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Unusual strain rate sensitivity of nanoscale amorphous CuZr/crystalline Cu multilayers

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ABSTRACT

Nanoscale amorphous CuZr/crystalline Cu multilayers were synthesized to study their strain rate sensitivity and plastic deformation mechanisms via nanoindentation testing. Despite the dramatically reduced microstructural length scale, nearly constant strain rate sensitivity of the multilayers was obtained at a wide range of individual layer thickness (10~100 nm). The scenarios that the two constituent layers mutually confined by each other might exhibit quite different strain rate sensitivities relative to the monolithic ones were proposed. Specifically, compared with the monolithic Cu and CuZr layers, respectively, the confined Cu layers should exhibit gentler increases in strain rate sensitivity while confined CuZr layers exhibit reduced strain rate sensitivity, as Cu layer thickness decreases. Correspondingly, corresponding deformation mechanisms accommodating the co-deformation of the two constituent layers were discussed in detail.

1. Introduction

Metallic glasses (MGs) are attractive for many technological applications due to their excellent physical, chemical and mechanical properties [1–4]. So far, however, the limited ductility with nearly no global plasticity has impeded the widespread use of MGs as structural materials. Numerous approaches have been undertaken to overcome the deleterious effects of shear banding deformation For instance, it has been reported that the adding of crystalline layers could suppress shear band instability in metallic amorphous/crystalline multilayers [5,6].

Nanoscale metallic amorphous/crystalline multilayers exhibited unique mechanical properties as the nanolaminate composites could be both ductile and strong [5–15]. Tremendous efforts have been devoted to understanding the underlying deformation mechanisms and exploring how ductility could be improved by combining the crystalline and amorphous layers [6,10,16–18]. It has been well documented that the main cause of instantaneous brittle fracture in MGs, i.e., nucleation and propagation of shear bands (SBs), could be suppressed by adding ductile crystalline metals into the MGs to construct new materials systems, e.g., amorphous/crystalline multilayers. Specifically, the confining effects (i.e., MG layers constrained by two adjacent crystalline metal layers) were proposed to play a key role in achieving simultaneous large ductility and high strength [6,16].

In contrast, strain rate sensitivity, as a crucial parameter in providing quantitative measures of the sensitivity of flow stress to loading rate and understanding the plastic deformation of MGs, has received much less attention in amorphous/crystalline multilayers [19,20]. High strain rate sensitivity combined with strong strain hardening generally means the ability to resist localized plastic deformation is high. Therefore, evaluating and interpreting the physical mechanisms underlying the strain rate sensitivity of amorphous/crystalline multilayers is important from both the scientific and engineering views. Previously, the strain rate sensitivity of both crystalline metals and MGs has been extensively studied, with ample deformation mechanisms and models proposed and debated [1,21–26]. However, nearly all of these studies concerned on the strain rate sensitivity of monolithic crystalline or MGs, in which the microstructural circumstances should be quite different from those of the crystalline and metallic glasses constituent layers in amorphous/crystalline multilayers.

Previously, by evaluating microstructural evolution and dynamics of shear bands, Guo et al. revealed the strain rate dependent shear band propagation velocities and strain rate sensitivity of flow stress in amorphous/crystalline CuZr/Cu nanolaminate pillar [19]. Despite that, the nature of nanoscale pillar may complicate the interpretation that how strain rate affects the plastic deformation mechanisms of amorphous/crystalline nanolaminates, as the extrinsic size effects attributed from the pillar size could significantly affect their mechanical properties [27,28]. Therefore, concerning only on the amorphous/crystalline

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nanolaminate structure itself but without the extrinsic size effects, one may thence ask: could the aforementioned mutual confined effects that succeeded in preventing shear banding deformation [6,16] also play a crucial role in determining the strain rate sensitivity of the amorphous/crystalline multilayers?

In the present study, nanoscale CuZr/Cu amorphous/crystalline multilayers, which have been widely studied in the open literature, were synthesized to evaluate their rate sensitivity of strength and the underlying deformation mechanisms. The main focus was placed upon the possible effects of confined crystalline and amorphous layers on the strain rate sensitivity of both constituent layers themselves as well as the entire multilayer.

