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Mathematical description of indentation creep and its application for the determination of strain rate sensitivity

Nguyen Q. Chinh *, Péter Szommer

Department of Materials Physics, Eötvös Loránd University, 1117 Budapest, Pázmány Péter s. 1/A., Hungary

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ABSTRACT

The creep process during the holding stage of nanoindentation test has been analyzed mathematically. It is shown analytically for the first time that the corresponding indentation depth–time relationship can be described by a power-law function, suggesting a direct application of nanoindentation for the determination of the strain rate sensitivity.

In the last three decades depth-sensing indentation (DSI) became widely used for mechanical characterization of solids. The reason for its popularity is that DSI enables the determination of several mechanical parameters even for a small volume of material without complicated sample preparation. Furthermore, the DSI is a very efficient technique for some materials and/or conditions to study the material properties where other methods are very hard to apply. For instance, the creep behaviors of human enamel [1], the effect of individual grains and grain size [2,3], mechanical properties of coatings [4,5] could be effectively studied – only – by using nanoindentation. Thanks to the development of techniques, the load and displacement can be applied and detected easily, with relatively high accuracy, making the analysis of the relationship between the recorded (load, depth, time) parameters more precise.

During the DSI process [6,7], a given geometrical indenter penetrates into the surface of the material with constant loading rate. When the load, P, reaches its selected maximum value, $P_{\max}$, the indenter can be held at this maximum load for a required time period, when creep may take place in the material zone under the indenter. The holding stage is then followed by the unloading when the load decreases time-linearly and the indenter moves backwards. Typical load–depth $(P-h)$ and depth–time $(h-t)$ curves can be seen in Fig. 1. The curves in this example were taken on an ultrafine-grained (UFG) Al–30Zn alloy which shows relatively intensive grain boundary sliding [8] and high ductility [9] at room temperature. In the basic application, the hardness, H, of the indented material can be measured according to the definition:

$$H = \frac{P_{\max}}{A} = C\frac{P_{\max}}{h_c^2}, \tag{1}$$

where A is the projected area of the hardness impression measured at the contact depth, h_c. Furthermore, the value of the constant, C, depends on the geometry of the indenter [6,7].

It is well-known that the most often used application of DSI is the determination of the hardness and the elastic modulus of materials. However, the method is suitable also for the investigation of e.g. stress induced phase transformations [10], deformation mechanism in amorphous materials [11], and plastic instabilities in metals [12,13]. Strain rate sensitivity can also be determined by measuring the hardness, H, at different loading rates, v, and analyzing the H–v connections [14–18].

Although the creep behavior and/or the ductility of the materials can be qualitatively studied by considering the holding stage, to our best knowledge, no mathematical analysis of this stage has been reported up to now. Our motivation in this work is to derive a constitutive equation for the description of the creep process in DSI.

For this purpose, we have to start from the following conventional relationships which are generally used to characterize creep

* Corresponding author. Tel.: +36 1 372 2845; fax: +36 1 372 2811.
E-mail address: chinh@metal.elte.hu (N.Q. Chinh).

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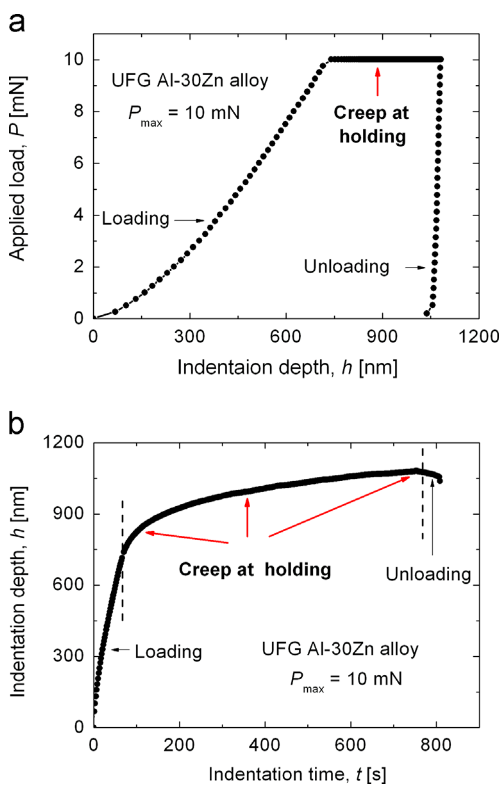

Fig. 1. Typical (a) load–depth (P–h) and (b) depth–time (h–t) curves showing the loading, holding and unloading stages during a depth-sensing indentation test. (The curves were taken on an ultrafine-grained Al–30Zn alloy [4] indented with load increasing by0.1 mN/s to $P_{\max} = 10$ mN maximum load, and with holding time of 700 s).

