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Indentation test for the investigation of high-temperature plasticity of materials

G. CSEH, N. Q. CHINH, P. TASNÁDI, A. JUHÁSZ
Department for General Physics, Loránd Eötvös University, Múzeum krt. 6-8, H-1088 Budapest, Hungary

High-temperature behaviour of materials has been investigated by indentation creep methods. Materials investigated belong to three typical groups of materials: glasses, pure metals and alloys. Indentation tests were performed with flat-ended cylindrical and hemispherical punches. It is shown that the hemispherical punch is also proper for the determination of the high-temperature creep characteristics of the materials investigated. It is also proved that there is a unique correspondence between the results obtained by the two indentation methods.

1. Introduction

It is well known that high-temperature behaviour of metallic materials can be described by the power-law creep in a wide range of the strain rate, $\dot{\varepsilon}$. During steady-state creep (secondary creep), the relationship between the strain rate, $\dot{\varepsilon}$, and the tensile stress, σ, at a constant temperature can be expressed generally by the equation

$$\dot{\varepsilon} = A\sigma^n \qquad (1)$$

where n is the stress exponent and A is a constant depending on the microstructural features and the activation energy of the deformation process.

Sometimes the deformation process is accompanied by the occurrence of a threshold stress [1]. In such cases, the creep process can be described more adequately by the use of an effective stress (the difference between the applied stress and the threshold stress) instead of the applied stress. Therefore Equation 1 should be modified into the following form

$$\dot{\varepsilon} = A(\sigma - \sigma_0)^n \qquad (2)$$

where σ_0 is the threshold stress.

Generally, the threshold stress and the parameter A depend strongly on temperature. In the case of pure metals, the value of the stress exponent, depending on the mechanism of the deformation, changes between 1 and 5. For glasses, due to the Newtonian viscous flow, the value of the stress exponent is 1.

Although for the investigation of the creep process, uniaxial tests are spread most widely, recently [2–7], indentation methods have also been applied successfully for the determination of the parameters of these processes. During indentation measurements due to a constant load, F, a cylindrical or a hemispherical punch is pressed into the surface of the sample. The indentation depth, h, of the punch is registered as a function of the elapsed time, t, so the velocity of the indentation, dh/dt, can be calculated.

The indentation creep test performed with a flat-ended cylindrical punch was proposed originally by Yu and Li in the '70s [2, 3]. Using this method for the investigation of the high-temperature plasticity of single crystals, they proved that results obtained by this method are equivalent to those obtained by the tensile creep test. Later, the method was successfully applied for measurements carried out on polycrystalline and superplastic materials [5–7]. It is worth mentioning that indentation measurements with hemispherical punches were made earlier for the determination of the viscosity of glassy materials [8–10]. However, this type of punch has been applied relatively rarely for the investigation of high-temperature creep of metallic materials.

The basic relationships used generally for the evaluation of indentation measurements with cylindrical and hemispherical punches, are summarized below.

1.1. Indentation test with a cylindrical punch

Applying constant load at constant temperature after a short initial transient, the punch penetrates at a constant impression velocity, $v = dh/dt$, into the sample. It has been proved that to compare the results of impression creep and those of uniaxial tensile tests, the impression velocity and the load must be converted into equivalent tensile stress, σ, and strain rate, $\dot{\varepsilon}$, by equations

$$\sigma = \frac{p}{k_1} \quad \text{and} \quad \dot{\varepsilon} = \frac{v}{k_2 d} \qquad (3a)$$

where $p = 4F/\pi d^2$ is the pressure just below the punch and d is the diameter of the punch, the values of the constants k_1 and k_2 vary between 3 and 3.3, and 0.7 and 1, respectively. Upto now, the validity of Equation 3a and b has been convincingly supported both experimentally and theoretically for several metals, alloys and ionic crystals [2–7].

