Microstructures and strengthening mechanisms of Cu/Ni/W nanolayered composites

J. W. Yan\textsuperscript{a}, G. P. Zhang\textsuperscript{a,*}, X. F. Zhu\textsuperscript{a}, H. S. Liu\textsuperscript{a}, C. Yan\textsuperscript{b}

\textsuperscript{a} Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, 72 Wenhua Road, Shenyang 110016, P. R. China

\textsuperscript{b} School of Engineering Systems, Faculty of Built Environment and Engineering
Queensland University of Technology, Brisbane, QLD 4001, Australia

\textsuperscript{*} Corresponding author: Tel. & Fax: +86-24-23971938, E-mail: gpzhang@imr.ac.cn
Abstract

Cu/Ni/W nanolayered composites with individual layer thickness ranging from 5 nm to 300 nm were prepared by a magnetron sputtering system. Microstructures and strength of the nanolayered composites were investigated by using nanoindentation method combined with theoretical analysis. Microstructure characterization reveals that the Cu/Ni/W composite consists of typical Cu/Ni coherent interface and Cu/W and Ni/W incoherent interfaces. Cu/Ni/W composites have an ultrahigh strength and a large strengthening ability compared with bi-constituent Cu-X (X=Ni, W, Au, Ag, Cr, Nb, etc.) nanolayered composites. Summarizing the present results and those reported in the literature, we systematically analyze the origin of the ultrahigh strength and its length scale dependence by taking into account of the constituent layer properties, layer scales and heterogeneous layer/layer interface characteristics including lattice and modulus mismatch as well as interface structure.

Keywords: metallic nanolayered composite, strength, length scale, interface, dislocation
1. Introduction

Layer-structured materials with multi-scale microstructures and heterogeneous interfaces, such as laminated steels [1], metal/glass multilayers [2, 3], metals with nanoscale twins [4], and even shells and bones in nature [5] etc., have exhibited some excellent mechanical properties, such as high strength and good toughness. Such a high performance may originate from the role of multi-scale microstructures and interfaces in controlling behaviors of microscopic defects (including dislocation nucleation, motion and its interaction with the interface, etc.) in the material. Obviously, a basic understanding of effects of length scales and heterogeneous interfaces in such layer-structured materials on dislocation activities would be beneficial to the design of high-performance structured materials.

Metallic nanolayered composites (NLCs) are composites stacked layer-by-layer by two or more different constituent materials with ultrafine layer thicknesses. Strength of such metallic NLCs is usually much higher than that expected by the rule of mixture, and exhibits a strong dependence on constituent layer thickness, i.e. the strength increases with decreasing the individual layer thickness [6-9]. Such the high strength can be attributed to constraints of fine length scale on dislocation motion [10, 11] and the resistance of layer/layer interfaces to dislocation transmission [6]. Firstly, as the individual layer thickness (\( \lambda \)) decreases from micrometer to nanometer scales, different strengthening mechanisms are operated and lead to the \( \lambda \) dependent strength. When \( \lambda \) is larger than about 50 nm, the strength of the NLCs increases linearly with \( \lambda^{-1/2} \) and is consistent with the Hall–Petch model. As \( \lambda \) decreases to tens of nanometers or less, too few dislocations reside in a dislocation pile-up and that cannot be treated as a continuum. Dislocation movement is confined to isolated layers. At the several
nanometer scale, a individual dislocation would glide to cross the interfaces, resulting in the saturation of the strength of metallic NLCs. Secondly, as a strong barrier for a dislocation to penetrate another layer, the interface structure is very important in determining mechanical properties of metallic NLCs, especially when the length scale is at a few nanometers, where the dislocation transmission through the interface might occur. An opaque interface in a layered composite where the two crystal structures are different, exhibits a higher resistance to slip transmission than a transparent interface, for which the slip planes and slip vectors are nearly continuous [12]. Concerning the strong confinement of the fine scale layer on dislocation motion and the barrier of the heterogeneous interface to the dislocation transmission, one would argue whether an introduction of more heterogeneous interfaces in a NLC would improve the strength of the material evidently.

