



# Intermetallic phase particles in 6082 aluminium alloy

G. Mrówka-Nowotnik\*, J. Sieniawski, M. Wierzbńska

Department of Materials Science, Rzeszów University of Technology,  
ul. W. Pola 2, 35-959 Rzeszów, Poland

\* Corresponding author: E-mail address: mrowka@prz.edu.pl

Received 15.10.2006; accepted in revised form 25.01.2007

## ABSTRACT

**Purpose:** In the technical 6xxx Al alloys besides the intentional additions Mg i Si, transition metals and impurities (Fe and Mn) are always present. Even not large amount of these impurities causes the formation a new phase components. The exact composition of the alloy and casting condition will directly influence the selection and volume fraction of intermetallic phases. During casting of 6xxx alloys, a wide variety of Fe-containing intermetallics phases Al-Fe, Al-Fe-Si and Al-Fe-Mn-Si are formed among the aluminium dendrites. The aim of this work was to examine the composition and morphology of complex microstructure of the intermetallics in 6082 aluminium alloy.

**Design/methodology/approach:** Light microscopy (LM), electron microscopy techniques (SEM and TEM) in combination with X-ray analysis (SEM/EDS), and X-ray diffraction (XRD) were used.

**Findings:** The examinations of the as-cast alloy after slow solidification at a cooling rate 2°C/min reveal that the microstructure consisted a wide range of intermetallics phases, namely:  $\beta$ -Al<sub>3</sub>FeSi,  $\alpha$ -Al<sub>15</sub>(FeMn)<sub>3</sub>Si, Al<sub>9</sub>Mn<sub>3</sub>Si,  $\alpha$ -Al<sub>12</sub>Fe<sub>3</sub>Si, Mg<sub>2</sub>Si.

**Research limitations/implications:** To facilitate confirmation of the achieved results it is recommended to execute supplementary analysis of the aluminium alloys, 6xxx series in particular.

**Practical implications:** Since the, what involves changes of alloy properties, From a practical position it is important to understand formation conditions of the intermetallics in order to control final components of the alloy microstructure. The importance of this is due to the fact that morphology, crystallography and chemical composition of the intermetallics strongly affect the properties of the alloy.

**Originality/value:** This work has provided essential data about almost all possible intermetallic phases precipitating in 6000 series aluminium alloys.

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

## MATERIALS

### 1. Introduction

The 6xxx-group alloys have a widespread application, especially in the building, aircraft and automotive industry due to their excellent properties. Increasing demands on such materials have resulted in increasing research and development for high-strength and high-formability aluminium alloys. 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 form during solidification - in the interdendritic regions, homogenisation and thermomechanical 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  $\beta\text{-Al}_3\text{Fe}_2\text{Si}_2$  (also called  $\beta\text{-Al}_3\text{FeSi}$ ) or  $\alpha\text{-Al}_{12}\text{Fe}_3\text{Si}$ . However Mn and Cr can substitute for Fe, and stabilize the formation of  $\alpha\text{-Al}_{12}(\text{FeMn})_3\text{Si}$  or  $\alpha\text{-Al}_{12}(\text{FeCr})_3\text{Si}$  are detrimental to the mechanical properties of the alloys. In addition, experimental studies have demonstrated that the formation of the intermetallic compounds is influenced by the alloy composition. It is very important to understand the formation and dissolution of these compounds. The present work was focused on the study on the study of the complex microstructures of 6082 aluminium alloy on the cast condition [6-8].

## 2. Material and experimental

The present investigation has been carried out using aluminium alloy 6082. The composition of the alloy is indicated in Table 1.

Table 1.

Chemical composition of tested 6082 Al alloy (wt%).

Element						
Si	Mg	Mn	Fe	Cr	Cu	Zn
1.2	0.78	0.50	0.33	0.14	0.08	0.05

Al - balance

Flat samples for structural observations were cut from the specimens along longitudinal axis. The sample surface was prepared using standard grinding and polishing procedure and etched in Keller solution (0,5 % HF in 50ml  $\text{H}_2\text{O}$ ). Microstructure observations were performed within a middle area of the specimen using an optical microscope - Nikon 300.

Specimens for transmission electron microscopy were punched as 3 mm diameter discs then grounded to 50-100  $\mu\text{m}$  and thinned by means PIPS 690-type ion thinning machine. Thin foils were examined by means of JEOL - JEM 2010 ARP transmission electron microscope at 200kV.

Morphology of the phases were examined in the scanning electron microscope HITACHI S-3400 (SEM), operating at 6-10 kV in a conventional back-scattered electron mode. 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 (ARL XTR'A Thermo diffractometer).

## 3. Results and discussion

Microstructure analysis of the as-cast 6082 alloy revealed five types of intermetallic compounds located at the grain boundaries. From Fig. 1 follows that their morphology is different. One can

recognized intermetallics nucleated in form of platelet, rod, polyhedron or "Chinese script".

