

Numerical simulation of the sintering process of multicomponent composite materials based on iron and glass

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Based on the mathematical description of the kinetics of compaction and volumetric shrinkage of a porous body represented by a mixture of cermet powders, taking into account the mechanisms of diffuse coalescence and coagulation, a system of nonlinear differential equations of porosity and granularity was obtained for the solid-phase and liquid-phase stages of the sintering process under conditions of sequential temperature annealing, with certain speeds changes and durations. The obtained equations can be used to control the sintering process by adjusting the level, speed and duration of individual temperature conditions and technical parameters of the mixture, as well as choosing the composition, consistency and geometric characteristics of the components of the powder mixture for the manufacture of a material with desired physical and mechanical properties. The differential equations of the sintering process include the coefficients of volumetric, grain boundary, and surface diffusion, the calculation of which for multicomponent systems requires the calculation of both the self-diffusion coefficients of the components of the system and the coefficients of their mutual diffusion. The sintering process of powders of iron, cast iron and ceramic is considered as the mutual diffusion of two binary alloys: cast iron (iron + carbon) and fayalite (iron + ceramic). The calculation of the coefficient of mutual diffusion of the resulting alloy was carried out according to the Darken formula. For the numerical solution of the problem, the scheme of the Runge-Kutta method of the fourth order of accuracy with a variable integration step is used. A program complex for solving the problem has been developed, the calculation results are shown on the example of an alloy of a powder mixture of iron, cast iron and glass.

Keywords: cermet materials, solid-phase sintering, liquid-phase sintering, melting, alloy, fayalite, sintering rheology, homogenization, glass metal, glass-metal materials, Runge-Kutta method, self-diffusion, heterodiffusion, mutual diffusion, grain-boundary diffusion, volumetric diffusion.

Чисельне моделювання процесу спікання багатокomпонентних композиційних матеріалів на основі заліза і скла. *Т.Г.Джаббаров, О.А.Дишин, М.Б.Бабанли, І.І.Аббасов*

На основі математичного опису кінетики ущільнення і об'ємної усадки пористого тіла, представленого сумішшю металокерамічних порошоків, з урахуванням механізмів дифузійної коалесценції і коагуляції, побудовано систему нелінійних диференціальних рівнянь пористості і зернистості для твердофазної і рідиннофазної стадій процесу спікання на послідовних температурних режимах відпалу, з певними швидкостями зміни і тривалістю. Отримані рівняння можуть використовуватися для контролю і управління процесом спікання шляхом регулювання рівня, швидкості і тривалості окремих температурних режимів і технічних параметрів шихти, а також вибору складу, консистенції і геометричних характеристик компонентів порошкової суміші для виготовлення матеріалу з заданими фізико-механічними властивостями. Диференціальні рівняння процесу спікання включають коефіцієнти об'ємної, зернограничної і поверхневої дифузії, обчислення яких для багатокomпонентних систем вимагає розра-

хунку як коефіцієнтів самодифузії складових компонентів системи, так і коефіцієнтів їх взаємної дифузії. Процес спікання порошків заліза, чавуну і ситалу розглянуто як взаємну дифузію двох бінарних сплавів: чавуну (залізо + вуглець) і фаяліту (залізо + ситал). Розрахунок коефіцієнта взаємної дифузії утвореного сплаву проведено за формулою Дарка. Для чисельного рішення задачі використано схему методу Рунге-Кутти четвертого порядку точності з перемінним кроком інтегрування. Розроблено програмний комплекс розв'язання задачі, результати розрахунку наведено на прикладі сплаву порошкової суміші заліза, чавуну і ситалізованого скла.

На основе математического описания кинетики уплотнения и объемной усадки пористого тела, представленного смесью металлокерамических порошков, с учетом механизмов диффузной коалесценции и коагуляции, построена система нелинейных дифференциальных уравнений пористости и зернистости для твердофазной и жидкофазной стадий процесса спекания при последовательных температурных режимах отжига, с определенными скоростями изменения и продолжительностями. Полученные уравнения могут использоваться для контроля и управления процессом спекания путем регулирования уровня, скорости и длительности отдельных температурных режимов и технических параметров шихты, а также выбора состава, консистенции и геометрических характеристик компонентов порошковой смеси для изготовления материала с заданными физико-механическими свойствами. Дифференциальные уравнения процесса спекания включают коэффициенты объемной, зернограничной и поверхностной диффузии, вычисление которых для многокомпонентных систем требует расчета как коэффициентов самодиффузии составляющих компонентов системы, так и коэффициентов их взаимной диффузии. Процесс спекания порошков железа, чугуна и ситалла рассмотрен как взаимная диффузия двух бинарных сплавов: чугуна (железо+углерод) и фаялита (железо+ситалл). Расчет коэффициента взаимной диффузии образованного сплава проведен по формуле Даркена. Для численного решения задачи использована схема метода Рунге-Кутты четвертого порядка точности с переменным шагом интегрирования. Разработан программный комплекс решения задачи, результаты расчета приведены на примере сплава порошковой смеси железа, чугуна и ситаллизованного стекла.

1. Introduction

The vast majority of modern machines and mechanisms require the creation of antifriction materials with a minimum coefficient of friction and high wear resistance. To meet these requirements, various kinds of antifriction materials based on non-ferrous metals such as bronze and babbitts, as well as tribological composite materials obtained by various materials have been created. Powder metallurgy makes a great contribution to the creation of new materials with enhanced tribotechnical properties that cannot be obtained by casting. One of the directions of obtaining wear-resistant materials is the creation of materials, the pores of which are filled with glassware. Recently, metal-glass materials (MG) based on iron and copper have been developing most intensively. The resulting materials differ from analogs by their increased wear resistance and low coefficient of friction. However, despite the great demand for this type of materials, metal-glass materials remain poorly understood. High brittleness of glass does not have a negative effect on the performance of products obtained from MG materials, due to the fact that small glass inclusions that melt and fill pores during sin-

tering are less susceptible to brittle fracture than large ones [1–3].

