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A critical appraisal of the extraction of creep parameters from nanoindentation data obtained at room temperature

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Abstract

Nanoindentation is being increasingly used to obtain creep parameters. The technique allows interrogation of small volumes and relatively rapid acquisition of data. However, the reliability of the methods used to extract creep parameters from experimental data is largely unproven. In the present work, commonly employed procedures are applied to a wide range of materials, for which the creep parameters are already known. The correlation observed between known values of the stress exponent, n, and those obtained using the indentation method is generally very poor. Possible reasons for this are considered. One concern is that primary creep, or other unsteady deformation mechanisms, may be strongly influencing the observed behaviour throughout the test. Taking such uncertainties into account, a procedure is proposed for ranking creep propensities exhibited during nanoindentation under specified conditions.

Keywords: Creep; Nanoindentation

1. Introduction

During nanoindentation, a fine scale indenter is used to load a sample in a controlled way, while the displacement is continuously recorded. The well-established procedure of Oliver and Pharr [1] can be applied to loading and unloading curves to deduce the hardness and modulus of the tested material. Other indentation-based procedures have been proposed to investigate other materials parameters, such as the fracture toughness [2] and equipment modifications been made, allowing fatigue and impact testing to be carried out [3]. The current work is focused on the use of indentation to assess creep parameters, in particular the stress exponent, n. Since the use of nanoindentation testing to characterize creep response is relatively novel, an outline is first presented of the relevant theoretical background.

2. Background to nanoindentation creep testing

2.1. Nanoindentation creep effects

The observation of creep (time-dependent) phenomena is common in indentation experiments, and is often considered to be a problem. For example, Chudoba and Richter [4] investigated how they can be eliminated during measurement of hardness and modulus. They pointed out that creep effects can sometimes lead to a net increase in indenter depth with decreasing load during the early stages of unloading. This is due to the material plastically deforming by creep faster than it is elastically recovering as the load is reduced. Since the Oliver and Pharr analysis for modulus measurement is focused on the initial rate of decrease in depth as the load is reduced, it is clear that the complications introduced can sometimes be substantial. To avoid such errors in hardness and modulus measurement, Chudoba and Richter recommended that the maximum load should be retained for a period (~10–60 s) before the onset of unloading. Longer dwell times can pose a problem, since

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this requires high machine stability if the loading and unloading curves are to be consistent. The purpose of the dwell is to allow the material to 'creep out', i.e. to deform sufficiently for creep deformation to be insignificant during unloading. Inherent in this rationale is the concept that the creep rate will fall off with time, possibly because of a decreasing contribution from primary creep.

However, since both load and depth are continuously monitored during nanoindentation, it is clear that information can be collected concerning creep behaviour during the hold period. Various attempts have been made to interpret such data so as to obtain creep parameters, as outlined below.

2.2. Equations used and assumptions made

All the nanoindentation-based methods, and most of those based on conventional indentation procedures, use the same basic equations in analysing creep data. These will be considered first, with the differences between the two types of test being examined subsequently.

2.2.1. Stress and strain fields under the indenter

Indentation of a surface generates various stresses and strains in different regions around and under the indenter. Early observations of the plastically deformed zone in conventional indentation indicated that the shape of the deformed zone is approximately hemispherical [5,6]. This suggested to Marsh [7] that the deformation could be represented as half of that produced by a spherical cavity expanding under internal hydrostatic pressure. He used Hill's equations [8] for this situation, in order to analyse indentation experiments. Johnson [9] took the description further by equating the expanding hydrostatic volume to the region of material under the indenter (see Fig. 1). Although not a perfect description of indentation (for example, the lack of constraint can lead to differences near the surface, particularly if there is significant pile-up or sink-in), this picture of indentation is the basis of several analyses, including those leading to equations commonly used for creep analysis.

Although not considered here, it seems likely that the situation will be more complicated for a spherical indenter than that for a self-similar indenter. For a spherical indenter, the size and shape of the stress and strain fields will vary in a complex way during the indentation process, since they are affected not only by the depth of the indent, but also by the ratio of indenter diameter to indent size.

2.2.2. Representative stress

The stress under an indent varies from high levels in the vicinity of the tip to vanishingly small values in remote regions. However, a characteristic (or effective) stress is commonly required for use in models. In an analogous manner to the procedure used to define hardness, the characteristic stress σ is taken as the applied load F, divided by the projected contact area A_p (the cross-sectional area of the indenter at the depth to which it is indented, rather than the real surface contact area):

$$\sigma = \frac{F}{A_p} \tag{1}$$

Choosing this as the characteristic value for analysis of creep follows the principles used in the early work of Mulhearn and Tabor [10]. It is also supported by the calculations of Bower et al. [11], who examined indentation of a solid creeping according to a power law relationship, i.e. one in which the uniaxial stress–strain behaviour is represented by

$$\dot{\varepsilon} = C\sigma^n \tag{2}$$

where C is a constant, σ is the applied stress, and n is the creep exponent. The equations produced were solved analytically for the complementary limiting cases of a

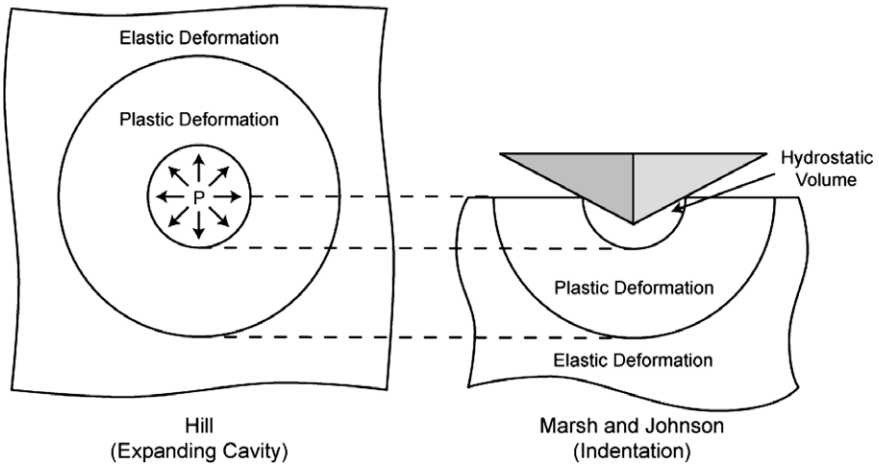

Fig. 1. Representation of the deformation field under an indent, as an expanding hemispherical volume subjected to hydrostatic pressure, as developed by Marsh [7] and Johnson [9]. As the hydrostatic volume increases in size, more material undergoes plastic deformation and the elastic–plastic boundary advances.

