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Role of interfaces in shock-induced plasticity in Cu/Nb nanolaminates

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Abstract

We investigate deformation of pure Cu, pure Nb, and 30 nm Cu/30 nm Nb nanolaminates induced by high strain rate shock loading. Abundant dislocation activities are observed in shocked pure Cu and Nb. In addition, a few deformation twins are found in the shocked pure Cu. In contrast, in shocked Cu/Nb nanolaminates, abundant deformation twins are found in the Cu layers, but only dislocations, in the Nb layers. High resolution transmission electron microscopy reveals that the deformation twins in the Cu layers preferentially nucleate from the Cu(112)//Nb(112) interface habit planes rather than the predominant Cu(111)//Nb(110) interface planes. Our comparative study on the shock-induced plastic deformation of the pure metals (Cu and Nb) and the Cu/Nb nanolaminates underscores the critical role of heterogeneous phase interfaces in the dynamic deformation of multilayer materials.

Keywords: Cu/Nb nanolaminate, shock loading, interfaces, deformation twinning
1. Introduction

In recent years, the multilayer structures have been the subject of many scientific investigations due to their unusual properties, such as ultra-high strength, radiation damage tolerance, high He solubility and mobility, and high thermal stability [1-9]. The interest in multilayer plasticity resides both in the fine scale structures, which demonstrate strength levels much higher than predicted by the rule-of mixtures[9-11], and in more macroscopic scale where important changes in the evolution of the deformation texture occur[12,13]. It has been confirmed by both experiments and molecular dynamics simulations that the interfaces play a critical role in controlling the process of microscopic scale plasticity (dislocation and twinning) and thereafter affect the macroscopic mechanical behavior (strength, texture, hardness, etc.) [1, 6-9, 12-15].

For possible broad applications of the multilayer materials, their performance, plastic deformation mechanism and the stability of the heterogeneous phase interfaces under extreme loading conditions, especially at high strain rate loading (e.g., shock wave loading), need to be considered. For example, Cu/Nb nanolaminates have been investigated widely in the past decade, and their microstructure, general mechanical behavior, radiation properties, etc., are well known [5-10]. Those previous studies provide sufficient background information for further exploring their deformation under high strain rate loading. However, there are few high strain rate experimental studies on nanocrystalline metals in general [16] and on nanolaminates in particular, although shock-induced deformation in solids at micron or submicron scales has been investigated extensively [17]. Here we take pure Cu, pure Nb and Cu/Nb nanolaminates as model materials to explore the role of heterogeneous phase interfaces on plastic deformation of the multilayer structures under high strain rate loading, using gas gun impact and electron microscopy. Drastically different deformation behaviors between the Cu/Nb nanolaminate and its single-component counterparts points to the critical role of the heterogeneous phase interfaces in plastic deformation of multilayered structure at nanoscales and high strain rate loading.

2. Experimental design and procedures
The Cu/Nb nanolaminates were synthesized via direct current magnetron sputtering at room temperature on Si substrates. Sputtering deposition was performed at a rate of ≈0.6 nm/s for Cu and 0.7 nm/s for Nb with a 4 mTorr Argon partial pressure; the power was 100 W and 350 W, respectively, for Cu and Nb deposition. The diameter of the as-deposited nanolaminate film is 75 mm, and the thickness is the same for the Cu and Nb layers (30 nm). The total thickness of the film is around 60 μm. The nanolaminate film was removed from the substrate, yielding a freestanding Cu/Nb multilayer foil with no evidence of damage introduced by the removal process. The freestanding foil was then punched into disc samples (4 mm in diameter) for shock experiments. In order to reveal the possible anisotropic shock deformation behavior of the Cu/Nb nanolaminates, we explored two shock loading directions, parallel and normal to the Cu/Nb interfaces. High purity Cu and Nb were commercially purchased and then annealed before shock experiments. The dimensions of the Cu and Nb samples used for shock loading are 1 mm thick and 4 mm in diameter.