1.2. Indentation test with a hemispherical punch

In this case, the pressure distribution varies monotonically under the punch during the indentation performed at a constant load. The penetration velocity of the hemispherical indentor decreases continuously, and therefore the results of this measurement can be hardly interpreted in the field of the creep of metals. However, indentation tests performed with a hemispherical indentor are proper for the determination of the viscosity of glasses. The theoretical interpretation of these measurements was developed by Douglas *et al.* [8] who derived the equation

$$\frac{\mathrm{d}h}{\mathrm{d}t} = \frac{3F}{16\eta}\frac{1}{[h(d-h)]^{1/2}} \tag{4}$$

by which the viscosity can be assessed from data taken experimentally. In Equation 4, η is the viscosity and the meaning of the other parameters is the same as in the case of a cylindrical punch. Direct integration of Equation 4 is difficult and its result would not be suitable for practical computations. However, supposing that $d \gg h$, Equation 4 can be rewritten in the following form

$$\frac{\mathrm{d}h}{\mathrm{d}t} = \frac{3F}{16\eta}\frac{1}{(dh)^{1/2}} \tag{5}$$

Integrating equation 5 and expressing η, we obtain

$$\eta = \frac{9}{32d^{1/2}}\frac{Ft}{h^{3/2}} \tag{6}$$

This equation can be applied relatively easily for the determination of viscosity from experimental data taken with a hemispherical punch. It is worth noting that the deviation between the results obtained by the use of Equation 6 and by that of the full theoretical form of Equation 4 can be generally neglected. In the case of a punch with diameter of 2 mm at relatively large penetration depths (0.3 mm) the two results differ only by about 5%. If the depth is only 0.05 mm, this deviation decreases to about 1%.

In this paper it is shown that a unique correspondence can be found between the results obtained by the two methods, which means that data taken by hemispherical punches can also be suitable for the determination of high-temperature creep characteristics of metallic materials.

2. Experimental procedure

Indentation measurements were made on three different materials (lead glass, pure aluminium and $AlSi_{12}Cu_1Mg_1Ni_1$ (M124) alloy) representing three typical groups of materials: glasses, pure metals and alloys. Cylindrical indentors with diameters of 0.8–3 mm, and hemispherical indentors with diameters of 1–4 mm, were applied. For all materials, the measurements were carried out in the high-temperature range, $T > 0.5T_m$, where T_m is the absolute melting point.

3. Results and discussion

To compare measurements performed with cylindrical and hemispherical punches some further formulae should be introduced. Arguing analogously to the case of the cylindrical indentor, let us define some similar parameters for the indentation performed with a hemispherical punch. Fig. 1 shows the geometry of the hemispherical indentor, where d is the diameter of the sphere, δ is the diameter of the imprint at a given indentation depth, h. During an indentation measurement, the pressure, p_s, under the punch, can be expressed as a function of the indentation depth

$$p_s = \frac{F}{(\delta/2)^2\pi} = \frac{F}{\pi(hd-h^2)} \tag{7}$$

Supposing that $d \gg h$, Equation 7 can be rewritten to the following form

$$p_s = \frac{F}{\pi hd} \tag{8}$$

In this case, the strain rate can be defined as the ratio of the impression velocity and the diameter of the imprint at the given indentation depth. Applying the same approach as in Equation 8 we obtain

$$\dot{\varepsilon}_s = \frac{v}{\delta} = \frac{v}{2(hd-h^2)^{1/2}} \approx \frac{v}{2(hd)^{1/2}}. \tag{9}$$

3.1. Indentation measurement with cylindrical and hemispherical punches

Fig. 2 shows the log–log plot containing the $\dot{\varepsilon} - p$ and $\dot{\varepsilon}_s - p_s$ data obtained on lead glass. These data were calculated by the use of Equation 3 with $k_2 = 1$ for the cylindrical punch, and Equations 8 and 9 for the hemispherical one. In the case of measurements performed with hemispherical punches, the impression

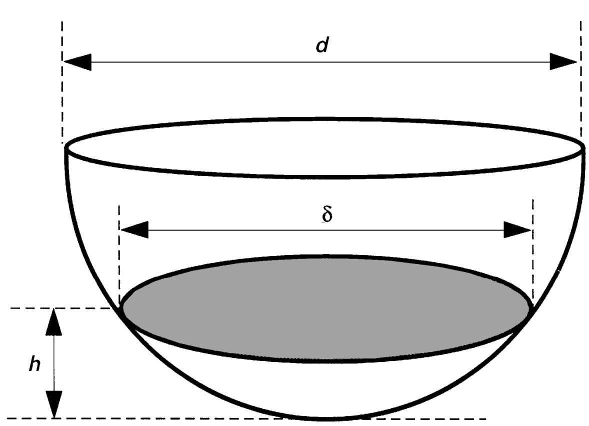

Figure 1 Geometry of the hemispherical indentor.

