Measurement of oxide layer thickness on 7050 aluminum alloy surface based on image processing technology

Maolin Zhang¹, Song Cai¹, Yongqing Huo², Zhiren Chen³ and Zhilin Wu¹,*

¹Nanjing University of Science and Technology, Nanjing 210094, China
²Chongqing Jialing Special Equipment Co., Ltd. Chongqing 400000, China
³Changzhou Habu Electromechanical Co., Ltd. Changzhou 213200, China

*Corresponding author e-mail: wuruinan-1994@njust.edu.cn

Abstract. This paper presents a method for measuring the thickness of the oxide layer on the surface of 7050 aluminum alloy based on image processing technology. As the 7050 aluminum alloy is oxidized to different degrees at different temperatures and different holding times, its surface color will also change significantly with the degree of oxidation. The image processing method was used to obtain the gray value of the sample photos with different degrees of oxidation, and the surface oxide layer of the sample was subjected to Ar⁺ layer-by-layer sputter peeling. XPS analysis was performed on the peeled surface to calculate the thickness of the oxide layer. The oxidation was found. Relationship between layer thickness and gray value. The thickness of the oxide layer can be obtained by extracting the gray value of the surface of the test object. This measurement method has the advantages of low cost, non-contact, reliable measurement, convenient and fast.

1. Introduction

7050 aluminum alloy has the characteristics of light weight and high specific strength. When it is used in the manufacture of aluminum alloy cartridges, it has the advantages of high ammunition carrying capacity and low wear and tear on weapon parts [1-2]. However, there are many problems in the manufacturing process of 7050 aluminum alloy cartridges. The current research basically focuses on the microstructure evolution of aluminum alloys in different heating processes and the extrusion molding of aluminum alloys, and ignores the aluminum alloy hot extrusion forming surface. Influence of oxide layer.

Zhang Zhaohui, Liu Sisi [3] found that during the hot extrusion process of aluminum alloy, it was found that micro-cracks would be produced on the surface of aluminum alloy due to the effect of scale; Deng Caiqin [4] research showed that the surface of aluminum alloy extruded profiles had “burrs”. It is related to the oxide layer on the surface of aluminum alloy; Cai Yingduo [5] studied that in the late stage of aluminum alloy extrusion, the oxide layer on the surface of the ingot will come out along the cross section of the profile and continue to squeeze. The detached oxide layer will enter the profile. Affects the metal continuity of the material. It can be seen that the oxide layer on the surface of aluminum alloy will not only cause the surface quality of the work piece, but also have a serious impact on the mechanical properties of the work piece. In the 7050 aluminum alloy cartridge forming process, the hot extrusion process is the most susceptible to oxidation. It is necessary to understand the
degree of oxidation of the 7050 aluminum alloy before hot extrusion in order to improve the heating conditions and control the billet oxidation.

Peng Zhihui [6] et al. Studied the effect of temperature and holding time on the thickness of the oxide layer on the surface of the 4004 aluminum alloy, and proposed that the gloss and color of the oxide layer on the surface of the aluminum alloy can be observed to determine the degree of oxidation, but only the 4004 aluminum alloy was obtained. The oxidation rule at 475℃ can only judge the degree of oxidation macroscopically at other temperatures, and the specific thickness of the oxide layer cannot be obtained. This paper proposes a method for measuring the oxide layer thickness of 7050 aluminum alloy based on image processing, which can greatly reduce the difficulty of measuring the oxide layer thickness and has the advantages of convenience and speed.

2. Sample Preparation

The aluminum alloy used for the test was 7050-H112 aluminum alloy bar stock provided by Southwest Aluminum. Its chemical composition is shown in Table 1. The cylindrical sample cut from the bar material was used to remove the surface oil and oxide layer, and then placed in a box Furnace heating. The high temperature oxidation test is performed at 400 and 450°C, and one sample is taken every 1 h. The samples taken in turn are oil-cooled and gently wiped to avoid damaging the surface oxide layer, and the corresponding temperature and time are marked. In order to distinguish the samples under different insulation systems.

| Cr   | Cu  | Fe  | Mg  | Mn  | Si  | Ti  | Zn  | Zr  | Al  |
|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 0.01 | 2.3 | 0.08| 2.1 | 0.01| 0.03| 0.02| 6.2 | 0.12| else|

3. Gray value extraction

3.1. Real-time photo shooting of samples

The specimens subjected to high temperature oxidation at 400 °C were photographed on the end face under the same lighting conditions. As shown in Figure 1, the photos are kept at 400 °C for different times. It can be clearly observed that the surface color of the sample gradually deepens with the extension of the hold time. In order to visualize the change of the surface color of the sample, the gray value of the photo was extracted with Matlab. In order to prevent the gray value of the photo boundary from being mistakenly taken as the gray value of the sample photo, a part of the photo is selected as the photo to be processed, as shown in Figure 2.

![Figure 1. Samples treated at different temperatures at 400°C.](image)

![Figure 2. Screenshot of the end face of the sample after oxidation at 400°C.](image)
3.2. Gray value curve
Open Matlab, call the function ‘imread ()’ to read the photos to be processed, and then call the function ‘rgb2gray ()’ to grayscale the photos, and you can use the command ‘imshow ()’ to view the processed images [7]. As shown in Figure 3, the image is gray-scaled after high-temperature oxidation at 400°C.

![Figure 3. Image after graying process.](image)

Calculate the average value of all elements in the gray value matrix corresponding to the image, and make a graph of the gray value change with the holding time, as shown in Figure 4. It can be seen that the 7050 aluminum alloy is oxidized at 400°C at high temperature. In the initial stage of oxidation, the surface of the 7050 aluminum alloy changes from silvery white to light gray, and the gray value decreases rapidly. With the extension of the holding time, the surface color of the sample gradually deepening, the rate of decline of the gray value gradually slowed down, and after 10 hours of oxidation, the gray value basically did not change.

