Alloying effect of Li and Y on the strengthening of Mg/T300 composites

S. Kúdela Jr.^{1*}, O. Bajana¹, Ľ. Orovčík¹, P. Ranachowski², Z. Ranachowski²

¹Institute of Materials and Machine Mechanics, Slovak Academy of Sciences, Dúbravská cesta 9, 845 13 Bratislava, Slovak Republic

²Institute of Fundamental Technological Research, Polish Academy of Sciences, Pawinskiego 5B, 02-106 Warszawa, Poland

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Abstract

Composite materials based on MgLi and MgY matrices reinforced with unidirectional T300 carbon fibres (45 vol.%) have been prepared by gas pressure infiltration technique. Nominal Li and Y alloying of Mg matrix were 0.5, 2, and 4 wt.% Li and 7, 13, and 20 wt.% Y, respectively. Back-scattered electron (BSE) observations and electron dispersive X-ray (EDX) analysis have revealed that penetration of lithium and formation of lithium carbide Li_2C_2 inside carbon fibres took place. Bending tests have shown that low Li alloying of Mg matrix (0.5 and 2 wt.% Li) has a favourable effect on the strength of MgLi/T300 composites. Higher Li content (4 wt.% Li) brings a much lower composite strengthening presumably due to degradation of carbon fibres with Li_2C_2 . A negative effect of high yttrium alloying on the strength of MgY/T300 composites is explained by poor adherence of YC₂ interfacial layer to carbon fibres.

Key words: magnesium matrix, magnesium composites, magnesium-lithium matrix, magnesium-lithium composites, magnesium-yttrium matrix, magnesiumyttrium composites, lithium carbide, yttrium carbide, reactive wetting

1. Introduction

To preserve the benefit of the low density of magnesium matrix composites, the most appropriate reinforcements are high-strength carbon fibres (CF) having density close to that of pure magnesium. The common manufacturing method of these composites is the pressure infiltration of carbon fibres preform with molten Mg and/or its alloys [1]. It is a relatively simple and time-saving process; nevertheless, its crucial problem is the formation of a sufficiently strong interfacial bond, enabling the load sharing between the matrix and the fibre. Adhesive bond based on the wetting of fibres with liquid metal is largely preferred. Magnesium does not wet and is chemically intact to CF at temperatures typical for the pressure infiltration ($\sim 700 \,^{\circ}$ C) so that the adhesion needs to be stimulated by chemical reactions [2]. Theoretical models explain this stimulating effect by an increase in adhesion work by additional term including the Gibbs potential of respective chemical reaction [3]. A suitable way seems to be the use of Mg-Al alloys, where the formation of Al₄C₃ and Al₂MgC₂ carbides significantly contribute to the interfacial strength. The problem is that these carbides nucleate and grow on the surface of CF, thereby affecting their structure and reducing the strengthening effect [4] sharply. Therefore other carbide-forming alloying alternatives have been examined, e.g., the use of transitive metals with a strong affinity to carbon (e.g., Zr) [5]. Preparation of these alloys is, however, rather complicated because of negligible solubility of transition metals in magnesium and their very high melting point.

Recently Mg matrix has been alloyed with rare earth elements Y or Gd to modify the interface of CF/Mg composites without affecting CF [6, 7]. In both cases, the segregation of Y or Gd at interfaces takes place forming fine particles Mg_2Y , Mg_24Y_5 , Mg_7Gd or oxidic layers Gd_2O_3 , and MgO. As a result, the interfacial strength has been greatly enhanced,

^{*}Corresponding author: tel. +421 2 32401029; e-mail address: ummskudm@savba.sk

reaching the maximum for 5.7 wt.% Y and 3.2 wt.% Gd. Higher concentrations of Y and Gd resulted in some strength decrease.

The present paper is focused on two topics. The first one is the study of the effect of low lithium alloying of Mg matrix (0.5-4 wt.% Li) on the performance of interfaces and strengthening in Mg/CF composites. Previous studies conducted with highly Li alloyed Mg matrices (8-12 wt.% Li) have revealed the destruction of CF due to excessive formation of lithium carbide Li₂C₂ [8]. Low Li concentrations are expected to minimize the damage of CF and stimulate the fibre/matrix adhesion through a moderate chemical reaction. Such an approach has not been explored until now. The second theme is the study of fibre/matrix interaction in MgY/CF composites based on highly yttrium alloyed magnesium matrix (7, 13, and 20 wt.%)Y). As mentioned above, low yttrium addition to Mg matrix favourably influences interfacial strength in MgY/CF composites [6]. Nevertheless, the formation of yttrium carbide YC₂ at interfaces can greatly influence the strengthening efficiency of composites prepared. Results obtained might, therefore, contribute to a better understanding of the strengthening and failure mechanisms of MgY/CF composites.