Although extensive studies on strength of bi-constituent metallic NLCs (Cu-X, X:Ni, Au, Cr, W, Nb, etc.) have been conducted for past decades [6-8, 13-16], less research is carried out to evaluate strength and strengthening mechanism of tri-constituent metallic NLCs [15, 17, 18]. In this paper, we present systematic examination of microstructures and strength of Cu/Ni/W nanolayered composites with different individual layer thicknesses and both transparent and opaque interfaces. The ultrahigh strength and the large strengthening ability in the NLCs were found. The strengthening mechanisms of the metallic NLCs were analyzed.

2. Experimental

2.1 Material selection and preparation
In this study, Cu/Ni/W NLCs were deposited on a 525 μm-thick Si substrate under ultrahigh vacuum (base pressure <1×10^{-7} Torr, working pressure 0.4 Pa) using a DC magnetron sputtering system. The NLCs were deposited in the order that the W layer was always bonded to the substrate first, and then the Ni and Cu layers were deposited, respectively. All the NLCs have the same total thickness \( h_f \) of 900 nm, but the individual layer thickness \( \lambda \) is 5, 30, 60, 100 and 300 nm, respectively. The whole deposition process was performed at a speed of 0.3 nm s\(^{-1}\) for the NLCs with the substrate kept at room temperature (RT). Microstructures of the NLCs were characterized by SEM (LEO Supra 35), X-ray diffraction (XRD, \( \theta-2\theta \) scanning) and transmission electron microscopy (TEM) (Tecnai F20, FEI).

2.2 Nanoindentation testing

To examine strength of the NLCs, a nanoindenter XP (MTS Nano Innovation Center, Oak Ridge, TN) with a Berkovich tip (tip radius ~ 50 nm) was used to determine hardness \( H \) and modulus \( E \) of the NLCs under continuous stiffness measurement with a constant strain rate of 0.025 s\(^{-1}\) at RT. A total of 10 indents with spacing more than 10 times the indent size were made for each sample. The mean values of \( H \) and \( E \) of the NLCs with different \( \lambda \) were then obtained at an indentation depth of about 90 nm in order to eliminate substrate effects [19].

3. Results

3.1 Microstructure characterization
X-ray diffraction (XRD) θ-20 scans presented in Fig. 1(a) show that both the Cu and Ni layers in the Cu/Ni/W NLCs have strong \{111\} out-of-plane textures, which indicates that the interface relationship between Cu and Ni layers follows typical \{111\}_Cu//\{111\}_Ni. The W layer in the NLCs has two different structures, i.e. a stable body-centered-cube (BCC) α-phase and a metastable A15 crystal structure, the so-called β-phase. In the λ=5 nm NLC, there is only an α-phase structure, while in the λ=300 nm NLC there is almost a β-phase. The W layers in other NLCs consisted of both α- and β-phases. The diffraction peaks of the α- and β-phases located at around 2θ=40° are very close to each other and cannot be easily separated. However, the α-phase (112) peaks and the β-phase (002) peaks can be easily found. The ratio of the relative intensity of the α-phase (112) peak to the β-phase (002) peak (Fig. 1(b)) reveals that with increasing λ the crystalline structure of the W layer is gradually dominated by the β-phase. Such the α-β phase transformation is consistent with previous findings on the deposition of nanoscale W thin films [20]. It is suggested that the occurrence and the stabilization of the β-phase during deposition are related to the burial of impinging impurities (e.g. O or Ar atoms) in the interstitial sites of the cubic cell. Furthermore, XRD θ-20 scans show that there is an fcc{111}/bcc{110} relation for the stable α-phase, and fcc{111}/β-W{002} relation for the metastable β-W structure. The orientation relation is consistent with the selected area diffraction pattern (SADP) and high resolution atomic images obtained from TEM observations shown below.

On the other hand, a small shift of the diffraction peak, such as α(112)/β(002) and Cu(111) peaks, indicates that a residual stress may exist in the NLCs, which seems increased with decreasing λ. The residual stress in a thin film usually arises
from the island nucleation and growth, island coalescence, and postcoalescence film growth of Volmer–Weber thin films [21]. Compressive stresses are generated by surface-stress effects, while tensile stresses are created during island coalescence and grain growth. The shift of α(112)/β(002) and Cu(111) peaks towards large diffraction angles indicates that a compressive stress exits in the NLCs with λ in the range of 30 to 300 nm. The residual stress for λ = 5 nm NLC is estimated by measuring the lattice strain from HREM images and using a method for layered epitaxial materials adapted from [22]. The results show a comprehensive stress in Cu layer and tensile in Ni and W layers. The residual stress is about 0.7, 0.4 and 1.1GPa in the Cu, Ni and W layers, respectively.