To improve interfacial interaction and intensify diffusion phenomena, and, consequently, increase the mechanical properties and wear resistance of iron-glass materials, they should be doped with components that are more wetted by glass [4]. For example, the use of cast iron powder in the composition of the charge can lead to this effect, since cast iron contains a relatively large amount of silicon and manganese, which can form very stable oxides upon heating. It is expected that during sintering these oxides are well wetted by glass and thereby contribute to its strong bond with the metal base [5]. As a result of research [6], the composition of the powder mixture "iron-cast-iron-glass" with the best strength was found (in percent): iron — 44, cast iron — 50, glass — 6. The optimum sintering temperature in the endogas environment for materials of the "iron cast iron" type "can be considered 1200°C. Available in the literature, mathematical models and methods for describing the kinetics of sintering processes of powder materials are mainly devoted to one or two-component systems [7–8]. Sintering processes and their computer simulation for multicomponent systems consisting of several metal and ce-

ramic powders and glass materials have been little studied because of the complexity of calculating the mutual diffusion coefficients [9].

The aim of this work is to construct mathematical models of the development of porosity and an increase in grain size at a certain rate in the form of differential equations corresponding to the sequence of temperature-time modes of sintering pressed briquettes from a mixture of metal powders containing a low concentration of high-silicon glass powder. These equations include the coefficients of volumetric, grain boundary, and surface diffusion, which are obtained for multicomponent systems by calculating self-diffusion coefficients of components of the system and their mutual diffusion coefficients.

2. Kinetic equations of sealing powder materials for sintering

At the heating stage, sintering of a powder body occurs by a diffusion (solid-phase) mechanism without the formation of a liquid phase. The following main processes are distinguished: volumetric and surface diffusion of atoms, shrinkage, recrystallization of a metal powder body (growth of some grains due to others of the same phase), transfer of atoms through the gas phase due to volumetric and surface diffusion, viscous flow and flow caused by external loads during sintering under pressure. This type of sintering is accompanied by the development of bonds between particles, the formation and growth of contacts (necks), the "healing" of pores (closing of through porosity) with their enlargement and spheroidization, compaction of the preform and its shrinkage; the latter is mainly associated with the volumetric deformation of particles due to bulk self-diffusion of atoms and adsorption of atoms (adatoms) on the surface of a substance (surface diffusion).

We introduce the following notations:

v_n and v_c — sample pore volumes before and after sintering, respectively;

d_n and d_c — density of the porous body before and after sintering;

Π_0 and Π_c — body porosity before and after sintering;

V_0 and V_c — body volume before and after sintering;

l_0 and l_c — linear body size before and after sintering.

From the dependences given in [10]

$$v_c/v_n = [d_n(d_k - d_c)]/[d_c(d_k - d_n)], \quad (2.1)$$

$$\Pi_c/\Pi_0 = (d_k - d_c)/(d_k - d_n).$$

After simple algebraic transformations, we obtain the relation between porosity Π_c and volumetric shrinkage $\Delta V/V_0$ ($\Delta V = V_c - V_0$):

$$\Pi_c = 1 - (1 - \Pi_0)/[1 - (\Delta V/V_0)^2]. \quad (2.2)$$

Assuming that the sintering is due to a viscous flow or diffusion in the boundary contact, which is capable of infinitely absorbing vacancies, the following relationship was obtained between volumetric shrinkage $\Delta V/V_0$ and linear shrinkage $\Delta l/l_0$ ($\Delta l = l_c - l_0$ [11]):

$$\Delta V/V_0 = 3\Delta l/l_0. \quad (2.3)$$

The initial stage of sintering is characterized by the formation and growth of a neck between particles, accompanied by shrinkage of the sample. Based on a simplified model of sintering of two spheres, the parameters of grain boundary diffusion were determined from measurements of shrinkage at the initial stage of sintering [10]. This model took into account both the energy of grain boundaries and its effect on the surface of the neck, as well as additional volumetric diffusion from the surface of the neck. Exact solutions were used to describe the geometry of the neck. For this, along with the radius of curvature of the neck surface (ρ_0), another parameter was introduced A_r — the area on the neck surface, which is reached by the atoms of the body due to volumetric diffusion:

$$A_r = 4\pi\rho_0 \left[\left(C - \frac{\pi - B}{2} \right) (r_g + \rho_0) \cos C - \rho_0 (\sin C - \sin \frac{\pi - B}{2}) \right], \quad (2.4)$$

where

$$C = \sin^{-1} \left[\frac{r_g - \Delta r + \rho_0 \sin(\pi - B)/2}{r_g + r_0} \right], \quad (2.5)$$

where r_g is the radius of the particle; Δr is the rapprochement of centers; B is the angle of solution of the grain-boundary groove in the neck, which is formed for local equilibrium between the surface tension of the grain boundary and the free surface of the neck. Assuming, as in previous works [10–12], that the specific linear

shrinkage $\Delta l/l_0$ of the powder sample is equal to $\Delta r/r_g$, i.e. :

$$\Delta l/l_0 = \Delta r/r_g. \quad (2.6)$$

The author of [13] obtained the following equation of the kinetics of shrinkage:

$$[X^3 \rho_0 / (r_g \cdot X + \rho_0 \cos(\pi - B)/2)] [d(\Delta l/l_0)/dt] = \quad (2.7)$$

$$= (2\gamma\Omega D_V / \pi T k r_g^3) \cdot (A_r / r_g^2 \cdot X) + (4\gamma\Omega b D_b / k T r_g^4),$$

where D_V and D_b are the coefficients of volumetric and grain boundary diffusion, respectively; b is the width of the boundary between particles; Ω is the atomic volume; k is the Boltzmann constant; γ is the surface tension associated with surface pressure σ in the case of a spherical particle, $\sigma = 2\gamma/r_g$, with a radius r ; T is the absolute sintering temperature; $X = x/r_g$ is the specific neck size; x is the radius of the neck. In this case, the transfer mechanisms that do not contribute to shrinkage (such as surface diffusion and transfer through the gas phase) were not taken into account.

From equation (2.7), by measuring the rate of shrinkage, neck size and the angle of the groove in the neck at the grain boundary, as a result of experiments in the sintering process, the values of the coefficients of volumetric and grain boundary diffusion are obtained by the formula

$$D_V = (\pi k T r_g^3 / 2\gamma\Omega) \cdot \lambda, \quad (2.8)$$

$$b D_b = (k T r_g^4 / 4\gamma\Omega) \cdot K,$$

where λ and K are, respectively, the slope and the segment ($A_r/r_g^2 = 0$) on the axis of the linear dependence of the expression $X^3 \rho_0 d(\Delta l/l_0)/dt / [r_g \cdot X + \rho_0 \cos(\pi - B)/2]$ on $A_r/r_g^2 \cdot X$.

