Study on the anodic oxidation performance of a rolled AA5252 aluminum alloy sheet with different thicknesses

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Abstract: Microstructures of AA5252 aluminum alloy sheets from surface to the center were observed by use of scanning electron microscope with electron backscatter detector assembly, and treated by anodic oxidation. Results showed that changes of the grain structures and textures of the AA5252 alloy sheets occur from the surface to the center layers. The anodic oxidation surface quality of the AA5252 aluminum alloy is closely related to the area ratio and distribution uniformity of Cube texture.

1. INTRODUCTION

AA5252 aluminum alloy is a kind of aluminum alloy that cannot be strengthened by heat treatment. The alloy has medium strength, good workability, welding performance and mechanical processing performance as well as satisfactory chemical polishing effect. After anodizing treatment, Abradability, corrosion resistance and colorful appearance of the alloy can be improved. It often appears that the strip defects on the surface of the aluminum alloy come from the difference in the anodic oxidation film [1, 2]. The areas with different colors are distributed in strips along the processing direction (such as, rolling direction) of the aluminum alloy, with a width of about 50μm to 5mm and a length ranging from a few millimeters to ten millimeters. During subsequently coloring process, the colorized difference becomes more significant because the oxidation film serves as an amplifier with color difference, which seriously affects the application of the aluminum alloy in electronics appearance [3, 4]. At present, there are few researches on the effect of the texture of AA5252 aluminum alloy on the anodic oxidation performance. In this paper, combined with the production process of rolling AA5252 alloy, the effect of anodizing from the surface to the center layers was studied, and the influence of grains and textures on the anodizing performance was preliminarily explored.
2. EXPERIMENTAL MATERIALS AND SCHEMES

2.1 Experimental materials
An AA5252 aluminum alloy was used as experimental material, which was made of large-size commercial ingots with a thickness of 650mm and a width of 1250mm by DC casting method. The surface of the ingot was milled out 20mm, and then homogenized at 480°C for 10 hours. After homogenization heat treatment, it was hot rolled to a gauge of 6.5mm and the final hot rolling temperature was 320°C. Finally, the hotband was cold rolled to with a thickness of 2.0mm according to the total cold reduction of 69%. After the cold rolling, the final annealing treatment was carried out at 230°C. The chemical composition of the AA5252 aluminum alloy is shown in Table 1.

Table 1. Chemical composition of AA5252 aluminium alloy sheets (wt.%).

|   | Si  | Fe  | Cu  | Mn  | Mg  | Zn  | Bal. |
|---|-----|-----|-----|-----|-----|-----|------|
|   | ≤0.02 | ≤0.04 | ≤0.10 | ≤0.10 | 2.2~2.8 | ≤0.05 | Al    |

2.2 Experimental materials
The alloy sheets with size of 75mm × 150mm were thinned sequentially from the surface to the center layers through the thickness direction, and 5 samples with different thickness were prepared: their thickness reductions were 0mm for 1# sample, 0.16mm for 2# sample, 0.37mm for 3# sample, 4# sample 0.46mm and 5# sample 0.93mm, respectively. The samples were immersed in 15% sodium hydroxide solution for 60 seconds at 60°C, and then 20% nitric acid solution for 30 seconds at 20°C. In an electrolyte with a sulfuric acid solution having concentration of about 18%, the temperature was about 20°C, the current density was 1.4 A/dm² by using constant current control, and the duration of anodizing treatment was 30 minutes. Under the D65 light source, the color difference on the surface of the anodized aluminum alloy was observed and evaluated. The effect of the anodizing treatment was assessed according to whether there was color difference of stripe patterns on the surface and the number of different color stripe patterns if any.

The above 5 samples were cut into 7mm×7mm squares, and then further were prepared for EBSD analysis as the following steps: 1) Mounting the samples (ratio of acrylic powder and solution (hardener) is 2: 1, waiting for drying); 2) Rough polishing and fine polishing of the samples on an automatic grinding and polishing machine (suspensions with 3μm particle size can be used for rough polishing and 0.2μm particle size can be used for fine polishing); 3) Ultrasonic cleaning in alcohol with a KH3200V ultrasonic cleaner for 4 minutes; 4) Electrolytic polishing (using an electrolyte with a sulfuric acid solution with concentration of 18%, polishing voltage 20V, current 1.0A, and polishing time 30 s). The prepared samples were observed at 100 magnifications on a JSM7800F field emission electron microscope equipped with an EBSD probe for the grain and texture analysis. The experimental data were collected and analyzed through the HKL Channel 5 software package. Considering the symmetry, only the texture test and organization observation were performed from the upper surface to the center layers.