Figure 2 presents cross-sectional TEM images of the Cu/Ni/W NLCs and the corresponding high resolution TEM (HRTEM) images of the interfaces. Firstly, a bright field TEM image in Fig. 2(a) shows the morphology of the λ=5 nm NLC. One can find that the nanoscale layers close to the substrate (the bottom of the image) are initially flat, and then gradually become wavy along the growth direction. The wavy morphology has a certain wavelength of 38.55±7.37 nm. The wavy morphology was also observed in other vapor-deposited metallic NLCs [23-25]. In contrast, no wavy morphologies are found in the λ=30 nm (Fig. 2(b)) and 100 nm (Fig. 2(c)) NLCs. It is suggested that the wavy morphology results from the release of the residual stress controlled by the deposition and diffusion process during non-equilibrium growth of NLCs [23-25]. Competitive mechanisms for interface roughening and smoothing, such as surface diffusion, thermal diffusion, Ehrlich–Schwoebel barrier [26, 27], deposition velocity and flux distribution all together may determine such the interface morphology.
Secondly, it is found that all the layers including Cu, Ni and W in the $\lambda=5$ nm NLC have a columnar-grain structure due to the in-plane grain size being larger than the layer thickness, as shown in Fig. 2(a). In contrast, the Cu layers in the NLCs except the $\lambda=5$ nm one consist of equiaxed grains (see Figs. 2(b) for $\lambda=30$ nm and 2(c) for $\lambda=100$ nm NLCs), and the Cu grain size scales with $\lambda$. A few deposition nanotwins were found in some Cu grains. The Ni and W layers in the NLCs have a columnar-grain structure, which is characterized as very uniform and thin grains with vertical grain boundaries, as indicated by arrows in Figs. 2(b) and 2(c). The sizes of Ni and W grains are approximately 30 nm, and almost independent of $\lambda$. The relation between in-plane grain size and layer thickness for all the NLCs are presented in Fig. 2(d). It can be seen that the aspect ratio ($\lambda/d$) for the Ni and W grains decreases from ~10 to ~1 with decreasing $\lambda$ from 300 nm to 30 nm, while the aspect ratio of Cu grains does not change with $\lambda$.

Figure 3(a)-(c) present HRTEM images of microstructures of Cu/Ni, Ni/W and Cu/W interfaces, respectively. The Cu/Ni interface with the relation of $\{111\}_{\text{Cu}}/\{111\}_{\text{Ni}}$ characterized by the corresponding SADP in the inset of Fig. 2(b) shows that the misorientation angle between the (111) slip planes in the Cu and Ni layers is about 15°, as shown in Fig. 3(a). The Cu and Ni constituents are miscible and the lattice mismatch is small (~2.5%). Misfit dislocations with $\frac{1}{2} <110>$ Burgers vector can be observed at the Cu/Ni interface for the $\lambda=30$ nm NLC. For Cu/W and Ni/W interfaces, HRTEM observations reveal that both interfaces exhibit a Kurdjumov–Sachs (K-S) orientation relationship: $\{111\}_{\text{fcc}}/\{110\}_{\text{bcc}}$, $<110>=\{111\}_{\text{bcc}}$, as shown in Figs. 3(b) and (c). The incoherent Cu/W and Ni/W
interfaces are sharp and without apparent interdiffusion. Misfit dislocations are observed at both interfaces through the thickness range shown in Figs. 3(b) and (c).

3.2 Hardness and modulus of Cu/Ni/W NLCs

Figure 4(a) presents the variation of hardness ($H$) and indentation modulus ($E$) of the NLCs as a function of $\lambda$. The values of $E$ for all the NLCs are around ~200 GPa, except that $E$ of the $\lambda=5$ nm multilayer is somewhat lower than that of the other NLCs with the larger $\lambda$. Usually, both stiffening and softening of elastic modulus of nanoscale multilayers have been reported [28-30]. The stiffening of elastic modulus of metallic multilayers is observed as $\lambda$ decreases, and is attributed to a strain-layered superlattice effect, where a dominant compression of the lattice stiffens the elastic constant in certain directions. The XRD scanning verified this mechanism in present NLCs with $\lambda$ in the range of 30 to 300 nm. As the layer thickness decreases to less than 10 nanometers, the softening of elastic modulus in W films was also observed in other studies [31], and is attributed to size effects on elastic properties due to the surface tension in the nanoscale range. For the present NLCs the softening in elastic modulus observed in the $\lambda = 5$ nm NLC can be related to the relatively large residual tensile stress in the Ni and W layers.

