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Microstructural deformation in fatigued nanotwinned copper alloys

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ABSTRACT

In this study, the uniaxial tension-tension fatigue behavior of fully nanotwinned magnetron sputtered Cu-6wt%Al, Cu-2wt%Al, and Cu-10 wt%Ni is presented. These alloys have average twin thicknesses ranging from 4 to 8 nm, average grain widths from 90 to 180 nm, and tensile strengths from 1 to 1.5 GPa. In the high cycle regime (10^3 to 10^7 cycles), the nanotwinned alloys exhibit fatigue strengths ranging from 210 to 370 MPa, which is higher than previously observed in nanotwinned Cu (fatigue strengths between 80 and 200 MPa). Fatigue strengths are normalized by tensile strength for Cu alloys with different microstructures to study the correlation between tensile and fatigue properties. Post-mortem analysis of the materials reveals a newly observed deformation mechanism, where localized detwinning leads to intergranular fracture between columnar grains. Overall, materials displaying detwinning as a deformation mechanism show lower normalized fatigue strengths in comparison to materials that deform with slip band like behavior.

1. Introduction

Nanotwinned metals have been of much interest largely due to their potential for simultaneous high strength and ductility [1–4] when compared to nanocrystalline metals which tend to have low ductility [5]. While the strength of a material is important in any design process, it has been estimated that 90% of mechanical service failures in metals occur due to material fatigue [6–8], and so it is important to understand the fatigue behavior of NT metals if they are to be utilized in engineering applications. Although some work has been performed investigating how NT Cu behaves under a cyclic load [9–14], the fatigue behavior of fully NT alloys has not been observed, and a fairly limited scope of microstructural parameters has been considered. This study aims to investigate the fatigue mechanisms in alloyed NT metals in order to understand how the deformation behavior affects their resultant fatigue properties.

Several previous studies have investigated uniaxial stress-controlled tension-tension fatigue of columnar NT Cu with varying microstructures [10,11], where the materials showed improved fatigue strengths compared to coarse grained Cu, yet little or no improvement was observed when compared to ultrafine grained (UFG) Cu. The mechanisms of fatigue vary for the different studies, where electrodeposited Cu by Pan shows the formation of 'zig-zag' persistent slip bands (PSB's) transferring through twin boundaries along with the formation of 'block' structures [11]; twin growth and formation of non-twinned regions are the primary fatigue mechanisms observed in magnetron sputtered Cu presented by Shute et al. [10] and Funk [15]. Twin growth was also observed as a fatigue mechanism in NT Cu as shown by Yoo et al. [13], where a high-throughput fatigue test was utilized to apply variable cyclic strain amplitudes to a NT Cu film. Similarly, a study by Li reveals that partially nanotwinned stainless steel 316 L (56% of grains twinned with 22 nm average twin thickness) performs better in uniaxial push-pull fatigue tests than coarse-grained stainless steel 316 L [16].

Preliminary studies investigating how different microstructures might influence the fatigue behavior in NT Cu have been performed. Crack propagation rates of pre-notched NT Cu samples with twin thicknesses of 32 and 85 nm were investigated by Singh et al. [12], who found that the sample with smaller twin thicknesses had a slower crack propagation rate at the same initial stress intensity amplitude, and so it is surmised that NT metals with smaller twin thicknesses tend to have better fatigue resistance. These results are further supported by Zhou et al. [14], where simulations showed that fatigue behavior tends to improve with decreasing

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twin thickness. In the simulations, both detwinning and crack closure at the crack tip are observed, believed to improve the fatigue resistance compared to nanocrystalline (NC) metals; note that these simulations investigate NT Cu in the low cycle regime (tens of cycles) with twin thicknesses ranging from 0.83 to 9.39 nm and grain sizes ranging from 10 to 20 nm, outside of the parameters typically seen in as-prepared NT films. Similar results are also obtained in a fatigue study on partially twinned NiCo alloy [17], where experiments and simulations showed that smaller twin thicknesses correlated to reduced crack nucleation rates in pre-notched samples. However, partially twinned structures do not isolate the influence of the nanotwinned structure and therefore further studies are still needed.

Since fatigue studies in NT metals are limited, and since it has been shown that twin thickness can influence the mechanical behavior similar to grain size [18–20], a comparison to nanocrystalline (NC) materials could provide further insights. Although the fatigue behavior of metals on the nanoscale is also fairly limited, in general, it has been observed that NC materials perform better under cyclic loading than either their ultrafine-grained or coarse grained counterparts [21–23]. NC metals exposed to cyclic loading have shown some similar deformation features to CG or UFG metals, where the presence of persistent slip bands, fatigue striations, shear lips, and dimple fracture has been observed [21,23]. However, in contrast to fatigue in CG materials, NC metals show grain growth as a primary microstructural deformation mechanism [23,24], which can lead to cyclic softening. All of these studies have investigated nanocrystalline materials with grain sizes ranging from 18 to 100 nm [23]; to date the fatigue behavior below a grain size of 18 nm has not been investigated.

