Influence of heat treatment on the precipitation of the intermetallic phases in commercial AlMn1FeSi alloy*

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1. INTRODUCTION

In the technical aluminium alloys, besides the intentional additions, transition metals such as Fe and Mn are always present. As it has already been presented earlier, even not large amount of these impurities causes the formation of a new phase components. Therefore, many efforts are focused on the possibility of the control of the processes of their formation, among other things, to eliminate and to minimise their negative effect on the functional properties of the material [1-6]. Processes of formation of these phases can occur during alloy solidification from the liquid phase [7,8] and during the cooling or the heat treatment in the solid state [1-6].

Occurring in the interdendritic regions phase of a cubic lattice α-$Al_x(FeMn)_ySi_z$, that is designated by Backerund as α-$Al_{15}(FeMn)_3Si_2$, can be either the eutectic component (1) or peritectic transformation product (2) [7,8] (in the temperature range of 640-589°C):

$$L \rightarrow \alpha\text{-}Al + \alpha\text{-}Al(FeMn)Si \quad (1)$$

$$L + Al_6(FeMn) \rightarrow \alpha\text{-}Al(FeMn)Si \quad (2)$$
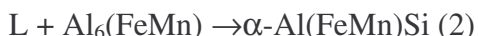
Primary precipitation of α-Al(FeMn)Si is limited by summary Fe+Mn+Si amount (less than 18%wt.) [8]. In the solid state the homogeneous nucleation of this phase was reported during a heat treatment at 550°C, but the heterogeneous nucleation on the interface: primary $Al_6(FeMn)/\alpha$-Al is considered the main process of its formation [1,5,10]. The process of the α-Al(FeMn)Si phase formation is controlled by chemical composition of the alloy, but in the case of the no equilibrium conditions the local fluctuations of the Fe, Mn and Si concentrations decide the precipitation course. When the local concentration of Fe is low (in relation to Mn) the cubic phase $Al_{12}Mn_3Si$ (Pm3 space group) occurs. Inside the microregions of locally increased Fe concentration, a cubic phase of bcc lattice (space group Im3), occurs [1]. A limiting concentration of Fe, limiting the space order of the crystal lattice has not been specified yet. Nevertheless, an increase in content of Fe causes distinct growth in volume fraction of the quaternary α-Al(FeMn)Si phase particles (because of the very low solubility of these elements in Al) whilst its morphology and size depend on a value of the Fe/Mn ratio [6,9,10].

The main task of this work was to analyse the influence of the heat treatment of AlMn1FeSi alloy on the evolution of either morphology and chemical composition of the particles of intermetallic phases containing of Mn, Fe, Si and Al.

2. MATERIAL AND EXPERIMENTAL

Material for the examination was the commercial AlMn1FeSi alloy of the chemical composition: Mn1.05%, Fe0.14%, Si0.10% (Al-bal.).

By means of the quenching BAHR´s dilatometer, type 805 A/D the temperature of the thermal effects in the material were registered. In order to determine the temperature of equilibrium processes a sample was heated up and cooled down with the rate of 4K/min in the scanning micro calorimeter - Nest DSC404C. The microstructure of the alloy (for the specific time-temperature conditions) was "frozen" by rapid cooling of the samples in the ice-water and examined using the light microscope Neophot 32, on metallographic microsections (etched with 1% HF in H_2O dist.). Chemical composition of the phase particles was estimated by means of X-ray microanalyser EDS (LINK ISIS 300 combined with Stereoscan 420) using standardless method. Measurements were carried out for each group of particles, classified on the basis of a mean contents of Si. Phase analysis was carried out by means of X-ray diffractometer TUR M62+HZG4A, with application of Cu-rays and Ni filter.
3. RESULTS

3.1 Results of dilatometry studies

On the base of the dilatometry examinations, the temperatures of characteristic points for either precipitation processes or dissolution of the structural constituents of the alloy, has been evaluated. The results of measurements are given in Table2.

Table 2. Results of the dilatometric analysis

Heating rate, °C/sec	Transformation temperature, °C			
	I	II	III	IV
0,08	290*	360 360*	418 460*	551
0,18		360	439	510

∗Results obtained by DSC method

Analysis of thermograms, obtained by DSC method, during heating, shows that within the temperature range 200-500°C, three transformations related to the thermal effects proceed: two endothermic peaks - 360°C and 460°C and one exothermic peak - 290°C were registered.

