

## **Length scale and strain rate dependent shear banding deformation in nanoscale Cu/W multilayers**

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### **Abstract**

Recently, nanoscale metallic multilayers have drawn worldwide attention due to their superior mechanical properties. Nanoscale metallic multilayers exhibited high yield strengths, which can approach 1/2–1/3 of the theoretical strength at room temperature. However, deformation instabilities in the form of shear bands are prone to occur as the layer thicknesses enter nanoscale. Therefore, understanding the underlying mechanism of the shear instabilities in nanoscale multilayers are in both scientific and engineering interests.

In present study, a series of nanoscale Cu/W multilayers with different bi-layer thicknesses were deposited by d.c. magnetron sputtering. Shear banding deformation was characterized by using the depth-sensing nanoindentation testing at a range of applied strain rate at room temperature. By evaluating the morphology of the residual indentation at various applied strain rate, contrary trends of the dependence of the shear banding behavior on stress were observed. A physical model was proposed and the possible dominant mechanisms were suggested and extendedly discussed.

### **1. Introduction**

In recent years, nanoscale metallic multilayers have drawn intense attention due to their unique mechanical properties, as their bi-layer thickness reduced from micro-scale to the nanometer scale (Anderson 2010; Mara 2010; Zhang 2011; Zhu 2011). In general, nanoscale metallic multilayers exhibited high yield strengths (approach 1/2–1/3 of the theoretical strength) and low ductility (less than 5%). Especially, deformation instabilities, e.g., shear banding deformation, were usually observed in metallic nano-multilayers (Li 2007; Wang 2011). Such highly localized plastic deformation strictly limited ductility and hence potential applications. Therefore, understanding the underlying mechanism of shear banding deformation in nano-multilayers is crucial for both scientific and engineering aspects.

Nanoindentation testing has been widely used to investigate the plastic instability of

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metallic multilayers. For larger bi-layer thickness, the morphology of the residual indentation was smooth and relatively uniform, while circular shaped shear bands (SBs) were formed as bi-layer thickness reduced below certain values. Previously, numerous studies have concerned on shear banding deformation in metallic multilayers, and several deformation mechanisms have been proposed, e.g., buckling assisted grain boundary sliding in nano-multilayers (Li 2010; Zhang 2006) and dislocation plasticity dominated instability in sub-micron scale multilayers (Li 2010). As the plastic deformation of both nanocrystalline metals and nano-multilayers exhibited very high strain rate sensitivity, understanding whether applied strain rate could affect the shear banding deformation in nano-multilayers is an interesting issue that needs to be addressed. Despite that, only a few studies, if any, have concerned on applied strain rate dependent shear banding deformation in metallic nano-multilayers.

In the present study, by characterizing residual indentation in Cu-W nano-multilayers, we report the applied strain rate dependent shear banding deformation behavior. In addition, related length scale effects and the dominant mechanism of the shear banding deformation were proposed and discussed.

## 2. Experiments

Cu/W multilayers with different bi-layer thickness  $\lambda$  of 3, 8, 14 and 24 nm were deposited by the magnetron sputtering on silicon substrates at room temperature. The base pressure of the chamber was less than  $5 \times 10^{-7}$  mbar. The deposition rates were 0.2 and 0.15 nm/s for Cu and W, respectively. The total layer thickness of all the multilayers was 600 nm. The first layer connected to the Si substrate was W, and the top layer was Cu. The microstructures of the Cu/W multilayers were examined by transmission electron microscopy (TEM, JEM 2100F operated at 200 kV accelerating voltages).

Nanoindentation tests were performed on a Dynamic Contact Module (DCM) device equipped with the Nanoindenter XP system (MTS, Inc), under continuous stiffness measurement (CSM) mode at room temperature. Upon calibration on standard fused silicon, the tip of the Berkovich diamond indenter was estimated to have a radius of  $\sim 50$  nm. All the indentation tests were performed by depth control mode. During testing, the indentation load was ramped at a constant loading strain rate to the prescribed depth limit. Then the indenter was unloaded and eventually withdrawn from the sample surface to terminate the test. All the morphologies of residual indentation were examined by scanning electron microscopy (SEM).

