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〈연구논문〉

Lateral growth of PEO films on Al7050 alloy in 0.1 M $NaAlO_2$

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Abstract

This paper investigated generation behavior of micro-arcs and growth behavior of PEO films on the AA7050 disc specimen in 0.1 M $NaAlO_2$ solution under the application of 1200 Hz anodic pulse current. Morphologies, thickness and surface roughness of PEO films were examined at the edge part and central part separately. Micro-arcs were generated first at the edge part and then moved towards the central part with PEO treatment time, indicating lateral growth of PEO films. The lateral growth resulted in uniform PEO thickness of about 5 μm and surface roughness of about 0.5 μm. Moving of the arcs from the edge towards the central part appeared only one time and large size arcs were generated at the edge before completing the central part with small size micro-arcs. This suggests that vertical growth starts before completing the lateral growth. Large size arcs generated at the edge resulted in the formation of relatively large size pores within the PEO films on the AA7050 disc specimen.

Keywords : Plasma electrolytic oxidation, Lateral growth, Anodic oxide film, AA7050

1. Introduction

Aluminum alloy is one of the most widely used engineering materials for automotive, aerospace and structural applications due to its high strength-to-weight ratio. Aluminum alloys are usually used after anodizing treatment for improved corrosion resistance [1-6]. In order to improve the resistance of Al alloys against corrosion and wear, their surfaces can be also covered with thick anodic oxide films using PEO (plasma electrolytic oxidation) method in which micro-arcs are generated due to dielectric breakdown of the surface oxide film. The PEO methods are studied extensively on aluminum alloys [7-14] and magnesium alloys [15-21].

PEO coatings are formed together with generation of micro-arcs under applied high electric field in liquid electrolytes [7-22]. Al alloys are easily corroded in alkaline solutions but anodic oxide film can grow if anodic current is applied in an alkaline electrolyte containing appropriate anions [8-13]. Growth of PEO films is closely related with arcing behavior on the specimen surface. PEO films are formed only where micro-arcs are generated. Thus, growth behavior and properties of PEO films could be

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expected by the observation of arcing behavior on the specimen surface during the PEO treatment. Lateral and vertical growths of PEO films were reported for the first time on Al1050 alloy [8]. Disk type of PEO film islands about 20 ㎛ diameter were formed first and they are sintered together and finally interconnected completely with treatment time to form a continuous PEO film with very smooth surface less than 1 ㎛ of surface roughness. Further PEO treatment after the complete interconnection of PEO films islands resulted in local thickening of PEO films by vertical growth [8]. The lateral growth of PEO films was observed first on AA1050 under the application of an anodic pulse current [8]. The lateral growth of PEO films is characterized by lateral movement of micro-arcs from the edge toward the center of the specimen. Despite of the advantage of low surface roughness of PEO films formed by the lateral growth mechanism, only a few of paper has been reported on the lateral growth of PEO films.

In this paper, generation behavior of micro-arcs was investigated on the AA7050 with PEO treatment time during the application of 1200 Hz anodic pulse current in 0.1 M $NaAlO_2$ solution. Growth behavior of PEO films was discussed in view of lateral and vertical growth mechanism.

