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COLOR ETCHING OF AZ91/SiC COMPOSITE

Abstract

Metal matrix composites, especially those based on magnesium alloys attracts growing interest because of wide application in industry. Silicon carbide particles may become the heterogeneous nucleation sites. That is why mechanical properties in composites improves not only because of the reinforcement particles presence but also because of microstructure refraining effect.

The aim of this paper was to choose the suitable etching solution for AZ91/SiC composites and to measure the mean grain size of few specimens of different composites. Authors of this article described the way of composites preparation and procedure of samples grinding and polishing. The etching procedure with suitable etchant was described. Presented microstructure photographs confirm that the chosen etching solution and specimens preparation procedure were suitable for this composite.

Keywords: *microstructure, color etching, AZ91, magnesium based composites, grain size*.

1. INTRODUCTION

The world industry is facing increasing challenges to reduce transportation impact on environment and to achieve low fuel consumption without sacrificing vehicles or other devices performance. In addition to a wide range of novel vehicle propulsion and power-saving technologies under development, light-weighting of elements is required to face these challenges [1, 2]. Magnesium alloys and their composites, because of their low density are fulfilling request for low specific weight materials. It may be an answer for novel market

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demands to industry plants. Grain size is one of the most important structural characteristic that determining mechanical properties. Knowing element properties the proper application regions for it can be chosen to achieve best mechanical properties and performance. Nowadays we can use simulation software to predict the element microstructure. For those programs very important is to have experimental data as a base for the nucleation and crystals growth model. Experimental data after applying statistical methods let us find approximated values of the so-called “fitting parameters” in the mentioned models [3].

During the grain measure the specimen must be cut. Due to dendritic microstructure of AZ91 on the polished section surface after etching we can observe grain boundaries, but the observed regions can be the arms of one dendrite. The colour etching can be answer to this problem. The grains with the same crystals directions will get the same colour, so we can add their surfaces and obtain total surface of cut grain. Having the surface we can calculate the mean radius of the grain. So the crucial problems during grain radius measurement are to choose suitable etchant and measure surface each of coloured regions. The goal of this work was to solve this problems for AZ91/SiC composite specimens.

2. METHODS OF COMPOSITE SPECIMENS PREPARATION

2.1 Composite casting

Three castings (A, B, C as detailed below) were performed for different composites. The casting procedure for all attempts were very similar (Table 2). There were used about 6000 g of AZ91(Table 1) and SiC(different weight percentage) particles of different sizes: casting A – 10 µm, casting B – 40 µm, casting C – 76 µm.

Table 1. Chemical composition of AZ91 alloy, wt. % determined using ICP-OES VARIAN Vista MPX

Al	Zn	Mn	Fe	Be	Si	Cu	Ni
8.5	0.64	0.23	<0.002	10 ppm	0.03	0.003	0.001

The SiC particles were pre-heated before inserting them into the liquid alloy. The samples were casted in standard thermoanalysis cups (Fig. 1). For each casting composite specimens were prepared:

- casting A specimens with: 0, 0.1, 0.5, 1, 2, 3.5 wt.% of SiC;
- casting B specimens with: 0, 0.5, 1, 2, 3.5 wt. % of SiC;
- casting C specimens with: 0, 0.5, 1, 2, 3.5 wt % of SiC.

Figure 1. Standard thermoanalysis croning sand cup with K type thermocouple

Croning sand cups with K type thermocouple were used to measure the temperature and thus determine the undercooling. From these castings 14 specimens were prepared. Specimens were taken from the region near to the thermocouple.

The specimens were then cut, ground and polished with the grinding machine. The water were used as lubricant for p80, p240, p320 SiC papers and for p600, p800, p1200 SiC papers ethanol was used. Automatic grinding parameters were set to pressure 2.3 bar, 150 rpm speed and 5 minutes time for all samples. The polishing procedure consisted of two stages. In the first stage 3μm diamond suspension was used, the automatic setup was set to the pressure 3.5 bar, 100 rpm speed, samples were polished for 4 minutes. In second stage OPS was used samples were polished non-automatically for 30 s at 120 rpm speed.

Table 2. Conditions comparison for castings made in Chair of Casting Research, Leoben

Casting conditions			
Casting symbol	**A**	**B**	**C**
Particles size, [μm]	10	40	76
AZ91 mass, [g]	5960	6250	5800
Ambient temperature, [°C]	22	22	24
Furnace temperature, [°C]	750	750	750
Particles temperature, [°C]	320	320	320
In-mould temperature, [°C]	100	100	100
Stirring time, [s]	240	180	180

2.2 Colour etching

To produce an etching solute we the procedure described by Maltais et al [4, 5, 6] was used. The chemical composition of the solute was [4]:

50 ml Destilled Water,
150 ml Ethanol,
1 ml Acetic Acid.

Described in [3,4] etching procedure was not very precised in details and the material was the AZ91 alloy. The improvements applied by authors to this method was found to be the best for AZ91/SiC composites. Specimens were immersed in the etching solution and gently agitated (by slowly moving around) for 80 – 95 s. They were then rinsed in ethyl alcohol (small amount). Then they were put dried from about 50 cm with a blast of room-temperature air. During drying stage the specimens were gently moved so that the remaining liquid still wet their entire surface.

