

MICROSTRUCTURAL EVOLUTION AS WELL AS MECHANICAL AND ELECTRICAL PROPERTIES OF AA 6101 WIRE DURING RECRYSTALLIZATION ANNEALING TREATMENT

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Abstract

AA 6101 wire alloy has been used in the electric transmission lines in company of the cable industries of Biskra (Algeria) for several years. In this work, the AA 6101 alloy was subjected to recrystallization annealing treatment at 400 °C and holding for 30 min, to see the effects of this treatment on the mechanical and electrical properties of drawn Al-Mg-Si wire. The recrystallization annealing treatment leads to the microstructural changes of the textured form, which causes the softening of the wire, that is to say an increasing of ductility. On the other hand, this heat treatment accelerates the recrystallization mechanism, accompanied by a softening of the wires and a reduction in the electrical resistivity. Characterization methods used in this work were: Electron back scattered diffraction (EBSD), X-Ray diffraction analysis (XRD), Vickers microhardness, tensile test, measuring electrical resistivity, scanning electron microscope (SEM) and energy diffraction spectrometer (EDS).

Keywords: AA6101 wire alloy, recrystallization, mechanical and electrical properties

1. INTRODUCTION

Aluminum today is the leading non-ferrous metal in use, especially in the sectors of transport, beverage containers, packaging, and electrical and aeronautics sectors.

Al-Mg-Si alloys (6xxx-series alloys) are structural hardening alloys. They have interesting general properties with a good ability to hot deformation by rolling and cold drawing, which facilitates their shaping.

Wire drawing is a metalworking process used to reduce the cross-section of a wire by pulling the wire through a single, or series of drawing. It mainly uses the plastic deformation for the reduction of the wire.

A cold deformation of this kind causes the work hardening. This work-hardening depends on the chemical composition of the material, deformation level and the deformation conditions (temperature, speed and deformation mode). In fact, the cold plastic deformation causes a significant increase in the dislocation density (i.e. stored elastic energy) in the material. The work-hardening lengthens the grains in the direction of traction, which results in giving the properties of the material an anisotropic character [1].

When the limit of the work-hardening is reached, it is necessary to perform an annealing heat treatment which restores mechanical and electrical properties of the wire. On the micrographic structure plane, the

recrystallization annealing results in the transformation of the cold (fibrous) structure into a recrystallized structure. This explains the decrease in the total number of crystalline defects in the material.

The scope of this work is to investigate the microstructural evolution and electrical properties of AA 6101 wire during recrystallization annealing treatment by using the electron back scattered diffraction (EBSD), X-ray diffraction analysis (XRD), Vickers microhardness, tensile test, measuring electrical resistivity, scanning electron microscope (SEM) and energy diffraction spectrometer (EDS).

2. EXPERIMENTAL METHODS

The Al-Mg-Si alloy was provided by the company MIDAL CABLES (BAHRAIN), in the form of wire coils from initial diameter of 9.5 mm with three different level of deformation ($\epsilon_1 = 21.3\%$, $\epsilon_2 = 69\%$ and $\epsilon_3 = 86.8\%$). The chemical composition of the investigated alloy is given in **Table 1**.

Table 1 Chemical composition (wt.%) of studied material aluminum alloy (6101 Series)

Al (97.34)	Mg	Si	Cu	Fe	Pb	V	Zn
	1.34	0.51	0.27	0.21	0.20	0.05	0.08

3. RESULT AND DISCUSSION

3.1. Characterization by (SEM / EDS) in non-annealed drawing

The chemical composition of the as received wire (**Figure 1**) and the strongly drawn wire (**Figure 2**) was carried out by EDS. The main elements are presented in **Table 1**. The Al-Mg-Si aluminum samples show a microstructure of elongated grains along the drawing wire axis, depending on the deformation level, the wire acquires a fibrous textured microstructure [2] (**Figure 2**).

