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Tensile behavior of micro-sized specimen fabricated from nanocrystalline nickel film

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ABSTRACT

This paper reports mechanical properties and tensile behavior of nickel films with average grain size of 8 nm. The nickel films were obtained from electroplating with supercritical carbon dioxide emulsion (EP-SCE). Micro-tensile test using a micro-gripper and micro-specimen both fabricated by focused ion beam was conducted to evaluate the mechanical properties, and the tensile behavior was observed by a scanning electron microscope (SEM). The micro-tensile specimen showed a high tensile strength of 3.0 GPa, yield strength of 2.4–2.5 GPa and plastic strain of 3–5%. SEM observation of the fractured micro-specimen after the micro-tensile test showed typical fractured patterns of ductile materials, which are necking phenomenon, shear lip and dimple pattern. This fractured mode is called cup and corn fracture. The high strength and ductility are suggested to be effects of grain refinement and suppression of grain boundary sliding by carbon contents introduced during the EP-SCE.

1. Introduction

Recently, demands on fabrication and evaluation of micro-components are increasing in the industrial field for miniaturization of the electronic devices. In particular, development of micro-electro-mechanical systems (MEMS) gathers a high attention because of the high applicability. For development of MEMS, fabrication and characterization of the micro-components are important for evaluating reliability of the MEMS devices. Electroplating method is often used in fabrication of the micro-components for MEMS, because the technique can be used to fabricate complicate metallic micro-patterns [1]. Moreover, properties of the materials electrodeposited, such as crystal orientation and grain size, can be easily controlled by the experimental conditions [2,3].

In our group, a new electroplating technique using supercritical carbon dioxide emulsified electrolyte (EP-SCE) was developed for fabrication of the micro-components [4,5]. The electroplating technique was reported to cause the effects of grain refinement and surface smoothening by a phenomenon called periodic-plating-characteristic [6]. The nickel films fabricated by EP-SCE are reported to be composed of nanocrystalline structure [7]. The nickel films fabricated by EP-SCE are promising material to be used in MEMS because the nanocrystalline metals are expected to give high strength and isotropic property [8].

Evaluating mechanical properties, such as strength and elongation, of the materials in micro-scale is important to determine reliability of the micro-components used in MEMS. In general, micro-compression and micro-bending test are usually conducted to understand the mechanical properties [9,10]. However, these tests cannot provide fractured strength and elongation properties of the micro-specimens. In the case of application into industrial material, fracture strength of the materials is an important property. Therefore, micro-tensile test is needed to evaluate the properties, but there are only a few reports on micro-tensile test [11]. The tensile test in micro-scale is difficult to conduct because alignment between the gripper and the specimen is difficult. In addition, preparation of the gripper for the micro-tensile test is also difficult. In this study, a micro-tensile test is conducted using a micro-gripper and micro-tensile specimen fabricated from a commercially available diamond tip micro-indenter and EP-SCE nanocrystalline Ni, respectively. The mechanical properties and fracture behavior are investigated from the micro-tensile test.

2. Experimental

2.1. Materials

Cu substrate with 99.99% purity was used to electroplate Ni after a pretreatment step. For the pretreatment, degreasing was

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conducted using two kinds of solution, which are 10 wt.% degreasing solution purchased from Okuno Industry Co., Ltd. and 10 wt.% HCl solution, for 1 min and 10 s, respectively. The Ni electrolyte was additive-free Watts bath composed of $NiSO_4{\cdot}6H_2O$ (300 g/l), $NiCl_2{\cdot}6H_2O$ (50 g/l), and H_3BO_3 (50 g/l). CO_2, and non-ionic surfactants, polyoxyethylene lauryl ether ($C_{12}H_{25}(OCH_2CH_2)_{15}OH$) were added to the aqueous electrolyte in a pressurized chamber. A micelle structure was formed when the emulsion is stirred using an agitator in the chamber [6]. Ni was electrodeposited on the pretreated Cu substrate at a constant temperature of 323 K, current density of 2 A/dm^2 and pressure of 15 MPa. Moreover, the Ni films fabricated in this condition were found to be composed of grains with average size of 8 nm [7,12].

2.2. Fabrication of the micro-gripper and micro-specimen

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 gripper and the specimen was both conducted using a scanning electron microscope (SEM, Hitachi: S-4500SE). The micro-gripper for the micro-tensile test was fabricated from a commercially available diamond-tip micro-indenter originally designed for a micro-bending test (Synton-MDP: Spherical tip). Fabrication scheme of the micro-gripper and a SEM image of the as-fabricated micro-gripper are showed in Fig. 1. Black and blue regions as shown in Fig. 1a are diamond tip of the micro-indenter. The grip part was fabricated by removing the blue region by FIB milling. Fig. 1b shows a SEM image of the as-fabricated micro-gripper taken along the direction of FIB irradiation. Diameter of the micro-gripper is about 50 μm.

