

# Strain hardening and ductility in a coarse-grain/nanostructure laminate material

X.L. Ma,<sup>a</sup> C.X. Huang,<sup>b,\*</sup> W.Z. Xu,<sup>a</sup> H. Zhou,<sup>a,c</sup> X.L. Wu<sup>d</sup> and Y.T. Zhu<sup>a,e,\*</sup>

<sup>a</sup>Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC 27695, USA

<sup>b</sup>School of Aeronautics and Astronautics, Sichuan University, Chengdu 610065, China

<sup>c</sup>National Engineering Research Center of Light Alloy Net Forming, Shanghai Jiao Tong University, Shanghai 200240, China

<sup>d</sup>State Key Laboratory of Nonlinear Mechanics, Institute of Mechanics, Chinese Academy of Sciences, Beijing 100190, China

<sup>e</sup>School of Materials Science and Engineering, Nanjing University of Science and Technology, Nanjing 210094, China

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A laminate structure with a nanostructured Cu–10Zn layer sandwiched between two coarse-grained Cu layers was produced by high-pressure torsion, rolling and annealing. Sharp interlayer interfaces with sufficient bonding strength were developed. Mechanical incompatibility between different layers during tensile deformation produced high strain hardening, which led to a tensile ductility higher than prediction by the rule-of-mixture. These observations provide insights into the architectural design and deformation studies of materials with gradient and laminate structures. Published by Elsevier Ltd. on behalf of Acta Materialia Inc.

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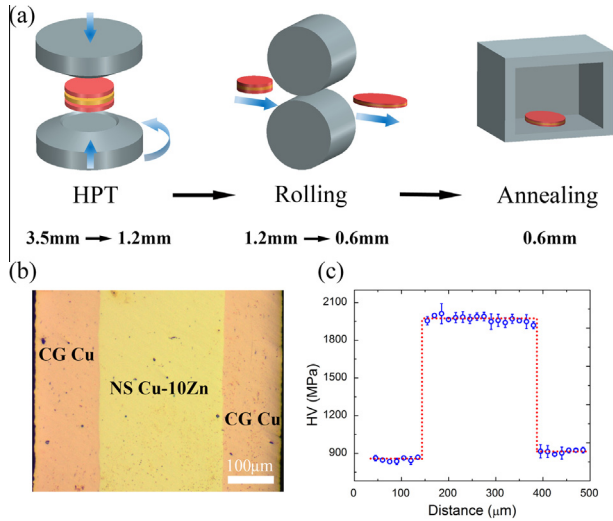
Gradient structures (GS) with a grain-size gradient have been recently introduced to optimize the mechanical properties of structural materials [1–5]. To date, some exceptional combination of enhanced strength and considerable ductility are reported in different material systems with multiscale grain-size structures [6–10], including GS and multi-layered materials. However, the fundamental principles that govern the deformation behaviors of GS are still not fully understood [1,5]. Elastic/plastic interface and stable/unstable interface caused by mechanical incompatibility during deformation have been reported to play a critical role in both strengthening and strain hardening of GS materials [2,5]. However, such interfaces migrate dynamically during the deformation of GS, which makes it hard to perform quantitative postmortem investigation [1,2,11]. In fact, GS can be approximately regarded as the integration of numerous layers and interfaces (laminate structure) [2]. Therefore, there are some similarities in the deformation behaviors of GS and laminate structures. For example, both structures have the mechanical incompatibility during deformation. The advantage of laminate structure is that its interfaces are stationary [12] and can be easily located after the deformation, making it easier

to analyze quantitative mechanics and investigate post-mortem microstructures. Therefore, it might be possible to use laminated (or sandwiched) structures to study some fundamentals in deformation behaviors of GS structure.

The fabrication of laminated nanostructured (NS)/coarse-grained (CG) structures with sharp interfaces is also a challenge since interfacial strength and selective grain refinement are required simultaneously. Here, we fabricated a laminate structure with a NS bronze layer sandwiched between two CG copper layers by utilizing two principles: (a) Different grain refinement effectiveness of Cu and bronze during deformation. It has been shown that the grain size of bronze can be refined much more effectively than that of copper during severe plastic deformation due to its lower stacking-fault energy [13]. (b) Different thermal stabilities of Cu and bronze. Alloying elements are generally effective in pinning grain boundaries and resisting grain growth [14]. Hence, bronze can remain much finer grain size compared to Cu after proper annealing. Using this approach, we can produce a laminate structure with sharp and well-bonded NS/CG interfaces. Another objective of this work is to use NS/CG sandwich to study the effect of grain-size-difference across the interfaces on mechanical behaviors.

Figure 1a schematically illustrates the procedure of sample processing. Commercial Cu (99.9 wt.%) and bronze (Cu–10 wt.%Zn) plates were punched into  $\phi$ -10 mm disks and polished to 3 groups of thickness. The total initial thickness of three disks was around 3.5 mm so that

\* Corresponding authors at: School of Aeronautics and Astronautics, Sichuan University, Chengdu 610065, China (C.X. Huang); Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC 27695, USA (Y.T. Zhu); e-mail addresses: [chxhuang@scu.edu.cn](mailto:chxhuang@scu.edu.cn); [ytzhu@ncsu.edu](mailto:ytzhu@ncsu.edu)



**Figure 1.** (a) Schematic illustration of fabrication of CG/NS/CG sandwich materials. Deformation history of thickness reduction is also provided below the corresponding step. (b) Optical microscopy observation of as-processed sandwiches with NS Cu–10Zn volume fraction 0.47. (c) Vickers micro-hardness indentation (with loading 25 g) on cross-sectional sample in (b).

sufficient thickness reduction ( $\sim 83\%$ ) after processing can be achieved to form strong interfacial bonding (thickness reduction history as shown in Fig. 1a). Mechanical polishing and ultrasonic cleaning were carried out before sandwiching the disks together. High pressure torsion (HPT) was applied at room temperature with 1 GPa for 10 revolutions at 1.5 rpm to obtain more homogeneous deformation along the radius direction [15]. Thereafter, as-HPTed sandwiches were rolled to 0.6 mm from 1.2 mm and then annealed at 240 °C for 2 h.

