STUDY OF THE PRECIPITATION IN AGS 6101 ALUMINUM ALLOY WIRES AT ROOM TEMPERATURE AND THE EFFECT OF DEFORMATION

Salim MESSAOUDI [1], Mosbah ZIDANI [1], Mohamed Djamel HADID [1], Marie Hélène MATHON [2], Anne-Laure HELBERT [3], François BRISSET[3] and Thierry BAUDIN [3]

[1]*Laboratory of Energy and materials, University of Biskra, Biskra, Algeria*
[2]*Léon Brillouin Laboratory, CEA (DSM-DRECAM) - CNRS, CEA Saclay, Gif sur Yvette, France, EU*
[3]*ICMMO, SP2M, Univ Paris-Sud, Université Paris-Saclay, UMR CNRS 8182, Orsay Cedex, France, EU*

Abstract

In this study, we will examine the phenomenon of precipitation that appears in the aluminum alloy AGS wires. Since the studied wires are drawn, we will try to see also the effect of the deformation on this phenomenon.

The precipitation was believed to be the main cause of hardening in the AGS 6101 wire rod. The hardening appears in this wire, after a period of storage, and causes the elevation of its hardness from 7% to 18% and its maximum tensile load from 4% to more than 22% depending on storage times.

Microstructural analysis of wire rod and drawn wires shows the existence of precipitates in the microstructures. Optical microscopy reveals the presence of two precipitates of different sizes and colors: whitish and dark gray. Under the scanning electron microscope, two types of precipitates appear: light gray and gray, ranging from 3 to 12 μm.

X-ray diffraction analysis did not reveal the presence of precipitates, observed by light microscopy and scanning electron microscopy. The EDS analysis allowed the measurement of the chemical composition of the light gray precipitates that were identified as the Mg_2Si intermetallic compound.

Note that the effect of deformation on precipitation in AGS 6101 alloy was not clearly found in this study. Because the wires used in this study were part of the same batch as the wire rod, in part. On the other part, the exploitation of background signal with XRD software does not seem very convincing.

Keywords: Aluminum alloy, AGS 6101 alloy, precipitation, wire drawn, intermetallic compound

1. INTRODUCTION

The phenomenon of precipitation in Mg-Si aluminum alloys is known since the 1950s [1, 2]. Since then, a precipitation sequence has been established for this system and assigned to the pseudo-binary system Al-Mg_2Si [3-6]. Although this sequence seems to depend solely on the ratio Mg / Si, however the overall precipitation reaction must take into account the presence of other elements in this alloy. There is a lot of works on the precipitation in this alloy after a heat treatment [7-15], but almost no work on precipitation at room temperature [16]. After a heat treatment, the precipitation leads to the formation of certain types of precipitates frequent and currently well-known like; Mg_2Al_3, the iron intermetallic precipitates such as, AlFeSi, $Al_xFe_ySi_z$ [3,4,8,14-17], AL_5FeSi [9], Al_7Cu_2Fe [18], others intermetallic precipitates: $Al_{12}Mn_3Si$, [16, 18] Al_2Cu [18] and the famous Mg_2Si [3-18].

In our case, the space of about five months, the microhardness measurements gave different values on the same wires. The values of the second measurement were significantly higher than that of the first measurement. This observation indicates the hardening of AGS 6101 alloy wires over time. So, to understand the cause of this hardening, we undertook this study.

2. STAYED MATERIAL

The studied material, presented in previous studies [19], is the aluminum alloy type AGS 6101, whose wire rod is produced by continuous casting. Note that for constraints related to the supplier of the material, we limit our study to four threads: the wire rod and three deformed wires; as illustrated in **Table 1**.

Table 1 The deformations (wires) studied

Designed wire	Wire rod	ε_1	ε_2	ε_3
Deformation level (%)	0	21	69	86.8≅87

3. NATUREL AGING

For the study of natural aging, the wires are left in the open air at room temperature. In the region where this study is carried out, the temperatures vary according to the season, in winter the temperatures are between 15 °C and 25 °C in a day and between 07 °C and 12 °C at night, in summer the temperature is in average between 40 °C and 55 °C. Measurements and characterizations were conducted according to the availability of means.