In the absence of mass transfer from the surface, equation (2.7) is simplified and for the first 3.5 percent shrinkage takes the form:

$$(\Delta l/l_0)^{2.06} [d(\Delta l/l_0)/dt] = \quad (2.9)$$

$$= \frac{2.63\gamma\Omega D_V}{k T r_g^3} \cdot (\Delta l/l_0)^{1.03} + \frac{0.70 \cdot \gamma\Omega b D_b}{k T r_g^4}.$$

In the case when volumetric diffusion prevails over grain boundary diffusion, differential equation (2.9) has a solution with respect to $y = \Delta l/l_0$:

$$y \cong (5.34\gamma\Omega \cdot D_V / k T r_g^3)^{0.49} \cdot t^{0.49}. \quad (2.10)$$

In the opposite case, when grain-boundary diffusion prevails over volumetric diffusion, equation (2.9) has the solution:

$$y \cong (2.14\gamma\Omega b \cdot D_b / k T r_g^4)^{0.33} \cdot t^{0.33}. \quad (2.11)$$

In the notation $y = \Delta l/l_0$, equation (2.2) is written in the form:

$$\Pi_c = 1 - \frac{1 - \Pi_0}{(1 - 3y)^2}$$

where do we find

$$y = \frac{1}{3} \left[1 - \left(\frac{1 - \Pi_0}{1 - \Pi_c} \right)^{\frac{1}{2}} \right]. \quad (2.12)$$

Differentiating the last equation with respect to t , we obtain the following differential equation with respect to Π_c :

$$\frac{d\Pi_c}{dt} = - \frac{6(1 - \Pi_c)^{3/2}}{(1 - \Pi_0)^{1/2}} \cdot \frac{dy}{dt}. \quad (2.13)$$

In view of (2.9), equation (2.13) can be written in the form:

$$\frac{d\Pi_c}{dt} = - \frac{6(1 - \Pi_c)^{3/2} \cdot 3^{2.1}}{(1 - \Pi_0)^{1/2} \left[1 - \left(\frac{1 - \Pi_0}{1 - \Pi_c} \right)^{1/2} \right]^{2.1}} \times$$

$$\times \left\{ \frac{2\gamma\Omega D_V}{3k T r_g^3} \left[\left(\frac{1 - \Pi_0}{1 - \Pi_c} \right)^{\frac{1}{2}} \right] + \frac{\gamma\Omega b D_b}{2k T r_g^4} \right\} \quad (2.13,a)$$

and equation (2.9) in the form:

$$\frac{d\Pi_c}{dt} = - \frac{6(1 - \Pi_c)^{3/2} \cdot 3^{2.06}}{(1 - \Pi_0)^{1/2} \left[1 - \left(\frac{1 - \Pi_0}{1 - \Pi_c} \right)^{1/2} \right]^{2.06}} \times$$

$$\times \left\{ \frac{2.63\gamma\Omega D_V}{3^{1.03} k T r_g^3} \left[1 - \left(\frac{1 - \Pi_0}{1 - \Pi_c} \right)^{1/2} \right]^{1.03} + \frac{0.70\gamma\Omega b D_b}{k T r_g^4} \right\}. \quad (2.13,b)$$

During sintering, a quantitative analysis of the kinetics of changes in the characteristic linear parameter of the porous microstructure L (L is the average grain size of the sample) can be carried out using the general theory of diffusion coalescence of dispersed systems [14]. If the main mechanism of mass transfer is surface diffusion,

then the kinetic equation for L takes the form [15]:

$$L^4 = L_0^4 + \frac{B_1 \gamma D_S \delta^4}{kT} \cdot t, \quad (2.14)$$

where D_S is the surface diffusion coefficient; $\Delta L = L - L_0$ is the thickness (of the order of the atomic diameter δ) of the layer in which surface diffusion occurs; t is the time; B_1 is the numerical constant approximately equal to 30; L_0 is the initial value of the parameter L before sintering.

Equation (2.14) satisfies many experimental data for oxide and metal powders [18]. For metal island films, coalescence occurs predominantly by the mechanism of surface diffusion of adatoms (adsorbing atoms) [15]. Differentiating (2.14) with respect to t , we obtain the differential equation of kinetics of change L :

$$\frac{dL}{dt} = \frac{B_1 \gamma D_S \cdot \delta^4}{4L^3 kT}. \quad (2.15)$$

In subsequent studies of liquid-phase sintering, theoretical results were based mainly on the compaction mechanisms discussed above, which develop sequentially during sintering. Further development of the theory of sintering with a liquid phase was obtained in the works of V.V.Skorokhod with co-workers [17] and Ya.E.Geguzin [15]; we took their results into account in the mathematical description of the kinetics of compaction at the stage of isothermal (liquid-phase) sintering under pressure P [18].

3. Calculation of diffusion coefficients by experimental annealing data

During diffusion annealing at the temperature T and annealing time τ , the material diffuses along the axis z into the bulk of the grain, as well as along the grain boundaries. The diffusion coefficient along the boundary turns out to be much larger than elsewhere, therefore, the bulk diffusion of material from the walls of the grain boundary into the surrounding crystals begins. Let D_V be the volumetric diffusion coefficient describing diffusion in grains, and D_b — the diffusion coefficient in the grain boundary layer, in this case, the direct contribution of diffusion from the surface decreases with distance much faster than the contribution of grain boundaries, and, therefore, far from the surface, diffusant enters the bulk practically only from

grain boundaries. The derivation of the diffusion equation is based on the following assumptions: 1) the laws of diffusion are satisfied both in the crystal and in the grains; 2) the diffusion coefficients D_V and D_b are both isotropic and independent of concentration, coordinates, and time; 3) the concentration of the diffusant $c(z, \tau)$ and its flow J_z in the direction z is determined by the first Fick law:

$$J_z = -D_V \cdot \frac{\partial c(z, \tau)}{\partial z} \quad (3.1)$$

and is continuous on the walls of the grain boundary, i.e. at $y = \pm\delta/2$; also, the flow J_z is continuous at the interface between grain and grain; 4) the thickness δ of the grain boundary is so small that a change in the concentration across the boundary (i.e., in the direction y) can be neglected.