## 3. Results

The bright field cross-sectional TEM images shown in Fig. 1 revealed the microstructure of the selected nano-multilayers, in which the dark and bright layers are W and Cu, respectively. As shown in the figures, the regular alignment of columnar grains was observed in all of the Cu/W nano-multilayers. The interfacial orientation relationship was  $\{111\}$  Cu //  $\{110\}$  W, which was determined through the cross-sectional TEM observation with the selected area diffraction at the interface.

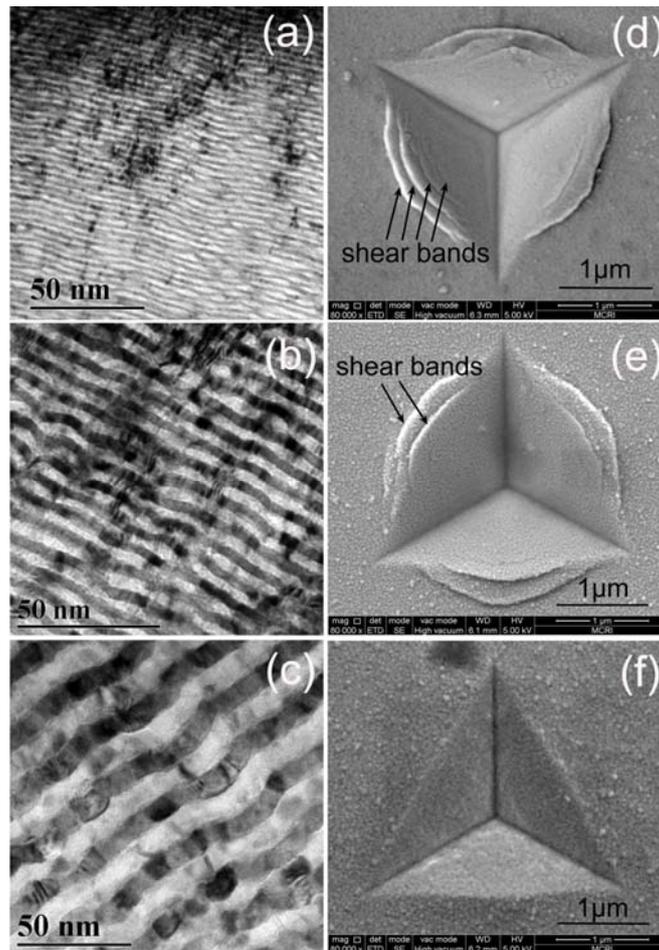


Fig. 1 Bright-field TEM cross-sectional images of nanoscale Cu/W multilayers with bi-layer thickness of (a) 3nm, (b) 8nm and (c) 24nm; and the corresponding SEM images of residual indentation morphology for the Cu/W multilayers are shown in (d), (e) and (f), respectively. Localized shear banding behavior observed in both 8nm and 3nm layered multilayers was indicated by arrows in (d) and (e), whereas no shear banding deformation was observed in 24nm layered multilayers as shown in (f)

Typical SEM images of residual indentation for selected multilayers with various bi-layer thicknesses were shown in Fig.2. For multilayers with  $\lambda > 8$ , no apparent pile-ups and shear band (SB) were observed around the residual indentation. When  $\lambda$  is reduced to 8nm or below, SBs appeared which could be identified by the circular deformation morphology around the indentation, as shown in Figs. 1(d) and (e). The observation that SB only occurred while  $\lambda$  was smaller than a certain value indicated that the shear banding deformation of the Cu/W multilayers exhibits apparent length scale dependence.

For the multilayers with bi-layer thickness of 8nm, specifically, Fig. 2 showed the indentation morphologies of the 8nm layered Cu/W multilayers deformed at various strain rate ranging from 0.2 to 0.001s<sup>-1</sup>. With decreasing strain rate, the number of shear bands appeared during nanoindentation testing decreases.

