

DETERMINING THE RHEOLOGICAL PROPERTIES OF COMPRESSIBLE
POWDER MATERIALS IN THE HIGH-TEMPERATURE RANGE

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We discuss the behavior of powder materials in the case of high-temperature deformation. We have ascertained rheological variables and the possible types of rheological curves for the case of unilateral compression. We propose methods for the determination of the rheological properties, based on experimental data.

Under conditions of high-temperature deformation powder materials are capable of flow, experiencing in this case great irreversible strains. It is precisely this property that is exploited in a variety of engineering processes in the field of powder metallurgy where materials are subjected to heat treatment under pressure [1]. Macroscopic flow in a material occurs under various conditions of self-propagated high-temperature synthesis, and these include the combustion of the original powder billets and the deformation of the products of combustion, which form to make up a hot porous mass [2].

Material that has been brought to the high-temperature state has been subjected to relatively little study in rheology, although familiarity with the properties of a hot porous mass is important from the standpoint of manufacturing which involves the use of SVS technology and hot pressing. The development of efforts in this area is held back by the absence of specialized equipment and methods of measuring the properties of such materials.

Among the specific unique features of the materials in the class which we are considering here we can include the following: first of all, in the area of high temperatures a powder billet heated from without or by means of an internal source represents a porous bonded structural frame which is formed on heating by the interaction of individual particles. Secondly, the existence of a large number of pores (up to 50%) and the change in their number during the deformation process defines the relationship between the shear resistance and compression of these materials relative to porosity. Thirdly, the material of the structural frame that is formed in the range of premelting temperatures exhibits considerable resistance to deformation.

By using the concepts of highly viscous compressible fluids, as applicable to such materials, we were able to study the quantitative relationships governing the flows of such materials in sintering processes and heat treatment under pressure [3, 4]. It is assumed here that the increased density of a porous body comes about as a consequence of the disappearance of pores from out of the volume of the material as a result of the fact that a certain amount of incompressible material is floating about in these pores, and it is this material that combines to form the framework of the porous body.

The mechanical behavior of porous materials subjected to high temperatures can be described by a rheological equation of state having the following form [5]:

$$\sigma_{ij} = \eta(T, \rho, \eta_1, I_k)(e_{ij} - \delta_{ij} \text{tr } e_{ij}/3) + \zeta(T, \rho, \eta_1, I_k) \delta_{ij} e_{ij}, \quad (1)$$

in which η and ζ are the shear and volume viscosities which are functions of the temperature T , the relative density $\rho = 1 - \Pi$, the magnitude of the shear viscosity η_1 of the material making up the framework, and finally, of the strain parameters. The latter are taken into consideration in the form of functions dependent on three principal invariants of the strain rate tensor I_k ($k = 1, 2, 3$). The quantity η_1 may also be a function of the strain parameters, as well as of the unique features of the material structure.

If we have no volumetric viscosity nor dependence on porosity, then Eq. (1) describes the rheological behavior of purely viscous incompressible fluids. The problems of the flow of such a class of materials have been thoroughly studied within the scope of theoretical and experimental rheology as it pertains to polymer materials [6]. For compressible highly viscous materials it is essential to develop methods to measure the fundamental rheological parameters and develop appropriate instrumentation. In the solution of these problems it is important to bear in mind the unique features of porous materials relative to the class of incompressible fluids: the presence of at least two viscosities and their dependence on the instantaneous porosity, the nonsteadiness of the compression process in the porous mass, etc. Consideration only of the above-enumerated features compels us once again to choose among the viscosimetric flows, rheological coordinates, and methods for the solution of the inverse problem.

In the present paper we examine the simplest type of isothermal flow of a porous material of uniform density, i.e., unilateral compression in a cylindrical shaping press, free of friction at the walls. This type of flow may be regarded as viscosimetric, since it provides for the obtaining of information relative to the volumetric and shear viscosities of the material, it is described by relatively simple and easily solvable equations [3, 4, 7], and it permits experimental realization. The presence of only a nonzero component of the velocity vector v and only a single nonzero component of the strain-rate tensor $\partial v/\partial z$ is characteristic. In this case, for the invariants of the cited tensor we have

$$I_1 = \frac{\partial v}{\partial z}; \quad I_2 = \left(\frac{\partial v}{\partial z}\right)^2; \quad I_3 = \left(\frac{\partial v}{\partial z}\right)^3,$$

and any relationship to these invariants on the part of the shear or volumetric viscosity leads only to a relationship dependent on the velocity gradient $\partial v/\partial z$. The stressed state in this case is characterized by three nonzero normal components of the stress tensor: σ_z , σ_r , σ_θ .