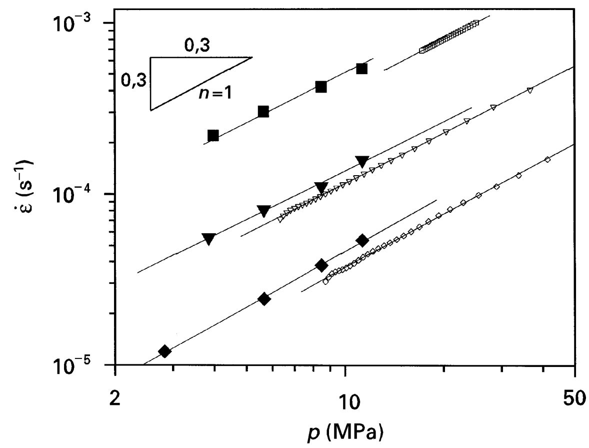

Figure 2 $\dot{\varepsilon}$–p and $\dot{\varepsilon}_s$–p_s for lead glass at different temperatures (logarithmic scale): (◆, ◇) 468 °C, (▼, ▽) 482 °C, (■, □) 497 °C. (◆, ▼, ■) cylindrical, (◇, ▽, □) spherical indentor.

velocity was determined by numerical derivation of the indentation–time curve. It is worth mentioning that, while in the case of cylindrical punches each data point corresponds to an individual measurement at different loads, by using a hemispherical indentor all data were obtained from the same test.

The curves given in Fig. 2 are straight lines, from the slopes of which a stress exponent of $n = 1$ is obtained. This means that the connections between strain rate and pressure data are linear in each case for lead glass. We emphasize that the linearity of the $\dot{\varepsilon} - p$ curves proves that the glass is deformed by Newtonian viscous flow. From Fig. 2 it can also be seen that the ratio of pressures, p and p_s taken at any given strain rate at constant temperature, is constant.

It was found that this ratio is constant not only for glasses. (Figs 3a, b and 4a, b show, for instance, some data series evaluated in the same way from the measurements performed on pure Al and M124 alloy, respectively, at different measuring parameters.) In each case, the following relationship can be written between p and p_s at any given $\dot{\varepsilon}$ and at constant temperature

$$p = Cp_s \tag{10}$$

where the value of C extracted from numerous measurements is 0.8 with $\pm$ 10% error. This means that the value of parameter C is a constant, which does not depend on material, the strain rate or temperature. Equation 10 confirms that indentation tests carried out with hemispherical indentors are suitable for the investigation of high-temperature plasticity of metals and alloys. It was shown previously [10] that the indentation test with both hemispherical and cylindrical indentors can be applied for the determination of the viscosity of glasses. It can be also seen (Fig. 3a and b) that the value of the stress exponent is about 4.5 for pure alluminium, which is in good agreement with that obtained for dislocation climb in aluminium [11].

3.2. Analysis of creep curves performed with hemispherical indentors

In the following it is supposed that Equation 2 describes the connection between the equivalent strain

