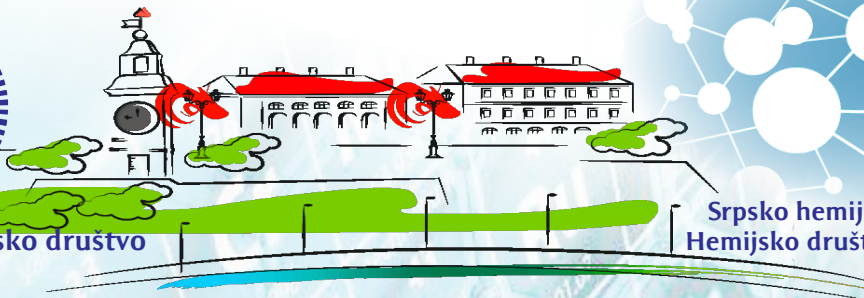




Srpsko hemijsko društvo



Srpsko hemijsko društvo  
Hemijsko društvo Vojvodine

# 55. savetovanje Srpskog hemijskog društva

# KNJIGA RADOVA

55<sup>th</sup> Meeting of  
the Serbian Chemical Society

# PROCEEDINGS

Novi Sad 8. i 9. juni 2018.  
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## Homogenization effect on microstructure Al-Mg-Si alloy containing low-melting point elements

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### Introduction

Extrudability of 6xxx series aluminium alloys (Al-Mg-Si-X) depends to the great extent on the microstructure of the homogenized billets [1]. In addition to the typical inhomogeneities such as elemental micro- and macro- segregations, the microstructure of as-cast state of Al-Mg-Si-X is characterized by the presence of iron-based intermetallic that can be detrimental for the deformability of these alloys. There are two types of Fe bearing intermetallics; the metastable,  $\beta$ -AlFeSi phase with monoclinic crystal structure and plate or needle-like morphology is impairing extrudability of the as-cast bullet by acting like stress concentrator and inducing local cracking and surface defects in the extrusions (2-4). One of the goals of the homogenization treatment is transformation of metastable  $\beta$ -AlFeSi into stable  $\alpha$ -AlFe(Mn)Si, with a cubic crystal structure and benevolent, globular morphology. The temperature range for  $\beta$ -AlFeSi  $\rightarrow$   $\alpha$ -AlFe(Mn)Si transformation is quite broad, temperature should be above 450 °C in order to achieve appreciable transformation kinetic caused by the low diffusivity of Fe and Mn, so the selection of the homogenization processing parameters depends very much on the Mg and Si content in the alloy [2,4]. The general rule is that the homogenization temperature should be above  $\beta$ -Mg<sub>2</sub>Si solvus in order to achieve an alloy's aging potential and desired mechanical properties, and below the solidus temperature of the pseudobinary Al-Mg<sub>2</sub>Si system for the given alloy composition. In the alloys with high Mg and Si content, like AA 6082 and its derivative AW6026, that is subject of this study, the homogenization temperature should be in a range 540°-580 °C [1]. However, the composition of AW6026 alloy is specific as it contains heavy metals Pb and Bi that are added in order to improve machinability of the alloy. These low melting point elements ( $T_m$ (Pb)=327 °C,  $T_m$ (Bi)=271.4 °C) are essentially insoluble in Al and have a tendency to segregate towards grain boundaries and free surfaces. There are reports [5] that at temperatures above 500 °C excessive migration of Pb and Bi toward surfaces takes place resulting in depletion of the low melting point phases required for good machinability. That would set homogenization temperature in significantly lower range than required for dissolution of Mg<sub>2</sub>Si. In addition, there are reports that wetting transition of grain boundaries by Pb occurs at temperatures 520-560 °C, depending on grain boundary type, increasing the propensity toward liquid metal embrittlement.

This study is aimed at evaluating the effect of homogenization parameters on completeness of  $\beta$ -AlFeSi  $\rightarrow$   $\alpha$ -AlFe(Mn)Si transition, Mg<sub>2</sub>Si dissolution and extent of loss of Pb and Bi in order to determine optimal homogenization parameters.

### Experimental procedure

The chemical composition of the studied AW6026 alloy, industrially cast in NISAL-Niš/Serbia, is presented in Table 1.