2. Experiments

2.1. Materials preparation

Nanoscale *amorphous CuZr/crystalline Cu* (referred to as CuZr/Cu hereafter) multilayers were deposited on Si (100) wafers by alternatively using direct current and radio frequency magnetron sputtering at room temperature. The individual layer thickness (h) of both CuZr and Cu was 10, 20, 50, 75 and 100 nm as the modulation ratio (η) was set as a constant of 1. The total thickness of all the CuZr/Cu multilayers was ~1200 nm.

2.2. Microstructure characterization

Field-emission scanning electron microscopy (FSEM, SU6600, HITACHI) was used to characterize individual layer thickness and residual indentation. Microstructural features of the thin films were characterized via X-ray diffraction (XRD, X-ray diffractometer 7000S) with Cu Ka radiation and transmission electron microscopy (TEM, JEOL, JEM-2100F).

2.3. Nanoindentation tests

Nanoindentation testing on CuZr/Cu multilayers was performed using Nanoindenter XP® system (MTS, Inc.) with a standard Berkovich diamond tip (radius ~50 nm) at room temperature. Penetration depths were set to be less than ~15% of total film thickness to avoid substrate effect. The hardness of the multilayers was measured using the continuous stiffness method at a constant strain rate of 0.05 s^{-1}; a holding segment of 10 s at maximum load was set before unloading. To evaluate the strain rate sensitivity, nanoindentation was performed at varying loading strain rate (0.2, 0.1, 0.05, 0.01 and 0.005 s^{-1}) at the maximum penetration depth of 200 nm. For all the multilayers, 9 indentions were performed at each indentation test and at least 5 effective data were used in subsequent data analysis.

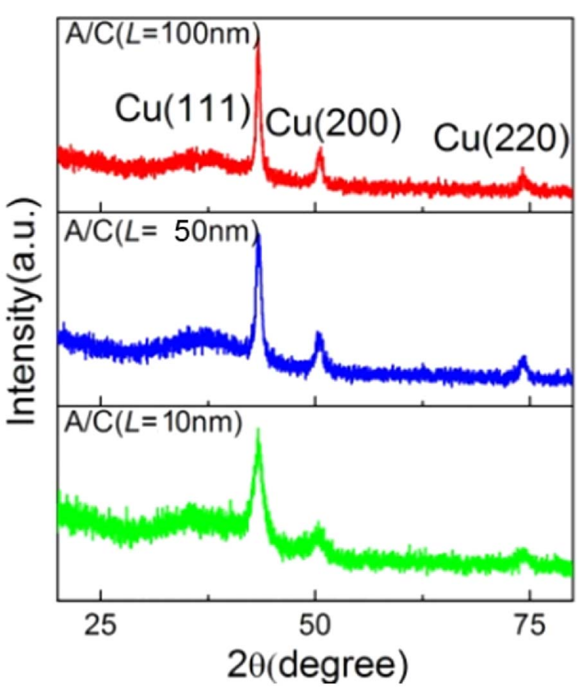

Fig. 1. XRD patterns of CuZr/Cu multilayers and monolithic CuZr films.

3. Results

XRD patterns of the multilayers in Fig. 1 revealed that the amorphous CuZr/nanocrystalline Cu multilayers exhibited Cu (111), Cu (200) and Cu (220) peaks, of which the intensity increased with the modulation period. A hump of amorphous CuZr was observed near the diffraction angle (38°), with a rather low intensity in all the CuZr/Cu multilayers. Selected cross-sectional TEM images of the multilayers with clear modulated layer structures (Fig. 2) indicated columnar grains in crystalline Cu layers and amorphous nature in glassy CuZr layers. Careful microstructure examinations showed that the average grain size of Cu in CuZr/Cu multilayers scaled with individual layer thickness. Specifically, the grain sizes of the Cu layers are 10 nm±3 nm, 45 nm±7 nm and 92 nm±11 nm, for the 10 nm, 50 nm and 100 nm layered multilayers, respectively. These observation indicated that it is reasonable to consider the grain size is nearly equal to the individual layer thickness of Cu constituent layers, consisting with the relation between the individual layer thickness and grain size of amorphous/crystalline multilayers reported in literatures previously [10,12,29–32]. Fig. 3(a) plotted the hardness of the present CuZr/Cu multilayers as a function of individual layer thickness h; and the h (or grain size d) dependent hardness follow the classical Hall-Petch relation except the multilayers with the minimum h(~10 nm) as shown in Fig. 3(b). The hardness changed dramatically from ~3.2 GPa for h=100 nm to ~5.9 GPs for h=10 nm. The non-dimensional strain rate sensitivity index, m, was evaluated as [33–35]:

$$m = \frac{\partial ln\,(H)}{\partial ln\,(\dot{\varepsilon})} \tag{1}$$

where H is the hardness and $\dot{\varepsilon}$ the applied strain rate. As shown in Fig. 4, the strain rate sensitivity of CuZr/Cu multilayers exhibited nearly a constant value of m=0.043 even though the individual layer thickness decreased from 100 nm to 10 nm.

4. Discussion

4.1. Unusual strain rate sensitivity of CuZr/Cu multilayers

As the individual layer thickness was systematically decreased, the nearly unchanged m derived from the present CuZr/Cu multilayers is unusual. Firstly, it has been well established that polycrystalline Cu exhibited enhanced m as its grain size increased, especially within the grain size regime which was enter nanoscale, i.e., less than 100 nm [22,36]. As shown in Fig. 4, the significant increment of m due to grain size refinement of the Cu layers should be compensated by an unknown mechanism, which corresponded to decreasing m with decreasing individual layer thickness, to achieve the nearly constant m for CuZr/Cu. Given the crystalline Cu layers, there were two only possible candidates, i.e., the amorphous CuZr layers and the amorphous/crystalline interfaces (ACIs), which might be responsible for such compensation mechanism(s).

For ACIs, previous studies indicated that heterogeneous interfaces were effective sites for both nucleation and annihilation of dislocations for crystalline constituent layers in amorphous/crystalline multilayers [6,37,38]. Also, interactions between dislocation and grain boundary

