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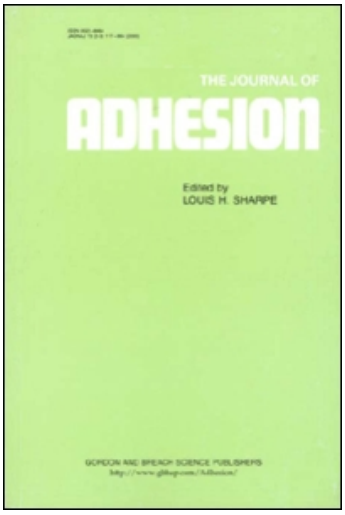

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Nano-Scale Indentation Creep Testing at Non-Ambient Temperature

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Nano-Scale Indentation Creep Testing at Non-Ambient Temperature*

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Depth sensing nanoindentation can be used to study the time-dependent deformation of very small volumes of materials, contacts, and thin films. Force modulation provides a continuous measure of the contact stiffness during an indentation, and minimises the adverse effect of thermal drift which is particularly important for sub-micron samples. Most of the nanoindentation experimental work so far has been carried out at room temperature. In this paper we describe a solid-state thermoelectric heating and cooling system which gives a straightforward way to vary the temperature of both sample and tip. The capabilities of the technique are demonstrated by observing the time and temperature dependent creep properties of high purity Indium. Hardness, its strain rate dependence, the stress exponent, and the activation energy for the creep process can all be directly measured from nanometre scale contacts, and the values obtained are similar to those from bulk conventional creep testing. The technique is likely to be of particular value for polymer thin films.

Keywords: Nanoindentation; creep; sub-micron testing; hardness; strain rate; activation energy

INTRODUCTION

Indentation hardness testing has been in use for over a century in the study of mechanical properties of materials [1]. Recently there has been a increased interest in studying the mechanical properties of extremely small volume of materials, thin films, surface coatings and

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interfaces over the length scale of few nanometers. Conventional hardness testing cannot be used for such small volumes because optical measurement of the indent size is not feasible. The more recent depth sensing indentation technique, known as Nanoindentation [2], avoids the need for imaging and also allows evaluation of other parameters, such as elastic modulus, continuously during a single indent. Nanoindentation is thus gaining much attention for sub-micron scale mechanical property measurements [3].

In depth sensing indentation a controlled, variable load is applied to the indenter and the resulting displacement as the indenter penetrates the specimen is monitored continuously. The load *vs.* displacement data obtained in an experiment are analysed using the known geometry of the indenter to give mechanical properties such as hardness and Young's modulus, with the aid of a suitable model [4]. The creep properties of the material can be obtained using techniques such as indentation load relaxation test, constant rate loading test and constant strain rate loading test [5–9].

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To date, nanoindentation testing has mainly been used to obtain the mechanical properties of materials at room temperature. To extend the technique to non-ambient temperatures it is essential that both tip and sample be set to the same temperature [10]. Recently, Oliver *et al.* [7] have reported the use of a high temperature micro probe for hardness testing. They showed that the creep properties and the activation energy for the creep process can be measured at a sub-micron scale, and at different temperatures under ultra high vacuum. Resistance heating and liquid nitrogen cooling were used to vary the temperature of the entire measurement system; this necessitated the use of a laser interferometer displacement sensing system.

In this paper we report a simple and cost effective solid-state heating and cooling system for existing Nanoindenters, enabling several tens of degrees excursion from room temperature. Only the immediate areas of sample and tip are heated. A solid state thermoelectric heat pump (Peltier cell) avoids thermal noise, has low mass, and it is simple to control the temperature with very high stability. We show that the thermal drift in the system is very small and, coupled with force modulated indentation, it can be used to study the creep properties over long time periods. We also show the equivalence of the creep data obtained by both direct displacement measurement and AC modula-

tion. Therefore, the data are from indents in excess of 1 μm in depth where, with care, DC measurements can be reliable. Much smaller indents can be observed with the AC method alone [8, 11].