2. Experimental

Al7050 alloy bar (wt.%, Zn 5.7~6.7, Cu 2.0~2.6, Mg 1.9~2.6, Zr 0.08~0.15, Fe < 0.15, Si < 0.12, Zn < 0.1, Ti < 0.06, Cr < 0.04 and Al balance) of 25 mm diameter was cut into 2 mm thick disc sample. The edge part of the disc sample was covered with porous anodic films for good adhesion with an epoxy resin. One surface of the disc sample was connected with Cu strip for electrical connection and then mounted in an epoxy. The other surface of the disc specimen was polished by SiC papers successively up to # 4000 SiC paper and then used for PEO experiment. The electrolyte used for PEO treatment was 0.1 M $NaAlO_2$ and STS316 plate was used for the counter electrode. The PEO coatings were formed by the application of 1200 Hz anodic pulse current of 200 mA/cm^2 and electrolyte temperature was kept to be lower than 30 ℃ by circulating cooling water using a cooling bath. Voltage between working and counter electrodes and digital photographs of the specimen surface were recorded with time during the PEO. Digital photographs of the PEO-treated AA7050 alloy surfaces were also taken after PEO treatments for 2 ~ 12 min. Surface and cross-sectional morphologies of the PEO films were observed using SEM (JSM-6610LV) near the edge and central parts of the Al7050 alloy disc specimen with PEO treatment time. Thickness and surface roughness of the PEO films were measured near the edge and central parts of the Al7050 alloy disc specimen with PEO treatment time. The PEO film thickness and surface roughness were measured by the eddy current method using ISOSCOPE FMP10 (Fisher) and surface roughness tester SJ-400 (MITUTOYO), respectively.

3. Results and discussion

Fig. 1 demonstrates typical PEO film formation voltage-time behavior of the AA7050 specimen in 0.1 M $NaAlO_2$ solution at 200 mA/cm^2 of 1200 Hz pulse current. The voltage increased rapidly with time up to about 270 V at 20 s and then its increasing rate is lowered down to almost zero up to about 6 min. The voltage increased slightly after 6 min and then it showed slight decrease after 9 min. The PEO film formation voltage is closely related with arcing and growth behavior of PEO the formation and growth of anodic films.

The initial rapid increase up to 20 s is normally attributed to the formation of barrier

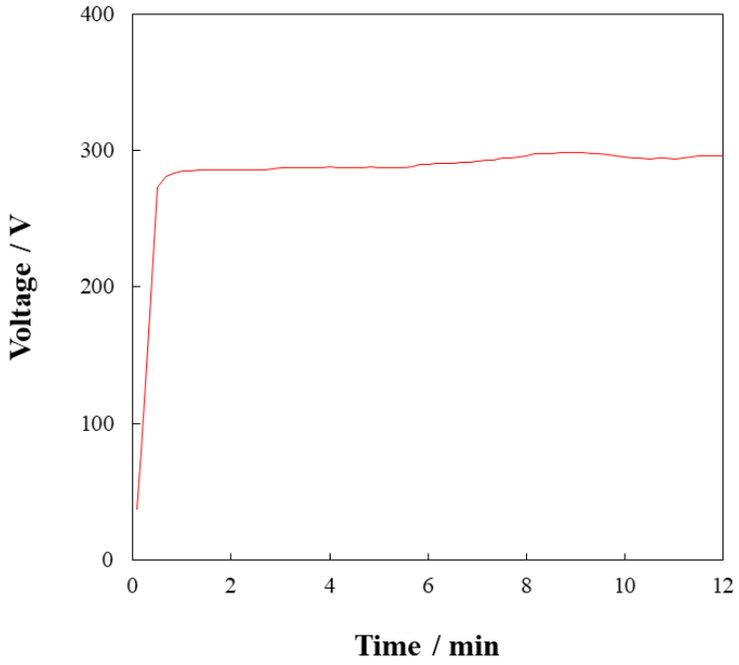

Fig. 1. Voltage-time behavior of Al7050 alloy at 200 mA/cm^2 of 1200 Hz pulse current in 0.1 M $NaAlO_2$ solution.

type of anodic oxide films. The lowered increasing rate of the voltage after 20 s in Fig. 1 should be related with micro-arc generation, as can be seen in Fig. 2(a). The reason why the voltage increases extremely slightly between 2 and 6 min in Fig. 1, is attributable to constant size and number of arcs, as exhibited in Figs. 2(f) ~ 2(j). A little bit large increase of the PEO film formation voltage between 6 and 8 min in Fig. 1, seems to be related with increased density of arcs resulting from the reduced area of arc generation (Figs. 2(j) ~ 2(l)). The slight decrease in the PEO film formation voltage

