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MODIFICATION OF THE CAST STRUCTURE OF AN EN AW 2011 ALLOY WITH HOMOGENIZATION

MODIFIKACIJA LITE STRUKTURE ZLITINE EN AW 2011 S HOMOGENIZACIJO

Maja Vončina[1*], Irena Paulin[2], Ana Debevec[3], Aleš Nagode[1]

[1]University of Ljubljana, Faculty of Natural Sciences and Engineering, Department for Materials and Metallurgy, Aškerčeva 12, 1000 Ljubljana, Slovenia
[2]Institute of Metals and Technology, Lepi pot 11, 1000 Ljubljana, Slovenia
[3]Impol Group, Partizanska 38, 2310 Slovenska Bistrica, Slovenia

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Aluminium alloys of group 2xxx contain copper as the main alloying element. Copper increases the strength and workability of the alloy, but also reduces the corrosion resistance and weldability. During casting, a nonequilibrium solidification occurs. Therefore, the cast alloy needs to be heat treated with a so-called homogenization process. Homogenization allows us to eliminate crystalline segregations and low-melting eutectics, and also causes changes in the morphology of intermetallic phases. The forming ability is in this way increased. In this research the subject of the investigations was the aluminium alloy with designation EN AW 2011 (AlCuBiPb), whereas the comparative analysis before and after homogenization annealing was made. Homogenization was conducted at 520 °C for 6 h. First, slices from two rods before and after homogenization were cut out, where three samples from each slice of the rod, namely in the middle, on D/4 and at the edge of the slice were prepared. Differential scanning calorimetry (DSC) was performed on all six samples, the results were compared with each other in order to establish the structure homogeneity before and after the homogenization process through the cross-section of the rod. Samples for light (LM) and scanning electron microscopy (SEM) were also prepared, whereas the phase composition and chemical homogeneity were analysed. Using the Thermo-Calc program, the nonequilibrium solidification was simulated and the phase formation during solidification was examined. From the obtained results, it was concluded that the homogenization was carried out successfully, due to a homogeneous chemical distribution in the examined phases and to a fairly homogeneous chemical composition throughout the cross-section of the rod slice.

Keywords: aluminium alloy EN AW 2011, homogenization, thermodynamic equilibrium, homogeneous microstructure

Aluminijeve zlitine iz skupine 2xxx vsebujejo kot glavni legirni element baker, ki zlitini povečuje trdnost in obdelovalnost, vendar ob tem zmanjša odpornost proti koroziji in varivost. Pri litju drogov prihaja do neravnotežnega strjevanja, zato je potrebno ohlajeno zlitino toplotno obdelati s tako imenovanim procesom homogenizacije. Ta omogoči, da odpravimo kristalne izceje in nizko taljive evtektike, povzroči pa tudi spremembo morfologije intermetalnih faz. S tem se poveča zmožnost preoblikovanja zlitine. V raziskavi je bila analizirana aluminijeva zlitina z oznako EN AW 2011 (AlCuBiPb), pri čemer je bila narejena primerjalna analiza pred in po homogenizacijskem žarjenju. Homogenizacija je potekala 6 h pri 520 °C. Iz dveh drogov, pred in po homogenizaciji je bila izrezana rezina litega droga, pri čemer so bili vzorci odvzeti na sredini, na D/4 in na robu rezine. Na vseh šestih vzorcih je bila izvedena diferenčna vrstična kalorimetrija (DSC), katere rezultati so bili primerjani med seboj z namenom ugotavljanja homogenosti strukture pred in po homogenizacijskem žarjenju po preseku rezine droga. Prav tako so bili vzorci pripravljeni za svetlobno (LM) in vrstično elektronsko mikroskopiranje (SEM), kjer je bila analizirana fazna sestava in kemijska homogenost. S pomočjo programa Thermo-Calc je bil simuliran potek neravnotežnega strjevanja in tvorjenje faz med strjevanjem. Iz pridobljenih podatkov med eksperimentalnim delom je bilo ugotovljeno, da je bila homogenizacija zlitine EN AW 2011 izvedena uspešno, saj ima zlitina po njej dokaj homogeno porazdelitev kemijskih elementov v fazah in homogeno kemijsko sestavo po celotnem prerezu rezine droga.

