Pergamon

Scripta Materialia, Vol. 34, No. 4, pp. 559-564, 1996
Elsevier Science Ltd

1359-6462/96 $12.00 + .00

0956-716X(95)00524-2

MICRO-HARDNESS OF ANNEALED AND WORK-HARDENED COPPER POLYCRYSTALS

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(Received July 20, 1995)

Introduction

Indentation size effects have been observed in a variety of materials (1–12). Generally, it is observed that the apparent hardness of the material increases as the size of the indent decreases. A variety of mechanisms can be responsible for these observations; they include the formation of microcracks under the indenter, the influence of harder surface layers and a dependence of flow strength on gradients of plastic strain. The present discussion is limited to materials which when indented deform plastically by glide of dislocations. In these materials, it is useful to think of three regimes of behaviour linked to the size scale of the indent. At very small indents (less than 100 nm), the flow stress of the material is thought to be controlled by the nucleation of single dislocations under the indentor (9,10) and depends on the crystallographic relationship between the indentor and the sample. At intermediate-sized indents (100 nm to 100 μm), deformation occurs by general plasticity involving large numbers of dislocations but hardness is still observed to be size dependent. Finally, when indent sizes are large (>100 μm) the hardness of the material becomes size-independent (11,12). The present work is primarily concerned with the intermediate size scale. It is one which appears in many other contexts such as inclusion problems, wear problems and crack tip problems: in all of these the size of the plastic zone is similar to that under the indentor, and in all of them size effects are also known. Indentation experiments are attractive because they are the most controllable method to examine plastic behaviour of metals in this regime.

One method for incorporating a scale dependence into plasticity was that suggested by Ashby (13), Brown and Stobbs (14) and others to describe the abnormally high initial work hardening rate observed in dispersion-hardened systems. In this approach, the flow stress of the material is increased due to the presence of what are known as *geometrically necessary dislocations* (15). These dislocations are required for compatibility reasons due to the presence of strain-gradients. Stelmashenko et al. (6) point out that these can explain the size dependence of indentation tests on tungsten single crystals. Fleck et al. (16,17) have recently reformulated classical plasticity theory to include the effect of strain-gradients.

The goal of the present work was to provide data on the size dependence of hardness measurements for a well characterized material such as copper. It was hoped that, by examining copper with two different starting dislocation densities (i.e. fully annealed and fully work-hardened), that information would be obtained on both the size dependence of the hardness measurement and the appropriate superposition laws

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for summing the effects of dislocations produced during indentation and the dislocations present in the starting material.

Experimental Procedure

Indentation tests were performed using a Vickers hardness indentor on a Shimadzu DUH-200 micro-hardness testing machine. This machine has the capability of performing tests at loads in the range of 1 mN-2 N. Load-displacement-time data was recorded during loading and unloading of the indentor at a loading/ unloading rate of 3.6 mN/s with a 1 second hold time between the end of the loading ramp and the beginning of the unloading ramp. The temperature of the testing machine was maintained at 22 ± 0.5°C during the test. The compliance and area-function for the indentor were determined by measuring the slope of the unloading curve in the way described by Oliver and Pharr (5). Polished tungsten, aluminum and soda glass (all nearly isotropic in their elastic properties) were used for the calibration procedure. Good agreement was observed between the area of the indents determined from the area-function and optical measurements of indent size. Measurements were then made of the indentation hardeness of annealed and work-hardened copper as a function of indent size, using flat, electro-polished surfaces.

Results and Analysis of Data

Figures 1a and 1b illustrate load-depth plots for tests performed on annealed and work-hardened copper samples to final loads of 100 mN and 2 N, respectively. In each case, two tests are presented for each condition to illustrate the excellent repeatability. In order to calculate hardness as a function of indentation depth, D, it is necessary to relate the depth of the indent to its area, A. This was accomplished using the approach described by Doerner and Nix (18) and Oliver and Pharr (5) which corrects for (i) elastic recovery of the indent upon removal of the load and (ii) elastic displacements in the testing machine:

$$D_{cor} = D_{measured} - \left(\frac{dD_{el}}{dP} + C_m \right) P \quad \text{with} \quad \frac{dD_{el}}{dP} = \frac{\sqrt{\pi}}{2E_r\sqrt{A}} \tag{1}$$