XRD diffractometry has been utilized to confirm the microscopic examination. The results has provided confidence in phase classification performed based upon metallographic study. The results of XRD analysis in form diffractogram is shown in Fig. 2. Combination of optical microscopic observation (Keller's solution is capable to colorize phases) and XRD results allow us to identify these intermetallic phases as  $\alpha\text{-Al}_3\text{FeSi}$  (dark phase),  $\alpha\text{-Al}_{15}(\text{FeMn})_3\text{Si}$  (grey phase),  $\text{Al}_9\text{Mn}_3\text{Si}$ ,  $\alpha\text{-Al}_{12}\text{Fe}_3\text{Si}$ ,  $\text{Mg}_2\text{Si}$  (black phase) (Fig. 1).

a)



b)

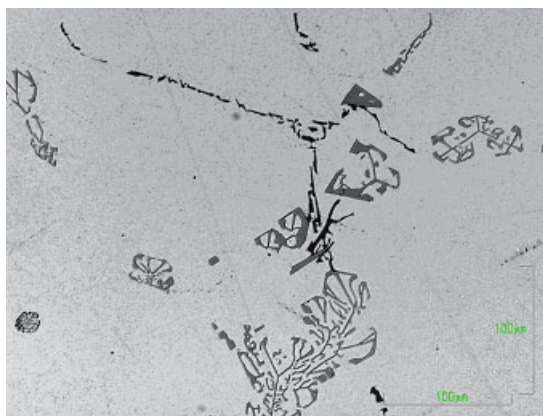


Fig. 1. As-cast microstructure of 6082 alloy

Some complex eutectic structures namely:  $\beta\text{-Al}_3\text{FeSi}$ ,  $\text{Mg}_2\text{Si}$  and  $\alpha\text{-Al}(\text{FeMn})\text{Si}$  and Si were also observed in the  $\alpha\text{-Al}$  matrix of as-cast 6082 alloy (Fig. 3-5). These intermetallics grew together into a eutectic cluster. In addition spherical in shape eutectic particles (Fig. 3d) were also noticeable.

$\text{L} \rightarrow \alpha\text{-Al} + \beta\text{-Al}_3\text{FeSi}$  eutectic reaction take place upon slow cooling ( $2^\circ\text{C}/\text{min}$ ) after casting. This reaction occurs when the eutectic composition is reached at the interaction front [9]. Literature data on the solidification process in this series alloy indicates that the precipitation temperature of  $\beta\text{-Al}_3\text{FeSi}$  intermetallic was about  $570^\circ\text{C}$  that corresponds to the amount of 10% liquid [9]. This intermetallic ( $\beta\text{-Al}_3\text{FeSi}$ ) generally precipitates in the interdendritic regions in form of plates (Fig. 6).