Newtonian viscous solid ($n = 1$) and a rigid, perfectly plastic solid ($n = \infty$), with the finite element method being used for intermediate values. The results showed how the creep behaviour defined by Eq. (2) leads to indentation behaviour in the form of a relationship between applied load and indent depth, and suggested that Eq. (1) gives a suitable measure of the characteristic stress.

2.2.3. Representative strain rate

In a similar way, there is no single value of the strain rate around an indent, but a characteristic value is often required. The origins of the most popular formulations for this parameter come from the description of the deformed zone as an expanding hemispherical cavity, as discussed above. The formulae given by Hill were used by Atkins et al. [12] to derive a relationship for the shear strain rate, as a function of the distance from the centre of the cavity r and the diameter of the hydrostatic volume d:

$$\dot{\gamma} = \frac{3}{2}\frac{d^2}{r^3}\dot{d} \tag{3}$$

This relationship was used by Pollock et al. [13], who selected the maximum strain rate (located at the boundary between the hydrostatic volume and the region deforming plastically) as the characteristic value. These workers also noted that, for a conical indenter, the value of d and the indentation depth h are linearly related, leading to the following equation

$$\dot{\varepsilon} = K\frac{\dot{h}}{h} \tag{4}$$

where K is another (dimensionless) constant. The final step to the now widely used equation for the strain rate during indentation was proposed by Mayo and Nix [14], who generalized the equation of Pollock et al. to pyramidal indenters, and also omitted the constant of proportionality, leading to

$$\dot{\varepsilon} = \frac{1}{h}\frac{\mathrm{d}h}{\mathrm{d}t} \tag{5}$$

As with the idea of a characteristic stress, the use of this formula for the characteristic creep rate is supported by the theoretical calculations of Bower et al. [11]. Attempts have been made to correlate this 'indentation strain rate' more directly with the actual (range of) strain rate experienced by the material under the indenter. Posil et al. [15] examined amorphous selenium and calculated that the indentation strain rate is approximately one order of magnitude higher. However, this conclusion is a rather limited and specific one.

2.2.4. Steady-state creep

All methods discussed so far assume the observed deformation to be solely due to steady-state creep deformation, such that the creep rate conforms to the standard (secondary) creep equation

$$\dot{\varepsilon} = C\sigma^n \exp\left(-\frac{Q}{RT}\right) \tag{6}$$

The parameters C (a constant), n (the stress exponent) and Q (the activation energy for creep) are all assumed to have the same values as would be obtained from conventional tensile creep tests. Of course, such tests involve homogeneous loading of the complete gauge length of the specimen and commonly last between a few hundred and several thousand hours [16], whereas indentation creep tests evidently involve highly non-homogeneous stress, strain and strain rate fields, and often last only a few minutes or a few hours. The reliability of this assumption that the deformation occurring during indentation can be described by Eq. (6) is a key issue.

It might be noted at this point that the question of creep mechanisms is closely related to microstructure. Glassy materials, in which complications associated with the presence of grain boundaries, dislocations, etc. will be absent, might thus be expected to behave in a simpler fashion. There has been relatively little work hitherto on nanoindentation creep of glassy materials, although Fatay et al. [17] found that the activation energy obtained in this way for a bulk metallic glass was consistent with that from homogeneous compression testing.

2.3. Analysis of indentation creep data

Initial study of creep during indentation was focused on conventional hardness tests at elevated temperature [10,18], which involved no monitoring of indenter depth. Analysis of such experiments was thus rather different from those more recently applied to nanoindentation. However, clarification of the underlying assumptions is nevertheless of interest, and the method of Sargent and Ashby [19] will be used to illustrate this. They used dimensional analysis to derive their final equations, but also pointed out that various approaches give solutions of similar form. The creep is assumed to conform to Eq. (6) and, for conical or pyramidal indenters, the stress and strain rate fields are assumed to be self-similar, i.e. while the values of stress and strain at each point will change during deformation, the ratios between values at different points in the field will remain the same. Using these assumptions, Sargent and Ashby arrived at essentially the same expressions for characteristic stress and strain rate as those given above, confirming that the analysis is basically the same for all scales of indentation.

A similar technique is impression creep [20,21], which involves using a flat-ended cylinder, rather than a pyramid or sphere. There is an underlying expectation that the stress state during impression creep will be closer to steady state. Under constant load, the indentation rate typically becomes approximately constant after about 100 min [20]. The creep parameters (stress exponent and activation energy) obtained by this method have been found [21] to be consistent with those from conventional creep tests for

a range of materials. However, most of the concerns with indentation creep, for example the probable influence of primary creep deformation in regions that have experienced relatively low strains, are expected to apply equally to impression creep.

2.4. Analysis of nanoindentation creep data

Several procedures has been used to analyse nanoindentation creep. These will be briefly discussed, although attention is primarily focused on the constant load method (Section 2.4.4).

2.4.1. Constant depth method

Attempts have been made to generate conditions such that the indenter remains at a constant penetration depth, and this is sometimes termed the indentation load relaxation test. The way that load decreases can be monitored, allowing analysis in a similar manner to a conventional stress relaxation (fixed grips) creep test. This method has been applied using both indentation [22] and nanoindentation [23] equipment. However, while it is relatively easy to hold an indenter in a fixed spatial position, keeping the penetrated depth constant is virtually impossible. This is due to the elastic strain in the whole sample being converted to plastic deformation solely in the region of the indenter, which is accompanied by an increase in the indented depth. The test conditions thus involve continuous changes in both depth and load, making analysis very complicated. The method has not proved popular.

2.4.2. Constant rate of loading method

Mayo and Nix [14] suggested that, with a given rate of load increase, a steady-state rate of depth increase will be attained, which can be combined with the equations for stress and strain rate (Eqs. (1) and (5)) to evaluate the stress exponent, n. Tests based on this method are called constant rate of loading (CRL) tests. The schematic curves given by Mayo and Nix are reproduced in Fig. 2a, which depicts a linear regime at greater depths (the 'large depth stage'). The observation that nanoindentation traces tend to become more linear as the indenter probes deeper is a common one (e.g. see the experimental curves of Oliver and Pharr [1], or the schematic curve in Fig. 2b), but this is just an effect of the relative rate of change of indent size being lower at greater penetration depths. For Berkovich indentation of an ideally plastic material (no work hardening), the depth–load plot (or depth–time plot for a CRL test) should follow a square-root relationship, which will show smaller gradient changes at larger depths. However, there will not actually be a different regime at higher loads, as implied by Fig. 2a, and the gradient does not in fact become constant (or indicative of a steady state).