The variation of $H$ with $\lambda$ exhibits the similar scale dependence observed in a number of bi-constituent metallic multilayers [12], i.e. $H$ increases with the decrease in $\lambda$. For comparison, the values of $H$ of some Cu/X (Ni, W, Cr, Nb, etc.) NLCs reported in the literature [7, 28, 32-35] are plotted as a function of $\lambda^{\frac{1}{2}}$ in Fig. 4(b), from which several findings can be obtained. First, the present Cu/Ni/W NLCs exhibit an ultra-high strength/hardness compared with the bi-constituent metallic NLCs
reported in the literature. Second, the variation of \( H \) with \( \lambda \) does not follow the H-P relation \( (H = H_0 + k\lambda^{-1/2}) \) until \( \lambda > 30 \) nm. Third, it is worth noting that the H-P slope \( (k) \) of the Cu/Ni/W NLCs is the largest in all the NLCs through comparing the slope reported directly by the authors or fitted with the hardness data from previous publications. Since the slope \( k \) in the H-P relation reflects a strengthening ability associated with the interface or grain boundary [6, 33], the present results imply that the Cu/Ni/W NLCs have a stronger interface strengthening ability.

4. Discussion

The above results clearly reveal that the present Cu/Ni/W NLCs have both ultrahigh strength and large interface strengthening ability. In addition to the well-known constraints of geometrical scales on dislocation motion, the present findings may also be attributed to the following factors,

1) Existence of abundant heterogeneous interfaces that may act as strong barriers to dislocation motion. Different from the bi-constituent NLCs with one kind of layer/layer interface, the present NLCs have three kinds of layer/layer interfaces.

2) Elastic modulus and lattice parameter mismatches. The fact that the shear modulus of the W layer is about 3 times larger than that of the Cu layer results in a large image force on dislocations in the Cu layer [28];

3) Other factors, such as coherency stress between the interfaces, the slip plane discontinuity between the layers etc., would play important roles in strength of the NLCs[8, 9].
In what follows, we will discuss the effects of both length scales and interfaces in details.

4.1 Comparison of interface strengthening ability

The H-P relation demonstrated in many metals and alloys is generally explained by the dislocation pile-up mechanism [36, 37]. The H-P slope reflects the strengthening ability of an interface that can block the dislocation in the pile-ups. Misra et al. [6] evaluated the interface strengthening ability of the Cu/Nb NLCs based on the slope \( k \) in the H-P relation that,

\[
\tau_{HP} = \tau_0 + k \lambda^{3/2},
\]

where \( \tau_0 \) represents the lattice resistance to dislocation gliding, and is taken as average friction stress associated with the movement of individual dislocations in the pile-ups, and \( \tau^* \) is the barrier strength offered by an interface to dislocation transmission. The slope \( k \) can be expressed as [6, 9, 38],

\[
k = \left( \frac{\mu b \tau^*}{\pi (1 - \nu)} \right)^{1/2},
\]

where \( \mu \) is the shear modulus, \( \nu \) is Poisson ratio, 0.3 is used here, and \( b \) is the Burgers vector of the soft constituent. For bi-constituent metallic NLCs, such as Cu-X (X=Ag, Au, Ni, Nb, Cr, etc.), a relation between \( k \) and interface properties was evaluated recently based on the concept of lattice mismatch \( (\delta_L) \) between two constituent crystals. \( \delta_L \) is defined as a ratio \( (\delta_L = \Delta a / \bar{a}) \) of the lattice constant difference \( (\Delta a) \) between the two constituent crystals to their mean value \( (\bar{a}) \) without taking into account of the detailed relationship of crystallographic orientation at the interface [33].
In calculations, a curve of the yield strength versus \( \sqrt{\lambda} \) was plotted following the traditional H-P relationship to extract the value of \( k \) by a linear fitting. Following such a method, the values of \( k \) of some typical bi-constituent (Cu-X) multilayers and the present NLCs as a function of \( \delta_L \) are shown in Fig. 5. It is found that the value of \( k \) for the bi-constituent NLCs increases with increasing \( \delta_L \), but the value of \( k \) of the present Cu/Ni/W NLCs is too high to be rationalized by the model proposed by Li et al. [33] if \( \delta_L \) is considered only.