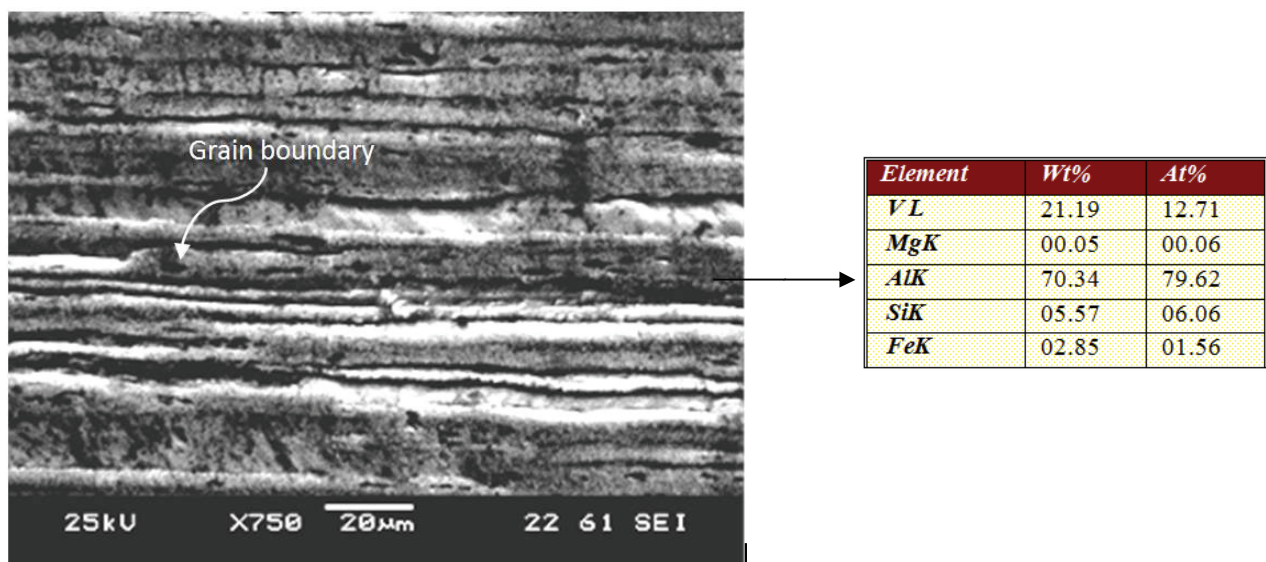


Figure 1 SEM/EDS analysis of as received wire $\epsilon = 0\%$ (USTHB Alger)

A difference in the amount of chemical elements such as Si and Mg were observed. On the other hand, at the level of the grain boundary, a distribution of small elongated black collector sizes was observed in the direction

of drawing. Analysis by EDX showed that the percentage of vanadium is greater than that of the other two chemical elements (Mg and Si).

A difference in the amount of chemical elements such as Si and Mg is observed in the two cases of drawn wire at $\epsilon = 86.8\%$ and as received wire $\epsilon = 0\%$. Moreover, a distribution of small elongated black collector sizes randomly dispersed within the grains was observed in the direction of drawing. Analysis by EDS showed that the percentage of vanadium is greater than that of the other two chemical elements (Mg and Si). This is probably due to the formation of AlVMg phase [3].

