THE STUDY ON RECRYSTALLIZATION ANNEALING THE PLATE OF HIGH-STRENGTH Al-Zn-Mg-Cu ALLOY

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Abstract: *High-strength Al-Zn-Mg-Cu alloy after casting ingot form is often rolled into plates of different thicknesses. To recover original structure after deformation, these aluminum alloy plates must be recrystallized. The recrystallization annealing process depends on the main factors such as annealing temperature and duration. Performance recrystallization was investigated through microstructure, scanning electron microscope (SEM) and hardness. By determining the microstructure, SEM and hardness of the plates of the Al-Zn-Mg-Cu alloy with different recrystallization annealing process, we chose the optimal process as followings annealing 415^oC for 150 minutes. The alloy plates after annealing achieve the uniform, fine microstructure and low hardness 67,2 HB.*

Keywords: Al-Zn-Mg-Cu alloy, 7075 alloy, Recrystallization.

1. INTRODUCTION

As we know it, high-strength Al-Zn-Mg-Cu alloy was being used widely in aerospace: valve, structure, air-frame; rocket engines and military equipment [4, 5]. The B95 alloy (Russia) equivalent to 7075Aluminium alloy (USA) is one of the most important engineering alloy [1, 3]. In recent years, we use B95 alloy to study the concave bullet instead of 40X steel (PG-7VL), stabilized wings of PG-9 bullet… The plate materials are used popular, so after the ingot molds rolled into sheets. At some defense factories, high-strength Al-Zn-Mg-Cu Aluminium alloy plate was manufactured from continuous-sold ingots (pouring temperature 690 °C ÷ 710 °C, casting speed 100 mm/min); annealing temperature 460^oC for 12 hours; extrusion at temperature 400^oC, thickness δ = 4,5 mm; the first rolling δ_1 = 4,3 mm, the second rolling δ_2 = 4,0 mm. After rolling into sheet, this plate material has changed the structure and properties, In order to return to the initial state the alloy need heat treatment (recrystallization) [1-3, 5, 6]. However, the choice of recrystallizating process after rolling ingots into sheets has not been fully investigated and published. So this paper, the authors investigated the suitable recrystallization process for the high-strength Al-Zn-Mg-Cu alloy produced at the factory to ensure stabilize the structure, fine grain, hardness is not high reached 67,2 HB.

2. EXPERIMENTAL

2.1. Equipment and materials for research

- Q4 TASMAN emission spectrometer, Germany;
- Nabertherm furnace temperature 30-650^oC, Germany;

- Struers cut-off equipment with cutting speed of 100-400 rpm, Denmark;
- Metal polishing equipment, USA;
- SiC paper have a particle size ranging from 120 to 2000;
- Cr_2O_3 polishing powder, size µm;
- Chemicals to etching: HCl, HF, HNO_3 of china; distilled water…
- AXIO A2M equipment, Germany;
- Brinel HP-250 Hardness Testers, Germany;

-JSM-7001FA FE-SEM Scanning Electron Microscope Analysis JEOL, Japan, magnification 2000 times at Shimane University, Japan.

2.2. Fabrication

*2.2.1. Fabricate r*recrystallization sample

- Fabricate the sample on Struers, cutting speed 200 rpm, size 10 x 10 x 4 mm;
- Examination of different tempering samples with a constant heat up rate of 5 °C/min in the temperature range of 250 ÷ 450 °C and a heat retention time of 30 ÷ 180 minutes; Cooling the same furnace.

2.2.2. Determine the microscopic organization of the sample

- Clip the sample using a dedicated tool or epoxy pour to position the flat smooth sample;
- Grinding SiC paper has different grain level increasing from 180 to 2000 ensuring flat, smooth, no visible scratches on the surface.;
- Polishing the specimen using a special machine, using Cr_2O_3 grinding powder ensure smooth surface;
- Etch based on Keller solution;

2.2.3. Determination of hardness, degree of recrystallization of sample

- Hardness of the sample on a Brinell HP-250 equipment with a load of 250 kg; D = 5 mm;
- The degree of recrystallization Xs (%) is determined by the expression: $Xs = (H_0-H)/(H_0-H_{rec})$ [5];

Where: Xs - crystallization; H_0 - initial plate hardness; H - hardness after recrystallization; H_{rec} - hardness after recrystallization completely.

3. RESULTS AND DISCUSSION

3.1. Survey of original materials

The sample was Al-Zn-Mg-Cu aluminum alloy rolled in the factory (symbol X59), the composition of Table 1.