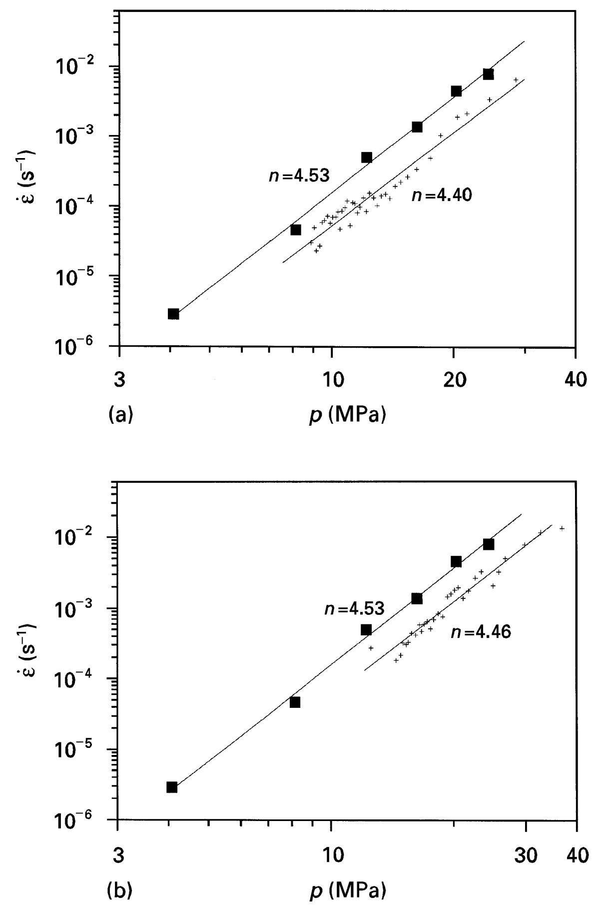

Figure 3 $\dot{\varepsilon}$–p and $\dot{\varepsilon}_s$–p_s functions for pure (99.99%) aluminium plotted at different loads and punch sizes at 447 °C: (a) (■) cylindrical $d = 1.25$ mm, (+) spherical $d = 2$ mm, $F = 20$ N; (b) (■) cylindrical $d = 1.25$ mm, (+) spherical $d = 3$ mm, $F = 40$ N.

rate and stress at any instant during the course of a measurement performed with a hemispherical punch. It means that the instantaneous strain rate and stress calculated by Equations 3, 8 and Equations 3, 9, respectively, obey the constitutive equation. In this case, Equation 2 can be regarded as a differential equation, which contains the indentation depth, h, and the elapsed time, t

$$\frac{1}{2(hd)^{1/2}}\frac{\mathrm{d}h}{\mathrm{d}t} = A\left(C\frac{F}{\pi hd} - \sigma_0\right)^n \tag{11}$$

Rearranging, it we obtain

$$\int_0^{h(t)} h^{n-(1/2)}\left(1 - \frac{3\pi hd\sigma_0}{CF}\right)^{-n}\mathrm{d}h = \int_0^t \frac{2AF^nC^n}{3^n\pi^n d^{n-(1/2)}}\mathrm{d}t \tag{12}$$

The expression in parentheses on the left-hand side of Equation 13 can be described in the form of a binomial series

$$\left(1 - \frac{3\pi hd\sigma_0}{CF}\right)^{-n} = 1 + \sum_{i=1}^{\infty}\left[\frac{\prod_{j=1}^{i}(n+j-1)}{i!}\left(\frac{3\pi hd\sigma_0}{CF}\right)^i\right] \tag{13}$$

