

Measurement of an indentation size effect for Indium as a function of temperature

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Abstract

We present compression and instrumented indentation experiments on high-purity Indium, at temperatures from 22 to -100°C. A strong indentation size effect is detected at all temperatures, corresponding to a large material length-scale. Therefore the large length-scale is not a consequence of creep mechanisms but an intrinsic property of the material.

Keywords: Indium, size effect, length-scale, indentation, compression, low-temperature.

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1. Introduction

In a paper recently appeared in this journal [1] we investigated experimentally the size-dependence of the plastic response of Indium in bending and indentation at room temperature. We showed that the material displays a pronounced size effect at mm-scale. As at room temperature Indium is at a homologous temperature of 0.69 and deforms by power-law creep, it remained unclear if the large length-scales associated with the observed size effects were a consequence of creep mechanisms, such as the occurrence of dislocation climb as opposed to glide.

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To clear this doubt we report instrumented Vickers indentation measurements, at temperatures from 22°C to -100°C, corresponding to a homologous temperature from 0.69 to 0.40. We calculate the indentation length-scale according to the Nix and Gao [2] model and we correlate this to the yield strain of the material. For the sake of brevity we refer the reader to [1] for a review of the literature relevant to the subject investigated.

2. Experimental methods

Specimens were fabricated from high-purity Indium (99.999%) with grain size of order 0.5 mm, identical to the material used in [1] for room-temperature tests. We refer the reader to [1] for details of the manufacturing procedure and annealing cycle.

The Indium ingot initially had an isotropic polycrystalline structure with grains of average diameter 0.5 mm. As the material was very soft and could not be polished mechanically, these large grains were revealed by gently pressing a microscope slide towards the ingot to obtain a locally flat surface, etching for 10 s with a 2% solution of HCl in distilled water, and subsequent observation in an optical microscope. Flat indium plates of thickness between 5 mm and 7 mm were used for the indentation tests, while cylinders of diameter of 3.5 mm and height 3 mm were used in compression experiments.

Compression tests were conducted with a DMTA (TA Instruments, RSA-G2) using nitrogen vapour to cool the atmosphere of the test chamber to temperatures of -20°C and -100°C. Force and displacement were measured by the sensors integrated in the DMTA, which also performed an automatic compliance calibration. The displacement rate was chosen to impose a nominal compressive strain rate of 10^{-3} s^{-1} . The maximum force our DMTA could exert was of approximately 35 N, corresponding to a maximum compressive stress of around 3.5 MPa.

Vickers instrumented indentation was carried out with the same DMTA analyser, at the same temperatures. A diamond Vickers indentation tip was displaced, at constant velocity $\dot{h} = 7.5 \cdot 10^{-3} \text{ mm/s}$, against a stationary, flat and smooth Indium plate, whilst recording the indentation force and the indenter displacement at a frequency of 100 Hz.

In both experiments the test chamber was purged with nitrogen vapour for at least 5 minutes, to prevent the formation of ice on the sample and loading fixtures.

3. Analysis of the indentation data

The force versus displacement measurements in indentation were processed to plot graphs of Vickers micro-hardness versus indentation depth, from knowledge of the indenter geometry and the assumption of negligible pile-up or sink-in, in line with microscope observation of the indentations. In [1] we developed a simple analytical model for indentation of a rigid-viscoplastic solid with a size-independent response. The model predicts

$$H = K\dot{h}^N h^{-N} \quad (1)$$

where H is the material hardness, h is the indentation depth, and K, N are parameters to be determined experimentally. Equation (1) predicts that the hardness decreases with increasing indentation depth for a size-independent material response.

Nix and Gao [2] formulated predictions of the hardness versus indentation depth for rate-independent materials displaying indentation size effects as

$$H = H_0 \sqrt{1 + \frac{h^*}{h}} \quad (2)$$

where H_0 and h^* are to be determined by fitting of eq. (2) to measurements; h^* represents a length-scale associated to the indentation size effect, while H_0 is the hardness at sufficiently large indentation depth ($h \rightarrow \infty$) and can be related to the yield strain ε_y by a relationship given by Tabor [3], as

$$\varepsilon_y = \frac{cH_0}{E} \quad (3)$$

In the following we will assume that the Young's Modulus is approximately independent of temperature and $E = 12.3$ GPa. The non-dimensional constant c is taken as 0.34, as suggested by [3]. We note that following the derivation in [2] and assuming a rate-dependent material response as in [1], one could obtain

$$H = K\dot{h}^N h^{-N} \sqrt{1 + \frac{h^*}{h}} \quad (4)$$

which represents the indentation response of a rate-dependent solid with size-dependent indentation response.