Mayo and Nix applied their procedure to pure Pb and Sn, testing samples with large grain sizes compared with the indent diameter, and also a finer-grained Sn sample. For the large-grained samples, the values they found ($n_{Pb} = 8.5$ and $n_{Sn} = 11.4$) were in approximate agreement with the literature values for coarse-grained materials ($n_{Pb} = n_{Sn} = 8$). However, since indentation was confined to single grains, it might be argued that a more appropriate comparison would have been with the single crystals values they quoted ($n_{Pb} = 1.2$ and $n_{Sn} = 4.8$). The fine-grained Sn sample gave a result ($n_{Sn} = 6.3$) in the range of the literature values ($n_{Sn} = 4.7$–8). Despite a measure of agreement, it is clear that these comparisons do not constitute a systematic confirmation of the validity of the approach.

2.4.3. Constant indentation strain rate method

Control systems in nanoindentation commonly allow programmable loading rates during a single indentation. A constant indentation strain rate can therefore be imposed. From Eq. (5), this requires the loading rate to be increased in such a way that it is constant when

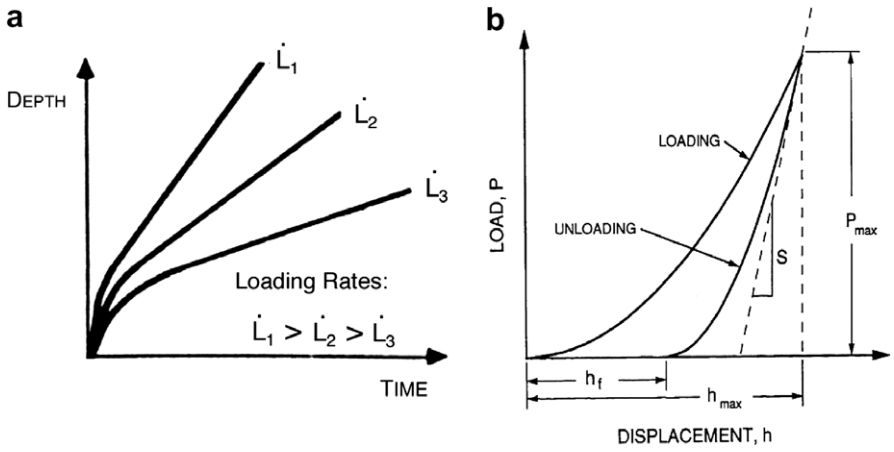

Fig. 2. Schematic indentation curves proposed by: (a) Mayo and Nix [14], for CRL creep, showing an approximately linear regime reached after a certain time; and (b) Oliver and Pharr [1], showing the typical form of an indentation curve, in which the loading curve becomes progressively more linear, without actually reaching a constant gradient regime. The two schematic plots illustrate the same characteristics, since depth and displacement mean the same thing, while time and load are equivalent when the loading rate is constant.

normalized by the instantaneous load. Of course, this assumes that Eq. (5) is applicable. Furthermore, it is still unclear whether a constant indentation strain rate corresponds in some meaningful way to steady-state creep conditions. In any event, the technique has been applied by Lucas and Oliver [24] to the testing of indium. They obtained good agreement with literature data for both n and Q ($n_{In} = 7.3$ and $Q_{In} = 77.9$ kJ/mol, compared with reported literature values of $n_{In} = 7.6$ and $Q_{In} = 75$–78 kJ/mol). The agreement they obtained for n using this procedure was slightly better than when they used a constant load method, which gave $n_{In} = 6.3$.

2.4.4. Constant load method

By far the most popular method is that involving a constant load, which simplifies measurement and analysis, and facilitates rapid data collection. It is the main focus of the current work. The technique was first reported by Mayo et al. [25], who recommended an extended dwell at constant load of at least 50 s duration, during which the further penetration of the indenter into the sample is monitored. The depth change during the dwell stage is plotted against dwell time, and the resulting curve analysed to deduce a value for n, as shown in Fig. 3. In their calibration experiments on Sn–38 wt.% Pb, Mayo et al. obtained $n = 1.8$, compared with a reported literature value of $n = 2$. However, in their experimental investigation of the strain rate sensitivity of TiO_2 (as a nanophase and in the form of single crystal rutile), values of n were found in the range 25–100. Although these materials have not been investigated in detail in conventional creep experiments, values of n of this magnitude are physically implausible for a creep-like process.

While such results are clearly a cause for concern, other workers have found good correlations between the literature and experiment using this method. For example, Raman and Berriche [26] examined Sn and found $6.7 \leqslant n_{Sn} \leqslant 8.1$, which compares well with a range in the literature of 7–8. The method may also be used to estimate the activation energy for creep, provided indentations can be made over a range of temperature (see Fig. 3). Such a procedure has been used, for example, by Asif and Pethica [27], who obtained good correlation between experiment and literature values for indium (experimentally $Q_{In} = 77$ kJ/mol, the literature value for self-diffusion, $Q_{In} = 75$–78 kJ/mol). However, it is probably fair to state that there have not really been any wide-ranging systematic studies of the reliability of the indentation technique for extraction of creep parameters. The experimental work described in the present investigation is aimed in this direction.

3. Experimental procedures

Experiments were performed using a NanoTest nanoindenter, supplied by MicroMaterials Ltd., Wrexham, UK. The indenter is mounted on a balanced pendulum and the direction of indentation is horizontal (see Fig. 4). Several pure element specimens have been studied, together with an alloy, an oxide and an ionic crystal (see Table 1). Each of these materials is included in Frost and Ashby's creep mechanism maps [28], and the parameters given in this reference have been taken as indicative of the results expected for each material.