EXPERIMENTAL

The experimental set-up is based on a Nanoindenter II [12], with Berkovitch diamond indenter tip. A schematic of the set up is shown in Figure 1. The indenter shaft is suspended by a system of leaf springs inside a housing head. The load is provided by passing a current through a coil and magnet assembly at the top of the shaft. The displacement of the indenter is measured by a capacitance gauge. The load resolution of the system is about 0.3 μN and the displacement resolution is < 0.05 nm. Parameters such as approach speed, load, and the rate of loading and unloading, are controlled by computer, and can be set over very wide ranges.

The temperature of the indenter tip and the specimen sample are varied using two separate thermoelectric Peltier cells. For the indenter tip assembly a thermoelectric heat pump is clamped between a small heat sink and small aluminium plate. The Berkovitch diamond tip is screwed to the small aluminium plate and the other side with the heat sink is screwed to the indenter shaft as shown in Figure 1. To monitor the temperature of the indenter a thermocouple is attached directly to the indenter tip. The overall mass of the indenter assembly complete with Peltier cell is less than 7.5 grams. The stiffness of the leaf spring along with the thermocouple and Peltier cell leads is approximately 70 N/m. The specimen stage consists of a thermoelectric Peltier cell clamped between a copper plate and an aluminium heat sink (Fig. 1). The heat pump is fastened to the heat sink by four acrylic screws. The specimen is clamped to the copper plate, and to monitor the specimen temperature a thermocouple is directly attached to the specimen surface. The specimen stage is fixed to the motorised X-Y stage which carries the specimen between the indentation position and optical microscope used to inspect the surface and pre-program placement of the indents. The whole system rests on a heavy table, which is pneumatically suspended to insulate from building vibrations. The indentation system is housed in an acoustically and thermally

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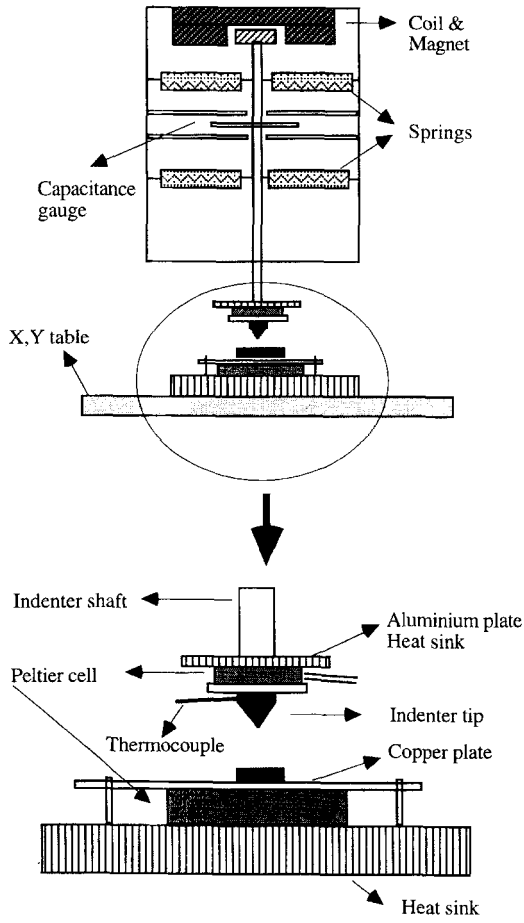

FIGURE 1 Schematic of variable temperature Nanoindenter.

insulated wooden cabinet. The temperature of the room in which the equipment is kept is carefully regulated to minimise the thermal fluctuation.

To accomplish precise temperature control of the indenter tip and the specimen stage, two separate custom built temperature controllers were used. The set temperature and the rate of heating or cooling can be adjusted manually or programmed through a computer. The temperature is monitored through a digital voltmeter and is

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continuously logged. The thermal stability of the system is better than 10 milli-Kelvin over a period of hours. Once the system reaches thermal equilibrium the displacement drift in the system is less than 0.05 nm/s. We note that this system enables cooling as well as heating. However, in our present system, the air in the test zone could not be dried sufficiently to prevent condensation and ice formation at temperatures near zero C – these obviously disturb the measurement. A controlled environment would enable measurements to below 230 K.

