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CHARACTERISATION OF MICROSTRUCTURE OF WE43 MAGNESIUM MATRIX COMPOSITES REINFORCED WITH CARBON FIBRES

In the paper the microstructures of WE43 matrix composites reinforced with carbon fibres have been characterised. The influence of reinforcement type and T6 heat treatment (a solution treatment at 525°C for 8 h, a hot water quench and a subsequent ageing treatment at 250°C for 16 h) on microstructure have been evaluated. The light microscope and scanning electron microscope investigations have been carried out. No significant differences in samples reinforced with non-coated textiles have been reported. The substantial changes in sample reinforced with nickel-coated textile have been observed. The segregation of alloying elements to the matrix-reinforcement layer has been identified. The T6 heat treatment caused the appearance of disperse precipitates of β phase, but the process cannot be considered as satisfactory (irregular distribution, low volume fraction, relatively large size).

Keywords: Metal matrix composite; Magnesium; WE43; Carbon fibre; Heat treatment.

1. Introduction

The trend of weight reduction determines the constant development of high performance light materials. Thus, the usage of lightweight metals and reinforced materials gains in importance and a big effort is laid on the replacement of conventional materials by metals like magnesium, aluminium or titanium [1].

Magnesium alloys possess the lowest density of all metallic constructional materials [2], however their application is limited comparing to the competing aluminium materials or polymers. Nevertheless, magnesium alloys are characterised by good damping capacity, excellent castability (suitable also for high-pressure die-casting) and weldability under controlled atmosphere. Moreover, this group of materials exhibits better mechanical properties, electrical and thermal conductivity and resistance to ageing in comparison with plastics [1,3-5]. Mentioned properties enable the application of magnesium alloys in many industries, but the major drawbacks, limiting the extensive usage of these alloys, are relatively poor corrosion resistance and insufficient mechanical properties at elevated temperatures. One of the possible ways of increasing the high-temperature properties of magnesium alloys is the addition of expensive rare earth metals, such as yttrium, neodymium or gadolinium [6]. Good creep properties are related to the presence of thermally stable and fine precipitates of Mg-Nd-Y intermetallic compounds (β" or β') [5,7-8].

Conventional alloying practice cannot ensure further improvement of the mechanical properties of magnesium alloys. For broadening its application it is of particular interest to use reinforcements. The need of high performance and light-weight materials in aerospace, aeronautical and automotive industries has become increasingly urgent in recent years, which leads to extensive research in the processing of magnesium matrix composites with cost-effective technologies [1]. Although there is no simple and clear definition of metal matrix composite, literature defines the magnesium matrix composite as a material consisting of rigid ceramic reinforcement and magnesium matrix. This kind of material combines the metallic properties of magnesium (low density, ductility and damping capacity) with ceramic characteristic (high strength, wear resistance), leading to greater strength and higher service temperature capabilities [3,9]. For instance, the magnesium matrix composite unidirectionally reinforced with continuous carbon fibre can readily show a bending strength of 1000 MPa with a density as low as 1,8 g/cm^3 to the temperature of up to 350-400°C [5]. To ensure certain properties fibre and particle reinforcements are used. The reinforcing material is usually Al_2O_3, SiC, carbon or combination of them.

The interface between matrix and reinforcement, including also the disperse precipitates in this area (products of chemical reactions between matrix and reinforcement or coating) is the critical region, which largely controls performance of the composite. One of the main problems in the fabrication of magnesium matrix composites arises from the reactivity of magnesium – some chemical reactions might cause the deterioration of reinforcement. Another problem is wettability. The presence of thermodynamically stable oxide on the surface of reinforcement inhibits wetting and infiltration. To overcome the

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processing problems coatings (copper, nickel, silicon carbide or titanium boride) and thermal treatments (the annealing of fibres to eliminate gases adsorbed on the surface of reinforcement) are applied. It provides the decrease of the contact angle and in consequence – improvement of wettability [2-5,10-12].

In order to optimize the microstructure and mechanical properties of magnesium matrix composites, a variety of fabrication methods have been developed over last two decades. The methods could be divided into two main groups – conventional and special. Four well established processing methods – stir casting, squeeze casting, powder metallurgy and liquid metal infiltration are considered as conventional methods, while pressure infiltration, in-situ reaction synthesis, mechanical alloying and spray forming are known as special methods [3,5,9,13].