By means of this most simple example we can trace the fundamental stages of the theoretical analysis which examines the rheological behavior of porous materials: the selection of the viscosimetric and rheological variables, the determination of the basic types of relationships between these, the manner in which these relationships are affected by the rheological properties of the material (the direct problem); the a priori determination of the unknown properties of the material, and the parameters governing the relationships of these properties to porosity, as for example, in terms of the experimentally measured viscosimetric variable (the inverse problem).

The process of consolidation, which we are studying here, is described by the well-known [4, 7] system of equations:

$$\frac{d\rho}{dt} = -\rho \frac{\partial v}{\partial z}; \quad \frac{\partial \sigma_z}{\partial z} = 0; \quad (2)$$

$$\sigma_z = \left(\frac{4}{3}\eta + \zeta\right) \frac{\partial v}{\partial z}; \quad (3)$$

$$\sigma_r = \sigma_\theta = \left(-\frac{2}{3}\eta + \zeta\right) \frac{\partial v}{\partial z}; \quad (4)$$

$$\eta = \eta_1 \left(\frac{\partial v}{\partial z}\right)^n \rho^m; \quad \zeta = \frac{4}{3} \eta_1 \left(\frac{\partial v}{\partial z}\right)^n \frac{\rho^\alpha}{1-\rho}, \quad (5)$$

$$t = 0: \rho = \rho_0; \quad H = H_0; \quad (6)$$

$$z = 0: v = 0;$$

$$z = H: \sigma_z = -N \quad \text{or} \quad v = -V.$$

In contrast to [4, 7], where we find a study of the change in the relative density over time as a function of the applied load, here we will be interested in the relationship between the stresses σ_i , the velocity v , the specimen height H , and the velocity gradient $\partial v/\partial z$ during the course of the experiment and the extent to which these are affected by the properties of the material, and namely, ρ and η_1 , as well as the exponent n of non-Newtonian flow and the exponents m and α representing the nonlinearity of the relationship between the properties and the relative density.

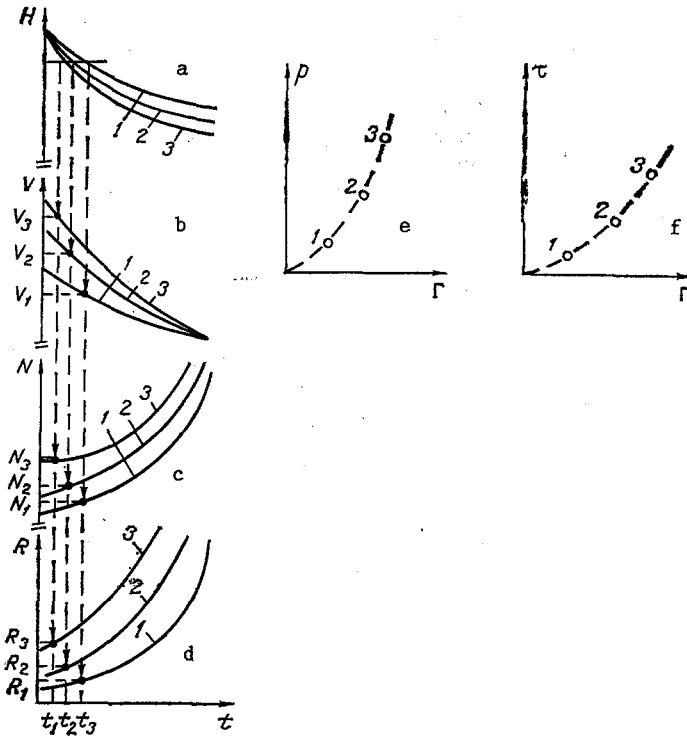


Fig. 1. Procedure for the construction of rheological curves $p(\Gamma)$ and $\tau(\Gamma)$ on the basis of data derived from the viscosimetric experiment.