3. RESULTS AND DISCUSSIONS

3.1 Anodizing quality
The anodizing treatment of aluminum alloy sheets is an electrolytic oxidation process. During this process, formation and dissolution reaction of an oxide film take place simultaneously, and finally an oxide film is formed on the surface of the aluminum alloy material. The quality (or depth) of the oxide layer depends on the speed of chemical reactions. This oxide film has some different functions, such as, protection, decoration, and insulation. In general, anodizing film of aluminum alloy is divided into two types: barrier and porous film. Barrier anodic oxide film, also was called shielded anodic oxide film, is generally formed in electrolytes with weak dissolving rate (such as neutral borate, neutral phosphate, etc.). This dense and non-porous film is very close to the surface of aluminum alloy, and
the thickness of this film is generally no greater than 0.1μm; While the porous oxide film is composed of an oxide film with two layers. The bottom layer is a dense and non-porous barrier layer connected to the metal surface, and the top part is a porous structure. The oxide film is used to protect and decorate the metal surface.

Figure 1. Surface features of anodic oxidated 5252 alloy at different layers of (a) and (b) 1#-0mm, (c) 2#-0.16mm, (d) 3#-0.37mm, (e) 4#-0.46mm and (f) 5#-0.93mm

Table 2 shows the anodic oxidation performance of 5 samples with different layers after reduction from surface. It can be seen in Table 2 that after anodization, the anodizing quality decreases with the increase in the thickness reduction from the surface to the center layers. That is, the anodizing effect transforms from the qualified surface layer to the unqualified center layer. This indicated that the anodic oxidation effect has a certain relationship with the grain orientations.

Table 2. Anodic oxidation quality of 5252 aluminum alloy at different layers

| No. | Thickness reduction | Anodic oxidation quality | Effect assessment |
|-----|---------------------|--------------------------|-------------------|
| 1#  | 0 mm                | A (Qualified, Better)    | Few strips with different colors on the surface |
| 2#  | 0.16mm              | A (Qualified, Better)    | Few strips with different colors on the surface |
| 3#  | 0.37mm              | B (Qualified, Good)      | Almost no strips with different colors on the surface |
| 4#  | 0.46mm              | C (Unqualified, Poor)    | Several strips with different colors on the surface |
| 5#  | 0.93mm              | D (Unqualified, Poor)    | Many strips with different colors on the surface |

3.2 Observation and analysis of grain structures

The evolution of the microstructures of aluminum alloy in the process of plastic deformation, mainly includes dislocations, grain structures and texture. The microstructures of aluminum alloy sheet along thickness direction distributed unevenly. Moreover, the deformation of the surface layer of the sheet and strip is severe, and the degree of deformation is larger compared to the deformation of the center layer. According to the experimental scheme, the experimental data were analyzed by HKL Channel 5 software package. Figure 2 shows the grain structures of the AA5252 alloy with different thicknesses
reduction to surface of the sheet. With the thickness reduction increasing, the crystal grains from the surface to the center layers are elongated. The crystal grains in surface layer are paraxial and small; the crystal grains near the center layer are elongated along the rolling direction, and distributed nonuniformly. This is mainly because the deformation degree on surface of the rolled AA5252 aluminum alloy sheet is higher than that of the center layer, and the higher storage energy recovers at below the recrystallization temperatures, so the equiaxial grains around the non-equiaxial grains were formed.

Figure 2. Grain structures at different layers (a)1#- 0mm, (b)2#- 0.16mm, (c)3#- 0.37mm, (d)4#- 0.46mm and (e)5#- 0.93mm

3.3 Texture analysis

In the process of cold and hot processing of aluminum alloy sheets, orientations of the crystal grains in the polycrystal material will be re-arranged along certain directions, crystalline texture. The change of metal material structures in the microstructure and texture during the plastic deformation process, and then these changes will have an very important impact on the final performance of materials. Table 3 shows the statistical results of the texture of the AA5252 alloy with different thickness reduction, and Figure 3 shows their texture maps.

As can be seen from Table 3 and Figure 3, the texture of the AA5252 alloy sheet includes: main rolling texture components Brass-{011}<211>, and Copper-{112}<111>; transitional texture components-Goss{011}<100>; recrystallization texture components Cube-{001}<100> and R-{124}<211>.