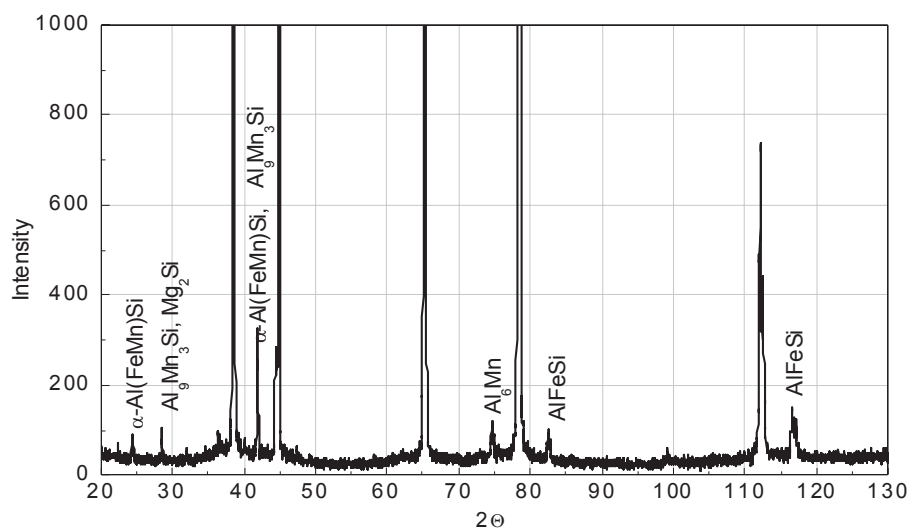


Fig. 2. XRD pattern of 6082 alloy in the as-cast state

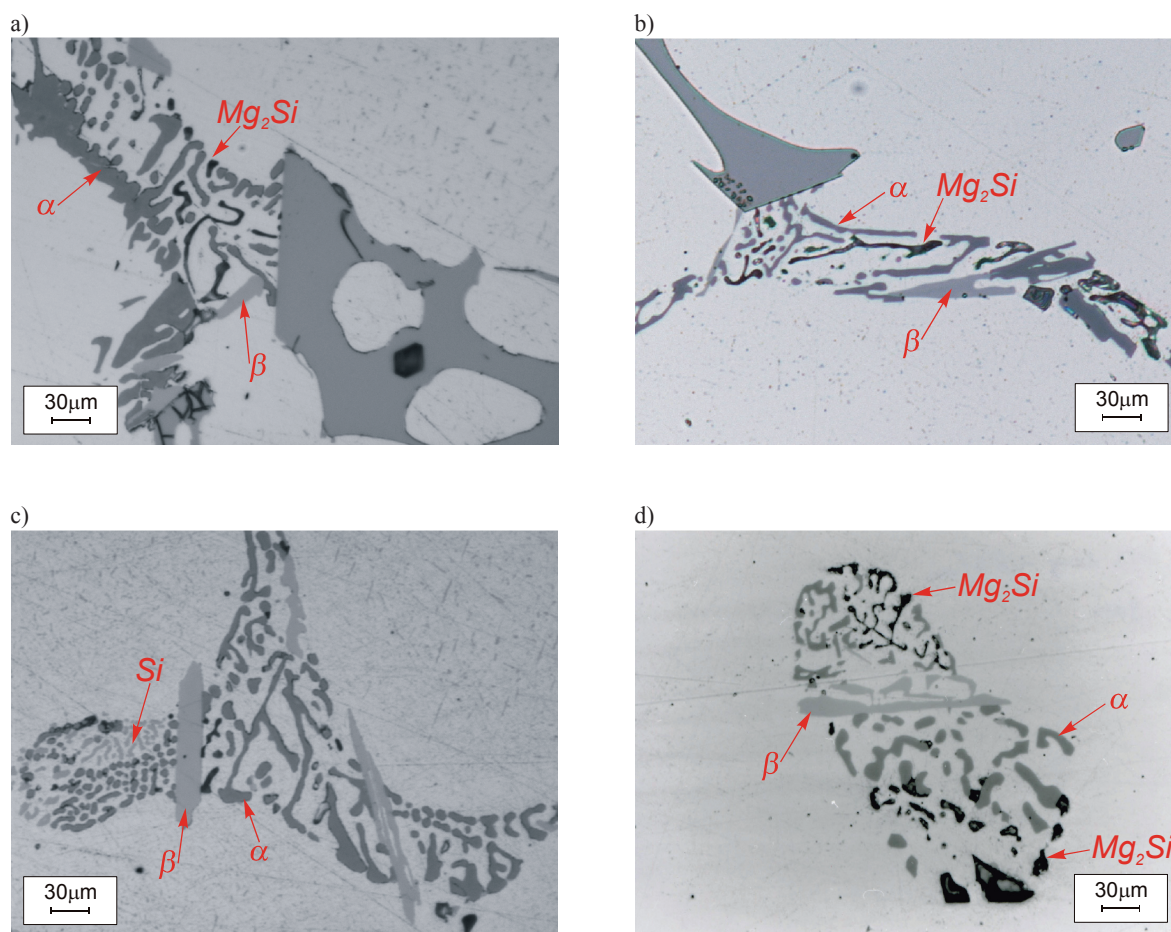


Fig. 3. The microstructure of 6082 alloy in the as-cast condition: a-b) ternary eutectic, c-d) the quaternary eutectic



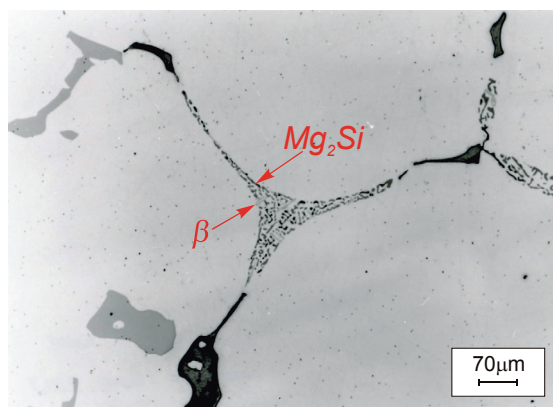


Fig. 4. Microstructure of AlSi1MgMn alloy formed via the ternary eutectic reactions  $L \rightarrow \alpha\text{-Al} + \beta\text{-AlFeSi} + \text{Mg}_2\text{Si}$

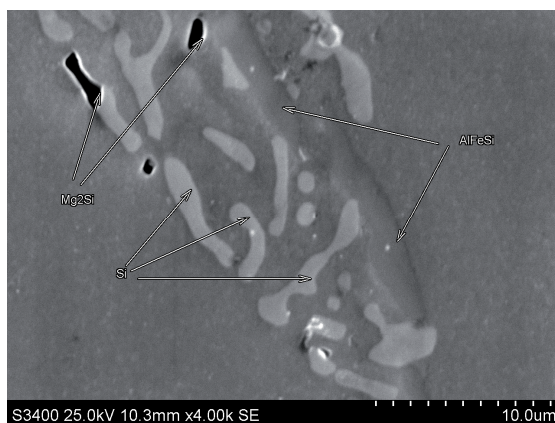


Fig. 5. The quaternary eutectic structure forming via the reaction:  $L \rightarrow \alpha\text{-Al} + \beta\text{-AlFeSi} + \text{Mg}_2\text{Si} + \text{Si}$

Table 2.

Chemical composition of determined intermetallic phases (%wt)

Phase	Si	Fe	Mn	References
$\beta\text{-Al}_3\text{FeSi}$	12-15	25-30		[10]
	12,2	25		[11]
	14,59	27,75		[9]
	13-16	23-26		This work
$\alpha\text{-Al(FeMn)Si}$	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

Since it is not easy to differentiate the morphology of intermetallic phases e.g. the  $\beta\text{-Al}_3\text{FeSi}$ , using only one technique, besides light optical microscopy examination, observation by scanning electron microscopy combined with EDS analysis of deep etched section were performed (Table 2). Results of EDS analysis have been compared to the selected reference data and are in excellent agreement with the result of [9-12] (Table 2).