3.2 Metallographic examinations

The microstructure of the alloy in the as-cast state consisted predominantly of the $Al_6(MnFe)$ particles (Fig. 1a) in form of the polyhedrons. The Chinese-script particles were formed during the succeeding steps of the heat treatment. Simultaneously, in solid solution more and more disperse precipitates were occurring and more clearly a precipitates-free zones were marked (Fig 1c).

a)   b)   c)   d)
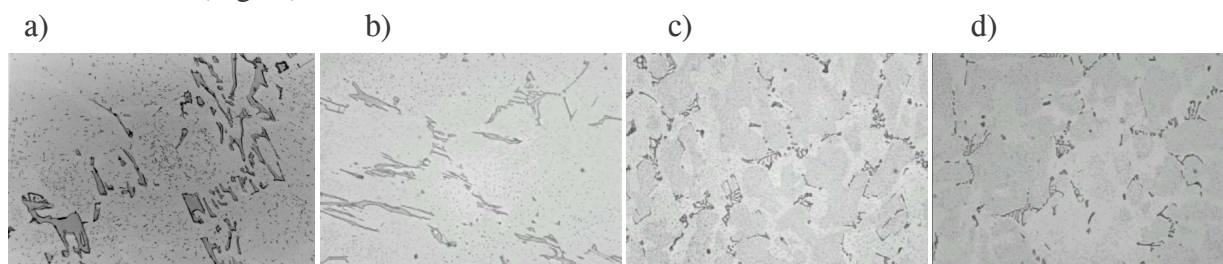
Fig. 1. Microstructure of examined alloy: a/ as-cast alloy, mag. x740, b /alloy after annealing (7h), frozen at 360°C, mag. x750, c/ alloy heat treated at 418°C/7 h, mag. x750, d/ alloy heated with the rate of 0.1°C/sec to 600°C and frozen, mag. x750

3.3 Results of the estimation of the chemical composition and structural identification of the phase precipitates in examined specimens

The chemical composition of the intermetallic phases are given in Table 3. On the base of the estimated chemical composition, the analysed precipitates can be identify as the $Al_6(MnFe)$ phase in form of both polyhedrons and Chinese-script, precipitated in the liquid state as well as from the solid solution at lower annealing temperature. A new phase (α-$Al_x(FeMn)_ySi_z$, where: x=18, y=3, z=1) was occurred above of 450°C. The phase composition of examined samples was confirmed also by X-ray diffraction analysis. The particles of the orthorhombic $Al_6(FeMn)$ phase and those of the cubic α-$Al_x(FeMn)_ySi_z$, were identified. Evaluated chemical composition of observed primary intermetallic phases, in the interdendritic spaces, was changing during heat treatment. It can be stated that as an annealing temperature increased, a tendency to increasing of Mn and simultaneously to decreasing of Fe contents in analysed precipitates appeared.

Table 3. Chemical composition of the intermetallic phases precipitates

Specimen	Morphology	Chemical composition, %wt			Chemical composition, %at			Fe/Mn
		Si,	Mn	Fe	Si	Mn	Fe	
AlMn1/ dilatometer	polyhedron	3,9	10,1	11,8	4,2	5,6	6,5	1,2
		0,6	12,6	11,8	0,6	7,1	6,5	0,92
	Chinese script	3,2	13,0	14,7	3,6	7,5	8,3	1,1
		0,3	8,8	11,4	0,3	4,8	6,2	1,3
AlMn1/465	polyhedron	4,0	11,1	13,1	4,4	6,3	7,3	1,15
		0,3	10,8	10,4	0,3	6,0	5,6	0,93
	Chinese script	0,8	11,6	14,6	0,9	6,6	8,1	1,22
AlMn1/450	polyhedron	0,5	11,9	11,3	0,6	6,6	6,2	0,9
	Chinese script	0,5	8,7	9,5	0,5	4,7	5,1	1,09
AlMn1/400	polyhedron	0,3	9,6	9,7	0,4	5,2	5,2	1,0
	Chinese script	0,8	9,3	9,5	0,8	5,1	5,1	1
AlMn1/350	polyhedron	0,3	11,1	12,5	0,3	6,2	6,9	1,1
	Chinese script	-	11,8	10,8	-	6,5	5,9	0,91
AlMn1/cast	polyhedron	0,6	11,1	12,9	0,6	6,2	7,1	1,14