4. EVOLUTION OF MECHANICAL PROPERTIES

Figure 1 illustrates the evolution of microhardness as a function of time, as can clearly be seen in this figure, there is an increase in the microhardness of the all wires over time. In five months the hardness of the wire rod has increased by about 07% and in about four years it exceeds by 35% its first measured value. It is the same trend for drawn wires; over the entire period of this study (approximately four years) the hardness of the deformed wire at 87% increased by approximately 22%.

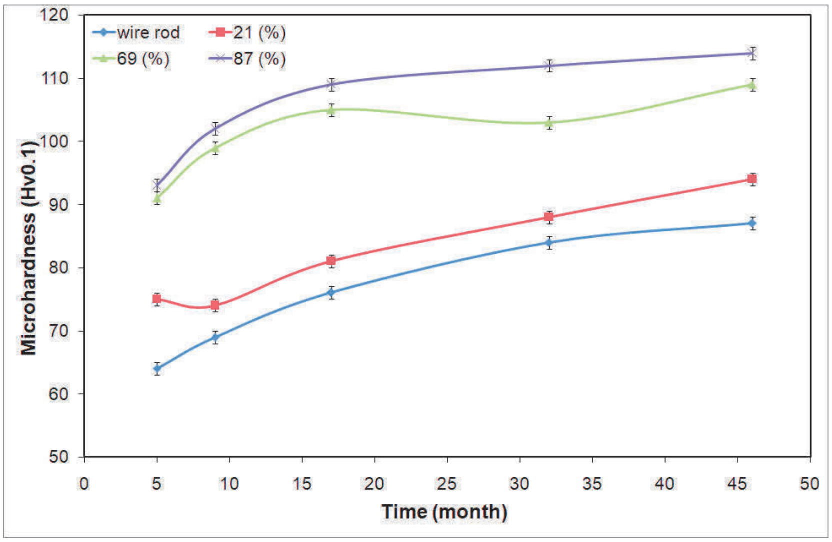

Figure 1 Evolution of the microhardness as a function of time

Tensile tests were also performed on these wires over time; **Figure 2** illustrates the data relating to the maximum tensile load. Similarly, as for the microhardness, it is clear in this figure the increase in the maximum load as a function of time for all the wires. Over the entire duration of the study, the increase in the load reached about 20% of its initial measurement for the wire rod and about 15% for the wire deformed at 87%.

These results reveal the hardening of the wire rod and the drawn wires of the AGS 6101 aluminum alloy. Due to a natural stress-free aging, the only phenomenon responsible for this hardening can only be attributed to the precipitation phenomenon known for these alloys. However, in spite of the differences which exist between the wire rod and the drawn wire, it can be said that the precipitation is more fluid in the wire rod than in the deformed wires.