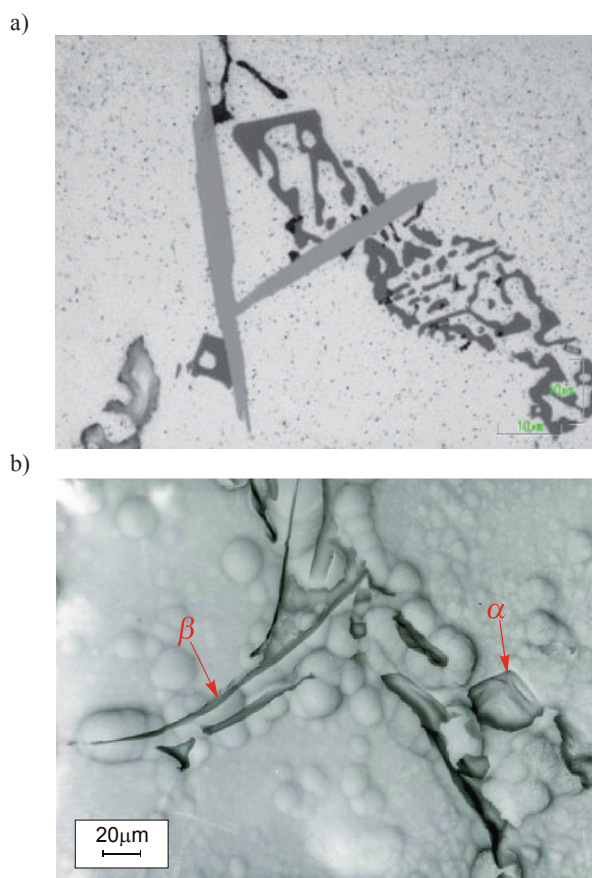


Fig. 6. Morphology of plate-like  $\beta\text{-Al}_3\text{FeSi}$  intermetallic phase (a) optical micrograph, (b) SEM micrograph

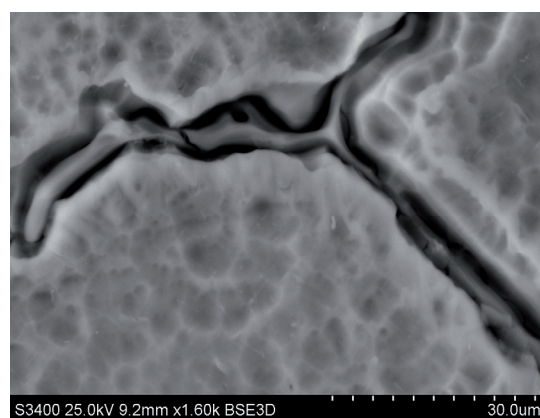


Fig. 7. "Chinese script"  $\beta\text{-Al}_3\text{FeSi}$  phase in 6082 alloy

$\beta\text{-Al}_3\text{FeSi}$  intermetallic phase was observed in the microstructure of the 6082 alloy as a ternary (Fig. 4) and a quaternary eutectic constituent (Fig. 3).  $\beta$  intermetallic can form via eutectic or peritectic reaction:

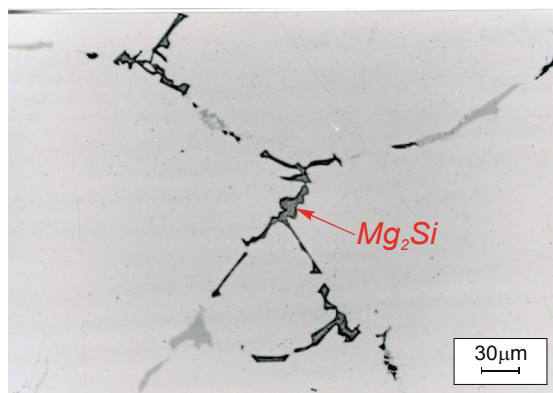
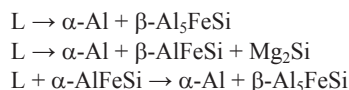


Fig. 8. The eutectic microstructure of  $\alpha\text{-Al} + \text{Mg}_2\text{Si}$  (LM)

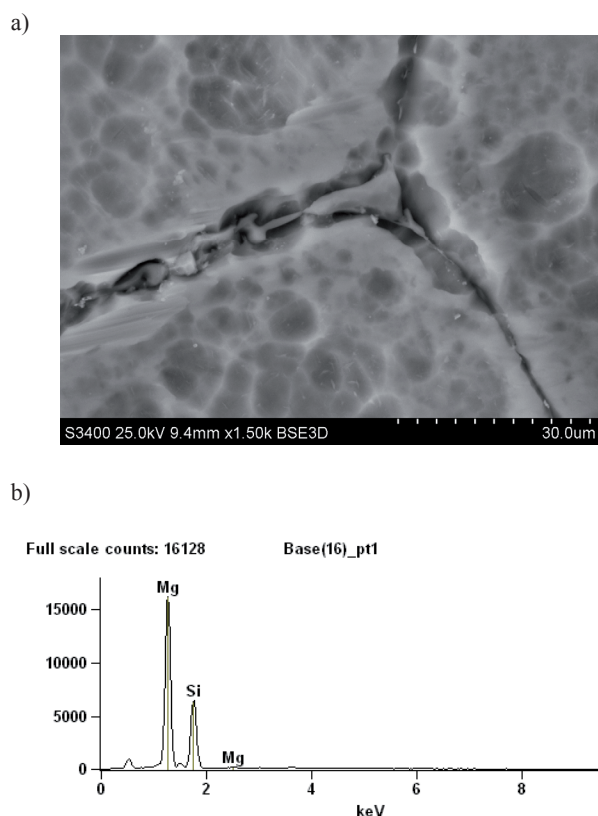


Fig. 9. Microstructure formed via the eutectic reaction of  $\alpha\text{-Al} + \text{Mg}_2\text{Si}$  (a) SEM micrograph, b) EDS spectra

