

Interfaces in the magnesium-matrix composites

Mejna območja v kompozitih z magnezijevo osnovo

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Abstract

Processes at the matrix/reinforcement interfaces strongly influence the properties of the composites. The basic task of the interfaces is to assure the strong bonding between the composite's constituents. In addition, they must be mechanically and thermodynamically stable. Therefore, the understandings how the bonds at the interfaces are formed, as well as the related processes, are of crucial importance by designing and manufacturing of the composites. This review paper describes the interfaces in the magnesium-matrix composites reinforced with different types of SiC, Al₂O₃, and SiO₂, and prepared by different methods.

Key words: magnesium-matrix, reinforcement, composite, interface, reaction product.

Izveček

Procesi v mejnih območjih med osnovo in utrjevalno sestavino močno vplivajo na lastnosti kompozitov. Osnovna naloga mejnih območij je zagotavljanje trdne povezave med sestavinami kompozita, prav tako morajo biti mejna območja mehansko in termodinamično stabilna. Zato je poznanje načina nastanka povezovanja v mejnih območjih in procesov, ki se zgodijo na njih, zelo pomembno za načrtovanje in izdelavo kompozitov. Pregledni članek opisuje mejna območja v kompozitnih materialih z magnezijevo osnovo, ki so utrjeni z različnimi oblikami SiC, Al₂O₃ in SiO₂ ter izdelani z različnimi postopki.

Ključne besede: magnezijeva osnova, utrjevalna sestavina, kompozit, mejno območje, reakcijski produkt

Introduction

Composites are modern materials, which consist of at least two chemically, physically, and mechanically different materials. The properties of the composites depend on the properties of the matrix and reinforcing phase, shape, fraction, distribution, and orientation of the reinforcing phase, interactions at the matrix/reinforcement interface, processing parameters, and heat treatment conditions. Often, these properties can be predicted, using the rule of mixtures^[1]:

$$L_c = L_m V_m + L_r V_r \quad (1)$$

where L_c is the property of the composite (e.g. Young's modulus, density, etc.), V the volume content, and indices m and r indicates the matrix and the reinforcement, respectively.

The interface between matrix and reinforcement is a region with different physical and chemical properties compared to the properties of the composite's constituents. Interface bonding arises from the adhesion of the constituents, which depends on the wettability. In the composites, the wettability is defined as the ability of the liquid matrix to spread over the solid surface of the reinforcement. Physical and chemical processes at the interfaces can strongly influence the mechanical, thermomechanical, and thermodynamic properties of the composites.^[1]

Reaction products are formed at the matrix/reinforcement interfaces as a result of the chemical reactions. These interfacial reaction products are usually brittle and could be strongly or weakly bonded to the reinforcement. There exists the critical thickness of the reaction products beyond which the composite properties becomes deteriorated.^[2] In the titanium-matrix

composites reinforced with SiC fibres, the critical thickness of the reaction products was 1 μm . The mechanical properties of the composite above this value were significantly decreased.^[3] Magnesium-matrix composites are promising materials because of their low density and high strength/weight ratio. The specific strength and stiffness of the magnesium-matrix composites should be greater than those of the aluminium-matrix composites. The selection of the ceramic reinforcement (chemical composition, shape, and volume fraction) and magnesium (alloy) matrix can be used to tailor the thermal conductivity of the composites. Particles (p), fibres (f), whiskers (w), and recently also different preforms (e.g. ceramic foam (cf)) are usually used as reinforcements (Figure 1). An advantage of the magnesium, compared to the aluminium, is that it can wet most of the ceramic reinforcement. A disadvantage is its reactivity with the reinforcements. In many cases, the reinforcements are very prone to oxidation. The oxidation behaviour and further reactions could influence interfacial structure and composition, and hence the nature and strength of the interfacial bonding. Undesirable reaction products at or near the interface may lead to loss of the load-bearing ability and thus change the mechanical properties of the composite.^[2]