Owing to the geometrical scale constraints, great stress intensity in metallic laminates is needed for transmission of plastic flow across grain boundaries and interfaces. As a result, the \( k \) value of metallic NLCs is expected to be larger than that for their bulk counterparts. Clemens et al. [39] found that there is no clear relationship between \( k \) of NLCs and that of metals, and argued that \( k \) is a reflection of multilayer system rather than individual constituent. To examine the effects of the modulus mismatch on the \( k \) value of the NLCs and pure metals, we present the \( k \) values of some typical metallic NLCs, bulk FCC and BCC metals in Fig. 6. The relation of \( k = 0.18 \mu \sqrt{b} \) (black dash line in Fig. 6) is used to estimate the \( k \) value [40], where for bulk metals \( \mu \) is shear modulus, \( b \) is the Burgers vector. For the NLCs, \( \mu \) is taken as the shear modulus of elastically stiffer constituent, \( b \) is taken as the Burgers vector of elastically softer constituent. It can be seen that the \( k \) values of BCC metals are larger than that of FCC metals, while the \( k \) values of NLCs are in between. It seems that the \( k \) values for FCC metals and metallic NLCs can fit well with \( k = 0.18 \mu \sqrt{b} \), and the \( k \) values for BCC metals are much larger than the predicted. This implies that the strengthening ability of heterogeneous layer/layer interface is larger than that of grain boundaries in FCC metals but lower than that of grain boundaries in BCC metals. For
bulk materials, the $k$ value is described as the microstructural stress intensity for transmission of plastic flow across grain boundaries [17]. Thus, $k$ is a reflection of both intrinsic properties of materials and extrinsic testing conditions, and can be affected by several factors, such as deformation mechanism, temperature and material microstructures. It is well known that $k$ of a metal deformed at low temperature is much larger than that at room temperature, and $k$ of metals deformed by twinning is larger than that by dislocation slip [36, 41]. This can be understood by the fact that great stress intensity values would be generated due to limited slip or twinning systems being available to accommodate the local grain boundary strains [36]. In general, stress intensity for pure FCC metals determined by the H-P relation is relatively small and can be correlated with the occurrence of multiple slip systems. For the present NLCs, the elastic modulus mismatch among constituent layers may offer additional blocking effects on dislocations, thus the interfaces has a larger strengthening ability than grain boundaries.

4.2 Interface barrier strength and peak strength of metallic nanolayered composites

The fact that the strength of NLCs increases with decreasing individual layer thickness from submicron to nanometer scales and finally reaches the peak strength at several nanometers leads to a question that what determines the peak strength of the NLCs. It has been shown that the direct shear deformation crossing the interface would happen when the driving force to dislocation motion being larger than the interface barrier strength [42-44]. Thus, it is expected that the peak strength for the NLCs is corresponding to the interface barrier strength ($\tau^*$), which may be evaluated by the interface properties. The $k$ value related to $\tau^*$ in Eq. (2) can thereby be rewritten as
All the parameters in Eq. (3) have the same meaning as mentioned before. By taking $k$ from the experimental values, $\frac{\tau^*}{\mu}$ is determined and shown in Fig 7.

