

SEM-EDAX INVESTIGATIONS ON ALUMINUM ALLOYS TYPE 2024 (AU4G1)

FLORINA VIOLETA ANGHELINA¹, ILEANA NICOLETA POPESCU¹,
CARMEN OTILIA RUSANESCU², DAN UNGUREANU¹

Manuscript received: 16.01.2019; Accepted paper: 22.05.2019;

Published online: 30.06.2019.

Abstract. In the selection of special alloys, account must be taken of their particular physico-mechanical properties such as breaking strength and plasticity much higher than the resistance of conventional alloys and last but not least relative corrosion resistance, especially if these alloys are used in the aeronautical industry. This paper presents the results of electron microscopy research on a sample taken from an aluminum 2024 (AU4G1) used for the aeronautical industry.

Keywords: aluminum 2024 (AU4G1), SEM-EDAX electron microscopy, germination and eutectoid growth.

1. INTRODUCTION

Particular advances in the technical field require a continuous improvement in the quality of metallurgical products and therefore an extension of the use of non-ferrous metals having certain properties superior to ferrous metal materials. The development of new alloys has mainly pursued the intensive exploitation of systems capable of undergoing solid transformations, so that today the largest number of marketed brands are registered in the ASTM 2000, 6000, 7000 groups, which group contain the structural hardening alloys [1-9]. In this paper are presented the results of the investigations carried out on a sample taken from a 2024 aluminum alloy batch (AU4G1) developed at Alro Slatina, destined for the aeronautical industry. The objective of the investigations is to assess the compliance of the alloy with the type specifications (2024) of the aviation rules [2-7, 10-12] in terms of chemical composition. Certain research data, such as heat treatment parameters, are the manufacturer's "know-how" and were not known. On the other hand, the aim of this paper is to optimize and implement atomic methods of structural and compositional analysis, and to prove their capacity. In this context, the data obtained is significant through the informations budget it provides to the manufacturer and the user on the efficacy and safety of the exploitation of the relevant alloys.

2. MATERIALS AND METHODS

Methods for investigating evidence may be either direct or indirect, by type of evidence and its quality. In direct methods, the sample is studied by inserting it directly into

¹ Valahia University of Targoviste, Faculty of Materials Engineering and Mechanics, 130004 Targoviste, Romania. E-mail: vianghelina@yahoo.com; pinicoleta24@yahoo.com.

² Politehnica University of Bucharest, Faculty of Biotechnical Systems Engineering, 060042 Bucharest, Romania. E-mail: rusanescuotilia@gmail.com.

the electronic microscope, and in the indirect methods one-step or two-step replicas are used. In the electronic scanning microscope, direct methods are generally used. The field of the field investigated by the electron beam is of the order of μm^2 or less. For this, a 16 mm diameter of the sample holder is considered sufficient. Type of material studied: duralumin for aviation 2024 (AU4G1). Condition of treatment: T 4 (specified by the manufacturer). In cases of dural samples were used metallographic processed samples.

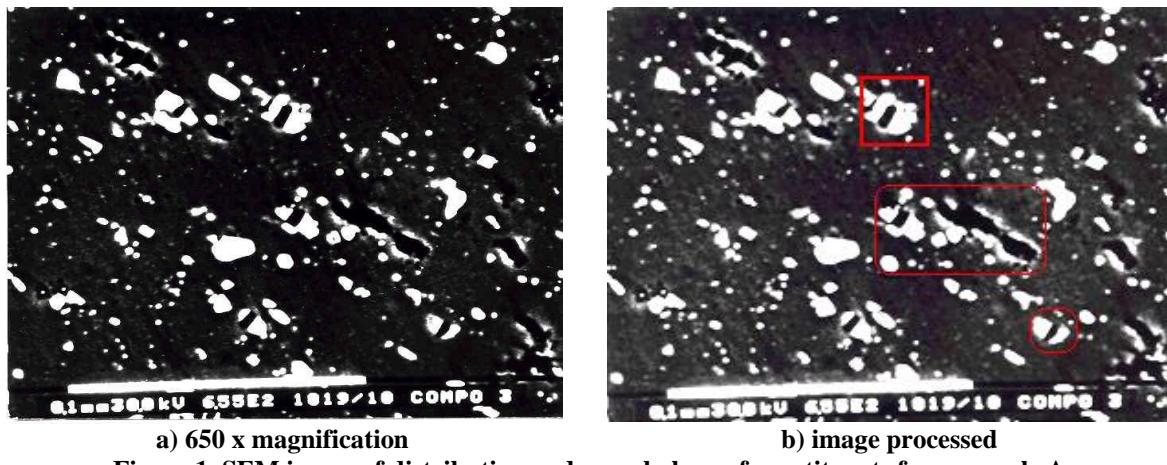


Figure 1. SEM image of distribution and morphology of constituents from sample A.

