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Effect of Inoculation on the Mechanical Properties of AZ91

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Abstract

The effect of Ca element on the microstructure evolution of the AZ91 magnesium alloy was investigated in this research. The magnesium-aluminium alloy AZ91 was inoculated with the Emgesal® Flux 5 to refine its microstructure and also improve its microstructure. Six different concentrations of the Emgesal® Flux 5 content were tested, ranging from 0.1 to 0.6% wt., and compared to the baseline of the AZ91 alloy without inoculation. Melted metal was poured into a preheated metallic mould. Samples to test were achieved after turning treatment. Formed microstructure was assessed using an optical microscope. The microstructure was refined for every tested samples. Mechanical properties such as tensile strength, elongation, Brinell hardness, Vickers microhardness, abrasion resistance and adhesive resistance were tested on the inoculated samples and compared to the non-inoculated AZ91. Introducing an Emgesal®Flux 5 inoculant caused a change in the tensile strength, elongation, Brinell hard-ness, Vickers microhardness, abrasive wear resistance as well as adhesive wear resistance in each examined concentration.

Keywords: Metallography, Solidification process, Magnesium alloys, Emgesal® Flux 5, DTA process

1. Introduction

For a few decades now, we have been observing an increase in the application of magnesium alloys in the automotive, aircraft, electronic and aerospace industry [1]. This is especially important now that the fuel prices are historically high and each reduction of mass entails decreased operating costs [2-3]. The more and more widespread application of magnesium alloys results from their low density coefficient with respect to their quite high mechanical properties.

An improvement of the mechanical properties of magnesium alloys as a result of obtaining a more refined microstructure of the cast can be achieved through the use of pressure casting and thixotropic casting methods as well as by means of alloying additions and intensive cooling [4-6]. Another way of microstructure refinement and thus also the mechanical properties is the application of inoculants [7-10].

For many years, both science and industry have been more and more frequently using Ca as an alloy addition, which, alone or together with other compounds, refines the microstructure as well as improves the mechanical properties of magnesium alloys. Jun H. et al. [11] examined that a simultaneous introduction of Ca and Y positively affects the creep resistance and tensile strength. Zhu G. et. al. [12] verified that a Ca addition in the amount of 0,47% significantly reduces the energy needed to exceed the yield point, which increases the workability of magnesium alloy casts. Wan Y. et. al. [13] and Yin P. et al. [14] demonstrated that magnesium alloys containing Ca exhibit good mechanical properties, high corrosion resistance and can be used in medicine owing to their high biocompatibility with the human body. In the study [14], it was observed that too high contents of Ca increase the brittleness of magnesium alloys. The works [14,15] prove that calcium has a tendency to form intermetallic precipitation $Ca_2Mg_6Zn_3/Mg_2Ca$ on the grain boundaries of the particular phases. The authors of the studies [13-15] show that good strength parameters and corrosion resistance values are obtained with the Ca concentrations not exceeding (1 – 1,5) %.

The aim of the study was to examine the effect of the Emgesal® Flux 5 concentration on the tensile strength, Brinell hardness, Vickers microhardness, abrasion resistance and adhesive resistance of AZ91 alloy casts obtained in metallic moulds.

2. Experimental

Within the investigations, 7 AZ91-based melts were prepared, of which 6 underwent inoculation with various concentrations of the Emgesal® Flux 5 substance. Emgesal® Flux 5 is a commercial product of Rheinkalk HDW GmbH & Co KG, the Lhoist Group. The compound, used in the refinement and modification of magnesium alloys, contains 5% CaF_2. It characterizes in density of 2.22 g/cm^3 and solidifies at 384 °C [16]. The schedule of the performed melts have been presented in Table 1.

Table 1.
Melt schedule

Melt number	Melt's chemical composition
W1	AZ91
W2	AZ91+0.1% (5%CaF_2)
W3	AZ91+0.2% (5%CaF_2)
W4	AZ91+0.3% (5%CaF_2)
W5	AZ91+0.4% (5%CaF_2)
W6	AZ91+0.5% (5%CaF_2)
W7	AZ91+0.6% (5%CaF_2)

Alloy AZ91 was elected for the tests. Its chemical composition has been given in Table 2.

Table 2.
Chemical composition of alloy AZ91 (W1)

Chemical composition, % wt.					
Mg	Al	Zn	Mn	Ca	Si
90.6	8.69	0.424	0.248	0.0011	0.0225

During each melt, the alloy was heated to 740 °C ±5 °C in a resistance furnace made of S235JRG2 steel [17] and mixed by means of a stirrer. In order to prevent oxidation, a gas mixture of argon and SF_6 was used.