Polished samples were mounted in the indenter, and constant load creep data were obtained. All tests were carried out at a stabilized chamber temperature of 28 °C. The

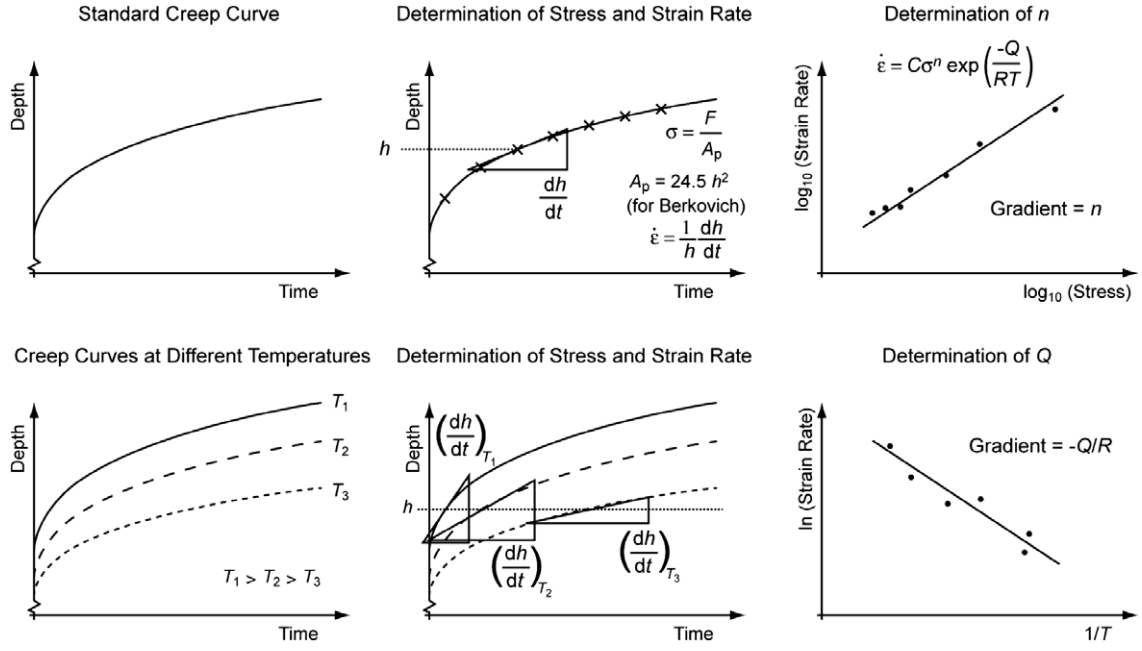

Fig. 3. Schematic depiction of how depth–time plots, obtained under constant load, can be used to determine values for the stress exponent, n, and the activation energy, Q. Stress and strain rate are found using Eqs. (1) and (5).

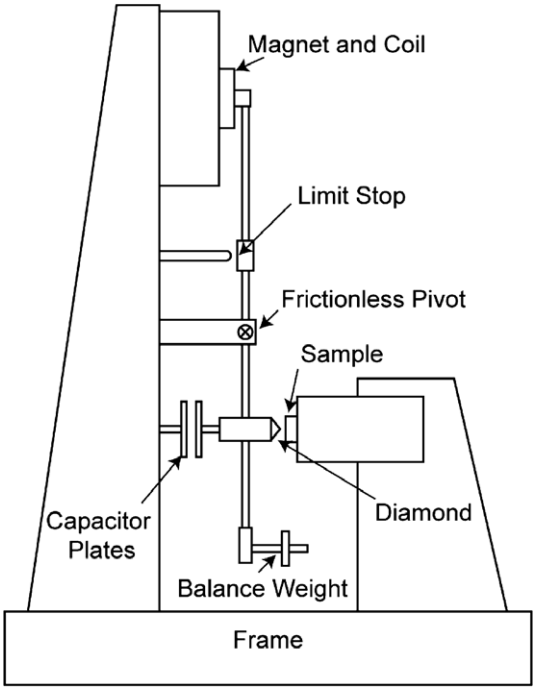

Fig. 4. Schematic diagram of the indenter used in the investigation.

Table 1
Comparison between the literature values of the stress exponent for various materials (obtained by conventional tensile testing of uniformly loaded specimens) and corresponding data obtained in the current work by constant load indentation creep testing

Material	Class	Literature values of n [28]	Experimental measurement of n		
			Average value	Number of tests	Standard deviation
Aluminium	fcc	4.4	12.5	9	6.2
Copper	fcc	4.8	6.7	3	3.4
Lead	fcc	5.0	5.9	4	8.9
Nickel	fcc	4.6	2.2	5	1.6
Silver	fcc	4.3	3.5	7	1.1
Chromium	bcc	4.3	7.7	8	6.8
Molybdenum	bcc	4.9	2.2	2	0.1
Tungsten	bcc	4.7	6.9	8	5.7
Vanadium	bcc	5.0	4.4	15	5.1
Cadmium	hcp	4.0	0.9	10	0.1
Magnesium	hcp	5.0	10.2	5	6.2
Zinc	hcp	4.5	3.9	6	1.2
316 Stainless steel	Alloy	7.9	4.2	8	3.3
Alumina	Oxide	3.0	5.4	4	2.1
Lithium fluoride	Ionic	6.6	7.7	12	6.5

load was selected (in the range 10–50 mN) so as to generate an indent of a suitable size (depth ~100–1000 nm). The indenter was loaded over a period of 20 s, and the creep load was then maintained for 1200 s, with the increase in depth during this period being recorded. (This dwell time is considerably greater than most of those previously used, since this allows study of the possibility of creep mechanism changes during the test.) The load was then decreased, again over a period of 20 s, in order to allow the modulus to be measured, using the method of Oliver and Pharr [1]. The data were corrected to account for any thermal drift, via the incorporation of a 60-s dwell at 20% of the maximum load on unloading, and for the compliance of the frame, using high load indents into a material of known properties (amorphous silica). Prior indents into silica over a range of depths were also used to correct for the deviation from the ideal Berkovich shape of the diamond. The constant load analysis described above was then performed, taking data every 60 s throughout the period, to evaluate the stress exponent for creep n.

4. Results and discussion

4.1. Comparison between measured and previously reported data

Examples of experimental plots are shown in Fig. 5. For some materials, noticeable discontinuities ('steps') were observed in the displacement–time plots, corresponding to rapid increments of indenter depth. Two examples are shown in Fig. 6. Some materials (notably copper, alumina, tungsten and stainless steel) commonly exhibited such effects, while others did not. The associated increment of displacement was particularly large in copper. It is thought that these steps were caused either by mechanical twinning or by sudden nucleation of dislocation cascades. Their presence suggests that mechanisms other than creep were exerting a strong influence, so the associated plots were eliminated from the study.

Data concerning the stress exponent, n, determined using the constant load method, are presented in Table 1. The relationship between these experimental data and the corresponding literature values [28] is presented graphically in Fig. 7. It can be seen that there is little or no correlation, and it is difficult to escape the general conclusion that use of this method to measure n is extremely unreliable. It can also be seen from the standard deviations (error bars) that reproducibility is poor, and that it is difficult to improve the precision or reliability of the method by increasing the number of tests. It is unlikely that this could be explained in terms of variations in microstructure between specimens, although it is true that no attempt was made to match microstructural features or impurity levels, etc. between the materials tested and those from which the literature data were obtained. Of course, there should also be error bars on the literature data, but these are expected to be relatively small.

