expression [13]:

$$\omega = \int_0^\tau W d\tau, \tag{4}$$

where: W – is the rate of changing the accumulated deformation of powder material; τ – is the period of time.

The rate of changing the accumulated deformation of powder material may be written in the following way [13]:

$$W = \frac{1}{\sqrt{1-\theta}}\sqrt{\psi e^2 + \varphi\gamma^2}, \tag{5}$$

where: ψ, φ – are porosity functions; e – is the volume changing rate; γ – is the shape changing rate.

Porosity functions may be calculated by expressions [13]:

$$\psi = \frac{2}{3}\frac{(1-\theta)^3}{\theta},\ \varphi = (1-\theta)^2, \tag{6}$$

and assuming of (Eq. 4) and (Eq. 5),

$$\omega = \int_0^\tau \frac{1}{\sqrt{1-\theta}}\sqrt{\varphi\gamma^2 + \psi e^2}\, d\tau. \tag{7}$$

The volume changing rate for uniaxial compression may be written as:

$$e = e_z + 2e_r, \tag{8}$$

a shape changing rate may be determined using the formula:

$$\gamma = \sqrt{\frac{2}{3}}\left|e_z - e_r\right|, \tag{9}$$

considering the equation:

$$\frac{e_r}{e_z} = \nu = \frac{r-1}{2+r}, \tag{10}$$

after substitution to (Eq. 8) the volume changing rate has transformed to the formula:

$$e = (1+2\nu)e_z. \tag{11}$$

A spherical component of the stress tensor:

$$p = \frac{1}{3}\sigma_z, \tag{12}$$

the intensity of shear stress is determined by the formula:

$$\tau = \sqrt{\frac{2}{3}}\left|\sigma_z\right|. \tag{13}$$

Loading surface of powder body with taking into account pore formation process may be implemented in the form of:

$$\alpha(p-a)^2+\beta\tau^2=c^2, \tag{14}$$

where: α,β,a,c – are coefficients that are dependent on the porosity [13].

The following equation that describes dimensional changes during uniaxial compression has obtained in [7] on the basis of the plasticity law (Eq. 14) while considering (Eq. 12) and (Eq. 13):

$$r=\frac{1}{3}\frac{\alpha}{\beta}\left(1-3\frac{\alpha}{\sigma_z}\right)= \\ =\frac{1}{3}\frac{\alpha}{\beta}\left[\frac{-6\alpha\beta-\sqrt{c^2(\alpha+6\beta)-6\alpha\beta a^2}}{\alpha a-\sqrt{c^2(\alpha+6\beta)-6\alpha\beta a^2}}\right]. \tag{15}$$

The equation of axial stress under uniaxial compression looks like:

$$\sigma_z=\frac{3\alpha a}{\alpha+6\beta}-\frac{\sqrt{6}\sqrt{c^2(\alpha+6\beta)-6\alpha\beta a^2}}{\alpha+6\beta}. \tag{16}$$

Therefore, changing of porosity and accumulated deformation during forming operation may be calculated from the expressions:

$$\frac{d\theta}{d\varepsilon_z}=(1-\theta)(1-2\nu), \\ \frac{d\omega}{d\varepsilon_z}=\frac{1}{\sqrt{1-\theta}}\sqrt{\frac{2}{3}\frac{1}{r^2}\varphi+\psi(1-2\nu)sign(e_z)}. \tag{17}$$

It is possible to determine the axial stress and accumulated deformation from (Eq. 16) and (Eq. 17). The right side of (Eq. 17) contains nonlinear functions of porosity, consequently, solution of differential equations' system was performed numerically by the Newton-Raphson method. The function (Eq. 3) describes a decrementing change of dislocations' density ρ [11]. The value of ρ asymptotically goes to the limiting value $\eta\varsigma/k$ at $\omega\to\infty$ and $\tau\to\infty$. The value of $\eta\varsigma/k$ depends on the deformation temperature and activation energy of thermally activated softening process according to (Eq. 2) at the condition $\eta\varsigma=const$.