The melts were poured into metal moulds preheated to 230±5 °C. The casts were cooled to room temperature. The ready casts were then subjected to mechanical treatment so that they would obtain the shape of strength test samples in accordance with the standard [18]. Additionally, samples for Brinell hardness tests were also prepared according to the standard [19], with the indenter diameter d = 2,5 mm and the applied force F = 490 N. The Vickers microhardness was also examined according to the standard [20], with applied the force of 300 N.

The abrasive and adhesive wear tests were carried out with the use of a device for tribological tests of the "pin on disc" type, whose schematics have been described in [21]. The samples were loaded with the force F = 5 N and the sample surface S = 113.097 mm^2. After these data were substituted into formula (1), the unit normal stresses were calculated σ = 0.045 MPa:

$$\sigma = \frac{F}{S} \tag{1}$$

The wear examinations were conducted with the following parameters:

- sample diameter 12 mm,
- counterspecimen rotational speed ω = 75 rot/min,
- test duration 4 h (measurement every 15 minutes).

The abrasive wear test was performed with the use of abrasive paper, grain gradation P240, whereas the adhesive wear test was conducted with the use of a counterspecimen made of aluminium, whose surface was earlier subjected to facing on a turning lathe. The masses were examined by means of a laboratorial scales Sartorius type L420P with the measurement accuracy of 0.001 g, whose measurement error equals ± 0,001 g. Each time before the mass measurement, the samples were cleaned with ethyl alcohol.

3. Results and discussion

3.1. Chemical composition

Table 3 provides a compilation of the results of the chemical composition analysis performed on the examined samples. The chemical composition of all the trials is in accordance with the standard PN-EN 1753:2001 [22]

Table 3.
Chemical composition of the analysed alloys

	Chemical composition, % wt.				
Melt no.	**Mg**	**Al**	**Zn**	**Mn**	**Ca**
W2	90.6	8.51	0.443	0.199	0.0048
W3	89.8	9.25	0.487	0.238	0.0022
W4	90.4	8.72	0.437	0.233	0.0040
W5	90.0	9.20	0.482	0.238	0.0030
W6	90.1	9.22	0.449	0.236	0.0019
W7	89.7	9.42	0.507	0.159	0.0025

3.2. Microstructure

Figure 1 presents the microstructure of a non-inoculated alloy AZ91 as well as the microstructure of alloy AZ91 inoculated with Emgesal® Flux 5 in the concentration of 0.4%. A comparison of the microstructures of the examined alloys shows grain refinement of primary phase α_{Mg} and eutectic $\alpha_{Mg}+\gamma(Mg_{17}Al_{12})$.