Table 1. *Component composition X59, B95 (Russia) equivalent to 7075 (USA).*

Alloy sample	Composition of aluminum alloy,%									
	Fe	Si	Mn	Ni	Cr	Ti	Al	Cu	Mg	Zn
B95	Max	Max	0,2-	Max	0,1-	Max	86,3-	1,4-	1,8-	5,0-

(Russia)	0,5	0,5	0,6	0,1	0,25	0,05	91,5	2,0	2,8	7,0
X59	0,338	0,094	0,390	0,006	0,181	0,023	89,286	1,836	2,103	5,668

According to Table 1, X59 is a high strength Al-Zn-Mg-Cu aluminum alloy and has the same composition as B95 (Russia) or 7075 (USA).

To better understand the X59 the high strength aluminum alloy sheet specimen, the team conducted a microscopic imaging (Figure 1), SEM image (Figure 2), and determined the hardness of the sample after rolling H_0 is quite high, reaching an average of 123 HB.

The microstructure (Fig. 1) and the SEM image (Fig. 2) after extruding in the factory exhibited uneven distribution phases in the roll strip. Cause when the particles are deformed by the rolling method: The small phase hardness is not broken but dispersed into strips in parallel with the rolling, so it will affect the properties of the product compared to the original pattern.

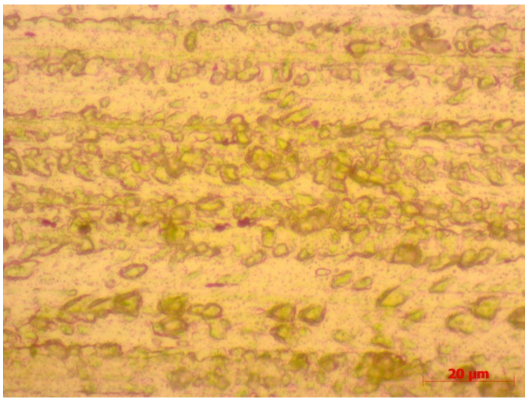


Figure 1. *Microstructure of the X59, X500.*

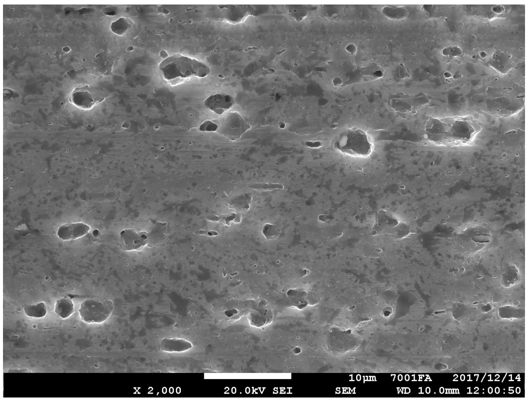
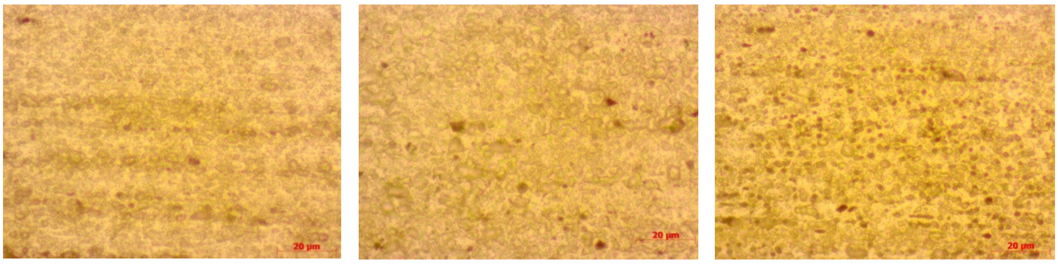


Figure 2. *SEM image X59, X2000.*

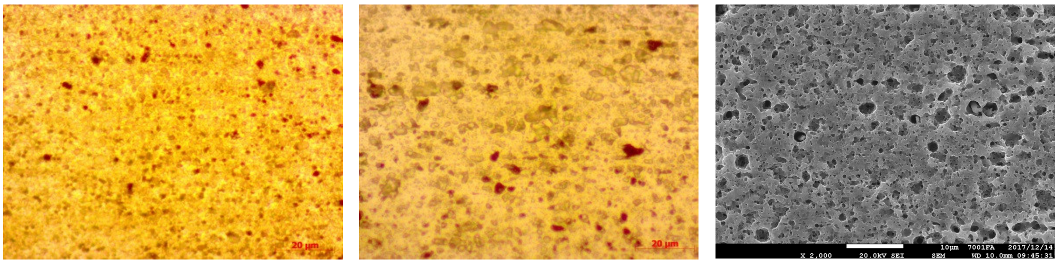
The alloys after rolling need to choose the suitable recrystallization mode for uniform component, stable structure and properties.