The ultimate density of dislocations under the given conditions of deformation goes to the value of $\eta\varsigma/k$ based on the expression (3). The average dislocations' density for maximum stress reached for this stage of deformation defined similarly to [12]:

$$\rho_k=\chi\frac{\eta\varsigma}{k}, \tag{18}$$

where: χ - is the ratio that is within $0<\chi<1$.

The dependence of the maximum flow stress σ_z for the certain deformation stage from the dislocation density ρ_k may be assumed in the following way [8]:

$$\sigma_z=A(\rho_k)^n, \tag{19}$$

where: A and n - are constants.

The expression (Eq. 19) after the substitution of (Eq. 18) looks like:

$$\sigma_z=A\left(\chi\frac{\eta\varsigma}{k}\right)^n. \tag{20}$$

After taking the logarithm, substitution of (Eq. 2) and transformations obtained:

$$ln\,\sigma_z=ln\left[A\left(\chi\frac{\eta\varsigma}{k_0}\right)^n\right]+\frac{nE}{RT}. \tag{21}$$

Obviously, the maximum flow stress σ_z for certain forming stage is corresponding to the accumulated deformation ω_k. Then, substituting the expression (3) the value of accumulated strain and equating (3) and (8), after transformations we obtain:

$$\omega_k=\frac{\varsigma}{k}ln\left[\left(\rho_0\frac{k}{\eta\varsigma-1}\right)(\chi-1)^{-1}\right]. \tag{22}$$

It has assumed from (Eq. 22) that the factor $ln\left[(\rho_0 k/(\eta\varsigma-1))(\chi-1)^{-1}\right]$ in case of balance between hardening and softening processes tends to a constant value for a certain flow stress. In this case, the rate of changing the dislocation density goes to zero. The following equation obtained from (Eq. 1) at a constant strain rate:

$$\frac{1}{\rho_к} = \frac{k}{\eta\varsigma}. \quad (23)$$

Then:

$$ln\left[\left(\rho_0\frac{k}{\eta\varsigma}-1\right)(\chi-1)^{-1}\right] = ln\left[\left(\frac{\rho_0}{\rho_ê}-1\right)(\chi-1)^{-1}\right] \quad (24)$$

It has accepted that the dislocation density of the hard phase before and after deformation are different by 10-100 times [14], and $\chi \approx 0{,}8 \div 0{,}9$, means that the value of (Eq. 24) may be approximately determined as $ln\,10 \approx 2{,}3$.

The equation (Eq. 22) was transformed to the form:

$$\omega_к \approx 2{,}3\frac{\varsigma}{k} \cong 2{,}3\frac{\varsigma}{k_0}exp\left(\frac{E}{RT}\right). \quad (25)$$

than, after taking the logarithm, obtained:

$$ln\,\omega_к = ln\frac{\varsigma}{k_0} - \frac{E}{RT}. \quad (26)$$

Equations (Eq. 21) and (Eq. 26) are relations connecting the main parameters of the deformation of the porous body flow stress, accumulated deformation of hard phase, strain rate, temperature and structural characteristics of the powder material ($\eta, k, E, \rho_к$), which characterizes softening process during deformation at the elevated temperatures. A linear character of dependences of the accumulated deformation and corresponding stress from the reciprocal temperature follows from these equations with a slope of the line equal to the activation energy of dynamic softening process flowing by one or another mechanism.

A slope of the function $ln\,\omega_к = f(1/T)$ is E/RT and corresponding to (Eq. 26). Consequently, the slope of the function $ln\,\sigma_z = f(1/T)$ is nE/RT according to (Eq. 21). Therefore, slopes of these functions different by the value n are characterizing a maximum possible density of dislocations.