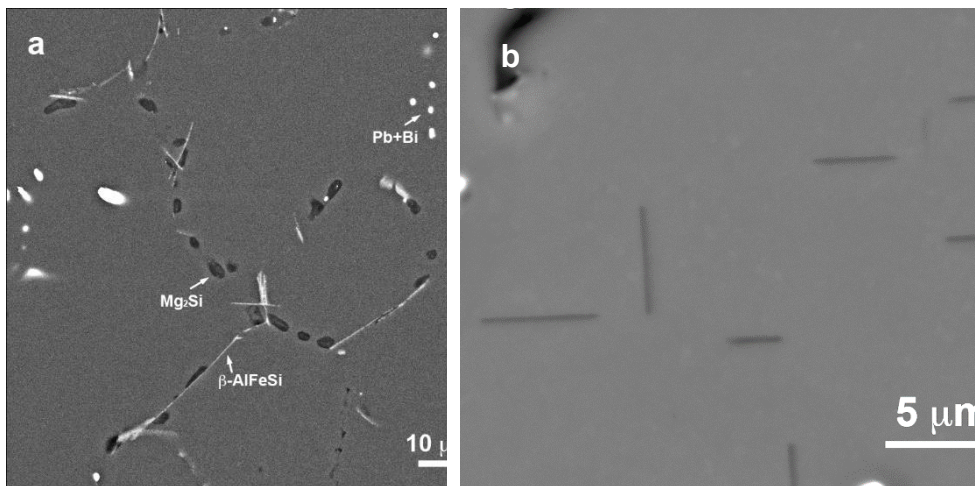
**Table 1** Chemical composition of AW6026 alloy

Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn
1.125	0.097	0.294	0.487	1.033	0.123	0.004	<0.002
Ti	Pb	Sn	Bi	V	Zr	Co	
0.009	0.252	0.006	0.697	0.010	<0.001	<0.002	

The specimens from the section of the as-cast bullet underwent homogenization treatments at a range of temperatures: 12h/480 °C, 12h/530 °C, 12h/550 °C and 6h/550 °C. The microstructural characterization of as-cast state and thermally treated specimens, previously mechanically polished by a standard procedure, was conducted in FEG SEM Tacan Mira and JEOL SEM equipped with EDS detector at 20 kV. For quantitative microstructural analysis, ImageJ software package was used. For evaluation of the effect of homogenization treatment on the grain size, specimens were electrolytically etched in Barkers reagent and characterized by optical microscopy in polarized light. In order to obtain representative statistics, the microstructural characterization and quantification were conducted on the area over 3x3 mm<sup>2</sup>.

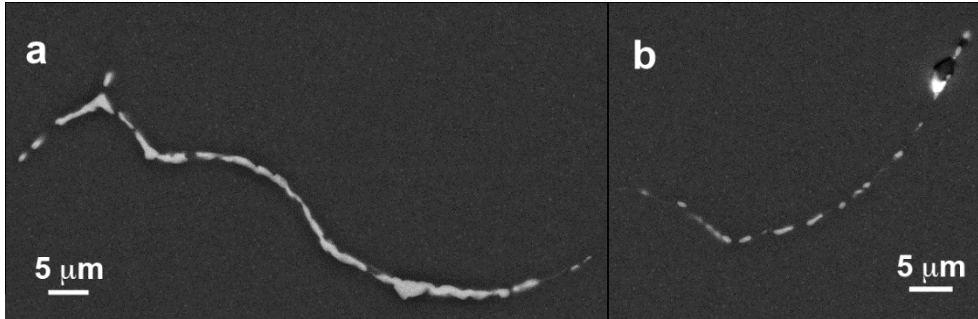
**Results and discussion**

Microstructure characterization showed that homogenization 12 h/480 °C has not resulted in  $\beta$ -AlFeSi  $\rightarrow$   $\alpha$ -AlFe(Mn)Si transformation to the significant degree. The slow diffusion of Fe at 480 °C is responsible for preservall of needle-like morphology (Figure 1a), since it controls the transformation kinetics. However, homogenization at 480 °C not only that it did not dissolve existing  $\beta$ -Mg<sub>2</sub>Si (Figure 1a), but resulted in additional  $\beta$ -Mg<sub>2</sub>Si precipitation since for the Mg and Si content in the studied alloy,  $\beta$ -Mg<sub>2</sub>Si solvus is around 540 °C. Although preferential sites for  $\beta$ -Mg<sub>2</sub>Si precipitation are interfaces of intermetallic phases and Al matrix, a fine plate  $\beta$ -Mg<sub>2</sub>Si precipitation that has crystallographic orientation relationship with Al matrix was observed to form (Figure 1b).