Ključne besede: aluminijeva zlitina EN AW 2011, homogenizacijsko žarjenje, termodinamično ravnotežje, homogena mikrostruktura

1 INTRODUCTION

Aluminium alloys are easy to machine, but form long chips during drilling or turning. For this reason, the EN AW 2011 alloy was developed in 1934 due to the need for a high-strength alloy that would not form long chips during machining. This problem was solved by adding small proportions of metals or low-melting elements such as lead and bismuth, which have limited solubility in aluminium. In aluminium, they form phases that are soft and therefore suitable for alloys that are further processed, as these phases help the premature tearing of chips and allows better lubrication of the tool. The advantage of bismuth in the alloy is also that it expands during solidification and thus compensates for the shrinkage of lead during solidification.[1,2] The microstructure of the EN AW 2011 alloy consists of primary α-Al crystals, a needle-shaped $Al_9Fe_2Si_2$ eutectic phase occurring at the crystal grain boundaries, an Al_2Cu eutectic phase, and solid lead and bismuth roundish particles.[3–5]

The alloy EN AW 2011 can be heat treated according to the purpose of use in various ways, namely by heat treatment T3, T4 and T8.[6] **Table 1** shows the typical me-

*Corresponding author's e-mail:
maja.voncina@omm.ntf.uni-lj.si (Maja Vončina)

chanical properties of the alloy EN AW 2011 after heat treatment T3 and T8, which are the most commonly used.[7]

Due to the high casting speed of rods and ingots, solidification is rather uneven and nonequilibrium, resulting in material defects such as crystal segregation, formation of low-melting eutectics, unfavourable shape of intermetallic phases and inhomogeneous distribution of alloying elements across rods or ingots and throughout the microstructure. The inhomogeneity of the chemical composition and the solid solution adversely affect the strength, heat deformability and corrosion resistance. In addition, it can lead to the formation of undesirable phases due to segregation in the material.[8] Therefore, it is necessary to heat treat the fast-cooled alloy with so-called homogenization annealing, which not only allows us to eliminate crystalline segregations and low-melting eutectics, but also causes a change in the shape of the intermetallic phases and the formation of fine precipitates.[7] The cast rods must be homogenized at a temperature below the solidus temperature before further processing into semi-products. This reduces the effect of micro-segregation or inhomogeneity due to the uneven solidification. Also, low-melting eutectics that could cause damage during further processing, are removed. In this way, precipitation is also controlled.[9]

Homogenization is a diffusion-controlled process. Thus, the rate of homogenization is higher at higher temperatures, since the diffusion rate is higher. At lower temperatures, low-melting eutectics are eliminated, and at slightly higher temperatures, crystalline segregations are eliminated. With the process itself, the change in the shape of the intermetallic phase and the formation of fine precipitates can be influenced, thus influencing various properties, namely:[10–12]

- enables the further heat treatment of products,
- improves the mechanical properties of the material, as it eliminates segregations,
- improves surface treatment,
- improves the ability for hot and cold plastic forming,
- raises the recrystallization temperature,
- causes a fine-grained microstructure.

Homogenization enables the diffusion of alloying elements from the grain boundaries and other elements-rich areas towards the middle. The time required for diffusion depends on the diffusion distance, which depends on the grain size or dendritic spacing. This can be written with the equation $x = \sqrt{(D \cdot t)}$, where x represents the distance, t time, and D the diffusion coefficient depending on the temperature, which is most important in controlling the process. It must also be considered which alloy is being homogenized, as the right conditions, such as processing time, homogenization temperature, and heating rate to homogenization temperature need to be chosen. Slow heating triggers accelerated nucleation, which ensures fine and even growth of the microstructure. Homogenization is also necessary because it can alter the dispersion and primary intermetallic compounds formed during casting.[13]

In this investigation, the microstructure of the as-cast and homogenized alloy EN AW 2011 was analysed, whereas the efficiency of the homogenization process was evaluated.

2 EXPERIMENTAL PART

In the experimental part, the homogeneity of the chemical composition was analysed across the cross-section of the rod slice from alloy EN AW 2011 (European standard EN 573-3, **Table 2**) before and after the homogenization annealing, whereas the chemical composition was partially modified. In order to predict the phase formation in a certain temperature range (homogenization temperature), which significantly affects the mechanical and forming properties of the rod, the equilibrium amount of all the phases regarding the temperature of EN AW 2011 alloy was calculated. From the chemical composition obtained by ICP analysis (ICP analysis was performed by inductively coupled plasma optical emission spectrometry (ICP-OES)), calculations were performed using the computer program Thermo-Calc and the TCAL6 database.