Morphologies of the interfaces

Figure 2 schematically presents distinct types of the matrix/reinforcement interfaces. At the interface type I (Figure 2a), the interfacial reaction products (IRPs) are directly in contact with the reinforcement. For the interface type II (Figure 2b), the interfacial reaction zone (IRZ) consists of two distinct layers. The first layer consists of the IRPs, and this layer is in direct

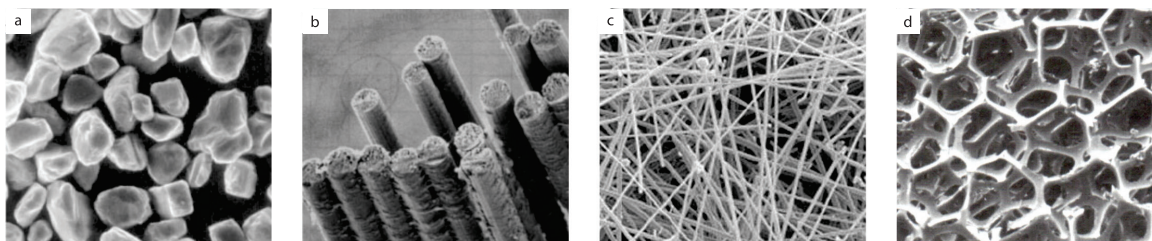


Figure 1: Shapes of the reinforcements. a) particles (p), b) fibres (f), c) whiskers (w) and d) ceramic foam (cf).

contact with the matrix. The second layer is in direct contact with the reinforcement, which consists of the matrix that extends along reinforcing surface. Thus, for interface type II the IRPs are not in direct contact with the reinforcement. The interface type III is very clean (Figure 2c) and the IRPs have not even formed at the interface at all.^[4] If the matrix does not wet the reinforcement, the cracks and debonding free interfaces are present between them. This interface can be marked as the interface type IV (Figure 2d).

Figure 3 shows the interfaces in the composite with AZ91 matrix reinforced with SiC particles (SiCp), where three kinds of the interfaces were observed.^[4] The reasons for existence of these three kinds of the interfaces in the present composite may arise from the conditions during stir-casting, which are very complicated. The friction between the melt and SiCp takes place during stirring and causes shearing during pouring. These actions can cause the formation of the interface III by breaking away the IRPs from the SiCp. However, the IRPs separated from the SiCp and SiCp are often pushed by the freezing front to the last solidified regions, and this leads to the formation of the interface II.^[5] When the IRPs do not break away from the SiCp

during stirring and pouring, the interface I will be formed.^[4]

Types of bonding at the interface

There are two types of bonding at the interface in the metal-matrix composites (MMCs): mechanical bonding and chemical bonding.

Mechanical bonding

Mechanical bonding is formed when the surfaces of the matrix and reinforcement are interconnected, and there are no chemical bonds between them. Interfaces in the MMCs are invariably rough, and the degree of the interfacial roughness increases the strength of the bond. In the MMCs reinforced with the ceramics, the metals generally have a higher coefficient of the thermal expansion than the ceramics. Thus, the metallic matrix in the composite will shrink more than the ceramic reinforcement on the cooling from a high temperature. This will lead to the mechanical gripping of the reinforcement by the matrix even in the absence of any chemical bonding. The matrix infiltrates into the cracks on the reinforcing surface, by the liquid flow or high temperature diffusion, which can also lead to some mechanical bonding.

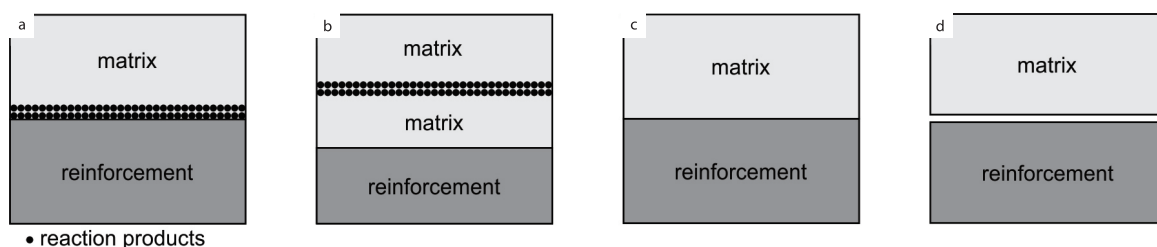


Figure 2: Schematic representation of the distinct interfaces. a) type I, b) type II, c) type III^[4] and d) type IV.

