Fabrication of hierarchical micro- and nano-structures on high purity aluminum plate using micro-indentation and anodic aluminum oxidation processes

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This paper presents a simple and cost-effective method for fabrication of the hierarchical micro–nano structure using micro-indentation and anodic aluminum oxidation processes on high purity aluminum substrate. To improve the surface roughness of high purity aluminum plate surface, ultra-fine planning with a single crystal diamond tool of 50mm in radius was used. Surface roughness of high purity aluminum plate was improved from 8~9μm to 20~25nm. Micro-indentation process was performed to fabricate a micro-pattern on the high-purity aluminum plate. Indentation tool was used the diamond tools of 25μm in radius. From micro-indentation process, hemispherical microstructures of 40μm-diameter and 10μm-depth were formed on aluminum surface. Using anodic aluminum oxidation technique, nano-pores of 200~300 nm in diameter were formed on the hemispherical microstructures. Polymer replica from the hierarchical micro- and nano-pores were fabricated by hot embossing of PMMA and UV embossing of NOA-61.

1. Introduction

Recently, micro-electromechanical systems and nanotechnologies are being used in various applications such as electrochemical nanobiochips, nanophotonics, ubiquitous sensors and nanopatterned displays. All of these applications require micro- and nano-patterns. In some special cases, micro–nano hybrid patterns are demanded to improve their performance.

Micropatterns are easily fabricated by micromachining technologies or precision mechanical machining. Sometimes, these micropatterns with various sizes and shapes are used as the mold for the injection molding and hot-embossing process for the multiple replications. In addition, nanopatterns fabricated by e-beam lithography, hologram lithography and nanoprobe lithography are also variously applied to the bio and optical applications. Also micro- and nano-pattern technology has been in the spotlighted in technology to implement surface structural coloring. [1-2]

Various fabrication methods for micro- and nano-scale structure have been developed. Conventional processes such as photo-lithography, electron-beam and focused ion beam (FIB) processes have guaranteed several micro- and nano-meters patterns. Moreover arbitrary micro- and nano-patterns could be fabricated. However, conventional methods are generally required for the high cost, long processing times, limited in applying to a large area, and are complicated processes. [3-6]

In this study, a simple and cost-effective method was presented for fabrication of the hierarchical micro–nano structure using micro-indentation and anodic aluminum oxidation processes on high purity aluminum substrate. This method was to fabricate a mold composed of micro- and nano-structure using mechanical and electrochemical methods. First, a micro structure was fabricated by using micro-indentation method. Then nano-holes were fabricated by Anodic Aluminum Oxidation (AAO).[7] Polymer replica from the hierarchical micro- and nano-pores were fabricated by hot embossing and UV embossing methods.

2. Fabrication of hierarchical micro- and nano-structures

2.1 Mold Fabrication

Figure 1 shows the hierarchical micro- and nano-structures fabrication process diagram. A pure aluminum (99.999%) plate (110 mm × 110 mm) of 5mm in thickness was used for fabrication of hierarchical micro- and nano-structures. Commercialized pure aluminum plates have bad surface roughness of 8~9 μm (Fig.1-step1). For that reason, mechanical ultra-fine planning was performed on high purity aluminum plate to obtain mirror-like surface.[8] Ultra-fine planning used a single crystal diamond tool having a radius of 50 mm. Mirror-like surface process was carried out by a pitch of 50 μm and DOC (depth of cut) of 35 μm as shown in Fig.1-step2. After ultra-fine planning, surface roughness, R_a, of high purity aluminum plate was improved from 8~9 μm to 20~25 nm (Fig.1-step3). Ultra-fine planning conditions were shown in Table 1. Micro-indentation process was performed to fabricate a micro-pattern on the mirror-like surface of high-purity aluminum plate. Indentation tool was the diamond tools with 25 μm in radius. Hemispherical micro-pattern array of the pitch 50 μm was fabricated by the micro-indentation process. The indentation process was performed by applying a voltage of 2.4 V at solenoid actuator (Azbil Co.). After micro-indentation process, hemispherical microstructures of diameter 40 μm and depth of 10 μm were fabricated. Conditions of micro-indentation process were shown in Table 2.