Table 3. Measured mean grain diameters for AZ91/SiC composite specimens (in μm)

SiC, wt. %	Particles size	10 μm	40 μm	76 μm
0		601.4	601.4	601.4
0.1		237.3	N/A	N/A
0.5		189.1	388.7	405.6
1		128.5	210.3	378
2		89.3	190.1	203.8
3.5		76.4	113	115

3. RESULTS

The etched specimens were examined using a light optical microscope Carl Zeiss AXIO Imager.A1 with cross polarized light and λ filter. The images on computer display revels arms of different dendritic grains as areas with different colours, see Fig. 2. The grain boundaries were marked semi-automatically and manually using the NIS-Elements 3.0 software. The measured grain sizes are gathered in Table 3. It can be observed that the grain size depends on both the weight percentage and size of SiC particles. If there is more particles the grains are smaller. Also the particles size has influence on the microstructure. When the particles of the same mass were used, the grains were smaller for smaller ceramic particles.

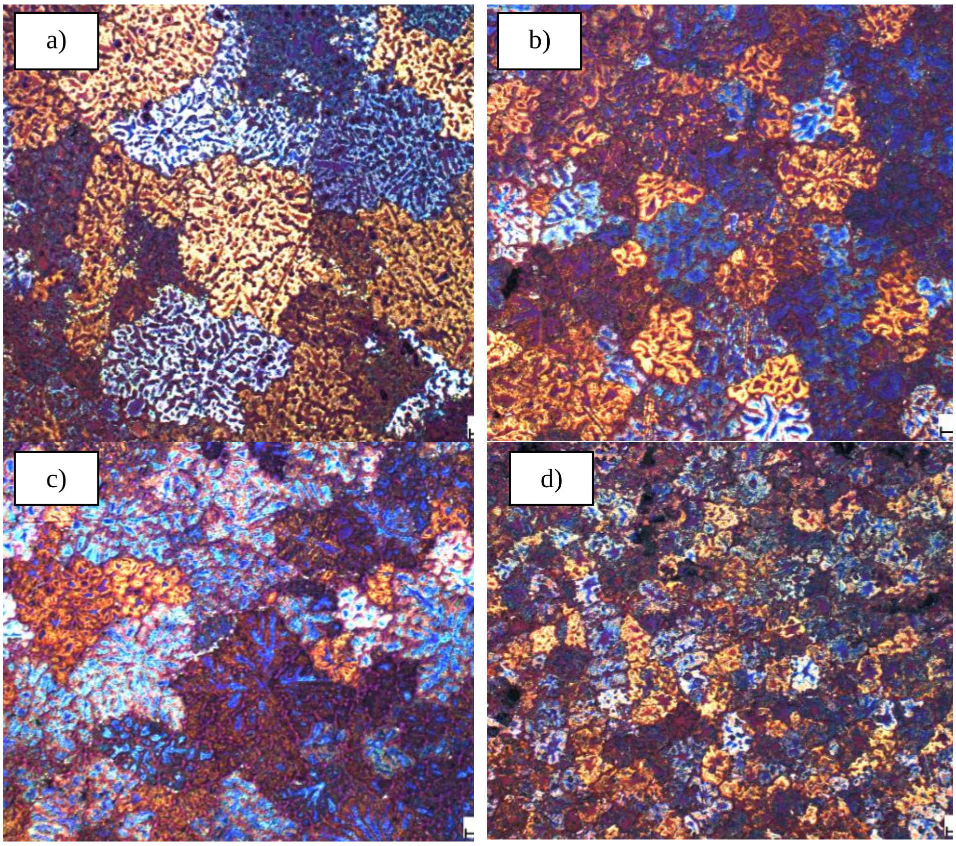

Figure 2. Optical micrographs of the etched samples viewed under cross polarized light using λ filter for different AZ91/SiC composites (Examples of the etching effect):
a) 0% of SiC; b) 2% of SiC, particles mean diameter 40 μm; c) 0.1% of SiC, 10 μm; d) 3.5% of SiC, 76 μm.

Conclusions

Ethanol and Acetic Acid based solute is suitable for colour etching of AZ91/SiC composite.

The particles content in composite has positive impact on composite microstructure.

Smaller particles produce better composite microstructure.

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References

[1] – **Luo A:** Heterogeneous *nucleation and grain refinement in cast Mg(AZ91)/SiCp metal matrix composites.* Canadian Metallurgical Quaretly, 35, 4, pp.375-383 (1996).

[2] **– Lu L, Dahle AK, StJohn DH:** *Heterogeneous nucleation of Mg-Al alloys.* Scripta Materiala, **54**, pp.2197-2201 (2006).

[3] – **Lelito J, Żak P, Suchy JS:** *The grain nucleation rate of the AZ91/SiC composite based on Maxwell-Hellawell model*, Archives of Metalurgy and Materials, 2009; Vol. 54(2), pp.347-350

[4] – **Maltais A, Dube D, Fiset M, Laroche G, Turgeon S:** *Improvements in the metallography of as-cast AZ91 alloy.* Material Characterization, 52, pp. 103-119 (2004).

[5] - **Maltais A, Fiset M, Dube D:** *Color etching of Mg-Al alloys.* Microsc Microanal **11**(2)**,** pp. 694-695 (2005).

[6] - **Maltais A, Dube D, Roy F, Fiset M:** *Optical anisotropy of a color-etched AZ91 magnesium alloy.* Materials Characterization, **54,** pp. 315– 326 (2005).