2. Experiment

2.1. Mg-Li and Mg-Y matrix alloy preparation

Magnesium-lithium alloys with nominal lithium alloying of 0.5 wt.% Li, 2 wt.% Li, and 4 wt.% Li marked as Mg0.5Li, Mg2Li, and Mg4Li, respectively, have been prepared by fusion of pure magnesium (99.9%, LMT Metalurgie) and lithium (99.0%, Merck) in a mild-steel crucible followed with casting into a cylindric steel mould. Both fusion and casting operations were conducted in the same chamber under an argon pressure of 1 MPa after the previous evacuation (10 Pa). In the same way, there have been prepared magnesium-yttrium matrix alloys using pure magnesium (99.9%, LMT Metalurgie) and yttrium (99.0%, Alfa Aesar) metals with nominal yttrium contents of 7, 13, and 20 wt.%, marked as Mg7Y, Mg13Y, and Mg20Y, respectively.

2.2. Composite fabrication

Composite samples were fabricated by infiltration of PAN-based carbon fibres T300 (Toray) with molten Mg-Li and Mg-Y alloys. Declared tensile strength of T300 carbon fibres is 3530 MPa and Young's modulus 230 GPa [9]. The melt infiltration was carried out by the vacuum – pressure technique in labour autoclave. Preheated fibrous preform $(15 \times 15 \times 70 \text{ mm}^3)$ containing unidirectionally arranged carbon fibres

 $(\sim 45 \text{ vol.\%})$ was immersed under vacuum into molten alloy at 750 °C, and then the argon pressure of 4 MPa was applied for 30 s and 120 s for MgLi and MgY matrices, respectively. The preform was then withdrawn from molten metal under argon pressure and cooled down.

2.3. SEM observations and EDX analysis

The structure of MgLi/CF and MgY/CF composites was examined using the scanning electron microscope (SEM, Jeol JSM 7600F) equipped with Retractable Backscattered Electron Detector (RBEI). Energy-dispersive X-ray spectrometer (EDX) operating at 15 kV was used for elemental analysis. Composite samples were metalographically polished using water-free isopropyl alcohol to a mirror-like finish without any etching. As the structure of MgLi/CF and MgY/CF composites is sensitive to atmospheric humidity, they were exposed to air for a limited time (≤ 1 h) before being SEM and EDX inspected.

2.4. Bending tests

The bending strength has been chosen as a criterion of the strength level instead of tensile strength itself. This seems to be reasonable as the bending failure occurs on the convex tensile strained side of the specimen [2]. Such an approach enables to avoid the problems with clamping forces typical for conventional tensile tests. Three rectangular plates $50\,\times\,10\,\times\,3\,\mathrm{mm^3}$ have been cut from each composite sample in the longitudinal direction to be bend strained in ZWICK Z100 apparatus with a crosshead rate of 1 mm min^{-1} . MgLi/T300 composites have been 3-point bend strained while MgY/T300 composite samples have been subjected to 4-point bending test. Both the bending strength and Young's modulus have been calculated from load vs bend displacement diagrams using standard procedures [10].

3. Results

3.1. Composite structure

Figure 1 shows a typical structure of Mg/T300 composite containing ~ 45 vol.% carbon fibres. There is certain recognizable inhomogeneity in fibres distribution exhibiting fibre-free regions between individual fibre yarns. The fibres within individual yarns are distributed nearly uniformly (each yarn contains about 3000 filaments). Similar features of fibres macro-distribution have also been observed in MgLi/T300 and MgY/T300 composites. Despite poor wettability between carbon fibres and pure magnesium, the fibres are closely bonded with Mg matrix without any sig-



Fig. 1. The overall structure of Mg/T300 composites displaying the CF distribution (SEM).



Fig. 2. Detail of the structure of Mg/T300 composites (SEM).

nificant incoherencies and/or casting defects (Fig. 2). As the carbon-magnesium system is chemically relatively stable, no reaction products occur at interfaces. Formation of MgC₂ and Mg₂C₃ carbides directly from elements is not possible because they are endothermic compounds that start to decompose at 500–650 $^{\circ}$ C [4, 11].

