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Tensile behavior of micro-sized specimen made of single crystalline nickel

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ABSTRACT

This paper proposed the effect of crystal anisotropy of micro-specimens evaluated by a micro-tensile. The micro-gripper and specimen were fabricated from a commercially available diamond-tip indenter and single crystal Ni disk, respectively, by focused ion beam. The stress–strain curves obtained from the micro-tensile test showed a few load drops, which is similar to serration contributed by occurrence of slip lines observed on surface of the specimens. Moreover, the difference in crystal orientations of ⟨111⟩ and ⟨223⟩ along the loading axis on stress–strain behaviors is evaluated. The results indicated the crystal anisotropy by Schmid factor is the same as the deformation behavior of face-centered-cubic bulk metals. Thus, the micro-tensile testing method used in this experiment successfully showed the effect of crystal anisotropy in micro-scale.

1. Introduction

A micro-mechanical testing method was widely used to investigate mechanical properties in micro-scale, such as sample size effects and crystal anisotropy [1–3]. Investigation of these characteristics in micro-scale is important to utilize the materials as micro-components, for example the mechanical parts used in micro-electro-mechanical systems (MEMS). In particular, micro-compression and micro-bending tests using micro-specimen are more frequently conducted than micro-tensile test, because gripping of the micro-specimen is difficult in tensile tests [1,4]. In the case of bulk materials, tensile experiments are often used to evaluate mechanical properties including fracture strength, elongation and fracture behavior, which are not provided by compression and bending tests [5]. There are only a few reports on micro-tensile test [6,7].

Materials used in an electronic component are often composed of polycrystals. In general, volume fraction of a single grain in the electronic component would be higher when size of the electronic component is decreased. In this case, the effect of crystal anisotropy would become more significant with decrease in size of the electronic component or device. The study of crystal anisotropy can be conducted using specimens fabricated from single crystal bulk material with controlled orientation. However, effects of crystal anisotropy on fracture strength, elongation and fracture behavior of the materials in micro-scale are difficult to evaluate since it is difficult to conduct the micro-tensile test. In fact, there is still no report on studying the effect of crystal anisotropy by a micro-tensile test.

Our group has reported several studies on micro-bending and micro-compression test with a specially designed micro-testing machine [8–10]. We also developed a method to conduct micro-tensile test with the micro-testing machine [12]. In this paper, the effect of crystal anisotropy on micro-tensile behaviors of micro-specimens composed of single crystal Ni (SCNi) will be evaluated using the micro-tensile test method we developed.

2. Experimental procedures

A micro-gripper for the micro-tensile test was fabricated from a commercial available diamond-tip micro-indenter (Synton-MDP: Spherical tip), shown in Fig. 1(a). Diameter of the micro-gripper is about 50 μm and distance between grip teeth is about 20 μm, shown in Fig. 1(b). Micro-tensile specimens were fabricated from a single crystal nickel disk with (111) crystal orientation purchased from the Nilaco Cooperation. The micro-gripper and micro-specimen for the micro-tensile test were both fabricated by a focused ion beam system (FIB, Hitachi:

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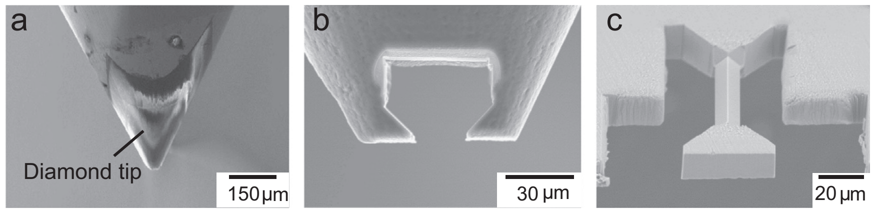

Fig. 1. SEM images of the (a) commercially available diamond-tip micro-indenter, (b) as-fabricated micro-gripper, and (c) as-fabricated micro-tensile specimen.

