Tailoring nanostructured Cu/Cr multilayer films with enhanced hardness and tunable modulus


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Length-scale dependent hardness (H) and indentation modulus (E) are systematically investigated in nanostructured Cu/Cr multilayer films with a wide modulation period (λ) range from 5 nm to 250 nm. H is gradually increased with reducing λ down to ~50 nm, whereafter a λ-independent effect is shown. The theoretical analysis of confined dislocation gliding and interface barrier strength quantitatively assess the variation of H. The indentation modulus E, however, exhibits a non-monotonic evolution with λ and attains a maximum at the same critical λ of ~50 nm. This unusual variation in E is related to the formation of interfacial amorphous layers, and can be reasonably explained in terms of a competition between the enhancement effect induced by compressed out-of-plane interplanar spacing of the constituent layers and the reduction effect associated with free volume in the amorphous intermixing layer. This result offers great benefits in engineering ductile nanolaminate materials with high strength and low modulus by tailoring interfacial structure and/or introducing amorphous layers.

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1. Introduction

Nanostructured multilayer films (NMFs) are increasingly being considered for various applications because of the improving capability to tailor the fabrication of these structures to meet specific property needs [1–4]. As layer thickness hM (one half of the modulation period λ) is decreased, three different regions are frequently observed in the variation of hardness/strength of NMFs [1–4]: the first region shows Hall-Petch (H-P) behavior, the second region shows an even greater dependence on layer thickness, and the third region exhibits a plateau or softening of hardness/strength. Correspondingly, three kinds of theoretical models have been proposed to explain the high strength/hardness of these materials as their characteristic dimensions shrinking toward the nanoregime, i.e., (i) the H-P like strengthening mechanism [4–6] based on the dislocation pileup against the interface applicable at the first region, (ii) the confined layer slip (CLS) mechanism [2–4] involving the single dislocation loop glide is confined to isolated layers applicable at the second region, and (iii) the interface barrier strength (IBS) mechanism [7–9] considering single dislocation cut cross the interface followed by glide of loops that span several layer thicknesses at the third region.

Accompanied with the significant increase in strength, the NMFs exhibit anomalous (either softening [10–12] or enhanced [13–15]) modulus compared with monolithic thin films of the constituent materials. The anomalous modulus effect remains controversial because researchers have not reached a consensus regarding the origin of effects, its magnitude or even its sign [13]. Strong softening of the effective shear and compression modulus with decreasing modulation period of the multilayers are usually observed. The softening of modulus has been interpreted as the result of the lattice expansion perpendicular to the film plane [10] or the effects of the disorder interface [11] and interfacial alloying [12]. At the same time, modulus enhancement in some immiscible systems [13–15] has also been reported. The modulus enhancement is often attributed to a strain-layered superelastic effect, wherein a dominant compression of the lattice softens it in certain directions [15].

Generally, high elastic modulus (E) and high hardness (H) are often discussed together because the processes are highly correlated: if a material shows low E, it tends to respond to large loads by plastic deformation, exhibiting low H. In this paper, a peak for the E at a critical modulation period (λ) ~50 nm is observed in the Cu/Cr NMFs with constant modulation ratio (η) of 1 (η defined the ratio of Cr layer thickness hCr to Cu layer thickness hCu, η = hCr/hCu), while the H monotonically increases within the whole λ-range spanning from 5 to 250 nm. When keeping λ constant and changing η from 0.11 to 1.0, the NMFs with λ ~25 nm (λ = hCr + hCu) exhibits higher H but lower E than that of ~50 nm ones. We illustrate the unusual mechanical properties by considering the competing effects of mixing-induced interfacial intermixing layer (or amorphous/disordered region) and the compression of the out-of-plane interplanar spacing of the Cr layer, and present an approach to

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design nanostructure materials with high hardness/strength and low (elastic) modulus.

