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ARTICLE

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The Effect of Different Heat Treatments on Surface Microstructures and Anodic Oxide Film Structures of Sheets of an Al-5.6Mg Alloy

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Abstract: The effect of different intermediate annealing heat treatments on the surface microstructures and anodic oxide film structures of rolled sheets of an Al-5.6Mg alloy was studied. The results show that when the continuous annealing is used instead of the static state annealing in intermediate annealing process to control microstructures of the sheets, the surface grain size of the sheets can be reduced by about 60 %, and size of the Mg precipitated phase (Mg_2Al_3) can be reduced by about 67 %. Under the combined influence of grain size, uniform precipitation phase, and texture, the highest glossiness can be obtained, which was attributed to continuous intermediate annealing and stabilization annealing at low temperature. The uniform grain and precipitation structures is beneficial to reduce the inhomogeneous dissolution of the oxide film and to obtain the anodic oxide film with uniform thickness and high gloss.

Key words: Heat treatment, Anodizing, Al-Mg alloy, Microstructures

Plates and sheets of AA5xxx aluminum alloys are widely applied to Computer, Communication, and Consumer Electronics (3C) industries because of their beautiful appearance surface and tinctorial diversity after anodizing, as well as high surface hardness and light weight [1]. However, in recent years, with the growing demand for lightweight, convenient, and aesthetically appealing aluminum products, there has been a greater focus on performance attributes such as higher strength, excellent surface gloss, and greater cost-effectiveness.

The many 5000 series aluminum alloys, especially high Mg content, such as AA5182 and AA5059 have low or poor external gloss after anodizing. This has become a significant reason in the efforts to improve and control the appearance quality of high magnesium content aluminum alloys in the aluminum industry. This is why the 5000 series aluminum alloys are currently a hot topic for research and development.

In the existing literatures, Ke Ma et al. [2-7] studied the effects of chemical compositions of the 5000 series aluminum alloys and anodizing process parameters on the appearance quality after anodized. Hebin Ding et al. [8] also investigated the corrosion rate of the second phase during anodizing. Additionally, G Beck et al. [9] found that single crystals with different crystal orientations after anodizing have different surface performances and K. KATO [10] observed that the reflectivity of the oxide film obtained after anodizing on different crystal planes was different.

However, there are few studies on the effect of heat treatment processes on the microstructures and anodic oxide film structures of sheets of 5000 series aluminum alloys with high Mg content. There are two main types of heat treatment: static state annealing and continuous annealing. Continuous annealing generally produces a fine-grained, high-strength aluminum alloy with uniform properties. To increase efficiency of production, continuous annealing frequently was used in aluminum industries [11]. With growing application of aluminum auto sheets, the number of continuous annealing lines has rapidly increased and is now widely used for various aluminum al-

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loy products to improve the properties of aluminum alloy sheets [12-14].

In this paper, an Al-5.6Mg aluminum alloy was studied and developed, and the effects of different heat treatments during intermediate annealing, this is, static state and continuous annealing, on the final microstructures and surface crystallization textures of the sheets and structures of oxide film after anodizing were analyzed by use of different equipment. The relationship between heat treatment, microstructures of Al-5.6Mg alloy sheets and performance of surface anodic oxide film was established. The findings from the observations and analysis in this work provide a guidance and improving direction for heat treatment processing in the development of the 5000 series aluminum alloy sheets with high Mg content, high strength and high gloss after anodic oxidation.

1 Materials and Experiments

The composition of the Al-5.6Mg alloy was designed and is listed as shown in Table 1. The composition of the aluminum alloy met with AA5023 alloy certification. It contains a very high wt.% Mg, while other elements were controlled in very low wt.% levels. A large ingot with commercial size of the Al-5.6Mg alloy was produced using high purity aluminum as raw material. The final composition of the ingot met the design requirements of the Al-5.6Mg alloy, as listed in Table 1.

Table 1 Chemical compositions of the Al-5.6Mg alloy (wt. %)

	Si	Fe	Cu	Mn	Mg	Cr	Zn	Rem.
5023	≤0.25	≤0.40	0.20-0.50	≤0.2	5.0-6.2	≤0.10	≤0.25	Al
Al-5.6Mg	≤0.10	≤0.12	≤0.10	≤0.2	5.0-6.0	≤0.05	≤0.05	Al
Ingot	0.04	0.08	0.02	0.2	5.56	0.03	0.02	Al

A hot band with the 3.7 mm thickness was obtained through conventional hot rolling process. To achieve full recrystallized grain structure, the final hot rolling temperature was set at 320 ℃. The hot band was then cold rolled by four passes to gauge of 1.0 mm, as shown in Table 2.