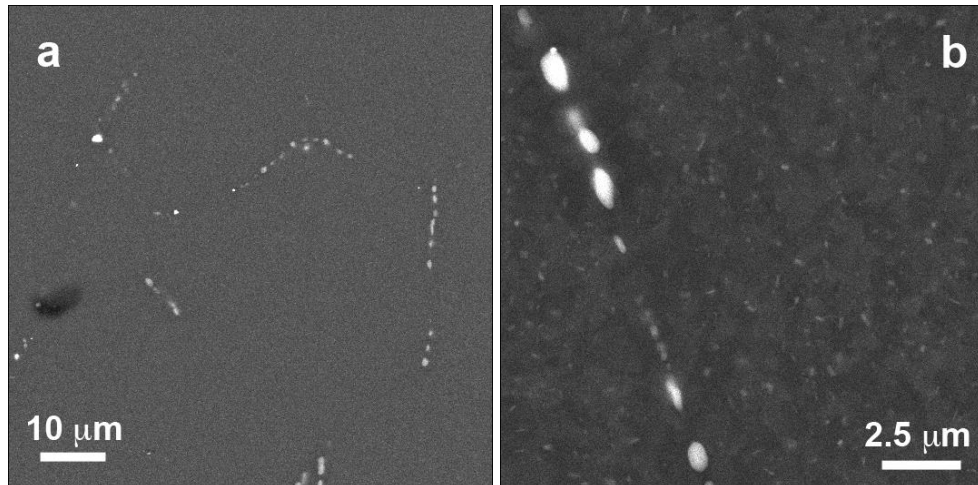
**Figure 1.** BE SEM micrographs of the microstructure after homogenization 12 h/480 °C:(a) Light, long needle-like particles are  $\beta$ -AlFeSi phase. Coarse particles in dark contrast are  $\beta$ -Mg<sub>2</sub>Si, that coagulate during homogenization; (b) Additional  $\beta$ -Mg<sub>2</sub>Si precipitated during the homogenization.

Homogenization at 530 °C resulted in partial  $\beta$ -AlFeSi  $\rightarrow$   $\alpha$ -AlFe(Mn)Si transformation (Figure 2). After 12 h of annealing, about 50-70 % of  $\beta$ -AlFeSi has been transformed, indicating that a longer thermal treatment is required to achieve desired transformation over 80 %.



**Figure 2.** BE SEM micrographs of the microstructure after homogenization 12 h/480 °C: (a) Continuity of  $\beta$ -AlFeSi is becoming interrupted indicating the initiation of  $\beta$  -  $\alpha$  transformation; (b) Particles of pearl-like morphology along the contour of original  $\beta$ -phase signalize complete transformation.

The increase in the homogenization temperature for 20 °C, to 550 °C, resulted in the much faster kinetics of the transformation. After 6 h of annealing, the transformation is almost complete ( $\geq 90\%$ ) (Figure 3a). Extending homogenization time to 12 h does not significantly change the microstructure with respect to the  $\beta$ -AlFeSi  $\rightarrow$   $\alpha$ -AlFe(Mn)Si transformation. One of the products of high temperature homogenization ( $T_{\text{homog.}} > 500$  °C) is precipitation of fine Al-Mn-Fe-Si dispersoids (Figure 3b).



**Figure 3.** BE SEM micrographs of the microstructure after homogenization 6 h/550 °C: (a) Pearl like morphology dominates the microstructure indicating complete  $\beta$  -  $\alpha$  transformation; (b) Fine dispersoids precipitate.