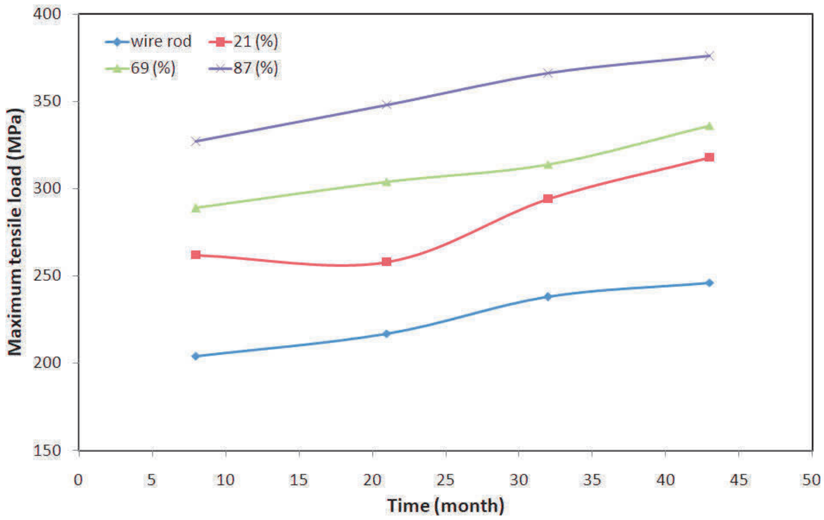

Figure 2 Evolution of the maximum tensile load as a function of time

5. MICROSTRUCTURAL ANALYSIS

5.1. Optical Microscopy (OM)

Figure 3 illustrates the optical micrographs of the wire rod and the 87% deformed wire. These micrographs were taken at two different periods; they clearly reveal the presence of precipitates in both wires. With unpolarized ligth, we can better see the contrasts and morphology of the precipitates, but we cannot identify there types clearly; we can only differentiate them according to their shape and color. On the wire rod, we can see dark-colored and light-colored precipitates. The dark-colored precipitates have a globular morphology, they are few but bigger than the light-colored ones. The light-colored precipitates are two kinds, globular shape, very numerous, precipitate in matrix and linear shape precipitates at the grain boundary. On the deformed wire (**Figure 3 (b)**), we also see two precipitates; light gray and dark gray. We can see the elongated and aligned precipitates (deformed precipitate), granular precipitates (not deformed precipitate) and tubular precipitates or intermetallic compounds according to the literature. This result confirms the hypothesis of further precipitation in the deformed wire similarly to that of the wire rod.

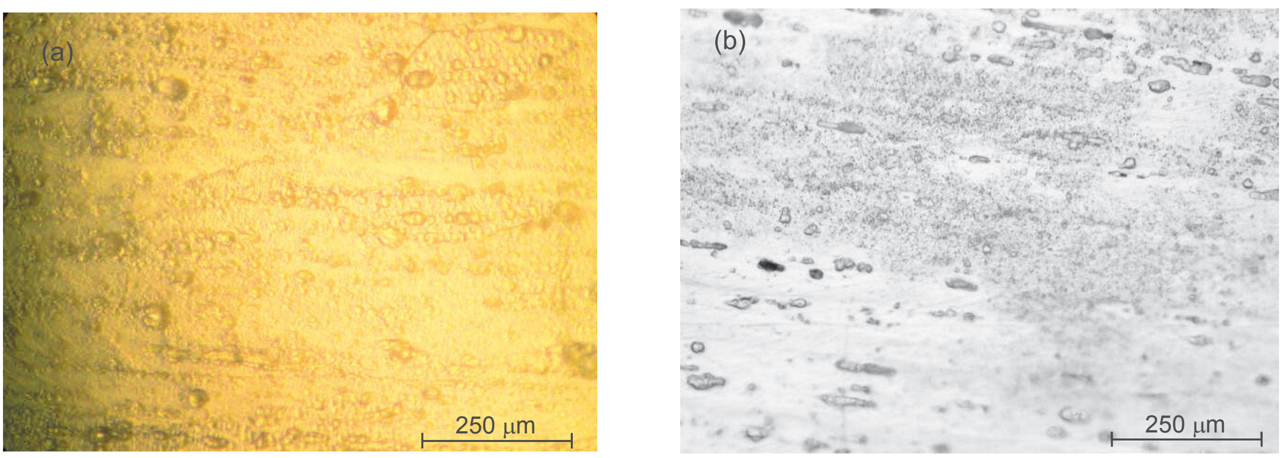

Figure 3 Optical micrographs: (a) wire rod, (b) wire drawn at 21%; observed at different time periods

5.2. Scanning Electron Microscopy (SEM)

For better observation and in order to identify the chemical composition of the classified precipitates, a microstructural analysis was carried out under a scanning electron microscope (**Figure 4**). Under the scanning electron microscope, the wire rod shows two types of precipitates appear: many of light gray precipitates and numerous of gray precipitates. On this wire, we have identified the famous precipitate Mg_2Si whose chemical composition is recorded in the **Table 2**. For the wire deformed, as can be seen in **Figure 4 (b),** two types of precipitates can also be distinguished; light gray precipitates and black precipitates. the light gray precipitates have two different morphologies, those elongated and aligned in parallel with the drawing axis of the deformed wire and those of granular shape less numerous and scattered in the matrix of the alloy. The black precipitates, hardly discernible, are also aligned along the drawing axis of the deformed wire.