Table 2 The cold rolling process of four passes to 1.0 mm

The first pass	3.7 mm to 2.5 mm
The second pass	2.5 mm to 1.8 mm
The third pass	1.8 mm to 1.3 mm
The fourth pass	1.3 mm to 1.0 mm

After the above cold rolling was completed, intermediate annealing was carried out. Two different intermediate annealing treatments were performed in a box furnace (a static state annealing) and a continuous annealing furnace (a continuous annealing) on industrial scale. The two annealing schedules are designed to obtain full recrystallization structures.

For static state annealing, the intermediate annealing was held at 330 ℃ for 2 hours, followed by cold rolling a final gauge to 0.8 mm. Stabilizing annealing was then performed at 100 ℃ and 200 ℃ for 2 hours, respectively, resulting in sample 1# and 2#.

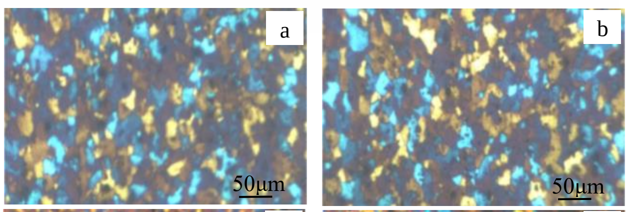
For continuous annealing, the goals were to obtain recrystallized sheet and to control size and number of precipitated phase particles. Taking industrial factors into account, continuous annealing was conducted at 380 ℃ for 15 seconds, followed by same cold rolling to a gauge of 0.8 mm and stabilizing anneal processing at 100 ℃ and 200 ℃ for 2 hours, respectively, resulting in sample 3# and 4#.

Grain structures and compound phase particles were observed by use of an optical microscope, and oxide film was examined by use of a scanning electronic microscope (SEM - EVO MA10). The grain size was measured using an optical method in accordance with ASTM E112-13 standard. The EBSD measurement and analysis were carried out by SEM (JSM 7800F) and Oxford software with advanced analysis capabilities. Surface roughness was measured with a roughness meter (TIME-3202) and the gloss was determined by a gloss meter (KSJ-MG268-F2).

2 Results and Discussion

Fig.1 shows the surface grain structures and the surface feature of Mg-containing phase from 0.8mm final sheets after direct corrosion by phosphoric acid solution. By comparing Fig.1a to Fig.1d, it can be observed that the two annealing processes lead to different grain sizes. The grain sizes of the samples (1# & 2#) from the static state annealing are obviously larger than those of the samples (3# & 4#) from the continuous annealing. The grain size measurement showed that the continuous annealing resulted in a grain size of about 12 μm, whereas the static state annealing resulted in a grain size of about 35 μm. The findings of S. Samberger et al. [15-16] indicated that the rapid annealing leads to smaller grain sizes after cold deformation, which is also reflected in the industrial results seen in Fig. 1a to 1d.

According to a study from Jingwei Zhao et al. [17], the size and quantity of Fe-containing phase do not change during cold rolling and annealing, so the Fe-containing phase will not be considered here because of low% Fe (see Table 1). Yong Yang et al. [18] noted that in 5000 series aluminum alloys with Mg wt. %>3.5 %, the Mg-containing phase, Mg_2Al_3 (precipitates), forms along the grain boundary during heat treatment. Popovic M et al. [19] proposed that a phosphoric acid solution with a concentration of 10 % and a temperature of 60 ℃ could be used to directly corrode the Mg-containing precipitates along the grain boundaries of 5000 series aluminum alloy. Following the corrosion method, the precipitations of Mg-containing phase were directly corroded, as shown in Fig.1e to Fig.1g. It was found that the precipitates are larger and more numerous after the static state annealing compared to those after continuous annealing.



Compared to Fig.1e and Fig. 1g, it can be observed that size of the corrosion pits of the sample 1# reaches to 15 μm from static annealing, which is three times the size of those in sample 3# from continuous annealing. This indicated that the precipitations of Mg containing phase can be reduced by about 67 % by use of continuous annealing. In addition, it was found that when other process conditions remain unchanged and only the stabilization annealing temperature is increased from 100 ℃ to 200 ℃, the grain size on the surface does not change, but the size and number of corrosion pits increase rapidly and the precipitates grow up.

