Hetero-deformation induced (HDI) hardening does not increase linearly with strain gradient


Abstract

Heterostructured metals and alloys have been reported to possess exciting potential of overcoming the strength-ductility tradeoff by introducing high-density of heterostructured domain interfaces [1–13]. These domain interfaces are different from grain boundaries and twin boundaries in conventional homogeneous materials. There are dramatic differences in strength and strain hardening capability across these interfaces [1–3,7–9]. Hetero-deformation caused by this mechanical incompatibility can introduce strain gradient near the interfaces [2–4,8,11]. As suggested by the strain gradient plasticity theory in micromechanics, pileup of geometrically necessary dislocations (GNDs) is needed to accommodate strain gradient near the interfaces of heterostructured domains. Here we report that HDI hardening does not increase linearly with increasing strain gradient in the interface-affected zone. This is because some GND pileups may be absorbed by the interface and consequently does not contribute to HDI hardening with increasing strain gradient. Higher mechanical incompatibility across interface produces higher strain gradient. The strain gradient-dependent strengthening effect of heterostructured interface mainly originates from the development of HDI stress.

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Microscale digital image correlation (μ-DIC) was used to characterize the evolution of strain gradient in the interface-affected zone (IAZ) during tensile testing. The result was compared with the evolution of HDI hardening, which was measured by unloading-reloading approach [2,9,19]. The comparison revealed that the HDI hardening does not have a linear relationship with increasing strain gradient in the IAZ.
Three types of laminate samples stacked with alternate sequences of copper and brass (Cu-10 wt% Zn), copper and brass (Cu-30 wt% Zn), copper and copper were accumulatively roll-bonded (ARB) to a layer thickness of ~62 μm at room temperature. The as-received brass-copper (Cu-10 wt% Zn) laminates were further rolled to a layer thickness of 31 μm, 15 μm, or 7.5 μm. Thereafter, all laminates were annealed at 220 °C for 2 h so that recrystallization occurs in Cu layers but not in Cu—Zn alloy layers. The Cu layers in as-annealed laminates are characterized with fully recrystallized coarse grains (CG), while the Cu—Zn alloy layers exhibit severely deformed nanostructures (NS) [11,12]. For simplicity, three types of laminates were labeled as CG/NS10Zn, CG/NS30Zn, and CG/CG, respectively, in which CG represents coarse-grained Cu layer.

Hardness was measured using an MTS Nanoindenter XP equipped with a Berkovich pyramid indenter. As shown in Fig. 1(a), sharp interfaces with significant hardness incompatibility in adjacent domains exist in heterogeneous CG/NS10Zn and CG/NS30Zn laminates, while no hardness difference across the interfaces in CG/CG sample.

Dog-bone-shaped small sample with the gauge length parallel to the rolling direction was prepared for μ-DIC strain measurement. Refined speckle pattern prepared by electrochemical etching was recorded by secondary electron imaging. The detailed test procedures are reported in our previous work [11]. Fig. 1(b–d) present the distribution of strain εx across interfaces. The strain distribution is averaged along the interface and plotted as a function of distance from an interface (Fig. 1(e)), which provides insights on the statistical nature of strain distribution near the interface. Obviously, interface-affected zones (IAZs) spanning ~10 μm with a negative strain gradient are formed at heterostructured interfaces in CG/NS10Zn and CG/NS30Zn laminates. In contrast, strain εx in CG/CG sample evolves uniformly across the homogeneous interface (the green curve in Fig. 1(e)). Comparing Fig. 1(a) with (e) reveals that the magnitude of strain gradient increases with higher mechanical incompatibility.

The negative gradient of strain εx was caused by the gradient distribution of dynamically generated Frank-Read sources near the heterostructured interface [22]. The mechanical incompatibility leads to synergistic constraint between heterogeneous domains during tension, which changes the local stress state and causes stress concentration near the interface [4,23,24]. The closer to the interface, the higher probability to activate Frank-Read sources because of the higher local stresses. The Frank-Read sources are essentially dislocation segments with various lengths between dislocation jogs, which are formed by the interaction of intersecting dislocations gliding on different slip planes [22,25]. Therefore, it is logical to expect that sites closer to the interface should experience more dislocation activities, and thus have higher measurable strain. Higher mechanical incompatibility induces larger stress gradient near interface, which will cause more intense Frank-Read source gradient. This is the reason why the strain gradient at the CG/NS30Zn interface is much steeper than that at the CG/NS10Zn interface (Fig. 1(a) and (e)).