The analysis conducted in [7] makes it possible, in the case of a density that is uniform through the volume, to regard all of the remaining characteristics of the flow to be uniform as well: components of the stress tensor σ_z , σ_r , σ_θ , the velocity v , and its gradient. In this case, expressing all of the variables in terms of the boundary values, we can write the solution for the system of equations (2)-(6) in the form

$$N = \frac{4}{3} \eta_1 \left(\rho^m + \frac{\rho^\alpha}{1-\rho} \right) \left(\frac{V}{H} \right)^{n+1}; \quad \rho = \rho_0 H_0 / H; \quad (7)$$

$$R = \frac{2}{3} \eta_1 \left(-\rho^m + \frac{2\rho^\alpha}{1-\rho} \right) \left(\frac{V}{H} \right)^{n+1}. \quad (8)$$

Selection of Rheological Variables. Relationships (7) and (8) suggest a set of four viscosimetric variables which must be measured experimentally: the guidepunch pressure N , the lateral pressure R , the specimen height H , all directly connected to the relative density ρ : $\rho H = \rho_0 H_0$, and the rate of change V in this height. Considering that $V = dH/dt$, we can assume that the measured functions $N(t)$, $R(t)$, $H(t)$ are adequate to determine the rheological properties.

From the above-enumerated set of viscosimetric variables we then have to find the so-called rheological variables whose relationship one to the other can be used to calculate the properties and it would be sensitive to changes in the rheological parameters n , m , α . The properties determined in this manner need not depend on the form of the stressed state, the deformation time, nor on the geometric dimensions of the measurement unit. For an incompressible material these variables include the shearing stress and velocity, while the relationship between these (the curve of the flow) determines the shearing viscosity and its relationship to the deformation parameters [6].

For porous materials, as follows from Eqs. (3), (4), and (7) and (8), we can at least assume three rheological variables: hydrostatic pressure p , the maximum magnitude of the tangential stress τ , and the velocity gradient Γ , which in the case of unilateral compression, such as we are considering here, represents the rate of change $\text{div} v$ in the volume and the strain rate in the direction of the pressing axis, simultaneously:

$$\rho = \sum_{i=1}^3 |\sigma_i|/3; \tau = |\sigma_z - \sigma_r|/2; \Gamma = |V/H|. \quad (9)$$

Let us note that the rheological variables in (9), accurate to the constant factor, represent the first two invariants of the stress tensor and the first invariant of the strain-rate tensor.

The function $\tau(\Gamma)$ determines the shearing viscosity of the porous body, while $p(\Gamma)$ defines the volumetric viscosity. The ratios τ/Γ and p/Γ , much like the assumptions in the rheology of incompressible fluids, must be treated as definitions of an effective means of measuring the resistance in shear and multilateral compression, i.e., the effective values of the shearing and volume viscosities η_e and ζ_e .

A study of such other relationships as, for example, $N(H)$, $N(V)$, $R(H)$, $V(H)$, is also of practical interest, since these are characteristics of a given method for the consolidation of a porous body. However, in the interpretation of these relationships it should be borne in mind that a change occurs along the time characteristic of the process which we are examining here, as well as of porosity, velocity, etc. Moreover, the characteristic which we are analyzing contains within it, in complex fashion, a combination of the volumetric and shearing viscosities of the material.

Analysis of Rheological Curves. Since the viscosity of the porous material depends on its density, for purposes of determining said viscosity it is desirable to construct flow curves $p(\Gamma)$ and $\tau(\Gamma)$, along which the density of the material would remain constant. It is possible to obtain such curves in a series of j experiments on the consolidation of this material, changing in each of these the initial value only for one of the parameters N , V , or H_0 . In Fig. 1 we find a graphic illustration of the procedure for the construction of the rheologic curves for the case in which $j = 3$.

Based on the $H(t)$ curves we select some value for the specimen height H which corresponds in this particular case to a specific value for the relative density ρ (Fig. 1a), and at the appropriate instant of time t_j (the time at which the chosen quantity H is attained in each of the experiments) from the curves $V(t)$, $N(t)$, $R(t)$ we determine the quantities V_j , N_j , R_j (Fig. 1b-d), needed for the calculation of the rheologic parameters p , τ , and Γ on the basis of (9). Thus we obtain the rheologic curves $p(\Gamma)$ (Fig. 1e) or $\tau(\Gamma)$ (Fig. 1f), along which the density is constant.

Repeating this procedure for the other value of the billet height or for its relative density, we might obtain a family of rheologic curves with a different but unchanging density along each of these curves. Figure 2 shows possible types of rheologic curves $p(\Gamma)$ and $\tau(\Gamma)$ for a viscous porous material at various values of the non-Newtonian index n (solid lines). The dashed lines show the evolutions of the curves of flow for non-Newtonian material ($n = 0$) with an increase the selected value of the relative density ρ . The ratios τ/Γ and p/Γ , as was noted earlier, determine the quantities η and ζ and their relationship to density.