The main reason for the observed difference is the varying heating up rates in the continuous and static state annealing processing. In general, the heating rate in continuous annealing exceeds 6 ℃/s in industrial scale, which is about 300-600 times faster than that of static state annealing [20]. Due to the rapid heating, the many nucleation points form uniformly in the sheets, resulting in small grain sizes. Additionally, the cooling rate in continuous annealing in this study is fast, closes to 10 ℃/s, which limits grain growth.

At the same time, the rapid cooling also effectively inhibits the precipitations of Mg phase. The finer grain size and Al_2Mg_3 phase distribution precipitated from the intermediate continuous annealing still are maintained even after stabilized annealing.

Table 3 lists the performance results of the oxide film of the samples after anodization. As shown in the table, the surface roughness of the samples after static state is higher, and the gloss is poorer compared to those from continuous annealing. Furthermore, increasing the stabilization annealing temperature from 100 ℃ to 200 ℃ reduces gloss. The higher the stabilizing temperature, the poorer the surface glossiness.

Table 3 Test results of performance of anodic oxide film

Number of samples		1#	2#	3#	4#
roughness	Ra /μm	0.462	0.541	0.454	0.457
	Rz /μm	3.483	4.421	3.166	3.262
glossiness	GU	28	24	48	27

It was found that the surface and profile morphologies of the oxide film of 2# and 3# samples with the lowest and highest gloss were shown in Fig.2. The uneven features of the oxide film surface in Fig.2a were significantly different from the local pits compared to Fig.2b. By comparing Fig.2c and Fig.2d, the thickness of oxide film of sample 2# from static state annealing is 11 μm, which is 50 % of the 20 μm from continuous annealing.

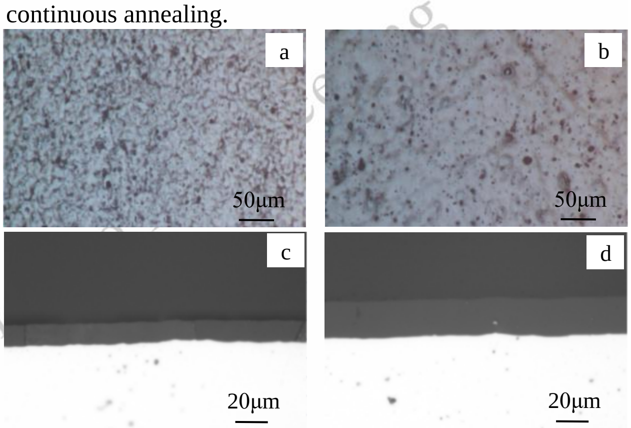


Fig.2 Surface morphology and profile morphology of anodic oxide film,(a)-surface of 2#,(b)-surface of 3#,(c)-cross sectional surface of 2#,(d)-cross sectional surface of 3#

In general, the growth of the anodic oxide film on aluminum alloys is accompanied by the interface between the oxide film & the aluminum matrix as well as the electrochemical dissolution of the oxide film formed on the surface. Dissolution first occurs at the grain boundary of aluminum alloy. Chao Zhang et al. [21] suggest that larger grain sizes can lead to more uneven dissolution of the oxide film during the process of anodization, resulting in a thinner oxide film. Due to the combined influence of more precipitated phase particles and larger grain sizes, the oxide film of the ample 2# undergoes uneven dissolution, leading to an uneven surface feature with reduced thickness. The uneven surface structure results in weaker reflection of incident light and lower gloss. It has been confirmed that a thick oxide film can achieve high surface gloss and protect better surfaces of aluminum alloys.

G Beck et al. [9] demonstrated that the oxide films on aluminum single crystals with (111), (110) and (100) orientations were formed in a two-step anodization process. It was found that the best ordering was obtained in nanoporous alumina on (100) lattice plane of aluminum single crystal compared to

(111) and (110) lattice planes. This indicates that the gloss after anodization is relative to surface crystal orientations of aluminum sheets.

