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# Analysis of intermetallic particles in AlSi1MgMn aluminium alloy

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**Materials**

## ABSTRACT

**Purpose:** The main objective of this study was to analyze the morphology and composition of complex microstructure of the intermetallic phases in AlSi1Mg alloy.

**Design/methodology/approach:** In this study, several methods were used such as: optical light microscopy (LM), transmission (TEM) and scanning (SEM) electron microscopy in combination with X-ray analysis (EDS) using polished sample, and X-ray diffraction (XRD) to identify intermetallics in AlSi1MgMn aluminium alloy.

**Findings:** The results show that the as-cast microstructure of AlSi1MgMn alloy after slow solidification at a cooling rate 2˚C/min, consisted a wide range of intermetallics phases. By using various instruments (LM, SEM, XRD) and techniques (imagine, EDS) the: $\alpha$-$A_{15}FeSi$, $\alpha$-$Al_{15}(FeMn)_3Si$, $A_{19}Mn_3Si$, $\alpha$-$Al_{12}Fe_3Si$, $Mg_2Si$ intermetallic phases were identified.

**Research limitations/implications:** In order to complete and confirm obtained results it is recommended to perform further analysis of the 6000 series aluminium alloys. Therefore it is planned to include in a next study, microstructure analysis of the alloy by using transmission electron microscopy technique (TEM).

**Practical implications:** Since the morphology, crystallography and chemical composition affect the intermetallic properties, what involves changes of alloy properties, from a practical point of view it is important to understand their formation conditions in order to control final constituents of the alloy microstructure. This paper has provided essential data about various intermetallic phases precipitating in 6000 series aluminium alloys.

**Originality/value:** This paper proposes the best experimental techniques for analysis of the intermetallic phases occurring in the 6xxx series aluminium alloys, provide wide description of the intermetallics formation through all possible reactions and data about their chemical composition, morphology and crystallography.

**Keywords:** Metallic alloys; Microstructure; Electron microscopy; Intermetallic phases

## 1. Introduction

The 6xxx-group contains magnesium and silicon as major addition elements. These multiphase alloys belong to the group of commercial aluminum alloys, in which relative volume, chemical composition and morphology of structural constituents exert significant influence on their useful properties [1-5].

In the commercial 6xxx aluminium alloys a wide range of intermetallic particles formed during solidification - in the interdendric regions, homogenisation and termomechanical processing [6]. In this aluminium alloys besides the intentional additions, transition metals such as Fe, Mn and Cr are always present. Even not large amount of these impurities causes the formation of a new phase component. The exact composition of the alloy and the casting condition will directly influence the selection and volume fraction of intermetallic phases [4].

Fe- bearing constituent phases typically found in 6xxx series alloys include of $\beta$-$Al_9Fe_2Si_2$ (also called $\beta$-$Al_5FeSi$) or $\alpha$ -$Al_{12}Fe_3Si$. However Mn and Cr can substitute for Fe, and stabilize the formation of $\alpha$ -$Al_{12}(FeMn)_3Si$ or $\alpha$ -$Al_{12}(FeCr)_3Si$ [3,7-8].

## 2. Material and experimental

The investigation has been carried out on the commercial AlSi1MgMn aluminum alloy. The chemical composition of the alloy is: 1.2%Si, 0.78%Mg, 0.5%Mn, 0.33%Fe, 0.14%Cr, 0.08%Cu, 0.05%Zn, Al bal. The alloy was melted in an electrical resistance furnace and cast in an iron mould. The specimens were remelted at 750$^o$C in ceramic crucible in air furnace. After being kept for 10 min at the temperature for melting the material entirely homogenizing the composition. The moltens were than cooled in the furnance at the cooling rate of 2$^o$C/min.

The microstructure of examined alloy was observed using an optical microscope - Nikon 300 on polished sections etched in Keller solution (0,5 % HF in 50ml $H_2O$). Morphology of specimens was made in the scanning electron microscope HITACHI S-3400 (SEM). Chemical composition of the intermetallics was made by EDS attached to the SEM using the software of Thermo Noran. Quantitative analysis of the microstructure components of AlSi1MgMn alloy was performed by X-ray diffraction (XRD, Thermo ARL diffractometer).

## 3. Results and discussion

Fig. 1 presents as-cast microstructures of AlSi1MgMn alloy. These images show fife types of intermetallic compounds located at the grain boundaries. These intermetallic particles had different morphologies, such as platelet, rod, polyhydron or “Chinese script”.

