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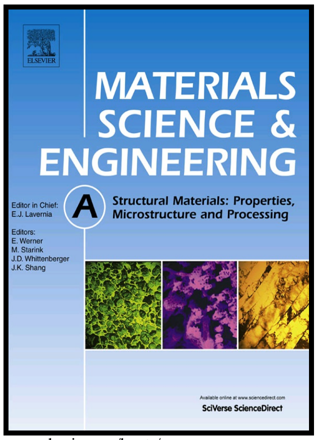


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# Effect of strain rate on mechanical properties of Cu/Ni multilayered composites processed by electrodeposition

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## Abstract:

Mechanical properties of Cu/Ni multilayered composites processed by electrodeposition were investigated by tensile tests at different strain rates in the range of $5\times10^{-5}$ to $5\times10^{-2}$ $s^{-1}$ at room temperature. With increasing strain rates, the strength and ductility of Cu/Ni multilayered composites increased simultaneously, while their strain rate sensitivity also increased, which is very different from the constituent pure Cu and Ni. The back-stress caused by the Cu/Ni layer interfaces also increased with strain rate. Strong back-stress work hardening is observed, which is the main reason for the observed good ductility.

Keywords: multilayered composite, strain rate, strength, ductility



## 1 Introduction

Improving mechanical properties of conventional engineering materials were inspired by the development of the laminated structures developed in biological materials (1). Therefore, metallic laminated composites have attracted the attention of more and more researchers in recent years (2-4). Many investigations have been carried out on mechanical properties of metallic laminated composites by controlling the constituent layers and interfaces, which revealed a combination of high strength with good ductility (5-9). The high performance of metallic laminated composites was believed to result from the fact that the hard layers can contribute to strength, while the soft layer can improve the uniform elongation by delaying the necking behavior due to the layer interface effects (3, 10-12). Metallic multilayered composites have been reported prepared by several methods including electron beam evaporation (13), bonding deposition (14, 15) and electrodeposition (16, 17). High strength has been observed (18, 19), whereas the ductility of multilayered composites turned out to be still needed to be improved (20, 21).

Generally speaking, strain rate may significantly affect mechanical properties of metals. Face-centered cubic (fcc) metals usually display higher tensile strength, but lower ductility with increasing strain rates (22-28). However, an electrodeposited

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nanocrystalline (NC) Cu has been reported to demonstrate an increase in ductility with increasing strain rates (29). Subsequently, the same phenomenon for ductility was found in NC-Ni (30), ultrafine-grained (UFG) Cu(31, 32) and Cu/Ni laminated composites (33, 34). Nevertheless, the mechanism of this abnormal tendency is not well explained.

In this study, we investigated tensile properties of the electrodeposited Cu/Ni multilayered composites with different deposition time and strain rates. Longer deposition time results in thicker individual layers. Effects of deposition time and strain rates on tensile strength and ductility of Cu/Ni multilayered composites will be discussed.

## 2 Experimental procedure

Cold-rolled Cu sheets 2 mm in thickness were fully annealed at 650°C for 2 h in order to decrease initial dislocation density and to increase good plasticity. Then the Cu sheets were mechanically polished with sandpapers from coarse to fine grades. The polished sheet was used as the substrate to produce Cu/Ni multilayered composites by electrodeposition at a temperature of 50°C. Cu layers were electrodeposited in an electrolyte solution containing 200 g. $L^{-1}$ $CuSO_4.5H_2O$, 10 g. $L^{-1}$ $H_3BO_4$, 0.08 g. $L^{-1}$ HCl and 0.05 g. $L^{-1}$ $H_2SO_4$ in deionized water and the PH value slightly lower than 3, while the Ni layers deposition solution was composed of 350 g. $L^{-1}$ $Ni(SO_3NH_2)_2.4H_2O$, 10 g. $L^{-1}$ $NiCl_2.6H_2O$ and 30 g. $L^{-1}$ $H_3BO_3$ in deionized water and the PH value slightly higher than 3. The Cu and Ni layers were alternately deposited, and the time of deposition was 10 min (10 min-sample) and 30 min (30 min-sample) for per layer. The deposition process lasted 4 hours. The first layer of deposition was Ni. The polished stainless steel sheet was used as the substrate to produce the individual pure Cu and Ni by electrodeposition under the same conditions and then removed. The prepared samples with a thickness of 500μm used for baseline study.