For the analytical description of grain-boundary diffusion, dimensionless variables ε , η and β are introduced, which correspond to y , z and τ , as well as a dimensionless parameter Δ characterizing the ratio of D_b to D_V :

$$\varepsilon = \frac{y - \delta/2}{(D_V \tau)^{1/2}}; \quad \eta = \frac{z}{(D_V \tau)^{1/2}} \quad (3.2)$$

$$\beta = \frac{(\Delta - 1)\delta}{2(D_V \tau)^{1/2}} = \frac{\delta D_b}{2D_V^{3/2} \tau^{1/2}}; \quad \Delta = \frac{D_b}{D_V}.$$

The physical meaning of the parameter η is that at a given depth z , it describes the effect of direct volumetric diffusion from the source into the crystal; the stronger this effect, the less η . For a given distance from the grain boundary plane, the parameter ε describes the contribution of volumetric diffusion from the grain boundary to the crystal bulk. The parameter Δ shows how much diffusion along grain boundaries is accelerated compared with bulk diffusion.

The equation for the concentration of diffusant in the grain $c_g(y, z, \tau)$ can be written in abbreviated form:

$$c_g(\varepsilon, \eta, \beta) = c_1(\eta) + c_2(\varepsilon, \eta, \beta), \quad (3.3)$$

where c_1 is the contribution of direct volumetric diffusion $\bar{c}(z, \tau)$ (average y value $c_g(y, z, \tau)$) from a source with a constant concentration to the bulk in the direction z , and c_2 is the contribution of the grain boundary.

Suzuoka [20] obtained an exact solution for the diffusion equation from an "instantaneous" source (or thin-film). Since c_1 decreases much faster than c_2 in an area far from the source, $c_1 \ll c_2$. Therefore, almost the entire diffusant located in this region comes there by volumetric diffusion from the grain boundary and, therefore, far from the diffusion source $\bar{c} = \bar{c}_2$.

The numerical method first applied in the approach of Levin and McCallum [21] showed that far from the surface with grain-boundary diffusion, the quantity $lg\bar{c}$ linearly depends on $z^{6/5}$, rather than on z ; using the advantage of this approach, Suzuoka formulated the following relationship between the slope of the graphs in coordinates $lg\bar{c}_2 - z^{6.5}$ and the parameter β for the source of finite thickness:

$$-\partial lg\bar{c}_2 / \partial z^{6/5} = 0.314 \cdot \beta^{-0.592}. \quad (3.4)$$

For the method of stripping layers, Le Clair established a very important relationship between δD_b , D_V , τ and the slopes of the average concentration profile z^n and $(\eta\beta^{-1/2})^n$, where n is a rational number (not necessarily a natural number):

$$\delta D_b = 2(D_V/\tau)^{1/2}(-\partial \ln \bar{c} / \partial z^n)^{-2/n}[-\partial \ln \bar{c} / \partial (\eta\beta^{-1/2})^n]^{2/n}. \quad (3.5)$$

Minus signs appear in front of the values in parentheses because the values themselves are negative.

For practical reasons, it was necessary to determine the value of the exponent n , at which the dependences $\ln \bar{c}$ on z^n and $\ln \bar{c}$ on $(\eta\beta^{-1/2})^n$ in the region of grain boundary diffusion are straight lines. In this case, the inclinations $-\partial \ln \bar{c} / \partial z^n$ and $-\partial \ln \bar{c} / \partial (\eta\beta^{-1/2})^n$ remain constant over the entire range of penetration depths where grain-boundary diffusion prevails, which facilitates the determination of the product δD_b . Expression (3.5) is valid for all solutions, regardless of the type of diffusion source, since it contains only the definitions η and β from equations (3.2).

In Whipple's exact solution for a diffusion source with a constant concentration in equation (3.5), $n = 6/5$

$$-\partial \ln \bar{c} / \partial (\eta\beta^{-1/2})^{6/5} = 0.78 \quad (3.6)$$

and equation (3.5) takes the form:

$$\delta D_b = 0.3292(D_V/\tau)^{1/2}(-\partial \ln \bar{c} / \partial z^{6/5})^{-5/3}. \quad (3.7)$$

For diffusion from an "instantaneous" source, Suzuoka found that in the area of grain boundary diffusion

$$-\partial \ln \bar{c} / \partial (\eta\beta^{-1/2})^{6/5} = 0.72 \cdot \beta^{0.008}. \quad (3.8)$$

The value $n = 6/5$ chosen by Suzuoka, was first proposed by Levin and McCallum for diffusion from a constant-concentration source into a polycrystalline sample and was well suited for the solution with an "instant" source.

Thus, the sought exponent in equation (3.5) is $n = 45$ for both the source with constant concentration and the "instantaneous" source.

In [20], simplified versions of equations for calculating the δD_b product using the solution of Suzuoka for an "instantaneous" source in various ranges of β parameter are given: 1) $\beta < 10^2$; 2) $10^2 < \beta < 10^4$; 3) $\beta < 10^4$.

1) For $\beta < 10^2$:

$$\delta D_b = 0.258 D_V^{91/194} \tau^{-103/194} (-\partial lg\bar{c} / \partial z^{6/5})^{-509/291}. \quad (3.9)$$

2) For $10^2 < \beta < 10^4$:

$$\delta D_b = 0.296 D_V^{585/119} \tau^{-121/138} (-\partial lg\bar{c} / \partial z^{6/5})^{-209/119}. \quad (3.10)$$

When deriving equations (3.9) and (3.10), equation (3.5) is used for $n = 65$, and for the expression $(-\partial \ln \bar{c} / \partial (\eta\beta^{-1/2})^{6/5})$ in the case $\beta < 10^2$ the relation obtained by Suzuoka is

$$-\partial \ln \bar{c} / \partial (\eta\beta^{-1/2})^{6/5} = 0.70\beta^{0.018}. \quad (3.11)$$

and in the case of the interval $10^2 < \beta < 10^4$, an improved, compared with the Suzuoka ratio [20],

$$-\partial \ln \bar{c} / \partial (\eta\beta^{-1/2})^{6/5} = 0.74\beta^{0.005}. \quad (3.12)$$

