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# STRUCTURE OF AZ91 MAGNESIUM MATRIX ALLOY COMPOSITE REINFORCED WITH GRAPHITE PARTICLES

**The results of structure investigations of magnesium matrix composite reinforced with graphite particles have been presented. The AZ91 (Mg - 9 wt. % Al - 1 wt. % Zn - 0.5 wt. % Mn) alloy was used as a matrix. The investigated composite was reinforced with synthetic graphite particles with a maximum diameter of 10 μm. A simple and non-expensive casting method involving mechanical mixing of liquid metal and the introduced ceramic particles under protective atmosphere was used to produce the investigated material. The obtained composite was characterized by uniform distribution of graphite particles within the matrix alloy. Graphite particles were observed both inside α phase dendrites and interdendritical sides. In order to quantitative description of homogeneity of the structure, coefficient of variation of graphite particles number at the surface (fields method) was determined. Coefficient of variation was equal 0.266, testifying uniform distribution of graphite particles. Microstructure of composite was typical for gravity cast AZ91 magnesium alloy (solidified under non-equilibrium condition). α solid solution, α+γ semi-divorced eutectic, discontinuous precipitates of γ phase ($Al_{12}Mg_{17}$) and $Al_8Mn_5$ intermetallic compound was observed in the composite microstructure, as a result of solidification process under non-equilibrium condition.**

Keywords: **magnesium matrix composite, graphite particles, structure**

## STRUKTURA KOMPOZYTU NA OSNOWIE STOPU MAGNEZU AZ91 UMACNIANEGO CZĄSTKAMI GRAFITU

**Przedstawiono wyniki badań struktury kompozytu na osnowie stopu magnezu umacnianego cząstkami grafitu. Jako osnowę zastosowano stop AZ91 (Mg - 9% wag. Al - 1% wag. Zn - 0,5% wag. Mn). Kompozyt umacniany były cząstkami syntetycznego grafitu o maksymalnej średnicy 10 μm. W celu otrzymania badanego kompozytu zastosowano prostą i niedrogą metodę odlewniczą, polegającą na mechanicznym mieszaniu ciekłego metalu wraz z wprowadzonymi cząstkami ceramicznymi w atmosferze ochronnej. Otrzymany kompozyt charakteryzował się jednorodnym rozmieszczeniem cząstek grafitu w stopie osnowy. Cząstki grafitu obserwowane były zarówno wewnątrz dendrytów fazy α, jak i w przestrzeniach międzydendrytycznych. W celu ilościowego opisu jednorodności struktury wyznaczono współczynnik zmienności ilości cząstek grafitu na powierzchni (metoda pól). Współczynnik zmienności wynosił 0,266, świadcząc o jednorodnym rozmieszczeniu cząstek grafitu. Mikrostruktura kompozytu była typowa dla odlewanego grawitacyjnie stopu AZ91 (krzepnącego w warunkach nierównowagowych). W mikrostrukturze kompozytu obserwowano roztwór stały α, częściowo rozsegregowaną eutektykę α+γ, wtórne wydzielenia fazy γ ($Al_{12}Mg_{17}$) oraz związek międzymetaliczny $Al_8Mn_5$ jako rezultat procesu krzepnięcia w warunkach nierównowagowych.**

Słowa kluczowe: **kompozyt magnezowy, cząstki grafitu, struktura**

## INTRODUCTION

Metal matrix composites (MMCs) have undergone a substantial development over the last years due to their promising advanced properties. A number of manufacturing techniques have been developed to produce a ceramic particulate reinforced with metal matrix composites, such as stir casting, mechanical alloying (MA), powder metallurgy (PM), squeeze cast, disintegrated melt deposition (DMD), infiltration, and self-propagating high-temperature synthesis (SHS). Among them, stir casting is the most economical (costs as little as one-third to one-tenth for mass production) as well as easily adopted method [1-8].