The most likely explanation for both the poor reproducibility and the poor correlation with tensile testing results is that the procedures and analysis currently employed in indentation creep testing are inherently unreliable and hence subject to large errors arising from small variations in test conditions or data interpretation. To explore this more deeply, the assumptions implicit in the method must be critically examined.

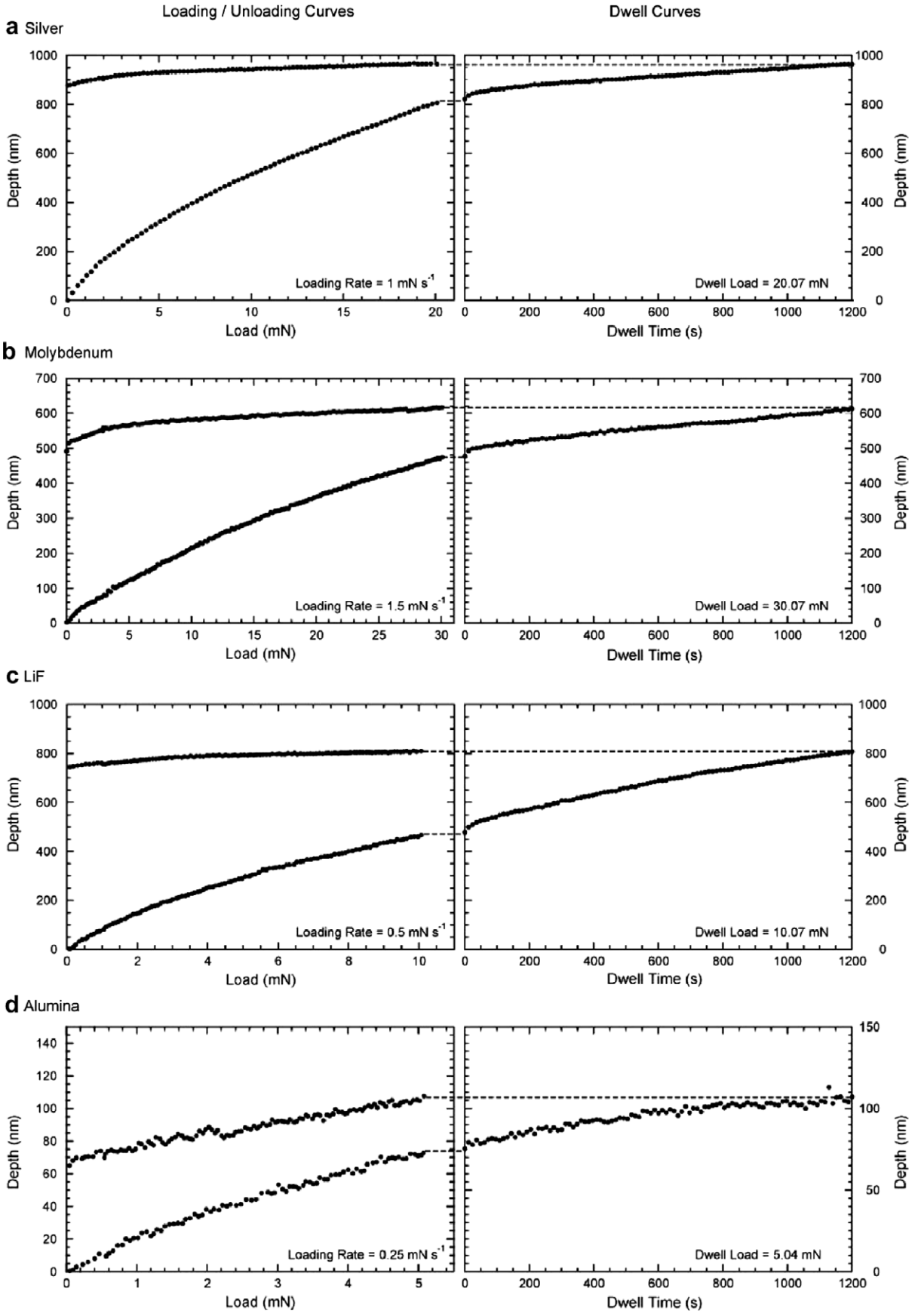

Fig. 5. Experimental load–displacement data obtained for various materials.