Coagulation of  $\beta$ -Mg<sub>2</sub>Si was already observed during homogenization at 480 °C (Figure 1a). However, the process of dissolution of  $\beta$ -Mg<sub>2</sub>Si only starts at higher temperatures. As it has

been expected degree of dissolution is higher at 550 °C than at 530 °C, since solvus lies between two temperatures. Longer homogenization time (12 h/550 °C) or even higher temperature (560 °C) might result in more Mg<sub>2</sub>Si dissolution, but the effect is not significant, particularly because an increase in exposure of the alloy to very high temperatures might give rise to a higher loss of heavy elements, Pb and Bi.

**Table 2.** Stereological parameters of heavy element particles (Pb and Bi)

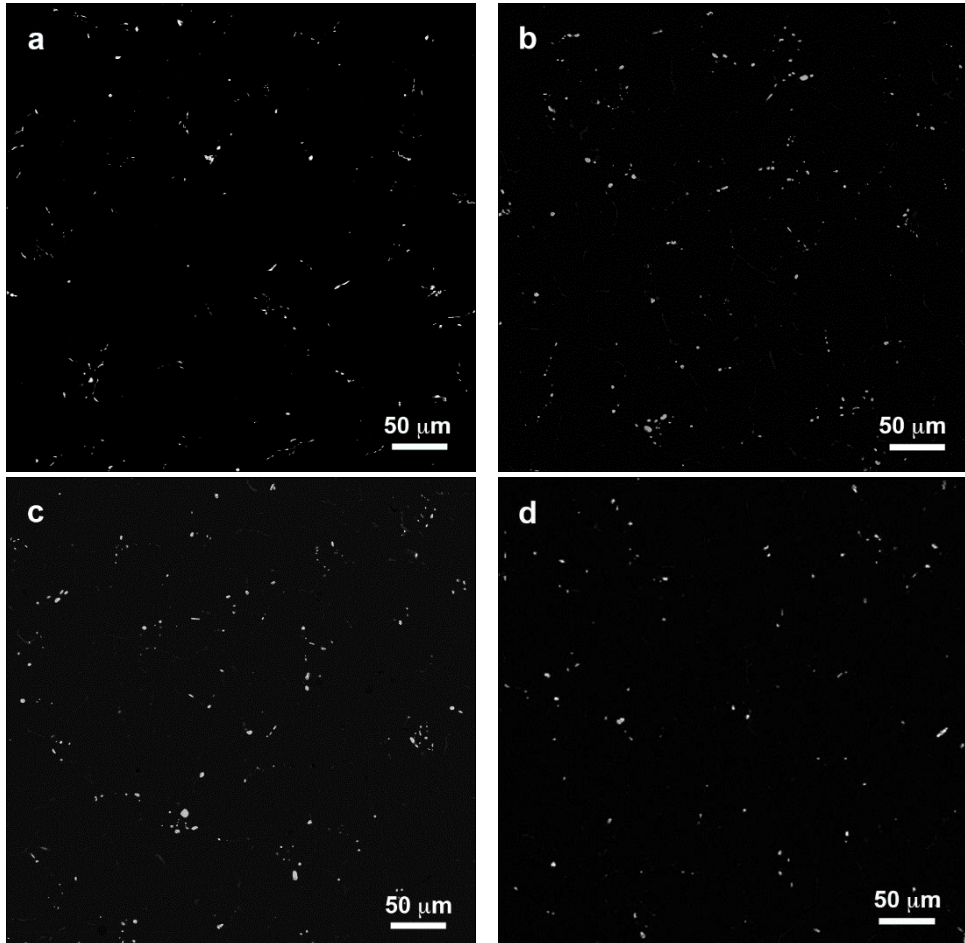
State	Area fraction, %	<particle size>, μm	roundness
12 h/480 °C	0.53	1.37	0.47
12 h/530 °C	0.48	1.21	0.57
6 h/550 °C	0.45	1.55	0.59

One of the critical issues with the homogenization at high temperatures is coarsening and escape to the surface of Pb and Bi particles. However, measurements of the area fraction of heavy element particles in as-cast state and after homogenization show a small drop (Table II). Similarly, the mean particle size is almost unchanged, but the increase in the roundness of the particles indicate some degree of shape change and coagulation. The absence of the significant change in fraction and distribution of heavy metal particles is evident in Figure 4.