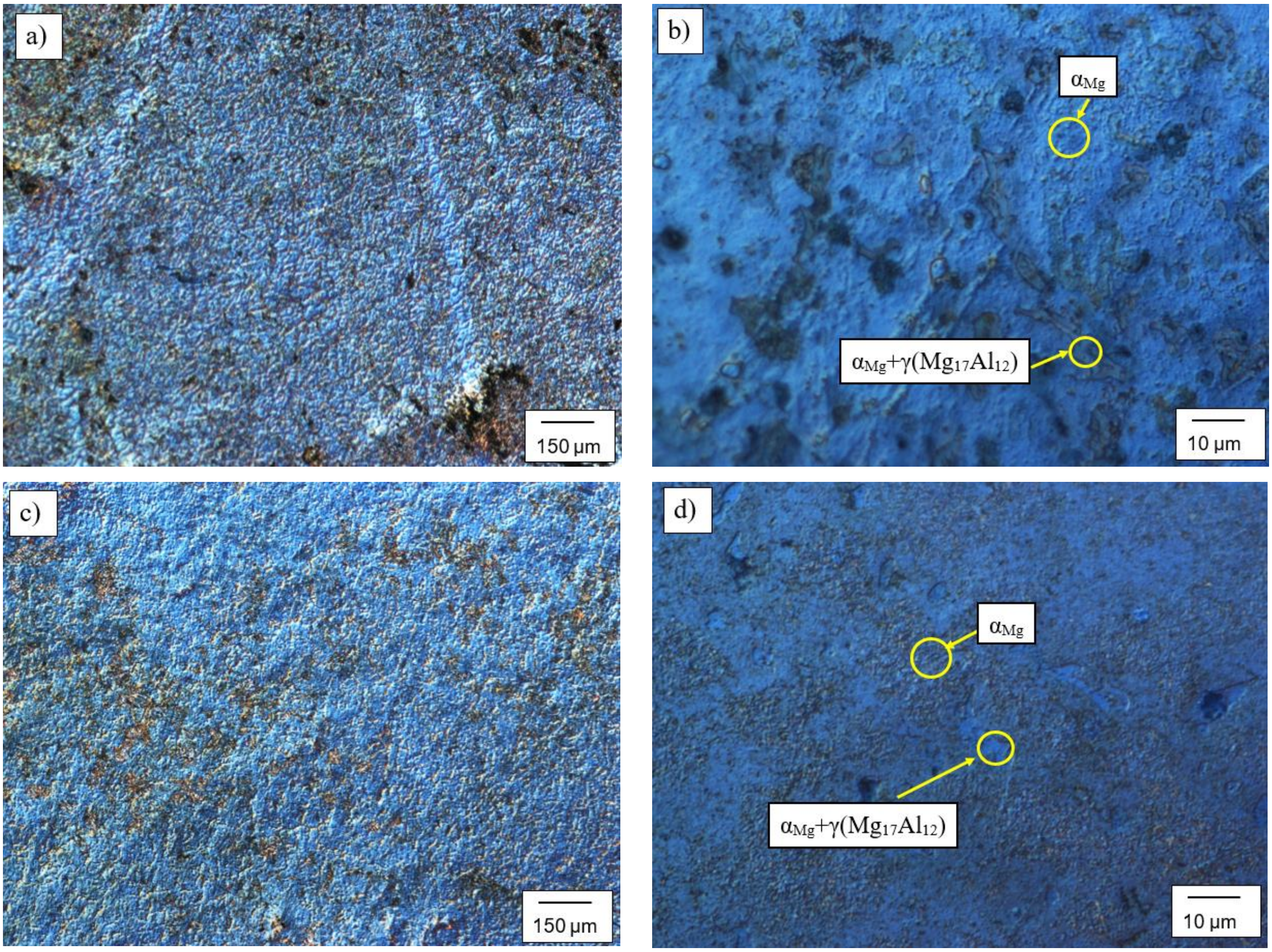

Fig. 1. Microstructure of the non-inoculated alloy AZ91 (a, b) and alloy AZ91+0.4%(5%CaF_2) (c, d)

3.3. Tensile strength

Figure 2 shows a diagram with the average ultimate tensile strength results (UTS) for the tested alloys. We can observe that, for the concentration of 0.1% (W2), the tensile strength value was close to the UTS value of alloy AZ91 without an inoculant (W1). In the case of concentrations of 0.2% and 0.6% (W3 – W7), the tensile strength of the investigated alloys was higher compared to the sample without inoculation. The highest value, obtained for the inoculant concentration of 0.6% (W7), equalled 125.8 MPa

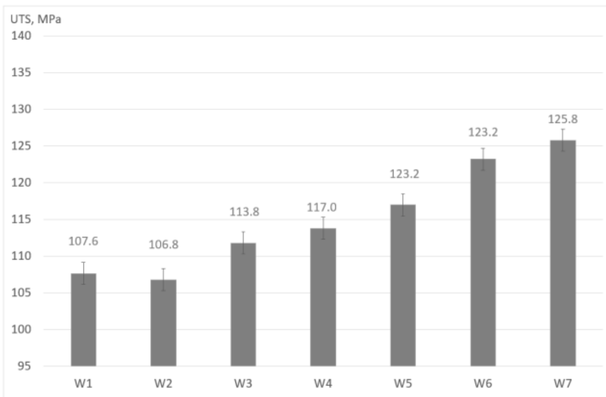

Fig. 2 Ultimate tensile strength at yield of analysed alloys

3.4. Elongation

Figure 3 presents a diagram with the average results of elongation A expressed in %. The diagram shows that the elongation for the inoculant concentration of 0.1% (W2) was close to the elongation of the non-inoculated alloy AZ91 (W1). For the remaining inoculant concentrations, the elongation value was higher than that of the base alloy. The highest elongation equalling 1.28% was obtained for the alloy which had been subjected to inoculation with Emgesal Flux 5 in the concentration of 0.6% (W7).