4. Results and discussion

Representative compressive true stress versus strain curves are shown in Fig. 1. The material shows yielding at negligible load, followed by considerable strain hardening; at room temperature we also observe a plateau phase, corresponding to dislocation saturation. At lower temperatures the material is clearly harder to deform; tests at low temperatures had to be terminated at nominal stresses of order 3.5 MPa due to limitation in force capacity of the DMTA and for this reason the saturation phase is not observed. The material's yield stress increases with decreasing temperature as expected; correspondingly, the hardness is expected to increase.

In Fig. 2 we present load versus indentation depth histories at three different temperatures, which confirm the increase in hardness as the temperature decreases. Three repetitions are shown at each temperature to illustrate scatter. The corresponding histories of Vickers microhardness as a function of the indentation depth are shown in Fig. 3. These were obtained assuming negligible pile-up and sink-in. We remind the reader that the specimens used for indentation had been produced by hot-compression of small pieces of the original ingot between microscope slides, to plastic strains of approximately 50%. The grain size in the direction of the indentation load was therefore of 250 μm , while the transverse grain size was approximately 700 μm . The maximum indentation depth was less than 250 μm .

Both material rate-dependence and size-dependence result in increasing hardness at decreasing indentation depth, via eq. (4). It is difficult, however, to separate the two effects, as this would require data at very large depths, which could not be obtained due to the small size of the indentation tips used.

We note that at room temperature the creep exponent for the material was measured as $N = 0.2$; correspondingly eq. (1) predicts, for the range of indentation depths shown in Fig. 3 (10 – 260 μm), an increase in hardness with decreasing depth of approximately 90%. However, the measured increase in hardness is much larger, of up to 300% in the same range of depths. At lower temperatures, the creep exponent is expected to be smaller than 0.2, and to be close to 0 at -100°C, such that the change in hardness due to rate effects is considerably smaller than 90%; in contrast, the measurements show an increase in hardness of 250 – 400%. We deduce that the material displays a strong indentation size effects at all temperatures explored.

To quantify the observed size effect and to compare it to the case of measurements on other metals we calculate moving averages of the experiments at each temperature (for the range $h > 10 \mu\text{m}$,

such to eliminate the effects of the roundness of the indentation tip) and we perform a best fit of this moving average to eq. (2); this allows obtaining the hardness and the indentation length-scale, H_0 and h^* ; the thicker lines in Fig. 3 correspond to the fit of eq. (2) to the data. The values of h^* determined are of order 100 μm . We note that fitting the Nix-Gao model (eq. (2)) to the data corresponds to neglecting rate effects; this may be appropriate at -100°C (homologous temperature 0.4) but less adequate as the temperature increase; nonetheless we use a fit of the Nix-Gao model to the data to quantify the magnitude of the size effect.

The indentation length-scale h^* , obtained by fitting eq. (2) to the measurements, and the corresponding yield strain at the three temperatures investigated in this study are presented in Fig. 4 together with published data for other metals and alloys at room temperature [4-9]. Indium displays the strongest size effect irrespective of the test temperature, with the length-scale h^* ranging from 66 to 144 μm . This suggests that the large observed material length-scales are not a consequence of creep mechanisms but are rather dependent on the atomic structure of the material and on the details of its dislocation distribution. The measurements at room temperature shown in Fig. 4 seem to support a correlation between length-scale and material yield strain, which in turn depends on the mean distance travelled by dislocations. The material yield strain is not an adequate indicator of the material length-scale when data at different temperatures are considered.

5. Conclusion

We measured the compression and instrumented indentation responses of high-purity Indium at temperatures in the range -100°C to 22°C , corresponding to homologous temperatures 0.4 to 0.69. Both responses were stronger at lower temperatures as expected. Material length-scales at different temperatures were obtained by fitting the Nix-Gao indentation model to the measured data; length-scales were of order 100 μm and were shown to vary only mildly in the temperature range explored. We concluded that these large length-scales are not a consequence of mechanisms associated to creep but depend on the atomic structure of the material and on the details of its dislocation distribution. Further analysis and observations at micron and sub-micron scale are required to shed light on the mechanisms responsible for the measured size effect.

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