On micro-structured high purity aluminum plate, nano-holes were fabricated by anodic aluminum oxidation process (Fig.1-step4). Platinum was used as counter electrode in AAO process. Also, anodic oxidation and etching were performed in the electrolytic equipment as shown in Fig.2. The AAO equipment constituted power supply and cooling plate which controlled the temperature of aluminum substrate.

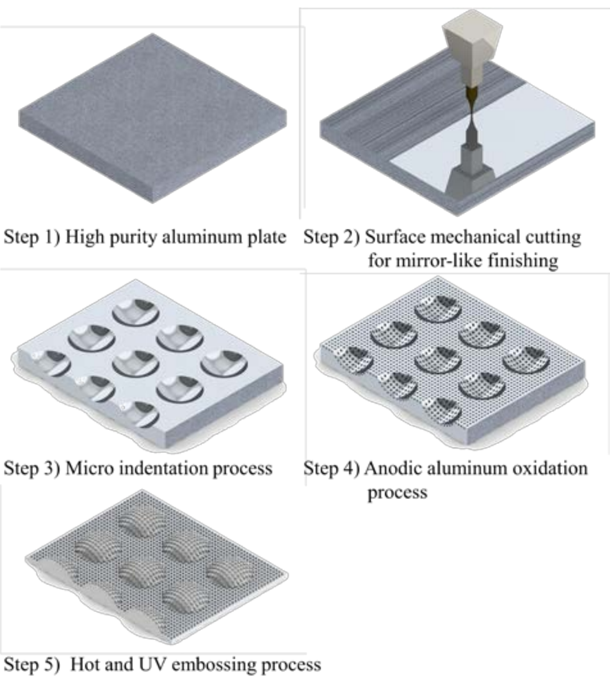

Fig. 1 Fabrication process of hierarchical micro- and nano structures

As the electrolyte for AAO process, 0.1 M phosphoric acid was used. The process was performed for 30 min. at the aluminum temperature of -10°C, and voltage of 200 V. As a result, nano-pores of 200~300 nm in diameter were formed on the micro- structured aluminum substrate. Figure 3 shows the photograph of hierarchical micro- and nano-holes on high purity aluminum substrate, SEM images of nanoholes.

Table 1 Detail conditions of ultra-fine planning

Cutting tool	Diamond tool radius 50 mm
Work piece (Area)	High purity aluminum (99.999%) (110 mm × 110 mm)
Pitch	50 μm
Depth of cut	35 μm
Cutting oil	ISOPAR-H Mist

Table 2 Detail conditions of micro-indentation

Cutting tool	Diamond tool radius 25 μm
Work piece (Area)	High purity aluminum (99.999%) (110 mm × 110 mm)
Pattern shape (Dimension)	Hemispherical pattern (diameter 40 μm, depth 10 μm)
Pitch	50 μm
Input voltage	2.4 V
Actuator	Solenoid (Azbil Co.)