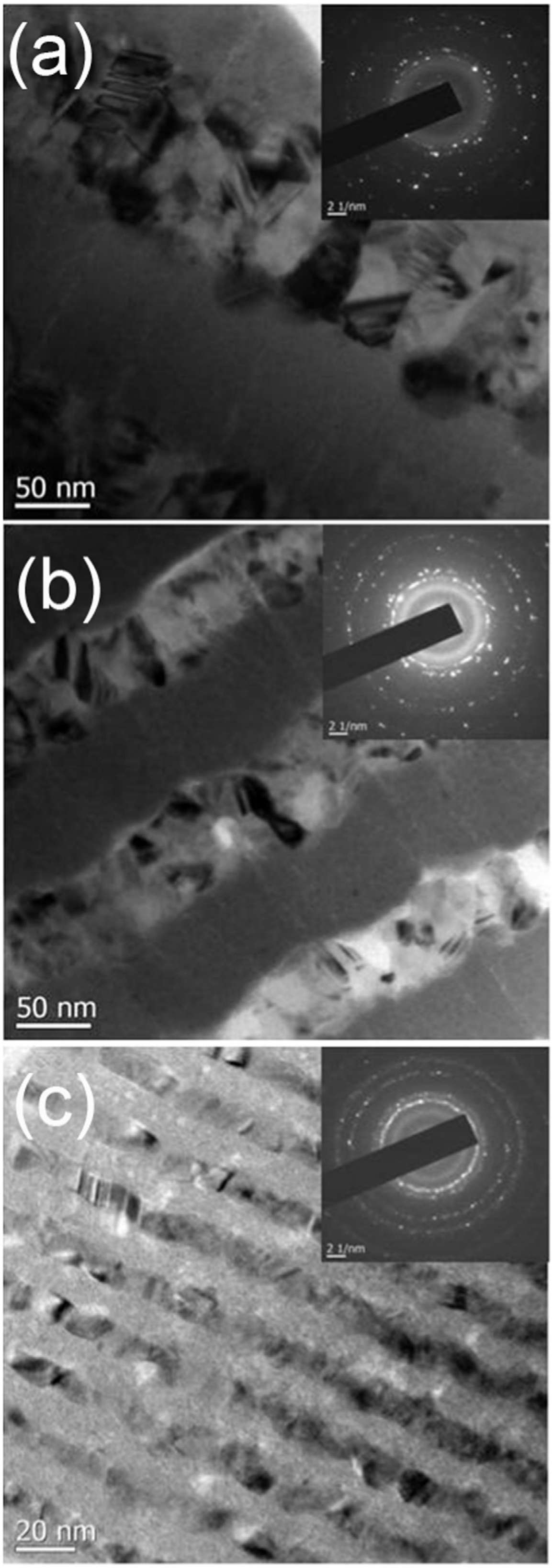

Fig. 2. Representative HRTEM images of (a) 100 nm layered CuZr/Cu multilayers and (b) 10 nm layered CuZr/Cu multilayers.

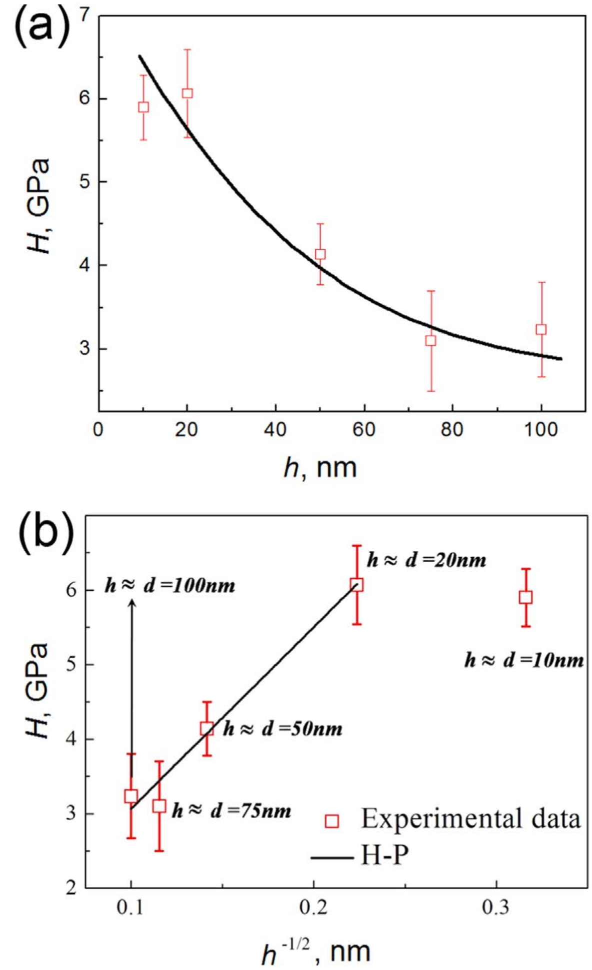

Fig. 3. Hardness plotted as a function of (a) individual layer thickness h and (b) $h^{-1/2}$ for the CuZr/Cu multilayers.

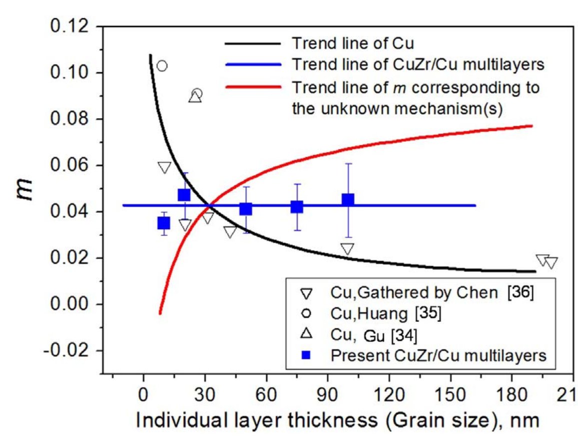

Fig. 4. Dimensionless strain-rate sensitivity, m, plotted as a function of individual layer thickness for CuZr/Cu multilayers, along with data of grain size dependent m for nanocrystalline Cu derived from Refs. [34,35,36]. To achieve the nearly constant m for the present CuZr/Cu multilayers, a unknown mechanism following the red trend line should exist so as to compensate the increased m due to reduced Cu grain size as indicated by the black trend line. Because no existing mechanism(s) could explain the red trend line (as discussed in the text), a new mechanism underlying the strain rate sensitivity behaviors of the confined Cu and CuZr constituent layers was proposed. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