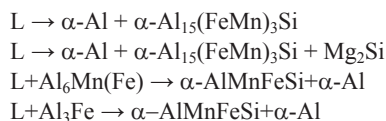
This  $\beta\text{-Al}_5\text{FeSi}$  phase in form of plates deteriorate the ductility and workability of 6xxx alloys. Their nucleation can be

avoided through additions of alloying elements, mainly of Mn and Be [13]. With the addition of the needle-type  $\beta\text{-Al}_5\text{FeSi}$  particles are modified to “Chinese script”. (see Fig. 10). Modified particles were found mostly inside of  $\alpha\text{-Al}$  dendrites (Fig. 7).

Examination of the as-cast microstructure of 6082 after slow cooling rate -  $2^\circ\text{C}/\text{min}$  alloy by light microscopy clearly showed the presence of  $\text{Mg}_2\text{Si}$  phase particles (Fig. 1, 3-5). After etching with Keller's reagent of polished surfaces of the alloy  $\text{Mg}_2\text{Si}$  particles appearing black in colour. All of the  $\text{Mg}_2\text{Si}$  particles were present in lamellar or “Chinese script” form. (Fig. 8,9). In all cases  $\text{Mg}_2\text{Si}$  primary particles solidified from the liquid followed by binary eutectic reactions  $L \rightarrow \alpha\text{-Al} + \text{Mg}_2\text{Si}$  (Fig. 8).

This eutectic reaction is completed at temperature of about  $577^\circ\text{C}$  [10]. The pseudo ternary:  $L \rightarrow \alpha\text{-Al} + \beta\text{-AlFeSi} + \text{Mg}_2\text{Si}$  and quaternary eutectic reactions  $L \rightarrow \alpha\text{-Al} + \beta\text{-AlFeSi} + \text{Mg}_2\text{Si} + \text{Si}$  occur when the eutectic composition is reached at the interface front (Fig. 3-5).

EDS analysis performed on particles showed in Fig. 10a,b indicated that those phases generally contained, apart from Al, Si and Mn, significant amounts of Fe (Fig. 10a,b). Thus these particles were identified as  $\alpha\text{-Al}_{15}(\text{FeMn})_3\text{Si}$  type. Fe-rich  $\alpha$ -phase also identified by formula  $\alpha\text{-Al}_8\text{Fe}_2\text{Si}$  was found to have a cubic structure and a compact morphology e.g. polyhedron (Fig. 10a,b). Because of its shape, these intermetallic give, despite of elevated hardness, a lower ductility than  $\beta\text{-AlFeSi}$  particles. Thus, they do not deteriorate mechanical properties of the alloy. The morphology of  $\alpha\text{-Al}_{15}(\text{FeMn})_3\text{Si}$  phase changed with the cooling rate and the amount of Mn and Mg [15-20]. At relatively low cooling rate the  $\alpha\text{-Al}_{15}(\text{FeMn})_3\text{Si}$  phase is formed as a primary crystals. When cooling rate increases, those crystals changing morphology to a typical “Chinese script” form (Fig. 1, Fig. 10c,d) or to a fine eutectic structure (Fig. 3). When Mn and Mg is present in  $\text{AlSi1MgMn}$  alloy,  $\alpha$  phase is solidified via the main eutectic and peritectic reactions [14]:



TEM study of the microstructure of 6082 alloy showed complex  $\alpha\text{-AlFeSi}$  particles (Figs. 11a,b and 13a,b). These particles appeared to predominantly represent interdendritic regions of the as-solidified  $\alpha\text{-Al}$  matrix. Diffraction study (Figs. 11c and 12c) revealed the cubic ordered crystal structure with a lattice parameter  $a = 12.56 \text{ \AA}$ .

## 4. Conclusions

In the 6082 aluminium alloy applied in this study besides  $\alpha\text{-Al}$  matrix a wide range of intrmetallics phases were observed. This alloy possessed a complex as-cast microstructure. Application of various instruments (LOM, TEM, SEM, XRD) and techniques (imagine, EDS) allow us to identify different Si - content intermetallics phases. After slow solidification at