High lithium reactivity with carbon leads to the formation of lithium carbide Li_2C_2 in MgLi/T300 composites. Nature of the fibre attack can be demonstrated by SEM inspection in BSE mode (Fig. 3a) and EDX map of oxygen OK α signal distribution in Mg2Li/T300 composites (Fig. 3b). The main observation is the occurrence of a dark interfacial zone (thickness of 1–2 µm) at the fibre perimeter. EDX analysis reveals that this peripheral zone coincides exactly with the OK α map. We attribute this strong OK α signal to LiOH/Li₂O species produced by hydrolysis of Li₂C₂



Fig. 3. BSE image of the structure of Mg2Li/T300 composite (a) and related $OK\alpha$ map (b).

upon contact with air humidity. Ionic Li_2C_2 is known as hydrophilic compound experiencing the hydrolysis [12]. Accordingly:

$$Li_2C_2(s) + 2H_2O(g) = 2LiOH(s) + C_2H_2(g).$$
 (1)

As a result, $OK\alpha$ signal designates Li_2C_2 occurrence within the fibre cross-section, which makes possible to monitor progress in carbidic reaction qualitatively by EDX. Note that Li is not detectable by the EDX technique. SEM and EDX observations indicate that Li_2C_2 is formed via reactive Li penetration into fibre interior producing continuous peripheral zone.

Other phenomena appearing in the structure of MgLi/T300 composites are bright regions surrounding carbon fibres. We consider them to be thin LiOH/Li₂O layer formed due to Li segregation around surfaces of individual carbon fibres. EDX technique cannot be used for its detection for principal reasons (thin film, Li undetectability). Note that similar effect in pure Mg matrix composites has not been observed.

Structure of highly Y alloyed MgY/T300 com-



Fig. 4. SEM of the structure of Mg13Y/T300 composite displaying needle-like crystals growing radially from CF into the matrix region.



Fig. 5. SEM image of CF cross-section in Mg13Y/T300composite showing thin, compact layer at the fibre periphery and needle-like crystallites growing into matrix region (a) and CF cross-section after sputtering (b).

posites is crucially influenced with excessive formation of yttrium carbide YC₂ at fibre-matrix interfaces. Both YC_2 and Li_2C_2 are ionic carbides that are hydrolyzed by air humidity producing both hydroxide and gaseous hydrocarbons according to reaction scheme (1). Figure 4 shows an SEM image of the structure of Mg13Y/T300 composite displaying needle-like (probably lamellar) reaction products growing radially from the interface into the matrix region. Detail of interfacial region in Fig. 5a reveals the presence of thin, compact product (thickness of $0.3-0.5 \,\mu\text{m}$) at the fibre periphery, which assists as a substrate for the growth of needle-like crystallites. The latter have apparently been formed additionally during contact with air as demonstrated by their disappearance after sputtering (Fig. 5b). It is also seen that the compact interfacial reaction product is prone to break away from the fibre, thus generating the interfacial cracks. These defects can critically influence the strengthening efficiency of carbon fibres and failure of MgY/T300 composites.

Interfacial chemistry in MgY/T300 composites can be demonstrated by EDX inspection of Mg20Y/T300composites in which very strong attack of CF has happened (Fig. 6). BSE image shows the massive formation of reaction product as a compact zone around individual fibres (thickness of $1-2\,\mu\text{m}$) and also the needle-like phase growing into the matrix region. EDX map suggests that both these products consist only of O and Y and are thought to be oxidic and/or hydroxide compounds produced by YC₂ hydrolysis. Magnesium apparently does not participate in the interfacial reaction. These observations suggest that yttrium segregates largely around the surface of the fibres to form yttrium carbide YC_2 as an authentic interfacial product which is additionally hydrolytically decomposed with air humidity.

One can conclude that both lithium and yttrium as alloying elements of Mg matrix react vigorously with carbon fibres during the melt infiltration process producing discrete Li₂C₂ and YC₂ phases. Although the chemical nature of both products is very similar (both are hydrophilic ionic compounds), their formation runs in different ways. Li_2C_2 is formed inside CF without disturbing their integrity significantly (at least for low Li matrix alloying) while yttrium reacts with outer fibre surface creating continuous YC₂ barrier at fibre/matrix interfaces exhibiting, however, poor adhesion with the fibre.