Dog-bone shape tensile specimens of pure Cu, Ni, and Cu/Ni multilayered composites were fabricated by wire-electrode cutting. The samples are 15 mm in gage length and 5 mm in width and their surfaces were polished to mirror finish. Uniaxial tensile tests were performed using a SHIMADZU Universal Tester, with different strain rates ($\varepsilon$) ranging from $5\times10^{-5}$ to $5\times10^{-2}$ $s^{-1}$ at room temperature. Jump tests were carried out over the strain rate ranging from $5\times10^{-5}$ to $2\times10^{-2}$ $s^{-1}$ and the loading-unloading-reloading (LUR) tests were conducted at the strain rates $5\times10^{-4}$ $s^{-1}$and $5\times10^{-2}$ $s^{-1}$. To ensure the repeatability of the stress-strain curves, at least three tests were carried out under each testing condition. The cross-sectional microstructure of the Cu/Ni multilayered composites with the deposition time of 10 min and 30 min per layer were performed on an optic microscope (OM, Leica DM 5000). The microstructure and grain orientation of Cu and Ni layers were observed on a field emission scanning electron microscope (FE-SEM, NOVA Nano SEM 450) equipped with an electron backscatter diffraction (EBSD) detector for image acquisition. The grain size and distribution of the Ni and Cu layers were estimated using EBSD images using the software (Channel-5), which was used to measure the grain area and subsequently estimate the grain size.

# 3 Results

The OM cross-sectional micrograph of the deposited Cu/Ni multilayered composites for 10 min-samples and 30 min-samples are shown in Fig. 1(a) and (b), respectively. The Cu and Ni layers for the 10 min-sample have a mean thickness of 5.34 μm and 6.15 μm, while for the 30 min-sample they are 13.11 μm and 12.84 μm, respectively. The total thickness is about 137 μm for 10 min-sample and 104 μm for 30 min-sample. The thickness ratio of Cu and Ni layers is 0.87 for 10 min -sample and 1.02 for 30 min- sample. The results of EBSD of Cu and Ni layers are shown in Fig. 1(c) and (d), respectively. Fig. 1(e) and (f) present distribution of the grain size of Cu and Ni layers, which exhibit that the average grain size is 1.26 μm and 1.47 μm, respectively.