Since the gradient on the left-hand side of (3.11) and (3.12) with $\beta \rightarrow \infty$ gradually approaches 0.78, the condition $\beta < 10^4$ is proposed in [2] to use the equality

$$-\partial \ln \bar{c} / \partial (\eta\beta^{-1/2})^{6/5} = 0.775. \quad (3.13)$$

3) For $\beta < 10^{-4}$, taking into account (3.13), the following formula is obtained for the calculation of δD_b :

$$\Delta = D_b / D_V,$$

whence follows

$$\delta D_b = 0.3257(D_V/\tau)^{1/2}(-\partial l \bar{g} / \partial z^{0.5})^{-5/3}. \quad (3.14)$$

Since we do not know a priori which of the three intervals β indicated above should be applied in each particular case, we will test all three hypotheses of the intervals β as competing, testing them using equation (2.11), which is valid if grain-boundary diffusion dominates over volumetric diffusion for any values β , and expressing the relationship of shrinkage $y = \Delta l / l_0$ with D_b at $t = \tau$, $b = \delta$:

In the case of $\beta < 10^2$.

$$\frac{\Delta l}{l_0} \cong (2.14\gamma\Omega\delta D_b / kTr_g^4)^{0.33}\tau^{0.33}. \quad (3.15)$$

Taking into account (3.4) and the expression β from (3.2), equation (3.3) recommended for calculation of δD_b at $\beta < 10^2$, can be written in the form:

$$\begin{aligned} \delta D_b &= \\ &= \frac{0.258 \cdot D_V^{0.6319}}{D_V^{1.5 \cdot 1.0172}} \cdot \frac{\tau^{-0.5309}}{\tau^{0.5 \cdot 1.0172}} \left(\frac{0.314}{2} \right)^{1.0172} \cdot (\delta D_b)^{1.0172} \end{aligned}$$

where do we find

$$\delta D_b^{0.0172} = 25.479 \cdot D_V^{0.8939} \cdot \tau^{0.023}.$$

Solving the last equation with respect to δD_b , we obtain

$$\delta D_b = 13.11 \cdot 10^7 D_V^{51.971} \cdot \tau^{1.3372}. \quad (3.16)$$

On the other hand, solving equation (3.15) with respect to δD_b , we obtain:

$$\delta D_b = \left(\frac{\Delta l}{l} \right)^{3.03} \cdot \frac{kTr_g^4}{2.14\gamma\Omega\tau}. \quad (3.17)$$

Equating the right-hand sides of equalities (3.16) and (3.17), we arrive at the equation

$$\begin{aligned} \left(\frac{\Delta l}{l} \right)^{3.03} \cdot \frac{kTr_g^4}{2.14\gamma\Omega\tau} &= \\ &= 13.11 \cdot 10^7 \cdot D_V^{51.971} \cdot \tau^{1.3372}, \end{aligned} \quad (3.18)$$

solving this for D_V , we obtain

$$\begin{aligned} D_V &= \\ &= 7.56 \cdot 10^{-9} \cdot \left(\frac{\Delta l}{l} \right)^{3.03} \cdot \left(\frac{kTr_g^4}{\gamma\Omega} \right)^{0.0192} \cdot \tau^{-0.0257}. \end{aligned} \quad (3.19)$$

Dividing equation (3.17) by D_V from (3.19), we obtain the following expression for the ratio $\Delta = D_b / D_V$:

$$\Delta = \frac{1}{2.148} \left(\frac{\Delta l}{l} \right)^{2.9717} \cdot \left(\frac{kTr_g^4}{\gamma\Omega} \right)^{0.09808} \cdot \tau^{-0.9743}. \quad (3.20)$$

In the case of $10^2 < \beta < 10^4$.

In view of (3.4), equation (3.10) for calculating δD_b with $10^2 < \beta < 10^4$ is written in the form:

$$\begin{aligned} \delta D_b &= \\ &= 0.2968 \cdot D_V^{4.916} \cdot \tau^{-0.5084} \cdot (0.314)^{-1.68} \cdot \beta^{0.9949}. \end{aligned} \quad (3.21)$$

Substituting the expression for β from (3.2) into (3.21) and setting $0.9949 \approx 1$, we obtain

$$\begin{aligned} \delta D_b &= \\ &= 0.2968 \cdot D_V^{4.916} \cdot \tau^{-0.5084} \cdot (0.314)^{-1.68} \cdot \frac{\delta D_b}{2D_V^{1.5} \cdot \tau^{0.5}}, \end{aligned} \quad (3.22)$$

where do we find

$$D_V = 0.9932 \cdot \tau^{0.295} \approx \tau^{0.295}. \quad (3.23)$$

Since $D_b / D_V = \Delta$, then (3.22) can be written as:

$$\begin{aligned} D_b &= \\ &= 0.1484 \cdot D_V \cdot \tau^{-1.0084} \cdot (0.314)^{-1.68} \cdot \Delta. \end{aligned} \quad (3.24)$$

Substituting the expression for D_V from (3.23) into (3.24) we find

$$D_b = 1.0089 \cdot \Delta \cdot \tau^{0.295} \approx \Delta \cdot \tau^{0.295}. \quad (3.25)$$

It is easy to see that approximate equality follows from (3.25) and (3.23) $D_b / D_V \approx \Delta$.

In the case of $\beta > 10^4$.