Among the various metal matrix alloys magnesium and its alloys with a low density and high stiffness to weight ratio is an excellent matrix material for composites. Magnesium alloys are light metallic structural materials and have a unique combination of properties, which are very attractive in such applications as the automobile, aerospace and electronic industries. Most commercial magnesium alloys are based on Mg-Al system. In magnesium alloys, aluminium constitutes the main alloying element, chiefly because of its low price, availability, low specific gravity, and the advantageous effect on the corrosion and strength properties. The alloy that is most widely used is AZ91 [9-14].

Cast composites based on a magnesium matrix and reinforced with ceramic particles constitute a new group of materials that feature the desired set of properties because of their low density, high specific stiffness and strength, high damping capacity, and good dimensional stability. Recently, different magnesium matrix alloys reinforced mainly by hard SiC particles and also $TiB_2$, TiC, $B_4C$, $Si_3N_4$/SiC particles have been manufactured [15-19]. The paper is focused on the AZ91 magnesium alloy reinforced with graphite particles.

## EXPERIMENTAL DETAILS

The commercial AZ91 magnesium alloy with a chemical composition given in Table 1 was used as a matrix of composite. A synthetic graphite particles with a maximum diameter of 10 μm were used as the reinforcing phase. The composite samples were obtained by means of a simple casting technique, involving mechanical mixing of liquid metal with the introduced particles and subsequent casting in metal moulds under protective atmosphere. Composite was cast into permanent mould.

TABLE 1. **Chemical composition of AZ91 alloy according to ASTM B93-94**

TABELA 1. **Skład chemiczny stopu AZ91 zgodnie z ASTM B93-94**

| Chemical composition, wt. %* | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Alloy | Al | Zn | Mn | Si max | Fe max | Cu max | Ni max | Others each max |
| AZ91 | 8.5÷9.5 | 0.45÷0.9 | 0.17÷0.4 | 0.05 | 0.005 | 0.03 | 0.002 | 0.02 |
| * Mg rest | | | | | | | | |

Microstructure examination was carried out by means of the light microscopes Neophot-32 and Neophot-21 (Carl-Zeiss Jena). A standard metallographic technique was used for sample preparation including wet prepolishing and polishing with different diamond pastes without contact with water. To reveal microstructure, samples were etched in a 1% solution of $HNO_3$ in $C_2H_5OH$ for about 60 seconds.

Quantitative analysis of graphite particles distribution in the composite was performed by fields method. This method allowed to determine the coefficient of variation ($\nu$) of a particles number in a field. At presented analysis a square with a 10 mm side (field $A$ = = 100 mm$^2$) was projected 130 times on micrographs (showing structure for non-etched surfaces) at magnification 80x. The number of graphite particles, N(A), appearing in each filed A was counted. Coefficient of variation ($\nu$) for $N(A)$ parameter was calculated from the equation:

$$\nu = \frac{s[N(A)]}{\overline{N(A)}} \tag{1}$$

where $s[N(A)]$ is a standard deviation of particles number, $\overline{N(A)}$ is an average number of particles in a field $A$. A more uniform microstructure results in lower coefficient of variation.

## RESULTS AND DISCUSSION

Figure 1 shows a typical structure of the composite characterized by very uniform distribution of graphite particles in the AZ91 matrix alloy. Neither the clusters of graphite particles nor any consequences of floating or sedimentation of the reinforced phase, frequently occurring in gravity cast composites, are observed. An impurities (oxides, iron intermetallics, etc.), which are possibly introduced due to an imperfect technological process were not observed either. In order to quantitatively describe the homogeneity of graphite particles distribution in the composite coefficient of variation from equation (1) was determined. Figure 2 presents diagram of distribution of graphite particles number in the AZ91 matrix alloy composite. Parameters of empirical distribution were equal: average number of particles in field $A$, $\overline{N(A)}$ = 16.6 and standard deviation, $s[N(A)]$ = 4.56. Hence, the value of the coefficient of deviation ($\nu$) calculated from equation (1) is equal to 0.266. This result testifies about uniform distribution of graphite particles in the AZ91 matrix alloy.