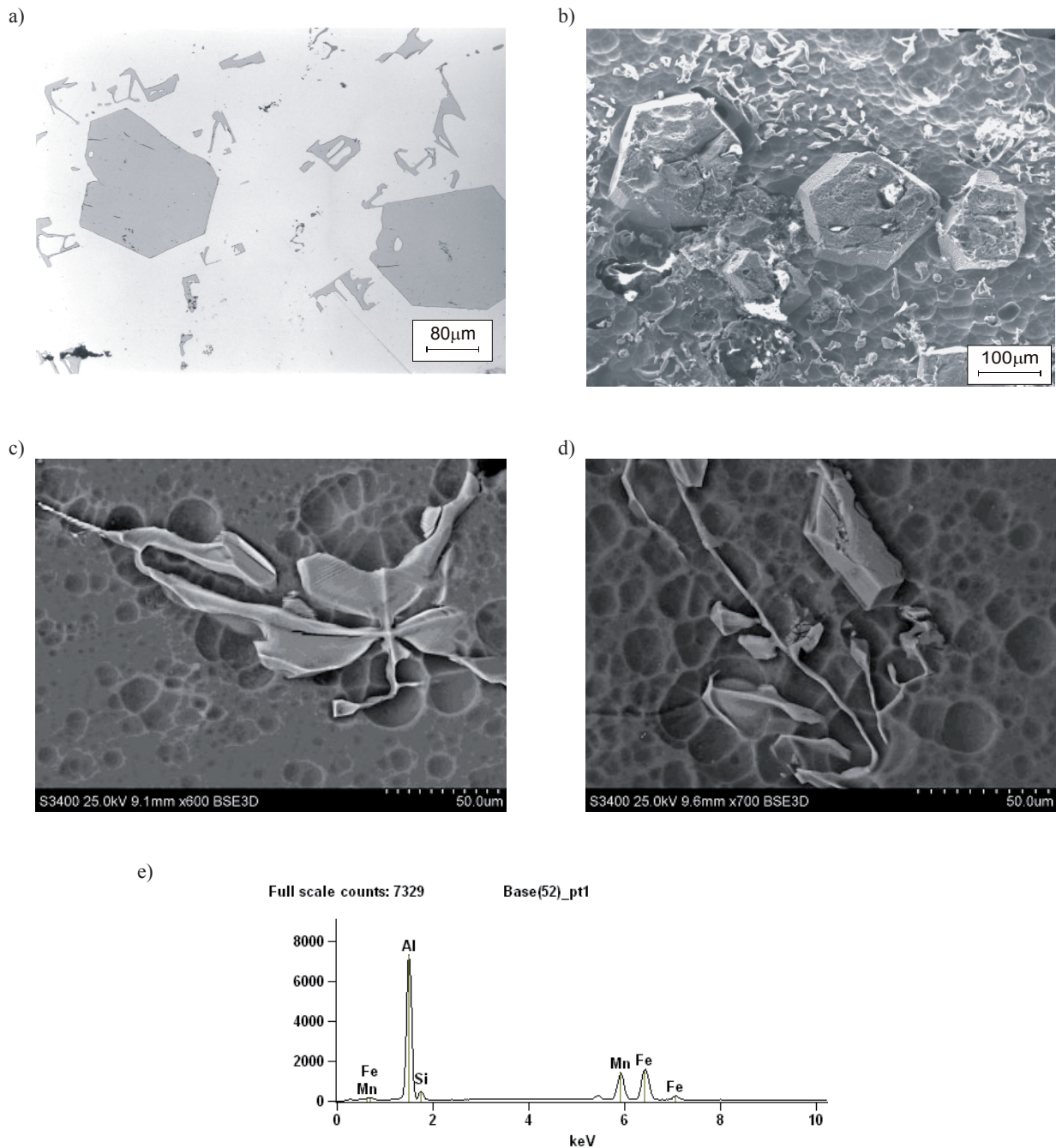


Fig. 10. Polyhedron (a) LM, (b) SEM; “Chinese script” (c, d) and columnar (d) morphologies of  $\alpha\text{-Al}_{15}(\text{FeMn})_3\text{Si}$  intermetallic phase, e) EDS spectra

a cooling rate of  $2^\circ\text{C}/\text{min}$ , the as-cast microstructure consisted seven phases, namely:  $\alpha\text{-Al}$ ,  $\beta\text{-Al}_3\text{FeSi}$ ,  $\alpha\text{-AlFeSi}$ ,  $\alpha\text{-Al}_{15}(\text{FeMn})_3\text{Si}$ ,  $\text{Al}_9\text{Mn}_3\text{Si}$ ,  $\text{Mg}_2\text{Si}$  and Si between the aluminium dendrites. Depending on the composition and cooling rate of the

alloy, the complex binary ( $\text{L} \rightarrow \alpha\text{-Al} + \beta\text{-Al}_3\text{FeSi}$ ,  $\text{L} \rightarrow \alpha\text{-Al} + \text{Mg}_2\text{Si}$ ) ternary ( $\text{L} \rightarrow \alpha\text{-Al} + \beta\text{-AlFeSi} + \text{Mg}_2\text{Si}$ ) and quaternary ( $\text{L} \rightarrow \alpha\text{-Al} + \beta\text{-AlFeSi} + \text{Mg}_2\text{Si} + \text{Si}$ ) eutectic structure in the solidified zone were also observed.