Regarding the micro-specimen, the Ni film fabricated by EP-SCE was sliced and mechanical polish to thin down to about 30 μm in thickness. Then the FIB was utilized to obtain a final shape of the micro-specimen. A FIB fabrication scheme and SEM image of the as-fabricated micro-tensile specimen are shown in Fig. 2. At first, the Ni film was milled roughly by the FIB, as shown in Fig. 2a. Secondly, the gripped part and gauge part were milled to a shape as shown in Fig. 2b. Gripped part of the micro-tensile specimen has a similar shape to the micro-gripper and was milled by FIB irradiation from the same direction as shown in Fig. 1b. Finally, gauge part of the micro-tensile specimen would have a square cross-section of $10 \times 10\ \mu m^2$ and 40 μm in the direction of long axis. More detail of the fabrication method can be found in a previous work on micro-compression test [13]. Fig. 2c shows a micro-tensile specimen with the final shape. With this configuration, there will be no influences from the film thickness to the gauge part.

2.3. Micro-tensile test and morphology observation

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. More details of the testing machine are described in a previous study [14]. Fig. 2d shows an optical image of the micro-tensile specimen and micro-gripper before the micro-tensile test taken by a CCD camera equipped on the testing machine, and then the micro-tensile specimen was loaded by moving the micro-gripper to an uniaxial direction only. The force and displacement were recorded every 33 ms. The fracture mode was evaluated by observing the fractured surface by SEM.

3. Results and discussion

3.1. Mechanical properties

Fig. 3 shows engineering stress-true strain curves obtained from the micro-tensile test. The micro-tensile test was conducted using two micro-specimens labeled as specimen A and B in the figure. The micro-tensile test for specimen A was conducted until fracture of the specimen. The micro-tensile test for specimen B was stopped and unloaded before failure of the specimen, which allowed observation of surface of the deformed specimen. In Fig. 3, both specimens show maximum stress of 3.0 GPa, yield strength of 2.4–2.5 GPa and plastic strain of 3–5%. After yielding, similar behavior to work softening at small strain were observed in both of the specimens. This behavior was also found in results of tensile test using bulk specimen composed of nanocrystalline structured metal [15,16]. In the case of nanocrystalline metallic materials, inhomogeneous deformation like a necking phenomenon is easily occurred at small strain compared with materials with large grain size because of low strain hardening rate and high strength [15]. Thus, the strain softening after yielding is considered to be an effect of decrease in diameter of the specimen by inhomogeneous deformation.

3.2. Deformation behavior

Observation of the before-fractured surface and fractured surface of the micro-specimens was conducted after the micro-tensile test for specimen B and A, respectively. Surface of specimen B is shown in Fig. 4a. Necking phenomenon often observed in ductile materials was observed at the gauge part, as shown in a magnified view above the specimen in Fig. 4a. On the other hand, deformation with shear bands was not occurred in the micro-tensile test. In a previous study on micro-compression test, shear band was observed in a micro-pillar made from Ni film with grain size lower than 15 nm [17], which is a fractured pattern of brittle materials. Fig. 4b and c show top-view and a view with tilted angle of 45 degree of the fracture surface of specimen A, respectively. A fracture pattern called shear lip at edge of the specimen was observed, indicated by white arrows in Fig. 4b. Moreover, in Fig. 4c, dimple patterns near center of the specimen were also observed. This fracture mode, referring to the dimple and shear lip, is called cup and corn fracture. This fracture mode is often observed in ductile

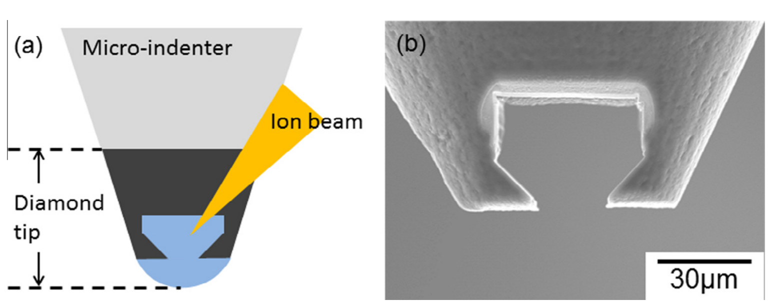

Fig. 1. (a) Schematic image showing fabrication method of the micro-gripper for the micro-tensile test and (b) SEM image of the as-fabricated micro-gripper.