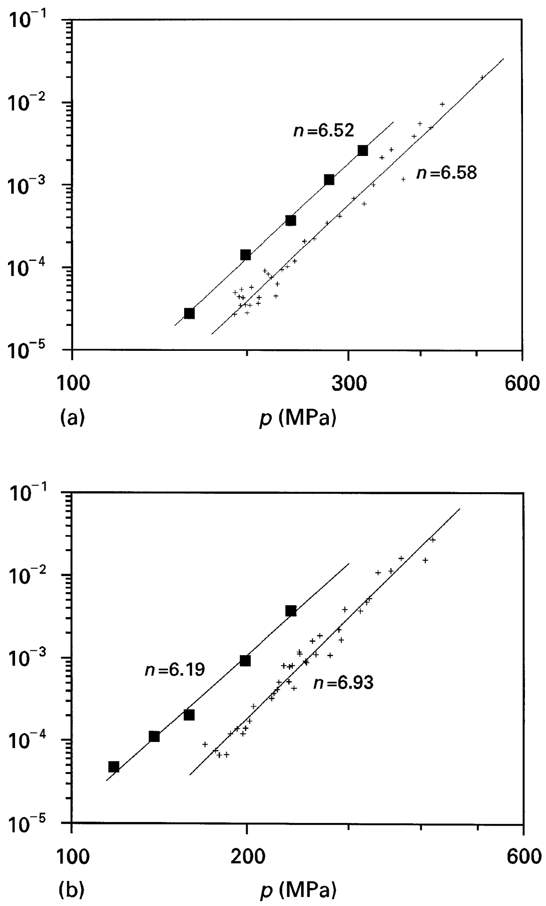

Figure 4 $\dot{\varepsilon}$–p and $\dot{\varepsilon}_s$–p_s functions for M124 alloy at different loads and punch sizes: (a) (■) cylindrical $d = 0.8$ mm, (+) spherical $d = 1$ mm, $F = 100$ N at 302 °C; (b) (■) cylindrical $d = 0.8$ mm, (+) spherical $d = 1$ mm, $F = 50$ N at 351 °C.

Substituting Equation 13 into Equation 12 we obtain

$$\frac{2AF^nC^n}{3^n\pi^n d^{n-1/2}}\int_0^t \mathrm{d}t = \int_0^{h(t)} h^{n-1/2}\,\mathrm{d}h + \sum_{i=1}^{\infty}\left[\frac{\prod_{j=1}^{i}(n+j-1)}{i!}\left(\frac{3\pi h d\sigma_0}{CF}\right)^i \int_0^{h(t)} h^{n+i-1/2}\,\mathrm{d}h\right] \tag{14}$$

from which:

$$t = (n+\tfrac{1}{2})^{-1}\frac{3^n}{2A}\frac{\pi^n d^{n-1/2}}{C^nF^n}h^{n+(1/2)} \times\left\{1+\sum_{i=1}^{\infty}\left[\frac{n+\frac{1}{2}}{n+i+\frac{1}{2}}\,\frac{\prod_{j=1}^{i}(n+j-1)}{i!}\left(\frac{3\pi h d\sigma_0}{CF}\right)^i\right]\right\} \tag{15}$$

According to this Equation, the elapsed time can be expressed as an explicit function of the indentation depth. This means that fitting Equation 15 to the indentation curve (h–t), the threshold stress and the stress exponent can be obtained from a single measurement. It should be noted that this analysis is valid for processes corresponding with the secondary creep.

By applying this procedure, we took into account the first five terms of the series of Equation 15. It can be shown that if the value of the threshold stress is lower than 25% of the minimum stress ($\sigma_0/\sigma_{min} < 0.25$) due to the load, then the error of the calculation must be lower than 4% if n is lower than 7.

Fig. 5a–c. show h–t data measured on lead glass, pure aluminium and the M124 alloy, respectively. It

