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 $\langle 111 \rangle$ and $\langle 223 \rangle$ 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.

Keywords: Micro-tensile test, Micro-sized gripper, Micro-tensile specimen, Single crystal nickel

1. Introduction

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, effect 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: 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 $\langle 111 \rangle$, and the one had a nearly $\langle 223 \rangle$. The difference in crystal orientation was ensured by making gauge part of the specimens to have $\langle 111 \rangle$ and $\langle 223 \rangle$ orientation and a square cross-section of 7.5×7.5 or $10 \times 10 \mu\text{m}^2$ and 40 μm in the loading axis. In order to check repeatability of the results, one more $\langle 111 \rangle$ and $\langle 223 \rangle$ specimen each with size of $10 \times 10 \mu\text{m}^2$ and 40 μm were prepared. Fig. 1(c) shows an SEM image of as-fabricated specimen having $\langle 111 \rangle$ 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 $\mu\text{m/s}$ 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 $\langle 111 \rangle$ and $\langle 223 \rangle$ to evaluate the effect of crystal anisotropy. Fig. 2 shows true stress-true strain curves obtained from results of the $\langle 111 \rangle$ and $\langle 223 \rangle$ specimens. The $\langle 111 \rangle$ specimens having different value of elongation were indicated as specimen A and B, respectively, and then the $\langle 223 \rangle$ 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 $\langle 111 \rangle$ and $\langle 223 \rangle$ specimens showed different behavior on the stress-strain curves as shown in initial region until 5 % true strain.

Fig. 3(a) and (b) show SEM images of the $\langle 111 \rangle$ and $\langle 223 \rangle$ specimen before the micro-tensile test, respectively. Fig. 3(c) and (d) show magnified images focusing at gauge part of the $\langle 111 \rangle$ and $\langle 223 \rangle$ 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 $\langle 223 \rangle$ 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].

The critical resolved shear stress (CRSS), which is an index of primary slip at the corresponding slip system, is an important factor to consider strength of single crystal metals [13]. The CRSS is defined by Schmid's law given in the following equation.

$$\tau_c = \sigma_y \cos \varphi \cos \lambda \quad (1)$$

where τ_c is the CRSS, σ_y is the yield stress in tension, and φ is the angle between the loading axis and normal direction to the slip plane, λ is the angle between the loading axis and slip direction and then $\cos \varphi \cos \lambda$ is the Schmid factor. The CRSS for pure nickel is 3.24-7.35 MPa [14]. Table 1 shows the top six highest Schmid factors of the specimens belonging in the slip systems of $\langle 110 \rangle$ directions and $\{111\}$ planes, which are the easily occurred slip events for face-centered-cubic (fcc) materials [13]. In Fig. 2(a) and (b), stress of the two specimens is very close until ~23 MPa. Yield strength of the specimens can be calculated using results listed in Table 1 and the Schmid's law, and then calculated σ_y of the $\langle 111 \rangle$ and $\langle 223 \rangle$ specimen by using each maximum Schmid factor are 9.81-22.27 MPa and 7.36-16.70 MPa, respectively. The calculation indicated the region until 23 MPa is regarded as plastic deformation.

The difference in the stress-strain curves shown in Fig. 2 is an evidence of the effect of crystal anisotropy on the deformation behavior after the plastic deformation. In particular, deformation behavior of the $\langle 223 \rangle$ specimen can be divided into three stages as shown in dashed line in Fig. 2(c). Moreover, two specimens listed as specimen C and D were indicated almost same behavior on stress-strain curves in initial region until 5% true strain. The three-stage behavior for $\langle 223 \rangle$ specimen is typically observed in single crystal bulk metals with fcc structure [14]. The regions divided by the dashed lines are named stage 1, 2 and 3 from left to right, respectively. In stage 1, slip system having a high Schmid factor such as $[101](-1-11)$ or $[1-10](11-1)$ easily glides at a low tensile strength. In stage 2, a multiple slip event is occurred due to other glide in slip systems with lower Schmid factor than the one already worked in stage 1. Thus, the gradient in stress-strain curve increases, which is due to work hardening. In stage 3, the primary glide disturbed by multiple slip is continued to glide by translating it into another slip plane which is called cross slip event. Then the gradient in the region decreases because the work hardening is