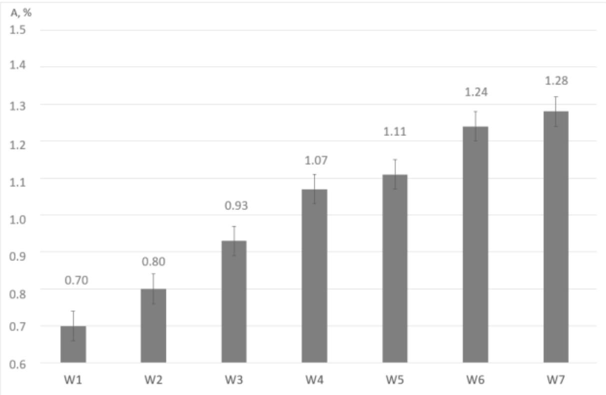

Fig. 3. Elongation of the examined samples

3.5. Brinell hardness

Figure 4 shows a diagram with the results of averages hardness measurements performed by the Brinell method. The diagram shows that, for the inoculant concentration of 0.1% (W2), the hardness HB reaches lower values than the non-inoculated AZ91 (W1). In the case of the Emgesal® Flux 5 concentration of 0.2% (W3) and 0.3% (W4), the hardness HB of the sample reaches a similar value to that of the base sample. For the inoculant concentrations of 0.4% (W5), 0.5% (W6) and 0.6% (W7), the hardness HB achieves higher values than the hardness of the AZ91 alloy not subjected to inoculation. The highest hardness equalling 69 HB was recorded for the sample enriched with 0.6% of Emgesal® Flux 5 (W7).

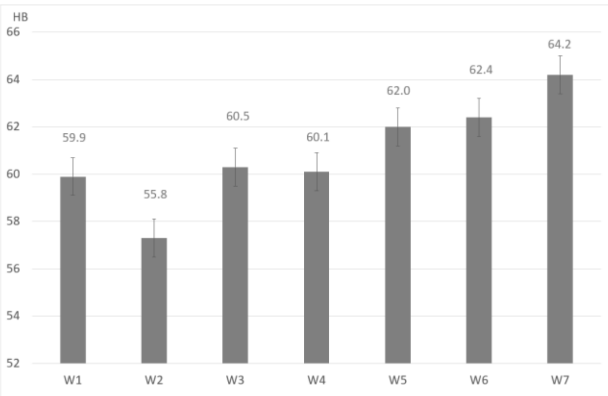

Fig.4. Brinell hardness of the tested alloy

3.6. Vickers microhardness

Figure 5 presents the averages results of the Vickers microhardness measurements of preliminary phase α_{Mg} (HV0.3). Based on the obtained results, it was stated that, for the inoculant concentration of 0.1% (W2), a slight drop of microhardness HV occurred, compared to the non-inoculated sample (W1). For the remaining concentrations, the microhardness HV increased, reaching the highest value of 61.4 HV for the inoculant concentration equalling 0.6% (W7), but for sample W5 microhardness HV was a little bit lower – 61.1 HV.

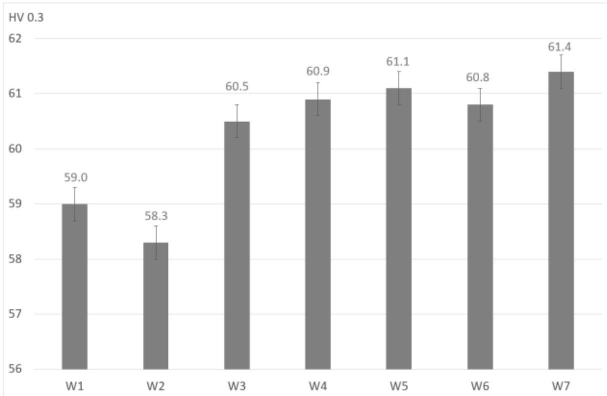

Fig. 5. Vickers microhardness HV0.3 for the examined alloys

3.7. Adhesive resistance

Figure 6 shows the total loss of mass U_{m1} resulting from the performed adhesive resistance test of the examined alloys. The diagram shows that the mass loss for the alloys with the inoculant in the concentration of 0.1% (W1) and 0.5% (W6) was close to the mass loss for the AZ91 alloy without an inoculant (W1). For the concentration of 0.2% (W3), the mass loss was identical to the case of the base alloy. For the sample with the inoculant concentration equalling 0.3% (W4), the mass loss was higher and equalled 0.035 g. In turn, for the samples in which the inoculant concentration was 0.4% (W5) and 0.6% (W7), the mass loss was lower than that of the sample made of the non-inoculated AZ91. The lowest mass loss equalling 0.034 g was recorded for the alloy with the inoculant content of 0.6% (W7).