Table 2 Local analysis of the chemical composition of the Mg_2Si precipitate carried out by EDX

Element	MgK	SiK
At (%)	64.86	35.14

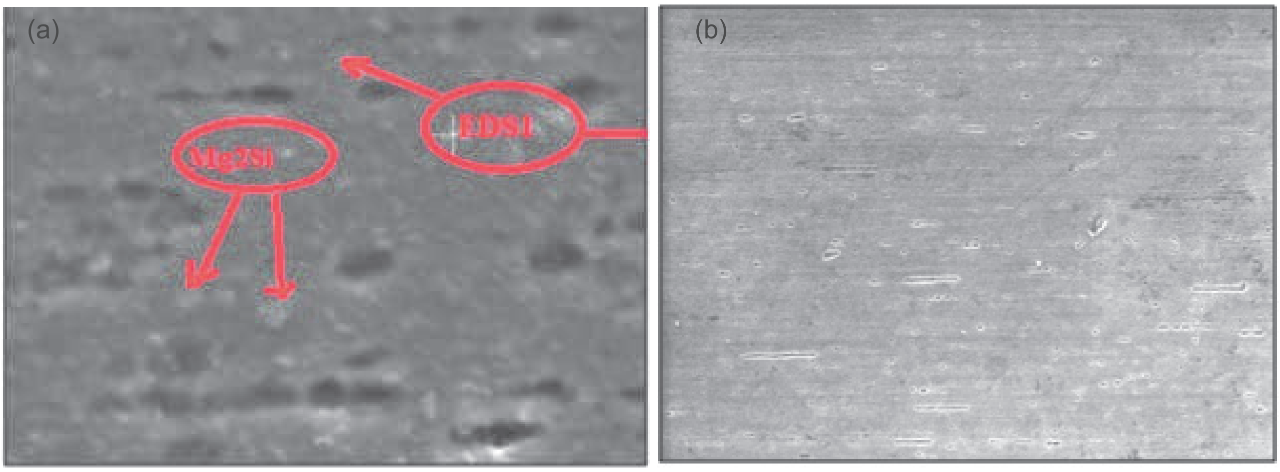

Figure 4 Scanning electron micrographs: (a) wire rod, (b) wire drawn at 87%; observed at same time

6. X-RAY ANALYSIS

X-ray diffraction analysis reveals only the diffraction peaks of the alpha phase (α-Al) of aluminum (α-Al) for all wires. But on the diagram of the strongly deformed wire (ε_3 = 87%), already presented in a previous study [19], after 3 years appears a new peak, with a good intensity, at the angle 2 Theta = 29.83° on diffraction (200). This peak was attributed to the precipitate Al_2Cu. The background analysis, by software XRD, makes it possible to extract some peaks masked by the background noise of the XRD technique. With this method, the famous precipitate Mg_2Si (observed by SEM) has been revealed at diffractions (111), (220), (222) and (311) (**Figures 5, 6**). Another precipitate of $AlMg_4Si$ type appears on the diffraction (111) for the deformations (69% and 87%). An iron-intermetallic compound $AlFe_3Si_{0.7}$ was also extracted at 2 Theta = 82.79° on diffraction (422) in deformed wire (21%). These results indicate that the deformation may have an effect on the precipitation in the AGS 6101 alloy. However, they are to be taken as a precaution, because as it was said the extracted peaks were confused with the background signal.

