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Formation of slip bands in poly-crystalline nano-copper under high-cycle fatigue of fully-reversed loading

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ABSTRACT

The purpose of this study is to investigate the effects of a nano-scale stress-field on fatigue damage in a nano-copper component under fully-reversed and high-cycle loading. A resonant fatigue experiment is carried out for a cantilever micro-specimen that has a polycrystalline nano-Cu sandwiched by Si, Ti and SiN. Crystallographic slip bands associated with extrusion/intrusion of about 30 nm width, which is much finer than that in the bulk copper ($\approx 1\ \mu m$), are formed on the Cu surface owing to the high-cycle fatigue loading. The new finding, the ultra-fine extrusion/intrusion, suggests the existence of different fatigue damage mechanisms in the nano-Cu from that in a bulk counterpart. The slip bands appear only in a particular grain though some others possess slip systems with higher Schmid factor. Detailed stress analysis, taking into account the Cu grains and surrounding dissimilar materials (Si, Ti, SiN), indicates that they are formed at a slip system with the highest resolved shear stress, which is in nano-scale. The formation stress is much higher than that in a bulk.

1. Introduction

In fatigue of a polycrystalline bulk metal at low or middle plastic strain amplitude ($\varepsilon_{pl.} = 1\times10^{-4} \sim 1\times10^{-2}$) under fully-reversed cyclic load, persistent slip bands (PSB) with widths in the range of several micrometers associated with extrusion/intrusion on the surface of the material are formed as in the case of a bulk single crystal [1–7]. While an activated slip system that forms the PSB has a geometric relation between the crystal orientation and loading direction in a single crystal [1], the activated slip system and PSB formation are governed by a local stress field due to deformation constraint between grains in the polycrystalline material [8]. Moreover, a crack is initiated owing to stress concentration at the PSB/matrix interface and at the intrusion. The collision of the PSB and grain boundary induces a favorable cracking site as well [9–11]. It should be careful that the PSB are rarely observed in tension–tension fatigue of bulk [12].

Electronic devices are typically composed of small components, which have nano-meter or micron scale. In the components, the stress distributes owing to its geometry and the deformation constraint by the surrounding materials. A metal is an important component, e.g., copper in wiring, and fatigue fracture of the metal caused by mechanical vibration and cyclic thermal stress in the nano-scale is a concern for reliability of a device. However, nanometer-scale metal does not have space to form the PSB, because the PSB width is several micrometers independently of the material size. Thus, it is an open question as to whether fatigue causes damage in a nanoscale metal.

Our group has carried out fatigue experiments of submicron-scale gold (Au) single crystals [13,14] using resonant vibration which can realize high-cycle and fully-reversed loading. Our studies revealed that extrusion/intrusion with a width of several nanometers was formed on the activated slip system with the maximum Schmid factor on the specimen surface [14]. However, polycrystalline metallic films consist of grains in the nanometer scale, and the local stress field due to deformation constraint between grains has a smaller size. Although there is a large body of research on films with submicron thickness [15–21], the effects of nanoscale local stress fields on fatigue damage of polycrystalline nano-scale metals have not been experimentally explored. It is also difficult to carry out fatigue experiment of film under fully-reversed load, which brings about clear PSB in a bulk metal [19].

In the present research, resonance fatigue experiment is carried out under fully-reversed load for a micro-specimen, which is carved out from a multi-layered material consisting of silicon (Si), titanium (Ti), polycrystalline copper (Cu), and silicon nitride (SiN). Using detailed observations and stress analysis, the fatigue damage in a micro-component with polycrystalline is investigated under nano-scale stress concentration.

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2. Experimental procedure

2.1. Material and specimen

After the native oxidized layer on a single crystalline silicon (Si) (100) substrate is removed by argon ion etching, layers of titanium (Ti) (thickness: 20 nm), copper (Cu) (thickness: 200 nm), and silicon nitride ((Si_3N_4 (hereinafter, it is described as "SiN"))) (thickness: 20 nm) are continuously deposited by magnetron sputtering. While the Cu and Ti layers are polycrystal, the SiN layer is amorphous. The multi-layered plate is annealed in vacuum (1×10^{-4} Pa) at 673 K for 1 h.

Fig. 1 schematically illustrates the specimen in the method used for the resonant fatigue experiments [13,14]. The specimen consists of a weight, a test section and a base. The test section is composed of the Si, Cu, Ti and SiN layers. The specimen is formed from the multi-layered plate using a focused ion beam (FIB) processing system (Hitachi, FB-2200) with accelerating voltage of 40 kV wherein the beam current is set to 1.17 nA.

The fabrication procedure used for the specimen is as follows.