RESULTS AND DISCUSSION

Verification of the mathematical model has performed for evaluation of the activation energy of dynamic softening processes at uniaxial compression of copper-titanium powder material. Samples were prepared from charge consists of copper powder PMS–1 and titanium powder BT1–0 with a mass fraction of titanium 0.5% and a porosity of 5-10% after sintering at 900 – 920 ºC during 3 hours into a synthesis gas atmosphere. Samples were deformed on the testing machine ZD–4 at temperatures of 100, 300, 400 and 600 ºC with strain rate 0.01 s^{-1} while recording of the indicator diagram.

The accumulated deformation values were calculated using (Eq. 17), axial stress values - by (Eq. 16), dependences $ln\,\sigma_z - 1/T$ and $ln\,\omega_к - 1/T$ (Fig. 1) have drawn.

The obtained dependences are straight lines, except for the points at 400 °C, in which there are gaps of functions due to deformation aging. Straight lines consist of two branches - low temperature branch that is characterizing deformation process at 100 – 300 ºC and high temperature branch for deformation process at 400 – 600 ºC. Slopes of these functions define the activation energy of dynamic softening passing through a particular mechanism. The degree of deformation characterizes by the stage N.

The low-temperature branch possess the lowest activation energy (Fig. 2a) with values within 0.20-0.39 eV at 5 % porosity samples and 0.12-0.28 eV at 10 % porosity, which is the result of dynamic recovery and polygonisation [9].

The activation energy at high temperatures grows more than 10 times (Fig. 2b) that indicates more intensive softening. The greatest increment of activation energy 2.3-2.6 eV has observed at high temperatures during the third stage. In this case, the activation energy of softening comparable with the activation energy of self-diffusion of pure copper, equal to 2.79 eV [14], so we can assume that softening during the third step carries out by dynamic recrystallization.

It should be noted that the value of activation energy of softening process depends on the initial porosity of samples and takes higher values at 5% porosity (Fig. 2).

The presence of pores decelerates the dynamic softening that has confirmed by experimental studies [15]. For example, E = 2.6 eV at θ_0 = 5% and E = 2.3 eV at θ_0 = 10% for high temperature deformation when softening takes place by dynamic recrystallization.

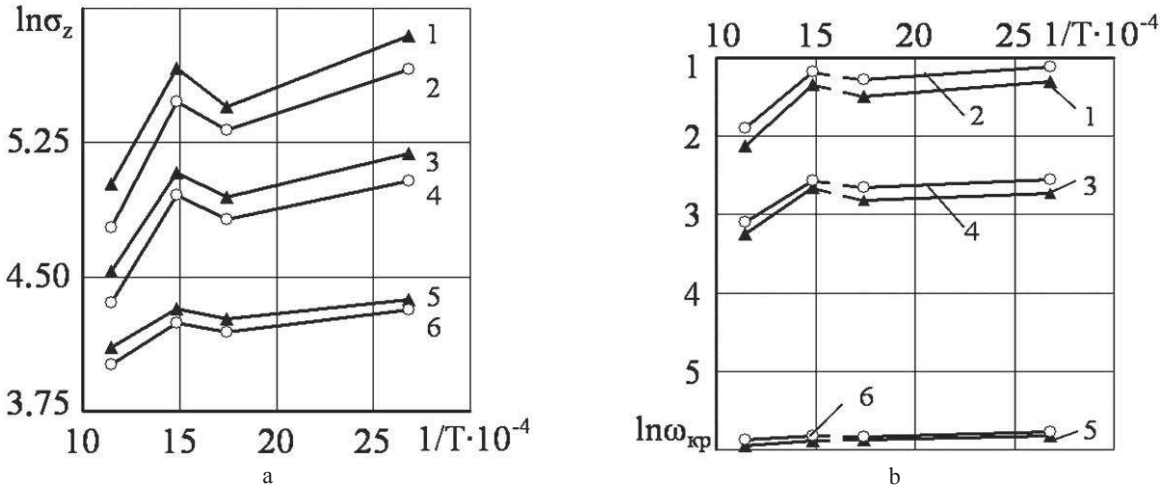

Fig. 1. The dependences $ln\,\sigma_z - 1/T$ – a, $|ln\,\omega_к| - 1/T$ – b: ▲ – θ_0 = 5 %; ○ – θ_0 = 10 %; 1, 2 – N = 1; 3, 4 – N = 2; 5, 6 – N = 3