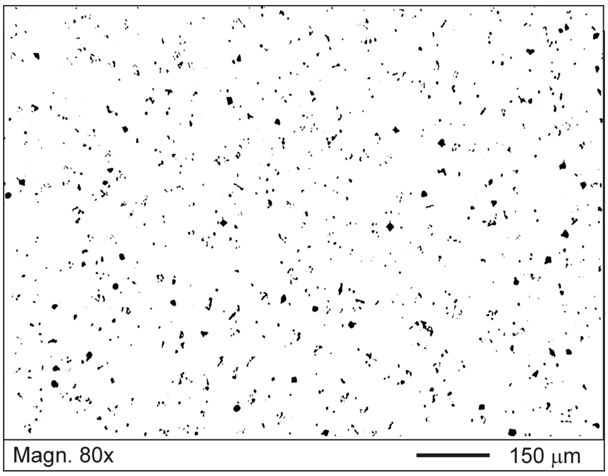


Fig. 1. Structure of the AZ91 matrix alloy composite reinforced with graphite particles (non-etched surface)

Rys. 1. Struktura kompozytu na osnowie stopu AZ91 umacnianego cząstkami grafitu (powierzchnia nietrawiona)

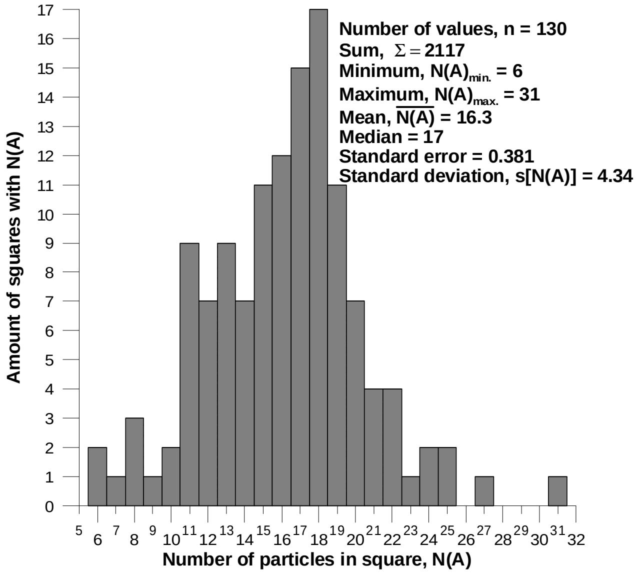


Fig. 2. Distribution of number of graphite particles in AZ91 matrix alloy composite (fields method)

Rys. 2. Rozkład liczby cząstek grafitu w kompozycie na osnowie stopu AZ91(metoda pól)

The microstructure of cast AZ91 composite reinforced with graphite particles was typical for cast magnesium matrix alloy, which is characterized by very heavy segregation of alloying elements. Mg-Al type alloys are prone to segregation due to relatively wide temperature spans between the liquids and the solids curves. Non-equilibrium solidification conditions caused the formation of large crystals of α phase (depleted in aluminium) and pushing the aluminium admixture away into interdendrical spaces. Figures 3 and 4 show images of microstructures revealed in etched surfaces. The microstructure was mainly characterized by solid solution of aluminium in magnesium (α phase - marked as 1 in Fig. 4) with a different composition of alloying element according to solidification rate and α+γ semi-divorced eutectic (marked as 2 in Fig. 4). γ-phase (called also β-phase) is the intermetallic compound $Mg_{17}Al_{12}$. It should be noted that commercial ternary alloys, for example AZ91, contain zinc as the additional alloying element but no new phases occur in the alloys if the Al to Zn ratio is greater than 3:1. Zinc substitutes aluminium in the γ-$Mg_{17}Al_{12}$ phase, creating ternary intermetallic compound ($Mg_{17}Al_{11.5}Zn_{0.5}$ or $Mg_{17}(Al,Zn)_{12}$ type) [3-5]. The discontinuous precipitates of γ phase was also observed (marked as 3 in Fig. 4). Discontinuous reaction, representing a solid-solid phase transformation, where a solid solution enriched in alloying element ($\alpha_0$) decomposes into a new solute-rich precipitate (γ) and near-equilibrium phase ($\alpha_n$) with the same crystal structure as the $\alpha_0$, according to the reaction: $\alpha_0 \rightarrow \alpha + \gamma$. This heterogeneous reaction leads to the formation of a lamellar structure due to the cellular growth of alternating layers γ and $\alpha_n$ phase. Because of non-equilibrium solidification conditions, aluminium is not uniformly distributed in α solid solution. During cooling of casts, below solvus temperatures, discontinuous precipitates of γ phase formed in the regions of solid solution enriched in aluminium. The presence of a small amount of manganese in commercial magnesium-aluminium alloys also caused a formation of $Al_8Mn_5$ intermetallic compound (marked as 4 in Fig. 4).

