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# THE INFLUENCE OF REMELTING PARAMETERS ON Mg ALLOY/GLASSY CARBON SUSPENSION STABILITY

The homogenous distribution of reinforcing phases in a liquid metal matrix determines the final properties of composite elements fabricated by the casting of a suspension. Ensuring not only a proper temperature and stirring conditions during suspension preparation, but also a relatively short time between the end of mixing and the start of suspension pouring into moulds is critical. In the article, investigations focused on the migration of glassy carbon particles (GCp) in two melted magnesium alloys, Mg3Al and Mg2ZnZrRE, were presented. The composites previously obtained by the stir casting method were remelted at the temperatures of 610, 640, 690 and 730°C (time 30, 60 and 90 min) and finally cooled at a temperature of  $20^{\circ}$ C. The particles segregation on the macro scale was analyzed on a longitudinal section of the obtained samples. It was revealed that both types of suspensions were stable at 610 and 640°C but at 690 and 730°C, a loss of stability was observed. In spite of the slightly less density of the glassy carbon than the applied magnesium alloys, the type of segregation was different and depended on the alloy chemical composition. In the suspension of Mg3Al-GCp, particle migration to the crucible top was observed only, while in the Mg2ZnZrRE-GCp suspension, two zones with a high particle concentration were formed, separated by a zone of pure metal.

Keywords: magnesium matrix composite, glassy carbon, stir casting, die casting

# WPŁYW PARAMETRÓW PRZETOPU NA STABILNOŚĆ SUSPENSJI STOP Mg/WĘGIEL SZKLISTY

W procesach odlewania kompozytów z zawiesin jednorodność rozmieszczenia fazy zbrojącej w metalowej osnowie decyduje o powtarzalności właściwości wytwarzanych wyrobów. Wymusza to zapewnienie w procesie technologicznym nie tylko odpowiedniej temperatury suspensji i warunków jej mieszania, ale również kontroli czasu pomiędzy zaprzestaniem mieszania a odlaniem wyrobu. W artykule przedstawiono wyniki badań segregacji cząstek węgla szklistego(GCp) w dwóch stopach magnezu Mg3Al i Mg2ZnZrRE. Eksperyment przeprowadzony w warunkach laboratoryjnych obejmował przetop kompozytów uzyskanych wcześniej w warunkach przemysłowych metodą mechanicznego mieszania komponentów i odlewania grawitacyjnego suspensji do form stalowych. Próbki wygrzewano w temperaturze 610, 640, 690 i 730°C w czasie 30, 60 i 90 minut i schłodzono w temperaturze 20°C. Analizowano makrostrukturę przekrojów wzdłużnych pod względem segregacji cząstek. Wykazano stabilność obu suspensji w temperaturze 610 i 640°C oraz jej utratę w temperaturze 690 i 730°C. Charakter segregacji cząstek był różny i zależał od składu chemicznego stopu, pomimo tego, że w obu typach suspensji gęstość cząstek była nieznacznie mniejsza od gęstości stopów. W suspensji Mg3Al-GCp cząstki migrowały do góry tygla, a w suspensji Mg2ZnZrRE-GCp utworzyły dwie strefy u góry i na dole tygla, które rozdzielone były strefą metalu bez cząstek.

Słowa kluczowe: kompozyt magnezowy, węgiel szklisty, odlewanie suspensji, odlewanie ciśnieniowe

## INTRODUCTION

Stir casting is a well-known method in the processing of aluminium and magnesium matrix composites reinforced with ceramic particles [1-5]. In the stir casting process, reinforcing phases in the form of particles, short fibers or whiskers, connect with a molten metal by means of mechanical stirring and a liquid metal-solid reinforcement suspension is obtained. A suspension with proper distribution of the reinforcing phases and viscosity can be cast by gravity or pressure die casting methods. The time between finishing suspension mixing in a protective atmosphere and the start of its pouring into moulds pays a very important role in the technological procedure because of the possibility of solid component segregation. The character of segregation is determined by the density of the components but its intensity depends on the temperature and time as well. That segregation may start in the crucible of a melting furnace and then in a ladle, before pouring the suspension into the moulds, which results in the manufacturing of products with different microstructure and properties. When we apply pressure die casting as the a process for composite products fabrication, an additional remelting procedure of the composite ingots is necessary and its parameters must be specified. However, direct and precise control of reinforcing phases migration in technological equipment both in industrial and laboratory conditions is difficult.