According to the standard, lead was replaced by bismuth and silicon in this investigated alloy, as lead is a heavy and toxic metal that is abandoned. The suitability of the homogenization annealing process was assessed using various analyses. The chemical composition through the cross-section of the rod slice was analysed before and after homogenization annealing, which was performed at 520 °C and lasted for 6 hours.

Three samples were taken from each slice of the rod, i.e., from the middle of the slice, at D/4 and at the very

Table 1*:* Mechanical properties of aluminium alloy EN AW 2011 after various heat treatments[7]

Heat treatment	Tensile strength (MPa)	Yield strength (MPa)	Hardness (HB)	Maximum shear deformation (MPa)	Fatigue resistance (MPa)	Modulus of elasticity (GPa)
T3	380	295	95	220	125	70
T8	405	310	100	240	125	70

Table 2: Chemical composition of alloy EN AW 2011 in mass fractions, (w/%)[14]

Element	Cu	Bi	Fe	Pb	Si	Al
Concentration	5.0–6.0	0.20–0.60	up to 0.70	0.20–0.60	up to 0.40	Rest

edge of the slice. Samples for differential scanning calorimetry (DSC analysis on NETZSCH STA 449c Jupiter) were turned on 4.5-mm-diameter rollers and sawn to a height of 3 mm. The heating and cooling rate of the sample was set at 10 K / min in the device, and the sample was heated to a temperature of 720 °C. The entire test was performed in a protective atmosphere of Ar.

For microscopy, samples were moulded into cold epoxy mass and prepared metallographically according to the standard metallographic procedure for aluminium alloys. Polishing was performed based on MDNAP 300 mm, to which a diamond suspension was added. The final polishing was performed based on MDChem using an OPS suspension.

For the microstructural analysis, an Olympus BX61 light microscope was used to image the microstructure at various magnifications. Using scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS), phase analysis before and after homogenization annealing was performed using a Thermo Fisher Scientific Quattro S FEG-SEM microscope with an Oxford Ultim Max SDD EDS analyser. EDS mapping and EDS line analysis through the as-cast and homogenized microstructure components were made.

3 RESULTS AND DISCUSSION

Figure 1 illustrates the results of the thermodynamic calculations, whereas the amount of all the phases regarding the temperature is presented. The results indicate that the temperature range for the dissolution is 450–540 °C. As shown in **Figure 1**, the equilibrium phases and the temperature ranges in which they are

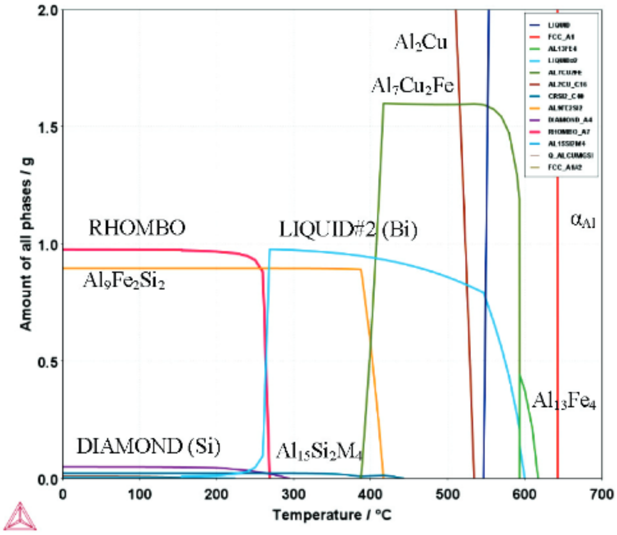

Figure 1: Thermo-Calc predicted phase stability in aluminium alloy EN AW 2011 regarding the temperature

present are: the $Al_{13}Fe_4$ phase up to 620 °C, the Al_7Cu_2Fe up to 590 °C, Bi up to 600 °C, Al_2Cu up to 530 °C, $Al_{15}Si_2Mn_4$ up to 440 °C and the $Al_9Fe_2Si_2$ phase up to 415 °C. This implies that by heating at 520 °C for a long holding time, $Al_{15}Si_2Mn_4$, $Al_9Fe_2Si_2$ will completely dissolve, whereas Al_2Cu and Al_7Cu_2Fe will only partially dissolve, but the iron phase $Al_{13}Fe_4$, as well as a fraction of the Al_7Cu_2Fe will still be present. It should also be noted that the homogenization treatment for these alloys should not be conducted at temperatures over 540 °C, as the alloy will start to melt.