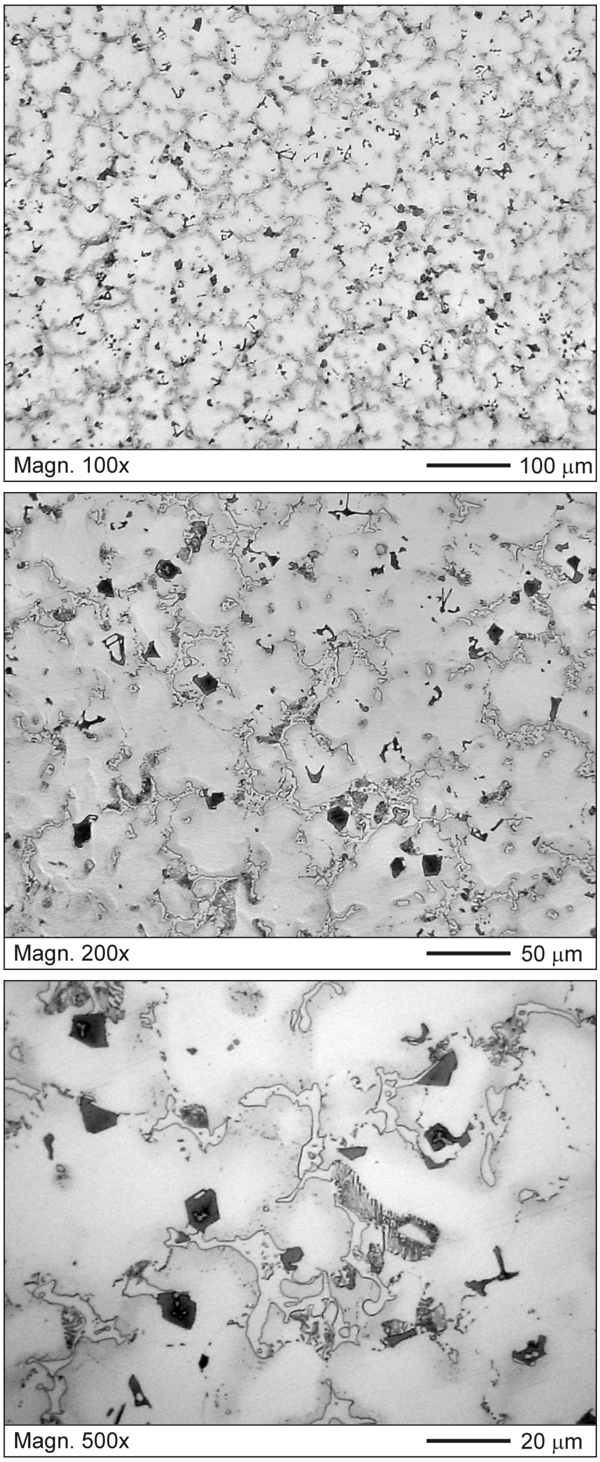


Fig. 3. Microstructure of the AZ91 matrix alloy composite reinforced with graphite particles (etched surface)

Rys. 3. Mikrostruktura kompozytu na osnowie stopu AZ91 umacnianego cząstkami grafitu (powierzchnia trawiona)

 

