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STRUCTURE ANALYSES OF Mg-Zn-Zr MATRIX ALLOY COMPOSITES REINFORCED WITH SiC PARTICLES

The results of microstructure investigations of magnesium matrix composites reinforced with silicon carbide particles have been presented. The Mg - 6 wt. % Zn - 0.9 wt. % Zr alloy was used as a composite matrix. The investigated composites were reinforced with 30 wt. % of SiC particles with a maximum diameter of 32 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 obtain the investigated materials. The obtained composites were characterized by uniform distribution of silicon carbide particles within the matrix alloy. Additionally, Mg_2Si phase was identified in the composites microstructure, as a result of reaction between the matrix alloy and SiO_2 film very often covering SiC particle. Moreover, the components did not reveal the creation of a reaction layers at the matrix/particles interfaces. Transmission electron microscopic analyses allowed to determine the presence of Zn-rich phases as well as the iron oxides precipitates at the interfaces between the silicon carbide particles and the matrix alloy.

Keywords: **magnesium matrix composites, SiC particles, structure**

ANALIZY STRUKTURY KOMPOZYTÓW NA OSNOWIE STOPU Mg-Zn-Zr UMACNIANYCH CZĄSTKAMI SiC

Przedstawiono wyniki badań mikrostrukturalnych kompozytów na osnowie stopu magnezu umacnianych cząstkami węglika krzemu. Jako osnowę zastosowano stop Mg - 6% wag. Zn - 0.9% wag. Zr. Badane kompozyty umacniane były 30% wag. cząstek SiC o średnicy 32 μm. W celu otrzymania badanych kompozytów zastosowano prostą i niedrogą metodę odlewniczą, polegającą na mechanicznym mieszaniu ciekłego metalu wraz w wprowadzonymi cząstkami ceramicznymi w ochronnej atmosferze. Otrzymane kompozyty charakteryzowały się jednorodnym rozmieszczeniem cząstek węglika krzemu w stopie osnowy. Dodatkowo została zidentyfikowana faza Mg_2Si w osnowie kompozytów jako wynik reakcji pomiędzy stopem osnowy i filmem pokrywającym bardzo często cząstki SiC. Ponadto komponenty nie wykazały tworzenia warstw reakcyjnych na granicy rozdziału cząstki/osnowa. Analizy wykonane przy zastosowaniu transmisyjnej mikroskopii elektronowej pozwoliły określić obecność faz bogatych w Zn oraz także wydzieleń tlenków żelaza na granicy rozdziału pomiędzy cząstkami węglika krzemu i stopem osnowy.

Słowa kluczowe: **kompozyty magnezowe, cząstki SiC, struktura**

INTRODUCTION

Metal matrix composites (MMCs) reinforced with ceramic particles are among the most recent structural construction materials. A number of manufacturing techniques have been developed to produce MMCs. Compared with other methods (like powder metallurgy, mechanical alloying, infiltration or self-propagating high-temperature synthesis), stir casting is the most economical (costs as little as one-third to one-tenth for mass production) and easily adopted method [1-9]. Obtaining expected properties in these materials depends mostly on achieving the desired structure characterized by the proper arrangement of the reinforcing phase in the matrix volume and on providing a strong bonding between the components. Reinforcement/matrix interfaces in cast MMCs are related to the wettability of the reinforcement phase by the molten matrix and to the interaction of the components during the production of the composite [10-14].

Among various MMC′s materials, magnesium matrix composites reinforced with ceramic particles deserve special considerations, because of their low density, high specific stiffness and strength, high damping capacity, and good dimensional stability. Among the various ceramic reinforcements in particulate reinforced magnesium matrix composites, silicon carbide is the most widely used. It should be noted that the magnesium – silicon carbide system is characterized by a very good wettability of SiC by molten Mg, a very high stability of SiC in liquid Mg and a precipitate-free and strongly bonded SiC/Mg interfaces with an adhesive character of the bond [15-18]. The influence, however, of all individual alloying elements on the nature of Mg/SiC interfaces is still not known.