THEORY

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In indentation creep, if the indentation depth, h, is monitored as a function of time, the mean strain rate, $\dot{\varepsilon}$, is commonly defined as [7]

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

The mean contact stress is

$$\sigma = \frac{P}{A} \tag{2}$$

where P is the load and A is the area of the contact; $A = 24.5\,h^2$ at larger depths for the Berkovitch indenter in use here. With the depth sensing indentation technique, as the scale of indentation decreases, so also do the displacements involved in creep tests. They thus become very sensitive to small thermal drifts in displacement, and this can render displacement measurements almost useless for creep studies if the experiments exceed several seconds. To overcome this problem, Weihs and Pethica [9] used AC force modulation, and measured the contact stiffness rather than displacement, as a function of time. The stiffness is directly determined simply by dividing the applied force modulation amplitude by the amplitude of the resulting AC displacement, which can be measured with good signal-to-noise by a lock-in amplifier. DC displacements, and hence drift, do not affect the measurement, which is typically made at a few tens of Hertz.

In an indentation experiment the contact stiffness, S, is proportional to the contact area, A [14, 13]

$$S = 2E^* \sqrt{\frac{A}{\pi}} \tag{3}$$

The effective strain rate can then be defined as

$$\dot{\varepsilon} = \frac{1}{S} \frac{dS}{dt} \tag{4}$$

and the mean stress or hardness, σ, can evaluated from

$$\sigma = \frac{P}{\pi} \frac{4}{S^2} E^{*2} \tag{5}$$

where P is the load and E^* is the reduced elastic modulus [9]. If the modulus of the material is known then the hardness of the material can be calculated. With the AC modulation technique, since the resolution of the stiffness measurement can be 50 N/m or better and E^* is often $> 10^{10}\,\mathrm{N/m^2}$, the motion of just a few atoms can be detected [9].

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INDENTATION CREEP OF INDIUM

Our apparatus was used to study the creep properties of electropolished high purity indium at different temperatures. Before the experiment commenced, the indenter tip and the specimen were heated to the desired temperature. Although it took only a few minutes to reach the desired temperature, more time was allowed to ensure that the system reached complete thermal equilibrium. To measure the contact stiffness continuously during an indentation, a small sinusoidal AC force of 8 μN at 40 Hz was added to the applied force, and the resulting oscillation in displacement was monitored using a lock-in amplifier. The amplitudes and the phase difference between force and AC displacement were used to calculate the contact stiffness as described by Pethica and Oliver [13].

After the system reached thermal equilibrium, the tip was brought to the specimen surface at a velocity of 4 nm/s. When the tip contacted

the specimen surface the resulting phase shift in the AC displacement signal was used to determine the initial point of contact. After the tip contacted the specimen surface, the load was held constant and the thermal drift monitored. The experiments were carried out only when the DC thermal drift was less than 0.05 nm/s. To measure the creep properties, the load was then ramped up to maximum load of 5 mN in one second. After reaching the maximum load, the load was held constant for 1600 sec. After this first hold period, the indenter was then unloaded to 20% of the original maximum load. It was then held at that load to check again the thermal drift during the experiment. After this second hold segment the indenter was completely unloaded.