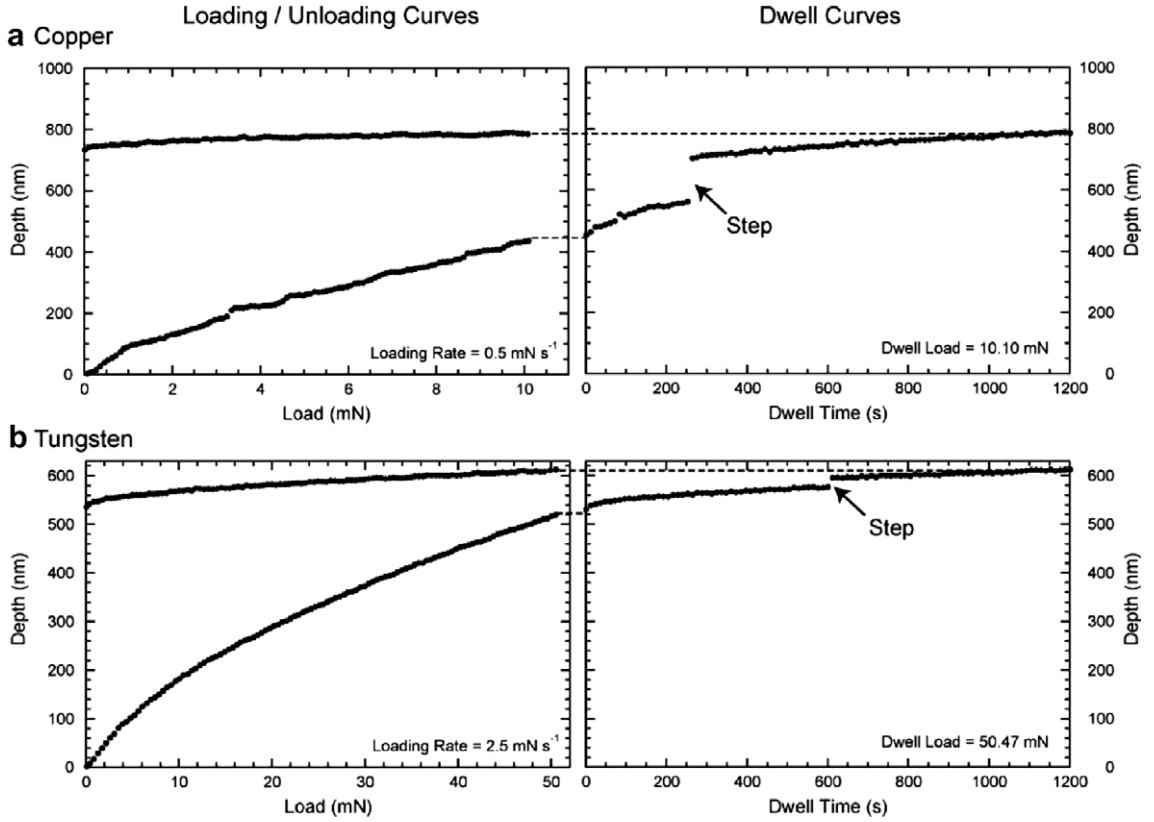

Fig. 6. Examples of the steps exhibited during the dwell stages in data for: (a) copper and (b) tungsten.

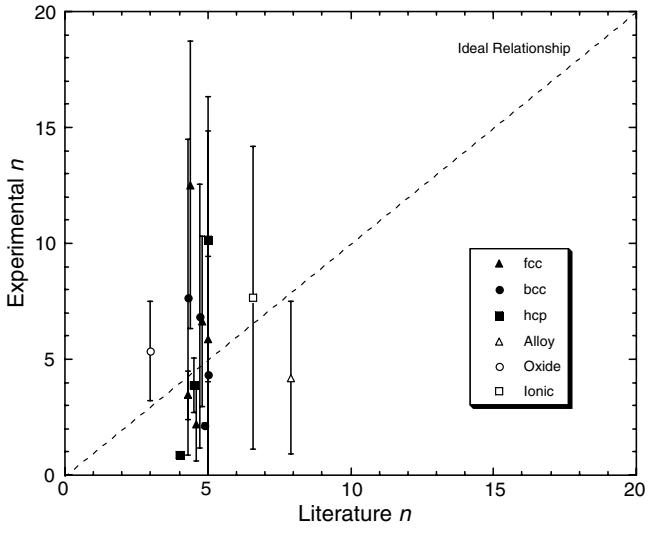

Fig. 7. Correlation between experimentally determined values of the stress exponent, n, obtained using the constant load method, and corresponding values from the literature [28].

4.2. Critique of assumptions used in current models

A key assumption behind the identification of characteristic stress and strain rate values is self-similarity of the stress and strain fields, which is common to most previous work. It has arisen from the expanding cavity model and is supported by the calculations of Bower et al. [11] described earlier. The other critical assumption is that of steady-state conditions. Creep is taken to be occurring in the steady-state (secondary) regime throughout the deforming volume of the specimen, or at least throughout the region within which deformation affects the movement of the indenter. This assumption certainly requires careful scrutiny, since it is well established that primary creep commonly generates much higher (and more variable) strain rates than secondary creep of the same material under the same applied stress.

One approach to exploring this issue further is to consider how a deformation mechanism map might be used to interpret a nanoindentation creep test. The local stress level (for example, the deviatoric component of the stress state) can be evaluated as a function of location at different stages of an indentation process. For example, finite element analysis can be used for this purpose [29–31]. Of course, accurate analysis would require data which may be unavailable (such as creep parameters), but a high level of precision would not be required in order to reveal the main features. Combination of the stress field at different stages during indentation with the mechanism map would at least allow an assessment of which creep mechanism is likely to be dominant in different regions and at different times during the test.

The concern about different mechanisms operating in different parts of the specimen, and about conditions

deviating markedly from those during tensile creep testing, is highlighted by the work of Li et al. [32], who formulated equations for the contribution to creep from a range of mechanisms, under typical indentation conditions. These equations predict that the dominant mechanism under virtually all conditions, except with very small ($\leqslant$0.4 μm) grain sizes (which promotes Coble creep) is expected to be dislocation glide plasticity. The reason for this is that the local stresses in the regions thought to control the indentation rate are exceptionally high.

In addition to such concerns, which relate primarily to local stress levels, there is also a timescale issue. The relatively short duration of most indentation tests, often required due to the limited thermal stability of the equipment, should be carefully noted. Typically, the test period is measured in minutes, rather than the hundreds or thousands of hours typical of tensile creep tests. Even if the stress fields were uniform, this difference would tend to raise the relative significance of primary creep. In combination with the fact that, throughout the test, previously unstrained material is continuously entering the creeping domain, this reinforces the conclusion that primary creep might be expected to exert a strong influence on the observed behaviour.

In response to such concerns, several workers [12,33,34] have suggested the use of a transient creep law, rather than the simple steady-state equation. However, perhaps because such an approach allows fewer deductions to be made about material behaviour over a range of conditions, these studies have not received much attention. Support for the use of a steady-state analysis comes from the calculations of Bower et al. [11], who noted that their analysis indicated that, for a material with $n > 5$, most of the loaded volume experiences a constant level of equivalent stress. This contention remains, however, open to question and further investigation.

Finally, it may be noted that most of the previous studies in which good correlation was reported between creep parameters obtained by indentation and by tensile testing were carried out on materials which creep rapidly at room temperature – notably Pb, Sn and In. In such materials, with very high secondary creep rates, the effects of primary creep, and the sensitivity to changes in creep mechanism, may be less significant than with more creep-resistant material. It is possible that the advent of equipment designed for routine indentation testing at substantially elevated temperatures may soon allow a much wider range of materials to be tested under such conditions, and lead to more reliable results. Certainly the capacity to test over a range of temperature, and hence to obtain activation energy values as well as stress exponents, will allow more systematic checks to be made on the internal consistency of the results obtained. At present, however, high temperature nanoindentation is still in its infancy, and it appears that the issues outlined above are inhibiting the use of nanoindentation for reliable measurement of creep parameters for many materials.

4.3. A proposed parameter characterizing resistance to creep indentation

The above discussion suggests that, at least in many cases, it may be difficult or impossible to evaluate by room temperature indentation testing the creep parameters measured in conventional tensile tests. However, differences are clearly apparent between the indentation creep behaviour of different materials, and it should at least be possible to quantify the indentation creep behaviour in some way. The current ISO standard method [35] for quantifying the extent of creep in nanoindentation, is quite conservative and does not include any analysis to obtain values of n or Q. The recommended procedure involves expressing the deformation during a specified period under constant load as a percentage of the deformation observed up to the start of the hold period. To compare different materials, the time and load must be the same. A potential deficiency with this method is illustrated in Fig. 8a. It is possible that quite different material behaviour could produce the same result. In effect, the gradient of the curve, as well as the distance the indenter has already moved, both reflect the propensity of the material to deform, up to the point concerned.