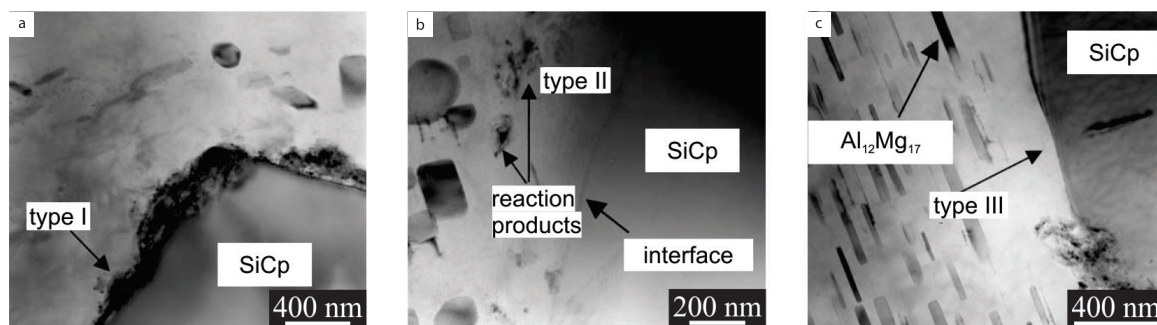


Figure 3: TEM of the three distinct types of interfaces between AZ91 and SiCp. a) interface I, b) interface II and c) interface III.^[4]

The radial gripping stress, σ_r , can be related to the interfacial shear strength, τ_i , by the equation 2^[6]:

$$\tau_i = \mu \sigma_r \quad (2)$$

where μ is the friction coefficient. It usually lies between 0.1 and 0.6. In general, the mechanical bond is a low energy bond vis-à-vis the chemical bond.

Chemical bonding

The metal/ceramics interfaces in the MMCs are generally formed at high temperatures. The diffusion and chemical reaction kinetics are faster at the elevated temperatures. Knowledge of the chemical reaction products and, if possible, their properties are needed. It is, therefore, imperative to understand the thermodynamics and kinetics of the reactions. In this way, the processing can be controlled, and optimum properties obtained. Chemical bonding in the MMCs involves atomic transport by the

diffusion, which causes the change of chemical compositions of the constituent phases at the interface. Thus, chemical bonding includes the solid solution and/or chemical compound formation at the interface (Table 1). For the diffusion controlled growth in an infinite diffusion couple with a planar interface, the important relationship is valid^[6]:

$$x^2 = Dt \quad (3)$$

where x is the thickness of the reaction layer, D the diffusivity, and t the time. The diffusivity, D , depends on the temperature in an exponential manner^[6]:

$$D = D_0 \exp\left(-\frac{\Delta Q}{kT}\right) \quad (4)$$

where D_0 is a pre-exponential constant, ΔQ the activation energy for the rate controlling process, k the Boltzmann's constant, and T the temperature.