Magnesium matrix composites exhibit rather high specific strength, wear resistance, excellent thermal and electrical conductivities, and good damping capacity and become attractive candidates for structural and functional materials [3].

2. Material and experimental methods

The main aim of the research was the evaluation of microstructures of fabricated composites. The influence of reinforcement type and heat treatment have been examined in order to achieve the most favourable microstructure and strength properties.

2.1. Material

The Mg-Y-Nd system is one of the most promising candidates for applications in aerospace and automotive industries due to its good castability, creep and corrosion resistance up to relatively high temperatures. The best properties are presented by WE43 and WE54 commercial magnesium alloys. Thus, WE43 magnesium alloy has been chosen for the matrix of investigated composites. Moreover, the mentioned alloy exhibits age-hardening properties, which may enable additional increase of composites' mechanical properties by application of T6 heat treatment [6,14-16]. The chemical composition of WE43 matrix alloy is given in Table 1 (RE represents the mixture of rare earth elements such as neodymium, dysprosium and ytterbium).

TABLE 1

Chemical composition of WE43 matrix alloy (weight %)

Mg	RE	Y	Zr	Zn	Mn	Fe	Ag
Balance	3.4	4.1	0.49	0.01	0.01	0.001	<0.01

The Tenax® – J HTS40 A23 12K 1420tex MC (by Toho Tenax America, Inc.) carbon fibres were used as the reinforcement of investigated composites. The properties of reinforcement fibres are listed in Table 2. Fibres were covered with organic size to aid in handling.

TABLE 2

Properties of reinforcement fibres according to Toho Tenax America, Inc

Property	Value
Tensile strength	2760 MPa
Tensile modulus	215 GPa
Elongation	1.28%
Density without size	2.70 g/cm^3
Linear density without sizing	1437 tex (g/km)
Sizing level	1.25%
Nominal filament diameter	$7.5 \cdot 10^{-6}$ m (including Nickel layer approx 0.25 μm thick, corresponding to 44% w/w nickel)
Twist	Never twisted
Specific resistivity	$1 \cdot 10^{-4}\ \Omega \cdot$ cm (typical)

The investigated samples were obtained from 3 casts of WE43 magnesium matrix composite reinforced with carbon fibres. Reinforcement was applied in the form of textile preforms:

- sample 1: one layer of non-coated 3D textile,
- sample 2: 3 layers of non-coated plain textile,
- sample 3: 3 layers of nickel-coated plain textile.

The samples of the matrix alloy have been included additionally to allow the comparison of the microstructures of the fabricated composites with the microstructure of non-reinforced WE43 alloy.

2.2. Composites fabrication technology

Composite samples were fabricated using the pressure infiltration technology. In the process carbon fibres were placed into a cuboidal mild steel mould. The mould and the silicon carbide crucible with the matrix alloy were fixed in the furnace and heated up to 720°C under the inert atmosphere of argon. During the heating process the carbon fibres were desized (temperature around 350°C). When the temperature reached the melting point the matrix alloy was homogenized and the mould with the carbon fibre perform was lowered beneath the surface of the melt. The pressure in the furnace was raised causing injection of molten WE43 alloy to the mould and infiltration of the carbon fibre perform. The last stage of the process was solidification of composite under the high pressure.

2.3. Experimental methods

The samples were prepared for investigation following the Struers specimen preparation system (grinding on abrasive papers with grit from 120 to 1200 and polishing on polishing cloths using the diamond pastes with grain size from 6 μm to 0.25 μm; the Struers OP-AN solution – 0.05 μm Al_2O_3 grains, was used for final polishing of the specimens). To reveal the elements of microstructure samples were etched using Nital solution (3% solution of HNO_3 in ethanol).

The images of microstructure were acquired with Olympus GX71 light microscope, equipped with Olympus DP70 12 Megapixels digital camera, using Olympus AnalySIS FIVE software. The bright field technique was applied. For detailed analysis of microstructure Hitachi S-4200 and Hitachi S-3400N scanning electron microscopes were used. Photomicrographs were registered using imaging techniques with a detection of secondary electrons (SE) and backscattered electrons (BSE). Chemical composition analysis were performed using Thermo NORAN System Six X-radiation EDS (energy dispersive spectroscopy) detectors coupled with scanning electron microscopes, with an accelerating voltage of 15 kV in a high vacuum.