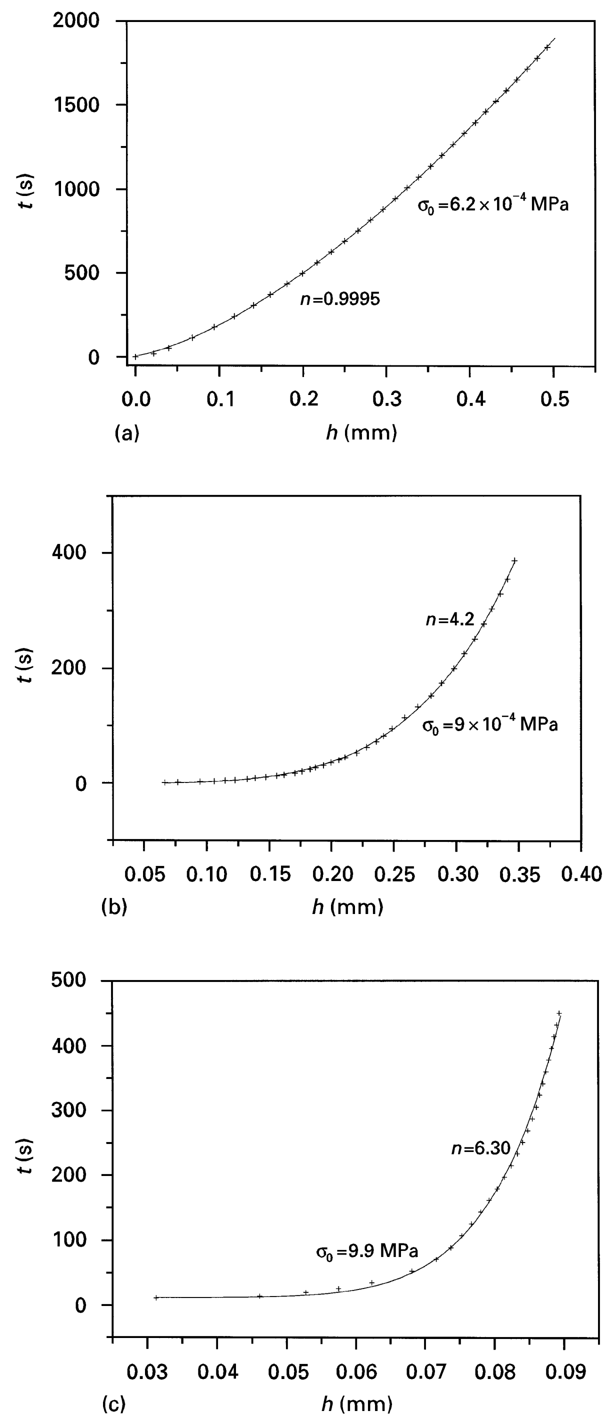

Figure 5 Time–indentation depth (t–h) taken with a hemispherical punch and fitted by Equation 15, for (a) lead glass, $T = 482$ °C, $d = 3$ mm, $F = 20$ N; (b) pure aluminium, $T = 644$ °C, $d = 4$ mm, $F = 10$ N; (c) M124 alloy, $T = 351$ °C, $d = 1$ mm, $F = 50$ N. (+) Measured values, (——) fitted function.

seems that the function determined on the basis of Equation 15 fits well to the measured points in each case. The values of the stress exponent obtained on this way are in good agreement with those obtained from the $\ln p$–$\ln\dot{\varepsilon}$ connections shown in Figs 2–4. In the case of lead glass and pure aluminium, the threshold stress, σ_0, has been found to be zero within the error. This fact also confirms the validity of Equation 15, because for glasses and pure metals at high temperature ($T > 0.5T_m$) the existence of a non-zero threshold stress cannot be expected. Namely, the appearance of a non-zero threshold stress is characteristic of extremely fine-grained materials [12] and materials containing finely distributed second-phase particles [1]. Non-zero threshold stress ($\sigma_0 \approx 9.9$ MPa at 351 °C) is obtained for only the M124 alloy, which contains a high volume fraction of precipitates. It can be supposed that these precipitates obstruct effectively the motion of the dislocations during the deformation. Problems referring to this threshold stress will be discussed in detail in a further paper.

It is worth mentioning that in the case of the zero threshold stress ($\sigma_0 = 0$; as was experienced in the case of lead glass or pure aluminium), Equation 15 can be simplified to the following form

$$h^{n+(1/2)} = \left(n + \frac{1}{2}\right)\frac{2A}{3^n \pi^n}\frac{C^n F^n}{d^{n-(1/2)}}t \qquad (16)$$

by which the indentation data obtained with hemispherical punches can be more easily treated. The use of Equations 15 or 16 leads to the same value of the stress exponent.

4. Conclusion

It was shown that a unique correspondence can be formed between the indentation measurements performed by cylindrical and hemispherical indentors; therefore, the indentation test carried out with a hemispherical indentor is also suitable for the investigation of the high-temperature plasticity of metals and their alloys.

The indentation curve taken using a hemispherical indentor can be described by an equation derived from the well-known constitutive equation of the high-temperature creep process. This equation can be fitted numerically to the measured curves, from which the values of the stress exponent and the threshold stress can be determined. It was pointed that, using this method, the creep parameters can be determined from only a single indentation curve, while in the case of cylindrical punches numerous measurements are required for the characterization of the creep process.

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