3.2. Bending tests

Representative curves stress vs deflection of Mg and MgLi matrix composites obtained from 3-point bending test are shown in Fig. 7. Bending strength and Young's modulus have been calculated using standard procedures from load-displacement diagrams, i.e., from maximum load value and a linear segment



Fig. 6. BSE image of the structure of Mg20Y/T300 composite (a), related EDX maps of yttrium YK α (b), oxygen OK α (c), and magnesium MgK α (d).

Table 1. Bending strength and Young's modulus values of Mg/T300, MgLi/T300 composites (3-point bending test), and MgY/T300 composites (4-point bending test)

Composite	Young's modulus (GPa)	$E_{\rm ROM}$ (GPa)	$E/E_{\rm ROM}$	Bending strength (MPa)
Mg/T300	108	128	0.84	828
Mg0.5Li/T300	121	128	0.94	934
Mg2Li/T300	119	128	0.93	1090
Mg4Li/T300	108	128	0.84	869
Mg7Y/T300	125	-	_	608
Mg13Y/T300	119	_	-	609
Mg20Y/T300	107	-	-	526

of bending plot, respectively, as a mean value taken from 3-bending samples (Table 1). Apart from some nonlinear effects in the initial part of flexural curves, the diagram stress vs deflection can conventionally be divided into three sections. In the first stage, the deflection increases linearly with the stress, thus indicating linear elastic behaviour. In the second stage, the deformation curve deviates from the linearity, which can be interpreted as the onset of inelastic phenomena. In the third stage, the stress falls after reaching its maximum, which indicates overall composite failure.

Stress vs deflection curves of Mg and MgLi matrix composites exhibit different slope in elastic deformation range which is reflected on different Young's moduli values. As seen in Table 1, low Li alloyed composites (Mg0.5Li and Mg2Li matrices) show significantly higher Young's moduli than those pure Mg-based while further increase in Li alloying (Mg4Li matrix) brings Young's modulus down to the level of



Fig. 7. Representative stress-strain curves of Mg/T300, Mg2Li/T300, and Mg4Li/T300 composites obtained from 3-point bending test.



Fig. 8. Representative stress-strain curves of Mg7Y/T300, Mg13Y/T300, and Mg20Y/T300 composites obtained from 4-point bending test.

unalloyed Mg matrix composites. Provided that elastic strains of the fibres and the matrix are equal theoretical Young's modulus can be calculated using the rule of mixture (ROM). A calculation has been performed for fibres volume fraction of 45 vol.%. There is to notify that Young's moduli of Mg and MgLi alloys are identical (45 GPa) [13]. Comparison of experimental Young's moduli with theoretical ones ($E/E_{\rm ROM}$ ratio) enables, therefore, evaluate the performance of interfaces in elastic deformation range. Application of this criterion confirms the positive effect of low Li content in MgLi matrix on the composite strengthening while higher Li alloying results in the drop in strengthening efficiency apparently due to degradation of CF caused by excessive Li₂C₂ formation.

The bending strengths of Mg/T300 and MgLi/T300 composites roughly correlate with Young's mod-

ulus values: the higher Young's modulus, the higher the bending strength. With the accumulation of decohesion events, the slope of bending curves of Mg and Mg2Li matrix composites gradually decreases until reaching the load maximum and then falls quite rapidly due to overall composite failure. Different fracture behaviour is apparent on the bending diagram of Mg4Li matrix composites. The featureless local stress maximum occurs in a rather broad plateau, and subsequent composite failure propagates slowly keeping still quite a high loading level. This manifests considerably higher consumption of fracture energy than that taking place in the failure of Mg and Mg2Li matrix composites.

Representative load vs displacement curves of 4-point bend strained MgY/T300 composites with high Y alloying are shown in Fig. 7. Determined strength parameters (Young's modulus, bending strength) are listed in Table 1 as mean values obtained from 3 bending experiments. It can be seen that the bending strengths of both MgY/T300 composites are much lower compared to that of Mg/T300 composites, thus demonstrating the detrimental effect of Y addition on the strength and fracture behaviour. Apart from little signs of inelasticity in Mg7Y/T300 composite other MgY/T300 composites show the symptoms of premature failure already in the elastic deformation range. The critical role may apparently play decohesion effects at interfaces observed in the structure of MgY/T300 composites (Fig. 5a).