In view of (3.4), equation (3.14) can be written as

$$\begin{aligned} \delta D_b &= \\ &= 0.3257 \cdot D_V^{0.5} \cdot \tau^{-0.5} \cdot (0.314)^{-1.67} \cdot \beta^{0.9866}. \end{aligned}$$

Substituting the expression (3.2) for β and setting $0.9866 \approx 1$, we obtain

$$\begin{aligned} \delta D_b &= \\ &= 0.3257 \cdot D_V^{0.5} \cdot \tau^{-0.5} \cdot (0.314)^{-1.67} \cdot \frac{\delta D_b}{2D_V^{1.5} \cdot \tau^{0.5}}, \end{aligned} \quad (3.26)$$

where do we find

$$D_V = 1.1262 \cdot \tau^{-1}. \quad (3.27)$$

Since $\Delta \approx D_b/D_V$, equation (3.26) can be written as

$$\delta D_b = 0.3257 \cdot D_V^{0.5} \cdot \tau^{-0.5} \cdot (0.314)^{-1.67} \cdot \frac{\delta}{2} \cdot \frac{\Delta}{2D_V^{0.5} \cdot \tau^{0.5}}$$

where do we find

$$D_b = 1.1262 \cdot \Delta \cdot \tau^{-i} \tag{3.28}$$

To test the hypotheses about the above three intervals β in a specific sintering procedure, we propose the following methodology. Let the solid-phase sintering process consist of n successive stages of annealing with temperatures T_i and durations τ_i ($i = 1, \dots, n$). The values of D_b , D_V and Δ corresponding to these steps are denoted by $(D_b)_i$, $(D_V)_i$ and Δ_i . For definiteness, we assume that throughout the entire sintering process under consideration, either the hypothesis $H_1: \beta < 10^2$ or the hypothesis $H_2: 10^2 < \beta < 10^4$ is true for β the parameter. The D_b , D_V values correspond to the hypotheses H_j ($j = 1, 2$); at Δ in the i -th stage of sintering, we denote $(D_b)_{i,j}$, $(D_V)_{i,j}$ and $\Delta_{i,j}$. When $j = 1$, the values $(D_V)_{i,j}$ are calculated by the formula (3.19) with $T = T_i$, $\tau = \tau_i$, $\Delta l/l = (\Delta l/l)$, $((\Delta l/l)$ is the shrinkage of the sample upon annealing with temperature T_i and duration τ_i), and $(D_b)_{i,j}$ by the formula

$$(D_b)_{i,j} = \Delta_{i,j} \cdot (D_V)_{(i,j)}, \tag{3.29}$$

where $\Delta_{i,j}$ is determined by the formula (3.20) with, $T = T_i$, $\tau = \tau_i$, $\Delta l/l = (\Delta l/l)$.

The quantities $(D_b)_{i,j}$, $(D_V)_{i,j}$ and $\Delta_{i,j}$ with $j = 2$ are determined in a similar way; the difference is that $(D_V)_{i,j}$ are determined now by the formula (3.23), and $(D_b)_{i,j}$ by the formula (3.25) with $\Delta_{i,j}$ determined by the formula:

$$\Delta_{i,j} = (D_b)_{i,j} / (D_V)_{i,j}. \tag{3.30}$$

We denote by $f_i(x)$ the probability density of the distribution of the value $(D_b)_{i,j}$ ($i = 1, \dots, n$), $j = 1, 2$, the approximate value of which is calculated by the histogram of values $(D_b)_{i,j}$ determined by formulas (3.17) and (3.25).

According to Fisher [19], the measure of information, the logarithm of the likelihood ratio $\log[f_1(x)/f_2(x)]$ is defined as information at the point x that must be distinguished in favor of H_1 versus H_2 ; the base of the logarithms is not significant, unless we are interested in the unit of measure-

ment; natural logarithms (base e) are usually used. The average information from an observation in favor of H_1 versus H_2 regarding a measure $\lambda(x)$ is defined as

$$I(1:2) = \int f_1(x) \log \frac{f_1(x)}{f_2(x)} d\lambda(x) \tag{3.31}$$

and the average information from observation in favor of H_2 versus the H_1 measure of H_1 as

$$I(2:1) = \int f_2(x) \log \frac{f_2(x)}{f_1(x)} d\lambda(x). \tag{3.32}$$

In addition, to determine the measure of discrepancy between two populations with densities $f_1(x)$ and $f_2(x)$, respectively, a minimum of discriminating information $I(1:2)$ is introduced, defined as the minimum value of expression (3.31) for given $f_2(x)$ and all such $f_1(x)$ for which $T(x)$ is a measurable statistics, so that $\Theta = \int T(x) f_1(x) d\lambda(x)$ and $M_2(\tau) = \int f_2(x) e^{\tau T(x)} d\lambda(x)$ exists for τ a certain interval.

For normal distributions $N(\Theta_j, \delta_j^2)$ ($j = 1, 2$)

$$I(2:1) = \frac{n(\Theta_1 - \Theta_2)^2}{2\sigma_2^2} + \frac{n-1}{2} \left(\log \frac{\sigma_2^2}{\sigma_1^2} - 1 + \frac{\sigma_1^2}{\sigma_2^2} \right). \tag{3.33}$$

The larger the value $I(2:1)$, the less "similarity" between the sample with density $f_1(x)$ and the population with density $f_2(x)$. Suppose, we have an observation x , which is a sample of independent observations n , and we want to test the null hypothesis H_2 that the observation belongs to a population with a density $f_2(x)$ with the alternative hypothesis H_1 that the observation belongs to a population with a density $f_1(x)$.

Taking $T(x) = \log(f_1(x)/f_2(x))$ as statistics, we will have

$$I(1:2) - I(2:1) = \log \frac{f_1(x)}{f_2(x)}. \tag{3.34}$$

Therefore, the critical region for testing the H_2 hypothesis is determined by the inequality

$$\log \frac{f_1(x)}{f_2(x)} \geq c, \tag{3.35}$$

where c is the given small positive number. Moreover, according to the main Neumann-

Pearson lemma, (3.35) determines the most powerful critical region. The hypothesis H_2 is rejected if inequality (3.35) holds.

Similar to the comparison of the hypotheses $H_1: \beta > 10^2$ and $H_2: 10^2 < \beta < 10^4$ a comparison of the hypotheses is performed $H_1: 10^2 < \beta < 10^4$ and $H_2: \beta > 10^4$. Now $(D_V)_{i,1}$ and $(D_V)_{i,2}$ are determined by formulas (3.23) and (3.27), respectively, and $(D_b)_{i,1}$ and $(D_b)_{i,2}$ — by formulas (3.24) and (3.28), respectively.