In the consolidation method being considered here, only one of the stress-tensor components may serve in the role of one of the rheological coordinates, and namely, the axial pressure, for example. The rheological curve $N(\Gamma)$, in analogy with the earlier considered $p(\Gamma)$ and $\tau(\Gamma)$, can be obtained from a series of experiments based on the above-described procedure. The qualitative form of the curves $N(\Gamma)$ for various ρ and n does not differ from the curves $p(\Gamma)$ and $\tau(\Gamma)$ shown in Fig. 2. However, the ratio N/Γ determines a certain effective quantity, i.e., the extent of resistance in the case of unilateral compression. According to (3), this quantity, in additive fashion, is made up of both of the viscosities. Nevertheless, if it is possible to choose the appropriate parameters of the exponential function (5) for their relationship to the relative density, then on the basis of the function $N(\Gamma)$ we can solve the inverse problem and determine the unknown properties. In principle, with these purposes in mind, the lateral pressure R is a suitable quantity, but it is a more complex problem to measure this quantity experimentally.

The degree of nonlinearity of the extent to which the properties η and ζ are functions of density (m and α) has little effect (even quantitatively) on the rheological curves. For a qualitative analysis of the effect of density on the properties and on the quantitative relationships of the flows in such materials we can limit ourselves to the simple forms of the functions $\eta(\rho)$ and $\zeta(\rho)$, for example, by assuming that $m = 1$; $\alpha = 0$. Of fundamental

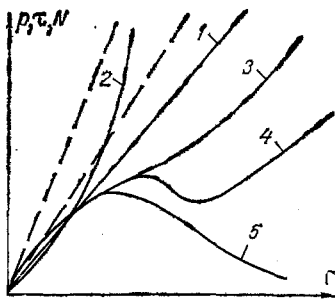


Fig. 2

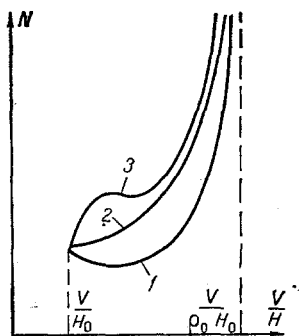


Fig. 3

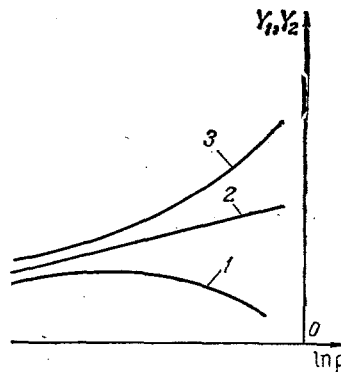


Fig. 4

Fig. 2. Possible types of rheological curves for viscous porous material at various density values (dashed lines) and for the values of the non-Newtonian indices (solid line).

Fig. 3. Viscosimetric characteristic $N(\Gamma)$ derived from one experiment under conditions of various rheological behavior of material.

Fig. 4. Representation of experimental data in rectification coordinates (12), (13).

significance is the inverse proportionality of the volumetric viscosity and the porosity of the material. It is precisely in this manner that all of the unique features of the stress-strain states are related in approximation of the nonpore state.

The non-Newtonian behavior of the material has a significant effect on the rheological curve of the material. Following [6], we will distinguish dilatational fluids whose viscosity increases as the velocity gradient V/H increases, from the pseudoplastic fluids whose viscosity diminishes with an increase in V/H . Among the former we include virtually all of the powder materials based on metals and alloys, while the latter include polymer-based fluids.

Characteristic of the dilatational materials are monotonically increasing rheological curves $p(\Gamma)$, $\tau(\Gamma)$, or $N(\Gamma)$, and this applies to the entire range of possible density values (Fig. 2, curves 1 and 2). The geometrically smaller non-Newtonian degree brings the rheological curve even closer to a straight line whose slope defines the corresponding viscosity value (Fig. 2, curve 2).

The rheological curves of the pseudoplastic materials are more diverse, i.e., among these we encounter those that are nonmonotonic (Fig. 2, curves 3-5). This diversity is brought about by the fact that the increasing stress in the material is offset by the drop in viscosity as the velocity gradient increases.