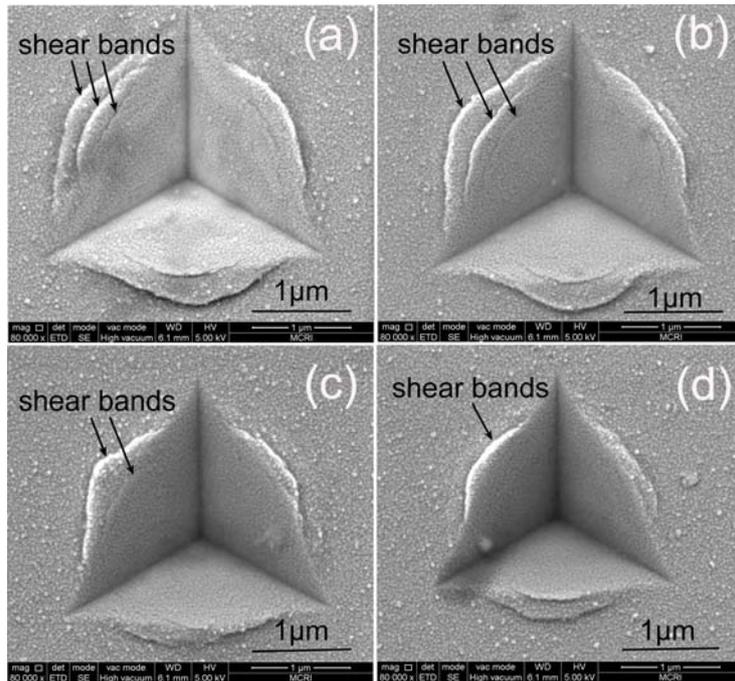


Fig. 2 SEM images of shear banding deformation at strain rate of (a) 0.2s<sup>-1</sup>, (b) 0.1s<sup>-1</sup>, (c) 0.01s<sup>-1</sup> and 0.001s<sup>-1</sup>, around residual indentation observed in 8 nm layered multilayers

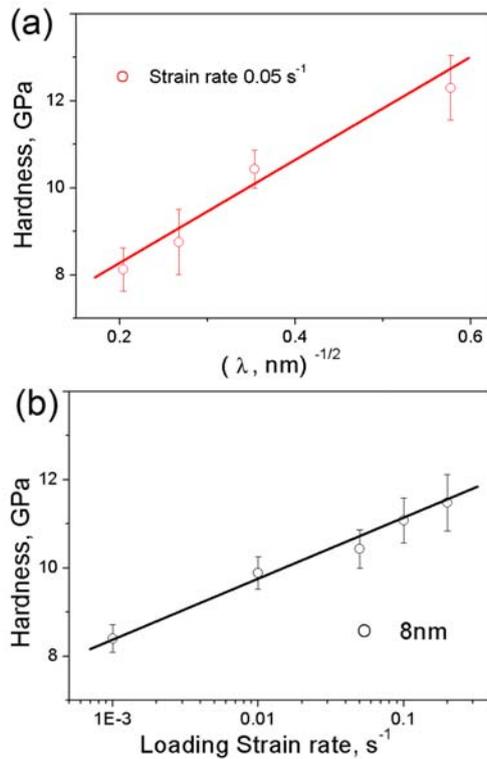


Fig. 3 Hardness as a function of (a) bi-layer thickness and (b) applied strain rate for the 8 nm layered Cu/W multilayers

The hardness of the nano-multilayers at various applied strain rates was presented in Fig. 3(a). Specifically, the hardness increases with decreasing bi-layer thickness; and also the hardness varies linearly with  $1/\sqrt{\lambda}$ , indicating a classical Hall-Petch behavior. For the 8nm layered nano-multilayers, Fig. 3(b) indicated that the hardness increases with increasing applied strain rate, showing a positive strain rate sensitivity.