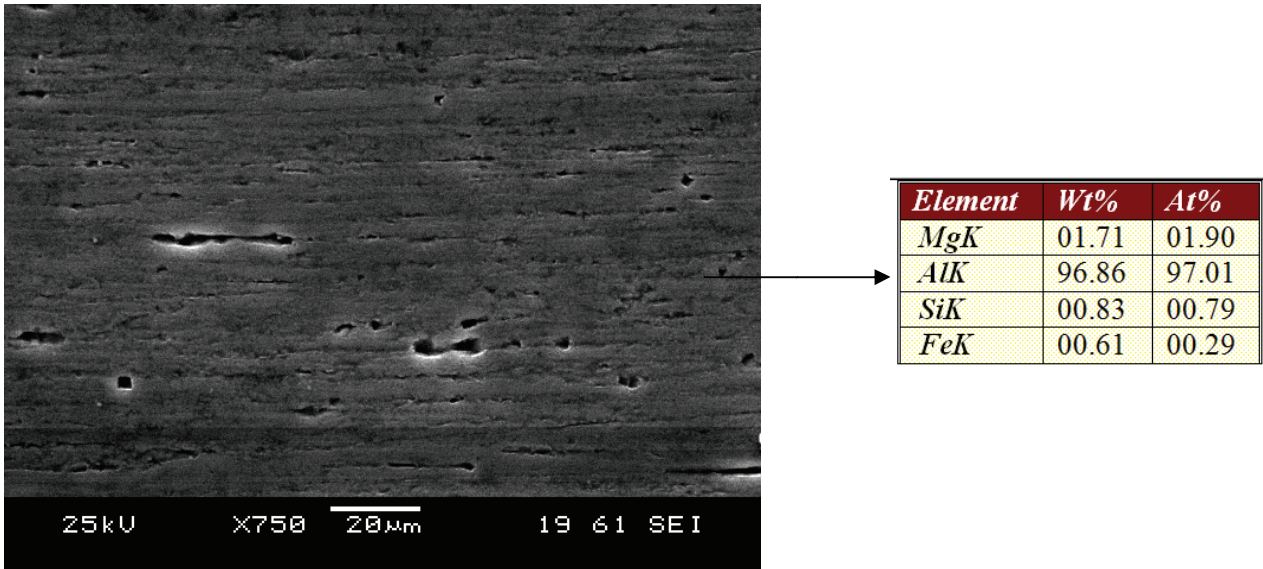


Figure 2 SEM/EDS analysis of drawn wire at $\epsilon = 86.8\%$ (USTHB Alger)

3.2. Tensile test and electrical resistivity

The electrical resistance measurements were performed by a four-point probe method, using a rig designed and made in our laboratory. The applied current was reversed (30 HZ) in order to eliminate uncertainty in measuring the voltage drop across the sample due to drift of the zero point. The current amplitude was kept low (10-20 mA) to inhibit the sample heating during the measurement period. Five Vickers hardness measurements were taken and averaged for each of the samples.

Results of tensile test, hardness measurements and Vickers hardness are presented in **Figure 3**.

From **Figures 3a** and **3b**, we can deduce that the elongation of the non-annealed drawing wire decreases and the mechanical resistance increases when the reduction rate increases compared to the as received wire [4]. This phenomenon is due to the increase in dislocation density during plastic deformation by drawing.

For an extruded drawn wire and annealed at 400 °C for 30 min we note a significant reduction in the tensile strength compared to the non-annealed drawing wire with an increase in elongation. This is due to the softening effect of the temperature, which tends to reduce internal dislocation density and residual stresses.

From the results shown in **Figure 3d**, it will be noted that the resistivity measurements show an increase in the resistivity of the non-annealed drawn wires with the deformation level. The annealing of the electrical wires at temperature 400 °C accelerates the recrystallization mechanism, accompanied by a softening of the wires and a reduction in the electrical resistivity.