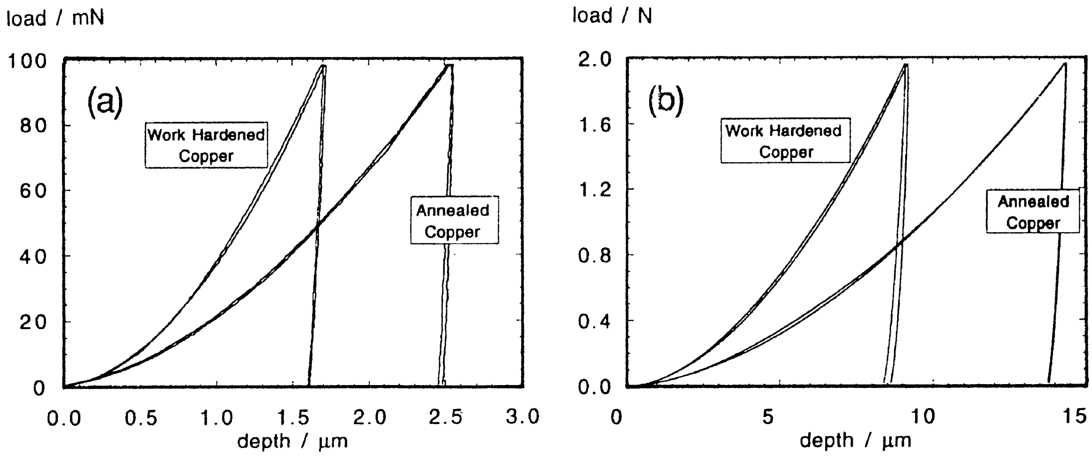

Figure 1. Load-depth curves for the micro-indentation of annealed and work-hardened copper, (a) maximum load of 100 mN, and (b) a maximum load of 2 N.

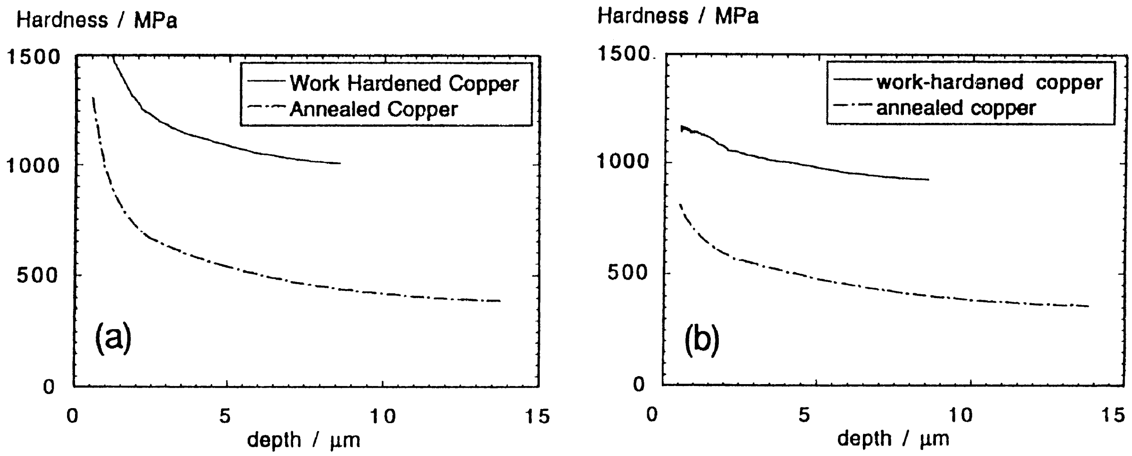

Figure 2. Hardness as a function of indent depth for annealed and work-hardened copper, (a) after correcting for machine compliance and (b) after correction for area function also.