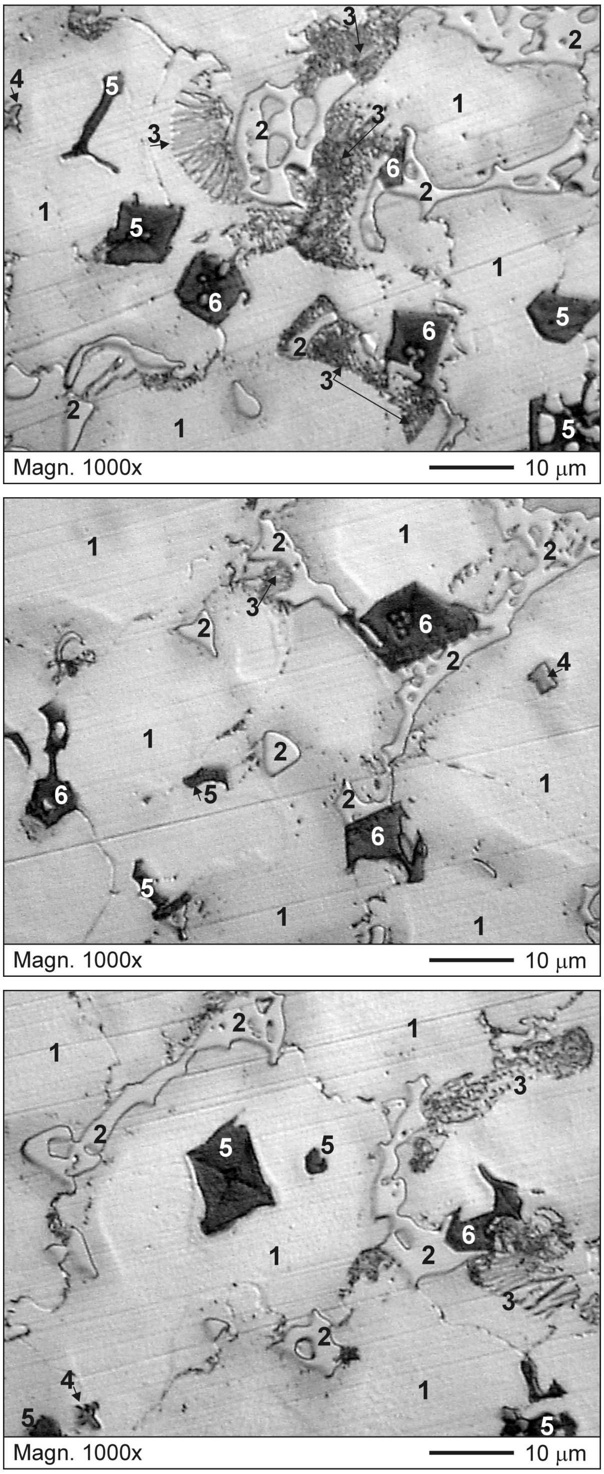


Fig. 4. Microstructure of the AZ91 matrix alloy composite reinforced with graphite particles (etched surface); 1 - α phase, 2 - α+γ eutectic, 3 - discontinuous precipitates of γ phase, 4 - $Al_8Mn_5$ intermetallic compound, 5 - graphite particles inside dendrites, 6 - graphite particles in interdendritics

Rys. 4. Mikrostruktura kompozytu na osnowie stopu AZ91 umacnianego cząstkami grafitu (powierzchnia trawiona); 1 - faza α, 2 - eutektyka α+γ, 3 - nieciągłe wydzielenia fazy γ, 4 - związek międzymetaliczny $Al_8Mn_5$, 5 - cząstki grafitu wewnątrz dendrytów, 6 - cząstki grafitu w przestrzeniach międzydendrytycznych

The presented microstructure images indicated also that graphite particles were located both inside dendrites (marked as 5 in Fig. 4) and interdendritical sides (marked as 6 in Fig. 4). Due to such location of graphite particles in cast matrix alloy it is difficult to describe the influence of particles on matrix crystallization process. On the other hand, homogeneity of graphite particles distribution and their location inside the dendrites, both testify about the wettability of used particles by molten matrix alloy.

## CONCLUSIONS

The produced AZ91 matrix alloy composite reinforced with graphite particles was characterized by a uniform distribution of the reinforcement phase in the volume of the matrix. Homogeneity of graphite particles distribution was regarded in terms of the coefficient of variation which was equal to 0.266. Neither the clusters of graphite particles nor any impurities, frequently occurring in gravity cast composites, were observed. The microstructure of the composite was typical for gravity cast AZ91 magnesium alloy (solidified under non-equilibrium condition). Graphite particles were observed both inside the dendrites and interdendritical sides. A uniform distribution of graphite particles and their location inside the dendrites testify the good wettability of particles by the molten matrix alloy and the easy formation of a bond between the components.