Table 1: Chemical reactions that can take place between magnesium and oxides, carbides, binding agents, and protective gases during the manufacturing of the MMCs

Phase	Chemical reaction	References
SiO ₂	$2\text{Mg}_{(l)} + \text{SiO}_{2(s)} \rightarrow 2\text{MgO}_{(s)} + \text{Si}_{(s)}$	{5.1} 4, 7, 8
	$2\text{Mg}_{(l)} + 2\text{SiO}_{2(s)} \rightarrow \text{Mg}_2\text{SiO}_{4(s)} + \text{Si}_{(s)}$	{5.2} 8
	$4\text{Mg}_{(l)} + \text{SiO}_{2(s)} \rightarrow 2\text{MgO}_{(s)} + \text{Mg}_2\text{Si}_{(s)}$	{5.3} 2, 8, 9
	$\text{SiO}_{2(s)} + \text{MgO}_{(s)} \rightarrow \text{MgSiO}_{3(s)}$	{5.4} 10
	$\text{SiO}_{2(s)} + 2\text{MgO}_{(s)} \rightarrow \text{Mg}_2\text{SiO}_{4(s)}$	{5.5} 10
	$4\text{Al}_{(l)} + 3\text{SiO}_{2(s)} \rightarrow 2\text{Al}_2\text{O}_3 + 3\text{Si}_{(s)}$	{5.6} 4
	$\text{Mg}_{(l)} + 2\text{Al}_{(l)} + 2\text{SiO}_{2(s)} \rightarrow \text{MgAl}_2\text{O}_4 + 2\text{Si}_{(s)}$	{5.7} 4, 9
	$4\text{Al}_{(l)} + 2\text{MgO}_{(s)} + 3\text{SiO}_{2(s)} \rightarrow 2\text{MgAl}_2\text{O}_4 + 3\text{Si}_{(s)}$	{5.8} 4
	$2\text{MgO}_{(s)} + 5\text{SiO}_{2(s)} + 2\text{Al}_2\text{O}_{3(s)} (+ \text{C}_{(s)}) \rightarrow \text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18(s)} (+ \text{C}_{(s)})$	{5.9} 11
	$2\text{Mg}_{(l)} + \text{SiO}_{2(s)} (+ \text{C}_{(s)}) \rightarrow 2\text{MgO}_{(s)} + \text{Si}_{(s)} (+ \text{C}_{(s)})$	{5.10} 11
SiC	$2\text{Mg}_{(l)} + \text{Si}_{(s)} \rightarrow \text{Mg}_2\text{Si}_{(s)}$	{5.11} 4, 7
	$2\text{Mg}_{(l)} + \text{SiC}_{(s)} \rightarrow \text{Mg}_2\text{Si}_{(s)} + \text{C}_{(s)}$	{5.12} 4
	$4\text{Al}_{(l)} + 3\text{C}_{(s)} \rightarrow \text{Al}_4\text{C}_{3(s)}$	{5.13} 4
	$4\text{Al}_{(l)} + 3\text{SiC}_{(s)} \rightarrow \text{Al}_4\text{C}_{3(s)} + 3\text{Si}_{(s)}$	{5.14} 4
Al ₂ O ₃	$3\text{Mg}_{(l)} + \text{Al}_2\text{O}_{3(s)} \rightarrow 3\text{MgO}_{(s)} + 2\text{Al}_{(l)}$	{5.15} 2, 12
	$3\text{Mg}_{(l)} + 4\text{Al}_2\text{O}_{3(s)} \rightarrow 3\text{MgAl}_2\text{O}_4 + \text{Al}_{(l)}$	{5.16} 11
	$\text{MgO}_{(s)} + \text{Al}_2\text{O}_{3(s)} \rightarrow \text{MgAl}_2\text{O}_4$	{5.17} 12
Al(PO ₃) ₃ (binding agent)	$9\text{Mg} + \text{Al}(\text{PO}_3)_3 \rightarrow 9\text{MgO} + \text{Al} + 3\text{P}$	{5.18} 13
N ₂ (protective gas)	$3\text{Mg}_{(g)} + \text{N}_{2(g)} \rightarrow \text{Mg}_3\text{N}_2$	{5.19} 14
	$\text{Mg}_3\text{N}_2 + 2\text{Al}_{(l)} \rightarrow 2\text{AlN} + 3\text{Mg}$	{5.20} 14

Interfacial reaction products

The interfaces between magnesium-matrix and reinforcements are not thermodynamically stable thus some interfacial reaction products can be formed (Table 2) as a result of the chemical reactions.