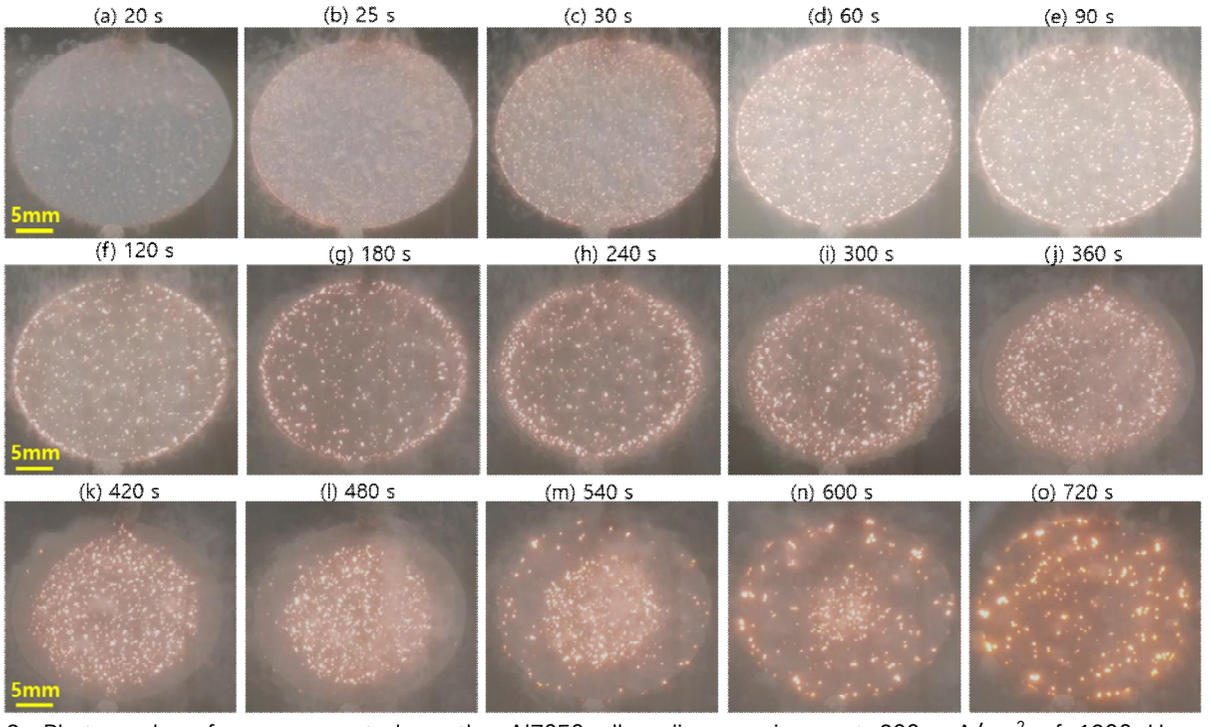

Fig. 2. Photographs of arcs generated on the Al7050 alloy disc specimen at 200 mA/cm^2 of 1200 Hz pulse current in 0.1 M $NaAlO_2$ solution.

after 9 min in Fig. 1, is ascribed to the generation of large size arcs near the edge part, as can be seen in Fig. 2(m).

PEO films are formed together with the generation of arcs. If arcs are generated locally at the same sites repeatedly, porous PEO film grows locally or local dissolution occurs through PEO coating formation and detachment. Thus, uniform arc generation over the whole surface is essential for the formation of uniform PEO films. The other case of uniform PEO film formation can be attained by lateral growth of PEO islands where arcs are generated at the same degree over the entire surface [8]. Such lateral growth was observed on the Al1050 alloy specimen under the application of anodic pulse current in an alkaline solution containing CO_3^{2-}, BO_2^- , SiO_3^{2-} and OH^-. In the present work, lateral growth of PEO films was also observed on the Al7050 alloy specimen in 0.1 M $NaAlO_2$ solution, as manifested by movement of arcs from the edge part towards the central part in Fig. 2. The centralization of the arcs from the edge part towards the central part with PEO-treatment time occurred twice on the Al1050 alloy specimen in 0.8 M Na_2CO_3 + 0.05 M $NaBO_2$ + 1 M Na_2SiO_3 + 0.4 M NaOH solution [8] but it appeared only one time in this work. It is noted that large size arcs were generated at the edge before completing the central part with small size arcs (Fig. 2(m)).