Planar shock wave loading experiments were conducted with a tabletop gas gun system, which utilizes the rapid expansion of compressed gas (He) to launch a flyer plate for its impact on a target assembly (Fig. 1(a); the gun labeled with an arrow was used in this study). The flyer plates were made of oxygen-free high-conductivity (OFHC) Cu, approximately 0.4-0.7 mm thick and 8 mm in diameter, attached to a polycarbonate sabot (Fig. 1(b)). The flyer plate assembly was accelerated in a smoothbore barrel by the expanding He gas upon the abrupt opening of a solenoid valve. The impact velocity was measured with two 670 nm diode lasers and corresponding Si photodiodes, placed along the barrel axis between the target assembly and the muzzle, ranging from 100 m/s to 400 m/s. The peak shock stress was calculated from the impact velocity and known shock equations of state (Hugoniots) of the flyer and target materials [17]. Both radial and longitudinal momentum traps were used to minimize the radial and longitudinal release effects on the target, as displayed in Fig. 1(c). The target assembly slid into a hollow steel cylinder after impact and was soft-recovered for metallurgical examinations (Fig. 1(d)). Details for such shock-recovery experiments can be found elsewhere [17,18].

Figure 1
The Cu/Nb nanolaminate disc was sandwiched between two annealed OFHC Cu cover discs (0.5 mm and 1 mm thick), and then pressed into a radial momentum trap fabricated from phosphor Cu, which is much stronger than OFHC Cu. Longitudinal momentum traps (thin Cu discs 0.1 mm thick) were placed behind the thicker cover disc. The shock direction is normal or parallel to the Cu/Nb interface for the normal (Fig. 2(a)) and parallel (Fig. 2(b)) loading, respectively. The peak shock pressure is ~6 GPa, the shock duration is ~0.25 μs, and the shock loading strain rate is ~10^6-10^7 s^-1 [see Ref. 19 for details on loading strain rate]. The shocked samples and Cu cover discs retained their shape after soft recovery, and the samples were retrieved for microstructural characterization. Shock loading of pure Cu and Nb were conducted in a similar way.

3. Results and discussion

3.1 Shock behavior of pure Cu and Nb

Figure 3 shows the microstructures of the as-annealed (preshock) and shocked pure Cu. The as-annealed pure Cu has a grain size of ~50 μm to 100 μm. Annealing twins were found in some grains with the width ranging from several μm to more than ten μm (Fig. 3(a)). Fig. 3(b) shows the EBSD images of the shock loaded pure Cu. Some subgrain structures formed during shock loading as indicated by the small contrast variations within a grain. However, no abundant
deformation twins were found in this sample after dynamic loading to ~6 GPa. DeAngelis et al. [20] investigated the dynamically loaded pure Cu and reported; abundant deformation twins only when the shock pressure was higher than 14 GPa, consistent with present investigation. However, due to local stress concentration, some of deformation twins might nucleate even at lower shock pressure, as revealed by the TEM observation shown in Fig. 3(c). In addition, profuse dislocation actives were revealed in the shocked pure Cu (Fig. 3(d)). Fig. 4 shows the microstructures of as-annealed and shocked pure Nb. The as-annealed pure Nb has grain size of several hundred μm (Fig. 4(a)). Fig. 4(b) displays a typical bright field TEM image of the shocked pure Nb, with the electron beam close to [111]. Profuse dislocation activities were found and no deformation twins nucleated in the present sample, similar to a previous investigation [21].