The T6 heat treatment was utilized to allow the additional improvement of the mechanical properties of fabricated composites and evaluation of its influence on their microstructure. The mentioned age-hardening heat treatment typically involves a solution treatment of 8 h at 525°C, hot water quench and subsequent ageing treatment of 16 h at 250°C. Depending on the ageing temperature, a precipitation sequence comprises the formation of β", β', β1 and equilibrium β phases (compositions given in Table 3). The aged microstructure at maximum hardness should contain metastable β' and equilibrium β phases as dispersed precipitates [8,17].

TABLE 3

Compositions of β", β', β_1 and β phases

Phase	Composition
β"	$Mg_3(Y_{0.85}Nd_{0.15})$
β'	$Mg_{12}NdY$ or $Mg_{24}Y_2Nd_3$
β_1	Mg_3RE (RE = Nd, Ce, La, Gd)
β	$Mg_{14}Nd_2Y$

3. Results and discussion

Basing on the conducted investigation of microstructures and the acquired photomicrographs the microstructures of fabricated composites have been compared with the microstructure of non-reinforced WE43 matrix alloy. The microstructures of samples after solution treatment and ageing treatment have been compared with the microstructure of as-cast composite. The results are presented in the following subsections.

3.1. Characterisation of microstructure

The microstructure of WE43 matrix alloy fabricated using pressure infiltration technology (Fig. 1) is a typical microstructure of WE43 casting magnesium alloy. The matrix (a) of the alloy is a solid solution of yttrium in α-Mg. The eutectic compound (b) composed of α-Mg solid solution and equilibrium β phase have been observed in the interdendritic regions and its morphology is typical for WE43 alloy. Small cuboidal particles (c) composed mainly of yttrium and rare earth elements (neodymium and dysprosium) have also been found. The results of EDS analysis are listed in Table 4.

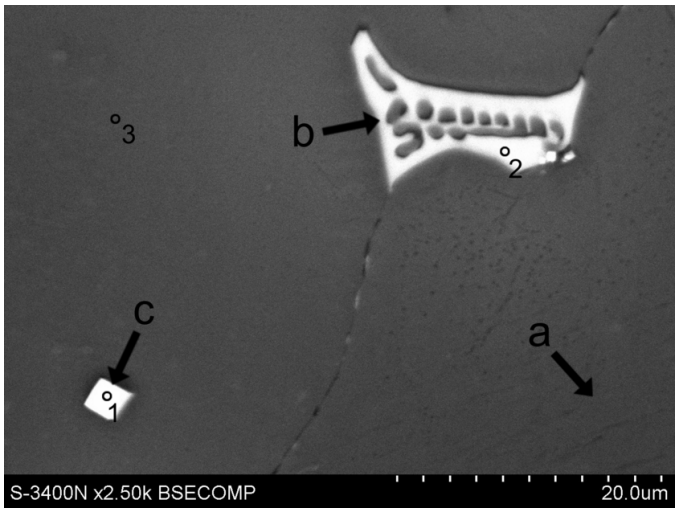

Fig. 1. SEM micrograph of non-reinforced WE43 alloy (2500×)

TABLE 4

Results of EDS analysis of non-reinforced WE43 alloy (atomic %)

	Mg-K	Y-L	Nd-L	Dy-L
Point 1 (c)	6.1	86.1	3.3	4.5
Point 2 (b)	87.2	4.8	8.0	
Point 3 (a)	98.3	1.7		

The microstructure of WE43 magnesium matrix composite reinforced with non-coated plain textile is shown in Figure 2. Microstructure of composite matrix is similar to that of cast WE43 alloy and consists of eutectic compound (α-Mg and equilibrium β phase) and solid solution of yttrium in α-Mg (a). At the interface α-Mg/C_f intermediate layer (c) composed of Mg, Y, Zr, Dy has been observed. Moreover, numerous fine particles (d) containing yttrium and zirconium have been identified in the vicinity of reinforcement (b). Table 5 contains the results of EDS analysis.