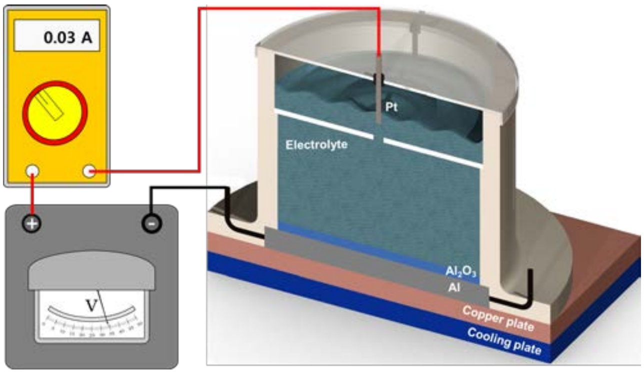

Fig. 2 Schematic diagram of anodizing system

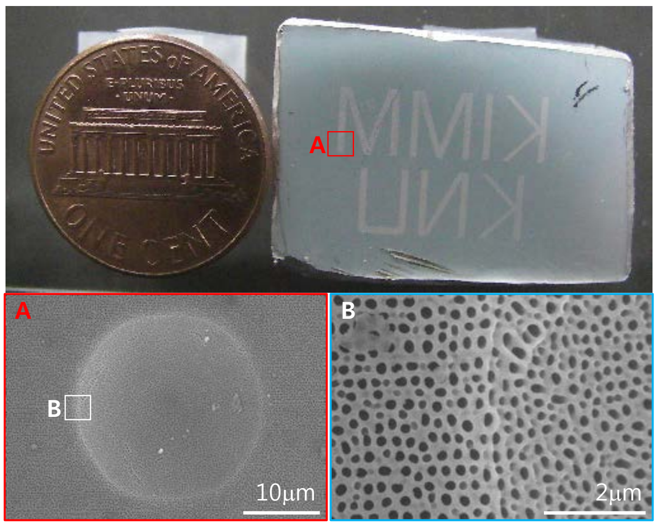

Fig. 3 Image of hierarchical micro- and nano-structure mold master and SEM image

2.2 Replication

For replication of mold, hot and UV embossing were used.(Fig.1-step5) Hot or thermal embossing is one of the most promising fabrication techniques for the micro–nano molding of a thermoplastic polymer that has a sub-10 nm resolution and high reproducibility.[9-10] In hot embossing process, the high purity aluminum plate with concave hierarchical micro- and nano-structures fabricated by using micro-indentation and AAO processes was used as a mold in order to replicate the convex hierarchical micro- and nano-structures on the polymethyl methacrylate (PMMA) sheet. The hot embossing system consists of upper and lower blocks with heating and cooling units operated by an air compressor. The mold was fixed on the top of the lower block, and a PMMA sheet was placed on the mold.

