

## Deformation induced alloying in crystalline – metallic glass nano-composites

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### ARTICLE INFO

#### Article history:

Received 12 December 2014

Received in revised form

20 January 2015

Accepted 21 January 2015

Available online 31 January 2015

#### Keywords:

Deformation induced alloying

Multilayers

Nanocrystalline

Metallic glass

Shear band

Atom probe tomography

### ABSTRACT

We study the mechanisms of deformation driven chemical mixing in a metallic nanocomposite model system. More specific, we investigate shear banding at the atomic scale in an amorphous CuZr/crystalline Cu nanolaminate, deformed by microindentation. Three CuZr/Cu multilayer systems (100 nm Cu/100 nm CuZr, 50 nm Cu/100 nm CuZr, and 10 nm Cu/100 nm CuZr) are fabricated to study the effect of layer thickness on shear band formation and deformation induced alloying. The chemical and structural evolution at different strain levels are traced by atom probe tomography and transmission electron microscopy combined with nano-beam diffraction mapping. The initially pure crystalline Cu and amorphous CuZr layers chemically mix by cross-phase shear banding after reaching a critical layer thickness. The Cu inside the shear bands develops a high dislocation density and can locally undergo transition to an amorphous state when sheared and mixed. We conclude that the severe deformation in the shear bands in the amorphous layer squeeze Zr atoms into the Cu dislocation cores in the Cu layers (thickness < 5 nm), resulting in local chemical mixing.

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### 1. Introduction

Nanometer scale amorphous/nanocrystalline (am/nc) multilayers [1–6] can simultaneously possess high strength and ductility that cannot be achieved by the constituent individual bulk materials alone [7,8]. The mechanical performance of such nano-sized layered structures is influenced by the internal interfaces of the crystalline layer (e.g. grain boundaries [9], twin boundaries [10]), but more importantly, governed by the hetero-interfaces [11,12]. On the one hand, by decreasing the thickness of the nanocrystalline layer, dislocation slip confinement can result in increased strength [11,13]. On the other hand, a further decrease of the nanocrystalline layer thickness reduces its dislocation storage capacity, and facilitates the cross-phase interface penetration of shear bands stemming from the amorphous layer. Shear bands in amorphous materials are accompanied by strain softening or even catastrophic failure. Recent works have shown that shear bands starting in the amorphous layers of am/nc nanolaminates can be arrested by the presence of nanocrystalline layers [5,6,12].

However, the influence of the thickness of the nanocrystalline layers on the structural and chemical properties of these shear bands is not well understood.

A more detailed analysis of the shear bands may enable controlling these strengthening/weakening processes, hence, guiding the design of such composite materials. Several works addressed certain features of shear bands in both, crystalline and amorphous materials. For example, in crystalline and nanocrystalline multilayers, the texture evolution and high density of dislocations [14–16] inside the shear bands indicate a crystallographically mediated process. Other features, such as amorphization [17] and chemical intermixing [18] were also observed in crystalline multilayers. In amorphous materials, nanocrystallization [19] may occur along the path of non-crystallographic [20] shear bands. There is less understanding though on the interplay among the shear bands of these two different phases. Here, we study the composition and structural changes occurring along the pathway of cross-phase shear bands in nano-structured multilayers.

The analysis also aims at better understanding the mechanism of deformation-induced alloying. In severe plastic deformation (SPD), the microstructure for each bulk metallic phase is firstly refined into a laminate structure by deformation and then further evolved into a chemically mixed structure upon further deformation [21]. However,

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since the deformed microstructure is complex and heterogeneous, it is very challenging to trace the structural evolution and the exact physical mechanism for the deformation induced alloying process. Several kinetic and thermodynamic explanations for chemical mixing of crystalline/crystalline bulk composites after SPD have been proposed. Previous experimental and theoretical works on crystalline/crystalline metallic layered structures [22,23], such as Cu–Ag [17,24,25], Cu–V [26], Cu–Nb–Ag [27] and Al–Ni [28], demonstrate in part that chemical intermixing occurs in a localized fashion via shear bands as a dislocation-mediated process. Consequently, it was observed that a critical strain is required to chemically mix heterogeneous crystalline solids [23].

Other explanations for deformation-driven intermixing, e.g. thermodynamic reasoning based on the Gibbs–Thomson effect [29] or kinetic reasoning based on vacancy-assisted diffusion [30], were also discussed.

Thus, we suggest that studying deformed amorphous/crystalline multilayers with well-defined initial thickness and composition may help identifying the underlying mixing mechanisms. On the one hand the analysis of cross-phase shear bands in well-defined multilayers provides a more precise reference state compared to metallurgically synthesized materials. On the other hand the amorphous layer may act as a “temperature tracer”, since any dissipative temperature increase surrounding the shear band would induce (detectable) crystallization. The initially uniform layer thickness allows us to exactly trace the local strain evolution by simply comparing the layer morphology, e.g. the offset, before and after deformation. Finally, atom probe tomography, when applied to such deformation-driven mixing phenomena, serves as a chemical tracer analysis device, revealing exactly which atoms have crossed the interface and where they were deposited [18,31].

