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Effects of loading rate on development of pile-up during indentation creep of polycrystalline copper

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ABSTRACT

Nanoindentation tests with loading rates spanning three orders of magnitude were carried out on annealed polycrystalline copper. In addition to the hardness increasing with loading rate, the formation and development of pile-up around the indentation sites were also found to be strongly rate-dependent. The development of pile-up with increased time at peak load was found to be sensitive to the prior loading rate, being much larger for tests at 50 mN/s than at 0.05 mN/s. The underlying mechanisms were investigated in terms of the kinetic aspects of the nucleation and interactions of dislocations, and can be well explained by the activation volume and the strain gradient plasticity theory.

1. Introduction

Dynamic deformation at the micro/nano scale has received great interest due to increasing demands for micro-electro-mechanical systems (MEMS). Nanoindentation has been successfully used to evaluate mechanical properties such as hardness (*H*) and elastic modulus (*E*) [1]. Rate dependence in the response of the near-surface region probed by indentation [2–6] also been intensively studied. Mann et al. [7] found that the mechanical deformation of an individual nano-scale contact is a highly dynamic phenomenon in which the loading rate affected the observed mechanical properties. Wang et al. [8] reported data on a $Ti_{0.5}Al_{0.5}N$ thin film suggesting that the onset of plasticity was sensitive to the loading rate. Chrobak et al. [6] suggested that the use of various loading rates can differentiate the dominant mechanism of incipient plasticity for GaAs, GaN, Si and Fe.

Pile-up is often observed for metallic materials during indentation [9] which can strongly increase the real contact area [10–13] leading to overestimation of the hardness and elastic modulus if the unloading curves are analysed assuming sinking-in behaviour. [14] A number of experimental [11,15–19] and modelling studies [20–26] have been carried out to investigate the main factors and the underlying mechanisms. It has been widely reported that the elastic and plastic properties affect the extent of pile-up. For a self-similar indenter, such as a Berkovich, pile-up depends on the ratio of *E*/*H* (related to elastic strain at yield) and the work hardening ability (related to plastic properties). [1,17,19,27,28] Based on finite element studies, Bolshakov and Pharr proposed [29] that an experimental parameter, the ratio of the final indentation depth to the maximum indentation depth (h_f/h_{max}), could also be used to express susceptibility to pile-up. It was suggested that in work-hardened samples $h_f/h_{max} \geq 0.7$ results in pile-up and $h_f/h_{max} < 0.7$ yields sink-in behaviour.

For a material with a given *E*/*H*, work hardening plays the dominant role. Pile-up is thus influenced by the dislocation density and distribution as work hardening is related to dislocation–dislocation (D–D) interactions. Gale et al. [30] investigated the effects of work hardening on the pile-up of a copper alloy. It was reported that more severely work-hardened regions showed higher pile-up height than in less work-hardened regions and thus proposed that higher resistance to dislocation motion is the fundamental mechanism driving pile-up. As dislocation movement is a kinetic process, the stress required for deformation depends on temperature and strain rate. [31–33] Formation and development of pile-up thus depend on the deformation rate and time. Charitidis et al. [23] noted that pile-up in Al alloys was slightly higher at the higher loading rate of 2.5 mN/s, and the pile-up was developed slightly during indentation creep. Rar et al. [34] observed that the Al produced a higher value of the pile-up/sink-in parameter when allowed to creep for a long time. Soare et al. [18] also

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reported that the pile-up of Al increased with time under load, but after unloading the differences are minimised due to the effects of elastic recovery. Finite element modelling was used by Taljat et al. [17] to model a wide range of materials with different elastic moduli, yield stresses, strain-hardening exponents, and friction coefficients. It was found that after initially sinking-in at small depths of penetration, there is a transition to pile-up in many materials that evolves and increases gradually as the indenter is driven into the material. Even when deformation enters the fully developed plastic stage, the pile-up geometry continues to change.

Although there have been previous reports of rate-dependence in the formation and development of pile-up, to date there have been few studies that have focused on the effects of loading rate, with the majority of these probing a small range of loading rate. Furthermore, the analysis of pile-ups was usually carried out under a constant stress field. As the loading rate affects the strain rate, stress distribution and gradient around indent, this could cause significant changes in the dislocations, mechanical properties and pile-up morphologies. Several researchers emphasized the importance of the stress gradient during nanoindentation and some new insights have been discussed in [35]. For example, strain gradient plasticity theory was employed to study the temperature and strain indentation size effect. [36] Voyiadjis et al. [37,38] found that the length parameter for plastic deformation depends on not only the temperature but also the strain rate. It is of fundamental scientific importance to improve our understanding of the effects of loading rate on dislocation-driven plasticity and pile-up phenomena under the stress and strain gradients. Moreover, the technologically important rate sensitivities of parameters determined from nanoindentation are studied over a wide range of loading rates.

Therefore, nanoindentation on a pure copper sample has been carried out to investigate the effects of loading rate on the formation and development of pile-up and the mechanical properties determined from the analysis of unloading curves. Utilizing the indentation curves, mechanical properties, activation volume and strain gradient plasticity theory, the underlying dislocation-driven mechanisms are discussed.