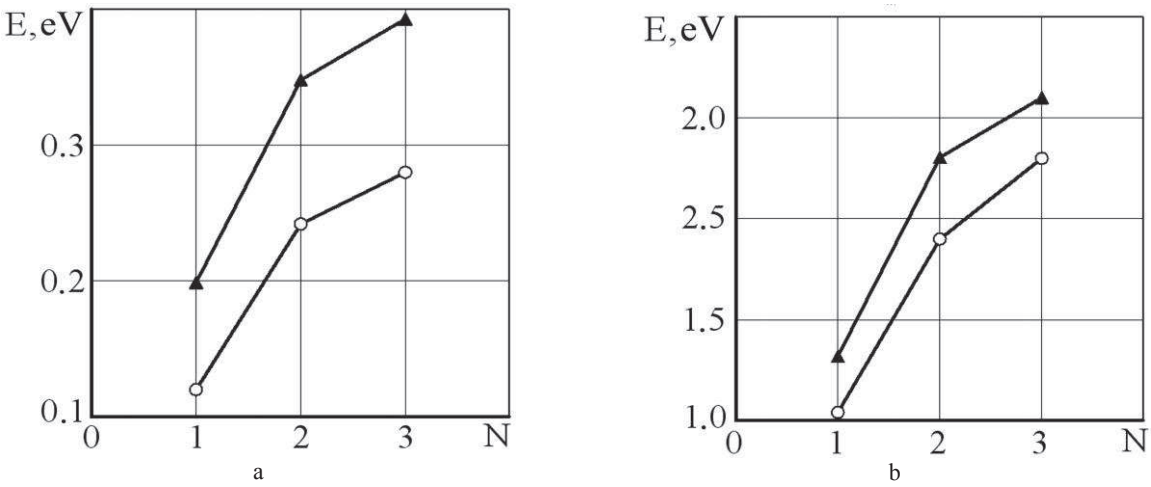

Fig. 2. Variation of activation energy during softening process: a – is the low-temperature branch; b – is the high-temperature branch; ▲ – θ_0 = 5 %; ○ – θ_0 = 10 %.

The existence of low-temperature and high-temperature branches on dependences $ln\,\sigma_z - 1/T$, that characterize softening mechanism has been verified by metallographic analysis. The character of structure changes in the powder material with 0.5 % Ti and 10 % porosity depends upon the temperature of deformation and observed at magnification ^{x}4000 (Fig. 3). Grain shape and condition of their boundaries characterize the processes occurring in the powder material during its deformation [16]. These experiments allowed to explain observed phenomena, establish the beginning of dynamic recrystallization and follow the kinetics of its development.

The starting structure after sintering of the powder material into a synthesis gas atmosphere at 900 – 920 ºC is characterized by the presence of copper grains, pores and titanium particles (Fig. 3, a). A considerable inequigranularity of microstructure has been observed. It has connected with inhomogeneous development of static recrystallization during sintering due to inhomogeneous stress state formed at compaction of charge from copper and titanium powders into a closed matrix [17].

Shapes of deformed copper grains observed on the photo of metallographic section of sample deformed by compression to the degree of deformation 0.69 at temperature 100 °C and strain rate 0.001 s^{-1} (Fig. 3, b) are different of equiaxed grains formed during sintering (Fig. 3, a). The dynamic recovery does not lead to rebuilding of structure, so distorted boundaries appeared as the consequence of severe deformation of hard phase. The refinement of grains has observed near titanium particles due to a local increase of internal stresses around the particles of alloying addition.