2. Experimental procedure

Two kinds of silicon-supported Cu/Cr NMFs with total thickness about 500 nm were synthesized by means of direct current (DC) magnetron sputtering at room temperature. The chamber was evacuated to a base pressure of $4 \times 10^{-6}$ Torr, and $(1-3) \times 10^{-3}$ Torr Ar were used during deposition. The target purities of Cu, and Cr were 99.99% and 99.95%, respectively. The first ones have a constant modulation ratio $\eta$ of 1 (η defined the ratio of Cu layer thickness $h_{Cu}$ to Cu layer thickness $h_{Cu}$, $\eta = h_{Cu}/h_{Cu}$) but a wide range of modulation period $\lambda$ ($\lambda = h_{Cu} + h_{Cr}$) from 10 to 250 nm. The second ones have a constant $\lambda$ (λ = 25 and 50 nm, respectively) but a wide range of $\eta$ from 0.11 to 1.0. In multilayer deposition, the first layer on the polyimide substrate was Cr and the last layer was Cu. The as-deposited Cu/Cr NMFs were annealed at 150 °C for 2 h to stabilize the microstructure and eliminate the residual stress. The X-ray diffraction (XRD) experiment was carried out using an improved Rigaku D/max-RB X-ray diffractometer with Cu Kα radiation and a graphite monochromator to determine the crystallographic texture, the out-of-plane interplanar spacing and the residual stress of the multilayers by using "sin² $\psi$ method" [16–18]. Transmission electron microscopy (TEM) observation was performed using the JEOL-2100F high-resolution transmission electron microscopy (HRTEM) with 200 kV accelerating voltages to observe the modulation structure and the interface structure. Scanning transmission electron microscopy (STEM) and energy dispersive X-ray (EDX) analyses to identify the elemental composition and the interface integrity of the specimens were performed, with Fischione a ultra-high resolution high-angle annular dark field detector (0.23 nm resolution in STEM image mode) and Oxford instruments EDX detector with a spatial resolution of ~1 nm for chemical analysis. For comparison reasons, the 1 μm-thick monolithic Cu films and Cr films were also prepared and treated following the same method mentioned above.

The measurements on $H$ and $E$ were performed by using a MTS nanoindenter XP, equipped with a Berkovich diamond indenter and patented Continuous Stiffness Method (CSM) technique, in a constant displacement rate of 2 nm/s. The maximum indentation depth was 250 nm, with a displacement resolution of <0.01 nm and a loading resolution of 50 nN. A frequency of 45 Hz was used to avoid sensitivity to thermal drift. Details of the technique and analysis method can be found in Ref. [19]. Indents with spacing of 50 μm between each other were made on every sample to minimize the deviation of the results after rejecting few extreme values. To avoid the substrate effects on the intrinsic modulus of the NMFs, we used the simple model developed by Doerner and Nix [20] to obtain the "indentation modulus", following the treatment in Ref. [15].

3. Results and discussion

3.1. Microstructure

The high-angle XRD spectra shown in Fig. 1 for Cu/Cr NMFs exhibited that all the multilayers are polycrystalline, with a strong Cu-(111) and Cr-(110) texture. The spacing of atomic planes of Cu-(111) is very close to that of Cr-(110) so that their peaks overlap with each other in the XRD pattern at small $\lambda$. It is interesting to see that the peak of the NMFs shifts slightly to larger angle with reducing $h_{Cr}$, indicating a slight decrease of the spacing of both Cu-(111) and Cr-(110) planes. This is favorable for the enhancement of the modulus of NMFs [15]. By using XRD and the "sin² $\psi$ method", the residual tensile stresses are determined for all the annealed Cu/Cr NMFs far less than their strength [16–18]. Cross-sectional views of the Cu/Cr NMFs from the transmission electron microscopy (TEM) observations are respectively displayed in Figs. 2 and 3, showing columnar grains in the Cu layers and ultra-fine nanocrystals in the Cr layers. The average grain sizes of Cu and Cr layers were determined from the cross-sectional TEM images, as is consistent with the statistical results from the planar-view TEM observations. The average grain sizes of both Cu and Cr scale with the layer thickness, as shown in Fig. 2(d). The corresponding selected area diffraction patterns (SADPs) indicated that the NMFs exhibited a strong Kurdjumov–Sachs (K–S) orientation relationship in the growth direction: $<111>$Cu/$<110>$Cr; $<110>$Cu/$<111>$Cr, supporting the XRD results.