Fig. 3 gives typical surface appearance of the AA7050 specimen surface obtained after PEO treatment at 200 mA/cm^2 of 1200 Hz pulse current in 0.1 M $NaAlO_2$ solution for various durations. Uniform PEO film is formed from the edge part and finally central part is

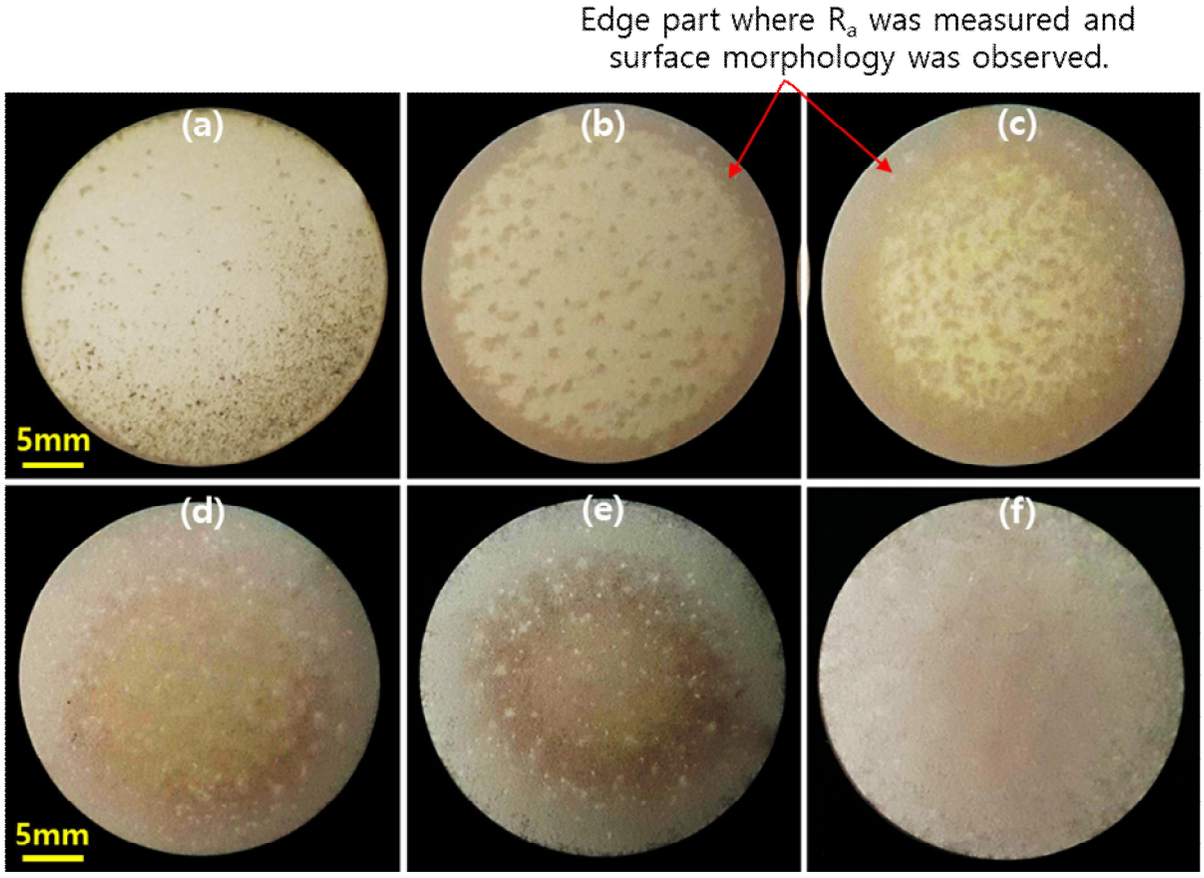

Fig. 3. Surface appearance of the PEO-treated Al7050 alloy disc specimen for various durations of (a) 2 min, (b) 4 min, (c) 6 min, (d) 8 min, (e) 10 min and (f) 12 min at 200 mA/cm^2 of 1200 Hz pulse current in 0.1 M $NaAlO_2$ solution.

covered with PEO film. It is clear that central part of the specimen is occasionally covered with island type of PEO films up to 6 min but fully covered with PEO film after 8 min of PEO treatment.