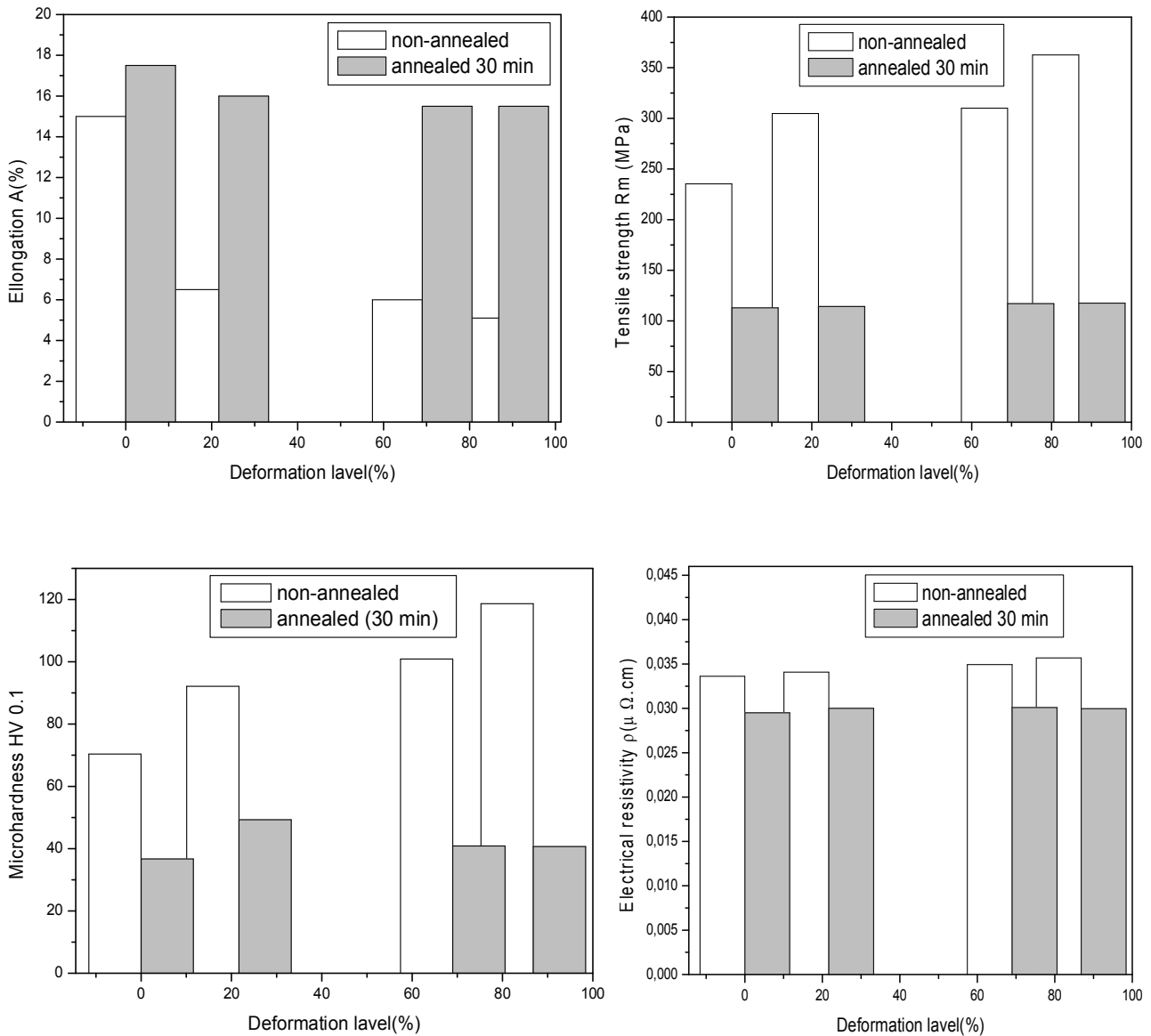


Figure 3 Results of tensile tests and electrical resistivity of as received ($\epsilon_1 = 0\%$) and drawn wires ($\epsilon_1 = 21.3\%$, $\epsilon_2 = 69\%$ and $\epsilon_3 = 86.8\%$) in non-annealed and annealed state determined by ENICAB Company of Biskra

3.3. Analysis by XRD patterns

X-ray diffraction analysis (XRD) gives us information on the evolution of the structure, the variation of certain crystallographic quantities. **Figure 4** shows the X-ray diffraction spectrum of drawn wire (deformation $\epsilon = 86.8\%$) in the non-annealed state and after annealing at 400 °C for 30 min

The Al-Mg-Si alloys (6xxx) are hardened by the metastable phase β'' (Mg_5Si_6) [5]. β'' was a coherent strengthening phase, which was in the form of needles coherent precipitate growing along a $\langle 100 \rangle$ direction of the Al matrix [6]. The structure of β'' phase was identified as monoclinic [7, 8] with lattice constants of $a = 0.77$ nm, $b = 0.67$ nm and $c = 0.405$ nm.

