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Deformation behavior of multilayered NiFe with bimodal grain size distribution at room and elevated temperature

Jochen Fiebig [a,*], Jie Jian [b], Lilia Kurmanaeva [a], Jon McCrea [c], Haiyan Wang [b], Enrique Lavernia [a,d], Amiya Mukherjee [a]

[a] Department of Chemical Engineering and Materials Science, University of California, Davis, CA 95817, USA
[b] Department of Electrical and Computer Engineering, Texas A&M University, College Station, TX 77843-3128, USA
[c] Integran Technologies Inc., Toronto, Canada
[d] Department of Chemical Engineering and Materials Science, University of California, Irvine, CA 92697, USA

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ABSTRACT

We describe a study of the temperature dependent deformation behavior of a multilayered NiFe-60 wt% Fe alloy with a layer thickness of 5 μm fabricated by electrodeposition. The structure of adjacent layers alternates between a nanocrystalline and a coarse grained. Uniaxial tensile tests at temperature between 20 °C and 400 °C and strain rate of 10^{-4}–10^{-2} were used to determine the mechanical behavior. Microstructure observations via transmission electron microscopy and fractography were performed to provide insight into the underlying deformation mechanism. The mechanical behavior is discussed in the context of the bimodal microstructure of multilayered samples and the contribution of each sub-layer to strength and ductility. The results reveal that even at higher temperatures the nanocrystalline layer determines the mechanical performance of multilayered materials.

1. Introduction

Multilayered (ML) materials are structures consisting of alternating layers, usually of different materials, that are designed to improve the properties compared to those of the individual components. An original study by Koehler [1] focused on improving the strength of ML materials and is based on the different line energies of the dislocations in the individual sub-layers. The mechanical properties of several systems (e.g. Cu/Ni [2–4], Cu/Nb [3,4] or Cu/Cr [3,4]) have been investigated to understand the deformation mechanisms in ML structures. The thickness of the individual layers was varied in these experimental studies [2–4] down to a few nanometers (e.g. 2–100 nm for Cu/Ni [2]), and an inverse Hall–Petch behavior, similar to that reported for nanocrystalline materials [5,6], was observed for Cu/Ni. Interestingly, all these works concluded that the influence of the interlayer interface on the mobility of dislocations is the key to understand the observed mechanical properties. To that effect, Misra and Wang published an overview of the influence of the layer thickness on the deformation mechanisms in ML structures [7]. In the range of layer thickness from tens of micron down to the submicron (≈ 50–100 nm [7]) the strength of multilayered materials is controlled by dislocation pile-ups at the interlayer interphases similar to the Hall–Petch relationship [8, 9] in conventional polycrystalline materials. The strength of the ML structure increases proportional to the inverse square root of the layer thickness. As soon as the layer thickness decreases below approximately 100 nm the concept of the so-called confined layer slip has been proposed [10,11]. In this case, a single dislocation loop moves parallel to the interface as demonstrated during in situ indentation of Cu/Nb with a layer thickness of 20 nm for Nb and 30 nm for Cu [12]. Yet, if the layer thickness decreases to a few nanometers (2–5 nm) experimental results indicated that the crossing of individual dislocations through the interlayer interface becomes the dominant deformation mechanism [13]. Further theoretical studies on this transition from multiple to single dislocation regime have been done by Misra et al. [14] and Fang and Friedman [15]. Misra et al. investigated a deformation map for ML materials with layer thickness and columnar grains size (grain boundaries are perpendicular to the interlayer interface) as variables to determine the dislocation activity. Depending on several parameters (Burgers vector, misfit of the adjacent layers and Poisson ratio of the softer layer) below a few nm of layer thickness and/or grain size, no dislocation pile-ups were formed [14]. A similar study was done by Fang and Friedman for polycrystalline ML materials [15]. Here also deformation maps depending on the layer thickness and grain size were determined.

Another important factor required to establish an

* Corresponding author.
E-mail address: jmfiebig@ucdavis.edu (J. Fiebig).

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understanding of the properties of ML structures involves the control of the interlayer interface structure. A study by Mara et al. presents the influence of the interface type on thermal stability of ML Cu/Nb with a layer thickness of 10 nm fabricated by accumulative roll bonding (ARB) [16]. In this material a highly ordered interface between Cu and Nb forms via deformation twinning and lead to high strength and exceptional thermal stability. A comparison of ML Cu/Nb with the same layer thickness of 20 nm but deformed by different ARB deformation routes (cross-rolled and longitudinally rolled) demonstrate the influence of the interface structure on the thermal stability [17]. The cross-rolled samples contained atomically flat Cu/Nb interfaces while the interface in the longitudinally rolled samples is faceted. These faceted interface stabilizes the material at high temperatures, which was validated by hardness measurements and obtaining the average layer thickness after certain heat treatments. Different combinations of pure elements in experiments and simulation, e.g. Cu/Nb [16,17], Cu/Ni [18,19] or Al/Ti [20,21] with different lattice structures (fcc, bcc, hcp) were investigated with regard to the influence of the interlayer interface on properties as strengthening, thermal stability or phase stability.