Figure 2 shows a comparison of DSC curves with respect to the sampling site in the as-cast and homogenized

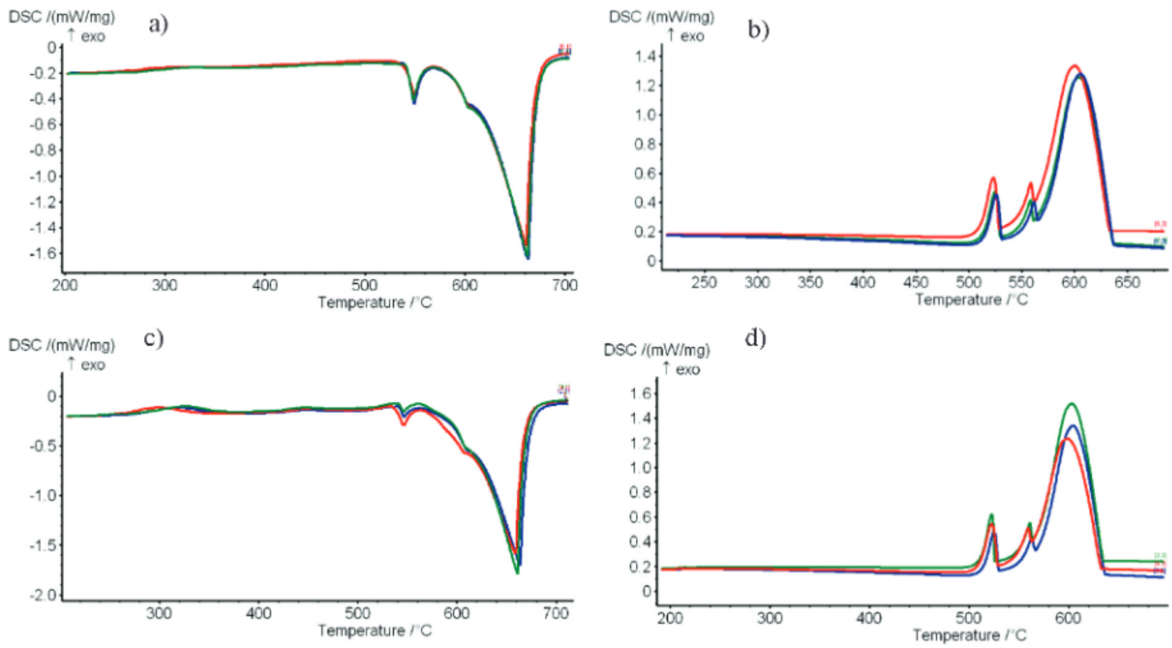

Figure 2: Comparison of: (a and c) heating and (b and d) cooling DSC curves of EN AW 2011 alloy taken from different places of the slice: centre (blue), D/4 (green) and edge (red); (a and b) for as-cast state and (c and d) homogenized state

state. From the comparison of heating (**Figure 2a**) and cooling (**Figure 2b**) DSC curves in the as-cast state, it can be noticed that the curves do not deviate noticeably from each other, which suggests that the chemical composition is quite homogeneous already in the as-cast state. The comparison of heating (**Figure 2c**) and cooling (**Figure 2d**) DSC curves after homogenization indicate that the melting and solidification of the alloy are very similar with respect to the sampling site, from which it can be concluded that the chemical composition after heat treatment is very homogeneous as a result of proper homogenisation.