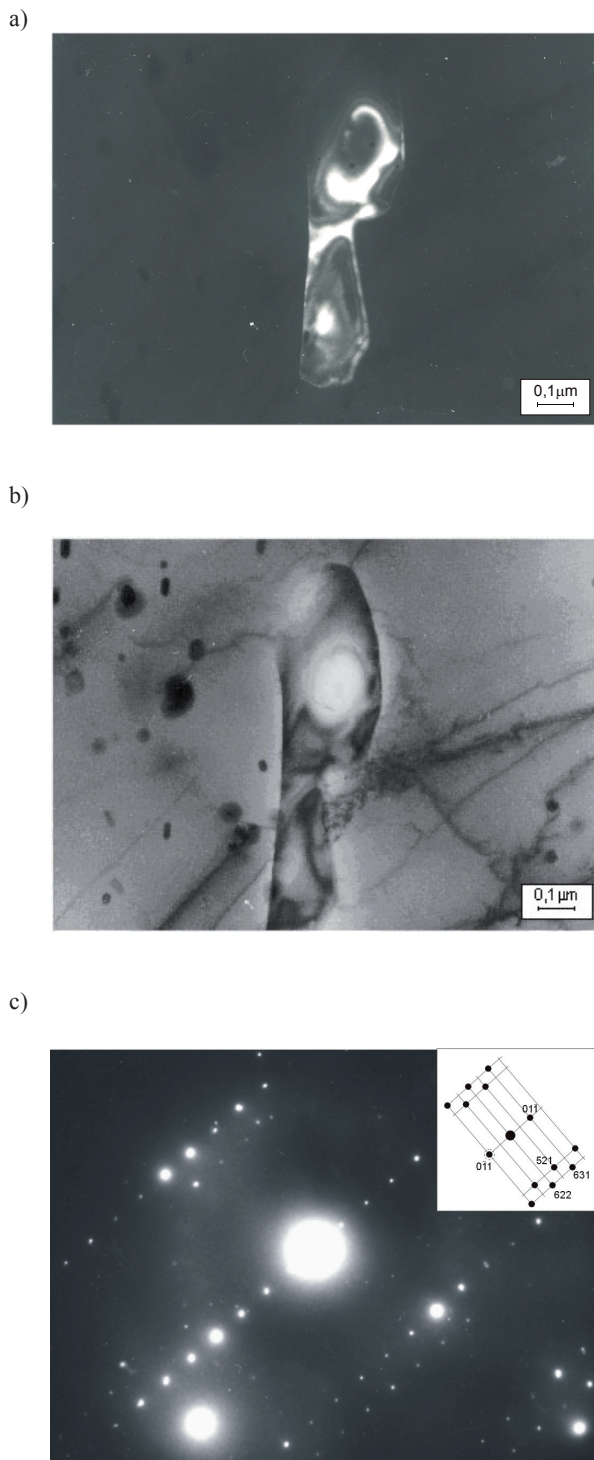


Fig. 11. (a) TEM dark-field and (b) bright field images of  $\alpha$ -AlFeSi particles found in the microstructure of 6082 alloy; (c) micro-diffraction pattern from one of the particles displaying  $[2-3-3]$  zone axis.

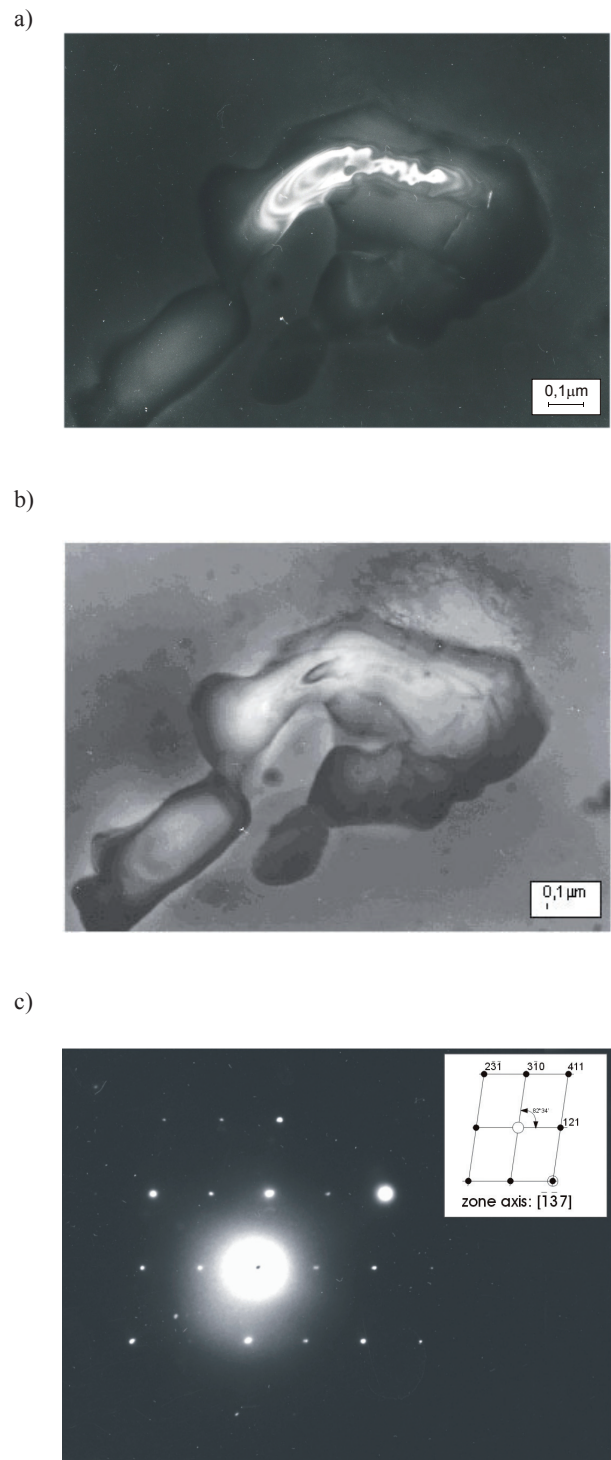


Fig. 12. (a) TEM dark-field and (b) bright field images of a cluster of  $\alpha$ -AlFeSi particles found in the microstructure of 6082 alloy; (c) corresponding micro-diffraction pattern with a  $[-1-37]$  zone axis.