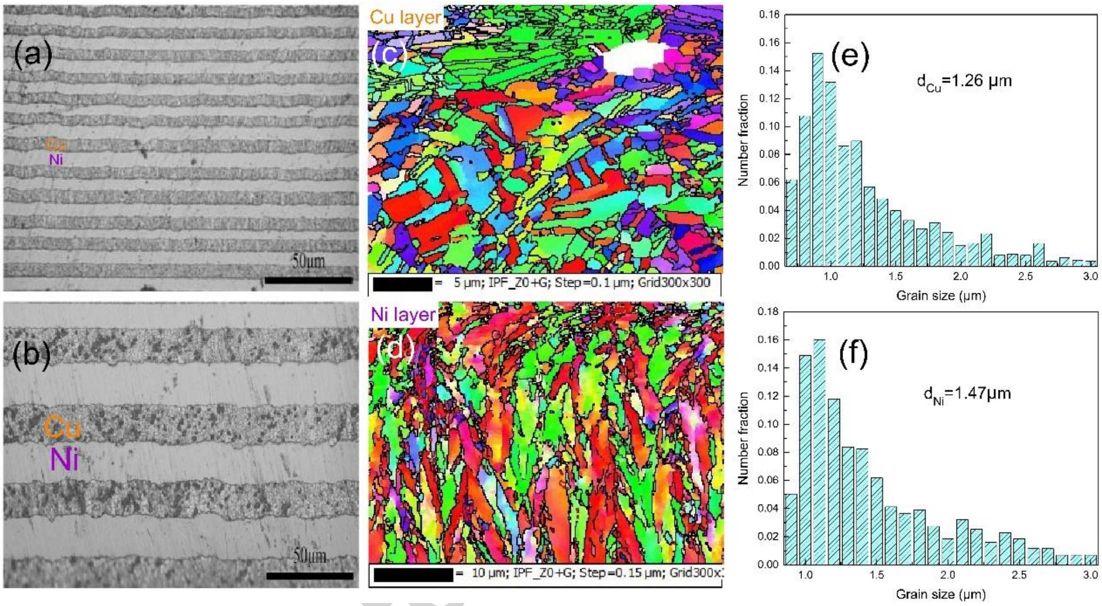


Fig. 1. The cross-sectional characterization of microstructures of Cu/Ni multilayered composites. OM in (a) 10 min-sample and (b) 30 min-sample; Results of the EBSD of (c) Cu layer and (d) Ni layer; Statistic distribution of grain size of (e) Cu and (f) Ni layers.



Figs. 2(a) and (b) display a series of typical uniaxial tensile engineering stress-strain responses of the 10 min-samples and the 30 min-samples at $\dot{\varepsilon}=5\times10^{-5}$-$5\times10^{-2}$ $s^{-1}$, respectively. As the strain rate ($\dot{\varepsilon}$) increased from $5\times10^{-5}$ to $5\times10^{-2}$ $s^{-1}$, the yield strength and ultimate tensile strength for both the 10 min-sample and the 30 min-sample increased gradually, while the uniform elongation also dramatically increased, as shown in Figs. 2(c) and (d). As can be also seen from Figs. 2(c) and (d), the yield strength for the 10 min-sample is higher than that for the 30 min-sample, and the ultimate tensile strength follows the same trend. However, the uniform elongation for the 10 min-sample is only slightly lower than that of the 30 min-sample.

In addition, the normalized strain hardening rate($\Theta = (1/\sigma)(\partial\sigma/\partial\varepsilon)_{\dot{\varepsilon}}$, where σ is the true stress and the ε is the true strain) decreased quickly with increasing $\varepsilon_t$ at different strain rates for all samples (Figs. 2(e) and 2(f)). It is clear that Θ of the 10 min-sample increased with increasing $\dot{\varepsilon}$ at the same strain, especially at $\varepsilon_t > 0.02$, so does the that Θ of the 30 min-sample. Furthermore, the Θ of the30 min-sample is slightly higher than the Θ of the 10 min-sample at the same strain and strain rate, which is why the 10 min-sample has slightly lower ductility. The necking will appear at Θ≦1. In other words, the onset of necking for the 10 min-sample is corresponding to $\varepsilon_t$ =0.192 at $\dot{\varepsilon}$=5×10$^{-5}$, and $\varepsilon_t$ =0.263 at $\dot{\varepsilon}$=5×10$^{-2}$s$^{-1}$; and for the 30 min-sample is in accordance with $\varepsilon_t$ =0.177 at $\dot{\varepsilon}$=5×10$^{-5}$, and $\varepsilon_t$ =0.297 at $\dot{\varepsilon}$=5×10$^{-2}$s$^{-1}$. It is generally known that the strain hardening ability is determined by the competition between the dynamic recovery and storage of dislocations (35), and is important for improving the uniform elongation (namely tensile deformation capacity). It is evident from (e) and (f) in Fig. 2 that the work hardening rate rises with increasing strain rate for both the 10 min-sample and the 30 min-sample. This is the best way to delay the onset of necking for the Cu/Ni multilayered composites.