In the presented study, the microstructure analyses of the Mg - 6 wt.% Zn - 0.9 wt.% Zr matrix composites reinforced with SiC particles were performed in order to describe the phenomena influencing the formation of the structure of the cast of those materials.

EXPERIMENTAL DETAILS

The Mg - 6 wt. % Zn - 0.9 wt. % Zr alloy was prepared as a matrix of composites. The α-SiC particles of the 6H type with a maximum diameter of 40 μm have been used as the reinforcing phase. The weight friction of SiC particles in composites was about 30%. The composites samples have been 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.

Microstructure examination was carried out by means of light microscope Neophot-32 (Carl-Zeiss Jena) on polished and etched (with a 0.5% nital solution) microsections. Analyses of distribution of the individual elements contained in the composite phases were performed by scanning electron microscopy (SEM), on an instrument equipped with an energy-dispersive X-ray analysis (EDX) device. The microstructures of the interfaces between the components were examined by transmission electron microscopy (TEM). Specimens for TEM investigations have been cut in the form of 3 mm discs and then thinned mechanically by means of the thinning device of Gatan production and polished using a double-jet Fischione polisher. Some of the foils were ion-thinned by means of a Gatan Duo Mill 600 equipped with a stand for cooling preparation in liquid nitrogen. Transmission electron microscopy observations were performed with a Philips CM20 microscope operating at 200 kV equipped with an X-ray energy-dispersive spectrometer (EDX). The bright field technique was used in microstructure observations.

RESULTS AND DISCUSSION

Figure 1 shows a typical microstructure of the composite characterized by a uniform distribution of SiC particles in the matrix alloy. Neither the clusters of SiC particles nor any consequences of floating or sedimentation of the reinforced phase, frequently occurring in gravity cast composites, are observed. Because of the relatively large size of the silicon carbide particles and relatively high crystallization speed of composites, the phenomenon of pushing or engulfing the reinforced particles by the growing matrix dendrites has not occurred.

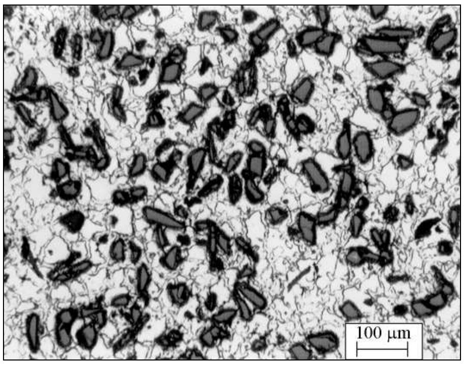

Fig. 1. Microstructure of the Mg - 6 wt. % Zn - 0.9 wt. % Zr matrix alloy composite reinforced with SiC particles

Rys. 1. Mikrostruktura kompozytu na osnowie stopu Mg - 6% wag. Zn - 0,9% wag. Zr umacnianego cząstkami SiC

The microstructure of the gravity cast matrix alloy is characterized by a very heavy segregation of the alloying elements. Magnesium-zinc alloys are prone to the segregation due to relatively wide temperature spans between the liquidus and the solidus curves. Non-equilibrium solidification conditions cause the formation of large crystals of the primary α-phase (depleted in alloying elements) and pushing the Zn admixture away into interdendritical spaces. At the last stage of solidification the MgZn-type intermetallic compounds are formed. The zirconium modifies the solidification process and causes the formation of the cellular microstructure.

SEM+EDX analyses indicate a presence of MgZn-type phase and also Mg_2Si phase in the composite matrix and near components interfaces. Figure 2 illustrates the microstructure obtained by scanning electron microscopy with superficial distribution of revealed elements, like magnesium, zinc, silicon and oxygen. The phase marked as “A” is the Mg_2Si compound while the phase marked as “B” is the Zn-rich intermetallic compound (MgZn-type). Silicon, which frequently covers the surface of SiC particles, could derive from the reaction between the matrix alloy and SiO_2 layer. It has been noted that the presence of the SiO_2 compound covering SiC particles due to the natural process of oxidizing has been reported by many research workers [4, 14].