Characterization of regions close to the edges of the specimens did not reveal depleted regions, while chemical analysis of homogenized specimens showed that Pb and Bi content is in the domain of statistical variation in the as-cast state. Lack of loss of the heavy elements is surprising since even Pb and Bi particles in Al matrix are expected to have high mobility at temperatures above 510-520 °C. This temperature range corresponds to the roughening transition of Al/Pb {111} interfaces. Namely, motion Bi and Pb particles in Al matrix, due to their lack of solubility in Al, does not occur by conventional vacancy mechanism but is controlled by Al self-diffusion and edge nucleation at the interface [6,7]. At roughening transition energetic barrier for edge nucleation becomes zero and particle migration becomes controlled by Al self-diffusion. Likely cause of small Pb and Bi loss is reaction with Mg. Bi has a strong affinity toward Mg to forming high-melting point compound and its function in the alloy is to act as a «getter» for Mg to keep Pb as low-melting elementary particles. EDS analysis showed that Bi particles contain Mg in the great amount. However, in the case of Pb, Mg content is a low indicating the formation of elementary core/shell structure elementary Pb/PbMg. So the formation of such shell can limit the mobility of Mg particles.

From a grain size measurements appears that the different homogenization treatments do not have a significant effect on grain size. Grain growth is limited during the homogenization due to the Zener pinning by dispersoids and intermetallic particles. Interestingly, it appears that Pb and Bi particles take part in Zener pinning as the regions with a lower content of Pb and Bi are characterized by the larger grains.





**Figure 4.** BE SEM micrographs of heavy metal particles (bright dots). Large difference in contrast is due to the difference in atomic number and scattering power. (a) as-cast state; (b) 12 h/480 °C; (c) 12 h/530 °C; (d) 6 h/550 °C.

### Summary

Low-temperature homogenization ( $T_{\text{homog.}} \leq 500$  °C) requires very long annealing times for  $\beta\text{-AlFeSi} \rightarrow \alpha\text{-AlFe(Mn)Si}$  transformation to take place. However, due to the high Mg and Si content in AW 6026 alloys, homogenization at 480 °C results in undesired precipitation of  $\beta\text{-Mg}_2\text{Si}$  particles.

Increase in homogenization temperature results in faster  $\beta\text{-AlFeSi} \rightarrow \alpha\text{-AlFe(Mn)Si}$  transformation kinetics, with annealing at 550 °C cutting homogenization time in half. Dissolution and coagulation of the existing  $\beta\text{-Mg}_2\text{Si}$  take place increasing the alloy aging potential in further processing. Importantly, high-temperature homogenization did not result in Pb and Bi loss, most likely due to the formation of compounds and core/shell structure around the particles of low-melting point compounds.

**Acknowledgement:** This work has been supported by Ministry of Education, Science and Technological Development of the Republic of Serbia, contract No. E!9992.

## Effekat homogenizacije na mikrostrukturu Al-Mg-Si legure koja sadrži nisko-topive elemente

*Predmet ovog rada je bilo ispitivanje i karakterizacija uslova homogenizacionog žarenja na mikrostrukturu Al-Mg-Si legure legirane nisko-topivim metalima Pb i Bi. Rezultati pokazuju da homogenizacija na temperaturama nižim od 500 °C zahteva izuzetno duga vremena žarenja da bi došlo do  $\beta$ -AlFeSi  $\rightarrow$   $\alpha$ -AlFe(Mn)Si transformacije, neophodne za sposobnost legure za presovanje. Dodatno, dolazi do izdvajanja  $\beta$ -Mg<sub>2</sub>Si faze, čime se smanjuje sposobnost legure za starenje u daljoj preradi. Visoko-temperaturna homogenizacija ne samo da rezultuje u potpunoj  $\beta$ -AlFeSi  $\rightarrow$   $\alpha$ -AlFe(Mn)Si transformaciji i rastvaranju  $\beta$ -Mg<sub>2</sub>Si faze, već i ne dolazi do očekivanog gubitka Pb i Bi usled izdvajanja na površini uzoraka.*

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