As NT materials evolve towards use in engineering applications, the use of alloyed nanotwinned systems will allow for a wider range of materials and properties. To the author's knowledge, no existing studies investigate the fatigue behavior of fully nanotwinned alloys (i.e. all the observed grains are nanotwinned). This manuscript aims to establish an understanding of fully nanotwinned alloys behavior under cyclic loading through coupling of the uniaxial fatigue behavior and the mechanisms that lead to fracture in the high cycle fatigue regime (10^3 to 10^7 cycles). A wide range of Cu alloys including Cu-6wt%Al, Cu-2wt%Al, and Cu-10 wt% Ni were tested at various conditions. The microstructural changes after fatigue were investigated to understand how the deformation mechanisms contribute to the fatigue properties. Furthermore, the results from this study are compared to previous works for both nanotwinned and nanocrystalline materials in order to highlight the role nanotwins play in the fatigue behavior of nanoscale metals.

2. Experimental

Fully nanotwinned 12–18 μm thick free standing films were sputtered following procedures outlined by Velasco et al. [25]. A total of four films were sputtered with three compositions: one Cu-10 wt%Ni, two Cu-2wt%Al with both higher and lower strength, and one Cu-6wt%Al. These films will be referred to as Cu10Ni, Cu2Al-HS, Cu2Al-LS, and Cu6Al respectively within this manuscript. Stacking fault energies for these alloys are 6, 37, and 60 mJ-m^{-2}, respectively, spanning an order of magnitude [26–28]. Thorough microstructural characterization was performed on each sample by TEM. Cross-sectional TEM samples were prepared by mounting the materials in silicon, preparing 3 mm discs, dimple grinding, and then ion milling with a FISCHIONE Model 1050 ion mill with a finishing accelerating voltage of 1 kV. TEM was then performed on each of the discs with a JEOL 2100F TEM, utilizing a combination of bright field and dark field TEM at an accelerating voltage of 200 kV. A minimum of 300 twins and 100 grains were measured for each film in order to determine the average twin thickness and grain width as well as distributions. Errors in the measured microstructural parameters were calculated as a combination of standard error from averaging and the maximum expected error in TEM measurements.

From each film, a minimum of one dog bone sample was prepared in order to perform tensile tests. The tensile behavior of some of the films (Cu6Al and Cu2Al-LS) had already been characterized and discussed in a previous study [29]. Thin strips were cut from the films and then mechanically polished in a die with a geometry proportional to ASTM E345-93 Type A with a reduced section of 0.8 × 3.6 mm. Sample geometries were measured utilizing a combination of optical microscopy (for sample widths) and SEM (for sample thicknesses) and tensile tests were performed utilizing a custom made tensile tester. For tensile tests, optical digital image correlation was utilized for strain measurements while a LC703-100 load cell was utilized for load measurements. Tensile tests were performed at strain rates of roughly 10^{-4}/s until fracture, following procedures described elsewhere [29]. Errors in material strengths were calculated from a combination of uncertainty in sample dimensions and maximum observed deviations found in thorough testing of calibration samples and nanotwinned samples.

Fatigue samples were formed utilizing a similar preparation method to the tensile dogbones, albeit with reduced sections of roughly 0.65 × 3.6 mm. Fatigue tests were carried out utilizing a custom built uniaxial fatigue test setup, which is described in more detail elsewhere [30]. Stress cycling was performed with a piezoelectric actuator and load measurements were made with a load cell. All fatigue tests were performed utilizing a 30 Hz sinusoidal wave with an R ratio (minimum to maximum stress) of 0.1 (tension-tension fatigue). Tests were stopped either when the sample completely fractured or when $5*10^6$ cycles was reached. Proportional control was used to reach the desired stress amplitude and mean load; this required a settling period of less than 400 cycles, and no overshoot during initial loading was observed. Only cycles beyond the settling period were considered for the number of cycles to failure. Optical images of the top surface of each sample were captured both before and after the fatigue test in order to identify any potential test anomalies. Initial fatigue tests were performed on Cu2Al-LS samples, where a total of two tests were performed at four different stress amplitudes of 210, 270, 340, and 370 MPa. In order to compare the behavior to the Cu2Al-HS, Cu10Ni, and Cu6Al alloys, two selected stress amplitudes (210 and 370 MPa) were applied to each material and at least two tests were performed for each amplitude.

Post-mortem morphological and microstructural analyses were performed on various samples utilizing a combination of SEM and TEM. Both a Zeiss and JEOL 7001F SEM were utilized to image the fracture surface morphologies at an accelerating voltage of 15 keV, although only images from the JEOL 7001F are presented in this manuscript. The entire fracture surface was imaged for one sample from each film at stress amplitudes of both 210 and 370 MPa. TEM was carried out on three select samples at the fracture surface in order to investigate any changes in microstructure during cyclic loading. TEM samples were prepared by FIB liftout, utilizing a JEOL 4500 FIB at an accelerating voltage of 30 keV, where the fracture surfaces were covered with a protective carbon layer prior to performing liftout in order to preserve the surface. A combination of bright field TEM and SAED were performed along and near to the fracture surface in order to understand any changes in microstructure along the fracture surface.

3. Results and discussion

Fig. 1 shows the microstructure, twin size distribution and