In the presented experiments, the effects of glassy carbon particles (GCp) segregation in two types of magnesium alloys, Mg3Al and Mg2ZnZrRE, regarding different time and temperature of the suspension were examined. Composite samples obtained from ingots manufactured in industrial conditions by stir casting were remelted at different parameters, cooled in air, and then their structure was characterized. The applied procedure showed the influence of the magnesium matrix composition, time and temperature on the suspension homogeneity.

# MATERIAL

Composite materials were obtained by stirring glassy carbon particles (GCp) of a granulation of

 $17\div160 \ \mu\text{m}$  [6] with two types of noncommercial magnesium alloys: Mg3Al with 3 wt.% Al and Mg2ZnZrRE alloy with 2 wt.% Zn, 1 wt.% Zr and 1 wt.% rare earth elements (RE). Suspensions of a mass of approximately 35 kg containing an Mg alloy and approx. 10 wt.% GCp were manufactured in an electrical furnace with a proper stirring system, in the protective atmosphere of an Ar and SF<sub>6</sub> gas mixture. Then they were gravity poured into steel moulds.

From the manufactured ingots, samples in the form of rollers with a diameter of 25 mm and height of 30 mm were prepared. The micrographs of the composites used in the experiments are presented in Figures 1 and 2. It is typical that in an Mg3Al-GCp composite, oxides and some phases from the Mg-Al system may accumulate around the glassy carbon particles (Fig. 1b), while in the Mg2ZnZrRE-GCp composite, besides oxides, micro-grains of zirconium are additionally detected (Fig. 2b).



Fig. 1. Micrographs of stir cast Mg3Al-GCp composite, SEM Rys. 1. Mikrofotografie kompozytu Mg3Al-GCp odlanego z suspensji, SEM



Fig. 2. Micrographs of stir cast Mg2ZnZrRE-GCp composite, SEM Rys. 2. Mikrofotografie kompozytu Mg2ZnZrREI-GCp odlanego z suspensji, SEM

# **EXPERIMENTAL PROCEDURE AND RESULTS**

The composite rollers prepared from both types of composites were put in ceramic crucibles and then covered from the top with a layer of a protective powder. Next they were placed in an electrical laboratory furnace without any protective atmosphere and held at the temperatures of 610, 640, 690 and 730°C respectively, for 30, 60 and 90 minutes at each temperature. The remelted suspensions were then cooled with the crucibles in air (Fig. 3a) and after their solidification, the ceramic crucibles and sintered protective powder were mechanically removed from the samples (Fig. 3b). The obtained research materials were cut and maroscopic observations of the longitudinal sections the samples were performed (Figs. 4-11).



Fig. 3. View of composite samples after remelting and cooling: a) in ceramic crucibles, b) after crucible removal

Rys. 3. Widok próbek po przetopieniu i schłodzeniu: a) w tyglach ceramicznych, b) po wyjęciu z tygli

#### Mg3AI-GCp composite

The longitudinal sections of the Mg3Al-GCp composite after remelting are presented in Figures 4-7. For the remelting temperatures of 610 and 640°C (Figs. 4 and 5), evident particle segregation was not observed but an increase in macroscopic pores was visible. An increase in remelting temperature up to 690°C and thereby reducing the metal viscosity, facilitated migration of the GCp to the top of the suspension. Therefore, for the time of 90 minutes, an evident zone of metal without particles was formed on the sample bottom. A further increase in temperature up to 790°C caused an increase in segregation intensity and zones without particles were observed for all the applied times of the remelting process. The generation of macropores was an additional effect occurring in the investigated Mg3Al-GCp suspension and its intensity increased with an increase in remelting time and temperature. The

origin of those pores is not clear. For the lower temperatures, one may explain their presence as a result of the coalescence of micropores formed in the composites during mechanical stirring and gravity casting. However, complete degradation of the suspension with an increase in temperature and time suggests that the reactions in the suspension are the main reason. In general, we can conclude that in the investigated Mg3Al-GCp suspension, only migration of GCp to the top of the crucible may occur, which is consistent with the effects previously observed during preparation of the initial suspension in industrial conditions. That effect can be explained clearly as a result of the slightly lower density of GCp than the magnesium alloy and is consistent with the expectations.