3. RESULTS AND DISCUSSION

The images (Fig. 1) show the compounds of a sample taken from a type (AU4G1) aluminum batch, which we will note with A. The image in Figure 1.a) is the original SEM image, and the image of Figure 1.b) is a processed version in which it is stressed that, by attack, chemical compounds can be evidenced relative to the matrix. Thus, in Figure 1.b), it is clearly evidenced the arrangement of the semi-embedded compounds in the matrix. It can be noticed that S and S' phases have irregular polygonal morphology and have a "black" color, making them hard to notice in SEM images. The Mg₂Si hardener compound is a dark, rounded and small-sized compound. The sample shows an agglomeration of Mg₂Si in the vicinity of the S phase, making it almost impossible to distinguish this compound from of contrast of the gray colour levels. Sample A contains a larger amount of Mg₂Si. Sample A presented a novel appearance of germination of a compound on the lateral surface of another compound.

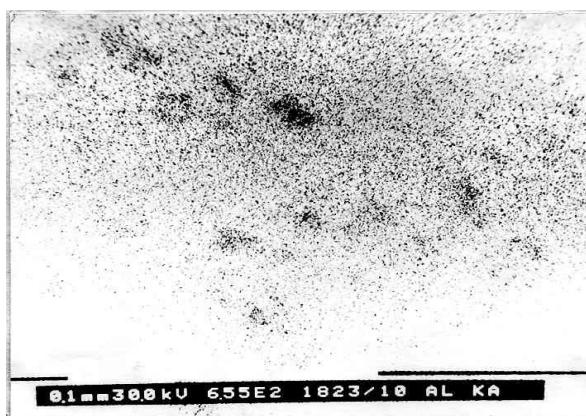


Figure 2. SEM detail images of the eutectic germination of constituents from sample A.

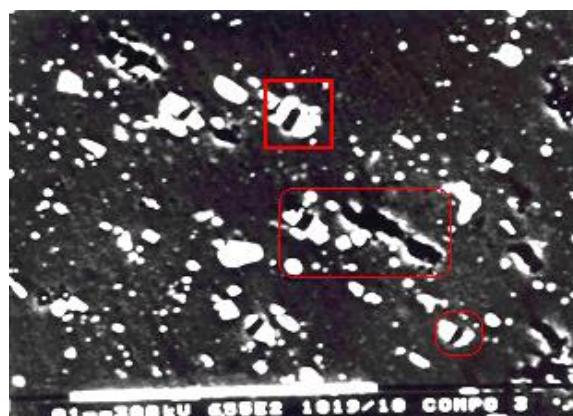
Thus, the compounds identified in Figure 1 and in the detailed images of Figure 2 exhibit particularities such as germination and eutectoid growth. In this regard, around a dark

colored parallelepiped compound, a „butterfly wing”-like compound grows and, together with the central compound, forms a distinct „butterfly” appearance. In the viewing area of Fig. 1 there are 7 "butterfly" formations and 5 "vermicular" formations that appear to be generated by the same growth mechanism, i.e "degenerate butterflies". The "butterflys wings" are $\text{Al}_7\text{Cu}_2\text{Fe}$ compounds, and the "butterflys body" are $\text{Al}_{23}\text{Cu}\text{Fe}_{24}$ compounds. Germination and eutectoid growth may be coherent if the two compounds have crystalline planar interfaces with close structural parameters.