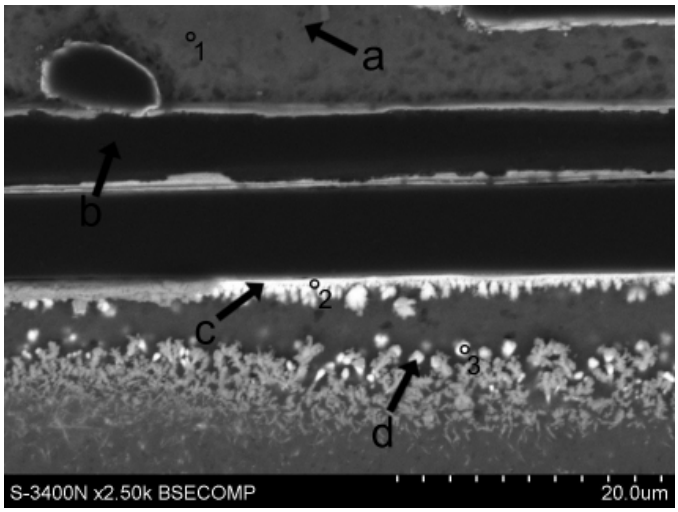

Fig. 2. SEM micrograph of WE43 magnesium matrix composite reinforced with non-coated plain textile (2500×)

TABLE 5

Results of EDS analysis of WE43 matrix composite reinforced with non-coated plain textile (atomic %)

	Mg-K	Y-L	Zr-L	Dy-L
Point 1 (a)	99.6	0.4		
Point 2 (c)	44.7	29.0	23.8	2.5
Point 3 (d)	64.0	20.7	15.3	

In the sample reinforced with nickel-coated plain textile (Fig. 3), lamellar eutectic (c) is visible in the vicinity of reinforcement that was not observed in WE43 composite reinforced with non-coated plain textile. The EDS analysis has shown that this eutectic compound consists of nickel and magnesium. Moreover, small amounts of yttrium and neodymium have been detected. Taking into account the Mg-Ni binary phase diagram and the obtained results it can be assumed that it is Mg_2Ni compound. The lack of alloying elements in the α-Mg matrix (a) has been observed. The matrix-reinforcement (b represents the reinforcement fibres) intermediate layer (d), containing magnesium, nickel and silicon has been identified. The results of EDS analysis are shown in Table 6.

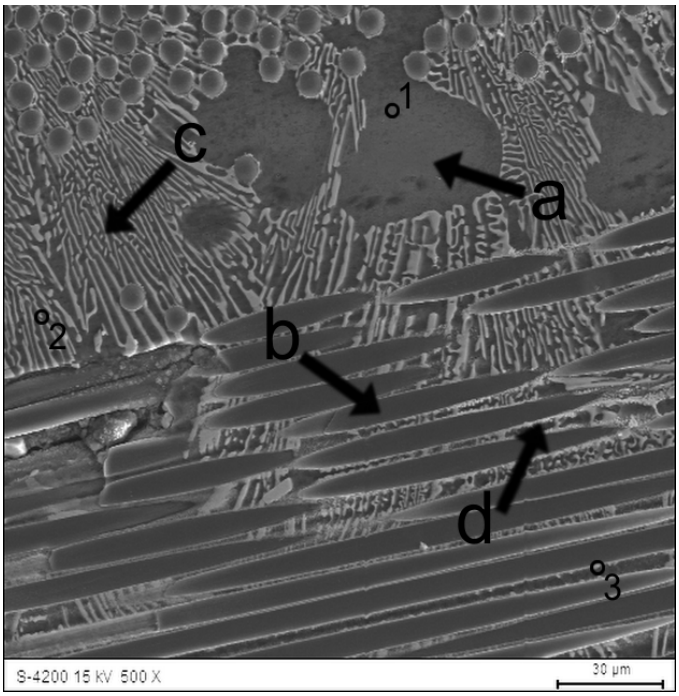

Fig. 3. SEM micrograph of WE43 magnesium matrix composite reinforced with nickel-coated plain textile (500×)

TABLE 6

Results of EDS analysis of WE43 matrix composite reinforced with nickel-coated plain textile (atomic %)

	Mg-K	Si-K	Y-L	Nd-L	Ni-K
Point 1 (a)	100.0				
Point 2 (c)	83.39		0.43	0.91	15.27
Point 3 (d)	79.84	0.64			19.52

The microstructure of composite reinforced with non-coated 3D textile (Figs. 4,5) is comparable with the microstructure of non-reinforced WE43 alloy. In the matrix of composite the α-Mg solid solution (a) and eutectic β phase (b) have been observed. The further investigation revealed the existence of intermediate layer (c) between the matrix and reinforcement fibres (d) that is composed of magnesium, yttrium and rare earth elements (neodymium and dysprosium). The results of EDS analysis are presented in Table 7.