flow under steady-state conditions:

$$\sigma = K\dot{\varepsilon}^m, \tag{2}$$

or

$$\dot{\varepsilon} = K^* \sigma^{1/m} \tag{3}$$

where K and $K^* = (1/K)^{1/m}$ are the material- and temperature-dependent constants respectively, σ is the flow stress, $\dot{\varepsilon}$ is the corresponding strain rate and m is the strain rate sensitivity (SRS). During the indentation creep with load $P_{\max}$, the equivalent flow stress, σ which is proportional to the mean contact pressure [6,19], can be given as

$$\sigma = C_1 H = C_1^* \frac{P_{\max}}{h_c^2}, \tag{4}$$

where C_1 and C_1^* are constants. In practical applications, for ductile materials, such as crystalline metals and alloys, the value of the contact depth, h_c, is almost the same than the actual depth, h ($h_c \approx 0.95h/0.97h$). For brittle materials, such as ceramics, glasses, these two quantities are rather proportional ($h_c \propto h$). Thus, in both cases the equivalent flow stress, σ, in Eq. (4) can be expressed by the actual depth, h, and another (C_2) constant as

$$\sigma = C_2 \frac{P_{\max}}{h^2}. \tag{5}$$

Furthermore, the equivalent strain rate, $\dot{\varepsilon}$, is dependent on the penetration rate, dh/dt, of the indenter according to the following formula [20,21]:

$$\dot{\varepsilon} = C_3 \frac{1}{h}\frac{dh}{dt}, \tag{6}$$

where C_3 is a constant.

During the loading stage the load is, in general, approximately proportional to the square of depth ($P \propto h^2$). When applying linearly increasing load with loading rate, v ($P = vt$), it can be easily shown on the basis of Eqs. (1)–(6) that $\dot{\varepsilon} \propto v$ and $H \propto v^m$, from which the SRS can be obtained [14–18]. It should be noted that the determination of SRS by using $H-v$ relationship requires several measurements to be done at different loading rates.

Using the creep stage, strain rate sensitivity can be obtained from the data of a single indentation measurement by analyzing the corresponding depth-time ($h-t$) relationship. So far there have been efforts to handle the $h-t$ data numerically [1], or by using empirical formula [22]. As it has been mentioned above, no analytical formula of $h-t$ connection has been reported for the description of the creep stage during indentation. We will show in the followings how to get this kind of formula by using Eqs. (1)–(6). In the present analysis some constants denoted as C_i (i from (1) to (6)) are used without direct physical meaning, as these constants are simply following from the mathematical derivation.

Substituting Eqs. (5) and (6) into Eq. (3) we obtain the following differential equation:

$$C_3 \frac{1}{h}\frac{dh}{dt} = K^* \left(C_2 \frac{P_{\max}}{h^2} \right)^{1/m}, \tag{7}$$

which can be rearranged as

$$h^{\frac{2}{m}-1} \frac{dh}{dt} = C_4 (P_{\max})^{1/m}, \tag{8}$$

where $C_4 = K^*(C_2)^{1/m}/C_3$.

During an indentation test including both the loading and holding stages, it can be supposed that the creep process – within the holding stage – starts from the onset characterized by the parameters (t_0, h_0). Separating the variables (t and h) and integrating Eq. (8) from the onset to a certain state given by (t, h), we get

$$\int_{h_0}^{h} h^{\frac{2}{m}-1} dh = \int_{t_0}^{t} C_4 (P_{\max})^{\frac{1}{m}} dt, \tag{9}$$

from which

$$\frac{m}{2} h^{\frac{2}{m}} = C_4 P_{\max}^{\frac{1}{m}} t - C_4 P_{\max}^{\frac{1}{m}} t_0 + \frac{m}{2} h_0^{\frac{2}{m}}. \tag{10}$$

Combining the constant terms, Eq. (10) can be rearranged to the form:

$$\frac{m}{2} h^{\frac{2}{m}} = C_4 P_{\max}^{\frac{1}{m}} t - C_5, \tag{11}$$

or simply

$$\frac{m}{2} h^{\frac{2}{m}} = C_6 (t - t_c). \tag{12}$$

The parameters C_5 in Eq. (11) and C_6, t_c in Eq. (12) are derived constants, $C_5 = C_4 P_{\max}^{1/m} t_0 - \frac{m}{2} h_0^{2/m}$, $C_6 = C_4 P_{\max}^{1/m}$, and $t_c = C_5 / C_6$.

According to Eq. (12), the $h(t)$ function can be written in the form:

$$h = B(t - t_c)^{\frac{m}{2}}, \tag{13}$$

where $B = (2C_6/m)^{m/2}$. This is the significance of the present analysis, showing that the indentation creep can be described by a simple power-law function, where the exponent depends only on the strain rate sensitivity, m.

Eq. (13) then allows us to determine conveniently the strain rate sensitivity, m, by analyzing the $h-t$ connection characterizing the indentation creep. Fitting the power-law function (13) to the experimental data obtained in this region, the value of the SRS, m, is double of the obtained fitting exponent.

Fig. 2 shows some examples demonstrating the validity of Eq. (13), as well as the determination of the m parameter in different cases. Careful nanoindentation experiments were made at room temperature using a three-side pyramidal Berkovich