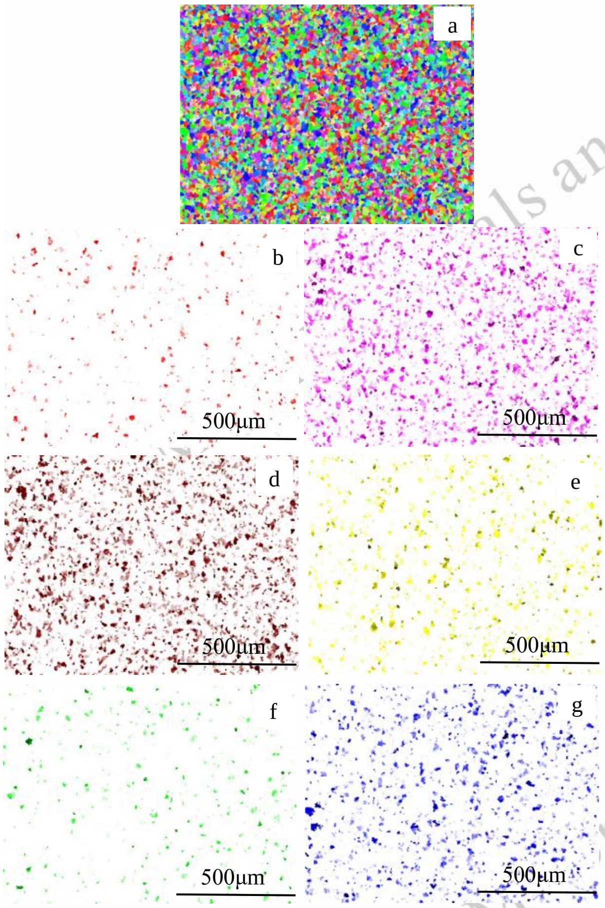


Fig. 3 Different crystal orientation distributions from EBSD on the surface of 2# sample (a)- orientation distribution, (b)-Cube, (c)-S, (d)-R, (e)-Brass, (f)-Goss, (g)-Copper

Fig.3 and Fig.4 show the result of EBSD analysis on the surfaces from Sample 2# and 3# and volume fractions of different texture components, i.e., different crystal orientations, are listed in Table 4. In general, the texture components on the surfaces in aluminum alloy sheets have main deformation textures: residual deformation texture: Brass-{011}<211>, Copper-{112}<111> & Goss {011}<100> and recrystallization textures such as Cube-{001}<100> & R-{124}<211>. Sometimes, Goss texture is classified as a recrystallization texture. Grain size analysis results from EBSD are consistent with the optical observation as shown in Fig.1, further indicating that continuous intermediate annealing reduces grain size.

In addition, based on the EBSD analysis from Fig.3 and Fig.4, it was found that the Cube texture is uniformly distributed on the surface. The deformation textures are located near and around the Cube texture components. Continuous annealing can produce more grains with Cube orientation compared to static state annealing. According to a study by G Beck, et al. [9], a high proportion of Cube grains can result in higher surface glossiness. As shown in Table 4**,** sample 3# has more Cube texture than sample 2#, which corresponds to the higher gloss of sample 3#. This indicated that a high-volume fraction of Cube components may lead to good surface performance after anodization.

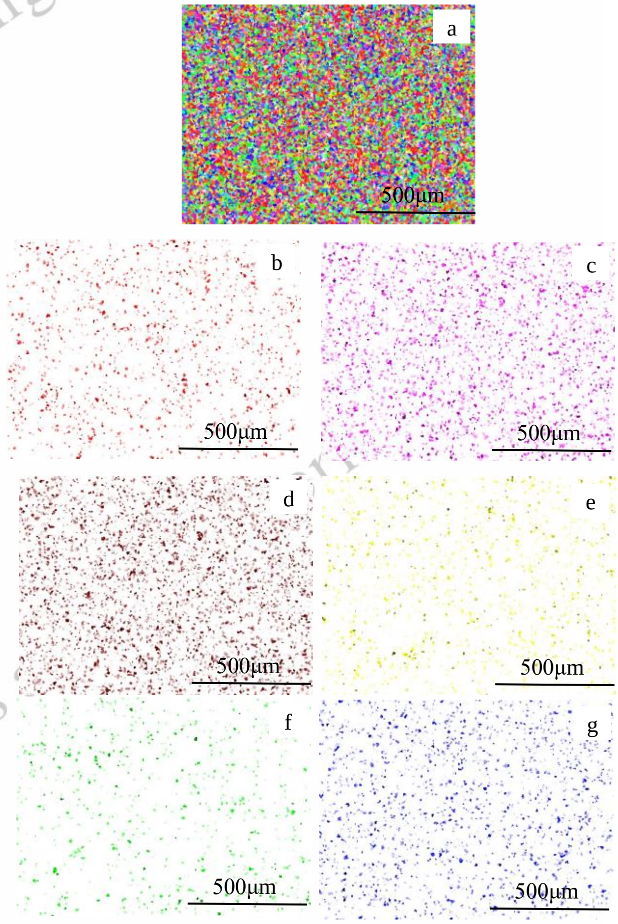


Fig. 4 Different crystal orientation distributions from EBSD on the surface of 3# sample (a)- orientation distribution, (b)-Cube, (c)-S, (d)-R, (e)-Brass, (f)-Goss, (g)-Copper