Aluminum concentration distribution, associated with Fig. 1. The nature of the compounds described above can be determined on the basis of the elemental distribution to be presented subsequently. Thus, in Fig. 3.a) the results of the SEM-EDAX type analysis on Aluminum element distribution are presented at a 650x magnification. The size of the scanned area is $184 \times 135 \mu\text{m}^2$.



a) 650 x magnification

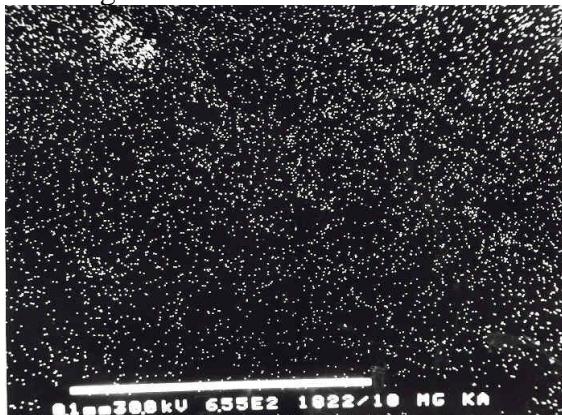


b) central detail at 200x magnification

Figure 3. Image of Al element distribution from sample A.

Aluminum distribution is relatively homogeneous in the matrix and non-homogeneous at the level of the compounds / phases. For an estimate of the variation of the superficial distribution of the aluminum concentration, that is to say, of the phases having different contents of aluminum, the SEM image of the analyzed surface is shown in Fig. 3.b). By comparing the two images, it is clear that the distribution of Aluminum is changed due to the compounds / phases. Thus, it can be appreciated that the aluminum has a lower concentration in the areas of the white compounds.

Magnesium concentration distribution, associated with Fig. 1. In Figure 4.a) the results of the SEM-EDAX type analysis of the Mg element distribution are presented at a 650x magnification.



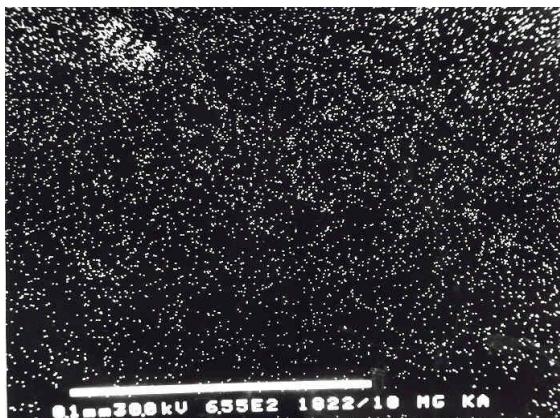
a) Mg distribution at 650x magnification



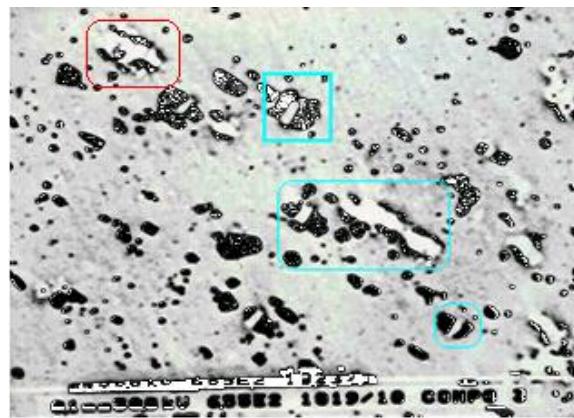
b) Phase distribution at 650x magnification

Figure 4. a) Image of Mg element distribution in sample A and b) associated SEM image

The size of the scanned area is $183 \times 135 \mu\text{m}^2$. The Mg concentration distribution is relatively homogeneous in both the matrix and the compounds. For the reasons given above, the analyzed SEM image is also presented in Figure 4.b). Analyzing the two images in Fig. 4 it can be stated that the Mg element has a relatively homogeneous concentration distribution in both the matrix and the compounds areas, with only one exception, in the upper left, of the image in Fig. 4.a) there is an agglomeration of Mg in the area of a "vermicular" formation of compounds Fig. 4.b). There are compounds with Mg, but more dispersed, that is, fewer. The magnesium agglomeration in compounds is much better outlined in (Fig. 5a, b) where the Magnesium distribution can be compared to the processed SEM image to better outline the morphology and distribution of the compounds.



a) Mg distribution at 650x magnification

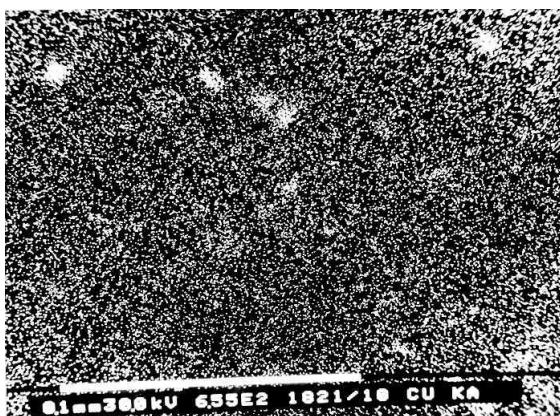


b) Phase distribution at 650x magnification

Figure 5.a) Image of the Mg element distribution in sample A and b) processed SEM image.