Reactions at the Mg/SiC interface

Magnesium and its alloys reinforced with the SiCp are very interesting because the reinforcement may lead to significant improvement of stiffness and strength.^[2] Reaction products at the magnesium/SiC interface depend on the manufacturing method of the composite.

Kaneda and Choh^[15] found that the MgO and Mg₂Si reaction products were formed at the pure magnesium/SiCp interface. The feature of this study was previous mixing of the SiO₂ powder infiltration agent with the SiCp reinforcement which is necessary for spontaneous infiltration phenomenon. The Mg-RE3 alloy wets the SiCp well, therefore, in this composite the RE₃Si₂ interfacial reaction products were formed in the form of the needles^[16] or thick reaction layer composed of the MgO, and Ce₃Si₂ fine particles.^[17] On the other hand, the interfacial reaction products were not observed in the composites with the pure magnesium, Mg-Al5, Mg-Al8, and Mg-Zn6 matrices reinforced with the SiCp and prepared by the melt stir techni-

que^[7, 17, 18]. Also, Cao et al.^[19, 20] did not find the interfacial reaction products in the Mg-Zn4, and Mg-Zn6 alloys reinforced with the SiC nanoparticles and prepared by the ultrasonic cavitation.

The AZ80/SiCp and AZ91/SiCp interfaces were without reaction products when the composites were prepared by the compocasting. Nevertheless, the particles of the Al₁₂Mg₁₇, and Cu₅Zn₈ compounds precipitated on the SiCp^[21, 22], indicating that the SiCp acted as nucleation sites. Similarly, the Mg(Cu, Zn)₂, and MgZn₂ compounds precipitated at the SiCp in the ZC63 - SiCp composite prepared by the melt infiltration into the powder and the melt stir technique. In the ZE63 - SiCp composite, which was prepared by the same procedure, the ZrO₂, and CeO₂ interfacial reaction products were formed.^[8] Further Wang et al.^[4] found the Al₄C₃, MgO, and Mg₂Si reaction products at the AZ91/SiCp interfaces when the composite was prepared by the melt stir technique. Directly at the reaction layer the carbon was present as a product of a chemical reaction between the magnesium and SiCp {5.12}. Magnesium does not have stable carbides but the aluminium, as an alloying element in the magnesium alloys, reacts with the carbon, and then the Al₄C₃ carbide can arise {5.13}. Also in this case, the SiCp acted as heterogeneous nucleation sites for Al₁₂Mg₁₇ and Al₈Mn₅ compounds. When the

Table 2: Interfacial reaction products formed at the interface between magnesium-matrix and different types of reinforcements

Matrix	Reinforcement	Interfacial reaction product
Mg		MgO, Mg ₂ Si
Mg-RE3	SiCp	MgO, RE ₃ Si ₂ or Ce ₃ Si ₂
ZE63		CeO ₂ , ZrO ₂
AZ91		Al ₄ C ₃ , MgO, Mg ₂ Si, MgAl ₂ O ₄ , AlN
AZ91		MgO
Mg	Al ₂ O ₃ f	MgAl ₂ O ₄ , MgO, Mg ₂ Si
AZ91		MgO
ZE41		MgO
AS21		MgO
AE44		MgO
AZ31	SiO ₂ cf	MgO, Mg ₂ Si
AZ61	SiO ₂ nano-p	MgO, Mg ₂ Si
AZ31	SiO ₂ -Al ₂ O ₃ cf	MgAl ₂ O ₄ , Mg ₂ Al ₄ Si ₅ O ₁₈ , Si, Mg ₂ Si
AZ91	SiC-SiO ₂ -C-Sicf	MgO, Mg ₂ Si

AZ91 - SiCp composite was prepared by the ultrasonic cavitation, the interfacial reaction products did not form.^[23]

Wu et al.^[24] investigated the interfaces between the AZ91 alloy and SiC whiskers (SiCw) in the composite prepared by the squeeze casting. They determined the MgO interfacial reaction products, while Zheng et al.^[25] did not find any interfacial reaction products in the same composite. When the $\text{Al}(\text{PO}_3)_3$ binding agent was added into the SiCw-preform, the MgO interfacial reaction products formed.^[13] In the AZ91 - SiC nanoparticles composite prepared by the ultrasonic cavitation, Lan et al.^[26] found the Mg_2Si interfacial reaction products, which were broken away from the AZ91/SiC nanoparticles interfaces because of the intensive ultrasonic cavitation.