Deformation at 400 °C leads to formation of complex-shaped boundaries (Fig. 3, c) that indicates processes associated with the migration of grain boundaries and formation of new ones. New

interfaces presented by coherent particles of precipitates have formed at 400 °C as the result of decomposition of a supersaturated solid solution during deformation of the copper-titanium materials [18, 19] that promotes the primary recrystallization [20]. However, the development of dynamic recrystallization inhibited due to blocking of grain boundaries by particles, thus preventing of their migration and resulting in assuming of serpentine shape (Fig. 3, c).

The microstructure of the sample after deformation at 600 ºC is characterized by the presence of equiaxial copper grains formed as the result of dynamic recrystallization (Fig. 3, d).

The recrystallized grains have a polygonal shape. Intragranular twins, appeared during dynamic recrystallization, have formed by separation of growing twins from double angle boundaries [21] as the result of recrystallized grain growth. It should be noted that equiaxial structure is formed by the volume of sample. It allows to suggest a uniform and complete flow of dynamic recrystallization at 600 ºC.

Increase of titanium content up to 2 % in the powder material of 5 % porosity at low temperature deformation leads to growing of the stress and ultimate degree of deformation that is corresponding to changing the activation energy and, consequently, the deformation mechanism. These processes provide grain refinement during compression to various degrees of deformation (Fig. 4). Different barriers like grain boundaries and pores, twin boundaries and titanium particles are places of defects concentration in copper-titanium powder materials, where the most fine- grained structure has been observed (Fig. 4, b). Grain refinement on boundaries of copper and titanium particles during plastic deformation is a consequence of differences in the plastic and elastic properties of copper and titanium.

Formation of finer grain size less than 2 μm has been observed near titanium particles (Fig. 5, a) as a result of compression of samples containing 2 % of titanium at 400 °C until the degree of deformation 0.081. High values of stresses and deformations in these places promotes to beginning of dynamic recrystallization leading to formation of new grains. However, intermediate phases, precipitated as a result of the strain aging [19], are inhibiting growth of dynamically recrystallized grains [22]. Consequently, anisomerous structure near titanium particles preserved up to degree of deformation 0.262 (Fig. 5, b). Further compaction to the degree of deformation 0.673 forms a homogeneous fine-grained structure