Interestingly, one can find that the interface of Cu on Cr (Cu/Cr interface) and the interface of Cr on Cu (Cr/Cu interface) are asymmetrical. There are bright fringes along the Cu/Cr interface (e.g., in Fig. 3(a)). Further HRTEM examinations and line scanning analysis reveal that these fringes are very thin intermixing amorphous layers, as typically shown in Fig. 3(b) and (c), respectively. This can be explained by considering the difference between the growth dynamics resulting from the different homologous temperatures of Cu and Cr. It is well known that the homologous temperature determines the degree to which adatoms are able to seek out minimum energy positions [15]. In the deposition process, Cu atoms with relatively high homologous temperature have greater mobility than Cr atoms. Thus, when Cu atoms deposit on the surface of Cr layer, a smooth surface tends to form with lower surface energy. By contrast, when Cr atoms deposit on a Cu surface the vapor atoms are incorporated epitaxially close their arrival point owing to their low mobility, and it is difficult for them to form a smooth surface or diffuse into the underlayer. When Cu is deposited on the Cr surface it will diffuse into the Cr layer to occupy defective sites and form

![Fig. 1. XRD patterns of the Cu/Cr NMFs with (a) $\eta = 1$ and (b) $\lambda = 25$ nm, respectively.](image-url)
a diffusive interface (see Fig. 3(c)). Once the Cu atoms are incorporated into the Cr layers near the interface, they compress the Cr lattice due to the relative small Cu atom volume [15], leading to the out-of-plane interplanar spacing compression of the Cr layer. The amorphous regions of intermixing layer between Cu and Cr (proved by the EDX analysis, see Fig. 3(c)) is mainly caused by the surface energy and the energetic non-equilibrium deposition process, which is similar to previous reports in the binary immiscible metallic multilayers such as Ag/Ni [21], Ag/Nb [22], and Cu/Ta [23,24]. The intermixing layer can play a decisive role in the mechanical properties and its thickness is insensitive to the layer thickness (about 1.5 ± 0.5 nm). These intermixing amorphous layers would be too thin to be detected by XRD. Wang et al. [25] have pointed out that the nanoscale amorphous/disordered intermixing layer not only sustains large tensile plasticity itself but also is likely to play the dominant mechanical role, exhibiting an extraordinary capacity to act as both a dislocation source and sink to mediate inelastic shear/slip transfer while avoiding extreme stress concentrations that lead to fracture initiation. It thus suggests that, compared with the NMFs with larger \( \lambda \), the NMFs with smaller \( \lambda \) may exhibit higher strength as well as superior deformability by introducing the very thin intermixing amorphous layers.

3.2. Length scale dependent hardness and modulus

Fig. 4(a) and (b) respectively shows the variation in hardness \( (H) \) and indentation modulus \( (E) \) with indentation depth for present \( \eta = 1 \) multilayers with \( \lambda = 100, 50 \) and \( 25 \) nm. One can see that plateau begins to occur for the hardness curves in the indentation depth about 200 nm as well as for the modulus curves. For simplicity, we therefore take the measured values at the depth of ∼200 nm to represent the intrinsic mechanical properties of the NMFs [15].

The dependence of flow stress \( \sigma \) (\( \sigma = H/3 \)) on \( \lambda \) is plotted in Fig. 4(c) for present Cu/Cr NMFs with \( \eta = 1 \). It is found that \( \sigma \) (or \( H \)) increases remarkably with reducing \( \lambda \) down to a critical \( \lambda^{\text{cri}} \) ∼ 50 nm, below which \( \sigma \) (or \( H \)) reaches a \( \lambda \)-independent plateau. This trend consists with Misra et al.'s results [2,5], indicating the transition of deformation mechanism. In Fig. 4(d), one can see that the result on Cu/W multilayers [15] showed a monotonic increase in \( E \) as \( \lambda \) reduced, just contrary to that of previously reported Ag/Nb NMFs [22]. The enhanced \( E \) with reducing \( \lambda \) results from the compression of out-of-plane interplanar spacing of Cr due to the Cu atoms diffuse into the Cr layer [15]. Thus, \( E \) should monotonically increase as the number of interface increases as well. On the other hand, the decreased \( E \) with reducing \( \lambda \) can be ascribed to the effect of amorphous region, which results from more interfaces in the NMFs with smaller \( \lambda \) [22]. Interestingly, over this \( \lambda \) range the \( E \) of Cu/Cr NMFs increases first, followed by a peak (∼178 GPa) at \( \lambda^{\text{cri}} \) ∼ 50 nm (see Fig. 4(d)). Below \( \lambda^{\text{cri}} \), \( E \) monotonically decreases with reducing \( \lambda \), analogous to that of Ag/Nb multilayers [22]. While above \( \lambda^{\text{cri}} \), a smaller \( \lambda \) leads to higher \( E \), similar to that of the reported Cu/W multilayers [15]. The different fashions between \( \sigma \) (or \( H \)) and \( E \) of present Cu/Cr NMFs suggests that the underlying deformations are fundamentally different: \( \sigma \) (or \( H \)) is conventionally regarded as the resistance to the plastic deformation and \( E \) reflects elastic deformation.