RESULTS AND DISCUSSION

The responses during the constant load hold segment, at three different temperatures, are shown in Figure 2. Figure 2(a) shows that the indenter displacement increases with time after the load step, and for any given time the indenter displacement increases with temperature. The same can be seen in Figure 2(b) which shows stiffness *vs.* time data. Both displacement and stiffness data can be used to calculate the

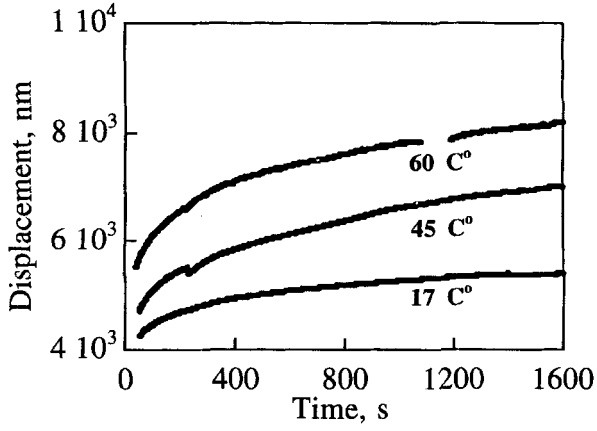

FIGURE 2 Time behaviour of DC displacement (a) and AC stiffness (b) following step increase in load.

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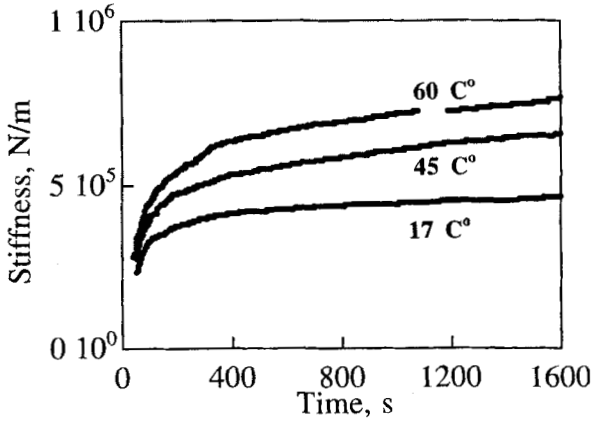

FIGURE 2 (Continued).

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mean contact stress independently using Eqs. (2) and (5). Figures 3(a) and (b) show the results. It can be seen that the mean contact stress relaxes as a function of time, and the rate at which it relaxes increases with temperature.

It should be noted that the displacement and the contact stiffness measurements are effectively independent. Contact stiffness gives information on changes in contact area with time, whilst vertical displacement of the indenter gives information regarding the relaxation perpendicular to the surface beneath the indenter. In principle, if geometric similarity of the indent is maintained, both the measurements should give the same result. However, in the initial stages of the indentation immediately after the rapid application of the load, there are material responses due to the high strain rates present which complicate this simple geometrical comparison. In the data described in this paper therefore, we only show the response at times greater than 100 seconds after the initial load application. Figure 4 shows the plot of hardness calculated from both displacement measurement and contact stiffness measurement. The excellent correlation between both measurements can be seen. We have specifically used data from relatively deep indents for this comparison, to minimise the effects of DC drift. The similarity between AC and DC data gives confidence that at much smaller indent depths, where only AC data can be used,