From the previous images it results that the Mg distribution is not significantly modified by the phases, which can be explained by the participation of Mg in the formation of all the compounds, but also by a "glow excitation" effect.

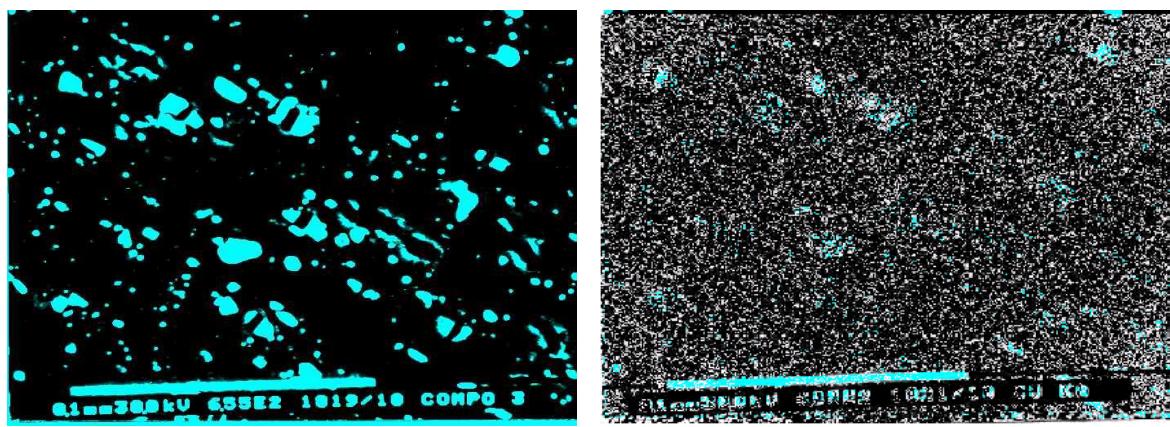
Copper concentration distribution, associated with Fig. 1. In Fig. 6.a) the results of the SEM-EDAX analysis on the copper element distribution are presented at a 650x magnification. The size of the scanned area is $184 \times 136 \mu\text{m}^2$. As the copper concentration distribution is non-homogeneous, a comparative image analysis of Fig. 6.a) was used with the associated SEM image and the processed SEM image (Fig. 6.c) to identify the phases which concentrating the copper element.



a) Cu distribution at 650x magnification



b) Phase distribution at 650x magnification



c) image b) processed

d) images a) and c) superimposed

Figure 6. a) Image of the Cu element distribution in sample A; b) the SEM image of the analyzed area; c) and b) the SEM image of the analyzed area processed; d) pictures a) and c) overlapped.

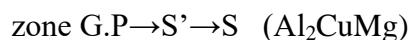
In Fig. 6.d) there is the distribution of the copper element superimposed over the image in Fig. 6.c). Fig. 6.d) clearly shows that "butterflies", more precisely "butterfly wings", contain more copper than the matrix but also more than the "butterfly body". This aspect explains the "butterfly" eutectoid growth mechanism. From the analysis (Fig. 6) it results that the Cu element has a significant concentration in the matrix and is predominantly agglomerated in the white phases.

4. CONCLUSIONS

Analyzes carried out on the aluminum batch have highlighted the following: the alloy fits in composition in type 2024 (AU4G1). Sample A confirms that the studied aluminum batch is in terms of the content of the compounds, according to the class of aluminum alloys (4.4% Cu, 1.5% Mg, 0.5 Mn) which usually identify Mg-Si, Al-Cu-Mg, Al-Cu and Al-Cu-Fe-Mg compounds. SEM-EDAX investigations show that the sample has significant compound contents. The compounds are inhomogeneously distributed on the surface of the sample forming aggregates of a constitutive and not mechanical nature. This gives rise to some hypotheses, such as: The chemical homogeneity of the batch is pronounced, starting from the elaboration; Putting in solution can not homogenize the chemical composition of the material.