Surface morphologies of PEO films formed near the edge part, indicated by arrow in Fig. 3 (b) were observed by SEM and their results are exhibited in Fig. 4 as a function of PEO treatment time. There appeared fan-cake type morphology and particulate oxide. There is not any big difference in the surface morphologies of PEO film up to 6 min with treatment time but more surface area is covered with fan-cake type oxide after 8 min with increasing PEO treatment time.

As shown in Fig. 5, surface morphology

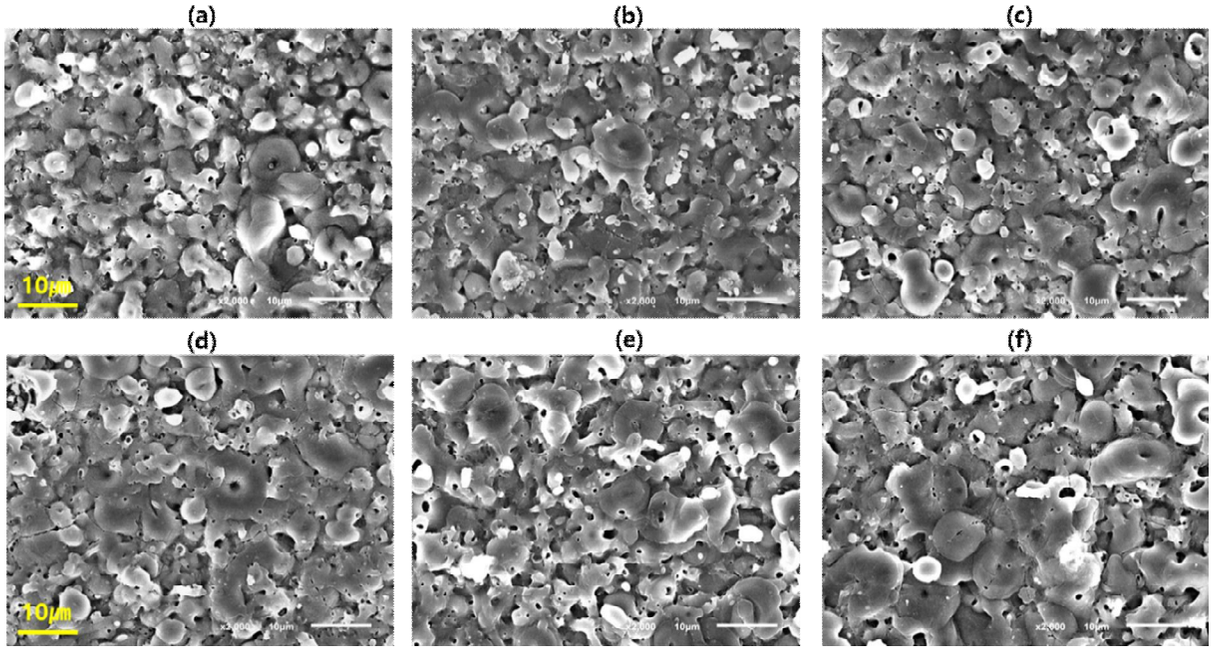

Fig. 4. Surface morphologies of PEO films formed at the edge of the Al7050 alloy disc specimen for various durations of (a) 2 min, (b) 4 min, (c) 6 min, (d) 8 min, (e) 10 min and (f) 12 min at 200 mA/cm^2 of 1200 Hz pulse current in 0.1 M $NaAlO_2$ solution.