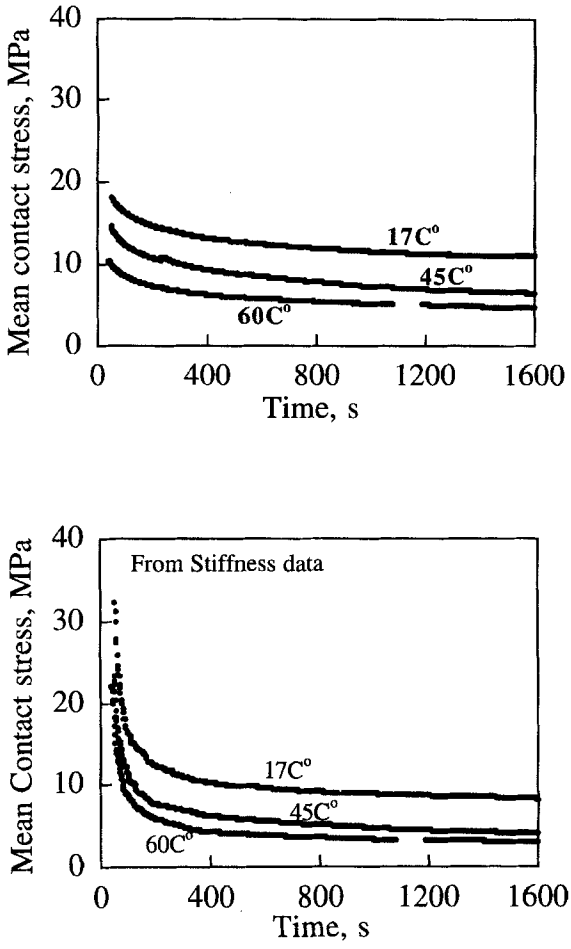

FIGURE 3 Evolution with time of mean contact stress following step increase in load, (a) from displacement, and (b) from AC stiffness.

the results give a realistic description of the material creep properties at that scale.

By simple (numerical) differentiation the rate of change of stiffness (dS/dt) can be obtained continuously during the constant load hold segment. The indentation strain rate can then be obtained with Eq. (4). The same can be done with the displacement *versus* time data. Figure 5 shows that the strain rates calculated by both measurements are again

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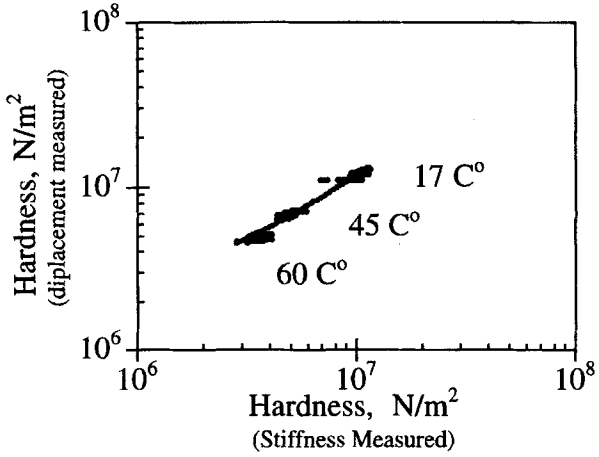

FIGURE 4 Comparison of hardness values determined from displacement and AC stiffness.

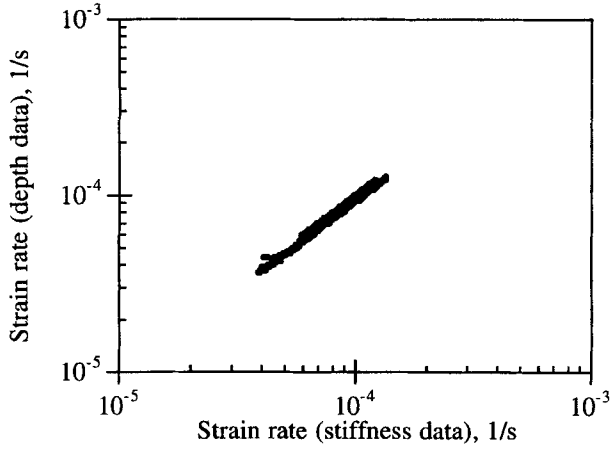

FIGURE 5 Comparison of strain rate values determined from displacement and AC stiffness.

in excellent agreement at all the temperatures studied. From the hardness and strain rate data at given times, one can produce plots of indentation strain rate *versus* mean contact stress. Figure 6 is a log-log plot of indentation strain rate *versus* mean contact stress, at temperatures of 17, 45 and 60 °C. The slope of the log-log plot is the stress exponent for the creep process. At 17 °C the calculated stress

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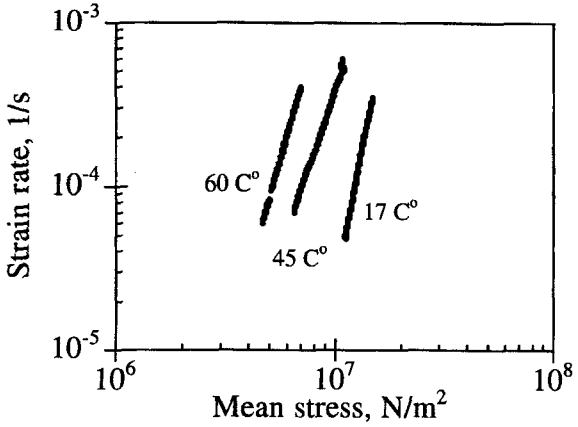

FIGURE 6 Mean stress *vs.* strain rate for three temperatures.