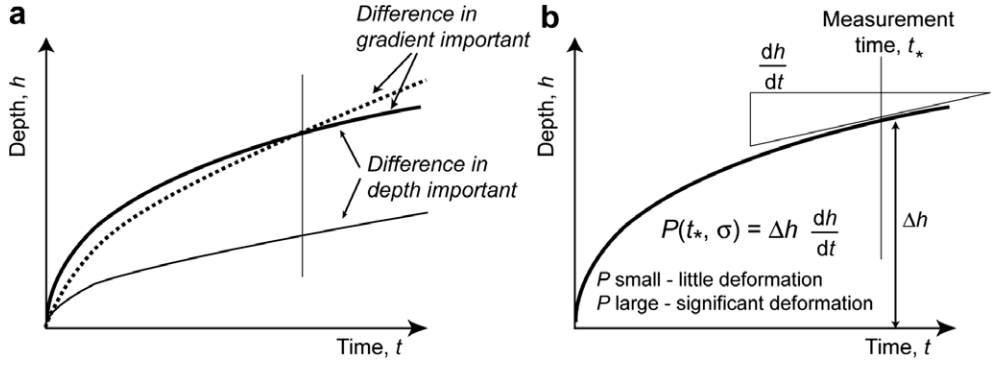

Fig. 8. (a) Illustration of how differently shaped constant load penetration curves could produce the same depth after a certain time, but be different at other times. Both the depth and the rate of depth increase are important: (b) analysis leading to a parameter, P, taking this effect into account.

Table 2
Calculated values of parameter P, designed to characterize the observed constant load deformation, after different dwell times

Material	Value of P (m^2/s)				Value of $\sqrt{(P/t)}$ (mm/s)			
	$t = 300$ s	$t = 600$ s	$t = 900$ s	$t = 1200$ s	$t = 300$ s	$t = 600$ s	$t = 900$ s	$t = 1200$ s
Alumina	1.0	0.5	1.7	0.9	57.7	28.9	43.5	27.4
Chromium	0.7	1.0	2.0	2.4	48.3	40.8	47.1	44.7
Aluminium	6.5	5.0	3.8	5.5	147.2	91.2	65.0	67.7
Zinc	36.1	42.9	45.3	42.5	346.9	267.4	224.3	188.2

Also shown are $\sqrt{(P/t)}$ values. All tests were performed at room temperature, using a force of 10 mN.

In order to account for this effect, a simple procedure is proposed, which generates a number representing the tendency of a material to deform under constant load indentation, $P(t^*, \sigma)$ (see Fig. 8b). For specified test conditions (temperature, indentation load and duration), the product of the increase in depth during the constant load period and the gradient of the curve at that point yield a number (P), which has units of m^2/s. If this number is low, the extent of deformation in the material has been, and should continue to be, small. A high value indicates that significant deformation is expected to occur under constant load indentation conditions. Validating this approach using data produced in the current work is complicated by the fact that different loads were used for different materials. However, several samples were tested with 10 mN loads, so evaluation of P for these cases after various times (300 s, 600 s, 900 s and 1200 s) allows some systematic data to be examined for comparative purposes, and calculated values of P for four materials are shown in Table 2. Also shown are values of $\sqrt{(P/t)}$, which has units of velocity and might be expected to give some sort of indication as to whether the average creep rate is changing much over these periods. It can be seen that $\sqrt{(P/t)}$ remains approximately constant in each case, at least within a factor of about 2.

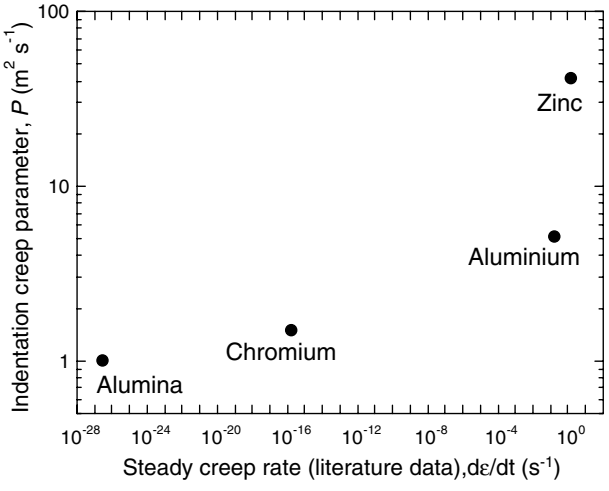

Fig. 9. Average values of P for four materials, obtained from the indentation test data shown in Table 2, plotted against corresponding calculated steady-state creep rates at ambient temperature, with an applied stress equal to that given by Eq. (1) for the indentation conditions concerned.

In Fig. 9, the average value of P is plotted against the corresponding predicted steady-state creep rate at room temperature, for an applied stress equal to the indentation stress at the start of the test. It can be seen that the parameter does at least rank these materials correctly, but it should be emphasized that it does not represent any fundamental creep characteristic, and this comparison is so limited that it is far from clear whether it could reliably be used even for ranking purposes. Probably the most positive general conclusion that can be drawn is that it may be possible to use data obtained during indentation testing to give at least some indication of creep characteristics, but much further work is needed to clarify the full capability and limitations of the approach. It seems likely that only when testing can be carried out over a range of temperature will comprehensive characterization be possible, since that will at least allow separation of stress and temperature sensitivities.

5. Conclusions

The following conclusions can be drawn from this work:

(1) Constant load nanoindentation creep testing at room temperature has been carried out on 15 different materials, with extensive repetition of individual experiments, and a well-established procedure used to measure creep stress exponents. However, there is virtually no correlation between these measured values and those in the literature for the materials concerned (obtained by conventional creep testing). This is unfortunate, since nanoindentation testing has several important advantages over conventional tensile testing, particularly for creep deformation.
(2) It is concluded from this result, and from detailed consideration of the theoretical basis for these procedures (in the light of actual conditions expected during nanoindentation creep testing), that the methodology employed hitherto is inherently unreliable, and hence subject to large errors arising from small variations in test conditions or data interpretation.
(3) A major source of error may be that associated with the fact that much of the material under stress is likely to be in the primary creep regime, in which case the associated strain rates will tend to be higher and more variable than during the steady-state creep which is assumed to be taking place. The relatively

short duration of most nanoindentation testing, the high gradients of stress and strain rate in the vicinity of the indenter and the continuously increasing size of the deformation zone may all be contributing to this effect.