Table 4 Volume fractions (%) of six typical texture components

Sample ID		2#	3#
Copper (Blue)	{112}<111>	12.5	8.38
S (Purple)	{123}<634>	21.4	15.2
Brass (Yellow)	{110}<112>	11.2	7.55
Goss (Green)	{011}<100>	3.46	3.87
Cube (Red)	{100}<001>	3.4	6.87
R (Dark red)	{124}<211>	23.8	18.8

The growth of anodized film along thickness direction on aluminum alloy is accompanied by the formation of oxide film on interface between the aluminum substrate and the electrochemical dissolution of the oxide film on the surface. Zhang Chao et al. [21] believed that a larger grain size leads to the more uneven dissolution of the oxide film during the anodizing process, making it easier to achieve a thinner oxide film. This indicated that the dissolution of the oxide film also depends on the crystal orientations. Specifically, grains with Cube texture may dissolve more slowly compared to other crystal orientations.

Therefore, because of the dual influence of a greater number of precipitated phases and larger grain size, the oxide film of sample 2# exhibits more uneven in the growth and electrochemical dissolution process, so resulting in an uneven surface and thinner oxide layers. By studying the impact of grain orientation on anodized oxide film in single crystals, K. Kato [10] concluded that the reflectivity of the oxide film varies with different lattice planes, with the reflectivity being highest on the {001} crystal plane compared to others.

After examining the anodized film formation process of aluminum alloy, G Beck et al. [9] proposed that the oxide film on the {001} crystal plane is the highest order, which contributes to achieving high reflectivity. In this study, under condition of a larger volume fraction of Cube texture, sample 3# obtains the highest reflectivity of the oxide film, thereby further enhancing the gloss of the aluminum alloy. The highest glossiness of sample 3# is attributed to the combined effects of grain size, precipitation phase, and texture, as well as the continuous intermediate annealing and stabilization annealing at 100°C.

3 Conclusions

In the present work, the effects of different intermediate annealing heat treatments on the surface microstructures and anodic oxide film structures of Al-5.6Mg alloy sheets were investigated. The following key conclusions were drawn:

(1) **Grain Size Reduction and Increase of Grains with Cube orientation**: For high Mg 5000 series aluminum alloy sheets, **continuous annealing** reduces the surface grain size by **60%**, achieving a finer grain size of **12 μm** compared to static annealing. Additionally, continuous annealing results in more grains with **Cube orientation**, which positively influences the material's properties.

(2) **Uniform Distribution of Precipitates:** Continuous annealing significantly decreases Mg-containing precipitates along the grain boundaries by **67%.** These precipitates are **uniformly distributed within the grains** in sheets processed via continuous annealing.

(3) **High Glossiness of Surface Oxide Film:** The combination of **smaller grain size, uniform precipitate phases, and Cube texture** leads to the highest surface glossiness, attributed to continuous intermediate annealing and subsequent low-temperature stabilization annealing. The uniform grain structure with Cube orientation and evenly distributed precipitates helps reduce inhomogeneous dissolution of the oxide film. This enhances the **uniformity and gloss** of the anodic oxide film, resulting in a **thicker, more consistent film** on the aluminum sheets.

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不同热处理对 Al-5.6Mg 合金板材表面组织和阳极氧化膜组织的影响

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摘　要：本文研究了不同中间退火热处理对 Al-5.6Mg 合金轧制薄板表面组织和阳极氧化膜组织的影响。结果表明:在中间退火过程中，用连续退火代替箱式退火，试样表面晶粒尺寸可减小约 60%，Mg 析出相（Mg_2Al_3）尺寸可减小约 67%；在晶粒尺寸、析出相和织构的共同影响下，经连续中间退火和低温稳定化退火后阳极氧化，获得了最高的光泽度。均匀的微观结构有利于减少氧化膜的不均匀溶解，获得厚度均匀、光泽度高的阳极氧化膜。。

关键词：热处理；阳极氧化；Al-Mg合金；微观结构

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