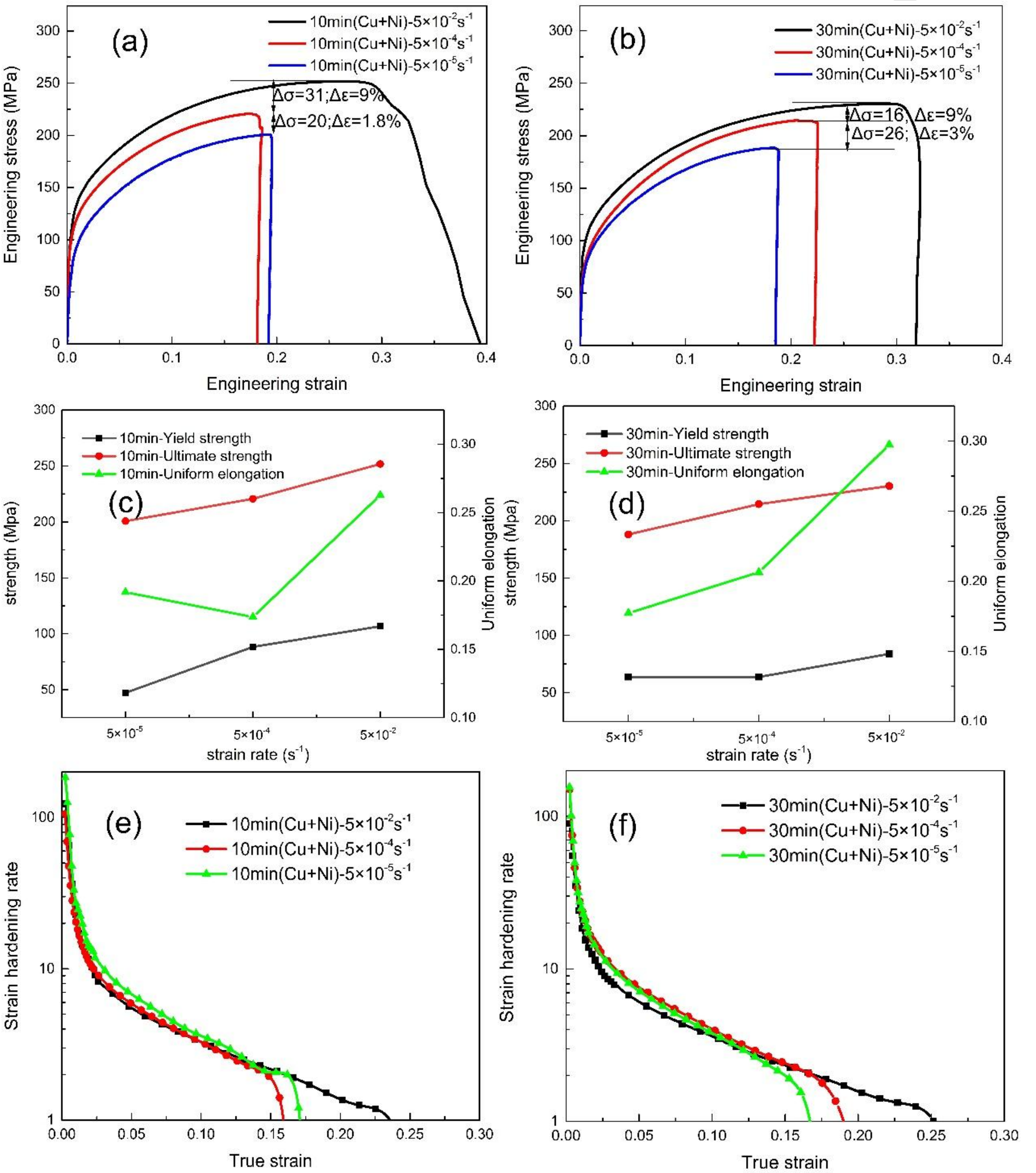

Fig. 2. The typical uniaxial tensile properties of Cu/Ni multilayered composites: engineering stress-strain curves of (a) 10 min-sample and (b) 30 min-sample, yield strength, ultimate tensile strength and uniform elongation vs. strain rate of (c) 10 min-sample and (d) 30 min-sample and the normalized strain hardening rate dependence on true strain of (e) 10 min-sample and (f) 30 min-sample.

It is well recognized that the mechanical properties of multilayered composites are greatly influenced by the interfaces of between Cu/Ni layers. In order to investigate the effect of strain rate on mechanical properties of the layered structures with a high density of interfaces, we conducted the tensile tests of pure Cu and Ni under different strain rates (Fig. 3(a)), respectively. It can be seen that the ultimate tensile strength increments of pure Ni is 87 MPa but no uniform elongation increment with increasing strain rate from $5\times10^{-4}$ to $5\times10^{-2}$ $s^{-1}$; while for pure Cu, the ultimate tensile strength is lower but the uniform elongation increased with increasing $\dot{\varepsilon}$. The ultimate tensile strength and the uniform elongation increments are 23 MPa and 4%, respectively.