(GB) in nanocrystalline metals could significantly enlarge m for nanocrystalline metals [39]. In this way, ACIs in the present multilayers should act as GBs in nanocrystalline metals, which might increase the strain rate sensitivity of the crystalline Cu layers as a result of enhanced dislocation activities caused by the presence of ACIs. Consequently, the strain rate sensitivity originated from the ACIs should increase as the individual layer thickness was reduced. For the present CuZr/Cu multilayers, this was obviously contrary to the aforementioned compensation mechanism.

For the amorphous CuZr layers, there existed no mechanism that could lead to enhanced m and compensate the significant reduction of m in the crystalline Cu layers as the individual layer thickness was increased from 10 to 100 nm. Interestingly, as shown in Fig. 4, the strain rate sensitivity of CuZr/Cu was even larger than that of crystalline Cu, by comparison with the well-established trend line of m verses grain size for Cu [36,40,41]. This indicated that the amorphous CuZr should exhibit a much higher m than that of crystalline Cu at larger individual layer thicknesses so as to achieve the nearly constant m for CuZr/Cu multilayers. However, this was hardly the case as the value of m for single-phase amorphous CuZr was usually close to zero or even negative.

Above all, at present, the combined mechanisms related to the crystalline Cu, the amorphous CuZr and the ACIs between them could not explain the unusual constant m observed in the present CuZr/Cu multilayers. Therefore, in the sections to follow, other mechanisms that might make the crystalline Cu and amorphous CuZr behave quite differently from their single-phase counterparts were proposed, i.e., the strain rate sensitivity of crystalline Cu and amorphous CuZr confined by each other in CuZr/Cu, and the effects of ACIs on the strain rate sensitivity of the confined Cu and CuZr layers.

4.2. The strain rate sensitivity of Cu confined by amorphous layers

To interpret the nearly constant m of the present multilayers as well as the observation that CuZr/Cu even exhibited a higher m than that of the corresponding Cu layers at large individual layer thicknesses, one should reconsider the m of crystalline Cu layers confined by amorphous CuZr layers, which might not change as significantly as the trend line derived from the single-phase crystalline Cu.

For a nanocrystalline metal having a much higher percentage of GBs compared with its coarse-grained counterparts, GB related mechanisms, e.g., interaction between dislocations and GBs, GB sliding, GB diffusion and GB migration, could be effectively enhanced as the grain size enters nanoscale, say, less than 100 nm [42]. As the grain size was further reduced to ~10 nm or less, dislocation-mediated processes became more difficult or even impossible, and GB self-deformation processes should play a key role [43,44]. Specifically, for face-centered cubic metals such as Cu, the enhanced GB related

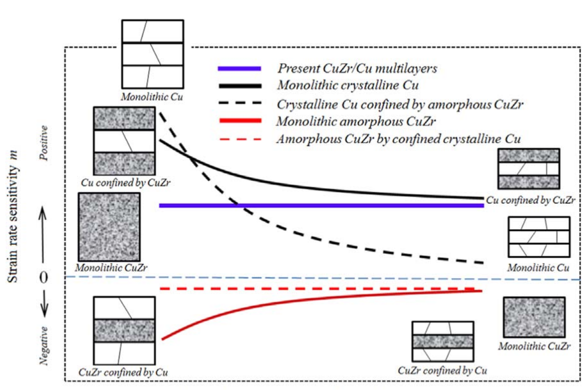

Fig. 5. Schematic illustration of the proposed mechanisms underlying the unusual constant strain rate sensitivity of CuZr/Cu multilayers.

mechanisms within nanoscale grains could significantly increase the strain rate sensitivity as previously reported [36,45]. For nanocrystalline Cu layers inside a CuZr/Cu multilayer, the microstructural circumstances around the Cu grains were changed dramatically.