In our research we selected the structure of the multilayered material to be different from that published in earlier studies. Instead of combining two different metals as in previous works, the studied ML material consists of alternating nanocrystalline (NC) and coarse grained (CG) layers of a NiFe alloy with similar composition. The NC layer strengthens the structure due to the grain boundary strengthening (Hall–Petch relationship) and the CG layer provides the ductility due to dislocation sources activity and the capability of dislocation multiplication and storage. In a related early study, Tellkamp et al. [60] explained the high strength and relatively good ductility of cryomilled Al by the existence of a few large grains within the nanocrystalline microstructure (average grain size of 35 nm). A similar rational was used by Wang and Ma [22] to explain the increase in strength in combination with good ductility of ultrafine grained Cu that was heat treated to generate a bimodal grain size distribution [22].

Instead of intermixing coarse and nanocrystalline grains the approach of the present study is to understand the mechanical behavior of a ML material consisting of NC and CG layers as it has been published by Kurmanaeva et al. [27] for a NiFe alloy and Daly et al. [61] for a ML NiCo alloy recently. Daly et al. reported that the yield and ultimate tensile strengths for the ML NiCo follow a rule of mixture relationship with respect to the tested monolithic NC and CG NiCo specimens. The tests were performed only at room temperature. In contrast to these results a rule of mixture was not observed by Kurmanaeva et al. [27] for ML NiFe, also tested at room temperature. Instead the measured yield and ultimate tensile strengths were higher than expected on the basis of the rule of mixture. Furthermore, the elongation to failure of the ML samples was less than that of the monolithic NC NiFe specimens [27]. Daly et al. reported a slightly enhanced ductility of the ML NiCo specimens compared with the NC NiCo [61].

Inspection of the published literature shows that most studies report mechanical behavior from testing performed at room temperature [2–4,27]. There are, however, some limited reports on tests at elevated temperature, such as a study of ML Cu/Nb by Mara et al. [23–26], for example. Our previous study on ML NiFe [27] compared the mechanical properties of ML NiFe alloy to those of monolithic CG and NC NiFe samples. The tests were only completed at room temperature and at one selected strain rate. In the present study we focused on the mechanical behavior dependence on strain rate and testing temperature in order to provide insight into the dominant deformation mechanisms. Fractographic and microstructure analysis was completed using secondary electron microscopy (SEM) and transmission electron microscopy (TEM), respectively, on samples studied after tests at different test temperatures.

2. Experimental methods

The multilayered Ni-60 wt%Fe samples were fabricated via pulsed electrodeposition by Integran Technologies Inc. in Toronto, Canada (details can be found in [67]). The modulated microstructure with alternating coarse and nanocrystalline layers was achieved by changing the electrodeposition parameters, namely voltage, current and pulse length. The layer thickness in the present study is 5 μm with a thickness ratio of 1:1. Thus, large sized sheets with a total thickness of 600 μm were produced by depositing on a passivated Titanium cathode. Afterwards the deposited material was mechanically stripped to obtain free-standing sheets from which the tensile specimens were sectioned by using EDM. The material contains a slight amount of impurities (Chemical composition C: 0.026%, S: 0.018%, N: 0.0004%, Co: 0.06%, Cu: 0.014% and Mo: 0.017%, all in wt% [27]).

Uniaxial tensile tests were performed with a mini-tensile tester with a displacement resolution of 5 μm and a load resolution of 0.01 N controlled by LabVIEW software. The test temperature was selected between room temperature (RT, $0.17T_m$) and 400 °C ($0.4T_m$) at strain rates between $10^{-4}\,s^{-1}$ and $10^{-2}\,s^{-1}$. The gauge section of the dogbone-shaped tensile specimens measured 3 mm × 1 mm × 0.3 mm. All surfaces were mechanically polished to a mirror-like quality. Prior to each high temperature tensile test the chamber was first evacuated for several hours and the samples were heated to 100 °C for 4 h to get rid of any moisture inside the chamber. The tests itself were obtained under a positive (1 Psi pressure) high purity argon atmosphere (99.999% Ar). Argon was used as a protective gas to avoid oxidation of the specimens during the tensile tests. The strain rate sensitivity was calculated by:

$$m = \frac{\partial \ln \sigma_{YS}}{\partial \ln \dot{\varepsilon}} \tag{1}$$

here m is the strain rate sensitivity, $\dot{\varepsilon}$ is the strain rate and σ_{YS} is the yield stress. The activation volume can be obtained by:

$$\Delta V = \sqrt{3}\, k_B T \frac{\partial \ln \dot{\varepsilon}}{\partial \sigma_{YS}} \tag{2}$$

here k_B is the Boltzmann constant, T is the absolute temperature, $\dot{\varepsilon}$ is the strain rate and σ_{YS} is the yield stress.

The thermal stability was investigated by using a differential scanning calorimeter (DSC) 404F3 by Netzsch. DSC measurements were performed at two different heating rates 20 and 30 K/min under a protective argon atmosphere. The sample weight was between 60 and 90 mg. The furnace of the DSC was purged for 10 min prior to each measurement. The peak temperatures were determined with the Proteus Analysis software provided by Netzsch.

A JEOL JEM-2010 microscope was used to perform the cross-sectional TEM (XTEM) and selected-area electron diffraction (SAED). The work was done under 200 KV with a double-tilt holder and a point-to-point resolution of 0.23 nm. Conventional TEM sample preparation routine including cutting, gluing, grinding, polishing, and final precision ion polishing was used to prepare the XTEM foils.

The fracture surfaces were studied using scanning electron microscopy FEI 30 SEM at 5 keV.

3. Experimental results

The stress–strain curves of ML NiFe samples at different strain rates at 20 °C (room temperature, $0.17 \cdot T_m$) and 400 °C ($0.4 \cdot T_m$) are