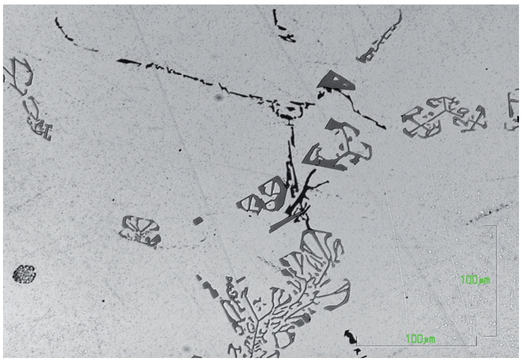



Fig. 1. As-cast microstructure of AlSi1MgMn alloy

Since it is rather difficult to produce detailed identification of intermetallic using only one method (e.g. microscopic examination) therefore XRD technique was utilized to provide confidence in the results of phase classification based on metallographic study. The results of this investigation are showed in Fig. 2. X-ray diffraction analysis of AlSi1MgMn alloy confirmed metalograffic observation. By combination of the XRD results and LM observation (morphology and different colours after eatching of Keller solution), these intermetallic phases were identified as a: β-$Al_5FeSi$ (dark phase), α-$Al_{15}(FeMn)_3Si$ (grey phase), $Al_9Mn_3Si$, α-$Al_{12}Fe_3Si$, $Mg_2Si$ (black phase) (Fig. 1).

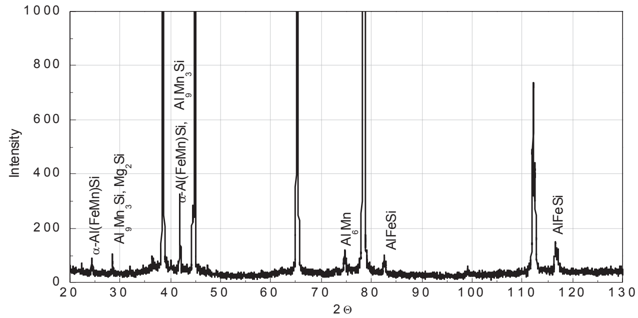
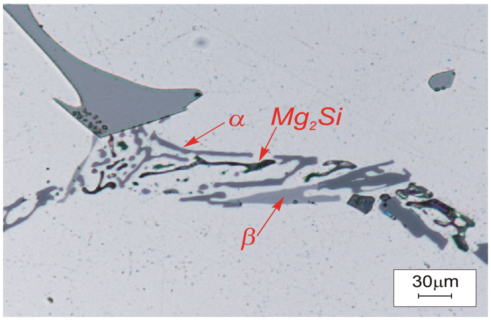



Fig. 2. XRD pattern of AlSi1MgMn alloy at the as-cast state

a)




b)

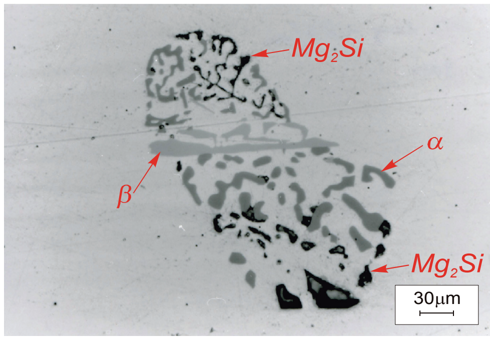



Fig. 3. As-cast microstructure of AlSi1MgMn alloy: a) the ternary eutectic, b) the quaternary eutectic

The alloy in as-cast state consists some complex eutectic structures as well (Fig. 3). Moreover the spherical in shape eutectic particles were also observed (Fig. 3b). It was found that three phases, namely: β-$Al_5FeSi$, $Mg_2Si$ and α-Al(FeMn)Si and Si are present in the α-Al matrix. These intermetallics grew together into a eutectic cluster (Fig. 3).

During slow cooling rate 2°C/min after casting, when the eutectic composition was reached at the interface front the eutectic reaction L→ α-Al + β-$Al_5FeSi$ followed [9]. According to the research on the solidification processing of this series alloy the precipitation temperature of β-$Al_5FeSi$ phase is about 570$^o$C were there is about 10% liquid [9]. The β-$Al_5FeSi$ with monoclinic crystal structure precipitates in the interdendritic regions generally as a plate-like morphology (Fig. 4).