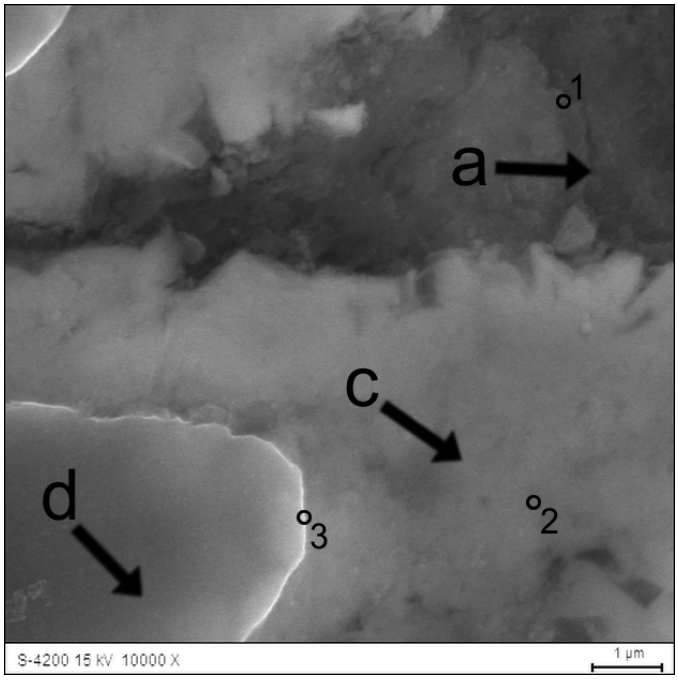

Fig. 4. SEM micrograph of matrix-reinforcement intermediate layer in WE43 matrix composite reinforced with non-coated 3D textile (10000×)

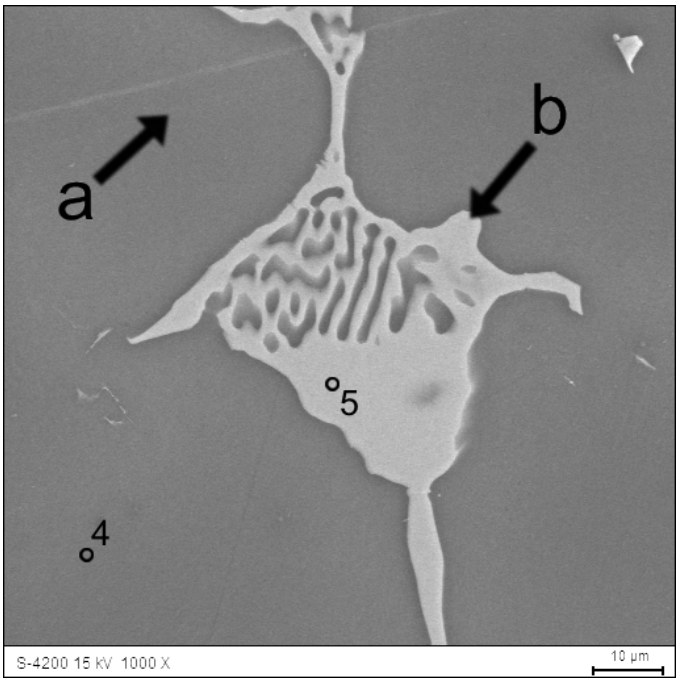

Fig. 5. SEM micrograph of the eutectic compound in WE43 matrix composite reinforced with non-coated 3D textile (1000×)

TABLE 7

Results of EDS analysis of WE43 matrix composite reinforced with non-coated 3D textile (atomic %)

	Mg-K	Si-K	Y-L	Nd-L	Dy-L
Point 1 (a)	86.83	1.56	11.61		
Point 2 (c)	27.43		69.95	2.62	
Point 3 (c)	15.91	0.21	72.84	3.79	7.25
Point 4 (a)	99.08		0.53	0.39	
Point 5 (b)	52.94		8.74	38.32	

3.2. Influence of reinforcement type on microstructure

As a result of the presence of carbon fibres in WE43 alloy, the existence of intermediate layer between reinforcement and matrix is observed. Microscopic observations indicate good bonding between matrix phase and fibres in all examined composites. Obviously, good bonding (adhesion) between matrix phase and reinforcement phase provides transfer of load, applied to the material to the reinforcement phase via the interface. Adhesion is necessary for achieving high level of mechanical properties of the composite.