3.3. Fractography

The fracture surface of Mg/T300 composites provides a messy picture in which besides the single fibre pull-out, there are also emerged the fibre bundles consisting of several tens fibres (Fig. 9a). Detail of the fibre bundle reveals that individual fibres are loosely put together with some matrix metal residues (Fig. 9b). This suggests that the failure of Mg/T300 composite has been governed with a double pull-out effect: the first one is the pull-out of fibre bundles with subsequent pull-out of single fibres from the bundle. Rather different features exhibit the failure of MgLi/T300 composites. The fracture surface of this composite is also characterized by the pull-out of fibre bundles in which, however, the single fibres are joined with the matrix alloy (Fig. 10a). Detail of the tip of the fibres bundle reveals that nearly flat fibre cross-fracture has occurred (Fig. 10b). Flat nature of fibre cross-fracture and absence of fibre pull-out are generally considered as the signs of fairly strong interfacial bond in metal matrix composites. Hence, the fractographic inspection suggests that the failure of Mg/T300 composites has occurred in "bundle pull-out + fibre pullout" mode while MgLi/T300 composites have failed



Fig. 9. SEM of the fracture surface of Mg/T300 composite demonstrating both the single fibres pull-out and fibre bundles pull-out (a) and detail of the top of fibres bundle (b).

in combined "bundle pull-out + fibre cross-fracture" mode.

4. Discussion

Lithium, as an alloying element, has been added to magnesium matrix to initiate its reaction with carbon fibres, thus stimulating the interfacial bond. As a result, a significant increase in bending strength and Young's modulus has occurred in MgLi/T300 composites if the lithium amount is low ($\leq 2 \text{ wt.\%}$ Li). Higher lithium content causes considerably lesser composite strengthening. No recognizable reaction layer has been formed at interfaces so the stronger interfacial bond should be ascribed to improved wetting of CF due to chemical reaction. Although the relationship between wetting and reactivity is still not satisfactorily understood, there exists a theoretical model describing the effect of chemical reaction on the wetting angle [14]:



Fig. 10. SEM of the fracture surface of Mg2Li/T300 composites displaying fibre bundles pull-out (a) and detail of the top of fibres bundle (b).

$$\cos\Theta = \cos\Theta^{\circ} - (\Delta\gamma_{\rm r}/\gamma_{\rm LV}) - (\Delta G_{\rm r}/\gamma_{\rm LV}), \quad (2)$$

where Θ° is the wetting angle in the reaction absence, $\gamma_{\rm LV}$ is the liquid-vapour interfacial energy, $\Delta \gamma_{\rm r}$ the change in interfacial energies due to the chemical reaction, and $\Delta G_{\rm r}$ is free energy change per unit area released by the reaction of materials in the immediate vicinity of metal/substrate interface. As for the practical use of relationship Eq. (2), the crucial difficulty is linked with the correct determination of $\Delta \gamma_{\rm r}$ and $\Delta G_{\rm r}$ parameters [15].

As no precipitation phenomena can occur in binary Mg-Li alloys and direct formation of MgC₂ and Mg₂C₃ carbides is thermodynamically forbidden, lithium carbide Li₂C₂ is the only reaction product in Mg-Li-C system [16, 17]. Li₂C₂ has been detected by EDX indirectly through OK α signal on the fibre cross-section either as peripheral margin in Mg2Li/T300 composites or whole fibre cross-section coverage in Mg4Li/T300 composites. The presumption that oxidic layer on the

cross-section of lithium affected CF is associated with Li_2C_2 presence has been confirmed experimentally by Auger analysis [18]. Lithium is thought to penetrate inside the fibres as a monoatomic gas via Knudsen's diffusion through interconnected fibre porosity. PANbased carbon fibres (density of $1.76\,\mathrm{g\,cm^{-3}}$) contain about 18 % microvoids at the nanometer scale, mainly due to the removal of H and N atoms on the fibre processing [19]. It is noteworthy that relatively strong MgK α signal distributed uniformly over the entire cross-section of MgLi melt affected T300 fibres has been detected by EDX which is indicative of percolation of gaseous Mg into the bulk of CF. Similar observation has also been reported by other authors [2]. Both Li and Mg are known as metals with high evaporating capacities [20]. In contrast, negligible MgK α signal has been detected in dense pitch-based GRA-NOC carbon fibres (density of $2.2 \,\mathrm{g \, cm^{-3}}$) after being infiltrated with molten Mg2Li alloy [18].