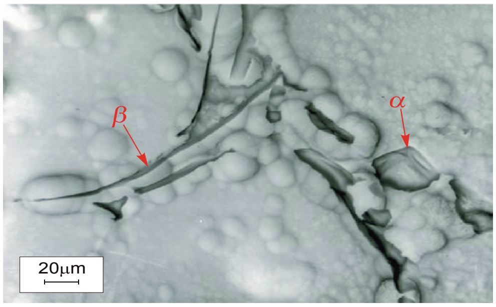



Fig. 4. Morphology of plate-like β-$Al_5FeSi$ intermetallic phase

It is difficult to distinguish the intermetallic phases e.g. the β-$Al_5FeSi$, using classical metallographic technique. Therefore, besides light optical microscopy examination, SEM observation connected with EDS analysis of deep etched section were also made. The results of EDS analysis made on the coarse intermetallic phases are present in Fig. 5. and Table 1. Comparison of selected reference data to the results obtained by EDS scans showed good agreement between them (Table 1).

Table 1.
Composition of the observed intermetallic phases (%wt)

| Phase | Si | Fe | Mn | References |
|---|---|---|---|---|
| β-$Al_5FeSi$ | 12-15 | 25-30 | | [10] |
| | 12,2 | 25 | | [11] |
| | 14,59 | 27,75 | | [9] |
| | 13-16 | 23-26 | | This work |
| α-Al(FeMn)Si | 10-12 | 10-15 | 15-20 | [10] |
| | 5,5-6,5 | 5,1-27,9 | 14-24,7 | [12] |
| | 5-7 | 10-13 | 19-23 | [9] |
| | 8-12 | 11-13 | 14-20 | This work |

The β-$Al_5FeSi$ phase was also observed as a ternary and a quaternary eutectic component (Fig. 3). The β-$Al_5FeSi$ phase during solidification can precipitates as a product of eutectic or perytectic reaction. According to literature the following reactions are expected during solidification and melting of 6xxx aluminium alloys:

L → α-Al + β-$Al_5FeSi$
L → α-Al + β-AlFeSi + $Mg_2Si$
L + α-AlFeSi → α-Al + β-$Al_5FeSi$

The β-$Al_5FeSi$ phase with a plate-like morphology weaken the ductility and workability of 6xxx alloys. Due to the detrimental effect of the β-$Al_5FeSi$ phase, their formation is avoided trough additions of alloying elements, mainly of Mn and Be [13]. They alter the composition and morphology of β-plates into "Chinesse script", found mostly inside α-Al dendrites.

Microscopic examination of the as-cast microstructure revealed the presence of $Mg_2Si$ phase particles (Fig. 1, 3). The $Mg_2Si$ phase can be readily identified according to its colour and morphology. Darker $Mg_2Si$ particles, in the as-cast state after slow cooling rate - 2°C/min, precipitate in the form of lamellar or "Chinese script" structure (Fig. 5). These intermetallic phases are the products of binary eutectic reactions L→ α-Al + $Mg_2Si$ which occur at temperature ~577°C [10]. When the eutectic composition is reached at the interface front, the ternary eutectic reactions L→ α-Al + β-AlFeSi + $Mg_2Si$ take place (Fig. 6).

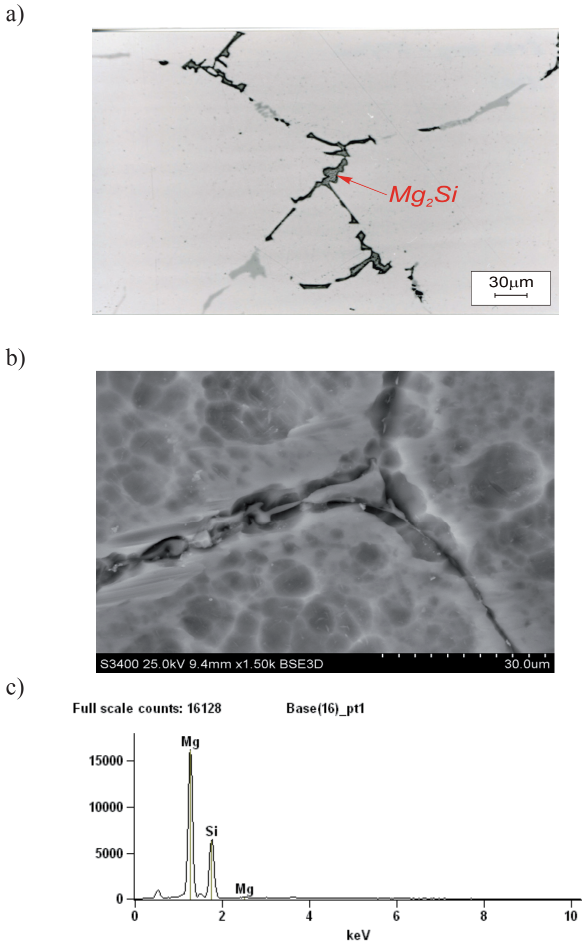



Fig. 5. The eutectic microstructure of α-Al + $Mg_2Si$ (a) LM, (b) SEM micrograph, c) EDS spectra