The mentioned layer is a zone of chemical reaction of reinforcing fibres with the matrix. A detailed analysis of the phase composition of intermediate layer was not performed in this work, whereas the EDS results suggest that it is composed mainly of magnesium, yttrium and rare earth elements. It cannot be excluded that the intermediate layer consists also of carbon. More detailed analysis of chemical and phase composition will be published elsewhere. The thickness of intermediate layers and its chemical composition depends on the kind of fibres. In the sample reinforced with plain textile, the intermediate layer is wider than in the sample reinforced with 3D textile. Moreover, the significant amount of fine precipitates, containing yttrium and zirconium has been observed in the vicinity of reinforcement fibres. It can be expected that formation of a layer enriched in alloying elements will lead to a reduction in content of yttrium and rare earth elements in the matrix composite.

In case of composite with nickel-coated plain textile significant changes of microstructure were observed (in comparison with the microstructure of matrix alloy and other composite samples). The nickel coating of reinforcing fibres has apparently dissolved in the magnesium matrix during the technological process, creating the Mg-Ni intermetallic compound (possibly the Mg_2Ni phase being a constituent of α-Mg+Mg_2Ni eutectic compound). The presence of a significant amount of this compound in the structure of the composite could be the direct reason of composite corrosion resistance deterioration (due to a large difference in electronegativity of Mg and Ni a galvanic corrosion cell is being created).

In all composite samples the segregation of alloying elements to the matrix-reinforcement intermediate layer has been identified. This segregation, causing the decrease of yttrium, neodymium and dysprosium concentration in the matrix, may result in a deterioration of mechanical properties of fabricated composites (decreased number or lack of disperse precipitates of β' and β phases, strengthening the matrix alloy). The presence of silicon could be justified by the possible reaction between the molten matrix alloy and the surface of silicon carbide crucible used in the composites fabrication process.

3.3. Influence of T6 heat treatment on microstructure

The influence of T6 heat treatment on microstructure of WE43 magnesium matrix composite reinforced with carbon fibres has been evaluated in terms of comparison of microstructures of composite samples after solution treatment and ageing treatment with the microstructure of non heat-treated composite. The typical aged microstructure of non-reinforced WE43 alloy at maximum hardness consists of the disperse precipitates of metastable β' ($Mg_{12}NdY$ or $Mg_{24}Y_2Nd_3$) and equilibrium β ($Mg_{14}Nd_2Y$) phases [8,17].

After applied solution treatment the microstructure of composite (Fig. 6) consists of α-Mg matrix (a), reinforcement fibres (b), needle-like precipitates composed of magnesium, yttrium and rare earth elements (c), clusters of fine Zr-Si particles (d), as well as small particles (e) containing magnesium, yttrium and rare earth elements (these precipitates are probably hydrides or oxides, which are formed during casting process and high-temperature heat treatment). Table 8 contains the results of EDS analysis.

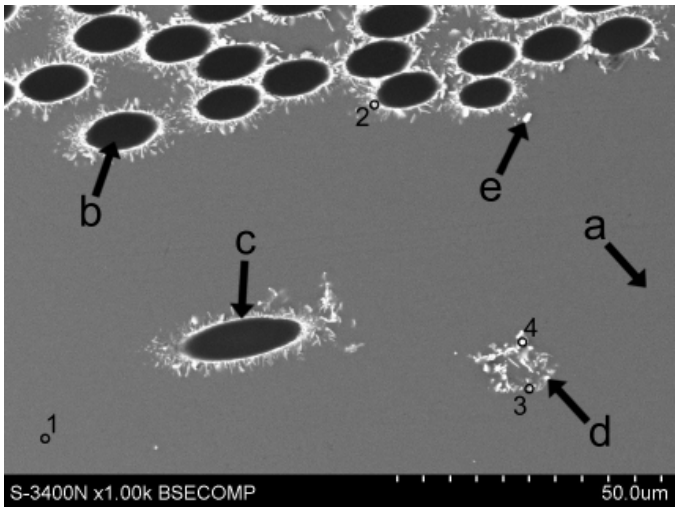

Fig. 6. SEM micrograph of WE43 magnesium matrix composite reinforced with carbon fibres after solution treatment (1000×)

TABLE 8

Results of EDS analysis of WE43 magnesium matrix composite reinforced with carbon fibres after solution treatment (atomic %)

	Mg-K	Y-L	Nd-L	Si-K	Zr-L
Point 1 (a)	100.0				
Point 2 (c)	82.4	15.9	1.7		
Point 3 (d)	87.8			2.5	9.7
Point 4 (d)	33.8			19.1	47.1

The investigation of microstructure of composite sample after ageing treatment (Figs. 7-8) revealed the existence of α-Mg matrix (a), reinforcement fibres (b), fine particles containing magnesium, yttrium and rare earth elements (c) within magnesium grains (some of the mentioned particles contain also traces of silicon), small irregular particles composed of magnesium, yttrium and rare earth elements (d) in the vicinity of reinforcement fibres and the Mg-Y-RE (RE = Nd, Dy) matrix-reinforcement intermediate layer (e). The results of EDS analysis are listed in Table 9.