The MgO, MgAl_2O_4 , and AlN interfacial reaction products and the $\text{Al}_{12}\text{Mg}_{17}$ compound were formed in the composite prepared by the melt infiltration of the AZ91 alloy into the premixed powder of the magnesium, aluminium, zinc, and SiC.^[27,28] The AlN reaction layer, which also contained magnesium, is the product of a chemical reaction between the Mg_3N_2 and aluminium {5.20}. The Mg_3N_2 layer around the particles of the powder was formed with reaction {5.19} between the magnesium and nitrogen, which was used as a protective gas.^[14] The MgAl_2O_4 interfacial reaction product formed in the composites with the aluminium- matrix reinforced with the SiC, and Al_2O_3 when the magnesium content in aluminium was smaller than the mass fraction of Mg 4 % or 2 %.^[9,28] The reactions {5.6}, {5.7}, and {5.8} did not take place because of the large chemical affinity of the magnesium to oxygen and the large content of the magnesium in the magnesium - SiCp composites.

Reactions at the Mg/Al₂O₃ interface

The composites, where the magnesium and its alloys are infiltrated into the reinforcing preform of the Al_2O_3 fibres ($\text{Al}_2\text{O}_3\text{f}$), are most often prepared by the squeeze casting^[29-35]. The preform of the $\text{Al}_2\text{O}_3\text{f}$ contains 3-4 % of the SiO_2 binding agent.

Rehman et al.^[36] investigated the matrix/fibre interactions in the composites with the pure magnesium, AZ61, and AZ91 matrices reinforced with the different $\text{Al}_2\text{O}_3\text{f}$. A few large

Mg_2Si particles were found in the pure magnesium reinforced with the $\delta\text{-Al}_2\text{O}_3\text{f}$ (Safimax) with standard density. In the case of the reinforcing with the $\eta\text{-Al}_2\text{O}_3\text{f}$ (Safimax) with low density, the fibres were reduced into the MgO and aluminium {5.15}. The fine MgO interfacial reaction products were observed in the AZ91 - $\delta\text{-Al}_2\text{O}_3\text{f}$ (Saffil) composite. It is viable that increasing the aluminium content in the magnesium matrix may reduce the interfacial reactions. Also, Hach^[37], Page^[38], Hallstedt^[39], Trojanová^[40] and Sklenička^[41,42] found the MgO particles at the matrix/fibre interfaces in the Mg - $\alpha\text{-Al}_2\text{O}_3\text{f}$, Mg - $\delta\text{-Al}_2\text{O}_3\text{f}$ (Saffil), ZE41 - $\alpha\text{-Al}_2\text{O}_3\text{f}$, AS21 - $\delta\text{-Al}_2\text{O}_3\text{f}$ (Saffil), and AZ91 - $\delta\text{-Al}_2\text{O}_3\text{f}$ (Saffil) composites. The sizes of these particles were higher at the ZE41/ $\alpha\text{-Al}_2\text{O}_3\text{f}$ interfaces than at the Mg/ $\alpha\text{-Al}_2\text{O}_3\text{f}$ interfaces.^[38] They were further increased by increasing casting temperature^[43] and longer reaction times.^[44] The presence of the spread MgO interfacial reaction layer in the AE44 - Al_2O_3 short-fibres (Saffil) composite has been reported also by Hu et al.^[45] Besides, they have also found the Al_2RE particles. Similarly to the SiCp also the $\text{Al}_2\text{O}_3\text{f}$ acted as nucleation agents because the $\beta\text{-Al}_{12}\text{Mg}_{17}$ compound at the AZ91/ $\delta\text{-Al}_2\text{O}_3\text{f}$ (Saffil) interfaces and the Al_2Nd , $\text{Mg}(\text{Ag})_{12}\text{Nd}$, and Mg_3Ag compounds at the QE/ $\delta\text{-Al}_2\text{O}_3\text{f}$ (Saffil) interfaces were precipitated.^[42] Shi et al.^[12] found that in the Mg - $\text{Al}_2\text{O}_3\text{f}$ composite the MgAl_2O_4 interfacial reaction product was formed, probably with reaction between the MgO and Al_2O_3 {5.17}. It should be noted that, in this study, the magnesium or aluminium powder was added into the preform of the $\text{Al}_2\text{O}_3\text{f}$ and that the reaction time was 4 h at the temperature of 1 123 K. Also in the study of wettability of the $\alpha\text{-Al}_2\text{O}_3\text{f}$ with pure magnesium the MgAl_2O_4 interfacial reaction product was found in addition to the MgO.^[46-48] This shows that the MgAl_2O_4 reaction product is formed after very long reaction times.