![Figure 4. Gray value curve of the sample at 400°C.](image)

4. 7050 aluminium alloy oxide thickness measurement

4.1. Measuring principle
The surface of 7050 aluminum alloy samples treated with different holding time was peeled layer by layer with Ar+ sputtering. XPS analysis was performed on the peeled surface to obtain the XPS spectrum. The XPS spectrum was used to determine the surface element composition and atomic fraction of the sample. When the peeling surface at a certain depth is reached, the atomic fraction of O atoms is basically unchanged, it is determined that the oxide layer has been peeled off, and the thickness of the oxide layer is marked at this depth [6]. Usually the etch rate of SiO₂ is taken as the standard, and the etch rate of other materials is divided by the etch rate of SiO₂ to obtain the relative etch rate. Therefore, it is only necessary to know the etching rate of SiO₂ under the same conditions to
know the etching rate of the required etching material. The thickness of the oxide layer can be calculated based on the atomic fraction diagram to obtain the time taken for the oxide layer to completely peel off.

4.2. X-ray Photoelectron Spectrometer (XPS) measurement results
The XPS analysis sample taken from the end face of the oxidation treated sample at 400 °C for 10 hours, the curve of Al, Mg, and O atomic fractions as a function of sputtering time was obtained. Before the complete stripping is completed, the O atom number fraction remains basically unchanged. As the stripping time increases, the O atom number fraction suddenly drops to a stable value, and then slowly decreases to gradually reach 0. It can be judged from Figure 5 that the time taken for the surface oxide layer of the 7050 aluminum alloy to completely peel off is 38 minutes.

![Figure 5. Ar+ sputtering after 10°C incubation O, Al, Mg atomic fraction.](image)

The instrument used for the measurement was an X-ray electron spectrometer manufactured by Japan Vacuum. The etching rate of SiO$_2$ was 8.5 nm / min, while the relative etching rate of Al$_2$O$_3$ was 0.5, and the known etching rate was 4.25 nm / min. From this, it can be calculated that the thickness of the oxide layer on the surface of the 7050 aluminum alloy after 16 hours of heat preservation is 161.5 nm.

4.3. Surface oxidation of 7050 aluminum alloy
After XPS analysis of samples with different holding times at 400 °C and 450 °C, the change rule of the thickness of the surface oxide layer of the 7050 aluminum alloy with holding time at 400 °C and 450 °C is shown in Figure 6. It can be seen that the oxidation rate of 7050 aluminum alloy increases significantly with the increase of the holding temperature, the limit thickness of the oxide layer also increases with the increase of temperature, and the higher the temperature, the shorter the time required to reach the limit thickness.

![Figure 6. Variation of oxide layer thickness with holding time.](image)

In addition, the thickness of the oxide layer of 7050 aluminum alloy becomes thicker with the increase of the heat preservation time. In the initial stage of heat preservation, the oxidation law roughly follows the logarithmic law. With the extension of the holding time, the oxidation law
generally follows the parabolic law. This is due to the partial recrystallization of the short-range ordered oxides of the oxide layer, and the γ-Al₂O₃ orientation nucleation, which destroys the continuity of the oxide layer [8], leading the samples continue to be oxidized. This is generally consistent with the rules found in the high temperature oxidation test of 4004 aluminum alloy by Peng Zhihui [6], etc. The difference is that the oxidation process of 7050 aluminum alloy directly transitions from a logarithmic law to a parabolic law, without a linear change law in the middle.

5. Conclusion
The one-to-one correspondence between the thickness of the oxide layer and the gray value of the sample is obtained, and the relationship curve shown in Figure 7 is made. It can be seen from the figure that the thickness of the oxide layer and the gray value are approximately linear, that is, the surface color of the aluminum alloy gradually deepens as the thickness of oxide layer increases.

![Figure 7. Relationship between gray value and oxide thickness.](image)

Therefore, in industrial production, only the gray value of the aluminum alloy surface needs to be extracted with Matlab, and the thickness of the oxide layer on the surface of the aluminum alloy can be obtained. The 7050 aluminum alloy that is kept at any time within the temperature range of 0 to 450°C has great practical value for enterprises.

References
[1] Wang Yusong. Research on forming process optimization and heat treatment process of 7050 aluminum alloy shell [D]. Chongqing University, 2015.
[2] Liu Xiaoxia. Research on Hot Extrusion Process of Aluminum Alloy Cartridges [D]. Nanjing University of Science and Technology, 2017.
[3] Zhang Chaohui, Liu Sisi, Sun Yuetao. Analysis of microcracks on cold rolled aluminum surface [J]. Journal of Beijing Jiaotong University, 2012, 36 (1): 108 - 111.
[4] Deng Caiqin. Discussion on the causes of "burrs" on the surface of 6063 aluminum alloy extruded profiles and preventive measures [C] .lw2004 Aluminum Profile Technology. 2004.
[5] Cai Yingduo. Common defects and countermeasures of 6000 series aluminum alloy extruded profiles [C]. National Academic Conference on Light Alloy Processing. 2000.
[6] Peng Zhihui, Wang Yan. Study on the surface constant temperature oxidation kinetics of 4004 aluminum alloy [J]. Journal of Central South University of Technology, 1998 (5): 479 - 482.
[7] Zhang Feige, Li Kai, Zhai Shaokang, et al. Application of MATLAB in Digital Image Processing [J]. Computer Technology and Development, 2019.
[8] Wefers K, Ren Binyin. Properties and characteristics of surface oxides on aluminum alloys [J]. Light Alloy Processing Technology, 1983 (3): 26 - 3.