Table 3. Anodic oxidation quality of 5252 aluminum alloy at different layers

| No. reduction | Copper (blue) | S (purple) | Brass (yellow) | Goss (green) | Cube (scarlet) | R (dark red) |
|---------------|---------------|-----------|----------------|--------------|----------------|-------------|
|               | {112}<111>    | {123}<634>| {110}<112>     | {011}<100>   | {100}<001>     | {124}<211>  |
| 1# 0 mm       | 23.9          | 17.9      | 8.6            | 5.3          | 8.1            | 17.8        |
| 2# 0.16mm     | 13.5          | 21.7      | 10.8           | 6.3          | 8.6            | 18.7        |
| 3# 0.37mm     | 9.3           | 14.8      | 15.5           | 5.7          | 8.0            | 15.4        |
| 4# 0.46mm     | 6.9           | 15.7      | 11.3           | 6.2          | 5.1            | 26.2        |
| 5# 0.93mm     | 17.2          | 13.0      | 23.4           | 6.3          | 4.3            | 16.4        |
Figure 3. Texture at different layers (a) 1#-0mm, (b) 2#-0.16mm, (c) 3#-0.37mm, (d) 4#-0.46mm and (e) 5#-0.93mm

Figure 4. The evolution of textures under different reduction of AA5252 aluminum alloy

By the statistical and calculating results of the texture components, the texture changes of the alloy sheet during the thinning process can be clearly shown in Figure 4. It can be seen intuitively in Figure 4 that with the thickness reduction increases, the Cube texture component generally show a downward trend distributed nonuniformly; while the Brass and Goss texture components generally show an upward trend and other textures show an upward or downward trends. When the thickness reduction is 0.46 mm, the orientation rate of cold-rolled texture component R increases, and when the thickness reduction is greater than 0.46 mm, the volumes of Copper and Brass textures increase.

3.4 The relationship between texture and anodizing quality

K. KAT [3] studied the influence of the grain orientation in the single crystal on the aluminum anodization film, and he believed that the reflectivity of light on different crystal planes was different. For example, the reflectivity of oxide film on the {001} crystal plane was higher than that on the other crystal planes. Secondly, in the process of studying the orientations of the holes on the surface, G Beck [4] proposed that the orientations of the crystal grains would cause the difference in the degree of order of the holes on the surface. The degree of order of the oxide film on the {001} crystal plane was
the highest. The anodic oxide film starts to form porous layers on the barrier layer. Due to the uneven crystal surface and the difference in crystal grain orientations, a large number of pores cannot grow perpendicular to the aluminum matrix interface and resulted in a decrease in the degree of order and uneven distribution of pores in the porous layer. When the surface of anodized aluminum is illuminated by a light source, the difference in the oxide film layer causes the different colors to appear on surface. In the AA5252 polycrystalline materials, it was found that the Cube {001}<100> texture has the greatest impact on the anodic oxide film, because the anodic oxide film grows on the grains oriented on the {001} plane. The crystallization speed of the oxide film on {001} is faster than that on the {011} surface and the {111} surface, resulting in uneven oxide film thickness and difference in reflectivity.

It can be seen from Table 3 that on the surface of the aluminum alloy material, the Cube texture area accounts for more than 8.0%, and the surface quality after the anodization is qualified; while in the central area of the aluminum alloy material, the Cube texture area accounts decreased and distributed nonuniformly, the surface after the anodizing treatment exposed many strip defects and unqualified. In the summary, from the surface to the center layers, the proportion of the Cube texture area gradually decreases, while the anodizing performance gradually deteriorates. This is mainly because the diminished and distributed unevenly of Cube texture area brings about a lower reflectivity than other texture areas, which leads to present different colors on the surface of anodized aluminum alloys.

4. CONCLUSIONS
1. From the surface to the center layers, the grains and textures occur changes of the AA5252 alloy.
2. The surface quality of the AA5252 aluminum is closely related to the proportion of Cube texture area. When the Cube texture area accounts for more than 8.0%, the surface after the anodizing treatment is qualified; and when the Cube texture area accounts for less than 8%, the surface after the anodizing treatment is unqualified.
3. The Cube and S texture area of the AA5252 aluminum from the surface to the center layers gradually decreases, and the anodizing performance of the AA5252 aluminum alloy sheet decreases correspondingly.

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