Next, we will compare the interface barrier strengths from the experiments with those from the theoretical calculations. Anderson et al. [9] has reviewed the factors contributing to $\tau^*$ (the barrier strength offered by an interface to dislocation transmission expressed in Eq. (2)), and pointed that the most important factor is the interface structure, which affects lattice mismatch, slip plane misorientation and dislocation-interface interaction. Thus, the magnitude of $\tau^*$ is generally dominated by lattice parameter mismatch, modulus mismatch (the Koehler barrier, which introduces a force between a dislocation and its image in the interface.), slip plane misorientation (slip discontinuity) and gamma surface (chemical) mismatch, which introduces a localized force on gliding dislocations due to core energy changes at or near the interfaces, etc. Rao and Hazzledine [45] qualitatively examined four contributors to $\tau^*$ for Cu/Ni NLCs by their atomistic simulations. They found that the Koehler barrier is about 0.01$\mu$ - 0.015$\mu$ and independent on the layer thickness and the dislocation character when the layer thickness is larger than the core width of a dislocation. The coherency stress (lattice parameter mismatch) made up to 0.02$\mu$ for the (111) interfaces at the coherent limit. The slip plane misorientation is the strongest barrier, and for 60° dislocations the blocking strength is estimated to be 0.03$\mu$ - 0.04$\mu$. As a result, the total barrier strength for the Cu/Ni interface is about 0.05$\mu$ - 0.075$\mu$. Following the formula given by Koehler [11], the barrier strength (the modulus mismatch barrier) for a full dislocation can be expressed by elastic modulus mismatch,
\[ \tau = \mu^* bR \sin \theta/(8M\pi). \]  

(4)

The blocking strength offered by misfit dislocations to glide dislocation is

\[ \tau = \alpha \mu (\frac{\delta a}{a} - \frac{b}{\lambda}), \text{ for } \lambda > \lambda_c \]  

(5)

where \( \alpha \) is Saada constant, taken as 0.5. \( \lambda_c \) is the critical thickness below which the interface loses coherency [45]. The critical thickness for breakdown of dislocation pile-up in the multilayers is about several tens of nanometers, while \( \lambda_c \) is at the nanometer scale. The experimentally-determined \( \tau^* \) (experimental values) [7, 16, 28, 32, 33, 35, 46, 47] and theoretical calculations (the modulus mismatch barrier, the lattice mismatch barrier and the sum of both of them) are compared in Fig. 7.

First, one can find that for most of the NLCs the contribution to the interface barrier strength by the lattice mismatch is larger than that by the modulus mismatch. The reason is that the image stress is relative small in the Hall-Petch regime comparing to the dislocation-dislocation interaction stress, and it only becomes significantly larger when the individual layer thickness decreases to several nanometers.

Second, for some NLCs in the left shadow region in Fig. 7 the experimental values for \( \tau^* \) is quite close to theoretical values if only considering the modulus mismatch and the lattice mismatch barriers. In this case, the elastic stiffer layer in the NLCs tends to have a lower stacking fault energy (SFE) (Cu/Ag, Cu/Au and Cu/330SS NLCs) or a lower elastic modulus mismatch (Cu/Nb, Cu/V NLCs) which makes the dislocation nucleation possible in the stiffer layer. The interfaces are semi-coherent considering the lattice parameter mismatch on the slip planes, which makes
the interfaces be potential sites for nucleation of dislocations in the stiffer layer. The multilayers will yield once these dislocation sources are activated by stress concentration built up by the dislocation piling-up in the elastic softer layer. It is worth noting that for Cu/W NLCs with a large modulus mismatch, the experimental value is also close to the theoretical, and that is believed to be a result of diffusion of Cu atoms at the Cu/W interface which was confirmed by TEM observation[28]. In general, for metallic NLCs with a lower stacking fault energy or lower elastic modulus in the elastic stiffer layer, the interface barrier strength is contributed mainly by both of the modulus mismatch and the lattice parameter mismatch.

Third, for other NLCs located at the middle and right shadow regions, the difference between the experimental value and the theoretical value is evidently large. This implies that in addition to the contribution by both of the modulus mismatch and the lattice mismatch barriers, other contributions, such as the slip plane misorientation, may play more important roles in dominating the interface barrier strength. Especially, the present Cu/Ni/W and the Cu/Cr (FCC/BCC) NLCs in the right shadow region tend to have such a larger difference between the experimental and the theoretical values than the Cu/Al and Cu/Ni (FCC/FCC) NLCs in the middle shadow region. In this case, the elastic stiffer layers seem to have a high stacking fault energy (Cu/Al and Cu/Ni NLCs) or large elastic modulus (Cu/Cr and Cu/Ni/W NLCs) which makes the dislocation nucleation difficult in the stiffer layer. Thus, the dislocations must be transferred from the elastic softer layer. The stresses for dislocation transmission through the interfaces depend on the slip-plane misorientation in adjacent layers. For FCC/FCC NLCs with a cube-on-cube orientation, the slip plane is nearly consistent in neighboring layers and the misorientation angle between (111) slip planes is very small[12]. While for FCC/BCC NLCs with K-S orientation relationship, the
misorientation angle between (111) slip planes in the FCC layer and (110) slip plane in the BCC layer is larger than 5° or 10°[13]. The misorientation angle for FCC/BCC NLCs is larger than that for FCC/FCC NLCs, thus the interface barrier strength $\tau^*$ of Cu/Cr NLCs is correspondingly larger.