To investigate the effect of Cu/Ni interfaces on mechanical properties, Zhang et al (33) assumed that there is a stress gradient near the interfaces. According to the ultimate tensile strength of pure Cu and Ni, the schematic illustration of stress gradient effect of Cu/Ni multilayered composites are shown in Fig. 3(b). We can see that the stress gradient near Cu/Ni layer interfaces increases with increasing strain rate. The existence of stress gradient will lead to the plastic strain gradient near the Cu/Ni interfaces, which needs to be accommodated by the geometrically necessary dislocations, which in turn produces back-stress (36, 37).

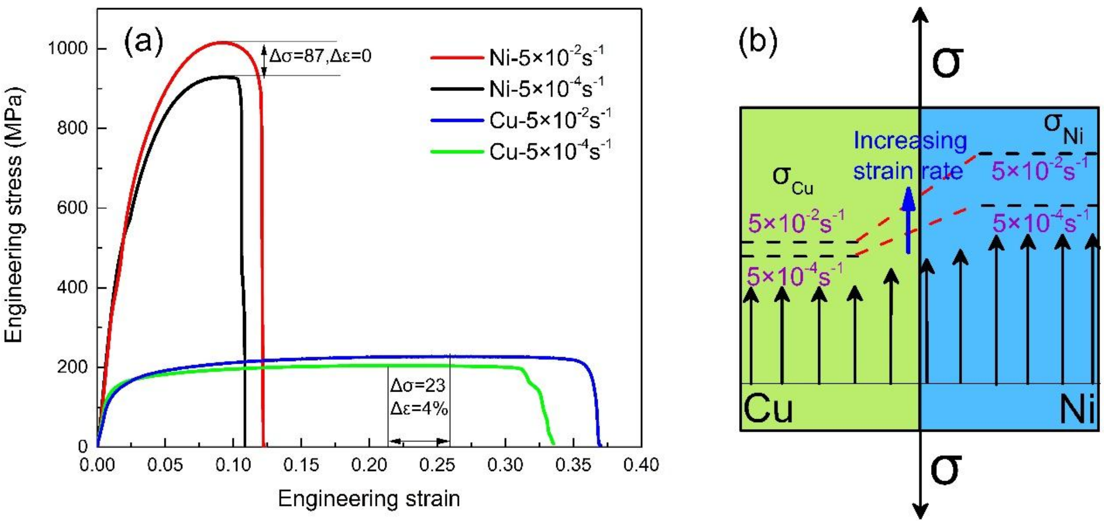


Fig 3. (a)Tensile stress-strain curve of pure Cu and Ni with a thickness of about 500 μm prepared at the same conditions as the multilayered composites at strain rates at $5\times10^{-4}$ $s^{-1}$ and $5\times10^{-2}$ $s^{-1}$. (b) Schematic illustration of stress gradient effect of Cu/Ni multilayered composites.

The back-stress, a long-range stress caused by geometrically necessary dislocations (37) can simultaneously increase strength as well as retain uniform elongation (38). The Cu/Ni multilayered composite is a type of heterostructured material according to the definition by Wu and Zhu (37), and for heterostructured materials, the strengthening of back-stress can be much higher than that of the statistically stored dislocations (3, 37).

We conducted loading-unloading-reloading (LUR) testing of Cu/Ni multilayered composites at the strain rates $5\times10^{-4}$ $s^{-1}$and $5\times10^{-2}$ $s^{-1}$ (Fig. 4(a)) to measure the back-stress using a procedure developed by Yang et al (39). Also, we investigated the back-stress dependence on strain rate and the relationship between the back-stress and the flow stress. Interestingly, it is evident from the inset in Fig. 4(a) that the Cu/Ni multilayered composites display the strong Bauschinger effect. Stronger Bauschinger effect is linked to higher back-stress (40). The back-stress dependence on the true strain of Cu/Ni multilayered composites at the strain rates $5\times10^{-4}$ $s^{-1}$and $5\times10^{-2}$ $s^{-1}$ is shown in Fig. 4(b). It can be seen that the back-stress of Cu/Ni multilayered composites increased with increasing true strain from 1% to 14% and strain rate.