- Fig. 4. Macrostructure of longitudinal section of Mg3Al-GCp composite samples remelted at temperature of 610°C during 30, 60, and 90 minutes
- Rys. 4. Makrostruktura przekroju wzdłużnego próbek kompozytu Mg3Al-GCp przetopionych w temperaturze 610°C w czasie 30, 60 i 90 minut



- Fig. 5. Macrostructure of longitudinal section of Mg3Al-GCp composite samples remelted at temperature of 640°C during 30, 60, and 90 minutes
- Rys. 5. Makrostruktura przekroju wzdłużnego próbek kompozytu MgAl-GCp przetopionych w temperaturze 640°C w czasie 30, 60 i 90 minut



Fig. 6. Macrostructure of longitudinal section of Mg3Al-GCp composite samples remelted at temperature of 690°C during 30, 60, and 90 minutes

Rys. 6. Makrostruktura przekroju wzdłużnego próbek kompozytu Mg3Al-GCp przetopionych w temperaturze 690°C w czasie 30, 60 i 90 minut



- Fig. 7. Macrostructure of longitudinal section of Mg3Al-GCp composite samples remelted at temperature of 730°C during 30, 60, and 90 minutes
- Rys. 7. Makrostruktura przekroju wzdłużnego próbek kompozytu Mg3Al-GCp przetopionych w temperaturze 730°C w czasie 30, 60 i 90 minut

## Mg2ZnZrRE-GC<sub>p</sub> composite

The longitudinal sections of the Mg2ZnZrRE-GCp composite after remelting are presented in Figures 8-11. For the remelting temperatures of 610 and 640°C (Figs. 8 and 9), the macrostructure observations did not reveal any changes in GCp distribution or the presence of macropores. The effect of remelting time on the segregation process was visible for the temperature of 690°C (Fig. 10), where the suspension after 30 minutes was still stable but after 60 and 90 minutes, evident segregation was observed. It was characteristic that three different zones were formed in the remelted samples. Two of them, enriched with glassy carbon particles were located on the top and on the bottom of the sample, and they were separated by a zone of a metal without particles.



- Fig. 8. Macrostructure of longitudinal section of Mg2ZnZrRE-GCp composite sample remelted at temperature of 610°C during 30, 60 and 90 minutes
- Rys. 8. Makrostruktura przekroju wzdłużnego próbki kompozytu Mg2ZnZrRE-GCp przetopionej w temperaturze 610°C w czasie 30, 60 i 90 minut



Fig. 9. Macrostructure of longitudinal section of Mg2ZnZnZrRE-GCp composite sample remelted at temperature of 640°C during 30, 60 and 90 minutes

Rys. 9. Makrostruktura przekroju wzdłużnego próbki kompozytu Mg2ZnZrRE-GCp przetopionej w temperaturze 640°C w czasie 30, 60 i 90 minut



Fig. 10. Macrostructure of longitudinal section of Mg2ZnZrRE-GCp composite sample remelted at temperature of 690°C during 30, 60, and 90 minutes

Rys. 10. Makrostruktura przekroju wzdłużnego próbki kompozytu Mg2ZnZrRE-GCp przetopionej w temperaturze 690°C w czasie 30, 60 i 90 minut

A similar effect was found in the samples remelted at 730°C (Fig. 11) independent of the remelting time. The effect of macropores formation was not registered. It means a different process of suspension homogeneity loss in the Mg2ZnZrRE-GCp system compared with the Mg3Al-GCp system. The reason is the different chemical composition of the applied magnesium alloys. The research by LM (Fig. 12) and SEM with EDS methods of the samples with segregated GCp showed a strong difference in the content of Zr depending on the sample region.