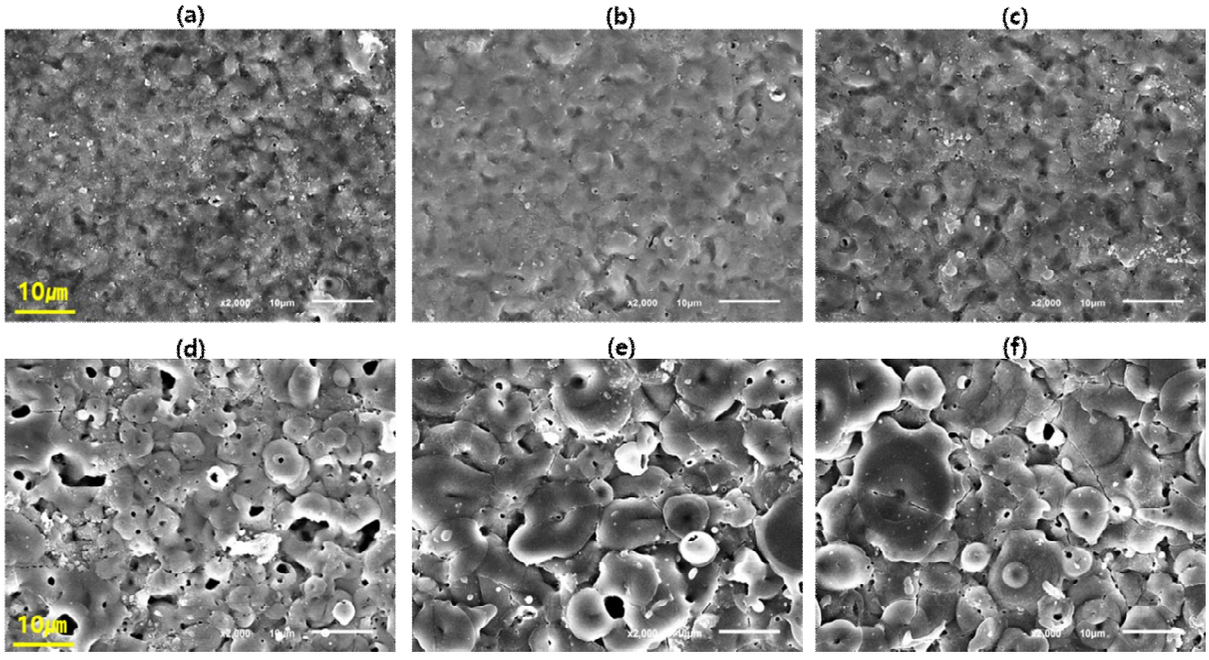

Fig. 5. Surface morphologies of PEO films formed at the central region of the Al7050 alloy disc specimen for various durations of (a) 2 min, (b) 4 min, (c) 6 min, (d) 8 min, (e) 10 min and (f) 12 min at 200 mA/cm^2 of 1200 Hz pulse current in 0.1 M $NaAlO_2$ solution.

