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Mechanics of fatigue crack initiation in submicron-thick freestanding copper films

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ABSTRACT

The mechanics of fatigue crack initiation in roughly 500-nm-thick freestanding copper (Cu) films were investigated by using single-side-edge-notched specimens and elastic finite element method (FEM) analyses. By applying cyclic loading at a stress ratio $R = 0$, an intrusion/extrusion was formed near a notch root, and a fatigue crack then initiated at the intrusion/extrusion, which penetrated the film in the thickness direction on a slip plane parallel to a Σ3 twin boundary. Local stress distributions were evaluated by three-dimensional FEM analyses, taking account of the individual grain shape and crystal orientation around the fatigue crack initiation sites. The results revealed that a fatigue crack was initiated on slip systems where (i) the slip deformation penetrated the film in the thickness direction without being blocked by grain boundaries or twin boundaries, and (ii) a high resolved shear stress occurred under the conditions in (i). The number of cycles to fatigue crack initiation increased as the intensity of the resolved shear stress field at the fatigue crack initiation site decreased. The resolved shear stress required for fatigue crack initiation in the Cu films is roughly one order of magnitude higher than that in a bicrystal or single-crystal bulk Cu of 30–35 MPa.

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1. Introduction

Submicron-thick metallic films have a characteristic structure compared with bulk metals such as a large surface-area-to-volume ratio and fine columnar grains with sizes of the same order of magnitude as the film thickness. Owing to the characteristic structure, the plasticity of the films exhibits size effects [1–4]: grain boundary (GB) strengthening [5,6], dislocation source starvation and annihilation of mobile dislocations at the free surface [7–11] occur in the films. Hence, the yield strength of the films is higher than that of bulk metals.

Because fatigue damage formation is brought about by an accumulation of cyclic plastic deformation, the characteristic plasticity of thin films influences their fatigue behavior. Many researchers conducted fatigue experiments of metallic films fixed on substrates, and reported the mechanisms and mechanics of fatigue damage formation at the submicron scale [12–21]. Zhang et al. [16] reported the size effects on the mechanisms of fatigue damage formation in Cu films with thicknesses ranging from 0.2 to 3.0 μm deposited on a polyimide substrate: extrusions on the free surface and self-assembled dislocation structures such as dislocation walls and cells inside the films were formed in the micron-thick films. This fatigue damage is often observed in bulk metals. In contrast, localized fatigue damage such as cracks along GBs and twin boundaries (TBs) and individual dislocations was observed in submicron-thick films. Trinks and Volkert [22] reported that extrusions were observed in films with a thickness of 200 nm and greater, whereas GB grooves and hillocks due to diffusion creep were formed instead of extrusions in sub-100-nm-thick films. Wang et al. [17] reported the size effects on the fatigue life of Cu films with thicknesses ranging from 50 nm to 3.0 μm on a polyimide substrate: thinner films needed higher strain ranges or more cycles to fatigue fracture. Sumigawa et al. [21] conducted fatigue experiments using 200-nm-thick Cu films sandwiched between rigid substrates and then conducted a finite element method (FEM) analysis which took the grains into account to evaluate the local stress. They reported that a fine intrusion/extrusion was formed on a slip system with the highest maximum resolved shear stress, and the films had a higher strength to fatigue damage formation than bulk Cu.

These studies, however, investigated the fatigue damage process or fatigue properties of metallic films fixed on substrates. In this condition, dislocation motion is constrained by the substrate [23,24], and pile-up of dislocations at the film/substrate interface induces the formation of voids, causing stress concentration and consequent fracture [14,17,18]. Furthermore, the evaluation of

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the mechanical properties of films is influenced by the residual stress due to the presence of the substrates [25]. For these reasons, it is difficult to evaluate accurate local stress fields at the fatigue damaged site. Hence, the use of freestanding films is desirable to investigate the intrinsic mechanisms and mechanics of fatigue in thin films. Moreover, freestanding thin films are used in micromachines such as microelectromechanical systems (MEMS), and it is also important, from an engineering standpoint, to clarify the mechanisms and mechanics of fatigue in freestanding thin films.

Some researchers conducted fatigue experiments using submicron-thick freestanding films [26,27]. Hu et al. [27] conducted fatigue experiments on 300-, 500-, and 700-nm-thick freestanding Cu films and reported that the fatigue life increased with decreasing thickness, and the mechanisms of fatigue crack initiation correspondingly changed from intrusion/extrusion formation to cracking along GBs as the film thickness decreased. The authors in [28] conducted fatigue crack propagation experiments in approximately 500-nm-thick freestanding Cu films and reported that intrusions/extrusions were formed ahead of the crack tip at stress ratios R of 0.1 and 0.5 in the region of maximum stress intensity factor (K_{max}) < 4.5 MPa $m^{1/2}$, and the fatigue crack then propagated preferentially through the intrusions/extrusions. These intrusions/extrusions were formed on the slip planes parallel to Σ3 TBs and penetrated the films in the thickness direction. In bulk metals, intrusions/extrusions have a significant impact on fatigue crack initiation, whereas they do not affect the fatigue crack propagation. The intrusions/extrusions, however, played a major role in fatigue crack propagation in submicron-thick freestanding films; therefore, the mechanics of fatigue damage formation are essentially important to understand not only fatigue crack initiation but also fatigue crack propagation. Because intrusions/extrusions are brought about by a local stress field, it is necessary to consider the local stress field that accounts for the influences of the constraints of the neighboring grains and the stress concentration at the GBs and TBs. However, this has not been investigated in submicron-thick freestanding films.