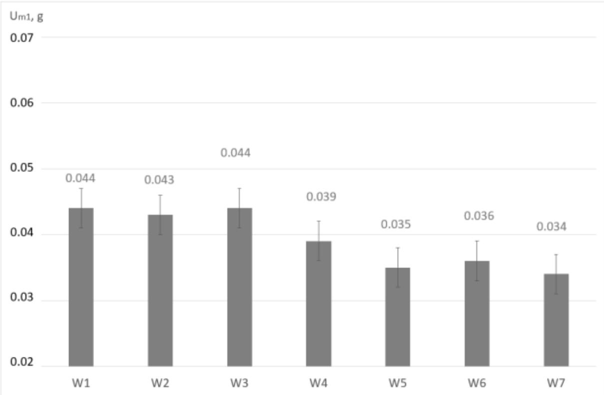

Fig. 6. Total mass loss U_{m1} occurring during the adhesive wear test

3.8. Abrasive resistance

Figure 7 presents the total mass loss U_{m2} resulting from the performed abrasive resistance test of the examined alloys. The diagram shows that the mass loss for the alloys with the inoculant concentration equalling from 0.2% (W3) to 0.6% (W7) was lower than the mass loss of the non-inoculated alloy AZ91. The lowest mass loss equaling 0.496 g was recorded for the sample containing 0.6% of inoculant (W7). For the samples in which the inoculant concentration was 0.1% (W2), the mass loss was higher than that of the non-inoculated alloy AZ91. The highest mass loss equalling 0.629 g was recorded in the case of the sample with the inoculant concentration of 0.1% (W2).

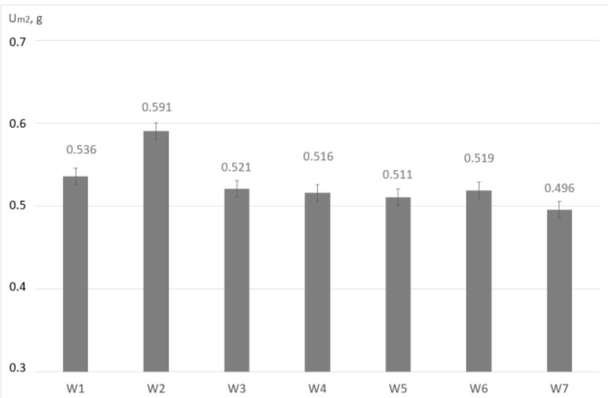

Fig. 7. Total mass loss U_{m2} occurring during the abrasive wear test

4. Conclusions

The presented results are one of the elements of studies aiming at obtaining the possibly most inoculated microstructure, which will contribute to the improvement of the strength parameters, hardness as well as abrasive and adhesive wear. Achieving the improvement of the mentioned properties without a significant change in the chemical composition of the alloy. The analysis of the obtained results made it possible to draw the following conclusions:

1. Introducing an Emgesal® Flux 5 inoculant caused a change in the tensile strength, elongation, Brinell hardness, Vickers microhardness, abrasive wear resistance as well as adhesive wear resistance in each examined concentration.
2. The highest tensile strength value equalling 125.8 MPa was obtained for the sample which contained 0.6% of the inoculant (W7).
3. The highest Brinell hardness equalling 64.2 HB was achieved for the sample with a 0.6% concentration of the inoculant (W7).
4. The highest Vickers microhardness equalling 61.4 HV 0.3 was reached for the sample containing 0.6% of the inoculant (W7). However, for the sample with the inoculant concentration of 0.4% (W5), the Vickers microhardness was not much lower and equalled 61.1 HV 0.3.
5. The highest adhesive wear resistance was observed in the samples containing the inoculant in the concentrations of 0.4% (W5) and 0.6% (W7).
6. The highest abrasive wear resistance was recorded in the samples containing 0.4% (W5) and 0.6% (W7) of the inoculant.
7. The biggest improvement of the mechanical properties was observed for the sample with the inoculant concentration of 0.6% (W7). However, for the sample containing 0.4% (W5) of the inoculant, the improvement was not much lower, and so, the concentration of 0.4% (W5) is considered as optimal.
8. Emgesal® Flux 5 as a inoculant improve tensile strength, elongation, Brinell hardness, Vickers microhardness, abrasive wear resistance as well as adhesive wear resistance as a result of fragmentation of microstructure of the samples.

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