lowered. In the final region of stage 3, the tensile test of the specimen D was conducted for a longer time than the specimen C. Therefore, the curve of the specimen D is extended to a higher point because of work hardening. However, the three-stage deformation behavior for the $\langle 223 \rangle$ specimen was not observed in the $\langle 111 \rangle$ specimen as shown in Fig. 2(b). For the $\langle 111 \rangle$ specimen, it is suggested that a multiple slip phenomenon was occurred at the initial region of plastic deformation, which is same as stage 1 observed for the $\langle 223 \rangle$ specimen. Then the difference comes from that there is no slip system having a Schmid factor that is much larger than the others as the case in the $\langle 223 \rangle$ specimen. In addition, the gap for the Schmid factor from the highest to the fourth is also much smaller when compared with the $\langle 223 \rangle$ specimen. This narrow gap in Schmid factor suggests that some slip systems having similar value on Schmid factor have probability of glide at the same time in initial region in the case of $\langle 111 \rangle$ specimen. Therefore, stress-strain behavior of specimen A and B in initial region until 5% true strain had different gradient contributed by the number of occurred slip systems. The behavior observed in the true stress-true strain curve for the $\langle 111 \rangle$ specimen is also similar to the stress-strain curve of for bulk single crystal metal composed fcc crystal structure having multiple slip [14,15].

4. Conclusions

In conclusion, this is the first report on the effect of crystal anisotropy for micro-specimens using a micro-tensile test. From the micro-tensile test, a few load drops were observed in the stress-strain curve, which are considered to be corresponded to the occurrence of distinct slip glides. Moreover, we found that the deformation behavior could be differentiated from Schmid factor of the specimens with different crystal orientation. Micro-tensile test results of the $\langle 111 \rangle$ and $\langle 223 \rangle$ specimen indicated different deformation behaviors which are similar to multiple slip behavior. Moreover, the results obtained in this study indicated that the micro-tensile test proposed can be applied to evaluate the crystal anisotropy of micro-components composed of materials other than nickel or other crystal structure.

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Captions

Table 1 Highest to the sixth highest value of Schmid factor in the slip systems of $\langle 110 \rangle$ direction and $\{111\}$ plane.

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

Figure 2. True stress-true strain curves obtained from the micro-tensile test with (a) $\langle 111 \rangle$ and (b) $\langle 223 \rangle$ direction along loading axis.

Figure 3. SEM images of the specimens before and after the micro-tensile test. (a) and (b) are SEM images of as-fabricated SCNi specimen with $\langle 111 \rangle$ and $\langle 223 \rangle$ orientation, respectively. (c) and (d) are gauge part of the micro-specimens (a) and (b) after the micro-tensile test, respectively.

Figure 1

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

Figure 2

Figure 2. (a) Optical image of the micro-gripper and micro-tensile specimen before the micro-tensile test and true stress-true strain curves obtained from the micro-tensile test with (b) $\langle 111 \rangle$ and (c) $\langle 223 \rangle$ direction along loading axis.

Figure 3

Figure 3. SEM images of the specimens before and after the micro-tensile test. (a) and (b) are SEM images of as-fabricated SCNi specimen with $\langle 111 \rangle$ and $\langle 223 \rangle$ orientation, respectively. (c) and (d) are gauge part of the micro-specimens (a) and (b) after the micro-tensile test, respectively.

Table 1 Highest to the sixth highest value of Schmid factor in the slip systems of <110> direction and {111} plane.

Loading axis: <111>

Slip system	Schmid factor
[110](-11-1)	0.33
[0-11](111)	0.32
[-101](1-11)	0.30
[-101](111)	0.28
[0-11](-111)	0.19
[110](-111)	0.19

Loading axis: <223>

Slip system	Schmid factor
[101](-1-11)	0.44
[1-10](11-1)	0.43
[-101](1-11)	0.35
[110](-11-1)	0.35
[0-11](111)	0.09
[0-11](-111)	0.08

**Evaluate Crystal
Anisotropy by the
Micro-Tensile Test**

Graphical Abstract