With regard to the \( E \), amorphous/disordered intermixing layers do play two competitive roles in influencing the \( E \): (i) intermixing...
layer can cause the compression of out-of-plane interplanar spacing of Cu and Cr layer \[14,15,21\], which increases with decreasing \( \lambda \), leading to \( E \) enhancement; (ii) the storage of excessive free volume in amorphous/disordered intermixing layers leads to the reduction in \( E \) \[12, 22, 26–29\]. It thus results in a decrease in \( E \) with decreasing \( \lambda \) due to more intermixing layers in the NMFs. Above \( \lambda \text{cri} \), the number of intermixing layers is small (as well as the amount of free volume), and the contribution of compressed out-of-plane interplanar spacing effects to the enhanced \( E \) is the controlling factor. Thus, reducing \( \lambda \) is favorable for increase in \( E \) (e.g., Cu/W \[15\]). Below \( \lambda \text{cri} \), the number of intermixing layers sharply increases but compression effects do not, which renders the reduced \( E \) of NMFs (e.g., Ag/Nb \[22\]). The two competing effects lead to the maximum in \( E \) observed in Fig. 4(d).

For the second kind of Cu/Cr NMFs with changing in \( \eta \), both \( \sigma \) (or \( H \)) and \( E \) monotonically increase with increasing \( \eta \) shown in Fig. 5(a) and (b), respectively. Interestingly, the Cu/Cr NMFs with \( \lambda = 25 \text{ nm} \) exhibits higher \( \sigma \) (or \( H \)) but lower \( E \) than that of the \( \lambda = 50 \text{ nm} \) NMFs at a given \( \eta \), as shown in Fig. 5. In other words, at a fixed \( \eta \) the smaller \( \lambda \) is, the higher \( \sigma \) (or \( H \)) and lower \( E \) is. It suggests that the number of interfaces play a positive role in enhancing \( \sigma \) (or \( H \)) and a negative role in enhancing \( E \). This \( \lambda \)-dependent \( E \) can also

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**Fig. 3.** (a) Bright-field cross-sectional TEM micrograph typically showing the microstructure of the \( \lambda = 25 \text{ nm} \) Cu/Cr NMFs with \( \eta = 1 \). (b) HR-TEM image typically showing the Cu/Cr interface with a very thin intermixing layer (~2 nm). Insert is the corresponding selected area diffraction pattern (SADP). (c) Line scanning analysis of the Cu/Cr interface for the \( \lambda = 50 \text{ nm} \) one with \( \eta = 0.33 \), showing the intermixing between Cu and Cr.

**Fig. 4.** (a) The hardness \( H \) and (b) elastic modulus \( E \) as a function of the indentation depth for the Cu/Cr NMFs with \( \lambda = 100, 50 \text{ and } 25 \text{ nm} \). Variation of (c) flow stress \( \sigma \) \( (\sigma = H/3) \) and (d) elastic modulus \( E \) with \( \lambda \). In (c), the solid curve is calculated from CLS model and the dash curve is from the IBS model. The hardness \( H \) and elastic modulus \( E \) of Cu/W \[15\] and Ag/Nb \[22\] are also included in (c) and (d) for comparison, respectively.
be explained by considering the effects of intermixing amorphous layer, i.e., the smaller $\lambda$, the more amorphous layers.

We next provide a quantitative explanation to the $\lambda$-dependent and independent $\sigma$ in this nanoregime, respectively. For present Cu/Cr multilayers composed of a softer Cu layer (the measured hardness is $\sim$2.2 GPa for 1 $\mu$m-thick films, falling in the range of reported values $\sim$1.8–2.8 GPa [1]) and a harder Cr layer (the measured hardness is $\sim$16.2 GPa for 1 $\mu$m-thick films, consistent with Firstov et al. results that the hardness is $\sim$15.5–21.6 GPa for 400–2000 nm-thick Cr films [30]), flow is controlled by the softer Cu phase rather than the harder Cr phase and thus the strengthening is closely linked to the smaller microstructural dimension of Cu between layer thickness and grain size [1–3,16–18]. In present Cu/Cr NMFs, the Cu layers have thickness much finer than the grain size except for the $\lambda$ = 250 nm ones, so the thickness of the Cu layer is the characteristic dimension controlling the flow behavior of the NMFs.