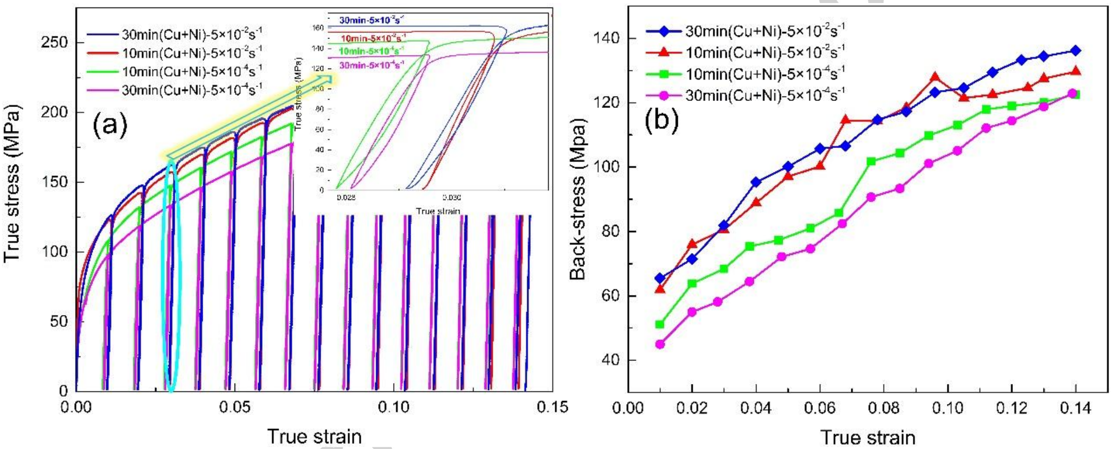


Fig. 4. Bauschinger effect and back-stress of Cu/Ni multilayered composites. (a) LUR stress-strain curves of 10 min-sample and 30 min-sample at the strain rates $5\times10^{-4}$ $s^{-1}$ and $5\times10^{-2}$ $s^{-1}$ and the magnified view of third hysteresis loop (inset). (b) The back-stress dependence on the true strain of Cu/Ni multilayered composites. The quick increase in back-stress with increasing strain indicates a strong back-stress induced work hardening.

In addition to back-stress, another mechanical property that might be related to the interface of Cu/Ni layers is the strain rate sensitivity (m). To measure the m on strength and uniform elongation of the Cu/Ni multilayered composites, we carried out strain rate jump tests on pure Cu, Ni and Cu/Ni multilayered composites over the strain rate ranging from $5\times10^{-5}$ to $2\times10^{-2}$ $s^{-1}$. Fig. 5(a) shows the jump tests of Cu/Ni multilayered composites. The m value for each jump test was calculated using the following formula (41)

$$m \equiv \left\{\frac{\partial \ln\sigma}{\partial \ln\dot{\varepsilon}}\right\}_{T,\varepsilon} \approx \left\{\frac{\ln(\sigma_2/\sigma_1)}{\ln(\dot{\varepsilon}_2/\dot{\varepsilon}_1)}\right\}_{T,\varepsilon}, \qquad (1)$$

where T represents the absolute temperature; $\sigma_1$ and $\sigma_2$ are the true stress before and after the strain rate jump immediately at the same strain and room temperature, respectively; $\dot{\varepsilon}_1$ and $\dot{\varepsilon}_2$ represent the strain rate prior to and after the strain rate-change test, respectively.

The m value of pure Cu, Ni and Cu/Ni multilayered composites at varying strain rates are shown in Fig. 5(b). The reported m value of 21 nm/21nm Cu/Ni multilayer thin films (34) is also shown in Fig. 5(b). It is worth noting that for the pure Ni the m is 0.014 when the strain rate is $5\times10^{-5}$ $s^{-1}$ and decreases to 0.007 at $2\times10^{-2}$ $s^{-1}$; the m value is relatively invariable for pure Cu with an average value of m 0.009. However, the m value increased from 0.004 to 0.017 when the strain rate increased from $5\times10^{-5}$ to $2\times10^{-2}$ $s^{-1}$ for the Cu/Ni multilayered composites. In other words, the strain sensitivity of the Cu/Ni layered composites behaves very differently from that of its pure Cu and Ni constituents and increased quickly with increasing strain rate.