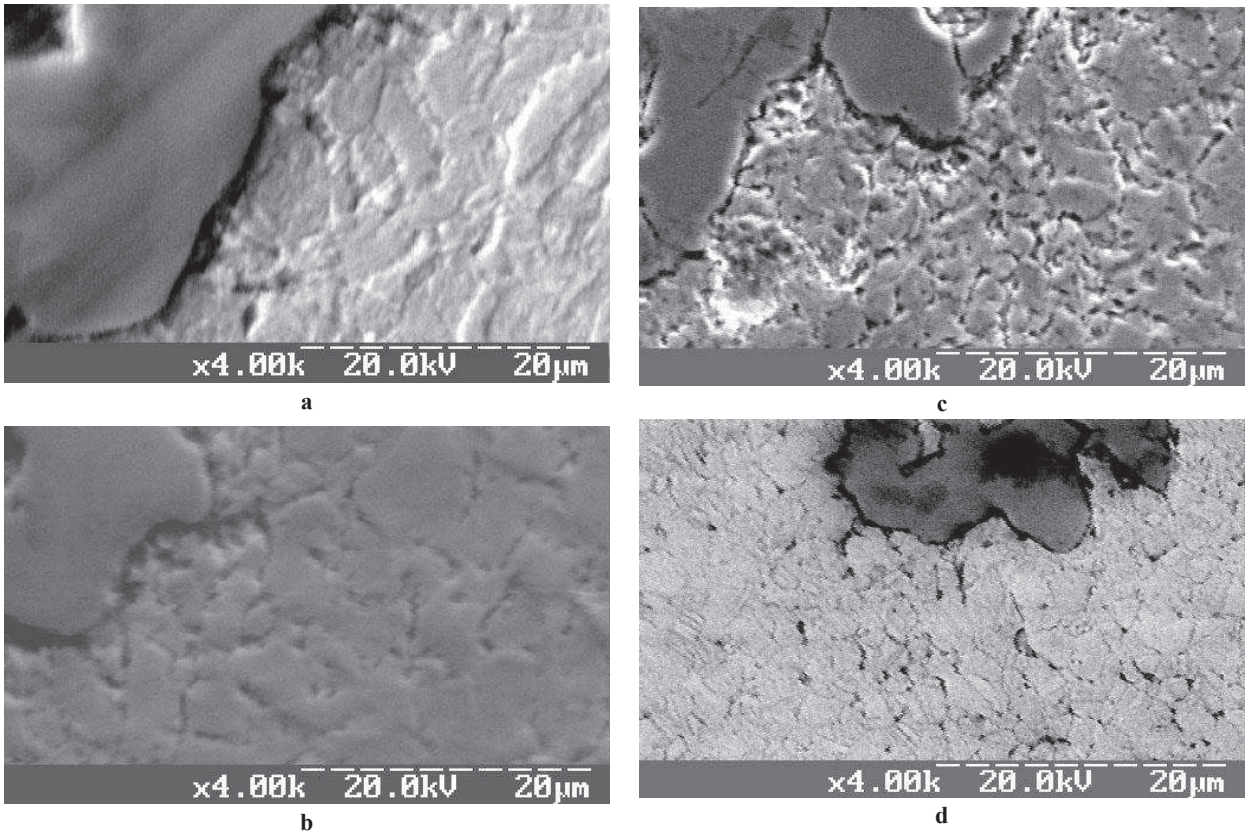

Fig. 3. Microstructures of samples with 0.5% Ti and 10 % porosity; a – after sintering; after deforming to degree of deformation 0.69 at strain rate 0.001 s^{-1}: b – t = 100 ºC; c – t = 400 ºC; d – t = 600 ºC.

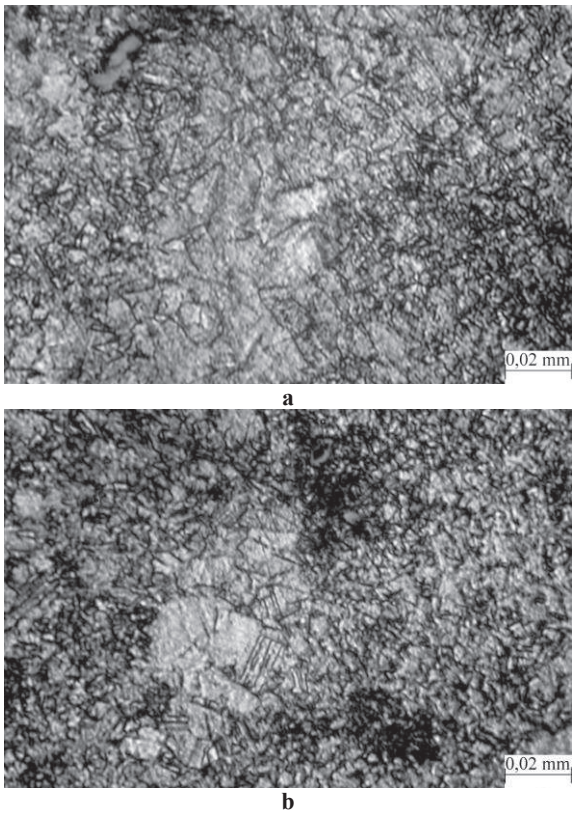

Fig. 4. Microstructure of samples: 2% Ti; 5% porosity; compression at 100 °C and strain rate of 0.001 s^{-1}: a - 0.079 the degree of deformation; b - the degree of deformation of 0.26

