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Length-scale-controlled fatigue mechanisms in thin copper films

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Abstract

Systematic investigations of fatigue damage and dislocation structures in thin Cu films with different thicknesses (0.2–3.0 μm) and grain sizes (0.3–2.1 μm mean diameter) were carried out using focused ion beam microscopy and transmission electron microscopy. The morphologies of fatigue-induced extrusions, cracks, and dislocation structures were studied and found to be controlled by film thickness and grain size. When either of these length scales is decreased below roughly 1 μm, the typical dislocation wall and cell structures found in fatigued coarse-grained bulk materials no longer develop and are replaced by individual dislocations. Similarly, the typical surface damage of fatigued bulk metals, such as extrusions and cracks near extrusions, is gradually suppressed and replaced by damage that is localized at interfaces, such as cracks, grooves, and voids along grain and twin boundaries. This gradual transition from damage characteristic of bulk metals to damage localized at interfaces is attributed to constraints on dislocation activity at submicrometer length scales. Based on the experimental results and a theoretical analysis of extrusion formation, a mechanistic map of fatigue damage behavior is proposed that summarizes this length scale dependence.

Keywords: Fatigue; Thin films; Length scale; Dislocation structure; Interfaces

1. Introduction

The strength of metal films with thicknesses of several micrometers or less is markedly higher than that of their bulk counterparts due to constraint effects exerted by the small dimensions on dislocation activity [1,2]. In general, the dimensions of a material refer not only to the geometry, such as film thickness or sample size, but also to the microstructural dimensions, such as grain size, particle size, etc., inside the material [2]. Recently, a number of studies of length-scale effects on mechanical properties have been conducted in small-dimensioned materials (such as thin films [3]), in fine-grained bulk materials (such as ultrafine grain [4]), and in nanocrystalline [5] materials, revealing special mechanical properties.

Fatigue, which has been investigated in bulk materials for more than a century, has recently begun to be investigated in materials with small dimensions [6–17] and grain sizes [18–27]. These investigations have shown that surface damage and dislocation structures as observed in fatigued bulk materials do not form when the film thickness and/or grain size are close to or less than the characteristic fatigue dimensions [14,16,26], such as the height and width of the extrusions and the spacing of the dislocation walls.

Fatigue properties of ductile metals with different geometric dimensions have been investigated by testing metallic wires [6], microbeams [7], and thin metal sheets and films [8–17]. Hofbeck et al. [6] reported that the absence of extrusion-like features at the surface of thin wires (30 μm in diameter) was due to the absence of persistent slip bands (PSBs). Judelewicz et al. [8] found that 20 μm thick Cu foils were almost free of extrusions and contained only a few

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grains with a low density of faint slip bands. Read [11] explained that the absence of clear slip steps on sample surfaces and of dislocation cells in 1.1 μm thick Cu films indicated that dislocations moved individually or in small groups. Recently, a systematic investigation of fatigue behavior of Ag and Cu films with thicknesses of micrometers or less by Schwaiger and Kraft [12–15] has shown that surface extrusions and voids at the film/substrate interface dominate fatigue damage of the thin films. Furthermore, the disappearance of fatigue dislocation structures in films thinner than 3 μm and a decrease in cyclic plastic strain accumulation with decreasing film thickness [16] have been found. All of these results indicate that fatigue damage formation is somehow hindered in thinner films. Therefore, the fatigue strength and the fatigue life of these small-dimensioned metals are expected to be greater than those of their bulk counterparts.

The effect of grain size on fatigue damage behavior has been studied in metals with micrometer- [18,19], submicrometer- [20–26], and nanometer-size grains [27]. In metals with micrometer-sized grains, Kawazoe et al. [19] found that the appearance of PSBs was extremely restricted when the grain size was smaller than 8 μm. Thiele et al. [26] examined the fatigue-induced dislocation structures in bulk polycrystalline Ni with mean grain sizes between 500 nm and 5 μm. They found that fatigue-induced dislocation structures did not form when the grain size was smaller than 1 μm. In submicrometer-grained metals, typical PSBs could not form in the submicrometer-sized grains [23,25,26], and instead of the formation of PSB-induced extrusions, shear banding and cracking along the shear bands were observed [25].

Despite the clear experimental evidence supporting the concept that fatigue damage formation is influenced by sample size and grain size, systematic and comprehensive investigations of the relationships among fatigue surface damage, dislocation structures, and material length scales have not yet been carried out. In this paper, we present the results of such a study on thin Cu films with thicknesses and grain sizes in the micrometer to submicrometer range. Two types of thin films were studied here: type I where the grain size is larger than the film thickness (columnar) and type II where the grain size is less than the film thickness. The mechanisms of fatigue damage in these highly constrained geometries are evaluated.

2. Experimental

The two types of thin Cu films (type I and II) were deposited by magnetron sputtering under ultrahigh-vacuum conditions (base pressure $<10^{-5}$ Pa) onto dog-bone shaped polyimide substrates with a thickness of 125 μm (gauge section: 22 mm length × 6 mm width). The type I films were deposited onto substrates cooled by liquid nitrogen and were then annealed at 100 °C for 2 h in the deposition chamber. The type II films were deposited onto substrates that were held at 300 °C and were then annealed for 2 h at 400 °C in the deposition chamber.

Film thicknesses and mean grain sizes were determined using focused ion beam (FIB) microscopy (FEI 200xP) [13,14] and are summarized in Table 1. The grain size is defined as the grain diameter of an equivalent circular area as determined from plan view FIB images. Twin boundaries were ignored in this determination. The grain sizes have a broad distribution, so that both submicrometer grains and grains with diameters of several micrometers were found in all of the films. However, as shown in Fig. 1, the mean grain sizes of the type I films are larger than 1 μm [14], while those of the type II films are smaller than 1 μm [13]. FIB cross-sectional images of the grain structure through the film thickness show a trend that grains with an in-plane diameter larger than the original film thickness are usually columnar. Thus, the type I films are predominately columnar and the type II films typically have two grains through the film thickness. X-ray diffraction

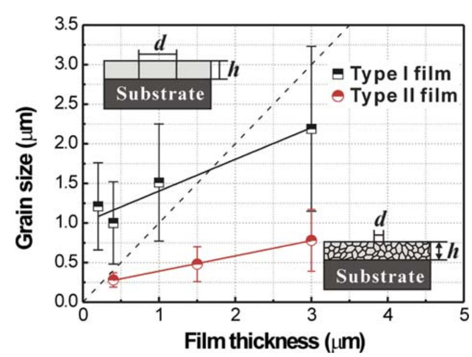

Fig. 1. Schematic of the relationship between geometric length scale (film thickness, h) and microstructural length scale (mean grain size, d) in the thin films studied.

Table 1
Summary of microstructural parameters and fatigue testing conditions for the type I and II films

Cu thin film	h (μm)	D (μm)	$\Delta\varepsilon_t$ (%)	$\Delta\varepsilon_{pl}$ (%)	N
Type I [14,30]	3.0	2.19 ± 1.04	1.0	0.91	5×10^3
	1.0	1.51 ± 0.74	1.0	0.87	5×10^3
	0.4	1.00 ± 0.52	1.0	0.86	1×10^4
	0.2	1.21 ± 0.55	1.0	0.83	2×10^4
Type II [13]	3.0	0.78 ± 0.39	1.3	1.01	6×10^4
	1.5	0.48 ± 0.22	1.3	0.92	6×10^4
	0.4	0.28 ± 0.09	1.7	1.22	6×10^4