In general, for the entire sintering process, the diffusion coefficients (grain-boundary, bulk, and surface) are represented in accordance with the Arrhenius law [23, 16] in the form of equality

$$D = D_0 \cdot \exp\left(-\frac{\Delta H}{k_B T}\right) \quad (3.36)$$

or equality

$$D = D_0 \cdot \exp\left(-\frac{Q}{RT}\right), \quad (3.37)$$

where R is the universal gas constant; D_i is the pre-exponential factor; ΔH is the activation enthalpy; Q is the activation energy; k_B is the Boltzmann constant; T is the absolute temperature, K.

We denote by D_j the values of the diffusion coefficient D at the stage j of the sintering process with a corresponding annealing temperature T_j .

Taking the logarithm of (3.36), we obtain

$$\ln D = \ln D_0 - \frac{Q}{k} \cdot \frac{1}{T}. \quad (3.38)$$

Introducing the notation $y = \ln D$, $x = 1/T$, $c_1 = \ln D_0$, $c_2 = -Q/k$ and applying the least squares (LSM) method to the linear regression $y = c_1 + c_2 x$, we obtain

$$c_1 = \frac{\sum_{i=1}^n x_i^2 \cdot \sum_{i=1}^n y_i - \sum_{i=1}^n x_i \cdot \sum_{i=1}^n x_i y_i}{n \sum_{i=1}^n x_i^2 - (\sum_{i=1}^n x_i)^2}, \quad (3.39)$$

$$c_2 = \frac{-\sum_{i=1}^n x_i \cdot \sum_{i=1}^n y_i + \sum_{i=1}^n x_i y_i}{n \sum_{i=1}^n x_i^2 - (\sum_{i=1}^n x_i)^2}. \quad (3.40)$$

In the notation used for equation (3.38), formulas (3.39) and (3.40) are written in the form

$$\ln D_0 = \frac{\sum_{i=1}^n \frac{1}{T_i^2} \cdot \sum_{i=1}^n \ln D_i - \sum_{i=1}^n \frac{1}{T_i} \cdot \sum_{i=1}^n \frac{1}{T_i} \cdot \ln D_i}{n \sum_{i=1}^n \frac{1}{T_i^2} - (\sum_{i=1}^n \frac{1}{T_i})^2}, \quad (3.41)$$

$$\frac{Q}{k_B} = \frac{-n \sum_{i=1}^n \frac{1}{T_i} \cdot \ln D_i + \sum_{i=1}^n \frac{1}{T_i} \cdot \sum_{i=1}^n \frac{1}{T_i} \cdot \ln D_i}{n \sum_{i=1}^n \frac{1}{T_i^2} - \left(\sum_{i=1}^n \frac{1}{T_i}\right)^2}. \quad (3.42)$$

Using formulas (3.41) and (3.42), the annealing results D_i , T_i ($i = 1, \dots, n$) can be used to calculate the pre-exponential factor D_0 and activation energy for diffusion of any kind (grain-boundary, volumetric, and surface) for both single-component and multi-component systems.

4. Determining equations of the processes of solid-phase and liquid-phase sintering of a powder mixture and their numerical solutions

We denote by $\Delta_{Ij} = (t_{j-1}, t_j)$ ($j = 1, \dots, n$) the time intervals of the solid-phase (non-isothermal) sintering with a temperature T_j maintained over an interval $\Delta_{I,j}$ of duration $\tau_{I,j} = t_j - t_{j-1}$. We denote by $\Delta_2 = (t_n, t_n + \tau_2)$ the time period of liquid-phase (isothermal) sintering with a duration τ_2 at which a constant temperature T_C is maintained. It is assumed that the temperatures T_j ($j = 1, \dots, n$) and T_C do not change over the entire volume of the furnace space with a temperature equal to the temperature of the sintered material; and at each interval $\Delta_{I,j}$, the temperature T_j rises with a rate of $w_j > 0$. The porosity and average grain sizes in the interval Δ_{Ij} ($j = 1, \dots, n$) are denoted by Π_{Ij} and L_{Ij} , and Δ_2 by Π_2 and L_2 , respectively.

Taking into account the relation (2.2) between porosity Π_C and volumetric shrinkage $\Delta V/V_0$ obtained in Section 2, on the basis of equations (2.13), the equation for kinetics of material compaction at the j -th ($j = 1,$

..., n) temperature stage is written in the form:

$$\frac{d\Pi_{1,j}}{dt} = -\frac{6(1 - \Pi_{1,j})^{3/2} \cdot 3^{2.1}}{(1 - \Pi_0)^{1/2} \left[1 - \left(\frac{1 - \Pi_0}{1 - \Pi_{1,j}} \right)^{1/2} \right]^{2.1}} \times \left\{ \frac{2\gamma\Omega D_{V,j}}{3kT_j r_g^3} \left[1 - \left(\frac{1 - \Pi_0}{1 - \Pi_{1,j}} \right)^{1/2} \right] + \frac{\gamma\Omega b D_{b,j}}{2kT_{1,j} r_g^4} \right\} \quad (4.1, a)$$

and for the first 3.5 percent shrinkage — in the following form:

$$\frac{d\Pi_{1,j}}{dt} = -\frac{6(1 - \Pi_{1,j})^{3/2} \cdot 3^{2.06}}{(1 - \Pi_0)^{1/2} \left[1 - \left(\frac{1 - \Pi_0}{1 - \Pi_{1,j}} \right)^{1/2} \right]^{2.06}} \times \left\{ \frac{2.63\gamma\Omega D_{V,j}}{3^{1.03} kT_j r_g^3} \left[1 - \left(\frac{1 - \Pi_0}{1 - \Pi_{1,j}} \right)^{1/2} \right]^{1.03} + \frac{0.70\gamma\Omega b D_{b,j}}{2kT_{1,j} r_g^4} \right\}, \quad (4.1, b)$$

where $D_{b,j}$ and $D_{V,j}$ are the coefficients of grain boundary and volumetric diffusion, m^2/sec ; Π_0 is the porosity before sintering.

The equation for the kinetics of grain growth of the material at the j -th ($j = 1, \dots, n$) temperature regime, based on equation (2.15), can be written in the form:

$$\frac{dL_{1,j}}{dt} = \frac{B_1\gamma D_{S,j}\delta^4}{4L_{1,j}^3 kT_j}, \quad (4.2)$$

where $D_{S,j}$ is the coefficient of surface diffusion; $L_{1,j} - L_0$ are the thickness of the layer in which surface diffusion occurs, which is usually taken of the order of the atomic diameter δ ; B_1 is the numerical constant approximately equal to 30 [17].