The purpose of the present study is to clarify the mechanics of fatigue crack initiation in submicron-thick freestanding Cu films. Fatigue experiments were conducted on roughly 500-nm-thick freestanding Cu film specimens with a single side edge notch. GBs and TBs ahead of the notch were identified by electron backscatter diffraction (EBSD) analyses. The fatigue crack initiation process was observed by optical and field-emission scanning electron microscopy (FESEM). Moreover, the local stress distribution at the fatigue crack initiation sites was evaluated by anisotropic elastic FEM analyses that take account of the individual grain shape and crystal orientation around the fatigue crack initiation sites. The mechanics of fatigue crack initiation were discussed on the basis of these results.

2. Experimental

2.1. Materials and specimens

The tested materials were Cu films with a thickness of roughly 500 nm. The purity of the Cu evaporant was 99.999%. The Cu films were deposited by electron beam evaporation at a pressure below 1.0×10^{-3} Pa. The substrate temperature during the deposition was not controlled. Fig. 1 shows a crystal orientation color map of the film surface obtained by EBSD (EDAX, DigiView) using FESEM (JEOL Ltd., JSM-7001F). Black lines indicate GBs, red lines indicate Σ3 TBs and blue lines indicate Σ9 TBs. Most of the TBs were Σ3 coherent TBs. The average grain sizes considering the TBs as GBs and not considering those as GBs were 370 nm and 1010 nm, respectively. The EBSD analysis of both surfaces of the freestanding film confirmed that the films had a columnar structure.

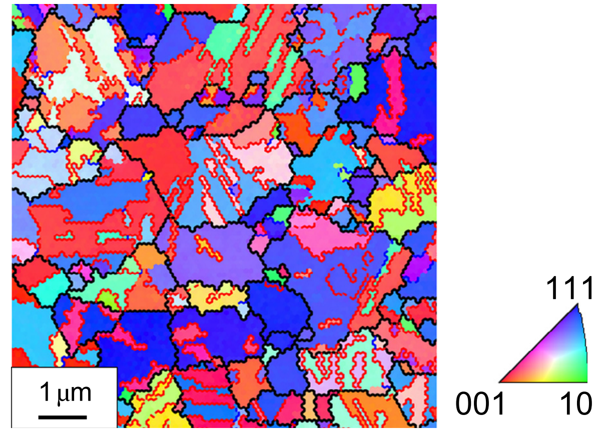

Fig. 1. EBSD orientation color map of the surface of a roughly 500-nm-thick freestanding Cu film (black lines: grain boundaries, red lines: Σ3 twin boundaries, blue line: Σ9 twin boundaries). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Freestanding film specimens were fabricated by a sacrificial layer etching method [28–30]. The shape and dimensions of the specimen are presented in Fig. 2. A single side edge notch was prepared in the center of the parallel section of the specimen. The specimens were fabricated by depositing through a specimen-shaped metal shadow mask. The thickness of each specimen was measured by using a stylus surface profiler (Tencor, AlphaStep 500; resolution: 0.1 nm), and the average thicknesses (B) are listed in Table 1. The thickness within 1–3 µm from the edge of the specimen deposited through the metal shadow mask became smaller than that of the inside region of the specimen. Therefore, the thinner region around the notch root was trimmed by using a focused ion beam (FIB) system (Seiko Instruments Inc. (now Hitachi High-Tech Science Corp.), SMI9200). The notch radii after FIB trimming (ρ), the notch lengths (a), and the stress concentration factors (α) are listed in Table 1. α is the ratio of the stress at the notch root to the stress at the parallel section without the notch (2 mm) and was evaluated by an elastic FEM analysis under the assumption of isotropic elastic materials. The shapes of the notch roots were designed so that α was 3.1–3.7. EBSD orientation color maps around the notch root were obtained before the experiments. The distance between adjacent EBSD analysis points was set to be 100 nm.

2.2. Experimental setup

Fatigue experiments were conducted by using a piezoelectric actuator-driven fatigue testing machine [28]. Fatigue experiments were conducted under load-controlled conditions. The maximum stress at the parallel section in the specimen without the notch (σ_{max}) was set at a constant value. The applied stresses are also listed in Table 1. A tension–tension cyclic stress with a sinusoidal wave was applied at a stress ratio (R) of 0 at a frequency (f) of 30 Hz. All experiments were conducted at 298 ± 5 K in ambient air.

Fatigue damage on the specimen surface was observed every 1.0×10^3 cycles by using a digital optical microscope (Hirox Co., Ltd., MXG-2500REZ; magnification: 2500×). To clarify the detailed fatigue crack initiation process, FESEM observation of the fatigue damage was conducted. The fatigue experiment was interrupted, and the specimen was detached from the experimental setup after several thousand cycles. Thereafter, fatigue damage was observed

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