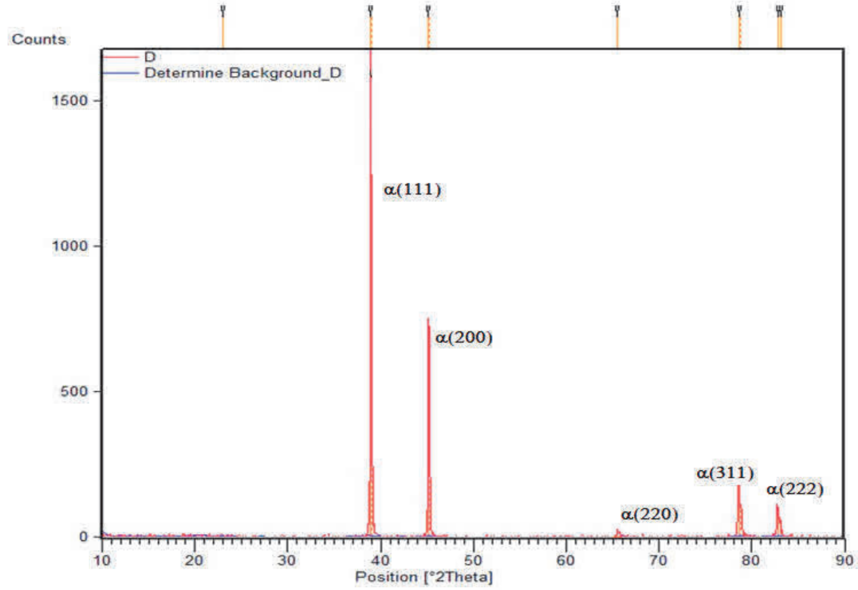

Figure 5 XRD-Background Analysis Method

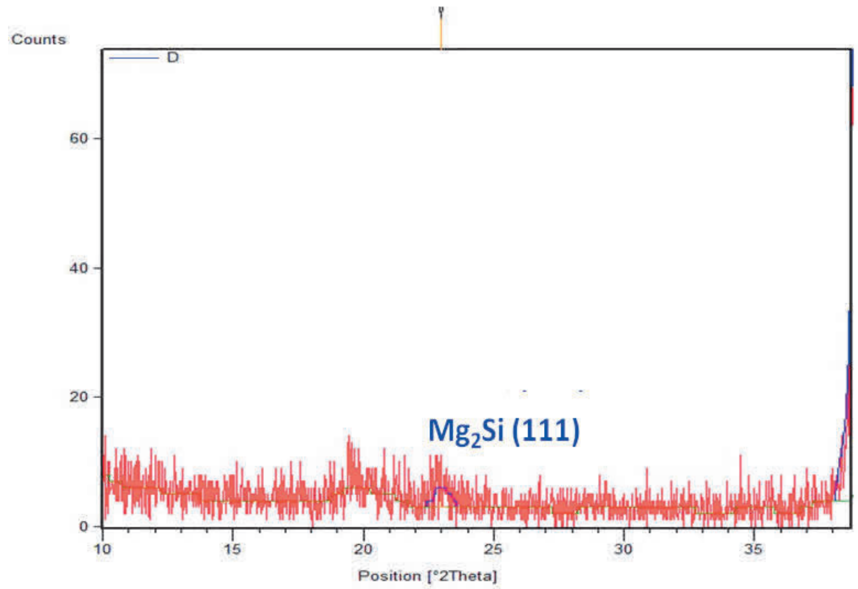

Figure 6 Example of Result of XRD-Background Analysis Method

7. CONCLUSION

From the results obtained previously, we can conclude the following points:

- The storage of AGS 6101 alloy wires under ambient conditions leads to their hardening.
- The phenomenon responsible for the hardening of the wire rod and drawn wire is precipitation.
- Precipitation is much more fluid over time in the wire rod than in the drawn wires.
- This results indicate that precipitation continues in the AGS 6101 alloy after deformation and highlighted in a global way the effect of deformation on precipitation.
- From all the observed precipitates only the Mg_2Si precipitate was been accurately identified.

REFERENCES

[1] LAMBOT, H. Etude cristallographique de la précipitation structurale dans les duralumins. *Rev. Met. Paris*.1950. vol. 47, no. 10, pp. 709-726.

[2] SAULNIER, A. Etude des alliages aluminium-magnésium. *Rev. Met. Paris*. 1956. vol. 53, no 4, pp. 285-297.

[3] HAMDI, I. Phénomène de précipitation des phases dans l'alliage d'aluminium (série 6000). *Doctoral Thesis*. Alegria: University of Biskra, 2014. p. 119.

[4] AOUABDIA, Y. Etude de la décomposition et de la précipitation dans des tôles des alliages Al-Mg-Si. *Doctoral Thesis*. Alegria: University of Constantine, 2011. p. 112.