Figure 3 and Figure 4

3.2 As grown Cu/Nb nanolamine microstructures

Figure 5 shows typical cross-sectional views of the microstructure of the as-sputtered Cu/Nb nanolaminates. As seen from the bright field TEM image (Fig. 5(a)), the Cu and Nb layer thicknesses are around 30 nm. The Cu/Nb layer interfaces are not exactly flat and show a wavy structure, similar to previous observations [6,10]. The corresponding selected area diffraction (SAD) pattern (inset to Fig. 5(a)) shows Bragg peaks corresponding to {110} bcc Nb and {111} fcc Cu, indicating the fiber texture along the normal of the multilayer. Fig. 5(b) displays a bright field TEM image of the as-sputtered Cu/Nb nanolaminates, with some growth twins in the Cu layers. The HRTEM images of the growth twins (Fig. 5(c)) suggest that those growth twins are with (111) twinning plane parallel to the Cu/Nb interface plane; similar results were reported for this nanolaminate system [6]. Overall, the growth twin density is low. A typical low magnification HRTEM image (Fig. 5(d)) shows the grain structures within the layers in the Cu/Nb nanolaminate. In each Cu or Nb layer, there is only a single grain across the layer thickness, while many grains with similar size (~30nm) but different orientations are observed in the direction parallel to the interface. Based on the atomic arrangement, the orientation relationship were identified and marked in Fig. 5(d). We examined different regions of the
sample, and found that the Kurdjumov-Sachs (K-S) orientation relationship ({110}Nb/{111}Cu and <111>Nb/<110>Cu) is the predominant one, while the Nishiyama-Wasserman (N-W) orientation relationship ({110}Nb/{111}Cu and <100>Nb/<110>Cu) is seen in dispersed locations (Fig. 5(d)). Figs. 5(e) and 5(f) are examples of the K-S and N-W orientation relationships. The interface planes in the present Cu/Nb nanolaminates are not all the ideal {110}Nb/{111}Cu type [6,22] due to the wavy feature of the layers, and can also be {112}Nb/{112}Cu or other types (Fig. 5(e) and 5(f)).

3.3 Shocked Cu/Nb nanolaminates

Typical microstructure of the shock-recovered Cu/Nb nanolaminates is displayed in Figs. 6 and 7, for normal and parallel loading, respectively. Abundant deformation twins are found in the Cu layers for both loading directions (Figs. 6(a) and (b) for the normal loading, and Figs. 7(a) and (b) for the parallel loading). This observation is in sharp contrast with the microstructures formed in shocked pure Cu at the same condition (Fig. 3), which indicates the critical role of interfaces in the plastic deformation mechanisms. Most of the deformation twins in the Cu layers originate from one side of the Cu/Nb interface and terminate at the opposite side (e.g., twins labeled by 1 in Figs. 6(a) and 7(a)), while some are formed between the interface and a Cu grain boundary (e.g., twins labeled by 2 in Figs. 6(a) and 7(a)). The SAD patterns (insets to Fig. 6(a) and Fig. 7(a)) reveal that the major orientation relationship between the Cu layer and Nb layer still remains as K-S, with {110}Nb/{111}Cu and <111>Nb/<110>Cu. This is further confirmed by HRTEM observation (Figs. 6(c) and 6(d), and Figs. 7(c) and 7(d)). The deformation twinning in Cu layers induces negligible changes to the orientation relationship of the Cu/Nb nanolaminates.
Compared to the microstructure in the as-sputtered Cu/Nb nanolaminates, shock compression induces obvious plastic deformation in the Cu layers and the density of deformation twins is high, while there is no clear evidence of deformation twinning in the Nb layer. To characterize the shock effect on the plastic deformation behavior of the Nb layers, we measured the full dislocation density inside the grains (not including interface dislocations) based on HRTEM images (Fig. 8). For each loading condition, we counted dislocations in at least five Nb grains for sufficient statistics. The measured dislocation density is on the order of magnitude of \(10^{16}/\text{m}^2\), higher than those in severely deformed pure metals [23], but it is normal for multilayer materials, as reported by Li et al. [24]. The dislocation density in the Nb layers increases after shock deformation, from \(\sim 2.25 \times 10^{16}/\text{m}^2\) to \(5 \times 10^{16}/\text{m}^2\). The dislocation density in the normal shock loading is slightly higher than in the parallel loading (Fig. 8); however, the difference becomes insignificant if we consider the statistical errors. It is indicated that the anisotropy in the shock deformation behavior is not pronounced in the present Cu/Nb nanolaminates. The Cu/Nb nanolaminate orientation relationship and structures remains stable even after shock loading to 6 GPa.

**Figure 8**

3.4 Deformation twinning in Cu/Nb nanolaminate

In general, deformation twinning is relatively difficult in coarse grained Cu due to the availability of multiple slip systems that can be easily activated [25,26]. In contrast, deformation twinning has been widely observed in nano-grained or ultrafine-grained Cu [27,28]. However, for the Cu/Nb nanolaminate, deformation twinning is not pronounced during monotonic loading even at applied stresses level approaching 2.5 GPa [9] or after rolling or nanoindentation [29, 30] although the grain sizes are in the range of nanoscales. Our findings of the deformation twins in the Cu layer in the Cu/Nb nanolaminate appear unusual compared to those experimental investigations.