At large length scale ($>100$ nm), the dislocations emitted from dislocation sources propagate toward an interlayer interface and are stopped by it to form a pileup. The dislocations in the pileup act cooperatively to overcome the interface barrier for multilayers to yield. The larger the layer thickness, the more dislocations present in the pileup and the softer the multilayers. Based on the dislocation pileup theory, the H-P scaling law is used to relate the hardness/strength to layer thickness [4–6]. At nanoscale, the deformation of Cu/Cr NMFs is mainly controlled by the nucleation and motion of single dislocation rather than dislocation pileups in soft or ductile phase. The CLS mechanism [3–5] involving the glide of single dislocation loop in soft phase bounded by two interfaces comes into operation.

Misra et al. [4] refined the CLS model by considering the effects of dislocation core spreading along the interface, interface stress and interface dislocation arrays on the confined layer slip stress to explain the increase in strength with decreasing layer thickness. According to the refined CLS model, the applied stress $\sigma_{\text{CLS}}$ required to propagate a glide loop of Burgers vector $b$ confined to one Cu layer is given as [4,17]

$$\sigma_{\text{CLS}} = \frac{M\mu^*b}{8\pi h} \left( 1 - \nu \right) \ln \frac{ah}{b} - \frac{\mu^*V_CE}{m(1-\nu)},$$

(1)

where $M$ is the Taylor factor, $h = h_{\text{Cu}}/\sin \psi$ is the layer thickness parallel to the glide plane, $\psi$ is the angle between the slip plane and the interface, $b$ is the absolute length of the Burgers vector, $\nu$ is the Poisson ratio for Cu, $\mu^* = (\mu_{\text{Cr}} - \mu_{\text{Cu}})/(\mu_{\text{Cr}} + \mu_{\text{Cu}})$ is shear modulus of Cu/Cr multilayers, can be estimated by the shear modulus $\mu_{\text{Cu}}$ and volume fraction $V_{\text{Cu}}$ of Cu layer and that of Cr layer, $\alpha$ represents the core cut-off parameter, $f$ is the characteristic interface stress of multilayer, $\varepsilon$ is in-plane plastic strain and $m$ is the Schmid factor $\sim$0.27 for the active slip systems. With the parameters of $M = 3.06$, $\mu_{\text{Cr}} = 48.3$ GPa, $\mu_{\text{Cu}} = 115.4$ GPa, $\nu = 0.343$, $b = 0.3356$ nm, $\alpha = 0.2$, $f = 0.5$, $\varepsilon = 1\%$ and $\psi = 70.5^\circ$, we plot $\sigma_{\text{CLS}}$ as a function of $\lambda$ in Fig. 4(c). One can clearly see that as $\lambda > \lambda^*\text{CLS}$, the CLS model can fit experimental data very well. However, when $\lambda < \lambda^*\text{CLS}$, this model far overestimates the strength and is invalid. It suggests the transition of deformation mechanism, i.e., single dislocations cross the interface instead of slipping in the confined layer once this peak strength is reached [4,5]. It should be pointed out that Eq. (1) also fits the experimental data for the NMFs with constant $\lambda$ well, see Fig. 5(a).

Since the interface barrier to the dislocation slip transition is characteristic of interfacial structure and strongly influenced by lattice mismatch and shear modulus mismatch between two constituent layers [4,7,8]. Hence, if the interfacial structure does not change with $\lambda$ (e.g., the intermixing layer thickness is independent of $\lambda$ in our samples), the IBS ($\sigma_{\text{IBS}}$) also remains independent of $\lambda$, and is given by [7,8]

$$\sigma_{\text{IBS}} = Mf\mu^* \left( \frac{\xi}{1 - \nu} - \frac{b}{\xi} \right) + \frac{\mu_{\text{Cu}} V_CE \sin \psi}{8\pi}. \quad (2)$$