4. DISCUSSION AND SUMMARY

Analysis of the microstructure allows revealing an evolution of its morphology: an increase in the volume fraction of the Chinese-script phase (Fig.2), dissolution of non-equilibrium interdendritic eutectic and globularization of the particles of intermetallic phases. The microstructure effects related to the composition of the phase precipitates, allows attributing the thermal effect observed above the temperature of 400°C to occurring a new equilibrium phase α-$Al_x(FeMn)_ySi_z$. It forms initially in form of the elongated polyhedrons (465°C) and after that (600°C) in form of the Chinese script (Fig. 1d). Since the diffusion process courses very slowly, the previously existing particles α-$Al_6(FeMn)$ remain covered with the of α-Al(FeMn)Si-phase envelope, even at high temperature and after long time of the annealing.

Mentioned in the literature [5,10] phenomenon of the heterogeneous nucleation of the equilibrium phase in a interface: matrix/phase Al_6(FeMn), might be related to the thermal effect which was observed at temperature of 360°C, when the process might already begin. However it goes on very slowly, because of low diffusivity of Fe and Si in the matrix. Developed interface of Chinese script precipitates can contribute to the local shortening of the diffusion path.
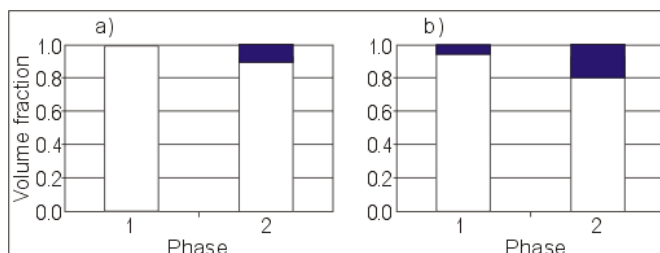

Fig. 2. Relative volume fraction of intermetallic phases of the alloy after isothermal annealing at: a) 465°C, b) 600°C (1 – Chinese script, 2 – polyhedrons, white field - Al_6(FeMn) phase, black field α-Al(FeMn)Si phase

Solubility of Si in Al_6(FeMn) is insignificant [8,10], hence within an analysed range of temperature, augmenting content of this element in the primary precipitates of this phase has not been observed. At the higher temperature of annealing the Mn atoms are included in the particles of Al_6(FeMn), replacing Fe atoms and changing the initial value of the Fe/Mn. It can be explained by faster diffusion of Mn atoms inside the particles of Al_6(FeMn) as well as in the matrix. In polyhedrons of Al_6(FeMn), about of 48 - 54% Mn atoms have been replaced with Fe atoms, likewise in precipitates of this phase in form of Chinese script. In α-Al(FeMn)Si phase, Mn atoms have filled about 43-46% sites of transition metals in crystal lattice. Such amount of this element is sufficient to stabilise the cubic bcc α-Al(FeMn)Si phase [9]. It has to be noted that the relative content of both Fe and Mn in the cubic phase α-Al(FeMn)Si, can vary significantly within quite extensive limits [1,2]. This is most likely due to Mn atoms occupying the sites in the crystal lattice without any fundamental transformations of the crystallographic system. Composition of α-Al(FeMn)Si particles determined by means of the EDS microanalysis, is characterised by to high contents of Al in comparison to the stoichiometry specified in the literature [6-8]. It arises from the presence of the some volume of the matrix in the analysed microregion. Nevertheless, assuming the very low solubility of Fe, Mn and Si in the matrix, obtained results may be used to estimate the relative concentration of these elements in the precipitates of the α-Al(FeMn)Si phase. Value of (Fe+Mn)/Si, near to 3, allows to assume that a quaternary intermetallic phase has an apparent stoichiometry α-Al_{12}(FeMn)$_3$Si.

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