In the present work, we fabricate amorphous CuZr/nanocrystalline Cu multilayered films with different initial Cu layer thicknesses. We then deform these samples by microindentation and use atom probe tomography (APT) and transmission electron microscopy (TEM) to map the local chemical and structural features associated with the co-deformation, specifically with the shear bands. Finally, we discuss the mechanism of deformation-induced alloying and the cause for the observed structural evolution.

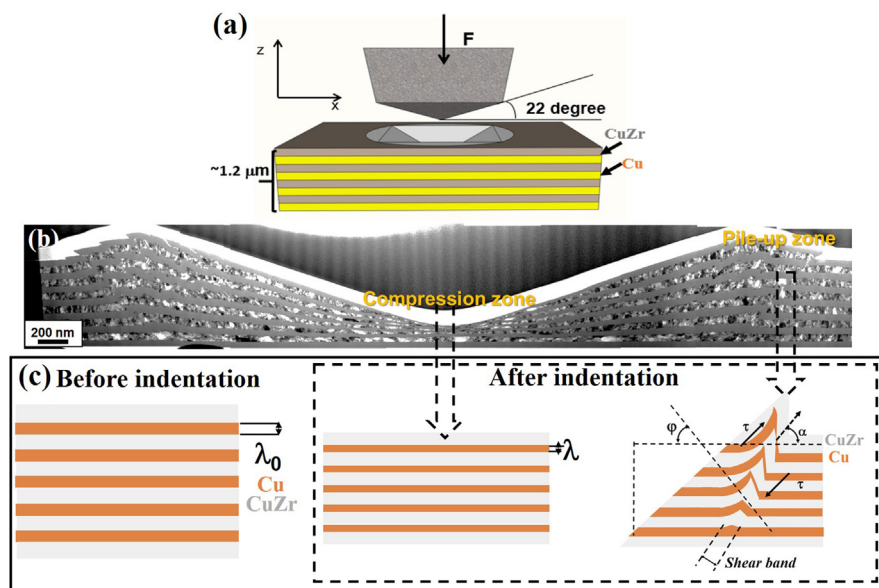
## 2. Experimental procedure

### 2.1. Materials synthesis

CuZr/Cu nanolaminates were deposited on Si (100) wafers by direct current magnetron sputtering at room temperature in a set-up described previously [32]. Cu (99.99 at%) and Zr (99.99 at%) targets were utilized to deposit alternating CuZr and Cu layers. The base pressure was  $4 \times 10^{-5}$  Pa or lower and the deposition pressure was  $4 \times 10^{-1}$  Pa using Ar (99.999%) gas. To deposit the CuZr layers, Cu and Zr targets were co-sputtered for 600 s at target power densities of 1.25 W/cm<sup>2</sup> and 5.8 W/cm<sup>2</sup>, respectively. For deposition of the Cu layers a shutter was placed in front of the Zr target. The deposition time for the Cu layers was 90, 450, and 900 s, respectively, producing three different multilayer samples with varying Cu layer thicknesses. The nominal CuZr layer thickness was  $\sim 100$  nm, and that of the Cu layers  $\sim 10$ ,  $\sim 50$ , and  $\sim 100$  nm, respectively. In the following these samples are referred to as MS10, MS50 and MS100, respectively. The CuZr/Cu multilayer stacks were synthesized with a total thickness of  $\sim 1200$  nm. APT analyses of as-deposited samples suggest that the laminates contain 0.8–1 at% impurity oxygen content incorporated during thin film growth from residual gas [33].

### 2.2. Microindentation and strain estimation

Fig. 1 illustrates the sample deformation. Each of the three CuZr/Cu multilayered samples was probed by a Vickers indenter using three different loads, i.e. 10, 25 and 50 g (Fig. 1a). The holding time at maximum load was 5 s. After indentation, the layers show different thinning behavior in different regions around the indent (Fig. 1b). Underneath the indenter tip, the layers are rather uniformly compressed. Underneath and next to the sides of the tool, pile-up zones with shear bands developed. Fig. 1c schematically shows the different regions after indentation. During loading, the center of the deformed region is primarily exposed to a compressive strain. The (true) compressive strain, i.e. the thickness reduction of the Cu layer within this compression zone can be



**Fig. 1.** (a) Schematic illustration of Vickers indentation, (b) Representative TEM cross-sectional view of indented 100 nm CuZr/100 nm Cu (MS100) multilayer and (c) Schematic illustration of thickness variation after indentation at different regions.

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