The first and second term is the influence of misfit strain and the modulus effect on IBS, respectively. Where $\beta$ is Saada’s constant, $R = (\mu_{\text{Cu}} - \mu_{\text{Cr}})/(\mu_{\text{Cr}} + \mu_{\text{Cu}})$, $\xi$ is the lattice mismatch of K–S orientation, $L = b/\xi$ [4] is a parallel array of glide loops of spacing, and other symbols have the same meaning as before. Taking $\beta = 0.4$, $\psi = 70.5^\circ$ and $\xi = 2.3\%$, using Eq. (2), the $\sigma_{\text{IBS}}$ is also presented in Fig. 4(c). For the present NMFs, the intersection point of the $\sigma_{\text{IBS}}$ line and CLS curve about 3.3 GPa yields a critical $\lambda$ = 50 nm, consistent with the experimental results. Above $\lambda^*\text{CLS}$, the rapid increase in $\sigma$ (or $H$) is caused by the nucleation and motion of dislocations within the confined layer, which is strongly suppressed by increased layer-to-layer interfaces (or intermixing layers) [1–4]. While below $\lambda^*\text{CLS}$, the plateau of $\sigma$ (or $H$) can be attributed to the single dislocations transmit cross the Cu/Cr interface [4,5,25], leading to a $\lambda$-independent peak $\sigma$ (or $H$) around 3.3 (or 10) GPa. It should be noted that, in the calculation of $\sigma_{\text{IBS}}$, we do not consider the variation of $\mu^* (or \mu_{\text{Cu}} and \mu_{\text{Cr}})$ of Cu/Cr NMFs caused by the competing effect between compressed out-of-plane interplanar spacing-induced modulus enhancement and amorphous intermixing layer-induced modulus reduction of the Cu/Cr NMFs and the possible strengthening effect caused by the very thin intermixing layer.

Finally, we will try to understand the difference in the length scale-dependent hardness of Cu/Cr, Cu/W and Ag/Nb multilayer, i.e., why the critical period can only occurs in Cu/Cr system but not in Cu/W and Ag/Nb system, based on factors that may determine the interface barrier strength, including mismatch strain, modulus mismatch, enthalpy of formation and dislocation core spreading along interfaces. The measured peak hardennesses of four fcc/bcc multilayer systems are listed in Table 1, together with the enthalpy of mixing, modulus mismatch and mismatch strain between fcc {1 1 1} and bcc {1 1 0} interplanar spacing. In earlier literature, it was postulated that factors such as large modulus mismatch (or Koeehler stress), large mismatch strain and high enthalpy of formation are usually considered favorable for hardness enhancement and play a dominant role in determining the peak hardness [2,7,8,22,23]. However, the peak hardness is the highest for Cu/Cr, then Cu/W [15], followed by Ag/Nb in descending sequence. Thus, there may be some other factors contributed to the highest hardness/strength of Cu/Cr, such as the intermixing layer at interface as well as the K–S oriented texture.
The saturation hardness is observed in Cu/Cr multilayers; nevertheless both the Cu/W and Ag/Nb show the monotonic increase in hardness. The underlying reason for the sharp enhancement of hardness of λ = 4 nm Ag/Nb multilayers originates from a unique microstructure where 1–3 nm thick amorphous Ag–Nb alloy regions form at the interfaces and grain boundaries of Ag nanoparticles [22], which induces the saturation hardness is not observed as λ in the range of 4–80 nm. While, in Cu/W multilayers the large interface and modulus mismatch and high enthalpy of mixing are main reasons responsible for the drastically monotonic increase in hardness with reducing λ [15], in addition to the influences of microstructure (random orientation and diffusive interface). It should be pointed out that there is the possibility that the critical modulation period occurs at smaller length scale, say 2–4 nm, similar to the case of Cu/Nb system (peak hardness at λ = 2.4 nm) [4].

4. Conclusions

The present results show that the Cu/Cr NMFs with intermixing layers can simultaneously possess high hardness/strength and low modulus at a critical modulation period about ∼50 nm. The theoretical analysis of confined dislocation gliding and interface barrier strength associated with lattice mismatch and shear modulus mismatch between two constituent layers confirm the crossover of the deformation mechanisms at which NMFs exhibit the strength plateau. The maximum indentation modulus can be reasonably explained by considering the competition between the enhancement effect induced by compressed out-of-plane interplanar spacing of the constituent layers and the reduction effect associated with free volume in the amorphous intermixing layer. It reveals a valuable scheme to synthesize ductile, high strength and low modulus nanolaminated materials by tailoring the interfacial structure and/or introducing amorphous layers and opens new avenues for improving the plasticity of crystalline materials.

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