If the function $\eta(\Gamma)$ is weak and rapidly saturated, then a bending point appears on the monotonically increasing rheological curve (Fig. 2, curve 3). When the viscosity values in the case of small and large velocity gradients differ significantly, two extrema appear on the rheological curve (Fig. 2, curve 4). However, if in the range of values for Γ , such as we are considering here, the relationship is not saturated, i.e., it diminishes monotonically,

the rheological curves $p(\Gamma)$, $\tau(\Gamma)$, or $N(\Gamma)$ pass through a maximum (Fig. 2, curve 5). These quantitative relationships do not change when the selected values of the density fixed along the entire rheological curve change. Precise quantitative calculations of the coordinates of the extrema, of the bending points, and of the other characteristics of the rheological curves are possible if the form and parameters of the function $\eta(\Gamma)$ are known.

The function $N(\Gamma)$, obtained in an experiment with a constant pressing speed, can also exhibit a nonmonotonic form (Fig. 3, curves 1 and 3). The nonmonotonicity in this case is due to the offsetting influence of two factors on the viscosity: its reduction because of the increase in V/H during the consolidation process and the increase that is brought about by the reduction in porosity. The effect of the latter always leads to an increase in $N(\Gamma)$ at the end of the process, but the initial consolidation stages due to the first factor may occur with a reduction in the stresses acting within the material.

In the consolidation process occurring in dilatational materials based on metals and alloys both of these factors lead to an increase in the effective viscosity, and the $N(\Gamma)$ curves in the consolidation regime $V = \text{const}$ always increase monotonically (Fig. 3, curve 2).

The effect of the shearing viscosity of the incompressible porous mass base is always uniquely defined: the greater this quantity, the higher the force needed to achieve a given density.

Solution of the Inverse Problem. To determine the properties of the material on the basis of experimental measurement results we will use the results of the analysis that we have carried out. The initial experimental material, as was noted earlier, may be represented by the functions $N(t)$, $R(t)$, and $H(t)$ measured in each of the experiments. Differentiation of the latter yields the function $V(t)$.

In the general case, determination of the rheological properties from among the above-enumerated experimental curves is accomplished in several stages. Initially, within the range of variations in density we choose a set of values for ρ_i , for each of which, by means of the above-described procedure, we construct the rheological curves $p_i(\Gamma)$ and $\tau_i(\Gamma)$. These make it possible to determine the non-Newtonian extent in dual rheological coordinates, as well as the effective value of the corresponding viscosity:

$$\begin{aligned} \ln p_i &= \ln \zeta_i + n \ln \Gamma, \\ \ln \tau_i &= \ln \eta_i + n \ln \Gamma, \end{aligned} \quad (10)$$

where the number i refers the derived value of the effective viscosity to the corresponding density. In this way we determine the relationship of the properties to the density, i.e., $\eta(\rho)$ and $\zeta(\rho)$. If these can be described exponentially in the form of (5), then in the coordinates

$$\begin{aligned} \ln \eta &= \ln \eta_1 + m \ln \rho, \\ \ln 3(1 - \rho)\zeta/4 &= \ln \eta_1 + \alpha \ln \rho \end{aligned} \quad (11)$$

we can determine the parameters of these relationships.

If we assume that the influence of the strain parameters on the viscosity of the materials can also be reduced to an exponential law analogous to the quantities $\eta(\rho)$ and $\zeta(\rho)$, then it is possible to determine the parameters of these exponential functions from only a single experiment on the consolidation of the porous material. A combination of Eqs. (7) and (8) gives us the following relationships:

$$\begin{aligned} N - R &= 2\eta_1 \rho^m \left(\frac{V}{H} \right)^{k+1}; \\ N + 2R &= 4\eta_1 \frac{\rho^\alpha}{1 - \rho} \left(\frac{V}{H} \right)^{k+1}, \end{aligned}$$

each of which contains only three unknown parameters. The latter can be determined in the appropriate rectification coordinates:

$$Y_1 \equiv \ln \frac{N-R}{(V/H)^{k+1}} = \ln 2\eta_1 + m \ln \rho, \quad (12)$$

$$Y_2 \equiv \ln \frac{(N+2R)(1-\rho)}{(V/H)^{k+1}} = \ln 4\eta_1 + \alpha \ln \rho. \quad (13)$$

Varying the as yet unknown non-Newtonian index k , we can achieve a situation in which the experimental data are represented in the form of a straight line whose slope defines the parameters m or α , with a truncated segment giving the quantity η_1 . A remarkably unique feature of this proposed method is the rectification of the experimental curve only in the event of the correct selection of the non-Newtonian index $k = n$ (Fig. 4, curve 2). If the chosen value of k is smaller than the true value ($k < n$), then in the coordinates of (12) and (13), in the region of large values for the relative density, the experimental curve will bend downward (curve 1 in Fig. 4), while if the selected value of k is greater than the true value ($k > n$), the experimental curve will bend upward (Fig. 4, curve 3).