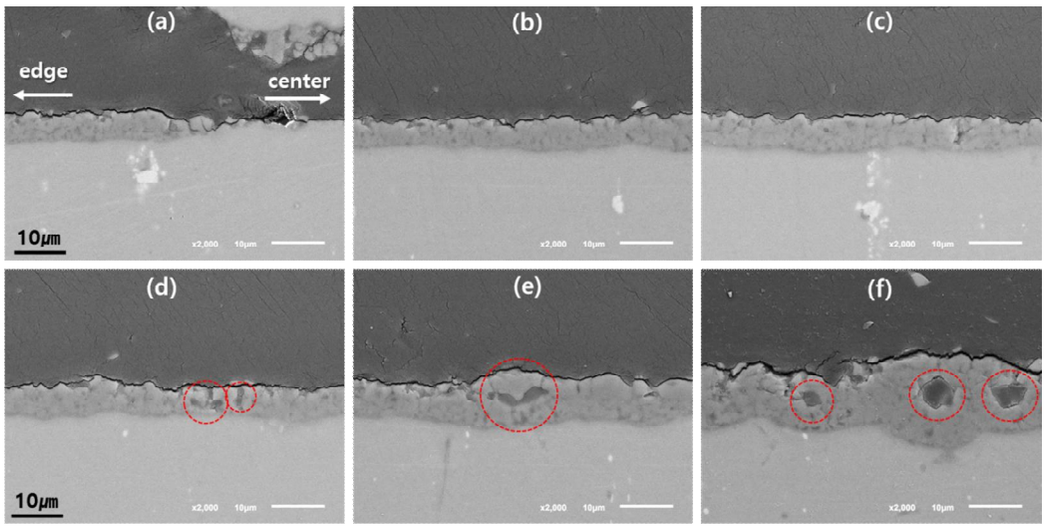

Fig. 6. Cross-sectional morphologies of PEO films formed at the edge of the Al7050 alloy disc specimen for various durations of (a) 2 min, (b) 4 min, (c) 6 min, (d) 8 min, (e) 10 min and (f) 12 min at 200 mA/cm^2 of 1200 Hz pulse current in 0.1 M $NaAlO_2$ solution.

of PEO films formed near the central part of disc specimen is changed drastically after 8 min of PEO treatment time from denser film into porous one. Larger size of fan-cake type morphology near the central part is observed on the PEO films with increasing PEO treatment time.

Cross-sectional morphologies of PEO films formed near the edge part are presented with PEO treatment time in Fig. 6. Relatively uniform thickness of PEO film was obtained near the edge up to 8 min of PEO treatment. This uniform thickness of PEO film seems to result from lateral growth of PEO film from the edge towards the center of the disc specimen. There appeared large size pores within the PEO films, as indicated by dotted red lines in Figs. 6(d) ~ 6(f). The formation of large size pores within the PEO films is related with the generation of large size arcs, displayed in Figs. 2(m) ~ 2(o), suggesting that large size of arcs appearing after finishing lateral growth of PEO films forms more porous and non-uniform PEO films. It is noted that the formation of large size pores results in the formation thicker PEO film, as can be seen in Fig. 6(f).

Fig. 7 depicts PEO film morphologies obtained from cross-section of central part of

