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Deformation behavior of a NiCo multilayer with a modulated grain size distribution

Matthew Daly [a], Jonathan Lawrence McCrea [b], Brandon Andrew Bouwhuis [b], Chandra Veer Singh [a], Glenn David Hibbard [a,*]

[a] *Department of Materials Science and Engineering, University of Toronto, 184 College Street, Suite 140, Toronto, Ontario, Canada M5S 3E4*
[b] *Integran Technologies Inc., 6300 Northam Drive, Mississauga, Ontario, Canada L4V 1H7*

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ABSTRACT

In order to gain a fundamental understanding of the deformation behavior of multilayer architectures composed of a periodic layering of hard and soft structural features, NiCo samples possessing modulated nanocrystalline and coarse-grained microstructures were fabricated using pulsed electrodeposition. Scanning electron microscopy imaging confirmed that the samples possessed an alternating grain size distribution and the desired 1:1 thickness ratio of constitutive layers. Uniaxial tensile testing showed a rule of mixtures relationship in yield and ultimate tensile strengths for the multilayer with respect to reference samples. Notably, an increase in elongation to failure was observed in the multilayer when compared to the nanocrystalline reference. The gains in elongation were found to be associated with improved neck stability due to dislocation activity within the coarse-grained layers. The morphology of the fracture surface, considered together with the available mechanical data, electron micrographs and texture measurements, forms the basis of a multiscale deformation mechanism which describes a failure pathway for multilayer architectures with nanocrystalline and coarse-grained structural features.

1. Introduction

Multilayers (ML)s are a layering of alternating material structures and represent an architecture which may be engineered to improve the relative mechanical properties of the constituent material components [1–4]. In traditional ML architectures, immiscible metallic species are selected for layering such that geometric confinement imposed by layer interfaces is augmented by lattice misfit between constituents, leading to increases in interfacial barrier strength [5,6]. The layer thicknesses may also be controlled in order to encourage specific dislocation-based strengthening mechanisms. For example, experimental and atomistic simulations have shown that as layer thicknesses reach into the nanometer regime, interfacial confinement effects suppress traditional Hall–Petch hardening and dislocation pile-up phenomena [7–9]. At these length scales, conventional dislocation-based plasticity is supplanted by dislocation confinement and transmission within individual layers, leading to significant gains in mechanical strength that exceed Hall–Petch predictions [7–9]. Once layer thicknesses approach sizes comparable to a dislocation core (~1–2 nm), interfacial barrier strength has been shown to reduce, promoting interlayer dislocation transmission and, consequently, a reversal of strength improvements from geometric scaling effects [10].

Although the presence of nanoscale geometric features in metallic MLs has led to impressive improvements in strength, as discussed by Anderson et al. [2], confinement from a high density of interfacial misfit dislocations may lead to notable decreases in ductility and a ductile–brittle transition in fracture behavior. For example, uniaxial tensile tests of Cu/Ag MLs show a two-fold decrease in elongation to failure at layer thicknesses of less than 40 nm, when compared against monolithic samples of the same materials [11]. A similar finding was echoed in a tensile investigation of Cr/Cu MLs [12], where the dislocation storing capability of the relatively ductile Cu constituent becomes compromised at layer thicknesses less than 100 nm. In nanoscale Cu/Au and Cr/Cu MLs, these reductions in elongation to failure manifest macroscopically as fracture surfaces exhibiting brittle failure [13]. Ultimately, lattice mismatch at interfacial structures in traditional MLs are generally detrimental to plastic flow, making preservation of the intrinsic ductility of such material architectures problematic. Additionally, in the case of hard-soft nanostructured MLs such as Cr/Cu, material elongation is further handicapped by a significant decrease in the dislocation storage capability of the Cu layer at nanometer thicknesses [13]. In a notable exception to this property trend, Mara et al. [14] have reported a measurement of ultra-high strength and a ductility of greater than

* Corresponding author.
E-mail address: glenn.hibbard@utoronto.ca (G.D. Hibbard).

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0.25 true strain in 5 nm Cu/Nb ML pillars under compressive loading. It is unclear from this study, however, if similar strains may be achieved under tensile loading. From a mechanics perspective, the direction of loading relative to the ML stacking direction is also an important consideration. In this work, the referenced literature refers to loadings directed perpendicular to the layering direction in the ML.

The effect of interfaces on strengthening and elongation to failure in MLs may be considered in the greater realm of nanostructured materials. Despite impressive Hall–Petch strengthening properties [15,16], bulk nanocrystalline metals have also typically suffered from reduced ductility relative to their polycrystalline analogs due to early plastic instability during tensile loading [17,18]. Recently, monolithic alloys possessing hybrid microstructures composed of a bimodal distribution of nanocrystalline (NC) and coarse-grained (CG) crystals have been suggested as material architectures possessing both high strength and good ductility [19–22]. This concept of a bimodal grain size distribution may be adopted into the ML architecture, permitting a modulated microstructure with a low interfacial threshold to dislocation mobility. In comparison to traditional MLs, a NC–CG architecture is expected to significantly reduce interfacial lattice misfit and encourage dislocation activity between constituent layers, thereby providing an avenue for assessment of deformation evolution in modulated microstructures. A recent study from Kurmanaeva et al. [23] examined the mechanical properties of a NiFe ML. Compared to a NC reference, however, the ductility of the ML was reported to be in fact slightly reduced. This surprising finding was found to be caused by the combination of FCC and BCC phases in the sample microstructures [23], further implicating the influence of lattice misfit on sample ductility. This result underscores the need for a monophase material, from which the impact of grain fidelity modulation and implementation of the ML architecture may be directly assessed.