[5] DE GEUSER, F. Interprétation et traitement des données de sonde atomique tomographique: application à la precipitation dans les Al-Mg-Si. *Doctoral Thesis. France:* University of Rouen, 2006. p. 135.

[6] MILKEREIT, B., WANDERKA, N., SCHICK, C. and KESSLER, O. Continuous cooling precipitation diagrams of Al-Mg-Si alloys. *Materials Science and Engineering A*. 2012. vol. 550, pp. 87-96.

[7] BIROL, Y. Effect of cooling rate on precipitation during homogenization cooling in an excess silicon AlMgSi alloy. *Materials Characterization*. 2012. vol. 73, pp. 37-42.

[8] AIZA, J., AZMI, R., ZUHAILAWATI, H. and ISMAIL, Z. Effect of Mg, Si and Cu content on the microstructure of dilute 6000 series aluminium alloys. *Journal of Alloys and Compounds*. 2011. vol. 509, pp. 8632- 8640.

[9] ADAMCZYK-CIESLAK, B., MIZERA, J. and KURZYDLOWSKI, K. J. Microstructures in the 6060 aluminium alloy after various severe plastic deformation treatments. *Materials Characterization*. 2011. vol. 73, pp. 327-332.

[10] WANDERKA, N.,LAZAREV, N., CHANG, C.S.T. and BANHART, J. Analysis of clustering in Al-Mg-Si alloy by density spectrum analysis of atom probe data. *Ultramicroscopy*. 2011. vol. 111, pp. 701-705.

[11] BANHART, J., LIU, M., YONG, Y., LIANG, Z., CHANG, C.S.T., ELSAYED, M. and LAY, M.D.H. Study of ageing in Al-Mg-Si alloys by positron annihilation spectroscopy. *Physica B*. 2012. vol. 407, pp. 2689-2696.

[12] TEICHMANN, K., D. MARIOARA, C., J. ANDERSEN, J. and MARTHINSEN, K. TEM study of β′ precipitate interaction mechanisms with dislocations and β′ interfaces with the aluminium matrix in Al-Mg-Si alloys. *Materials Characterization*. 2013. vol. 75, pp. 1-7.

[13] ABID, T., BOUBERTAKH, A. and HAMAMDA, S. Effect of pre-aging and maturing on the precipitation hardening of an Al-Mg-Si alloy. *Journal of Alloys and Compounds*. 2010. vol. 490, pp. 166-169.

[14] ABID, T. Etude Calorimétrique et Dilatométrique d'un alliage Al-Mg-Si. *Magister Thesis.*Alegria : University of Constantine, 2007. p. 91.

[15] HENGCHENG, L., YUNA,W. and KE, D. Hardening response and precipitation behavior of Al-7%Si-0.3%Mg alloy in a pre-aging process. *Materials Science & Engineering A*. 2013. vol. 560, pp. 811-816.

[16] SHEN,Y., GARNIER, J., ALLAIS, L., CRÉPIN, J., CALDEMAISON, D., HERIPRE, E., HIVER, J.M.and FORGET, P. Etude de l'anisotropie d'endommagement de l'alliage d'aluminium 6061-T6. *Fédération française des matériaux*. *Submitted on 17 Mar2011.*<hal-00572454>. p. 12. Available from https://hal-mines-paristech.archives-ouvertes.fr/hal-00572454.

[17] HONG-YING, L., CUI-TING, Z., MAO-SHENG, H., JIAO-JIAO, L. AND XIAO-CHAO, L. Time−temperature−property curves for quench sensitivity of 6063 aluminum alloy. *Transaction of. Nonferrous Metals. Society of China*. 2013. vol. 23, pp. 38−45.

[18] BARLAS, B. Etude du comportement et de l'endommagement en fatigue d'alliages d'aluminium de fonderie. *Doctoral Thesis. France:* National School of Mines of Paris, 2004. p. 231.

[19] MESSAOUDI, S., ZIDANI, M., CHENAFI, M., MEGHAZI, A., MATHON, M. H. and BAUDIN, T. Monitoring the Evolution of the Properties of Aluminium AGS Alloy Wire during Drawing. In METAL2016:*25th International Conference on Metallurgy and Materials*. Ostrava: TANGER, 2016, pp. 327-332.