Formation of Li_2C_2 proceeds by destroying of graphene hexagons via localization of two free electron pairs at carbon atoms thus giving rise to highly ionic Li_2C_2 product consisting of acetylide anion $|C \equiv C|^{2-1}$ and Li^+ cation [12]. There was reported orthorhombic crystal structure for Li₂C₂ at room temperature that transforms to cubic phase at about $500 \,^{\circ}\mathrm{C}$ [17, 21]. As Li_2C_2 is structurally incoherent with the turbostratic graphitic structure of PAN-based T300 fibres, its accumulation in carbon fibres causes their degradation. Excessive Li₂C₂ production may, therefore, be a reason for lower strengthening efficiency of carbon fibres in Mg4Li matrix composites. On the other hand, higher lithium alloying of h.c.p. magnesium matrix increases its plasticity by activation of pyramidal slip planes wherein solid solution strengthening is insignificant up to $\sim 5 \text{ wt.}\%$ Li [22]. This can be considered a reason for higher fracture energy of Mg4Li matrix composites compared to those pure Mg and Mg2Li alloy based.

Previous studies concerning carbon fibres reinforced MgY matrix composites showed that Y alloying enhanced greatly interfacial strength. Even adding of 1 wt.% Y increased bending strength of composites studied by 27 % [23]. Interfacial strength reached its maximum at 5.7 wt.% Y and further increase of yttrium content to 8.5 wt.% caused only a little weakening [6]. The authors did not mention, however, the occurrence of yttrium carbide despite sufficiently high thermodynamic potential for YC_2 formation at temperatures of 400–1000 $^{\circ}\mathrm{C}$ [24]. Our study is focused on higher Y concentration (7-20 wt.% Y), and bending tests reveal that bending strength of highly Y alloyed Mg matrix composites is well below the strength level of unalloyed Mg matrix ones. Structural investigations clearly indicate that thick interfacial carbidic layer formed in highly yttrium alloyed MgY/T300 composites shows poor adherence to carbon fibre creating interfacial cracks that assist in premature composite failure. A gap between the carbidic layer and the fibre is probably caused by volume changes accompanying YC_2 formation (Pilling-Bedworth rule). High Y alloying of the magnesium matrix should cause considerable solution and precipitation hardening [25]. However, because of premature composite failure, the contribution of higher matrix strength is insignificant.

5. Conclusions

Mg-Li and Mg-Y matrix composites reinforced with T300 carbon fibres (~45 vol.%) have been prepared by the vacuum-pressure infiltration at 750 $^{\circ}C/30$ s and 750 $^{\circ}C/120$ s, respectively, to evaluate the effect of matrix alloying on the structure, bending strength and Young's modulus of composites prepared. Results obtained are as follows:

1. Structure of Mg-Li matrix composites (0.5, 2, and 4 wt.% Li) reinforced with T300 carbon fibres is characterized by penetration of Li into fibre bulk producing lithium carbide Li_2C_2 inside the fibres. Li_2C_2 has been monitored on fibre cross-sections by EDX through OK α signal by utilizing the hydrolysis of Li_2C_2 with air humidity to LiOH. No visible reaction products occur at interfaces.

2. Low lithium alloying (0.5 and 2 wt.% Li) of Mg matrix results in considerably higher bending strength and Young's modulus of MgLi/T300 composites in comparison with those of unalloyed Mg/T300 composites. Higher lithium content (4 wt.% Li) causes much lower strengthening slightly above that of Mg/T300 composites. This is attributed to the degradation of the CF structure due to excessive Li₂C₂ formation in the fibre bulk.

3. The prominent feature of the structure of highly yttrium alloyed (7, 13, and 20 wt.% Y) MgY/T300 composites is massive segregation of Y around carbon fibres. Interfacial product has been found as a barrier around CF, and it acts as a substrate for the growth of needle-like crystals into the matrix region. Interfacial products have been identified by EDX as oxidic Y-O phase. Authentic interfacial product appears, however, to be yttrium carbide YC₂ experiencing the hydrolysis with air humidity to form Y-O phase.

4. MgY/T300 composites based on highly yttrium alloyed Mg matrix show considerably lower bending strength than unalloyed Mg/T300 ones. There are signs of premature composite failure on stress vs deflection curves. A reason for that are considered interfacial defects connected with poor adherence of carbidic layer to CF.

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