3.2. Survey of recrystallization temperature

The recrystallization temperature of the alloy was investigated by the authors at different temperatures from 250 °C to 450 °C. The results of the microstructure analysis, SEM images are shown in Figure 3.



a) At 250 °C b) At 300 °C c) At 350 °C



d) At 415 ^oC e) At 450 ^oC f) At 415 ^oC

***Fig. 3**. a-e) Image of the alloyed microstructure after recrystallization at different temperatures at 500 times magnification and f) SEM image at 415 °C, X2000*

The result of microscopic image of the alloy after recrystallization (Figure 3) showed that the post-recrystalline alloy samples had a more uniform structure, smoother fineness, and progressive strip loss than the original sample.

However, at different annealing temperatures there are different microstructures, at the higher temperature, the better the microscopic distribution, the gradual strips are removed. At the temperatures of 250 °C, 300 °C, 350 °C arranged in milled leaves are still but fading. This proves that recrystallization process at these temperatures is effective but not yet high. However, the annealing temperature is not sufficient to ensure that the structure is oriented smoothly. Increase recrystallization temperature to 415^{o}C, the microstructure of the alloy is dispersed phase, small smooth, no longer see the rolling strip (Figure 3d). This is explained that at 415^{o}C, the first recrystallization process occurs completely, the microstructure is uniform, not yet recombined. This is also consistent with the SEM image observation (Figure 3f) showing that the dispersed phases are smooth, no longer smooth strips such as the original X59 (Figure 2). As the temperature rises to 450^{o}C, the microstructure of the alloy consists of several large phases mixed with little phase and no strips in the microstructure of the sample (Figure 3e). This may be due to the fact that at a temperature of 450 °C, the first recrystallization process has occurred completely and the phenomenon of the second recrystallization phase begins. The second recrystallization process is undesirable as it affects the uniformity of the microstructure and the properties of the reduced material.

To better understand the influence of temperature on the mechanical properties of materials, the team determined the hardness of the alloy at different recrystallization temperatures. The results are shown in table 2.

The results showed that the alloys after tempering decreased hardness compared to the X59 sample (123 HB). However, at different annealing temperatures, the hardness decreases and the degree of recrystallization of the alloy is different.

Table 2. Hardness of the alloy after recrystallization at different temperatures.

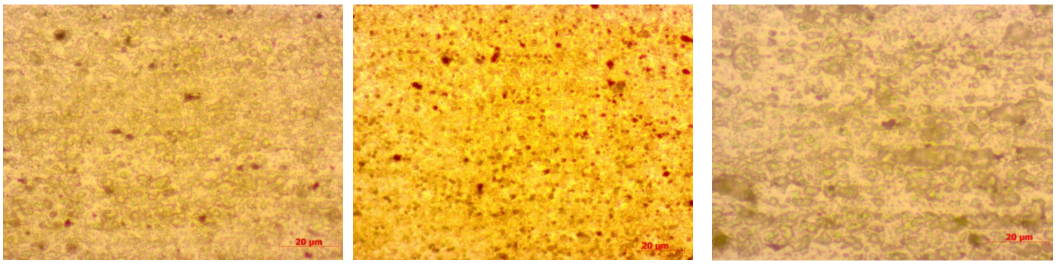
Recrystallization temperatures, °C	Hardness, HB						Xs, %
	At 1	At 2	At 3	Average	H_0	H_{rec}	
250	76,0	77,0	76,0	76,3	123	67,2	83,69
300	72,0	72,5	73,0	72,5	123	67,2	90,50
350	68,9	68,9	68,9	68,9	123	67,2	96,95
400	67,7	67,8	67,9	67,8	123	67,2	98,93
415	67,2	67,2	67,2	67,2	123	67,2	100
450	67,1	67,2	67,3	67,2	123	67,2	100

As the annealing temperature increases, the hardness decreases, the recrystallization efficiency increases. The smallest hardness was 67,2 HB, the recrystallization efficiency was 100% at annealing temperature of 415°C to 450°C. This result is perfectly consistent with the microstructure obtained in Figure 3.

