Fabrication of Electrochemical Micro-reactor by Photon Rupture Method and Electrochemistry

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Introduction

Micro-reactor and micro miniaturized total analysis system (μ-TAS) have been studied because of reduce system size and west solutions and chemicals, high reaction rate. These equipments were fabricated on silicon, grass and organic sheet by mask process, namely photo lithography and Galvano-fomung und Abformung (LIGA) process. However, these process use harmful chemicals and take long time to fabricate.

Recently, oxide film stripping by photon rupture (focused pulsed Nd-YAG laser beam irradiation) method has been reported[1)]. The irradiation of a pulsed laser beam is able to strip the oxide film at extremely high rate without any contamination from film stripping tools. This technique has been applied to form 2 dimensional and 3 dimensional metal and organic structure[2-4)], and micro pores and grooves on aluminum substrate[5)].

The purpose of this study is to form electrochemical micro-reactor on aluminum by using photon rupture method and electrochemistry, and to investigate electrochemical performance of the reactor.

Experimental

Specimen : A pure aluminum sheets, 99.9mass %, 1.0 mm thick, were cut into 15 x 15 mm^2 with a handle. The specimens were degreased in an ethanol ultrasonic bath, mechanical polishing, and the electro-polished in a mixture of 70 mass % perchloric acid and acetic acid, volume ratio 1 : 4, at a constant voltage of 28 V, cell voltage at 286 K. After electro-polished the specimens were rinsed in doubly distilled water and acetone.

Anodizing : Porous type anodic oxide films were formed by anodizing at 293 K in 0.22 $kmol/m^3$ oxialic acid solution with a constant current density, i_a = 100 A/m^2 for t_a = 3.6 ks. After anodizing, specimens were dying dipped in 0.03 $kmol/m^3$ alizarin red S solution for 300 s at 323 K, and sealing in boiling distilled water for 600 s.

Laser irradiation : After anodizing, the specimen was immersed in solutions and irradiated by pulsed Nd - YAG laser (Sepctra Physics GCR - 130) through a lens and quartz window. The laser beam was the second harmonic wave, wave length was 532 nm, wave duration was 8 ns and frequency was 10 s^{-1}. The irradiation time, t_i, was changed between 0.18 s and 3.6 ks. The laser power, P, was 30 mW. To fabricate micro-channel, through hole and micro-electrochemical cell, the distance between the specimen and lens, and the irradiated position were controlled by an XYZ stage.

Au plating and re-anodizing : After fabricated micro-channel and through hole, sample was anodized again in the solution. And then fabricated micro cell, after that gold was deposited on the electrode surface by electrochemically. Figure 1 shows schematic drawing of cross section of electrochemical micro-reactor, which total solution volume is 1.5 μl.

Cyclic voltammetry : Cyclic voltammogram of the micro-electrochemical cell was measured in 2 mol/m^3 and 20 mol/m^3 $K_3Fe(CN)_6$ / $K_4Fe(CN)_6$ at static condition at different sweep rate.

Results

Figure 2 shows cyclic voltammogram obtained from micro-electrochemical reactor in 20 mol/m^3 $K_3Fe(CN)_6$ / $K_4Fe(CN)_6$ at static condition. Oxidation and reduction peak can be seen. The peak currents of both reduction and oxidation increase with increasing sweep rate.

Reference

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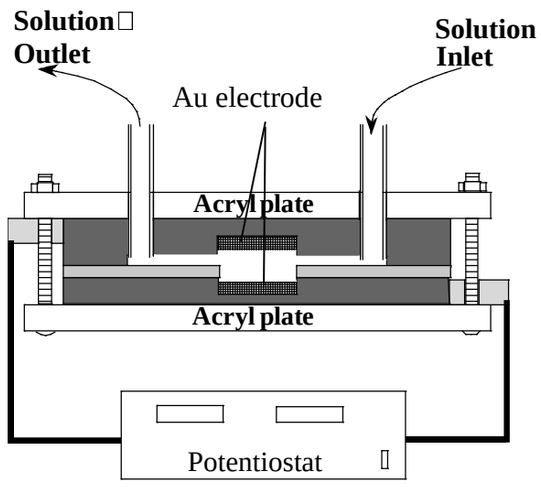

Fig. 1 Schematic drawing of the cross section of electrochemical micro reactor and electrochemical unit.

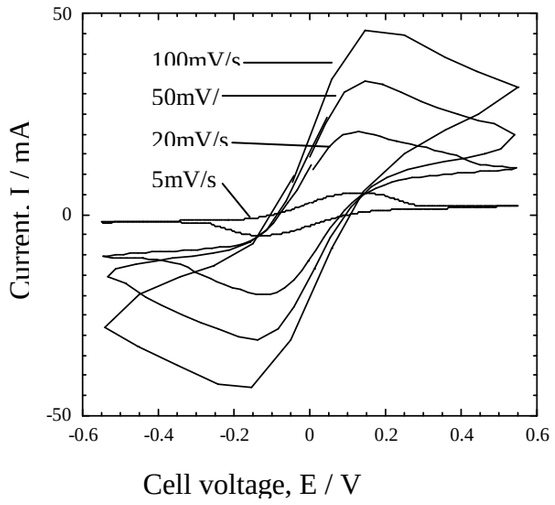

Fig. 1 Cyclic voltammogram obtained from micro-reactor in 20 mol/m^3 $K_3Fe(CN)_6$ / $K_4Fe(CN)_6$. Solution volume : 1.5 ul