As has been shown previously, in the germination and growth of certain compounds, there are coupled segregation phenomena of alloying elements whose "driving forces" are not known, but can only be anticipated as having a thermodynamic nature, that is, by subtracting the free energy of the material. At first sight, the increase of the compounds by segregation is somewhat contrary to the theory of diffusion, which implies another hypothesis, so the driving force of segregation is the opposite of the driving force of diffusion and is higher.

In the batch studied, there is a lack of Al_2Cu equilibrium compound, which is specified by the literature [8, 13-21] as the most important durifier. This can only be explained by the fact that, in the case of the investigated batch, the dominant precipitation reaction leads to the formation of a phase S (Al_2CuMg) and / or an S`-phase according to the scheme:



In order to elucidate the stoichiometry of the white compounds in the above mentioned figures, it is necessary to investigate their structure by electron diffraction on

microarrays and / or EDAX - quantitative precision analysis, what undoubtedly results from previous analyzes is that segregation mechanisms are coupled. From the point of view of metal physics and material science, the germination of compounds and coupled growth represent a new field of study, both theoretically and practically important, for controlling the properties of aluminum alloys for aviation .

REFERENCES

- [1] Zamarca, S., Vasiliu, F., Pencea, I., Studiul durificarii duralurilor, *Studii si cercetari de fizica*, **109**, 124, 1988.
- [2] ***** SAE AMS 4037N, *Aerospace materials specification*; NA 41.100, NA 41.101, NA 41.110, NA 41.118.
- [3] ***** EN 515, *Aluminium and aluminium alloys, Wrought products-Temper designation; Aluminum standards and data*, Aluminum Association, 2009.
- [4] ******Aluminium Standards and Data*, Aluminium Association, 2004.
- [5] ***** SR-EN 485/2/2004, *Aluminium and aluminium Alloys-Sheet strip and plate-Mechanical Properties*, Aluminium Asociation, 2004.
- [6] ***** *Metallic Materials Properties Development and Standardization* (MMPDS)\MMPDS – 03, Vol. 2a (Chapter 3, Part 1), 2006.
- [7] ***** SR-EN 515/1995, *Aluminiu si aliaje de aluminiu – Produse Deformabile. Simbolizarea starilor*, 1995.
- [8] ***** EN 573-3, *Aluminium and Aluminium Alloys Chemical composition and form of wrought products*, Part 3 Chemical composition, Aluminium Association, 2014.
- [9] Fara, I., *Aluminul de la materia primă la produse finite*, Ed. Tehnica, 2000.
- [10] Poinescu, A.A. et al., *Revista de Chimie*, **65**(10), 1245, 2014.
- [11] Poinescu, A. et al., *Journal of Science and Arts*, **1**(42), 275, 2018.
- [12] Lungu, J. et al., *Journal of Optoelectronics and Advanced Materials*, **12**(9), 1969, 2010.
- [13] Engdahl, T. et al., *Materials Science and Engineering*, **327**(1), 59, 2002.
- [14] Constantin A.M., *Bulletin of Transilvania University of Brașov, Series IX: Sciences of Human Kinetics*, **11**(60)-1, 39, 2018.
- [15] Constantin A.M., *Bulletin of Transilvania University of Brașov, Series IX: Sciences of Human Kinetics*, **11**(60)-2, 155, 2018.
- [16] Buha, J. e al., Positron Studies of Precipitation in 6061 Aluminium Alloy, *Proceedings of the 9th International Conference on Aluminium Alloys*, p. 1028-1033, 2004.
- [17] Suciu, M.V., Cercetări privind determinarea parametrilor optimi de tratament termomecanic în vederea îmbunătățirii caracteristicilor mecanice ale aliajelor Al-Zn-Mg-Cu, destinate industriei aerospatiale, *Buletinul U.P. Bucuresti – seria Metalurgie*, 1985.
- [18] Ionita, I. et al., *Journal of Optoelectronics and Advanced Materials*, **10**(11), 2859, 2008.
- [19] Setnescu, R. et al., *Revista Materiale Plastice*, **43**(1), 1, 2006.
- [20] Bucurica, I.A. et al., *Romanian Journal of Physics*, **64**(7-8), 2019.
- [21] Bucurica, I.A. et al., *Bulletin of Materials Science*, **41**, 71, 2018.