(4) An indentation creep parameter is proposed, which can readily be obtained experimentally and may be used to give an indication of the significance of creep under the conditions concerned. However, it is not a fundamental creep parameter, and it is not clear whether it can be related in a systematic way to creep characteristics obtained during conventional steady-state creep testing with a uniform stress field.

(5) Further experimental and theoretical work in the area is clearly required. It seems likely that systematic creep characterization by nanoindentation testing will only be possible if the testing can be carried out over a range of temperatures, in which case activation energy data can be obtained and the internal consistency of deduced combinations of Q and n values can be systematically checked. Equipment suitable for carrying out high and low temperature nanoindentation may thus be useful in this context, although the uncertainties and sources of error highlighted in the present paper are such that reliable extraction of basic creep parameters may prove difficult even when such testing facilities are available.

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References

[1] Oliver WC, Pharr GM. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. J Mater Res 1992;7:1564–83.
[2] Lawn BR, Marshall DB. Hardness, toughness and brittleness: an indentation analysis. J Am Ceram Soc 1979;62:347–50.
[3] Beake B, Goodes SR, Smith JF. Micro impact testing: a new technique for investigating thin film toughness, adhesion erosive wear resistance and dynamic hardness. Surf Eng 2001;17:187–92.
[4] Chudoba T, Richter F. Investigation of creep behaviour under load during indentation experiments and its influence on hardness and modulus results. Surf Coat Technol 2001;148:191–8.
[5] Samuels LE, Mulhearn TO. An experimental investigation of the deformed zone associated with indentation hardness impressions. J Mech Phys Solids 1957;5:125–34.
[6] Mulhearn TO. The deformation of metals by Vickers-type pyramidal indenters. J Mech Phys Solids 1959;7:85–96.
[7] Marsh DM. Plastic flow in glass. Proc Roy Soc Lond A 1964;279:420–35.
[8] Hill R. The mathematical theory of plasticity. London: Oxford University Press; 1950.
[9] Johnson KL. The correlation of indentation experiments. J Mech Phys Solids 1970;18:115–26.
[10] Mulhearn TO, Tabor D. Creep and hardness of metals: a physical study. J Inst Met 1960–1961;89:7–12.
[11] Bower AF, Fleck NA, Needleman A, Ogbonna N. Indentation of a power law creeping solid. Proc Roy Soc Lond A 1993;441:97–124.
[12] Atkins AG, Silverio A, Tabor D. Indentation hardness and the creep of solids. J Inst Met 1966;94:369–78.
[13] Pollock HM, Maugis D, Barquins M. Characterisation of submicrometre surface layers by indentation. In: Blau PJ, Lawn BR, editors. Microindentaion techniques in materials science and engineering. Philadelphia (PA): ASTM; 1986. p. 47–71.
[14] Mayo MJ, Nix WD. A micro-indentation study of superplasticity in Pb, Sn, and Sn–38 wt.% Pb. Acta Metall 1988;36:2183–92.
[15] Posil WH, Oliver WC, Fabes BD. The relationship between indentation and uniaxial creep in amorphous selenium. J Mater Res 1995;10:2024–32.
[16] Voorhees HR. Assessment and use of creep rupture properties. In: Kuhn H, Medlin D, editors. ASM handbook. Mechanical testing and evaluation, vol. 8. Materials Park (OH): ASM International; 2000. p. 383–97.
[17] Fatay D, Gubicza J, Szommera P, Lendvai J, Blétry M, Guyot P. Thermal stability and mechanical properties of a Zr-based bulk amorphous alloy. Mater Sci Eng A 2004;387–389:1001–4.
[18] Westbrook JH. Temperature dependance of the hardness of pure metals. Trans ASM 1953;45:221–48.
[19] Sargent PM, Ashby MF. Indentation creep. Mat Sci Technol 1992:594–601.
[20] Chu SNG, Li JCM. Impression creep; a new test. J Mat Sci 1977;12:2200–8.
[21] Li JCM. Impression creep and other localised tests. Mater Sci Eng 2002;A322:23–42.
[22] Hannula SP, Li CY. Repeated load relaxations of type-316 austenitic stainless-steel. Scripta Metall 1984;18:225–9.
[23] LaFontaine WR, Yost B, Black RD, Li CY. Indentation load relaxation experiments with indentation depth in the submicron range. J Mater Res 1990;5:2100–6.
[24] Lucas BN, Oliver WC. Indentation power-law creep of high-purity indium. Metall Mater Trans 1999;30A:601–10.
[25] Mayo MJ, Siegel RW, Narayansamy A, Nix WD. Mechanical properties of nanophase TiO_2 as determined by nanoindentation. J Mater Res 1990;5:1073–81.
[26] Raman V, Berriche R. An investigation of the creep processes in tin and aluminium using a depth-sensing indentation technique. J Mater Res 1992;7:627–38.
[27] Asif SAS, Pethica JB. Nano-scale indentation creep testing at non-ambient temperature. J Adhesion 1998;67:153–65.
[28] Frost HJ, Ashby MF. Deformation mechanism maps – the plasticity and creep of metals and ceramics. Oxford: Pergamon; 1982.
[29] Sun Y, Bell T, Zheng S. Finite element analysis of the critical ratio of coating thickness to indentation depth for coating property measurements by nanoindentation. Thin Solid Films 1995;258:198–204.
[30] Fivel M, Verdier M, Canova G. 3D simulation of a nanoindentation test at a mesoscopic scale. Mat Sci Eng A 1997;234:923–6.
[31] Bressan J, Tramontin A, Rosa C. Modeling of nanoindentation of bulk and thin film by finite element method. Wear 2005;258:115–122.
[32] Li WB, Henshall LJ, Hooper RM, Easterling KE. The mechanisms of indentation creep. Acta Metall Mater 1991;39:3099–110.
[33] Lucas BN, Oliver WC. The elastic, plastic and time dependent properties of thin films as determined by ultra low load indentation. MRS Symp Proc 1992;239:337–41.
[34] Ogbonna N, Fleck NA, Cocks ACF. Transient creep analysis of ball indentation. Int J Mech Sci 1995;37:1179–202.
[35] Metallic materials – instrumented indentation test for hardness and materials parameters – part 1: test method. In: International Standard ISO 14577-1; 2002.