

# Large strain deformation of bimodal layer thickness Cu/Nb nanolamellar composites

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## ABSTRACT

Nanolayered composites have garnered much attention due to their ability to withstand deformation to large strains, shock deformation, and irradiation induced microstructural damage. These behaviors have been attributed to high densities of bimetal interfacial content. Although they exhibit yield strengths approaching theoretical limits, multilayered materials with layer thicknesses less than 10 nm have shown limited ductility in rolling. In this study, bimodal 4 nm/40 nm Cu/Nb multilayers are rolled to 30% thickness reduction without the onset of shear instability. The stacking order used allows focus to be drawn specifically to the ductility by the boundary crossing mechanism exhibited in multilayered materials with layer thicknesses below 10 nm. Through the geometric constraint offered by alternating 4 nm and 40 nm layer thickness modes, the onset of localized shear is avoided and the 4 nm layers can be rolled to large strains.

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## 1. Introduction

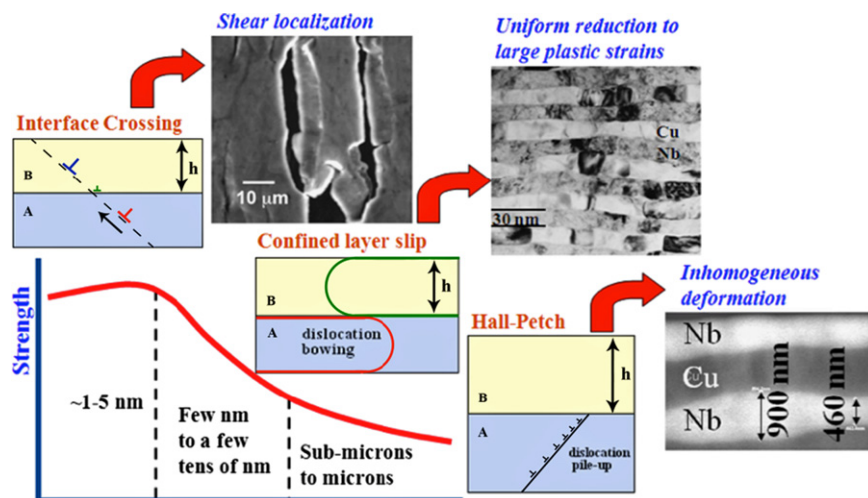
Nanolamellar composites have attracted much attention over the past decade due to their ultrahigh strengths [1,2], deformability [3], fatigue resistance [4], and radiation tolerance [5]. By reducing layer thicknesses to the nanometer scale, many of these materials are approaching the theoretical strength limits of their constituents, while maintaining a high degree of deformability [6–9]. More specifically, Cu/Nb multilayers provide favorable qualities that make them ideal candidates for investigations into interface physics: immiscibility of Cu and Nb at room temperature, formation of a well-characterized semi-coherent interface, and the pre-existence of suitable interatomic potentials for atomistic modeling [10]. When synthesized by magnetron sputtering, a semi-coherent, energetically favorable interface will form between {111} Cu and {110} Nb planes, with  $\langle 1\bar{1}0 \rangle$  Cu ||  $\langle 1\bar{1}1 \rangle$  Nb, and a lattice mismatch of 10.5%. This interface plane also defines the orientation relationship (OR) known as the Kurdjumov–Sachs (KS) OR, which is very common in fcc–bcc systems. The existence of this OR/interface plane allows experimental data to guide and verify models in development [7,11].

Previous work on the Cu/Nb system has mostly been performed in single bilayer thickness mode scenarios, i.e. one repeating bilayer thickness throughout the material. These studies using

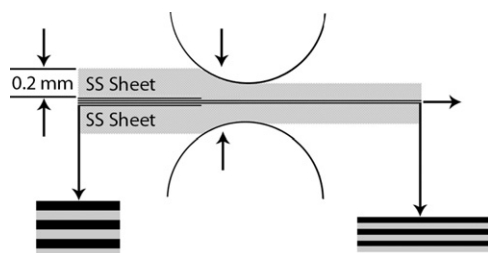
nanoindentation, rolling, tensile testing, and micropillar compression exhibit various deformation behaviors throughout a range of layer thicknesses [12–14]. Nanoindentation results show that hardness increases with decreasing layer thickness only to a certain point, depending on the constituents [15]. Through all systems, hardness increases down to layer thicknesses of approximately 50 nm, following the Hall–Petch relationship ( $\sigma = \sigma_0 + kd^{-1/2}$ , where  $d$  is layer thickness). Below 50 nm and depending on the system being tested, the material will deform by the confined layer slip (CLS) mechanism to a thickness of a few nm. In the CLS regime, dislocations behave according to the Orowan bowing mechanism, with the interfaces acting as barriers to dislocation transmission across interfaces; the result is an evenly distributed array of dislocation loops that accommodate symmetric slip in both constituents during deformation to large strains [16–19]. Below this layer thickness, the motion of single dislocations across the interfaces is the dominant deformation mechanism [20]. These length scale-dependent mechanisms correspond to results of rolling experiments (Fig. 1) [21].

