Differential Scanning Calorimetry through the Example of AICu15Mg5 Alloys

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Abstract. The effect of titanium content on the microstructure and properties of aluminium-coppermagnesium alloy (AlCu15Mg5) was examined using differential scanning calorimetry (DSC), Xray diffraction (XRD), hardness and compression testing. DSC analysis of AlCu15Mg5 alloys has shown that increasing of Ti content shift the temperature of two endothermal peaks. The value of enthalpy for the first peak was increased, while for the second endothermal peak it was decreased with the Ti addition in Al-Cu15Mg5 alloy. From the enclosed XRD results of the testing it can be seen that addition of titanium causes the modification of the structure, i.e. making finer grains. Increases of Ti addition improve the hardness and compression strength level.

Introduction

Excellent strength vs. density ratio, formability and corrosion resistance, make high-copper AlCuMg alloys a potential candidate for a number of industrial applications [1]. Developed in the early times in the aeronautical field, they have been then considered for a wide range of different applications, even though, due to their high specific strength, they are mainly considered as a substitution of iron-based materials for structural parts in the transportation industry. Several technical compositions are presently standardized and new alloys based on that metallic system are now being considered and developed [2]. We have found that forming and phase transformation in these alloys are mostly influenced by magnesium and titanium content. In this investigation the main attention was paid to the influence of titanium content on the microstructural changes and mechanical properties in Al-Mg15-Cu5 alloys (0 wt.%Ti, and 0.3 wt.%Ti). DSC analysis and XRD study were performed in order to investigate the effects of titanium content on the microstructural changes and mechanical properties in Al-Mg15-Cu5 alloys.

Experimental

Two Al-Cu-Mg alloys with chemical composition given in Table 1 have been used in this study. These alloys were produced by melting and casting. The solidification structure of one alloy was modified by the addition of the AlTi5B1 in order to produce an Al-Cu-Mg alloy containing 0.3 wt.% Ti.

Alloy	Al	Fe	Si	Cu	Zn	Mg	V
AlCu15Mg5 (0%Ti)	78.32	0.20	0.10	15.31	0.086	5.897	0.004
AlCu15Mg5 (0.3%Ti)	77.15	0.21	0.11	15.44	0.080	5.645	0.011

Table 1. Chemical composition of the investigated alloys (in wt.%).

Microstructural characterization of cast samples has been done by differential scanning calorimetry (DSC) and X-ray powder diffraction (XRD) method. DSC analysis was performed using differential scanning calorimeter type "Shimadzu DSC-50" under protective argon

atmosphere, at a heating rate of 10°C/min, up to the maximum temperature of 725°C [3]. Mass of tested samples was in range of 15.02-15.16 mg.

The X-ray diffraction analysis was performed on the AlCu15Mg5 (0%Ti and 0.3%Ti) samples using a wide range of angles (2 θ) from 5 to 100° with a step of 0,02° and a holding time of 0,50 seconds at each step. A diffractometer with a graphite monochromator and a constant divergence slit (*D*) of 1mm was used. The current and the voltage of the X-ray tube during the analysis were 30mA and 40kV, respectively. The width of the receiving slit (*R*) was 0,1mm, corresponding to fine focused X-ray tubes. The radiation was Cu K α_1/α_2 , doublet ($\lambda \alpha_1 = 0,154178$ nm and $\lambda \alpha_2 = 0,154438$ nm).

Hardness of tested AlCu15Mg5 alloy (0%Ti and 0.3%Ti) was measured by Brinell method. Compression tests were performed using a universal electronic tensile/compression testing machine of $1x10^4$ kg. The initial dimensions of samples were as follows: diameter $D_o=10$ mm and height $H_o=20$ mm. The samples were tested until the fracture occurred.

Results and Discussion

Differential scanning calorimetry analysis. DSC traces obtained at the heating rate of 10°C/min for the investigated alloys are shown in Fig. 1. Thermograms obtained for high-copper AlCu15Mg5 alloys can be briefly described as follows: a) an "intermediate temperature" section between 500°C and 550°C, where only endothermal effect can be detected, and b) a "high temperature" section between 550°C and 700°C where a broad endothermic effect is observed.



Fig. 1. DSC traces for the alloys AlCu15Mg5 (0%Ti) and AlCu15Mg5 (with 0.3%Ti) at 10°C/min.

DSC thermograms in Fig. 1 have shown that the first endothermal peak occurs at a temperature of around 525°C. This peak refers to a phase transition with the value of the enthalpy of -59.74J/g for alloy AlCu15Mg5 (0%Ti), and -75.75 J/g for alloy AlCu15Mg5 (0.3%Ti), as it is given in Table 2.