Microstructure observations realized by transmission electron microscopy allowed to determine strongly connected and clean interfaces between the matrix and the silicon carbide particles. Figure 3 illustrates the transmission electron micrograph of a typical matrix/SiC particle interface observed in the investigated composites (with a visible dislocation structure in the matrix alloy). Although thermodynamic analyses suggest a possibility of reaction of zirconium with SiC particles, tests performed on composites made on the Mg - 6 wt. % Zn - 0.9 wt. % Zr matrix alloy did not reveal these tendencies.

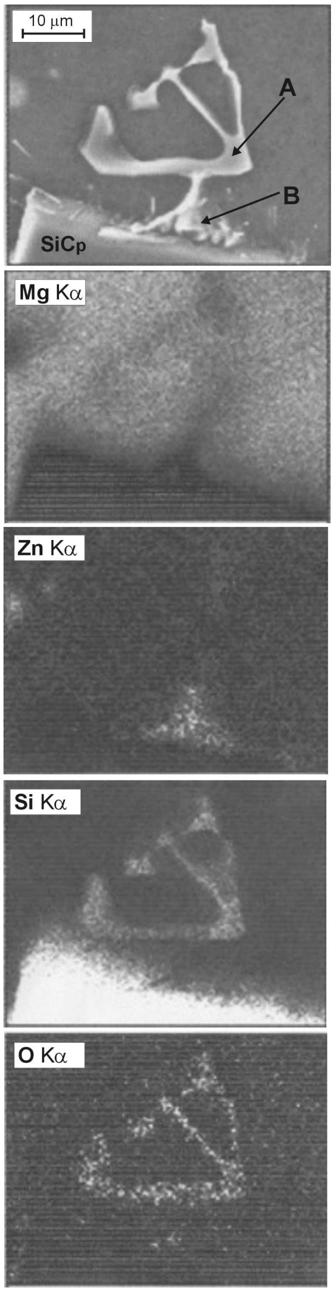

Fig. 2. Secondary electron image of the investigated composite with superficial distribution of revealed elements; SEM+EDX

Rys. 2. Obraz elektronów wtórnych badanego kompozytu wraz z rozkładem powierzchniowym ujawnionych pierwiastków; SEM+EDX

In some cases, fine crystals of the intermetallic compounds are observed near the interfaces between SiC particles and the matrix alloy. The results of the superficial and the point X-ray analyses of elements in observed microstructure, performed by TEM+EDX, are shown in Figure 4. The local high concentration of zinc near component interfaces (forms in the last stage of the matrix alloy solidification process as the result of an alloying element segregation) suggests that silicon carbide particles are not likely the nucleation sides for the magnesium matrix.