Figures 3 and **4** show the microstructure of the EN AW 2011 alloy in the as-cast state (a, b and c) and after homogenization (d, e and f) at 50× and 200× magnification. Samples were taken from the middle, on D/4 and the edge, and are presented from left to right. In the figures it can be seen that the microstructure at the edge of

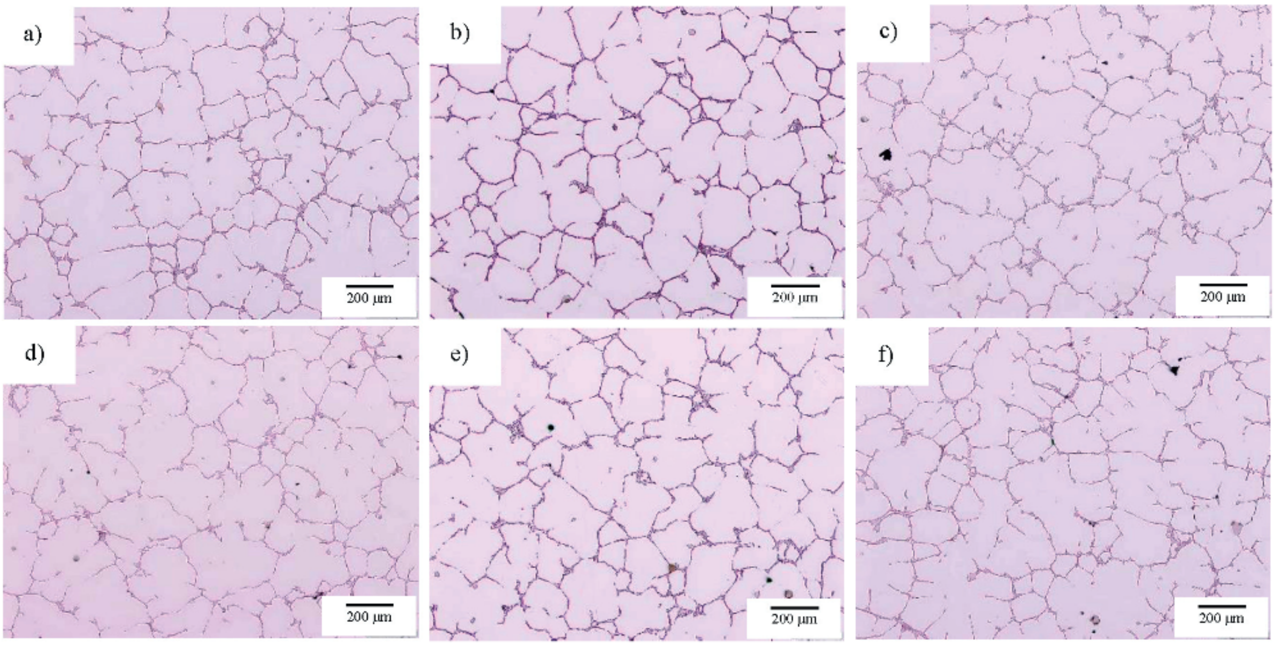

Figure 3: Microstructure of: (a, b and c) as-cast and (d, e and f) homogenized sample taken from: (a and d) the centre, (b and e D / 4) and (c and f) edge