## Additional information

The presentation connected with the subject matter of the paper was presented by the authors during the 12<sup>th</sup> International Scientific Conference on Contemporary Achievements in Mechanics, Manufacturing and Materials Science CAM3S'2006 in Gliwice-Zakopane, Poland on 27<sup>th</sup>-30<sup>th</sup> November 2006.

## Acknowledgements

This work was carried out with the financial support of the Ministry of Science and Information Society Technologies under grant No. 3T08B 078 27.

## References

- [1] C.D. Marioara, S.J. Andersen, J. Jansen, H.W. Zandbergen, The influence of temperature and storage time at RT on nucleation of the  $\beta''$  phase in a 6082 Al-Mg-Si alloy, *Acta Materialia* 51 (2003) 789-796.
- [2] N.C.W. Kuijpers, W.H. Kool, P.T.G. Koenis, K.E. Nilsen, I. Todd, S. van der Zwaag, Assessment of different techniques for quantification of  $\alpha$ -Al(FeMn)Si and  $\beta$ -AlFeSi intermetallics in AA 6xxx alloys, *Materials Characterization* 49 (2003) 409-420.
- [3] J.E. Hatch Ed., *Aluminium. Properties and Physical Metallurgy*. ASM Metals Park, Ohio 1984.
- [4] S. Zajac, B. Bengtsson, Ch. Jönsson, Influence of cooling after homogenization and reheating to extrusion on extrudability and final properties of AA 6063 and AA 6082 alloys, *Materials Science Forum* 396-402 (2002) 399-404.
- [5] G. Mrówka-Nowotnik, J. Sieniawski, Influence of heat treatment on the microstructure and mechanical properties of 6005 and 6082 aluminium alloys, *Journal of Materials Processing Technology* 162-163 (2005) 367-372.
- [6] A.K. Gupta, P.H. Marois, D.J. Lloyd, Review of the techniques for the extraction of second-phase particles from aluminium alloys, *Materials Characterization* 37 (1996) 61-80.
- [7] R.A. Jeniski Jr, Effect of Cr addition the microstructure and mechanical behavior of 6061-T6 continuously cast and rolled redraw rod, *Materials Science and Engineering A237* (1997) 52-64.
- [8] M. Warmuzek, G. Mrówka, J. Sieniawski, Influence of the heat treatment on the precipitation of the intermetallic phases in commercial AlMn1FeSi alloy, *Journal of Materials Processing Technology* 157-158 (2004) 624-632.
- [9] Y.L. Liu, S.B. Kang, H.W. Kim, The complex microstructures in as-cast Al-Mg-Si alloy, *Materials Letters* 41 (1999) 267-272.
- [10] L.F. Mondolfo, *Aluminium Alloys: Structure and Properties*. Butterworths, London-Boston, 1976.
- [11] M. Warmuzek, K. Rabczak, J. Sieniawski, The course of the peritectic transformation in the Al-rich Al-Fe-Mn-Si alloys, *Journal of Materials Processing Technology* 162-163 (2005) 422-428.
- [12] M. Warmuzek, J. Sieniawski, K. Wicher, G. Mrówka-Nowotnik, The study of distribution of the transition metals and Si during primary precipitation of the intermetallic phases in Al-Mn-Si alloys, *Journal of Materials Processing Technology* 175 (2006) 421-426.
- [13] S.R. Claves, D.L. Elias, W.Z. Misiolek, Analysis of the intermetallic phase transformation occurring during homogenization of 6xxx aluminum alloys. *Materials Science Forum* 396-402 (2002) 667-674.
- [14] M. Warmuzek et al., Influence of heat treatment on the precipitation of the intermetallic phases in AlMn1FeSi, *Proceedings of the 11<sup>th</sup> International Conference Achievements in Mechanical & Materials Engineering, AMME'2002, Gliwice-Zakopane, 2002*, 601-604.
- [15] L.A. Dobrzański, W. Borek, R. Maniara, Influence of the crystallization condition on Al-Si-Cu casting alloys structure, *Journal of Achievements in Materials and Manufacturing Engineering* 18 (2006) 211-214.
- [16] G. Mrówka-Nowotnik, J. Sieniawski, M. Wierzbńska, Analysis of intermetallic particles in AlSi1MgMn aluminium alloy, *Journal of Materials Processing Technology* 20 (2007) 155-158.
- [17] Y. Birol, The effect of processing and Mn content on the T5 and T6 properties of AA6082 profiles, *Journal of Materials Processing Technology* 173 (2006) 84-91.
- [18] C.D. Marioara, S.J. Andersen, J. Jansen, The influence of temperature and storage time at RT on nucleation of the beta, "Phase in a 6082 Al-Mg-Si Alloy", *Acta Materialia* 51 (2003) 789-796.
- [19] M.M. Haquea, A.F. Ismailb, Effect of superheating temperatures on microstructure and properties of strontium modified aluminium-silicon eutectic alloy, *Proceedings of the 13<sup>th</sup> Scientific International Conference Achievements in Mechanical and Materials Engineering, AMME'2005, Gliwice-Wisła, 2005*, 267-270.
- [20] G. Mrówka-Nowotnik, J. Sieniawski, M. Wierzbńska, Analysis of intermetallic particles in AlSi1MgMn aluminium alloy, *Journal of Achievements in Materials and Manufacturing Engineering* 20 (2007) 155-158.