Here D_{cor} and $D_{measured}$ are the corrected and measured displacement of the indentor respectively, P is the load applied to it, C_m is the compliance of the testing machine, A is the projected area of the indent and E_r is given by

$$\frac{1}{E_r} = \frac{(1 - \nu^2)}{E} + \frac{(1 - \nu_i^2)}{E_i} \qquad (2)$$

Here, E and ν are the Young's modulus and Poisson's ratio of the test material and E_i and ν_i are those of the indentor (1147 GPa and 0.07, respectively, for diamond). For an ideal Vickers indentor, the area is related to the square of the displacement of the indentor, but a further correction is needed to allow for the roundness of the indentor tip (5). Having obtained the corrected displacement of the indentor the hardness can be calculated using the conventional definition:

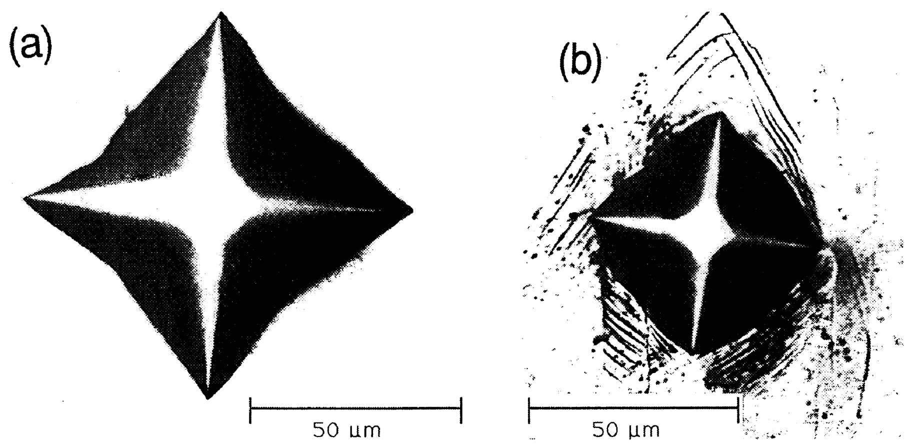

Figure 3. The profile of an indent in (a) annealed and (b) work-hardened copper.

$$H = \alpha \frac{P}{A} \tag{3}$$

where α is a constant ($\alpha = 927$ for H in MPa where P is in N and A is in $(\mu m)^2$). Figure 2a shows the result of calculating hardness as function of depth. In both the annealed and work-hardened condition, the hardness of the copper increases as the indent size decreases. Figure 3a and 3b shows micrographs of the indents in the annealed and work-hardened copper, respectively. In both cases the sides of the indent are curved, but in the opposite sense, i.e. inward curving for the annealed material and outward curving for the work-hardened material (12).

Discussion

A simple model to describe the size dependence in terms of the strain gradients can be developed as follows. The hardness is representative of the flow stress, σ, of the material ($H \approx 3\sigma$). The shear flow-stress in an FCC material like copper is related to the dislocation density, ρ, by (19):

$$\sigma = \alpha \mu b \sqrt{\rho} \tag{4}$$

where α is a constant ($\approx$0.3 for FCC materials (19)), μ is the shear modulus, *b* is the Burgers vector and ρ is the dislocation density. Then,

$$H^2 = C_1 \rho \tag{5}$$

where $C_1 = (3\alpha\mu bM)^2$ and M is the Taylor factor (M = 3.06) The dislocation density, ρ, can be considered to be made of two components which add in a linear manner (expected of obstacles of a similar strength (20)), i.e.

$$\rho = \rho_s + \rho_g \tag{6}$$

Here, ρ_s describes the density of dislocations which would accumulate in a uniform deformation of the material and ρ_g describes the additional density made necessary by the gradient of strain, γ:

$$\rho_g = \frac{1}{b} \frac{\partial \gamma}{\partial x} \tag{7}$$

For an indent, the strain-gradient, $\partial\gamma/\partial x$, can be approximated by $\Delta\gamma/w$ where w is the width of the indent ($w = 2D/\tan\beta$). Since $\Delta\gamma = \tan\beta$ we obtain:

$$\frac{\partial \gamma}{\partial x} = \frac{\Delta \gamma}{w} = \frac{\tan^2(\beta)}{2D} \tag{8}$$

where β is the inclination angle between the face of the indentor and the surface of the material being indented (22° for a Vickers indentor). Substituting (6), (7), and (8) into (5) gives:

$$H^2 = C_1 \left(\rho_s + \frac{\tan^2(\beta)}{2bD} \right) \tag{9}$$

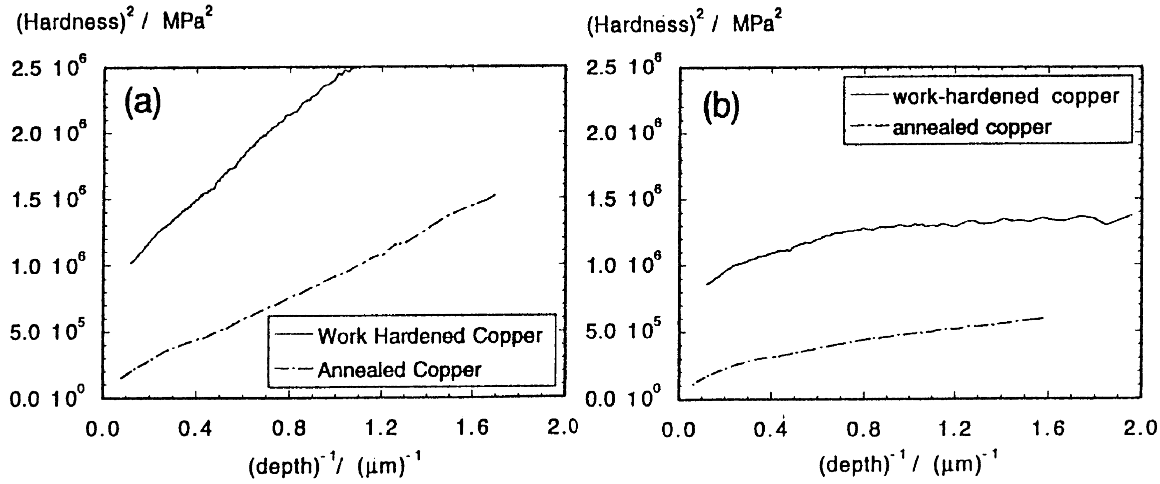

Figure 4. Plots of H^2 against D^{-1} for annealed and work-hardened copper which test the model, (a) after correcting for machine compliance and (b) after correction for area function also.

This is essentially the same result given by Stelmashenko et al. (6). It implies that a linear relationship exists between H^2 and D^{-1} if the two components of the dislocation density add in a linear manner. We have found that this method of plotting the data is very sensitive to the addition law which was used, allowing a test of superposition rules. Figure 4 shows that an approximately linear relationship exists for both the annealed and work-hardened copper. The slope is steeper for the work-hardened material, implying a steeper gradient of strain in the work-hardened copper than in the annealed copper—understandable, since the annealed material has a high work hardening rate which tends to spread the plasticity over a wide volume. This is supported by Figure 3a and 3b which show much more localized deformation around the indent in the work hardened material. The linearity of the data when plotted in this manner implies that a linear addition law for ρ_g and ρ_s is appropriate.

From equation (9) with $\mu = 42$ GPa, $b = 2.5 \times 10^{-4}$ μm, $\alpha = 0.3$ and $C_1 = 1$, the predicted slope $d(H^2)/d(D^{-1})$ has a value of 0.27 GPa².μm. The measured slope for annealed copper in Figure 4(a) is 0.85 GPa².μm—much too large. After making the area correction (Figure 4(b)) the slope becomes 0.3 GPa².μm, in good agreement with the model.

Conclusions

1) The experimental measurements of the size dependence of hardness in annealed and work hardened copper can be explained by a dislocation model incorporating geometrically necessary dislocations due to the presence of strain gradients in the deformation zone around the indent.
2) Micro-indentation offers an experimental tool for the testing and validation of theories of strain-gradient plasticity.
3) The experiments suggest that the hardening law may be taken as a simple sum of the densities of statistically-stored and geometrically-necessary dislocations.

Acknowledgements

The authors wish to express their gratitude to the U.S. Office of Naval Research under contract number N00014-91-J1916 and NSERC (Canada) for financial support, and to acknowledge the help of Alan Heaver with the experiments.

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