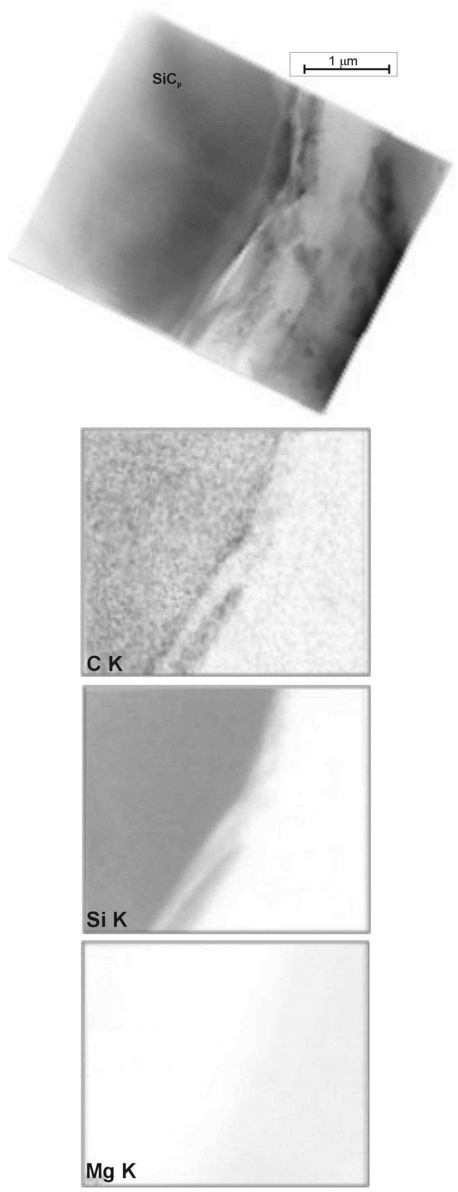

Fig. 3. A typical TEM image of the interface between the Mg - 6 wt. % Zn - 0.9 wt. % Zr matrix alloy and SiC particle

Rys. 3. Typowy obraz TEM granicy rozdziału pomiędzy Mg - 6% wag. Zn - 0,9% wag. Zr stopem osnowy oraz cząstką SiC

Occasionally, in same cases interfaces between components also contained precipitates with a different size and square morphology. Figure 5 shows the TEM image of the interface between the magnesium matrix and SiC particle with visible fine square precipitates. X-ray maps preformed from the observed area revealed the presence of iron and oxygen at the component interfaces. This result suggests that analyzed precipitates are the iron oxides. The iron is probably introduced into the investigated material during the mechanical mixing of the liquid composite suspension as a result of the damage process of the steel equipment used, such as crucibles and rabbles. These impurities, which were introduced due to an imperfect technological process, were located mostly along the interfaces between the matrix alloy and reinforced particles.