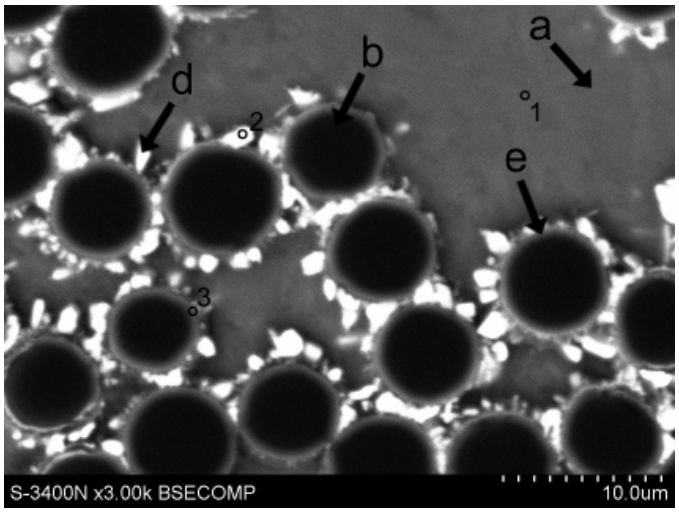

Fig. 7. SEM micrograph of the matrix-reinforcement intermediate layer in WE43 magnesium matrix composite reinforced with carbon fibres after ageing treatment (3000×)

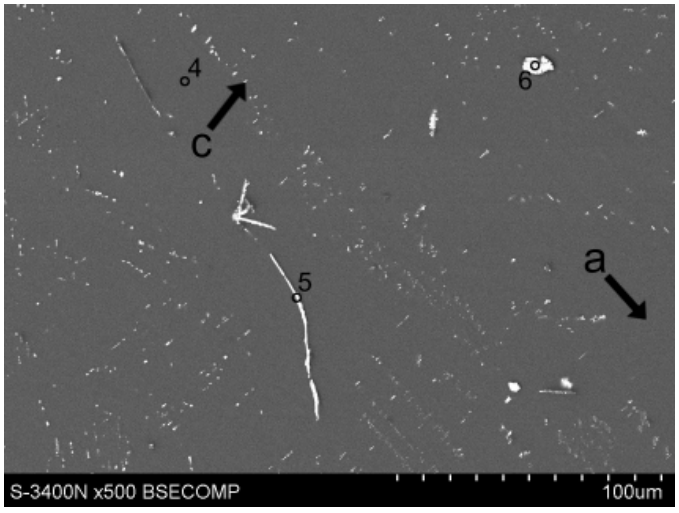

Fig. 8. SEM micrograph of the WE43 matrix after ageing treatment (500×)

The evaluation of the microstructure of a composite sample after solution treatment revealed the presence of Mg-Y-RE needle-like precipitates in the vicinity of reinforcement fibres. This indicates further segregation of alloying elements to the intermediate layer and a progressive reaction between matrix and reinforcement. No α-Mg+β eutectic compound has been observed (it suggests, that the eutectic compound has been dissolved in the matrix). The coagulation of Mg-Y-RE needle-like precipitates has been reported in the composite sample after ageing treatment, however the small amounts of needle-like precipitates have been observed as well. The presence of small Y- and Nd-rich precipitates in the matrix has been reported (Fig. 8 point c). Their size and literature data [15] indicates, that they might be the β phase precipitates. The irregular distribution, small volume fraction and relatively large size of these precipitates shows the invalid process of ageing treatment. It could be justified by the presence of yttrium and neodymium in precipitates found in α-Mg/Cf interfacial boundary (these amounts of Y and Nd do not form the strengthening phases during the precipitation process). The mentioned factors cause, that the mechanical properties of composite matrix could not be improved by application of the heat treatment, typically used for WE43 alloy.