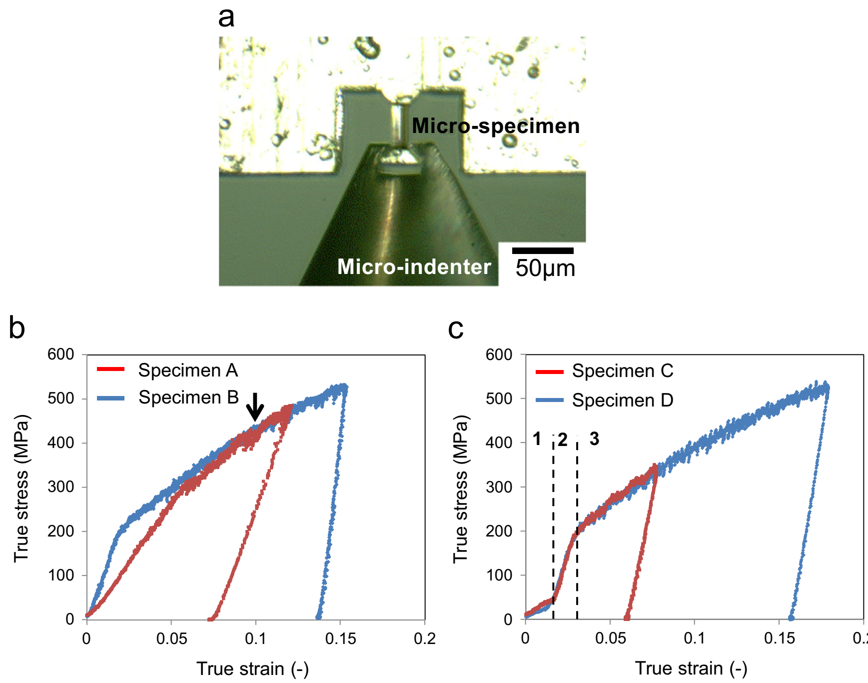

Fig. 2. True stress–true strain curves obtained from the micro-tensile test with (a) ⟨111⟩ and (b) ⟨223⟩ direction along loading axis.

FB2100) operated at 40 kV. Observation of the specimens was conducted using a scanning electron microscope (SEM, Hitachi: S-4500SE). Details of the fabrication procedures could be found in a previous report [12].

The micro-tensile test was conducted using specimens with two different crystal orientations. One of the specimens had orientation close to ⟨111⟩, and the one had a nearly ⟨223⟩. The difference in crystal orientation was ensured by making gauge part of the specimens to have ⟨111⟩ and ⟨223⟩ orientation and a square cross-section of 7.5×7.5 or $10 \times 10\ \mu m^2$ and 40 μm in the loading axis. In order to check repeatability of the results, one more ⟨111⟩ and ⟨223⟩ specimen each with size of $10 \times 10\ \mu m^2$ and 40 μm were prepared. Fig. 1(c) shows an SEM image of as-fabricated specimen having ⟨111⟩ crystal orientation at the gauge part. The crystal orientations against the loading axis were confirmed by electron backscattered diffraction (EBSD) analysis before the test.

The micro-tensile tests were conducted by controlling a constant displacement rate at 0.1 μm/s under a uniaxial loading using a testing machine designed for micro-sized specimens. The force and displacement were recorded every 33 milliseconds. The micro-tensile tests were stopped and unloaded before fracture of specimens for observation of surface of the specimens.

3. Results and discussion

In this work, we conducted micro-tensile tests for specimens with two different crystal orientations of ⟨111⟩ and ⟨223⟩ to evaluate the effect of crystal anisotropy. Fig. 2 shows true stress–true strain curves obtained from results of the ⟨111⟩ and ⟨223⟩ specimens. The ⟨111⟩ specimens having different value of elongation were indicated as specimen A and B, respectively, and then the ⟨223⟩ specimens were also indicated as specimen C and D as shown in Fig. 2(b) and (c), respectively. In Fig. 2(b), a few load drops in the stress–strain curve as indicated by the arrow were observed. Moreover, the ⟨111⟩ and ⟨223⟩ specimens showed different behaviors on the stress–strain curves as shown in initial region until 5 % true strain.

Fig. 3(a) and (b) shows SEM images of the ⟨111⟩ and ⟨223⟩ specimen before the micro-tensile test, respectively. Fig. 3(c) and (d) shows magnified images focusing at gauge part of the ⟨111⟩ and ⟨223⟩ specimens after the micro-tensile test, respectively. Large numbers of slip line were observed on the surface of both specimens while fewer numbers of slip were observed on the ⟨223⟩ specimen, which should be contributed by the difference in loaded strain on both sample.

The load drops observed in stress–strain curves and the SEM images showing surface of the specimens after the micro-tensile test were similar to previous work reported on tensile testing of single crystal copper micro-specimen [7]. The load drops are similar to the serration behavior, which suggest presence of the effects of distinct slip glide during the micro-tensile test. The load drops observed in Fig. 2(b) is weaker than that reported by Kiener et.al since the specimen size used in this study is larger than Kiener's. Effect of the distinct slip loading on load drop is size dependent. The changing in stability of the stress–strain behavior with specimen size was reported in other studies on micro-mechanical test [7,11].