1. A block of 25 μm × 25 μm × 25 μm dimensions is carved out of the multi-layered material (Fig. 2(a)).
2. The block is cut from the substrate after a microprobe is attached to the upper surface by tungsten (W) deposition (Fig. 2(b) and (c)).
3. The block is placed on a 5 mm × 5 mm Si substrate (thickness 550 μm) (Fig. 2(d)) and is mounted by W deposition (Fig. 2(e)). The microprobe is separated by FIB.
4. Similarly, a block of weight is carved out of an Au plate and is picked up by a microprobe. The Au block is mounted on the SiN layer of the multi-layered block, which was mounted on the Si substrate, by W deposition (Fig. 2(f) and (g)).
5. A test section that includes the Si substrate and layers of Ti, Cu and SiN is fabricated using y- and z- direction beams with low currents of 0.02 nA (Fig. 2(h)). The upper surface of the test section is flattened by a weak x-direction beam in order to avoid any difference in level at the interfaces (Fig. 2(i)).
6. In order to remove damaged and re-deposited layers introduced by FIB, argon (Ar) ion milling (HITACHI, Gentle mill-Hi; accelerating voltage: 0.3 kV, current: 8 μA, processing time: 5 min) is performed on the surfaces of the test section.

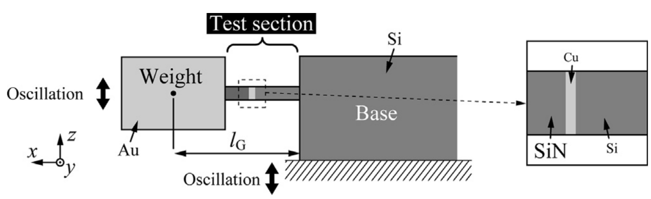

Fig. 1. Schematic illustration of the resonant fatigue experimental method. A specimen consists of a weight, a test section and a base.

By inducing oscillation in the vertical direction to the base, as shown in Fig. 1, a fully-reversed bending load is applied to the test section. The resonant frequency of the nano-/micro-scale material is usually more than a few tens of GHz. However, too high a resonant frequency brings about difficulties in control of the fatigue cycle because a number of 10^7 cycles, commonly defined as the number of cycles of the fatigue limit, is attained in less than 1 s. This can be reduced by removing the weight attached to the cantilever tip. The resonance frequency, f_0, of a cantilever (cross-section of w (width) × h (height)) with a heavy weight at the end is approximately evaluated by the following equation.

$$f_0=\frac{1}{2\pi}\sqrt{\frac{k}{m}}\quad\left(k=\frac{Ewh^3}{4l_G^3}\right) \tag{1}$$

here, k is the spring constant of the test section in the vibration direction, m is the mass of the weight, E is Young's modulus of the test section and l_G is the length from the test section root to the

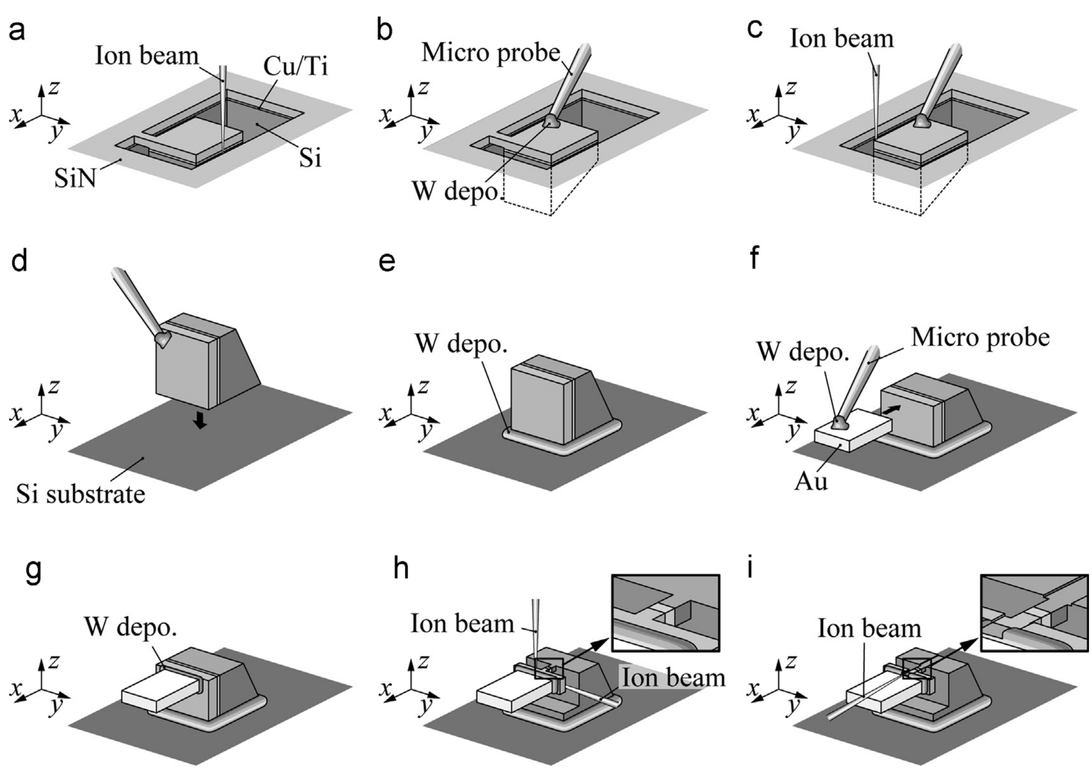

Fig. 2. Schematic illustration of the specimen preparation method using FIB.