**Figure 9**
Deformation twinning in coarse-grained Cu normally occur under extreme conditions, where the large stresses induce twins via pole mechanisms inside the grain [31-34]. In nanocrystalline Cu, atomistic simulations and experiments have shown that deformation twinning nucleates from grain boundaries through the glide of Shockley partial dislocations that lie on parallel, neighboring (111) glide planes [27,28,35-37]. However, the heterogeneous phase interface between the Cu and Nb layers likely play an important role in the deformation twin in the present investigation. In contrast, the growth twins in Cu nanograins are mostly originated and terminated at columnar grain boundaries, with the (111) plane parallel to the interface plane in the as-sputtered nanolaminates. However, Figs. 6 and 7 reveal that the deformation twins tend to be inclined at a characteristic an angle with the Cu/Nb interface planes, and its distribution is shown in Fig. 9 based on measurements from the TEM micrographs. For ~70% of the deformation twins, the angle is around 20° to 25°, indicating that deformation twinning is related to particular interface planes. Since the (111) plane is at angle of 19.5° with (112), the (112) interfaces might be the preferred sites for deformation twinning during straining.

**Figure 10**

Fig. 10 shows a representative HRTEM image of deformation twins nucleated at the heterogeneous phase interface, with the deformation twins inclined at an angle of ~20° with respect to the interface plane. The orientation relationship between the Cu and Nb layers in this region is K-S, while there are two types of interface planes, approximately (110)Nb//(111)Cu and (112)Nb//(112)Cu (Fig. 10(a)). Some of the twins in this region are originated from the interface plane, (112)Nb//(112)Cu, while the others, from the triple junction or from the grain boundaries in the Cu layer. Fig. 10(b) is the fast Fourier transformation (FFT) pattern of the region marked by the rectangle in Fig. 10(a), and Fig. 10(c) shows the corresponding indexed pattern. Based on the diffraction pattern at the interface, we can confirm that the interface planes are Cu(112)//Nb(112). However, part of the interface plane has been altered to become approximately Cu(110)//Nb(112) type (with few degrees discrepancy) after twinning. It appears that deformation twinning in the Cu layers of the Cu/Nb nanolaminate cannot change the orientation relationship between the Cu and Nb layers, while
it may adjust the interface plane type. Fig. 10(d) shows the enlarged HRTEM image of the region marked in Fig. 10(a). There are some twins and stacking faults at an angle with the interface; two distinct regions are marked as A and B (Fig. 10(d)). In order to identify the twin-interface structures, we performed inverse FFT in this region using the twin diffraction spots marked as circles in the Fig. 10(b), and the result is shown in Fig. 10(e). The two bright regions (A and B) in Fig. 10(e) correspond to the twinning regions (A and B, respectively) in Fig. 10(d). In Fig. 10(e), a series of interface defects are found between the front of deformation twins and the Nb layer (the exact nature of the defects is unclear). It is possible that such special interface defects at the Cu(112)//Nb(112) interfaces may play a key role in the nucleation of those deformation twins. Wang et al. [38] proposed that deformation twining in the Cu layer of the Cu/Ag multilayer can nucleate via the twin transmission from the Ag layer across the interface because twinning in Ag is much easier and nucleates first. However, a most plausible mechanism in our case appears to be that twinning nucleates from the Cu(112)//Nb(112) type interface plane defects. The present investigations point to the role of Cu(112)//Nb(112) type interface plane and related defects in deformation twinning, while the detailed mechanism is still unclear and will be investigated with molecular dynamic simulations.