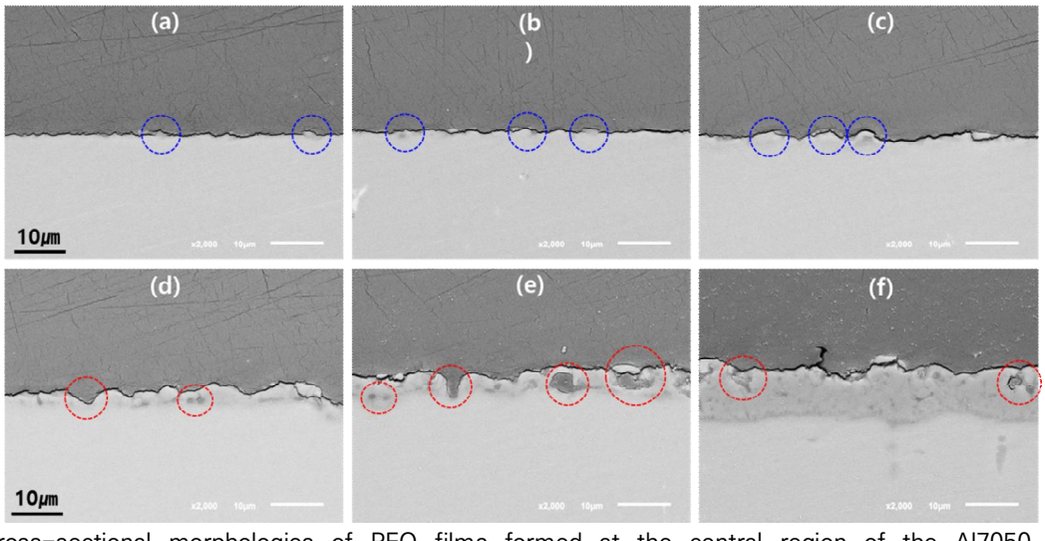

Fig. 7. Cross-sectional morphologies of PEO films formed at the central region of the Al7050 alloy disc specimen for various durations of (a) 2 min, (b) 4 min, (c) 6 min, (d) 8 min, (e) 10 min and (f) 12 min at 200 mA/cm^2 of 1200 Hz pulse current in 0.1 M $NaAlO_2$ solution.

disc specimen. Island type of PEO films, indicated by dotted blue circles, was formed partly up to 6 min of treatment time and they are connected after 8 min of PEO treatment (Fig. 7(d)). Large size pores of several microns were observed within the PEO films, as indicated by dotted red circles. The formation of large size pores seems to result from the generation of large size arcs after 9 min of PEO treatment time, as exhibited in Fig. 2(m).

PEO film thickness measured by the eddy current method and surface roughness are plotted as a function of PEO treatment time in Figs. 8(a) and 8(b), respectively. It is interesting to note that the PEO film thickness and its roughness R_a measured near the edge part show constant values of about 5 μm and 0.5 μm between 4 min and 8 min of treatment time. The PEO film thickness at the central part reached the same value as that at the edge part after 12 min of treatment time. It is worthwhile to mention that the PEO film thickness at the central part is lower than that at the edge part up to 10 min treatment time. This is related with the surface appearance in Fig. 3 where whole surface of PEO films becomes uniform after 12 min but still PEO film-covered central parts are darker than the edge part for 8 min and 10 min PEO-treated surface (Figs. 3(d) and 3(e)).

The PEO film at the central region grew after 6 min of treatment time but the surface roughness at the central region increased with increasing treatment time. Considering that PEO film thickness is measured at a point while surface roughness is measured along the line of 4 mm long, the increased surface roughness with treatment time from 2 min represents the presence of island type of PEO film, which is manifested in the cross-sectional observation of PEO films in Fig. 7. Thus, it is concluded that vertical growth starts on AA7050 alloy specimen in 0.2 M $NaAlO_2$ solution under 1200 Hz pulse current before completing lateral growth. Such vertical growth results in non-uniform thickness and increases of surface roughness.

4. Conclusions

In this work, generation behavior of micro-arcs and growth behavior of PEO films were investigated at the edge part and central part of the AA7050 disc specimen with PEO treatment time in 0.1 M $NaAlO_2$ solution under the application of 1200 Hz anodic pulse current. The following conclusions were drawn from the analyses of experimental results.

1. Micro-arcs generated on the AA7050 disc specimen moved from the edge towards the central part with PEO treatment time and formation of PEO films proceeded from the edge part to central part, indicating lateral growth of PEO films.
2. Moving of the arcs from the edge towards the central part appeared only one time for the AA7050 disc specimen time in 0.1 M $NaAlO_2$ solution under the application of 1200 Hz anodic pulse current.
3. The lateral growth in 0.1 M $NaAlO_2$ solution produced PEO films with thickness of about 5 μm and surface roughness of about 0.5 μm on the AA7050 disc specimen.
4. Large size arcs were generated at the edge before completing the central part with small size arcs on the AA7050 disc specimen time in 0.1 M $NaAlO_2$, suggesting that vertical growth starts before completing lateral growth.
5. The PEO film thickness at the central part reached the same value as that at the edge part after 12 min of treatment time.

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