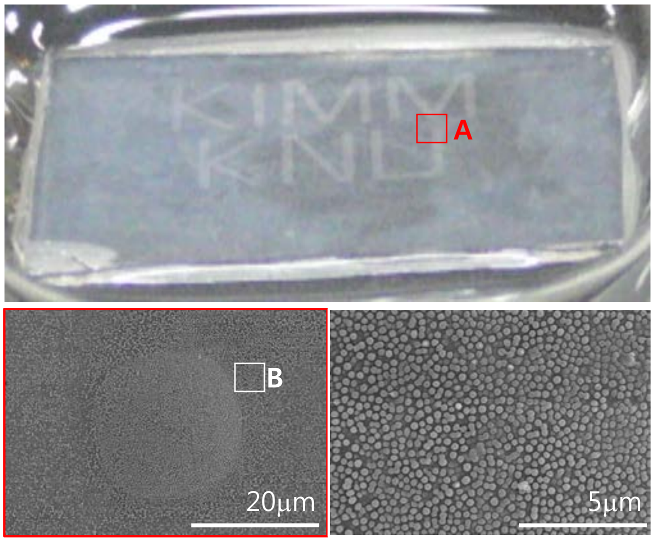

Fig. 4 Image of replicated convex micro- and nano-structures on the PMMA sheet

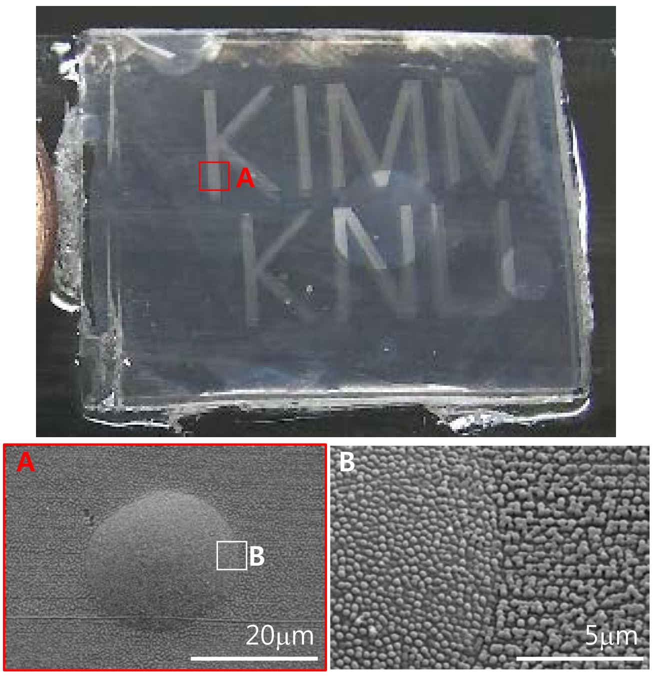

Fig. 5 Image of replicated convex micro- and nano-structures on the UV resin

The mold and the sheet were then compressed together after heating both the upper and the lower blocks to the desired temperature. The temperature of both the blocks in the hot embossing system was maintained to 60°C at an idling state. An embossing load of 7.0 kN was applied for 60 s after the temperature of both the blocks was increased up to 130°C within 30 min. Finally, both the heated blocks were cooled down to 60°C within 5 min, and then the PMMA replica was released from the mold. The hot embossing process was generally performed around the glass transition temperature of a polymer plate. The glass transition temperature of a PMMA plate ranges from 80 to 165°C. In this study, PMMA with a glass transition temperature of 110°C was used. Considering the heat dissipation in ambient air and the thermal conductivity of PMMA, we determined the hot-embossing temperature to be 135°C, which was slightly higher than the glass transition temperature of the PMMA sheet. As shown in Fig.4, nano-structure of 200~300nm on the micro structure were successfully fabricated by the hot embossing.

UV embossing was performed, and replica was compared with replica prepared by hot embossing. First, slide glass was cleaned thoroughly. Then, the surface of glass was treated by oxygen plasma, and then 3-Aminopropyl triethoxysilane (3-APTES) was coated on the glass to enhance the bonding strength of glass surface and UV resin by dipping in the solution at 85°C.[9] On the other hand, Teflon-AF was coated on aluminum mold to reduce the surface energy for easy detach of aluminum mold and UV replica. 0.03wt% Teflon-AF was coated, and then the aluminum mold was baked at 155°C for 20 min. and 175°C for 20 min.

UV embossing was performed using UV resin of NOA-61 (Norland Inc.). In order to increase UV resin filling into nano-pores, UV embossing process was carried out at 50 °C. Figure 5 shows photograph and SEM images of replicated micro- and nano-structures. Both hot and UV embossing methods are suitable to replicate micro- and nano-structures. However processing time of UV embossing was slightly faster than that of hot embossing process.

3. Conclusions

This paper presents a simple and cost-effective method for fabrication of the hierarchical micro–nano structure using micro-indentation and anodic aluminum oxidation processes on high purity aluminum substrate. Using ultra-fine planning process, the surface roughness of high purity aluminum plate was improved. Surface roughness of high purity aluminum plate was reduced as much as 20~25nm. Micro-patterns was formed by micro-indentation process with diamond tool of 25μm-radius. From micro-indentation process, hemispherical microstructures of 40μm-diameter and 10μm-depth were formed on aluminum surface. Using anodic aluminum oxidation technique, nano-pores of 200~300 nm in diameter were formed on the hemispherical microstructures. Nano-structure of 200~300 nm on the micro-structure of 40 μm were successfully fabricated by the hot embossing as well as UV embossing process. Hierarchical micro- and nano-structures could be applied to super-hydrophobic surface and structural coloring applications.

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