3.3. Survey of recrystallization time

The authors investigated the tempering samples at 415 °C, the heating rate of 5 °C/min with the period time varied from 30 minutes to 180 minutes. The results of the microstructure analysis are shown in Figure 4.

It has been observed from Figure 4, that the recrystallization time for 120 minutes, the microstructure of the alloy consists of small, alternating, unevenly distributed layers (Fig. 4a). When the period time is increased to 150 minutes, the microstructure of the alloy is smooth, evenly distributed, no longer recognizing the strips (Fig. 4b). This will be done because of annealing temperature at 120 minutes the first recrystallization has been done but hasn't been done completed when the last 150 minutes the first recrystallization has been done completely. If continue to increase at 415°C for 180 minutes, the alloy microstructure includes several multi phase, many large phase and low fine phase, lost rolling strip. This showed that, when increasing the time to 180 minutes, the second recrystallization occurred, the merged phases grew and the fine phases had been dissolved. This is an unwanted crystallization stage in the recrystallization process of the alloy.



a) At 120 minutes   b) At 150 minutes   c) At 180 minutes

Figure 4. *The microstructure image of the alloy after crystallization at 415°C for different period times with magnification of 500 times.*

To understand the effect of recrystallization time to be better, the authors investigated the hardness of the alloy at different period times (Table 3).

Table 3. *Hardness of the alloy after recrystallization at different times.*

Recrystallization time, minute	Hardness, HB						Xs, %
	At 1	At 2	At 3	Average	H_0	H_{rec}	
30	76,4	76,4	76,7	76,5	123	67,2	83,33
60	70,7	70,6	70,5	70,6	123	67,2	93,91
90	70,4	70,1	70,1	70,2	123	67,2	94,62
120	69,5	69,6	69,4	69,5	123	67,2	95,88
150	67,2	67,2	67,2	67,2	123	67,2	100
180	67,1	67,2	67,3	67,2	123	67,2	100

The results showed that the sample after annealing had a much lower hardness than the original alloy. Hardness decreases much and efficiency increases as the annealing time increases. Recrystallization occurs completely when the period time is 150 minutes. When increasing the annealing time for 180 minutes, the hardness of the alloy does not change.

Thus, the recrystallization optimized for the X59 aluminum alloy plates is 150 minutes at 415 °C, the obtained product is organized micro-fine phase, smooth distribution, no longer strips, hardness achieved smallest 67,2 HB and the first recrystallization process occurred completely.

4. CONCLUSION

From the results of the study on the recrystallization of the X59 alloy plates after rolling the high-strength Al-Zn-Mg-Cu aluminum alloy, the authors show some conclusions:

- The aluminum alloy sheets after rolling should be tempered to recrystallize to stabilize the organization, structure and properties.
- The recrystallization was completely crystallized for the X59 specimens after rolling at a temperature of 415 °C for 150 minutes, speed of heat up 5 °C/min. After recrystallizing, the alloy is no longer oriented according to the rolling direction, the fineness is smooth, dispersed, low hardness average 67,2 HB.

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TÓM TẮT

NGHIÊN CỨU CHẾ ĐỘ Ủ KẾT TINH LẠI TẤM HỢP KIM NHÔM
ĐỘ BỀN CAO HỆ Al-Zn-Mg-Cu

Hợp kim nhôm độ bền cao hệ Al-Zn-Mg-Cu sau khi đúc dạng thỏi thường được cán thành các tấm có chiều dày khác nhau. Để trở lại tổ chức ban đầu sau khi biến dạng, các tấm hợp kim nhôm phải tiến hành ủ kết tinh lại. Chế độ độ ủ kết tinh lại phụ thuộc vào các yếu tố chính như: nhiệt độ, thời gian ủ. Chất lượng ủ kết tinh lại được đánh giá bởi tổ chức tế vi, ảnh hiển vi điện tử quét (SEM) và độ cứng của hợp kim. Bằng phương pháp xác định tổ chức tế vi, ảnh SEM và độ cứng của hợp kim nhôm dạng tấm hệ Al-Zn-Mg-Cu ở các chế độ ủ kết tinh lại khác nhau cho thấy chế độ ủ kết tinh lại hợp lý là nhiệt độ 415 ^{o}C, giữ nhiệt 150 phút. Phôi tấm sau khi ủ có tổ chức đồng đều, độ cứng đạt 67,2 HB.

Từ khóa: Hợp kim Al-Zn-Mg-Cu, Hợp kim 7075, Ủ kết tinh lại.

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