For determination of equations of solid-phase sintering, along with equations (4.1) and (4.2), the following equations are used: — equation for temperature rise

$$\frac{dT_j}{dt} = w_{T_j}, \quad w_{T_j} = (T_j - T_{j-1})/(\tau_{1,j}), \quad t_{j-1} < t \leq t_j \quad (4.3)$$

— initial conditions

$$\Pi|_{t=0} = \Pi_0, \quad L|_{t=0} = L_0, \quad T|_{t=0} = T_0, \quad (4.4)$$

— conditions for conjugation of temperature stages in time

$$\Pi_{1,j}|_{t=t_{j-1}} = \Pi_{1,j-1}, \quad L_{1,j}|_{t=t_{j-1}} = L_{1,j-1}, \quad T_j|_{t=t_{j-1}} = T_{j-1}, \quad j=2, \dots, n \quad (4.5)$$

The conditions (4.5) are used for the "fitting" of solutions obtained at individual temperature stages in order to obtain a continuous solution. The solutions for porosity $\Pi_{1,j}$ and granularity $L_{1,j}$ obtained for $j = n$ in this way are used as initial data for the description of liquid-phase sintering.

The kinetics of compaction at the stage of liquid-phase sintering will be analyzed based on the rheological description of the deformation of the porous structure, in which diffusion-viscous flow and compaction of a porous body in a homogeneous stress state under conditions of uniform compression are considered [23].

Taking into account the dependence of the shear viscosity coefficient of a porous body η on porosity Π [24].

$$\eta = \eta_0(1 - \Pi)^{5/3}, \quad (4.6)$$

the differential equation of a porous body can be written in the form:

$$\frac{d\Pi}{dt} = -\frac{3}{4} \cdot \frac{\Pi}{(1 - \Pi)^{5/3}} \cdot \frac{P}{\eta_0} \quad (4.7)$$

where P is the hydrostatic pressure.

For small porosities, we can restrict ourselves to a linear dependence η on Π , assuming $(1 - \Pi)^{5/3} \approx 1 - 5/3 \cdot \Pi$. Then we obtain the following law of compaction of the porous body:

$$\frac{d\Pi}{dt} = -\frac{3}{4} \cdot \frac{\Pi}{1 - \frac{5}{3}\Pi} \cdot \frac{P}{\eta_0} \quad (4.8)$$

Based on equation (4.7), the kinetics of material compaction at the stage of liquid-phase sintering is written in the form of a differential equation:

$$\frac{d\Pi_2}{dt} = -\frac{\Pi_2}{(1 - \Pi_2)^{5/3}} \cdot \frac{3P}{4\eta_0}, \quad t \in \Delta_2 \quad (4.9)$$

and the kinetics of grain growth of the material, in accordance with equation (2.15), is expressed by a differential equation

$$\frac{dL_2}{dt} = \frac{B_1\gamma D_{s,n}\delta^4}{4L_2^3 \cdot kT_c} \quad t \in \Delta_2, \quad (4.10)$$

where

$$D_{s,n} = D_{s,0} \cdot \exp\left(-\frac{Q_s}{RT_c}\right) \quad (4.11)$$

Q_s is the activation energy of grain boundary diffusion.

The basic equations of the liquid phase sintering process, in addition to equations (4.9), (4.10), include the initial conditions at $t = t_n$, which are written in the form

$$\Pi_2|_{t=t_n} = \Pi_{1,j}|_{t=t_n}, L_2|_{t=t_n} = L_{1,j}(t)|_{t=t_n}; \quad (4.12)$$

$$T_c = T_n.$$

The solution of equations (4.1)–(4.5) and (4.10)–(4.12) was performed using the Runge-Kutta method (fourth order of accuracy) for a system of differential equations of the first order.

Thus, the system under study for sintering a powder mixture of the three components of iron, carbon and glass, is divided into two half-systems — gray cast iron (iron/carbon) and fayalite (iron/sitall-glass), for which it is easy to calculate the volumetric, surface, and grain-boundary diffusion coefficients separately, reducing it to the consideration of the gray cast iron/fayalite binary system and the calculation of its diffusion coefficients according to the Darken formula.

This approach extends to composite systems from any number of components of the powder mixture. It is enough to divide the system into a finite number of subsystems of approximately equal power (i.e., the number of constituent components) for which the pre-exponential coefficients of the Arrhenius formula for the coefficients of volumetric, surface, and grain-boundary diffusion can be calculated. However, some components of the system may be included in several subsystems. Then, step-by-step clustering is applied to the subsystems using one of the well-known methods of irreversible aggregation. In this case, the "particle + cluster" method is the most successful, according to which small "particles" (in our case, low-power subsystems) are attached to large "particles" (a higher-power subsystem). At each step of clustering, in essence, a binary system will be considered, the diffusion coefficients of which are calculated by the Darken formula. At the final step of the described procedure, we obtain a cluster that coincides with the initial sintering system.

5. Conclusions

The proposed method for numerically simulating the sintering process of multi-component metal-ceramic composite materials is based on the governing equations describing the kinetics of compaction and grain growth of the material for the solid-phase and liquid-phase stages of the sintering process, taking into account volumetric, surface and grain boundary diffusion, as well as mechanisms of mutual diffusion, is proposed.

To calculate the diffusion coefficients from experimental annealing data, a statistical procedure for comparing hypotheses was developed to determine the interval of application of the parameter associated with the ratio of the volume and grain boundary diffusion coefficients.

The applicability of the Zener theory to calculating the pre-exponential factor of the diffusion coefficient for metal alloys is shown, and the corresponding numerical calculations for fayalite (an alloy of iron with glass) are performed. Using the alloy "gray cast iron + fayalite" as an example, the diffusion mechanism of sintering of their powder mixture is analyzed and the dependences of the coefficients of volumetric, surface, and boundary diffusion on the sintering temperature are determined. The coefficient of mutual diffusion of this alloy, which is considered as a binary system, is calculated.

The proposed approach to numerical modeling the sintering process in the multi-component composite systems can be applied to an arbitrary number of constituent components.

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