Consider next the strain rate sensitivity of Cu constituent layers having the smallest individual layer thickness, i.e., 10 nm. Unlike those surrounded by Cu grains in monolithic crystalline Cu, within the present multilayers, Cu grains were confined by amorphous CuZr grains. The heterogeneous ACIs formed between the crystalline Cu and amorphous CuZr could effectively prevent the deformation mechanisms related to high m, such as GB sliding and GB migration. Then, the GB self-deformation within the Cu layers could accommodate plastic deformation only in the direction parallel to the ACIs, resulting in a lower m relative to that of monolithic Cu with identical grain size. It should be mentioned that the ACIs could be the effective sites for nucleation and annihilation of dislocations within Cu grains as pointed by Wang et al. [6], thus causing a higher m than that of the corresponding monolithic Cu. However, the increment of m attributed from the ACIs-associated dislocation mechanism should be much lower than the decrement of m derived from preventing the GB self-deformation mechanism via the ACIs. Therefore, by combining the two effects of the ACIs, the strain rate sensitivity of the Cu layers having the smallest individual layer thickness should be not as high as that derived from the monolithic Cu as previously reported.

In comparison, the strain rate sensitivity of Cu layers having the largest individual layer thickness (i.e., 100 nm) should be higher than that of the monolithic Cu with a grain size of 100 nm, as the presence of ACIs could promote the nucleation and annihilation of dislocations [6], even causing a m higher than that of the CuZr/Cu multilayers. In this way, the present Cu layers might have a trend line of m not as steep as that of the monolithic Cu, increasing mildly as the individual Cu layer thickness was reduced, as shown in Fig. 5.

4.3. The strain rate sensitivity of CuZr confined by crystalline layers

Unlike FCC crystalline metals exhibiting in general a positive and grain-size-dependent m, the hardness/strength derived at various strain rates for MGs generated quite different strain rate sensitivity, as negative, close to zero, or even positive values of m were reported [24,25]. While inconsistence existed in reported absolute values of m, few studies concerned the size-dependent m of MGs [20]. In particular, there existed no study on the strain rate dependent hardness of MG thin films with a film thickness less than 100 nm.

Recently, Wang et al. [20] systematically evaluated the strain rate sensitivity of CuZr/Cu multilayers (bi-layer thickness fixed at 100 nm) having different modulation ratios (ratio of amorphous layer thickness to crystalline layer thickness), ranging from 0.1 to 9.0. For comparison, the size dependent hardness and m of monolithic CuZr amorphous films with film thickness varying from 100 to 3000 nm were also presented [20]. Despite the lacking of experimental data for both hardness and strain rate sensitivity at nanoscale film thicknesses, the fit curves based on the data derived from these films indicated monolithic CuZr amorphous films exhibited size insensitivity in both hardness (4.84 GPa) and m (−0.022) when the film thickness was less than 100 nm [20]. As the layer thickness of CuZr amorphous layers in all the specimens was less than 100 nm, the CuZr layers should have a negative m. The negative m of MGs was consistent with the well-documented relation that a higher strain rate generated more free volumes [46], thus causing softening of the MGs.

Given that the m of Cu constituent layers increased monotonically with decreasing individual layer thickness, the contribution of CuZr layers to m should increase correspondingly so as to achieve the nearly constant m of CuZr/Cu as shown in Fig. 4. At this stage, two scenarios might simultaneously occur in CuZr/Cu subjected to indentation deformation.

Firstly, for a composite with two constituent materials, it has been established that plastic deformation should initiate in the softer material (as the harder one needs a higher stress to yield), serving thus as the subordinate processes upon plastic deformation [15]. For the present CuZr/Cu multilayers, Cu was the soft one and should accommodate more fraction of plastic deformation of the whole multilayer than CuZr. However, the hardness of Cu was strongly size-dependent, increasing dramatically as its grain size was reduced in the range of 10–100 nm [47]. Given the unchanged hardness of CuZr as the individual layer thickness became less than 100 nm [20], the contribution of CuZr to plastic deformation should increase as the Cu layers were strengthened when its individual layer thickness was reduced. In this way, the value of m derived from the CuZr layers (which exhibited a negative m) was increased, even if the strain rate sensitivity of CuZr was constant as proposed by Wang et al. [20]. Then, the increased m due to the refining Cu layers should be compensated by the negative m of CuZr layers by participating a higher fraction of plastic deformation in CuZr/Cu multilayers having smaller individual layer thicknesses.