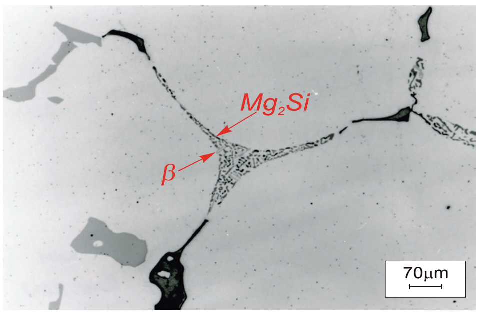



Fig. 6. Microstructure of AlSi1MgMn alloy formed through the ternary eutectic reactions L → α-Al + β-AlFeSi + $Mg_2Si$

The LM and SEM observation connected with EDS clearly confirmed that the Fe-rich intermetallics, in the investigated AlSi1MgMn alloy, were mainly of the α-$Al_{15}(FeMn)_3Si$ type. The α-$Al_{15}(FeMn)_3Si$ phase, also known as α-$Al_8Fe_2Si$ phase have a cubic structure and a compact morphology e.g. polyhedron (Fig. 7), which does not initiate cracks in the cast material to the same extent as the β-$Al_5FeSi$ despite its elevated hardness. When Mn and Mg are added to 6xxx alloys, e.g. AlSi1MgMn alloy, the $\alpha$-$Al_{15}(FeMn)_3Si$ is formed during solidification at the main eutectic and perytectic reaction [14]:

$L \rightarrow \alpha\text{-}Al + \alpha\text{-}Al_{15}(FeMn)_3Si$

$L \rightarrow \alpha\text{-}Al + \alpha\text{-}Al_{15}(FeMn)_3Si + Mg_2Si$

$L{+}Al_6Mn(Fe) \rightarrow \alpha\text{-}AlMnFeSi{+}\alpha\text{-}Al$

The $\alpha$-$Al_{15}(FeMn)_3Si$ phase shows, in contrast to the $\beta$-$Al_5FeSi$, some variations in composition and quite different morfologies depending on the cooling condition [15]. At low cooling rates the $\alpha$-$Al_{15}(FeMn)_3Si$ phase is formed as a primary crystals. When cooling rate increases, those crystals changing morphology to a typical "Chinese script" form (Fig.1, Fig.7) or to a fine eutectic structure (Fig. 3).

a)

b)

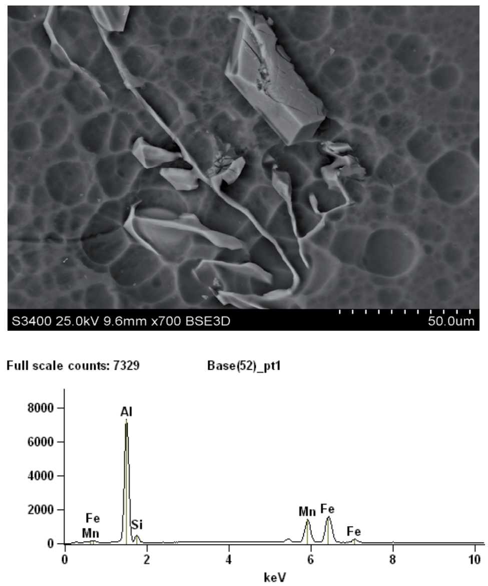



Fig. 7. "Chinese script" and columnar (a) morphologies of $\alpha$-$Al_{15}(FeMn)_3Si$ intermetalic phase, b) EDS spectra

## 4. Conclusions

The alloy used in this study possessed a complex as-cast microstructure. By using various instruments (LM, SEM, XRD) and techniques (imagine, EDS) a wide range of intermetallics phases were identified. After slow solidification at a cooling rate 2$^{o}$C/min, the as-cast microstructure included six phases, namely: $\alpha$-Al, $\beta$-$Al_5FeSi$, $\alpha$-$Al_{15}(FeMn)_3Si$, $Al_9Mn_3Si$, $Mg_2Si$ and Si. Depending on the composition and cooling rate of the alloy, the complex binary, ternary and quaternary eutectic structure in the solidified zone were also observed.

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