Ductility of Cu/Nb nanocomposites was explored in a series of rolling experiments by Misra and Hoagland [8] and Misra et al. [12]. In Cu/Nb multilayers with layer thicknesses above a few hundreds of nanometers, layers deform heterogeneously across the sample—a result of the formation of dislocation networks in Nb and sub-grain cell structures in Cu, as well as the stochastic nature of slip in coarse-grained materials. At this length scale the layers experience a change in crystallographic texture; individual layers evolve their respective bulk rolling textures, no longer

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**Fig. 1.** Dislocation mechanisms and rolling results under various layer thickness regimes. Materials with sub-micron layer thicknesses and higher deformation as bulk constituents by the Hall–Petch mechanism. When layer thickness ranges from a few to tens of nm, the CLS mechanism is active, allowing uniform layer thickness reduction. Below layer thickness of a few nm, the energy for dislocation bowing is exceeded and dislocations pass directly through interfaces, subjecting layers to shear localization. [8].



**Fig. 2.** Schematic of experimental rolling setup used in this study and prior rolling studies [8]. Stainless steel was used to accommodate the thin film specimens.

exhibiting the KS OR after deformation to large strains. Other experiments, where Cu/Nb multilayers of initial layer thicknesses from 32 to 75 nm were rolled between stainless steel sheets, all to a final layer thickness of 30 nm, resulted in homogeneous deformation throughout the thickness of the layers. This experimental setup, also used in this study, is shown in Fig. 2. These samples experienced over 50% thickness reduction without exhibiting signs of localized shear or texture change. Both results are attributed to the inherent symmetric slip present in CLS materials; the evenly distributed dislocation distribution in this deformation mode allows for homogeneous deformation to large strain while maintaining texture. When the starting layer thickness was 4 nm, the rolled sample exhibited through-thickness cracking (Fig. 1) after rolling to a reduction of only a few percent in thickness. At this layer thickness regime, slip transmission becomes more favorable than confined layer slip, and dislocations pass directly through the interface. A bimodal sample was tested in rolling as well; the material consisted of alternating bilayers of Cu/Nb with 40 nm and 4 nm layer thicknesses. This sample was rolled to 20% reduction and showed no shear cracking. Cu/Nb bilayers of both thicknesses co-deformed without decohesion [8]. It must be noted that each 4 nm bilayer was between two 40 nm bilayers, and ductility in the thinner layers may be attributed to the presence of CLS adjacent to each bilayer surface. As a result, the ability of the interface crossing mechanism to accommodate deformation to large strains in regions without CLS interactions is not clearly demonstrated.

Tensile tests, performed on 40 nm Cu/Nb multilayers with tensile direction parallel to the interface planes, directly revealed the onset of failure as well as the ductility of the multilayers.

Results of tensile testing show considerable work hardening with ductile failure occurring at a flow stress of 1.55 GPa and a strain of 3.4%. This flow stress value closely matches the flow stress determined by nanoindentation using the H/2.7 correlation factor. The limited ductility exhibited was a product of the Considère's criterion, a result of geometric instability [13].

Cu/Nb multilayers were also tested in compression using a micropillar compression technique [22,23]. These micropillars were 4  $\mu$ m in diameter by 8  $\mu$ m in height, oriented with the pillar axis in the growth direction of the multilayer. Results of micropillar compression tests showed significant deformability of both 5 nm and 40 nm layer thickness Cu/Nb in compression. In fact, the 5 nm material deformed to nearly 30% thickness reduction before failing by shear instability, again showing that the material is capable of deformation to large strains under geometries not limited by Considère's criterion. TEM cross-sections of these pillars, in addition to regions below indents, were extracted using a Focused Ion Beam (FIB). These cross-sections showed no signs of through-thickness cracking of layers beneath the deformed regions, elucidating the continuity of individual layers through the thickness of the material and, hence, the ductile nature of these materials with layer thicknesses of a few nanometers [14,21].

Despite localized shear cracking in the case of 4 nm layer thickness Cu/Nb in rolling, this material exhibits significant deformability as shown by TEM analysis of compressed micropillars and regions beneath nanoindents. This leads to the hypothesis that the Cu/Nb multilayers with layer thicknesses of a few nanometers are limited in their ductility only by the onset of geometric instability. These instabilities may be caused by the stochastic nature with which materials deform by the Hall–Petch mechanism, as illustrated in Fig. 1; the rough features that form at the surface of the stainless steel cladding create points of stress concentration at the surface that result in through-thickness cracking of the 4 nm layer thickness material [8]. We introduce a high strength multilayer of layer thickness where CLS is the predominant deformation mechanism in contact with the stainless steel envelope, utilizing the material's inherent ductility facilitated via the dominant symmetric slip mechanism. Localized stress from the stainless steel cladding is distributed over a large volume, and shear cracking may be avoided. This geometry, as well as the “higher” or “increased” number of layers deposited at each thickness mode, will allow clear determination of the

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