#### 4. Discussion

As bi-layer thickness reduced into tens or a few nanometers, numerous studies indicated that shear banding deformation would occur in metallic multilayers, e.g., Cu/Ta (Wang 2011), Cu/Au,(Li 2009; Zhang 2006) Cu/Cr (Li 2009) and Cu/Nb (Bhattacharyya 2009; Zhu 2011) multilayers. Moreover, it has been indicated that shear bands were easily formed in multilayers with smaller bi-layer thickness. For Cu/Nb multilayers as an example, shear bands formed easily in 5nm layered Cu/Nb multilayers when compared with 20nm layered one (Bhattacharyya 2009). It was proposed that confined layer slip (CLS) model dominates the plastic deformation of the 20 nm layered multilayers (Misra 2005; Misra 2001), inducing relatively homogeneous plastic deformation because of confining single dislocation loops within individual layers. Those observations were consistent with the present experimental result that no shear band appeared in the multilayers with larger bi-layer thickness as shown in Fig. 1(f). While bi-layer thickness reduced to below a certain value, i.e., 8nm in present study, shear banding deformation occurred as numerous shear bands were identified around the residual indentation.

In general, for multilayers consisted of two constitute layers with different strengths, plastic deformation occurs in the soft layer first whilst the stiff one undergoes elastic deformation simultaneously, because the stress of plastic deformation in W layer is much larger. Soft layer was preferentially thinned and squeezed out, prior to hard layer, as observed in other multilayers (Liu 2010; Zhang 2012). In this length scale deformation of Cu layer can be attributed to the slip activity of dislocation in the isolated crystalline layer with low shear strength that are susceptible to sliding (Liu 2010; Zhang 2012).

Therefore, numerous studies (Li 2010; Wang 2011; Wen 2009; Xie 2011) indicated that the bi-layer thickness dependent shear banding deformation may be controlled by grain boundary related mechanisms, because decreasing of layer thickness indeed leads to decreasing of mobile dislocation. In this length scale, homogenous deformation is very limit because of lacking of effective slip system and active dislocation. Large plastic deformation only can be carried by grain boundary other than dislocation. In a nanocrystalline multilayer with random grains, large incompatibility and stress concentration could build up inside or near the grain boundary or interface during deformation. Deformation process will release this stress concentration accompanied by piling up or grain rotation (Li 2010; Xie 2011). At this stage, the pile up forms and expands as the indenter penetrates deeper. As the pile up increases, the shear stress concentration generates in the W layers. Simultaneously, grain boundary was rotated to direction which is benefited to shear deformation and eventually shear band forms in the region where the maximum shear stress exceeds a critical value that the W layers

cannot tolerate. Above all, shear banding behavior in nano-multilayers was dominated by both grain boundary sliding and relaxations of stress concentration built between the two constitute layers.

Based on aforementioned mechanisms, the strain rate dependent shear banding deformation observed in Fig. 2 could be interpreted as following. At higher strain rates, internal stress generated under indenter is relatively large, i.e., higher strain rates results higher hardness as shown in Fig. 3(b). Noticing that the multilayers with smaller bi-layer thickness possess higher hardness as indicated in Fig. 3(a), the higher internal stress generated either by reducing bi-layer thickness or higher applied strain rate may play a crucial role in forming shear bands in the Cu/W nano-multilayers.

## 5. Conclusions

In this study, a series of Cu/W multilayers with different bi-layer thicknesses were prepared by magnetron sputtering. The length scale and strain rate dependent shear banding behavior in Cu/W multilayers was investigated using the depth-sensing nanoindentation method at room temperature. By evaluating the morphology of the residual indentation, the microstructural length scale dependent shear banding behavior could be divided into two regimes. For larger bi-layer thickness, dislocation-mediated mechanism dominates the plastic deformation of the nano-multilayers, and no shear bands were observed. While bi-layer thickness reduced below a certain value, ~ 8 nm in present study, grain boundary sliding will take over and become dominant plastic deformation mechanism; numerous shear bands appeared. For all the nano-multilayers deformed at various applied strain rates, the stress generated during indenter penetration play a crucial role in forming shear bands in the Cu/W nano-multilayers.

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