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exponent is 7.7. This is in good agreement with the stress exponent obtained in a conventional, uniaxial creep test [14]. However, at 45 °C the exponent is 4.5 ± 0.5, and at 60 °C the exponent is 4.8 ± 0.5. These latter results were obtained a number of times and are repeatable within the bounds given.

Creep at temperatures above 0.5 T_m is expected to follow an Arrhenius type of behaviour, and the strain rate can be written as

$$\dot{\varepsilon} = \dot{\varepsilon}_0 \exp(Q/RT)$$

where Q is the activation energy, R is the gas constant, and T is the absolute temperature. The activation energy, Q, of the creep process can, therefore, be obtained from the slope of the semi-log plot of strain rate *versus* $1/RT$. Figure 7 shows the result for our three temperatures. The activation energy deduced is 77 KJ/mol. This is in good agreement with the activation energy for self diffusion in pure indium which is in the range of 75–78 KJ/mol [15].

However, it should be noted that although the measured data are quite repeatable, the interpretation as a single Arrhenius plot is an almost certainly an oversimplification. The significant changes in apparent strain rate index with temperature mean that a true activation energy is not easily defined. The removal of the short-time response data, described above, should also be borne in mind. We also

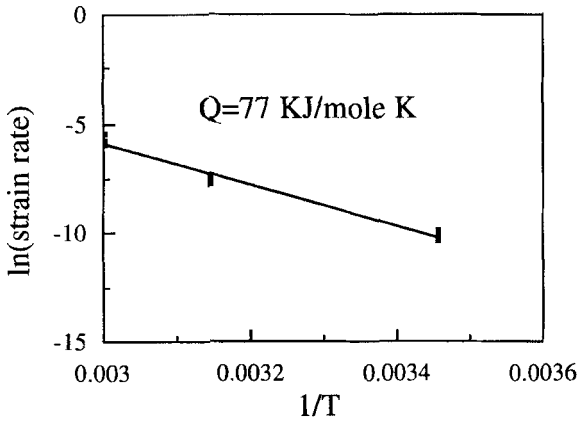

FIGURE 7 Strain rate at fixed stress, *vs.* temperature. The slope gives the activation energy within a simple Arrhenius model.

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recall that in an indentation test the sample has a very wide range of actual strains present at any given time, along with a complex non-uniform elastic field, and so differs significantly from the single strain state in a uniaxial test. It is perhaps, therefore, not reasonable to expect a high degree of accuracy and comparability with traditional creep testing. At least in the experiments described here the loading state is defined, since it is not necessary to remove the indenter to measure an indent area.

In spite of the complexities, we consider that the above results serve to confirm the utility of the effective strain rate defined in Eqs. (1) and (4), and the repeatable observation of temperature dependence of creep. The method has the great merit of the ability to test very small sample volumes, and therefore may be able to investigate local spatial variations across samples.

CONCLUSION

We have shown how a relatively straightforward modification to a Nanoindentation apparatus allows study of the temperature-sensitivity of time-dependent mechanical deformation in sub-micron volumes. The results are in quite good agreement with both earlier

indentation tests, and traditional uniaxial test methods. In addition to stress exponents for creep, the new apparatus gives a good measure of creep activation energies. The technique should enable measurement of hardness, creep, and modulus (G' and G'', as a function of both temperature and modulation frequency) in systems such as polymer thin films and small adhesive junctions, which are not amenable to traditional testing techniques.

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