2. Materials and methods

The as-rolled copper sample (purity ≥ 99.6% and size 10 mm × 10 mm × 3 mm) was annealed in argon atmosphere at 700 °C for 1 h to minimize the original dislocation density, and then gently ground and polished. The surface roughness, R_a is about 9.6 nm. The grain size of the annealed copper was about 70 µm. The surface consists of mainly of (111) orientation, with smaller fractions of (200) and (220).

The nanoindentation tests were performed using a NanoTest Vantage (Micro Materials Ltd) with a Berkovich diamond indenter. The load was increased at different rates (0.05, 0.5, 5 and 50 mN/s) to the peak load of 10 mN. The load was held at the peak value for 100 s before unloading at 1 mN/s. The instrument has the excellent thermal stability required to perform tests over the x1000 difference in loading rate in conjunction with a hold of sufficient duration at peak load. The unloading curves were analyzed by the Oliver and Pharr method, power-law fitting from 100–20% of the unloading curve. [14] Each test was repeated 10 times. Additional tests at the loading rates of 0.05 and 50 mN/s were carried out with and without dwell periods at peak load to assess the influence of loading history on the evolution of pile-up during the dwell period. The surface morphology around the indents was recorded using an integrated piezo-driven SPM (Scanning Probe Microscopy) nanopositioning stage after indentation. Each condition was repeated three times.

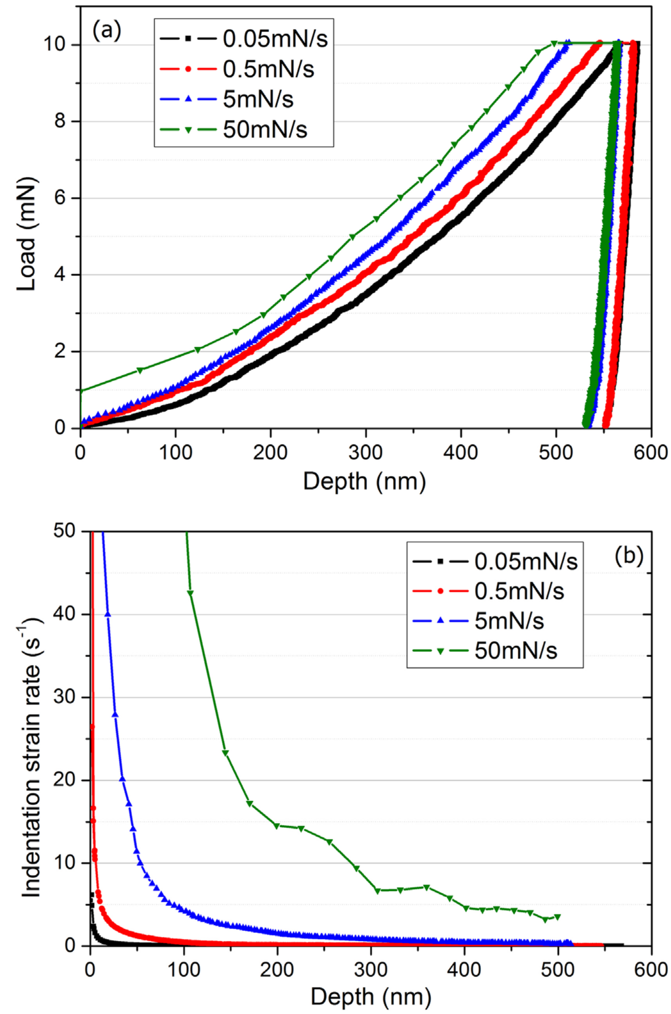

Fig. 1. (a) Load–displacement curves at different loading rates, (b) the indentation strain rate.

3. Results

Fig. 1(a) illustrates typical load-displacement curves at different loading rates. The indentation curves were clearly affected by the loading rate. With an increase of the loading rate, the depth at the end of loading (h_L) in Table 1 was decreased. For a pyramidal indenter, the indentation strain rate (*SR*) during loading can be calculated [39] as

$$SR = {}^{1/h}\!/_{dh/dt} \quad (1)$$

where h is the depth and t is the time. Generally, the *SR* in Fig. 1b exhibited a high value at the initial contact and then dropped rapidly, until it finally stabilized, similar to the Ref. [40] The different loading rates can significantly change the levels of the *SR*, especially at the initial contact. For example, the *SR* at 50 mN/s was very high, over 40 s^{-1} at the depth of ~100 nm, decreasing to ~10 s^{-1} at ~250 nm, finally to ~3 s^{-1} at 500 nm. For 0.05 mN/s, the initial *SR* was only ~0.1 s^{-1} when the depth is 8 nm, finally stabilized ~0.002 s^{-1}.

In all cases the ratio of the final depth to the maximum depth was around 0.95, suggesting that pile-up could be significant. The h_L(= depth at the end of the loading period), creep depth h_d(= increase in depth during the 100 s hold at peak load), and the derived hardness H, elastic modulus E are summarised in Table 1.

Fig. 2 shows typical depth vs. hold time curves after loading at