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Sample size effect on micro-mechanical properties of gold electroplated with dense carbon dioxide

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ABSTRACT

Micro-mechanical properties of gold electroplated with dense carbon dioxide contained electrolyte (EP-DCE) were investigated by micro-compression tests using micro-pillar specimens for applications as movable components in MEMS devices. The effect of grain size on the micro-mechanical properties was also investigated using a gold film fabricated by conventional electroplating (CONV-EP). The gold film fabricated by the EP-DCE showed a finer average grain size when compared with that of the CONV-EP. Because of the finer grain size, the micro-pillar fabricated from the EP-DCE film showed a higher strength than that of the micro-pillar fabricated from the CONV-EP. An increase in the compressive flow stress from 740 to 810 MPa was observed as dimensions of the micro-pillars decreased from 20 × 20 × 40 to 10 × 10 × 20 μm^3, which was a result of the sample size effect.

1. Introduction

Gold materials prepared by electroplating (EP) are widely used in electronics industries as conducting materials for interconnections owing to their superior properties such as high chemical stability, corrosion resistance, electrical conductivity, and density [1,2]. Recently, micro-electronic-mechanical systems (MEMS) accelerometers utilizing movable components made of gold-based materials were reported to have better sensitivity than the conventional silicon-based devices [3]. However, gold is known to be a relatively soft material (i.e., yield strength of bulk gold: 55–200 MPa [4]), especially when compared with the silicon [5]. The low mechanical strength becomes a concern in further miniaturization of the device. Therefore, strengthening of the gold materials is required to allow further improvement of the MEMS accelerometer.

Grain boundary strengthening mechanism based on Hall–Petch relationship is often applied in strengthening of metallic materials [6–8]. Strengthening of the metallic materials is achieved when the average grain size (d_g) reaches submicron- or nano-scale [9]. On the other hand, d_g of electroplated metallic materials can be easily controlled by the electroplating parameters such as the current density, pH value, and plating bath temperature [10–12]. An alternative electroplating process conducted in high pressure environment, pressurized by introducing dense carbon dioxide into the electrolyte, was reported to be effective to refine the d_g to nano-scale (d_g < 100 nm) as demonstrated in electroplating of Ni and Cu [13–16]. A significant enhancement in the mechanical property was reported in Ni films fabricated by the electroplating with dense carbon dioxide contained electrolyte (EP-DCE) method [17]. Through application of the EP-DCE, further grain refinement in d_g of the electroplated gold can be realized and leads to further enhancement in the mechanical strength.

Mechanical properties of materials having dimensions in micro-scale are different from those of bulk materials. The phenomenon is known as the sample size effect. Since the first report of the sample size effect in single crystal specimens [18], many subsequent investigations have demonstrated the effect in the mechanical strength of specimens composed of fcc and bcc metals when dimensions of the specimen are decreased to micro- or nano-scale [19,20].

In order to utilize the grain boundary strengthening mechanism, the specimen evaluated in this study will be fabricated from gold films prepared by the EP-DCE method, which will be composited of gold crystals having the d_g in nano-scale order [21]. Components in MEMS devices often have dimensions in micrometer or tens of micrometer order. For practical applications of the EP-DCE in fabrication of

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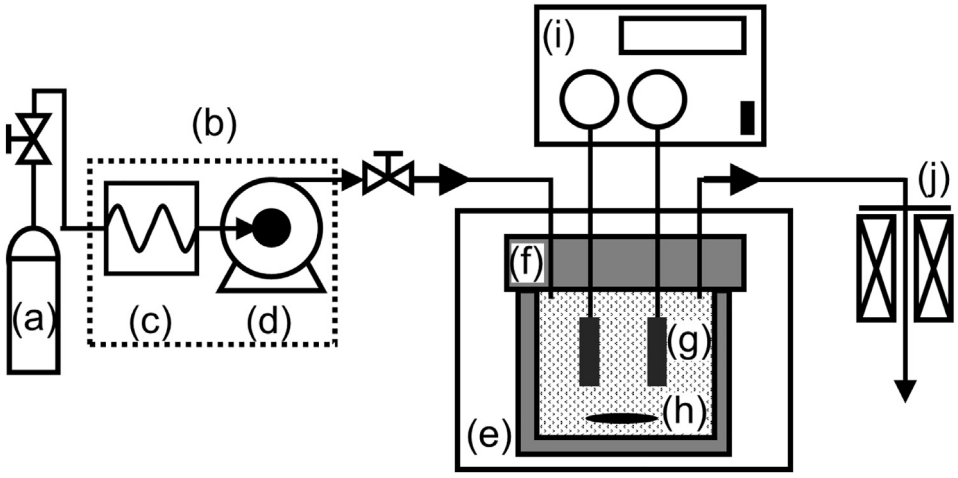

Fig. 1. High pressure apparatus: (a) CO_2 gas tank, (b) CO_2 pressurization unit, (c) liquidization unit, (d) high-pressure pump, (e) box oven, (f) reaction cell, with PEEK coating on the inner wall, (g) substrates, (h) stirrer, (i) programmable power supply, and (j) back-pressure regulator.

components used in MEMS devices, it is necessary to evaluate the mechanical property using specimens having dimensions in micro-scale. For polycrystalline specimens, there are limited reportes on the sample size effect [22–24], especially for specimens composed of gold and having dimensions in micro-scale.