Reactions at the Mg/SiO₂ interface

The SiO_2 is seldom used as a reinforcing phase in the form of the particles or in any other form. Most often it is used as a binding agent by the manufacturing of the reinforcing preform of the Al_2O_3 fibres.

The melt is infiltrated into the pores and struts of the ceramic foam at the manufacturing of

interpenetrated phase composites. The Mg_2Si , and MgO reaction products and $Al_{12}Mg_{17}$ compound were formed in the pores and struts of the SiO_2 ceramic foam, which were filled with the AZ31 matrix.^[49] Lee et al.^[50] incorporated the SiO_2 nanoparticles into the AZ61 matrix by the friction stir processing. The SiO_2 nanoparticles reacted with the magnesium {5.3} and the Mg_2Si , and MgO reaction products were formed.

Reactions at the $Mg/SiC + Al_2O_3 + SiO_2$ interface

The reinforcing phase can consist of two or more carbides or oxides in the different preforms, e.g. the ceramic foam (cf). Zeschky et al.^[11] found at the AZ31/ SiO_2 - Al_2O_3 /cf interface the $MgAl_2O_4$ {5.16}, and $Mg_2Al_4Si_5O_{18}$ {5.9} reaction products and the silicon, which further reacted with the magnesium and the Mg_2Si {5.11} was formed. In the AZ91 - oxidized SiC - SiO_2 - C - Si (cf) composite, at the interfaces the MgO , and Mg_2Si reaction products, into the struts of the ceramic foam very small content of the MgO , and in the centre of filled pores of the ceramic foam the MgO , Mg_2Si , and $\gamma-Al_{12}Mg_{17}$ were formed. In the case of reinforcing the AZ91 matrix with the non-oxidized SiC - SiO_2 - C - Si (cf), the cracks and debonding free interfaces were obtained between the metal and ceramic skeleton.^[10]

Conclusions

During the manufacturing of the magnesium-matrix composites, the strong bonding between the matrix and reinforcement, without the reaction products at the interfaces should be attained. However, most of the observed interfaces in the magnesium-matrix composites were covered with the interfacial reaction products. This means that the systems magnesium alloy - reinforcement (SiC , Al_2O_3 , and SiO_2) were thermodynamically unstable. The main interfacial reaction products were the MgO , and Mg_2Si . Their size increased with increasing casting temperature and longer reaction time while the increasing the aluminium content into the magnesium matrix reduced the interfacial reactions. The types of the interfacial reaction products were also depended upon the

manufacturing method. Therefore, in order to obtain adequate properties of the magnesium-matrix composites, it is necessary to choose the appropriate combination of the constituents and suitable manufacturing process.

Acknowledgement

This work was partly financed by the Slovenian Research Agency (ARRS), projects 1000-09-310152, and L2-2269.

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