Secondly, the confining effects of Cu layers might change the strain rate sensitivity of CuZr layers. As discussed above, the hardness of CuZr remained unchanged while the Cu layers were strengthened as the individual layer thickness was reduced. Under such conditions, the stress exerted from the Cu layers onto the CuZr layers should be quite different as the strain rate and/or individual layer thickness were varied [12]. For CuZr/Cu multilayers having smaller individual layer thicknesses, intra-granular stresses in the Cu layers should spread over a wider range, as the m of Cu increased with decreasing grain size. Then, the higher stresses exerted on the CuZr layer nearby could generate more free volume, and vice versa. In this scenario, the strength of CuZr with smaller individual layer thickness might be softened further as the applied strain rate was increased, leading to a more negative m.

The two scenarios as discussed above might operate simultaneously to compensate the increased m due to the adding of Cu layers, thus yielding the nearly constant m of CuZr/Cu multilayers, as sketched in Fig. 5.

4.4. Further remarks

Nanoscale amorphous/crystalline multilayers are complex materials systems. So far information concerning their strain rate sensitivity is limited [19,20]. Among those few studies, interestingly, Guo et al. [19] proposed that the confinement of crystalline layers could alter the velocity of shear bands propagation in the amorphous constituent layers, and the instantaneous velocity of the shear bands decreases at higher Cu layer thicknesses. Furthermore, the velocity of single shear band in amorphous/crystalline multilayers could be different while deformed at different applied strain rates. Note the amorphous/crystalline multilayers used in Ref. [19] were fabricated to be nanopillars, for which an additional extrinsic length scale, i.e., the pillar size, was involved. Then, the microstructural circumstances of the amorphous/crystalline nanopillar multilayers may be quite different from that of the present CuZr/Cu multilayers is some certain ways. Despite that, as shear banding deformation is the main carrier of plastic deformation of amorphous materials, the intrinsic relevance between shear band velocity and the strain rate sensitivity of the amorphous/crystalline multilayers is a crucial issue need to be further explored.

Also, to better understand such composites consisting of amorphous and crystalline layers, systematic experimental studies on the mechanical behavior and microstructural evolution of both constituent layers at nanoscale are needed. Especially for the amorphous one, as deformation mode transition from heterogeneous to homogeneous processes is involved at the length scale concerned, it is even more challenging to interpret its strain rate sensitivity although it is a crucial issue to be addressed in the future. The experimental results of Wang et al. [20] clearly indicated that the strain rate sensitivity of CuZr/Cu multilayers could change in a wide range from negative to positive by reducing the modulation ratio of amorphous layer thickness to crystalline layer thickness. The present work further demonstrated that the strain rate sensitivity of the two constituent layers might exhibit quite different behaviors from those in free standing status, i.e., without mutual confinement effects on each other. These findings pave a new way to interpret the rate-sensitive mechanical properties of amorphous/crystalline multilayers. However, more sophisticated experimental and simulation studies are needed to reveal the deformation mechanism underlying the microstructural evolution of such complex composites materials systems.

5. Conclusion

The strain rate sensitivity and plastic deformation mechanisms of amorphous CuZr /crystalline Cu multilayers were studied by nanoindentation testing at room temperature. Although the individual layer thickness was varied from 10 to 100 nm, nearly constant strain rate sensitivity (m) was achieved in these multilayers. Such unusual rate-sensitive mechanical behavior was interpreted by considering the mutual confining effects of the two constituent layers, which might exhibit quite different strain rate sensitivities compared with those in free standing status. Specifically, the crystalline Cu layers confined by the amorphous CuZr layers showed more gentle increase in strain rate sensitivity as the individual Cu layer thickness was reduced, while the strain rate sensitivity of the confined CuZr layers decreased. Acting altogether, these mechanisms enabled a CuZr/Cu multilayer to exhibit nearly constant strain rate sensitivity as its individual layer thickness was varied at the nanoscale.

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