In an effort to understand the intrinsic deformation behavior of ML structures comprised of hard and soft features, the current study investigates the mechanical properties of a $Ni_{80}Co$ (wt%) ML with an alternating grain size distribution. The NiCo system was selected for study as the metallic species exhibit solid solubility in a FCC unit cell for material chemistries greater than 40 wt% Ni [24] and NC NiCo is known to possess enhanced thermal stability [25]. The purpose of this study is two-fold: firstly, improvements in material plasticity achieved from a combination of NC and CG microstructures are examined; secondly, the unique architecture of the NiCo ML offers the opportunity to study the deformation behavior of relatively soft coarse-grained microstructures confined by hard nanocrystalline structural features. Due to its monolithic construction, deformation may proceed in the hard–soft ML without inhibition to dislocation activity from interfacial lattice misfit. In an effort to rigorously assess the deformation behavior of the ML architecture, microscale architectural features and plasticity must be considered. Therefore the current study investigates both micro and macroscale deformation events in order to provide a comprehensive multiscale characterization of the mechanical response of MLs with modulated microstructures.

2. Methods and materials

Free-standing NiCo plates were fabricated at Integran Technologies Inc. using pulsed electrodeposition (PED) on an inert stainless steel cathode from a proprietary electroplating solution and process. This procedure is known to create high quality samples and the PED process is described in greater detail in [26–28]. CG and NC layers were deposited by modifying the cathodic pulse shape to achieve the desired microstructure. The PED parameters were varied to create a ML with a 1:1 CG–NC thickness ratio. Bulk CG and NC sheets were also fabricated to serve as a comparative reference for mechanical testing. Bath chemistries were maintained to produce a nominal $Ni_{80}Co$ (wt%) alloy composition. Following PED, the sheets were mechanically stripped from the stainless steel cathode. Tensile coupons with a 100 mm length, 10 mm grip width, 3.5 mm grip fillet radius, and a nominal gauge dimension measuring 33 mm long (L_o) by 3 mm wide (W_o) were cut from the bulk sheets using a waterjet. The resulting coupons were deburred and ground to at least a grit of 600 with SiC abrasive papers to remove any obvious surface flaws from the gauge length. The coupons all possessed thicknesses (t_o) ranging from 0.5 to 0.7 mm. A small taper was present in the tensile coupon cross-sections due to the waterjetting process, creating a slightly trapezoidal gauge area. All measurements reported in this study use the caliper dimension for calculation where applicable. Deviations in nominal measurements due to tapering of the gauge area are rather low and were measured to be in the range of 3–5% for the specimens examined in this study. It should be noted that the effects of this measurement error did not alter the statistical significance of the presented data.

The as-deposited microstructure of the NiCo ML was imaged using scanning electron microscopy (SEM) and PED alloy chemistries were confirmed with energy dispersive X-ray (EDX) analysis using a Hitachi SU 3500 instrument. For SEM imaging, samples were progressively ground to a grit of 1200 and then polished using 50 nm colloidal silica suspension. Vibratory polishing in colloidal silica suspension was then performed for 4 h as the final polishing step. Colloidal silica residue was removed through Ar ion milling at a 6 kV accelerating voltage with a gracing angle of 8° for 10 min. At low magnification (Fig. 1a), it can be seen that the NC and CG layers appeared to have a near equal thickness throughout the full sample cross-section. The orientation of the tensile coupon dimensions relative to the features of the ML are indicated in the figure. At higher magnifications (Fig. 1b and c), a clear interface between NC and CG layers was observed, and the elongated morphology of the CG microstructure became evident. Based on the collected micrographs, the CG and NC layers were measured to be in the range of 5–10 μm thick. An asymmetric grain morphology associated with the PED growth direction (image down) of the CG layer was visible at high magnification (Fig. 1c). The facets of the terminal structures created a characteristic roughness in the ML interface on the order of 1–2 μm, which has been previously observed in Fe MLs [29]. EDX analysis confirmed that the PED sample chemistries were near target, with Co compositions of 19, 17, and 20 wt% measured for the CG, ML, and NC samples, respectively. Note that previous studies of NiCo manufactured by a similar PED procedure report sulphur and carbon concentrations on the order of several hundred ppm [25].

The texture and the grain size of the CG layers were characterized using electron backscatter diffraction (EBSD) mapping. EBSD maps were collected with a step size of 300 nm and an indexing of over 85% was achieved. Due to the high number of interfaces in the PED samples, a conservative interpolation algorithm was utilized to smoothen EBSD results, which had a negligible impact on the orientation distribution of the collected measurements. According to the collected EBSD maps (Fig. 2), the morphology of the CG layers was columnar, with major and minor grain axis measurements in the range of approximately 5–8 and 3–5 μm, respectively. For EBSD analysis, grain boundaries were defined as adjacent regions with a greater than 10° misorientation. Fig. 2a and b presents the sample texture in the t_o (growth orientation) and L_o directions and the corresponding orientation distribution functions of the collected EBSD data is provided in Fig. 2c and d, respectively. According to the EBSD texture data, the CG layer possess a strong ⟨100⟩ fiber texture along the growth orientation (Fig. 2c), which is common in PED materials [30] and was reported in NiFe MLs [23]. Given this orientation restriction, the EBSD pole figures along the loading axis (L_o)