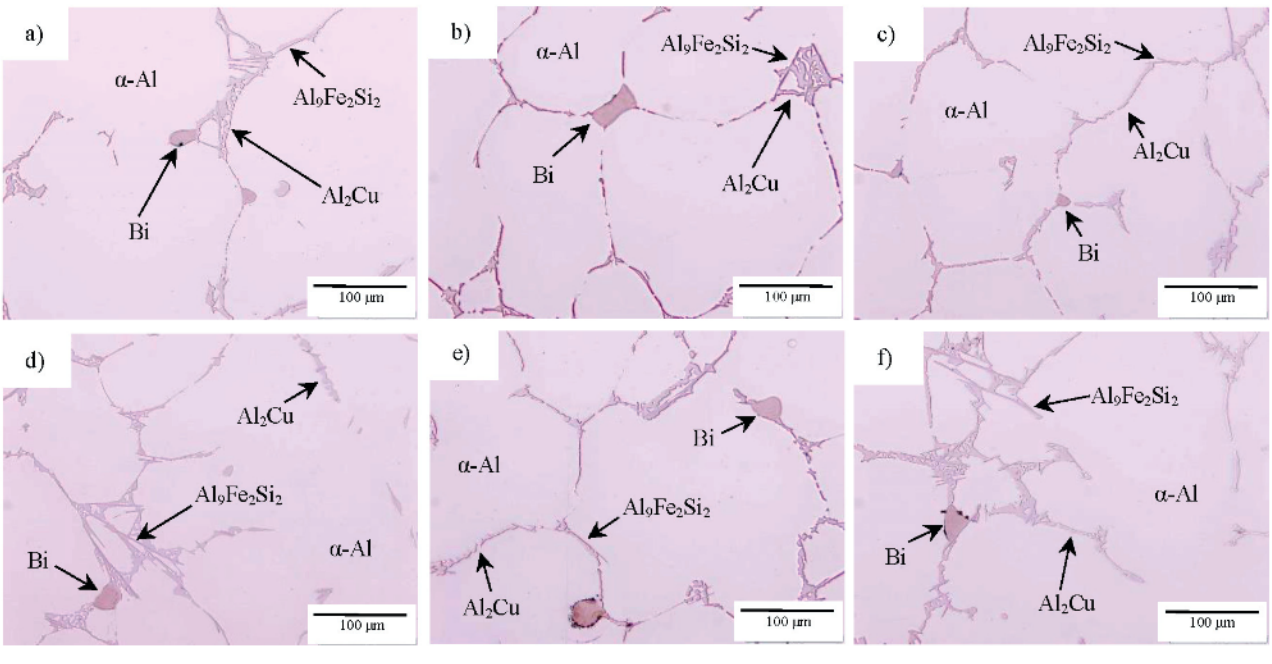

Figure 4: Microstructure of: (a, b and c) s-cast and (d, e and f) homogenized sample taken from: (a and d) the centre, (b and e) D / 4 and (c and f) edge with marked microstructural constituents

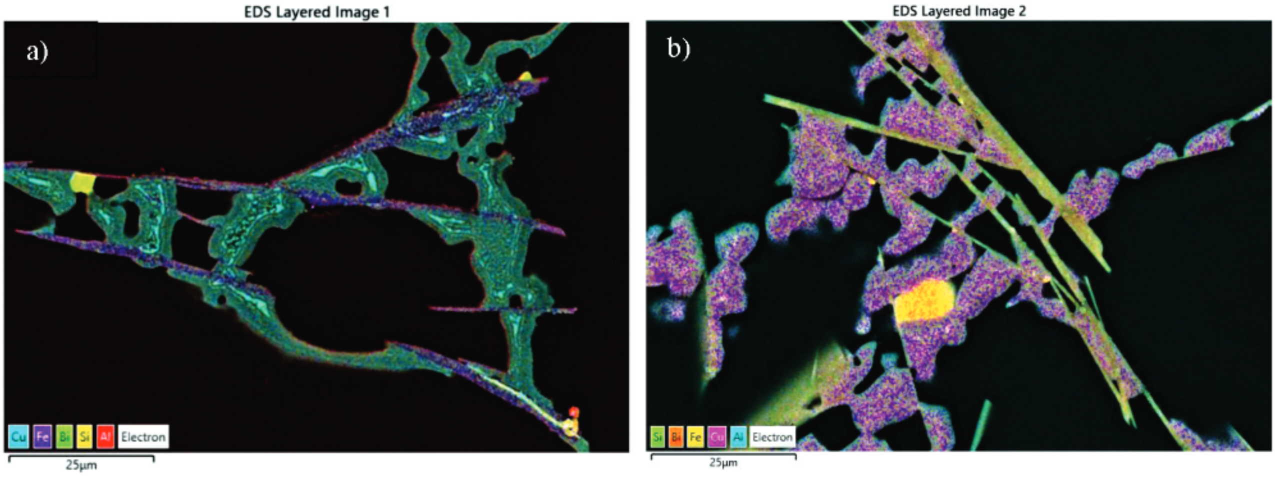

Figure 5: Mapping EDS analysis of: a) as-cast and b) homogenized sample from EN AW 2011 alloy taken at site D/4

the sample is coarser than in the middle. In the sample taken from the middle, smaller round shape Bi-particles than at the edge can be found (**Figure 4**). The reason for this is that the edge cools faster than the middle and consequently most of the Bi is precipitated/solidified already at the edge. Thus, the proportion of Bi is decreasing from the edge towards the middle. In homogenized samples, however, it is observed that the homogeneity of the microstructure (size and distribution of microstructural components) is uniform throughout the cross-section of the rod slice. Homogenization at a temperature of 520 °C for 6 h ensured a more uniform size and distribution of Bi-particles in the microstructure, and the eutectic Al_2Cu phase was more evenly distributed and more homogeneous (**Figure 3** and **4**). From this it can be concluded that homogenization was successfully carried out. Furthermore, if the as-cast and homogenized microstructure in **Figure 3** at a higher magnification is compared, it can be seen that in the as-cast state the eutectics form a continuous network, which is partially broken by homogenization. It can also be seen that less redish phases (Al_2Cu) are present in the microstructure in the as-cast state than in the homogenized one. This is due to the fact that Cu in the as-cast state was nonequilibrium solidified, which was solved by heat treatment where the Cu-phase was partially dissolved.