TABLE 9

Results of EDS analysis of WE43 magnesium matrix composite reinforced with carbon fibres after ageing treatment (atomic %)

	Mg-K	Y-L	Nd-L	Gd-L
Point 1 (a)	100.0			
Point 2 (d)	67.7	18.7	10.9	2.7
Point 3 (e)	83.5	4.8	11.7	
Point 4 (a)	100.0			
Point 5 (c)	76.8	1.2	22.0	
Point 6 (c)	72.9	13.8	13.4	

4. Conclusions

- The microstructure of WE43 matrix composites reinforced with non-coated plain textile and non-coated 3D textile consists of solid solution of yttrium in α-Mg (matrix), carbon fibres (reinforcement), equilibrium β ($Mg_{14}Nd_2Y$) eutectic compound and matrix-reinforcement intermediate layer (reaction zone). The composition of intermediate layer is Mg-Y-RE. Additionally the presence of Y-Zr particles in the vicinity of reinforcement has been reported in the sample reinforced with plain textile (wider reaction zone).
- Microstructure of sample reinforced with nickel-coated plain textile consists of α-Mg matrix, carbon fibres (reinforcement), Mg-Ni eutectic compound (traces of yttrium and neodymium have been registered as well) and Mg-Ni-Si matrix-reinforcement intermediate layer. The presence of Mg-Ni eutectic compound is a result of the reaction between the nickel coating and magnesium matrix during a technological process.
- The influence of reinforcement on the microstructure of samples reinforced with non-coated textiles is limited to the existence of matrix-reinforcement intermediate layer (reaction zone) and the segregation of alloying elements (yttrium, neodymium, dysprosium) to this area.
- The fabricated composites could be considered as susceptible to heat treatment. The presence of dispersed precipitates

of β' and β phases, containing magnesium, yttrium and rare earth element has been reported. However the results of applied age-hardening treatment might be unsatisfactory due to the segregation of alloying elements to the matrix-reinforcement intermediate layer, resulting in a formation of small Mg-Y-RE particles in the vicinity of reinforcement (the potential lacks of alloying elements in the matrix causing disorders in formation of disperse precipitates of β' and β phases).

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REFERENCES

[1] W. Hufenbach, M. Andrich, A. Langkamp, A. Czulak, J. Mater. Process. Tech. **175**, 218-224 (2006).
[2] B.L. Mordike, T. Ebert, Mater. Sci. Eng. **A302**, 37-45 (2001).
[3] L. Chen, Y. Yao, Acta Metall. Sin. **27**(5), 762-774 (2014).
[4] M. Svoboda, M. Pahutová, K. Kuchařová, V. Sklenička, K.U. Kainer, Mater. Sci. Eng. **A462**, 220-224 (2007).
[5] H.Z. Ye, X.Y. Liu, J. Mater. Sci. **39**, 6153-6171 (2004).
[6] T. Rzychoń, M. Dyzia, I. Pikos, Solid State Phenom. **211**, 101-108 (2014).
[7] A. Kiełbus, T. Rzychoń, Procedia Eng. **10**, 1835-1840 (2011).
[8] A. Kiełbus, T. Rzychoń, L. Lityńska-Dobrzyńska, G. Dercz, Solid State Phenom. **163**, 106-109 (2010).
[9] D.B. Miracle, Compos. Sci. Technol. **65**, 2526-2540 (2005).
[10] T.P.D. Rajan, R.M. Pillai, B.C. Pai, J. Mater. Sci. **33**, 3491-3503 (1998).
[11] S. Abraham, B.C. Pai, K.G. Satyanarayana, V.K. Vaidyan, J. Mater. Sci. **25**, 2839-2845 (1990).
[12] F. Wu, J. Zhu, Compos. Sci. Technol. **57**, 661-667 (1997).
[13] J. W. Kaczmar, K. Pietrzak, W. Włosiński, J. Mater. Process. Tech. **106**, 58-67 (2000).
[14] Z. Trojanová, Z. Száraz, F. Chmelík, P. Lukáč, J. Alloy. Compd. **504**, L28-L30 (2010).
[15] P. Mengucci, G. Barucca, G. Riontino, D. Lussana, M. Massazza, R. Ferragut, E. Hassan Aly, Mater. Sci. Eng. **A479**, 37-44 (2008).
[16] T. Rzychoń, J. Szala, A. Kiełbus, Arch. Metall. Mater. **57**(1), 245-252 (2012).
[17] K. Davey, S. Bounds, J. Mater. Process. Tech. **63**, 696-700 (1997).