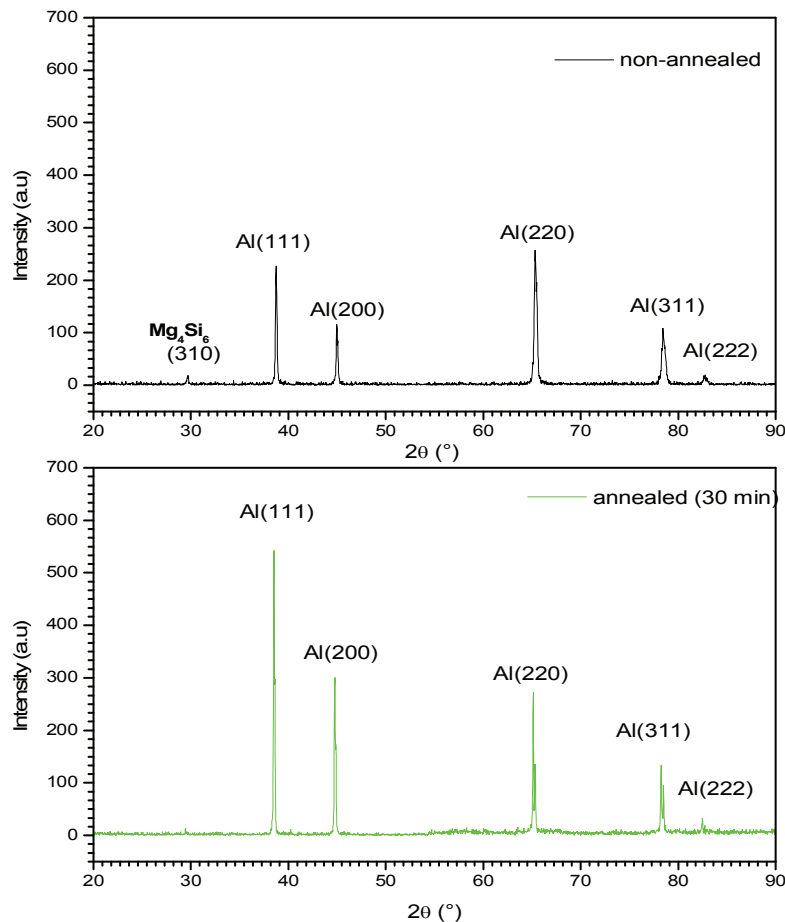


Figure 4 XRD pattern of AGS drawn wire at $\epsilon = 86.8\%$. (Non-annealed and annealed 30 min)

In the case of the sample deformed at $\epsilon = 86.8\%$ a new peak was observed at the position of about $2\theta = 29.66^\circ$. This recorded peak corresponds to Mg_4Si_6 precipitates (based on ASTM files), which explains the presence of intermetallic monoclinic crystalline system whose mesh parameters [$a = 1.516$ nm, $b = 0.405$ nm and $c = 0.674$ nm] in a solid solution of aluminum. We observe the displacement of the peaks towards the small angles. The intensity of the peaks during annealing is observed. The increase of the peak intensity during annealing was observed. That is, the elementary mesh of the crystal lattice of the material was affected.

4. CONCLUSION

In the annealing heat treatment and holding for 30 minutes, the recrystallization process abruptly reduced the breaking load (R_m), the microhardness (HV) and the electrical resistivity (ρ).

From the X-ray diffraction results, the increase in the intensity of the peaks and shifts of the peaks towards the small angles during annealing was observed. This recrystallization eliminated structural defects such as the intermetallic compound element Mg_4Si_6 .

REFERENCES

- [1] MONTEIRO, W. A., ESPÓBITO, I. M., FERRARI, R. B., BUSO, S. J. Microstructural and mechanical characterization after thermomechanical treatments in 6063 aluminum alloy. *Materials Sciences and applications*, 2011, no. 2, pp. 1529-1541.
- [2] ZIDANI, M., HADID, M. D., DJIMAOUI, T., FARH, H., MESSAOUDI, S., BESSAIS, L., MATHON, M. H., BAUDIN, T. Annealing effect at low temperature on the evolution of the microstructure, mechanical and electrical properties

- of a drawn aluminum wire. In *METAL 2016: 25th International Conference on Metallurgy and Materials*. Ostrava: TANGER, 2016, pp. 1595-1599.
- [3] MENG, Y., CUI, J., ZHAO, Z., ZUO, Y. Effect of vanadium on the microstructures and mechanical properties of an Al-Mg-Si-Cu-Cr-Ti alloy of 6XXX series. *Journal of alloy and compounds*, 2013, vol. 573, pp. 102-111.
- [4] ZIDANI, M., HADID, M. D., DJIMAOUI, T., MESSAOUDI, S., BESSAIS, L., MIROUD, D., FARH, H., MATHON, M. H., BAUDIN, T. The influence of aging on industrially cold drawn aluminum alloy (6101) used in the electric transmission lines. *International Journal of Engineering Research in Africa*, 2016, vol. 24, pp. 9-16.
- [5] BENOIT, A., BAUDIN, T., PAILLARD, P., MOTTIN, J. B. Homogeneous welding of the 6061 aluminum alloy using MIG CMT. In *Proceedings of 9th International Conference on Trends in Welding Research*, June 4-8 2012, Chicago, USA, 2013, pp. 125-128.
- [6] ANDERSEN, S. J., ZANDBERGEN, H. W., JANSEN, J., TUNDAL, U., REISO, O. The crystal structure of the β'' phase in Al - Mg - Si alloys. *Acta Metallurgica*, 1998, vol. 46, pp. 3283-3298.
- [7] MATSUDA, K., NAOI, T., FUJII, K., UETANI, Y. Crystal structure of the β' phase in an Al-1.0 mass.% Mg 0.4 mass% Si alloy. *Materials Science and Engineering A*, 1999, vol. 262, pp. 232-237.
- [8] ANDERSEN, S. J., MARIOARA, C. D., TORSÆTER, B. M., EHLERS, R. F. J., HOLMESTAD, R., REISO, O., RØYSET, J. Behind structure and relation of precipitates in Al-Mg-Si and related alloys. In *12th International Conference on Aluminium Alloys*, September 5-9, 2010, Yokohama, Japan Aluminium Alloys, pp. 413-419.