4. Conclusion

The dynamic deformation of pure Cu, pure Nb, and Cu/Nb nanolaminates induced by high strain rate shock loading (\(\approx 10^6 - 10^7 \text{ /s}\)) was investigated. Abundant dislocation activities were observed in shocked pure Cu and Nb. In addition, a few deformation twins can be found in the shocked pure Cu. In contrast, abundant deformation twins are found in the Cu layers, and only dislocations, in Nb layers after shock loading of the Cu/Nb nanolaminates. The anisotropy in the shock-induced deformation is not pronounced in the Cu/Nb nanolaminates. Current shock loading does not change the orientation relationship between the Cu and Nb layers, while deformation twinning can alter the local interface plane type. The deformation twins in the Cu layer are mostly at an angle of “20° with the interface plane. High resolution TEM reveals that most of the deformation twins nucleate from the Cu(112)//Nb(112) interface planes, as opposed to the predominant Cu(111)//Nb(110) interface planes. The interface defects of
Cu(112)//Nb(112) interface planes may play an important role in the nucleation of deformation twins in the Cu layer within Cu/Nb nanolaminates. The comparison of the plastic deformation behaviors of the shocked pure metals (Cu and Nb) and the Cu/Nb nanolaminates demonstrates the critical role of interfaces on the deformation of multilayer structures under high strain rate loading.

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References


Figure Captions

Figure 1. Gas gun shock experiment setup: (a) the tabletop gas guns; (b) a sabot with a flyer plate attached; (c) the sample holder with the radial momentum trap installed; (d) the assembled recovery chamber.

Figure 2. Schematic sample configurations for normal (a) and parallel (b) shock loading. The Cu cover discs and nanolamintes are 4 mm in diameter. The thicknesses of the Cu cover discs are about 0.5 mm and 1 mm (a) and 1 mm and 1 mm (b), respectively.

Figure 3. EBSD images of annealed pure Cu before (a) and after (b) shock loading, and TEM images (c,d) of shocked Cu, showing shock induced the deformation twinning (c) (The electron beam close to [011]) and (d) abundant dislocation activities (The electron beam close to [001]).

Figure 4. (a) EBSD image of annealed pure Nb (unshocked). (b) TEM micrograph of shock-recovered Nb, displaying dense dislocations.

Figure 5. Typical TEM micrographs of the as-sputtered Cu/Nb multilayer’s (unshocked): (a) a low magnification bright field TEM image showing the wavy layer structure; (b) a local bright field TEM image demonstrating the growth twins; (c) the corresponding HRTEM image showing the growth twins in (b); (d) a low magnification HRTEM image displaying the grain structures and the orientation relationship between layers; (e) and (f) are HRTEM examples of different orientation relations between Cu and Nb layers.

Figure 6. Typical TEM and HRTEM images of the Cu/Nb nanolaminates recovered from normal loading: (a) and (b) are typical bright field TEM images, showing high density deformation twins in the Cu layers. (c) and (d) are the typical HRTEM images displaying the twin structure in the Cu layer. The inset to (c) is the FFT pattern for the deformation twins.

Figure 7. Typical TEM and HRTEM images of the Cu/Nb nanolaminates recovered from parallel loading: (a) and (b) are typical bright field TEM images, showing high density deformation twins
in the Cu layers; (c) and (d) are typical HRTEM images displaying the twin structures. The inset to (c) is the FFT pattern for the deformation twins.

Figure 8. Dislocation density in Nb layers for the as-sputtered and shock-recovered Cu/Nb nanolaminates. The inset micrograph shows the way to identify the edge dislocations using FFT image.

Figure 9. The statistics of the interacting angle between the deformation twins and the interface planes. The inset shows the way to measure the angle.

Figure 10. (a) A typical HRTEM image showing the deformation twins near the Cu/Nb interface; (b) the FFT pattern of the area marked by the white rectangle in (a); (c) the indexed pattern of (b); (d) the enlarged image of the area marked by the rectangle in (a); (e) the corresponding inverse FFT image showing the defects at the interface between the twin front and the Nb layer. The spots used for this image are marked by circles in the FFT pattern in (b).
Figure 1
Figure 2

Cu/Nb nanolaminates

(a) Normal loading

(b) Parallel loading
Figure 3
Figure 6
Figure 8

Dislocation density in Nb layers, \( \times 10^{16} \text{ m}^{-2} \)

- As deposited
- Parallel shock
- Normal shock