The described methods assume that in each experiment on unilateral compression of the porous material the axial N and the lateral R pressures are measured, as is the height of the specimen and the rate of change. However, it is possible to determine the properties of the material in the measurement of only two quantities: $N(t)$ and $H(t)$. In this case the velocity is determined through differentiation of the $H(t)$ curve. The constructed $N(\Gamma)$ curves for various density values in the $\ln N - \ln \Gamma$ coordinates determine the non-Newtonian exponent n and the relationship between density and the defective resistance to unilateral compression, which in additive fashion is combined out of the shearing and volumetric viscosities of the porous bodies.

If the material exhibits non-Newtonian properties, then it is impossible to divide the measured value of the level of resistance into volumetric and shearing viscosity such as in (5). However, in view of the limited influence exerted by the parameters m and α on the rheological curve, with good approximation it is possible to determine both of the viscosities, assuming that $m = \alpha$.

In the case of non-Newtonian behavior of the material, these parameters can be determined independently of the structure of the rheological curve $N(\Gamma)$. For this we have to use the solution of problem (2)-(6) for the kinetics of change in density. An extrapolation formula of this solution was proposed in [8], on the basis of which we can write the relationship between the experimentally measured quantities in the form $[\rho = \rho_0 H_0 / H(t)]$

$$\ln \frac{1-\rho_0}{1-\rho} - \rho + \rho_0 + \frac{\rho^m - \rho_0^m}{m} - \frac{(\alpha-2)(\alpha+1)}{4(\alpha-1)} (\rho^2 - \rho_0^2) = \frac{3}{4\eta_1} \int_0^t N(t) dt.$$

When we present the experimental results in the rectification coordinates:

$$Y_3 = \frac{(\alpha-2)(\alpha+1)}{4(\alpha-1)} + \frac{1}{\eta_1} X, \quad X = \frac{3}{4(\rho^2 - \rho_0^2)} \int_0^t N(t) dt, \quad (14)$$

$$Y_3 \equiv \left(\ln \frac{1-\rho_0}{1-\rho} - \rho + \rho_0 + \frac{\rho^m - \rho_0^m}{m} \right) / (\rho^2 - \rho_0^2),$$

for the constants of the straight line $Y_3(X)$ we obtain the sought quantities η_1 , α , and m , and for best rectification the quantity m varies in limits of $2.5 < m < 3.0$.

Each of the methods described above, that is both the complete and the shortened set of measured parameters, exhibit their own advantages and shortcomings. The measurement of the complete set of parameters is an experimentally more complex problem, but the results obtained in this manner are independent of the initial theoretical concepts. On the other hand, the rather general assumptions regarding the exponential form of the sought functions, making it possible to obtain the rectification coordinates, and the simplicity of the experimental execution make up the advantages of the second method.

Let us take note of the fact that the first solution of the inverse problem of determining the rheological properties of porous materials was the method involving the curtailed number of measured parameters. The analysis conducted in [4] of the various methods of consolidating porous materials allowed us not only to estimate the shearing viscosity η_1 of the

incompressible base, but also to determine the non-Newtonian index of the flow for a large group of solid alloys. A drawback of this study involves the fixed form of the relationship between the viscosities of the porous body and the relative density. In the present study it is included among the parameters to be determined. Let us also note that the method of determining the non-Newtonian index described in the present study, in combination with other rheological parameters, is suitable for arbitrary (including fractional) values of n and this sets it advantageously apart from the method covered in [4].

NOTATION

σ_{ij} , stress tensor component; e_{ij} , strain-rate tensor component; η , shearing viscosity of the porous material; ζ , volumetric viscosity of the porous material; T , temperature; ρ , relative density of the porous material; Π , porosity; η_1 , shearing viscosity of incompressible structural frame; I_k , strain rate tensor invariants; δ_{ij} , Kronecker symbol; v , velocity of the material in the direction of the pressing axis; z , coordinate along the pressing axis; t , time; ρ_0 , relative density of the material at the initial instant of time; H_0 , original height of the billet; N , dyepunch pressure; V , dyepunch speed; n , non-Newtonian flow index; m , degree of nonlinearity for the shearing viscosity of the porous body as a function of density; α , the same, for the volumetric density; η_e , ζ_e , effective values of the shearing and volumetric viscosities.

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