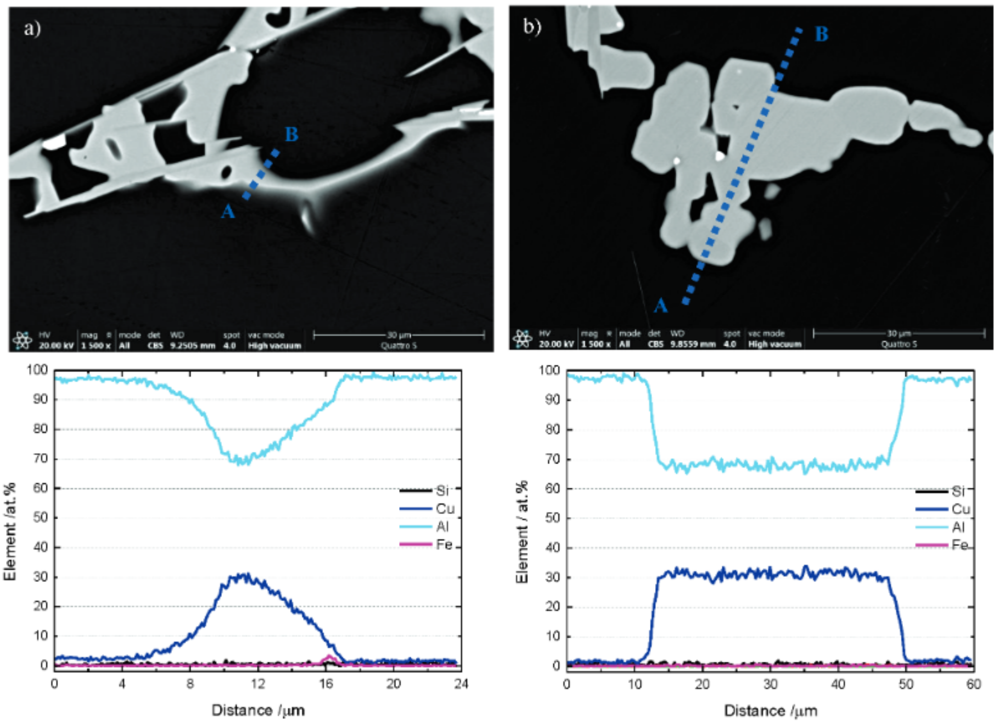

Figure 6*:* SEM micrographs and corresponding EDS line-scan analyses across the Al_2Cu eutectic phase in: a) as-cast sample and b) homogenized sample

Using a FEG-SEM microscope, the phases occurring in the microstructure of the samples in the as-cast and homogenized state were identified and confirmed. **Figure 5** shows an EDS mapping, where each chemical element in the microstructure is coloured with a different colour, which makes it easier to identify the distribution of the elements in the microstructural constituents. It is evident that in the as-cast sample the distribution of copper in the eutectic Al_2Cu phase is not uniform or homogeneous, with a noticeably higher copper concentration inside the phase, marked blue in **Figure 5a**. The homogenized sample shows the influence of the homogenization process on the dissolution and diffusion of copper, as it was evenly distributed throughout the eutectic Al_2Cu phase. This can be seen as very fine purple dots, evenly distributed where the Al_2Cu phase is present in **Figure 5b**. In addition, the Cu-phase in the homogenized sample is more fragmented and rounded than in the as-cast sample, due to the dissolution process and lowering of the surface energy.