Fig. 2(a) shows the statistical evolution of strain εx in CG/NS10Zn laminate with increasing applied strain εy. The ratio of the height (H) and the width at half height (W) of strain concentration peak, H/W, can be used to quantify the mean intensity of strain gradient |dεx/dx| in IAZ. As shown in Fig. 2(b), the mean |dεx/db| in the IAZ increases almost linearly with applied strain. In other words, the strain gradient in the IAZ increases linearly with applied strain. As discussed latter, this can be attributed to the formation and deactivation of dislocation pileups near the heterostructured interface.

Since the width of the IAZ is not affected by layer thickness until adjacent IAZs start to overlap [11,12], the volume fraction of IAZ (VIAZ) is primarily determined by the interface density. The VIAZ in the four groups of CG/NS10Zn samples with layer thicknesses of 62 μm, 31 μm, 15 μm and 7.5 μm are approximately 16.8%, 33.6%, 69.5% and 100%, respectively. These samples are used to deduce the strain gradient-related strengthening effects of heterostructured interfaces. Tensile samples with a gauge length of 12 mm and a width of 2 mm were machined for both uniaxial and loading—unloading—reloading (LUR) tensile tests. HDI stress is deduced from hysteresis loops (Fig. 3(a)) using the procedure that was used to measure the “back stress” [2,9]. As shown in Fig. 3(b), the measured HDI stress (σHDI) increases with applied strain, and the sample with higher VIAZ has higher HDI stress.

Note that microscopically plastic strain produced by dislocations is intrinsically heterogeneous in polycrystalline materials [26]. The back

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Fig. 1. (a) Hardness profiles of three types of laminates. Every data point was averaged from 5 nanoindentations. The εx strain maps at the tensile strain of ~10%: (b) CG/NS10Zn, (c) CG/NS30Zn, and (d) CG/CG. In the coordinate, Y is the tensile direction, and X is the sample thickness direction perpendicular to interface. (e) The distribution of statistical average strain εx as a function of distance from the right interface. The white lines in (c) mark the shear banding direction.
stress and forward stress can also be built up in homogeneous materials due to the non-uniform dislocation pileups against grain boundary or dislocation walls [16]. For example, pronounced hysteresis effects were observed in work-hardened polycrystalline and freestanding NS samples [9,16,25]. Therefore, the above measured HDI stress at a certain applied strain should be the total $\sigma_{HDI}$ of laminate sample which can be expressed as

$$\sigma_{HDI} = (V_{NS}\sigma_{HDI,NS} + V_{CG}\sigma_{HDI,CG}) + V_{IAZ}\sigma_{HDI,IAZ}. \tag{1}$$

where $\sigma_{HDI,NS}$ and $\sigma_{HDI,CG}$ are the HDI stress intrinsic to NS and CG components, respectively. $\sigma_{HDI,IAZ}$ represents the HDI stress developed from per unite IAZ volume fraction, i.e., the HDI stress induced by the hetero-deformation in the IAZ. The contribution of these HDI stress components in a laminate can be evaluated basing on the volume fraction of their origin.

Fig. 3(c) shows the fitting of the total $\sigma_{HDI}$ as a function of $V_{IAZ}$ according to the linear relationship described in Eq. (1). As shown, the slop and intercept of the fitting line represent the magnitude of $\sigma_{HDI,IAZ}$ and $V_{IAZ}\sigma_{HDI,NS} + V_{CG}\sigma_{HDI,CG}$ at a certain applied strain, respectively. Fig. 3(d) shows the evolution of the $\sigma_{HDI,IAZ}$ with increasing applied strain. Surprisingly, the $\sigma_{HDI,IAZ}$ increased quickly at the early strain stage, and then slowed down at a tensile strain of ~2%. Note that the derived $\sigma_{HDI,IAZ}$ is the upper limit of HDI stress in a laminate with non-overlapping IAZs. The evolution of $\sigma_{HDI,IAZ}$ with increasing applied strain (Fig. 3(d)) is different from the linear evolution of strain gradient intensity (Fig. 2(b)), which indicates that the HDI stress in the IAZ does not increase linearly with increasing strain gradient in the IAZ. This observation contradicts earlier theory and assumption that the development of HDI stress depends proportionally on the evolution strain gradient [1,11,12,21].