5. Conclusions

(1) Cu/Ni/W NLCs deposited by the magnetron sputtering system contain two kinds of fcc/fcc and fcc/bcc interfaces. Only the $\alpha$-phase structure appeared in the W layers of the $\lambda=5$ nm NLCs, while for the $\lambda=300$ nm NLCs there are almost $\beta$-phase in the W layers.

(2) The Cu/Ni/W NLCs show the ultrahigh strength, which increases with decreasing the individual layer thickness. The strength can follow the H-P relation until $\lambda<30$ nm, and after that the deviation from the H-P relation occurs.

(3) The strengthening ability of the present NLCs is much larger than that of bi-constituent (Cu-X) NLCs reported in the literature, and is rationalized by the blocking effects offered by modulus mismatch, lattice parameter mismatch and slip plane misorientation.

(4) The interface barrier strength of the NLCs is determined experimentally. For the metallic NLCs with a lower stacking fault energy or lower elastic modulus in the elastic stiffer layer, the interface barrier strength is contributed mainly by both of the modulus mismatch and the lattice parameter mismatch, while for the NLCs with a larger stacking fault energy or larger elastic modulus in the elastic stiffer layer, other barriers, such as the slip plane misorientation, may become more dominant.
contributions to the interface barrier strength than that by the modulus mismatch and the lattice mismatch.

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References

Figure 1 (a) X-ray 0-20 scans showing the (111) texture in the Cu and Ni layers and the phase transformation of the W layer of the Cu/Ni/W nanolayered composites (NLCs) with decreasing individual layer thickness. (b) Variation of the relative intensity of $\alpha$-W (112) peak to $\beta$-W (002) peak with individual layer thickness.
Figure 2 TEM cross-sectional characterization of microstructures in the Cu/Ni/W NLCs with (a) $\lambda=5$ nm, (b) $\lambda=30$ nm and (c) $\lambda=100$ nm, (d) grain size vs. individual layer thickness for Cu, Ni and W layers in Cu/Ni/W NLCs. The grain size is determined based on TEM observations.
Figure 3 HRTEM images of the microstructures of (a) Cu/Ni, (b) Ni/W and (c) Cu/W interfaces in the Cu/Ni/W NLCs.
Figure 4 Nanoindentation tests of the Cu/Ni/W NLCs with different individual layer thicknesses ($\lambda$), (a) hardness (H) and indentation modulus (E) of the multilayers as a function of $\lambda$. (b) Linear fitting is used to calculate the slope ($k$) in Hall-Petch (H-P) relation $H = H_0 + k\lambda^{-\frac{1}{2}}$. Only the linear part of the data is used for the data fitting.
Figure 5 Hall-Petch slopes ($k$) vs. lattice parameter mismatch ($\delta_L$) for Cu-based metallic multilayers. The H-P slope for bi-constituent multilayers follows a linear relation with $\delta_L$, however, the Hall-Petch slope for Cu/Ni/W NLCs indicated by a horizontal red dash line is much higher than that of all bi-constituent NLCs.
Figure 6 Comparison of H-P slope for various metals. Red circle points for BCC metals[37], blue triangle points for FCC metals[37], black square points for metallic NLCs[7, 32-35, 46, 48, 49] including the present metallic NLCs, and dash line is the linear fitting by the formula $0.18 \mu \sqrt{b}$.
Figure 7 Interface barrier strength ($\tau^*$) normalized by shear modulus ($\mu$) for various metallic NLCs showing differences in $\tau^*/\mu$ between experimental values and theoretical values. Based on the difference between the experimental value and the theoretical value, three regions can be roughly identified by a little difference in the left pink-shadow region, a quite large difference in the right blue-shadow region and the medium difference in between.