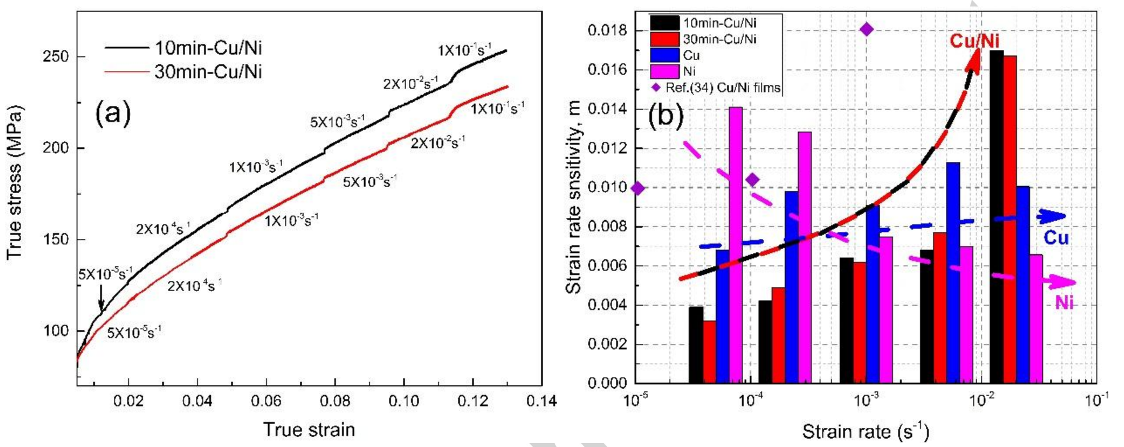


Fig. 5. (a) The tensile true stress-strain curves obtained in strain rate jump tests with strain rate ranging from $5\times10^{-5}$ to $2\times10^{-2}$ $s^{-1}$ for the Cu/Ni multilayered composites at room temperature. (b) Strain rate sensitivity vs strain rate plot of pure Cu, pure Ni, Cu/Ni multilayered composites in our work and 21 nm/21nm Cu/Ni multilayer thin films from the reference (34).

# 4 Discussions



In terms of strength of Cu/Ni multilayered composites, according to the formula $\sigma \propto \dot{\varepsilon}^m$ (29) ($\sigma$, $\dot{\varepsilon}$, and m represent flow stress, strain rate and strain rate sensitivity, respectively), the flow stress increases with increasing $\dot{\varepsilon}$ and m. And the strain rate sensitivity (m) of both the 10 min-sample and the 30 min-sample are shown in Fig. 5(b), where m value rises with increasing $\dot{\varepsilon}$. On the other hand, according to the relation between the strain rate and the dislocation motion $\dot{\varepsilon} = b\rho\bar{\upsilon}$ (41) (b represent the magnitude of Burgers vector; $\rho$ is the density of mobile dislocations and $\bar{\upsilon}$ is the average dislocation velocity related to resolved shear stress $\tau$) and the fact that when some dislocations begin to move, the stress needed to move the dislocations can drop and the average dislocation velocity can decrease (41), the density of mobile dislocations increased with increasing $\dot{\varepsilon}$, which can generate a great number of dislocation interactions and pile-up and lead to a greater strain hardening. This is beneficial to enhancing the ductility.

In addition, as can be seen from Fig. 4(b), the back-stress of Cu/Ni multilayered composites increased with increasing strain rate, which is mostly due to the higher geometrically necessary dislocations near the Cu/Ni layer interfaces at higher strain rate associated with back-stress. Furthermore, back-stress is related to plastic strain gradient, and the plastic strain is accommodated by geometrically necessary dislocations (3, 42). Consequently, the pile-up of a great amount of geometrically necessary dislocations creates strain gradient as well as stress gradient. This is consistent with the assumption of Zhang et al (33). This stress gradient will be increased with increasing $\dot{\varepsilon}$ (33), which is also evidenced in Fig. 3. In other words, the higher stress gradient means the higher pile-up of geometrically necessary dislocations of Cu/Ni multilayered composites at higher strain rate, which produces higher back-stress. And higher back-stress will enhance the strength of Cu/Ni multilayered composites. Also, a larger strain gradient of Cu/Ni layer interface at higher strain rate can promote the back-stress induced work hardening during the tensile tests, which consequently helps with achieving a higher uniform elongation (ductility).