In order to verify and emphesize the influence of homogenization annealing on the microstructure homogenity, an EDS line analysis through the eutectic Al_2Cu phase is presented in **Figure 6**. The analysis was performed on the straight line running across the eutectic phase A-B, as shown in the SEM micrographs in **Figure 6**. The distribution of the alloying elements present in atomic % is shown. According to the EDS line-scan profile in **Figure 6a**, the concentration of copper is increasing, and aluminium is decreasing towards the centre of the phase and decreasing and increasing towards the edge of the phase, respectively. In the centre of the Al_2Cu phase the concentration of copper is the highest and of aluminium is the lowest. On the other hand, in the homogenized sample in **Figure 6b,** the concentration of copper and aluminium does not change, and it is constant throughout the cross-section of the Al_2Cu phase.

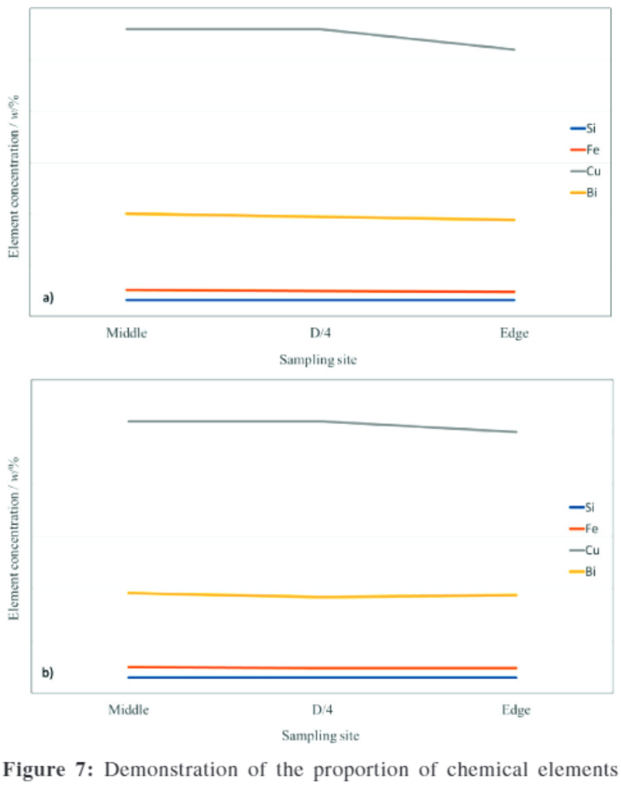

Figure 7: Demonstration of the proportion of chemical elements through the cross-section of the rod slice in: a) as-cast state and b) after homogenization

Figure 7a shows a graphical change in the mass fraction of the analyzed chemical elements in the EN AW 2011 alloy depending on the sampling site for the as-cast state. Through the cross-section from the centre to the edge, the proportions of copper and bismuth locally changes, which is the result of rapid nonequilibrium solidification during the casting of rods. As with the as-cast sample, the homogenized sample (**Figure 7b**) shows that the proportion of copper locally slightly decreases from the middle to the edge, but not to the same extent as with the as-cast sample. This suggests that the homogenization achieved a more homogeneous local distribution of chemical elements through the cross-section of the rod slice. The remaining three elements shown in the diagram, however, have almost complete chemical homogeneity through the cross-sectional after heat treatment.

4 CONCLUSIONS

The purpose of the research was to analyse the homogeneity of the chemical composition and modification of the as-cast microstructure from the alloy EN AW 2011 with homogenization annealing. With the help of literature data and research results the following can be concluded:

- The course of the DSC curves indicates that in the as-cast state the alloy is not completely homogeneous through the cross-section of the slice of the rod, and that the homogenization of the alloy EN AW 2011 was successful.
- According to the microstructure analysis, the following phases were identified: α-Al, Al_2Cu, $Al_9Fe_2Si_2$ and Bi. The microstructure was changed after homogenization, as eutectics changed shape and became more spherical and more evenly distributed. This change is shown with the distribution of elements, especially copper, being more uniform and homogeneous in the eutectic Al_2Cu phase after the homogenization.
- Chemical analysis of samples before and after homogenization showed that, despite the relatively homogeneous chemical composition of the rod through the cross-section, heat treatment is necessary and, in this case, appropriate, as it ensures a more homogeneous local distribution of elements in the microstructure and along the entire cross-section of the rod slice.
- With the selected homogenization annealing, a suitably homogeneous composition was achieved over

the entire cross-section of the rod slice, thus ensuring uniform and improved mechanical and forming properties over the entire cross-section of the EN AW 2011 alloy rod slice.

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