The development of HDI stress from IAZ is caused by the piling up and accumulation of GNDs [18]. Specifically, the spatial gradient of

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**Fig. 2.** Evolution of strain $\varepsilon_x$ in CG/NS$_{10Zn}$ with increasing applied strain $\varepsilon_y$: (a) statistical averaged distribution [11], (b) the mean strain gradient $|d\varepsilon_x/dx|$ in IAZ. The intensity ($H$) and the width at half intensity ($W$) of strain concentration peak at interface are extracted from the Gaussian fitting (red dotted line). Mean $|d\varepsilon_x/dx|$ is approximately equal to the ratio $H/W$.

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**Fig. 3.** Deduction of the HDI stress developed from per unit volume fraction of IAZ ($\sigma_{HDI,IAZ}$). (a) LUR curves and hysteresis loops of CG/NS$_{10Zn}$ laminates with different layer thickness, i.e., CG/NS$_{10Zn}$ samples with varying volume fraction of IAZ ($V_{IAZ}$). (b) Total $\sigma_{HDI}$ of laminates. (c) Linear fitting of the total $\sigma_{HDI}$ as a function of $V_{IAZ}$, showing the deduction of $\sigma_{HDI,IAZ}$ at a certain strain. (d) Evolution of $\sigma_{HDI,IAZ}$ with applied strain.
The HDI stress at a specific position can be in theory calculated by integrating the stress field of each individual GND as \( \sigma_{\text{HDI}} = \nabla \rho_{\text{GND}} \). This expression can be further rephrased as \( \sigma_{\text{HDI}} = \nabla H \eta \) if one assumes that \( \rho_{\text{GND}} \) is proportional to the strain gradient \( \eta \). Accordingly, during straining, the HDI stress caused by GND pileups in local strain gradient zone should develop proportionally with the evolution of strain gradient intensity. In other words, an increase in strain gradient should lead to a constant increase of HDI stress. This is why HDI stress was assumed or hinted to increase linearly with strain gradient development in IAZs at the large plastic strain stage.

The true stress-strain curves of CG/NS10Zn samples are plotted in Fig. 4(a). As shown, the flow stress increases with decreasing layer thickness. The measured flow stress \( (\sigma_f) \) of laminates at a certain applied strain can be expressed as

\[
\sigma_f = (V_{\text{NS}}\sigma_{f,\text{NS}} + V_{\text{CG}}\sigma_{f,\text{CG}}) + V_{\text{IAZ}}\sigma_{f,\text{IAZ}}.
\]

where \( \sigma_{f,\text{NS}} \) and \( \sigma_{f,\text{CG}} \) are the flow stress intrinsic to NS and CG components, respectively. \( \sigma_{f,\text{IAZ}} \) is the flow stress developed from per unit IAZ volume fraction, i.e., the extra strengthening caused by GND pileups in an IAZ. Similar to the fitting process shown in Fig. 3(c), the \( \sigma_{f,\text{IAZ}} \) at certain strain can be extracted by extracting the slope of the linear relationship between \( \sigma_f \) and \( V_{\text{IAZ}} \). The green curve in Fig. 4(b) shows the evolution of \( \sigma_{f,\text{IAZ}} \) with applied strain.

Since the piling up of GNDs in IAZ can also produce extra dislocation strengthening \( (\sigma_{\text{dis,IAZ}}) \), i.e., Taylor strengthening due to the increase of total dislocation density \([14,15]\), the \( \sigma_{f,\text{IAZ}} \) is the sum of \( \sigma_{\text{HDI,IAZ}} \) and \( \sigma_{\text{dis,IAZ}} \):

\[
\sigma_{f,\text{IAZ}} = \sigma_{\text{HDI,IAZ}} + \sigma_{\text{dis,IAZ}}.
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