Furthermore, the strain rate sensitivity (m) could have an effect on uniform elongation. Based on Hart's criterion for strain rate sensitive materials (43):

$$\frac{1}{\sigma}\left(\frac{\partial\sigma}{\partial\varepsilon}\right)_{\dot{\varepsilon}} - 1 + m \leq 0, \qquad (2)$$

where the first term and m represent the normalized strain hardening rate and the strain rate sensitivity, respectively. From Hart's criterion, for rate-sensitive materials, the m is an important factor to effectively delay the onset of necking and to improve tensile ductility (41). For grain boundary deformation mechanisms including grain boundary sliding and Coble creep, m could be 0.5-1(44), i.e. much higher than what we observed. Therefore, over the strain rate ranging from $5\times10^{-5}$ to $2\times10^{-2}$ $s^{-1}$, the deformation mechanism of present pure Cu, Ni, and Cu/Ni multilayered composites is based on dislocation-based plasticity.

Some investigations of strain rate sensitivity for multilayered films have been reported. Cao et al. (45, 46) found the m value for Ti/Ni multilayered films remains largely constant with increasing strain rate. Wang et al. (47) believed that the m value decreased with increasing strain rate for the Cu/Ta multilayered films because a faster loading strain rates (LSR) enabled more interface activities per unit time and easier interface transmission of dislocations at the Cu/Ta incoherent interfaces, resulting in less pile-up at the interface and lower back-stress. However, it is evident from Fig. 4(b) that the m value for Cu/Ni multilayered composites increased with increasing strain rate, which is similar to the trend of 21nm/21nm Cu/Ni multilayer thin films (34). This is not observed in pure Cu, Ni, the electrodeposition NC Ni (25) and other multilayered films (45-47). Carpenter et al.(34) thought the increase in m implies that the strong evolution of dislocation interaction is not observed in nanocrystalline metals, even including nano-twinned grains.

Does the m value of present Cu/Ni multilayered composites, whose grain size is different from thin films, increase for the similar reason? For the dislocation-based mechanism, the m should increase when the dislocation density is increased in a metal with a given grain size. But the increase of m with dislocation density is relatively small for large grain size (22, 48). It is known that in fcc metals, the dynamic recovery of dislocations could be restricted with increasing the strain rate (31). This means the higher storage of dislocations at higher strain rate. However, it is puzzling that the m value increased for Cu/Ni multilayered composites, which is opposite to pure Cu and Ni.

It is well known that the strain rate sensitivity (m) is inversely proportional to the activation volume (V*). As discussed above, there is a great amount of geometrically necessary dislocations with very small spacing near the Cu/Ni layer interfaces, which can act as concentrated obstacles to the dislocation movement (49). This leads to a small activation volume near the Cu/Ni layer interfaces. Besides, the geometrically necessary dislocations increase with increasing strain rate and eventually increases strain rate sensitivity. In other words, the effect of Cu/Ni layer interfaces on m value of Cu/Ni multilayered composites is relatively small at the low strain rate and relatively large at the high strain rate. Because the m increases with increasing $\dot{\varepsilon}$**,** it can help with improving uniform elongation as shown in Figure 2(a) and 2(b).

# 5 Conclusions

The Cu/Ni multilayered composites synthesized by electrodeposition displayed simultaneously increased in strength and ductility with increasing tensile rates from $5\times10^{-5}$ to $5\times10^{-2}$ $s^{-1}$ for both the 10 min-samples and the 30 min-samples. The increase of strength is mainly due to the back-stress of Cu/Ni multilayered composites caused by the Cu/Ni layer interface, which increases with increasing strain rate. And the increase of uniform elongation can be attributed to the increase of strain rate sensitivity and the back-stress induced work hardening by the Cu/Ni layer interfaces, which help delay the onset of necking during tensile tests.

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