Alloy	I P	EAK	II PEAK		
	T, °C	-ΔH, J/g	T, °C	-ΔH, J/g	
AlCu15Mg5 (0%Ti)	523.1	59.74	596.0	44.06	
AlCu15Mg5 (0.3%Ti)	526.9	75.75	578.7	28.28	

 Table 2. The value of enthalpy for tested Al-Cu15-Mg5 alloys

These results have shown that the value of enthalpy, for the first detectable endothermal effect at around 525°C, was increased when the titanium content was increased. It was supposed that the first peak is due to the localized melting, probably a ternary eutectic, with the formation of a melt rich in copper and magnesium in presence of the α -solid solution and the compounds Al₂Cu and Al₂CuMg [3]. In DSC thermograms in Fig. 1 the second detectable thermal effect is a high temperature endothermal peak occurs at around 596°C, for the alloy without Ti, and 578°C, for the alloy with Ti. This peak refers to a phase transition with the reaction enthalpy of -44.06 J/g for alloy AlCu15Mg5 (0%Ti) and -28.28 J/g for alloy AlCu15Mg5 (0.3%Ti). From these results it can be concluded that the second peak is due to the localized melting of the binary eutectic. The results have shown that the value of enthalpy for the second endothermic peak was decreased when the content of titanium was increased (see Table 2). Also the temperature of the second peak is decreased from 596.0°C for the alloy AlCu15Mg5 (0.3%Ti), as it is shown in Table 2.

X-ray analysis. Using X-ray powder diffraction we have found that in tested AlCu15Mg5 alloys the tetragonal intermetallic compound Al_2Cu and orthorhombic intermetallic compound Al_2CuMg were formed. Using the X-ray diffractograms it was possible to calculate the average sub-grain size for both alloys (Table 3).

Alloy	Average sub-grain size, nm		
AlCu15Mg5 (0%Ti)	491		
AlCu15Mg5 (0.3%Ti)	496		

Table 3. Average sub-grain size in the crystallographic direction [112] for different titanium contents in Al-Cu15-Mg5 alloy

The sub-grain size is in the range of the lattice of the crystal grain from which the X-rays are coherently diffracted. The sub-grains are separated by dislocation walls and have a space orientation which is different by several angle minutes. Using X-ray diffraction of polycrystals, the sub-grain is defined as a range of quantitative values, starting from the average length in a definite crystallographic direction, through the average volume, to their dimensional distributions. In alloys containing high titanium the average sub-grain size in the crystallographic direction [112] were found to increase [2].

It is well known that in the binary aluminium-copper system, the aluminium-rich terminal solid solution is in equilibrium with the intermetallic compound , which has approximately the formula CuAl2, although some solid solubility exists. The addition of magnesium allows the formation of more intermetallic compounds, such as CuMgAl2, CuMg4Al6, CuMgAl and Cu6Mg2Al5 [2]. The AlCu15Mg5 alloy used in this study, containing 0,3 wt.%Ti, has typical cellular-dendritic structures. The second phase can be an intermetallic compounds that do not necessarily contain aluminium (Mg2Cu or MgCu2); or an alloying element, such as copper or magnesium, depending on the composition of the alloy. Cast AlCuMg alloys contain soluble phases: Al2Cu or Al2CuMg which appear in various amounts and at various locations in the microstructure, depending on the thermal history of the specimen.

Mechanical properties. The values of Brinell hardness and compression strength are given in Table 4. The changes in chemical composition of the alloy cause changes in the structure and these changes are reflected in the Brinell hardness and the compression strength. By increasing the content of titanium the hardness and compression strength of Al-Cu15-Mg5 alloy were increased.

Alloy	HB _(av)	$R_{0,2p}$ (MPa)	R_{m} (MPa)
AlCu15Mg5 (0%Ti)	161,2	372,8	705,5
AlCu15Mg5 (0.3%Ti)	167,0	379,9	712,2

Table 4. Hardness and compression strength of Al-Cu15-Mg5 alloy (0%Ti and 0.3%Ti)

Summary

Different experimental procedures performed on Al-Cu15-Mg5 alloys enable us to explain the influence of titanium content on the mode of formation and the structure in that system. From the results obtained in this study the following can be concluded:

- DSC analysis of AlCu15Mg5 alloys has shown that the first endothermal peak occurs due to the localized melting, probably a ternary eutectic, producing a high copper and magnesium melt in presence of α -solid solution and the intermetallic compounds Al₂Cu and Al₂CuMg. Increasing of the titanium content in the AlCu15Mg5 alloy the value of enthalpy for the first peak is increased. The second endothermal peak occurs due to the local melting of the binary eutectic. The value of enthalpy for the second endothermic peak was decreased when the content of titanium was increased.

- Using X-ray diffraction it was established that the tetragonal intermetallic compound Al_2Cu and orthorhombic intermetallic compound Al_2CuMg are formed for both tested AlCu15Mg5 alloys.

- Compression strength and hardness of the tested Al-Cu15-Mg5 alloy increase with an increase of titanium content.

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