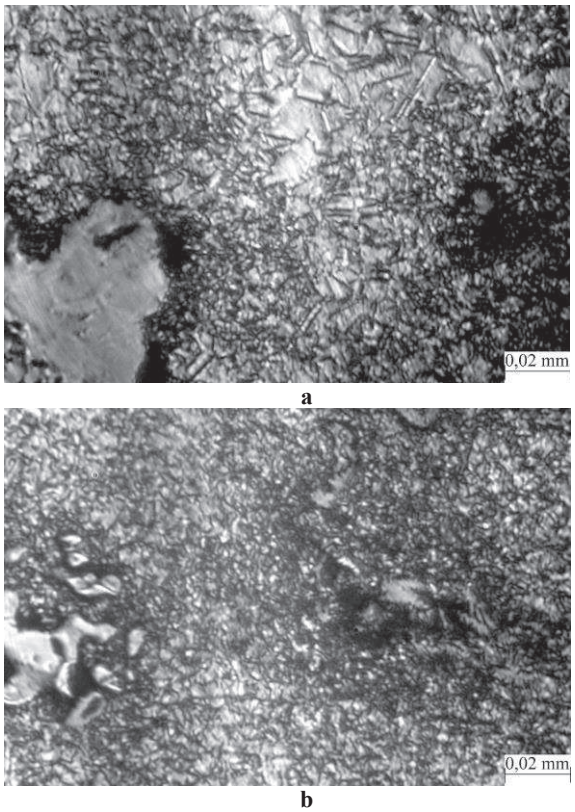

Fig. 5. Microstructure of samples: 2% Ti; 5% porosity; compression at 400 °C at a strain rate of 0.001 s^{-1}: a - 0.081 the degree of deformation; b - the degree of deformation of 0.262.

with the copper grain size 7.28 μm by improving of completeness of the dynamic recrystallization.

Deformation at 600 ºC follows by intensive decrease of copper grain size while increasing degree of deformation, which is typical for copper-titanium powder material with 0.5 % and 2 % of titanium (Fig. 6). Metallographic studies have shown that anisomerous structure observed at low deformation degrees of 0.040 – 0.046 and caused by inhomogeneous development of dynamic recrystallization. Formation of a homogeneous fine-grained structure due to increasing of dynamic recrystallization rate have occurred, while increasing of deformation degree to 0.118.

The microstructures of samples with 2 % and 0.5 % Ti after compression is characterized by copper grains with serrated boundaries (Fig. 6). The structure of samples with 0.5% Ti is less stable, grain boundaries are extremely tortuous and include a large number of "tips." The reason of serration is local heterogeneity of deformation in the border volumes and the consequent difference in the density of defects into local volumes on both sides of the original boundary that is stimulating migration of small areas to the volume with higher density of defects. Toothed shape of boundaries indicates the dynamic recrystallization [23]. Reduction of curvature of grain boundaries in