## REFERENCES

[1] Chua B.W., Lu L., Lai M.O., Influence of SiC particles on mechanical properties of Mg based composite, Composite Structures 1999, 47, 595-601.

[2] Zheng M., Wu K., Yao C., Sato T., Tezuka H., Kamio A., Li D.X., Interfacial bond between SiCw and Mg in squeeze cast SiCw/Mg composites, Materials Letters 1999, 41, 57--62.

[3] Magnesium Alloys and Their Applications, WILEY-VCH, Edited by K.U. Kainer, 1998.

[4] Magnesium Alloys and Their Applications, WILEY-VCH, Edited by K.U. Kainer, 2000.

[5] Magnesium Alloys and Their Applications, WILEY-VCH, Edited by K.U. Kainer, 2003.

[6] Zheng M., Wu K., Yao C., Effect of interfacial reaction on mechanical behavior of SiCw/AZ91 magnesium matrix composites, Materials Science and Engineering 2001, A318, 50-56.

[7] Cai Y., Tan M.J., Shen G.J., Su H.Q., Microstructure and heterogeneous nucleation phenomena in cast SiC particles reinforced magnesium composite, Materials Science and Engineering 2000, A282, 232-239.

[8] Lianxi H., Erde W., Fabrication and mechanical properties of SiCw/ZK51A magnesium matrix composite by two-step squeeze casting, Materials Science and Engineering 2000, A278, 267-271.

[9] Saravanan R.A., Surappa M.K., Fabrication and characterisation of pure magnesium-30 vol.% $SiC_P$ particle composite, Materials Science and Engineering 2000, A 276, 108-116.

 

[10] Hassan S.F., Gupta M., Development of high performance magnesium nano-composites using nano-$Al_2O_3$ as reinforcement, Material Science and Engineering 2005, A392, 163--168.

[11] Zhang Xiuqing, Liao Lihua, Ma Naiheng, Wang Haowei, The effect of heat treatment on damping characterization of TiC/AZ91 composites, Materials Letters 2006, 60, 600-604.

[12] Jinhai Gu, Xiaonong Zhang, Yongfu Qiu, Mingyuan Gu, Damping behaviors of magnesium matrix composites reinforced with Cu-coated and uncoated SiC particulates, Composites Science and Technology 2005, 65, 1736-1742.

[13] Hassan S.F., Gupta M., Development of a novel magnesium/nickel composite with improved mechanical properties, Journal of Alloys Compounds 2002, 335, L10-L15.

[14] Braszczyńska K.N., Lityńska L., Zyska A., Baliga W., TEM analysis of the interfaces between the components in magnesium matrix composites reinforced with SiC particles, Materials Chemistry and Physics 2003, 81, 326-328.

[15] Easton M.A., Schiffl A., Yao J-Y., Kaufmann H., Grain refinement of Mg-Al(-Mn) alloys by SiC additions, Scripta Materialia 2006, 55, 379-382.

[16] Xiuging Z., Haowei W., Lihua L., Xinying T., Naiheng M., The mechanical properties of magnesium matrix composites reinforced with ($TiB_2$+TiC) ceramic particulates, Material Letters 2005, 59, 2105-2109.

[17] Chen H., Liu J., Huang W., Corrosion behavior of silicon nitride bonding silicon carbide in molten magnesium and AZ91 magnesium alloy, Materials Science and Engineering 2006, A415, 291-296.

[18] Wang Y., Wang H-Y., Xiu K., Wang H-Y., Jiang Q-Ch., Fabrication of $TiB_2$ particulate reinforced magnesium matrix composites by two-step processing method, Materials Letters 2006, 60, 1533-1537.

[19] Xiuqing Z., Lihua L., Naiheng M., Haowei W., Effect of aging hardening on in situ synthesis magnesium matrix composites, Materials Chemistry and Physics 2006, 96, 9-15.

 