In this study, mechanical properties of gold films fabricated by the EP-DCE will be investigated by micro-compression tests using non-tapered micro-pillars fabricated from the electroplated gold films. Gold micro-pillars with three different dimensions, which are 20 × 20 × 40, 15 × 15 × 30, and 10 × 10 × 20 μm^3, and the same aspect ratio would be fabricated by focus ion beam (FIB) system. Micro-compression tests with a displacement controlled mode would be applied to evaluate the mechanical property.

2. Experimental

Details of the high pressure electroplating apparatus are shown in Fig. 1. The gold electrolyte used in this study was a commercially available sulfite-based gold electrolyte purchased from Matex Japan (Matex Gold NCA). The electrolyte was composed of 50 g/L of Na_2SO_3, 50 of g/L $(NH_4)_2SO_3$, 21.63 g/L of $Na_3[Au(SO_3)_2]$ with pH of 8.0 and 5% sodium gluconate. For the EP-DCE, pressure in the reaction cell was elevated by pressuring liquefied CO_2 into the cell and was controlled at 10 MPa during the entire electroplating process by a back-pressure regulator. A piece of gold film fabricated by the conventional electroplating (CONV-EP) method, which was conducted at atmospheric pressure, was prepared to be used as a comparison with the EP-DCE. The current density and the plating temperature for both CONV-EP and EP-DCE were fixed at 5 mA/cm^2 and 40 °C, respectively. Cu plates and Pt plates with the same dimensions of 1.0 × 2.0 cm^2 were used as the cathode and the anode, respectively. Thickness of the gold films was controlled at about 50 μm.

Micro-pillars with a square cross-section and an aspect ratio of 2:1, which is the long-side over one side of the square cross-section, were fabricated from the gold films using FIB (Hitachi FIB-2100). Dimensions of the micro-pillars were either 20 × 20 × 40, 15 × 15 × 30, or 10 × 10 × 20 μm^3. Details of the fabrication method are reported in a previous work [17]. The Ga ion implantation from the FIB fabrication process is ignored due to shallow penetration depth of the Ga ions (less than few tens of nanometer) [25,26] when compared to dimensions of the specimen evaluated in this study. The micro-compression test was performed with a testing machine equipped with a diamond flat-punch indenter. The micro-compression test was conducted at a constant displacement rate of 0.1 μm/s using a piezo-electric actuator. A strain gage type load cell was used to record the data of displacement and load at a rate of 30 points per second. The engineering strain-stress (*SS*) curves were subsequently converted by the following equations:

$$\sigma = P/A_0 \quad (1)$$

$$\varepsilon = L/L_0 \quad (2)$$

where σ is the engineering stress, P is the load, A_0 is area of top surface of the micro-pillar, ε is the engineering strain, L is the displacement, L_0 is the length of micro-pillar. The microstructure and deformation behaviors of the gold micro-pillars were observed using a scanning ion microscope (SIM) equipped within the FIB and a scanning electron microscope (SEM, JEOL 7500F).

3. Results and discussion

3.1. Mechanical properties of gold micro-pillars with different grain size

Fig. 2 shows SIM images of the micro-pillars fabricated from the CONV-EP and the EP-DCE gold films before and after the micro-compression tests. Dimensions of the two pillars were both 20 × 20 × 40 μm^3. Camouflage-like patterns were observed on surface of side-wall of the CONV-EP pillar. Different level of brightness observed in the patterns of SIM image, as shown in Fig. 2(a) and (b), are usually results of the difference in crystal orientation. Hence, boundaries of the patterns are often identified as boundaries of grains. For the CONV-EP gold film, the d_g was ranged in several hundreds of nanometer as shown in the SIM image. d_g of the CONV-EP film prepared under the same condition was ~0.8 μm evaluated by electron backscatter diffraction reported in a previous study [21]. After the compression test, the pillar showed typical deformation behaviors of polycrystalline structures [23], which the pillar deformed into the shape of a barrel as shown in Fig. 2(b).

On the other hand, patterns with different level of brightness were also observed on surfaces of the EP-DCE pillar, but the sizes were much smaller than those in the CONV-EP pillar, which implied d_g of the EP-DCE film was much smaller than that of the CONV-EP film. The d_g was roughly estimated from side-wall of the EP-DCE pillar, Fig. 2(c), and the d_g was below 100 nm. In a previous study on gold electroplating with dense carbon dioxide, the gold film fabricated under the same conditions used in this study showed an average grain size at ~13 nm [21]. The grain refinement effect observed in the EP-DCE film is suggested to be caused by the same mechanisms reported in electroplating of Ni using the EP-DCE, which are the periodic-plating-characteristics (PPC) [14] and co-deposition of carbon contents from reduction of carbon dioxide dissociated in the electrolyte [13,16,21]. Similar barrel-shape deformation behavior was observed in the EP-DCE pillar after the

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