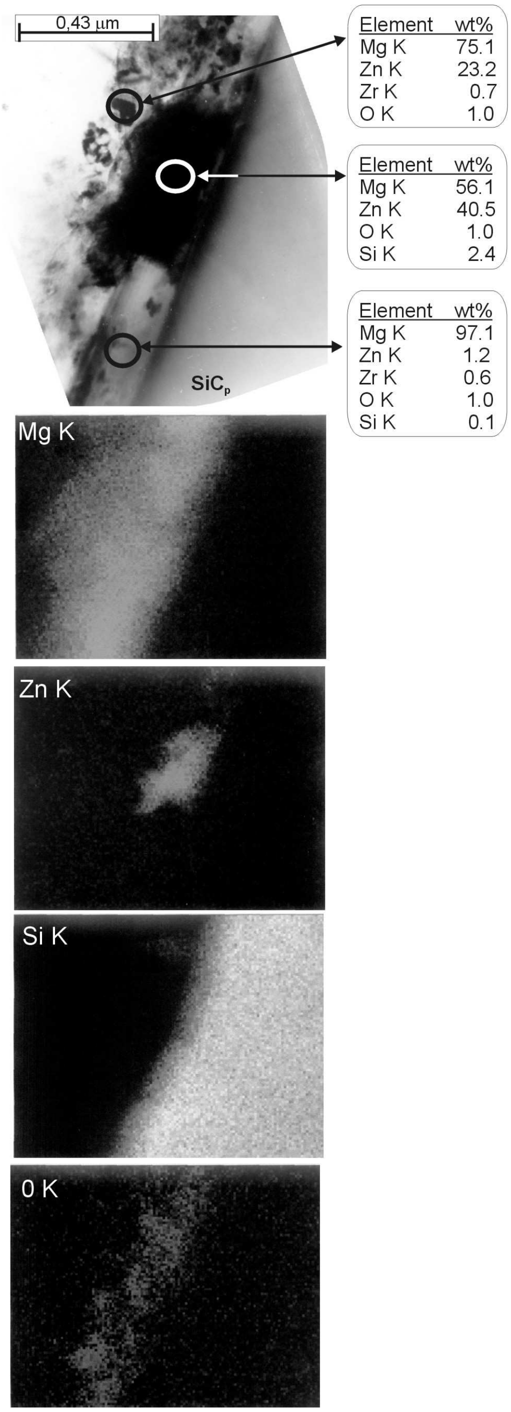

Fig. 4. TEM image of the interface between the Mg - 6 wt. % Zn - 0.9 wt. % Zr matrix alloy and SiC particle and X-ray maps and point analyses of an elements at the observed interface, TEM+EDX

Rys. 4. Obraz TEM granicy rozdziału pomiędzy stopem osnowy Mg - 6% wag. Zn - 0,9% wag. Zr i cząstką SiC oraz rozkłady powierzchniowe i analizy punktowe pierwiastków na obserwowanej granicy, TEM+EDX

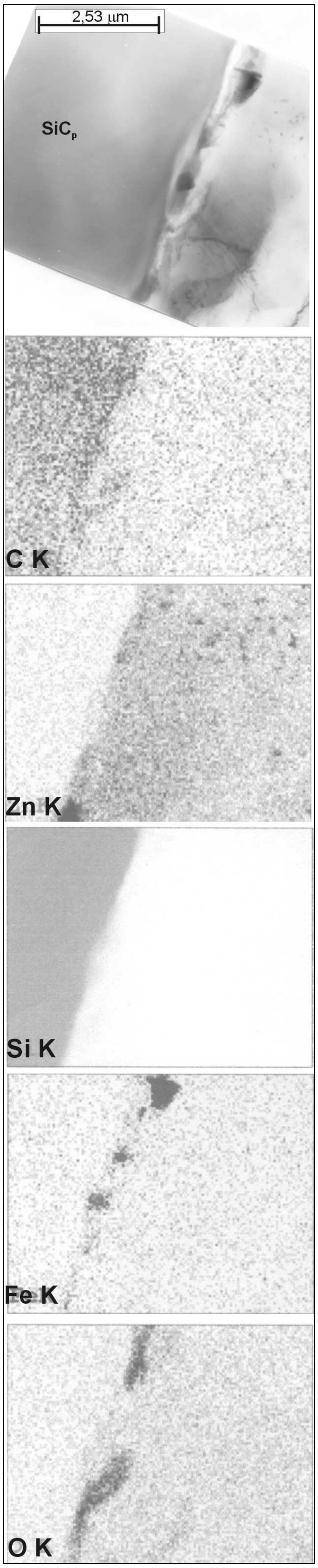

Fig. 5. TEM image of the interface between the Mg - 6 wt. % Zn - 0.9 wt. % Zr matrix alloy and SiC particle and X-ray maps showing the distribution of C, Si, Zn, Fe and O at the observed interface, TEM+EDX

Rys. 5. Obraz TEM granicy rozdziału pomiędzy stopem osnowy Mg - 6% wag. Zn - 0,9% wag. Zr i cząstką SiC oraz rozkłady powierzchniowe obrazujące rozkład C, Si, Zn, Fe i O na obserwowanej granicy, TEM+EDX

CONCLUSIONS

The produced Mg - 6 wt. % Zn - 0.9 wt. % Zr matrix alloy composites reinforced with silicon carbide particles are characterized by a uniform arrangement of the reinforcement phase in the volume of the matrix. Such a uniform distribution has been possible owing to the good wettability of the SiC particles by the molten matrix alloy and the easy creation of the bond between the components. It should be noted that the assurance of proper production process parameters is the primary condition for obtaining the correct structure of composites. One of the consequences of failing to satisfy this condition is the occurrence of slag contamination. Additionally damage process of the equipment used (such as crucibles and rabbles), which should be made of steel causes the introduction of iron oxides into the composite material, which most often locate at the interface of components and give rise to mechanisms destructive to composites.

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