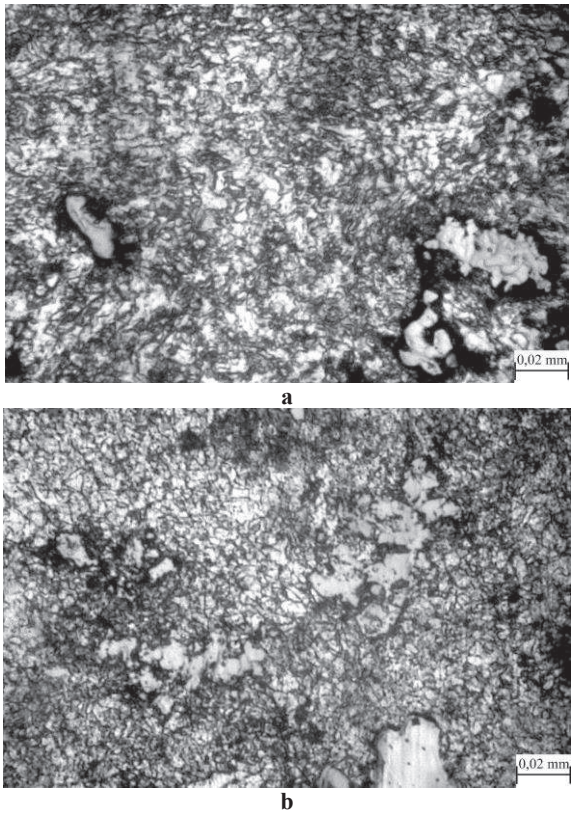

Fig. 6. The microstructure of samples of 10% porosity after compression strain rate of 0.001 s^{-1} at a temperature and deformation degree 600 ºC 0.916: and - 0.5% Ti, b - 2% Ti.

samples with 2 % Ti is associated with inhibition of grain boundaries' migration by pores, titanium particles and disperse precipitations (Fig. 6).

Increasing of the deformation temperature causes softening of hard phase and, as shown by metallographic analysis, softening of copper begins at 100 °C for the account of recovery and above 300 ºC – by the dynamic recrystallization.

Commercially pure titanium (BT1–0) recrystallizes at temperatures 750 – 800 ºC [24]. Therefore, we can assume that in the investigated temperature range (100 – 600 ºC) softening takes place for the account of the dynamic recovery.

Obviously, the dynamic recovery does not allow to complete internal release of stresses removal and titanium particles are crushed because of high intensity of plastic deformation.

CONCLUSIONS

1. Theoretical analysis of the kinetics of softening processes has been conducted for deformation at high temperatures and based on the dislocation concepts of plastic deformation and plasticity theory of porous powder bodies. Constitutive equations that are relating the main parameters of the deformation - flow stress, accumulated deformation of hard phase, strain rate, temperature and structural characteristics of the material - ability to accumulate dislocations, Boltzmann function, activation energy, density of dislocations, which may be used for evaluation the mechanism of dynamic softening during deformation of porous powder materials at the elevated temperatures.
2. The linear character of the dependencies $ln\sigma_z - 1/T$ and $ln\omega_\kappa - 1/T$, which allows to determine the mechanism of dynamic softening of porous bodies has found. It has established by estimating of activation energy that at low temperatures the deformation mechanism is dynamic softening and recovery polygonization, at high temperatures - dynamic recrystallization. The presence of porosity phase reduces the activation energy of softening and makes its development less intensive.
3. The mechanism of dynamic softening was confirmed by metallographic examination of structure evolution in compression samples from copper-titanium powder materials of different titanium content.

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ВЗАИМОСВЯЗЬ ПАРАМЕТРОВ ПЛАСТИЧЕСКОГО ДЕФОРМИРОВАНИЯ И СТРУКТУРООБРАЗОВАНИЯ В ПОРОШКОВЫХ МАТЕРИАЛАХ

Резюме. Проведен теоретический анализ кинетики разупрочняющих процессов, проходящих в порошковом пористом теле при деформировании в области повышенных температур. Получены определяющие уравнения, связывающие параметры деформации и параметры структурообразования, которые характеризуют процессы динамического разупрочнения при деформировании. Показана линейная зависимость логарифма осевого напряжения и накопленной деформации твердой фазы в функции обратной температуры. Выполнена оценка энергии активации динамического разупрочнения при одноосном сжатии. Показано, что при низких температурах деформации механизмом разупрочнения является динамический возврат и полигонизация, при повышенных температурах – динамическая рекристаллизация. Пористость снижает энергию активации динамического разупрочнения. Металлографический анализ подтверждает механизм динамического разупрочнения порошкового материала.

Ключевые слова: математическая модель, возврат, полигонизация, рекристаллизация, энергия активации.