Microstructures near interfaces were characterized by FEI Quanta 3D FEG with Ion Channeling Contrast Microscopy (ICCM), JEOL-2010F Transmission Electron Microscopy (TEM) operated at 200 kV and Electron Dispersive X-ray Spectroscopy (EDS) mapping in FEI Titan 80-300. Dog-bone shaped samples with gauge dimension of  $0.6 \times 2 \times 8.4 \text{ mm}^3$  were cut from the middle of sandwiches and tested under uniaxial tension at a strain rate of  $9 \times 10^{-4} \text{ s}^{-1}$ . Scanning electron microscopy (SEM) was used to examine fracture surface and interface.

Three groups of samples with varying volume fraction of central NS Cu–10Zn layer (A: 0.10, B: 0.22, C: 0.47) were fabricated. Figure 1b shows a typical optical micrograph (cross-sectional view) of sample C. Different colour contrast clearly indicates three layers with two sharp interfaces. As shown in Figure 1c, microhardness within each layer is rather homogeneous, while there are abrupt transitions at the interfaces, indicating a great difference of yield strength between central and outer layers. These hardness levels in each layer do not change much with variation of volume fractions.

Figure 2a is a channeling contrast image showing the typical microstructures near the interface. On the left side is typical CG Cu with a grain size of  $\sim 4 \mu\text{m}$ , while on the right side is NS bronze with a grain (sub-grain) size of  $\sim 100 \text{ nm}$ . Magnified image of the NS/CG interface is shown in Figure 2b, which reveals a void-free transition from NS bronze to CG Cu. The exact Cu/bronze interface is hard to tell in TEM and can be identified by EDS

mapping. Figure 2c is a typical high-angle annular dark-field (HAADF) image including a Cu/bronze interface, whose precise location is unknown. EDS mappings in Figure 2d and e show the elemental distribution of Cu and Zn and resolve the exact interface (marked by dotted lines) in Figure 2c. Concentrations of Zn are measured as 0.64 wt.% in the left side and 10.22 wt.% in the right side, respectively. This agreement of specific composition in Cu and bronze implies no significant bulk diffusion from each side during sample processing. Therefore, Figure 2c–e confirm the generation of well-bonded and sharp Cu/bronze interface.

Figure 3a shows the tensile stress–strain curves of laminated samples, pure CG Cu and NS bronze samples. Pure samples were made from the sandwiches by polishing off other layers. The yield strengths (0.2%-strain offset stress) for samples A, B and C were measured as 142 MPa, 201 MPa and 266 MPa, respectively, while their uniform elongation (engineering strain) were 27.1%, 19.6% and 12.3%, respectively. The yield strength of the laminate sample increases and its tensile ductility decreases with increasing volume fraction of central NS bronze layer. Note that the uniform plastic deformation of NS bronze layer in sandwich is much higher ( $>12\%$ ) than that ( $\sim 0.7\%$ ) of its pure counterpart. This is because its early necking tendency was constrained by the stable CG Cu layers from both sides via the two interfaces.

In the rule of mixture (ROM) for laminated composite structure, yield strength ( $\sigma_{ys}$ ), strain hardening ( $d\sigma/d\varepsilon$ ) and uniform elongation ( $\varepsilon_{UE}$ ) (true strain) are expressed as [2,16,17]

$$\sigma_{ys} = \sum V_i \sigma'_{i,ys} \quad (1)$$

$$\frac{d\sigma}{d\varepsilon} = \sum V_i \frac{d\sigma_i}{d\varepsilon} \quad (2)$$

$$\varepsilon_{UE} = \frac{\sum V_i \sigma_{i,UE} \varepsilon_{i,UE}}{\sum V_i \sigma_{i,UE}} \quad (3)$$

where  $V_i$  is the volume fraction of component  $i$ ,  $\sigma'_{i,ys}$  is the flow stress of component  $i$  alone at 0.2% plastic strain of the composite sample [2],  $\sigma_i$  is the true stress of component  $i$ ,  $\sigma$  and  $\varepsilon$  represent the true stress and true strain of integrated composite sample,  $\sigma_{i,UE}$ ,  $\varepsilon_{i,UE}$  stand for the true stress and true strain of component  $i$  at necking point.

It is found that the yield strength of laminate samples agrees well with the ROM (Eq. (1)), as shown in Figure 3b. Comparisons of strain hardening and uniform elongation (true strain) between experimental results and theoretical prediction from ROM are shown in Figure 3c and d. When calculating the strain-hardening rate, we assume a constant engineering stress after necking for pure bronze. This assumption provides the maximum applied stress that a standalone bronze layer can sustain according to Considère criterion. Namely, the derived strain-hardening rate is the upper limit based on ROM and would give a conservative comparison to experimental observation. For Eq. (3), it is also assumed that during the uniform elongation, the strain hardening of each component can be expressed by Hollomon law of  $\sigma_i = k_i \varepsilon^{n_i}$  [16]. In fact, the Hollomon law has been used on NS materials in compression test [18,19] despite limit reports of applications for tensile tests due to the plasticity instability. Figure 3c shows that the strain-hardening rate of the

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