- Fig. 11. Macrostructure of longitudinal section of Mg2ZnZnZrRE-GCp composite sample remelted at temperature of 730°C during 30, 60, and 90 minutes
- Rys. 11. Makrostruktura przekroju wzdłużnego próbki kompozytu Mg2ZnZrRE-GCp przetopionej w temperaturze 730°C w czasie 30, 60 i 90 minut



- Fig. 12. Micrograph of MgZnZrRE-GCp composite sample remelted at 690°C for 30 minutes, metal zone without particles (top) and zone with particles segregated at bottom of crucible (bottom), LM
- Rys. 12. Mikrofotografia próbki kompozytowej MgZrRE-GCp przetopinej w temperaturze 690°C w czasie 30 minut, widoczna strefa metalu bez cząstek (góra) i strefa z cząstkami segregującymi na dnie tygla (dół), LM

An increase in Zr content in the zone located at the bottom and an almost total absence of Zr both at the top of the composite zone and the metallic zone were detected. That effect of Zr segregation on the bottom of the furnace crucible is well known in the metallurgical processes of magnesium alloys with Zr [7] and therefore the application of a special mixing procedure is necessary. It confirms the results obtained for a suspension of GCp and magnesium alloy with Zr.

# SUMMARY

The presented results of investigations on a laboratory scale focused only on one of the technological aspects of Mg-GCp suspension casting, namely conservation of their proper homogeneity after ceasing the stirring procedure. The influence of the remelting parameters on the alloy-particles interface microstructure was not analyzed in this paper. The described effects of suspension disintegration with temperature and time for the Mg alloy-GCp system confirmed GCp movement in the direction of the crucible top which was previously observed directly during preparation of the initial suspension, independent of the magnesium alloy composition. Additionally, it was revealed that the character of homogeneity loss is more complicated when a magnesium alloy with zirconium is applied.

The mains conclusions are as follows:

- The homogeneity of a suspension of an Mg alloy-GCp composite can be lost after ceasing the mixing procedure and it depends on time and the temperature of the suspension. That phenomena must be controlled during composite processing.
- In homogenous Mg3Al-GCp and Mg2ZnZrRE-GCp composite systems, a remelting temperature of 610÷640°C (time 30÷90 min) did not induce a loss of homogeneity.
- For the temperature of 690°C, the suspensions of both the composite systems were stable after 30 minutes but an increase in remelting time

involved particles segregation. A further increase in temperature implicated segregation in the suspensions for all the examined times, and very intensive destruction of the Mg3Al-GCp suspension, as well.

- The segregation effects of glassy carbon particles in magnesium alloys depended on the alloy composition. In the Mg3Al alloy, the particles migrated to the top of the crucible while in the Mg2ZnZrRE alloy, two zones of the alloy with particles were separated by a pure metal zone.

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## REFERENCES

- Rajan T.P.D., Pillai R.M., Pai B.C., Characterization of centrifugal cast functionally gradient aluminum-silicon carbide metal matrix composites, Material Characterization 2010, 61, 923-928.
- [2] Deng K.K., Wu K., Wu Y.W., Nie K.B., Zheng M.Y., Effect of submicron size SiC particulates on microstructure and mechanical properties of AZ91 magnesium matrix composites, Journal of Alloys and Compounds 2010, 504, 542-547.
- [3] Ye H.Z., Liu X.Y., Review of recent studies in magnesium matrix composites, Journal of Materials Science 2004, 39, 6153-6171.
- [4] Braszczyńska K.N., Zyska A., Braszczyński J., Ocena przydatności osnowy z różnych stopów magnezu w kompozycie umacnianym cząstkami SiC, Kompozyty (Composites) 2003, 3, 8, 253-258.
- [5] Olszówka-Myalska A., Myalski J., Hetmańczyk M., Kiełbus A., Technnologiczne uwarunkowania stabilności zawiesiny stop magnezu-cząstki węgla szklistego, Rudy i Metale 2013, 835-839.
- [7] Qian M., Zheng L., Graham D., Settling of undissolved zirconium particles in pure magnesium melts, Journal of Light Metals 2001, 1, 157-165.