PHYSICS OF STRENGTH AND PLASTICITY

PACS numbers: 61.66.Dk, 62.20.-x, 68.37.Hk, 81.05.Je, 81.05.Ni, 81.20.Ev, 81.70.Jb

Characterization and Properties of Mg–TiO₂ Composites Produced *via* Ball Milling and Powder Metallurgy

B. Stalin, V. S. Vidhya*, M. Ravichandran**,
A. Naresh Kumar, and G. T. Sudha***

*Anna University, Department of Mechanical Engineering,
Regional Campus Madurai,
Madurai-625019, Tamil Nadu, India*

**Ananda College, Department of Chemistry,
Devakottai-630303, Tamil Nadu, India*

***K. Ramakrishnan College of Engineering,
Department of Mechanical Engineering,
Trichy-621112, Tamil Nadu, India*

****Government Polytechnic College,
Department of Mechanical Engineering,
Peelamedu, Coimbatore-641014, Tamil Nadu, India*

The new magnesium matrix composites reinforced with different weight fractions (0, 4, 8, 12) of TiO₂ particles are fabricated by powder metallurgy. The ball milled powders are compacted and sintered for further mechanical and characterization studies. With the increasing weight percentage of TiO₂ particles, the particles gradually show more homogeneous distribution in the matrix. Compared with the matrix, the compressive strength of the composites is improved. With the increasing TiO₂ contents from 0 to 12% wt., the ultimate compressive strength increases from 82 to 158 MPa, respectively, while the corresponding strain decreases. The improvement of the ultimate compressive strength and hardness of the Mg–TiO₂ composites is due to the homogeneous microstructure of TiO₂ in Mg matrix. Energy dispersive X-ray spectroscopy and scanning electron microscopy are used to study the morphology and distribution of reinforcements in the matrix material. An identical distribution of TiO₂ parti-

---

Corresponding author: B. Stalin
E-mail: stalin1312@gmail.com

Citation: B. Stalin, V. S. Vidhya, M. Ravichandran, A. Naresh Kumar, and G. T. Sudha, Characterization and Properties of Mg–TiO₂ Composites Produced *via* Ball Milling and Powder Metallurgy, *Metallofiz. Noveishie Tekhnol.*, **42**, No. 4: 497–509 (2020), DOI: [10.15407/mfint.42.04.0497](https://doi.org/10.15407/mfint.42.04.0497).

cles is obtained throughout the matrix in the Mg–TiO₂ composites.

Key words: magnesium based composites, microstructure, mechanical properties, powder metallurgy.

Нові композиційні матеріали з магнієвою матрицею, зміцнені частинками TiO₂ різних масових часток (0, 4, 8, 12), виготовлялися методом порошкової металургії. Порошки, отримані в кульовому млині, ущільнювалися та спікалися для подальшого вивчення механічних властивостей і характеристик. Зі збільшенням масового відсотка частинок TiO₂ вони демонструють поступово зростаючу однорідність їх розподілу в матриці. Міцність на стиск композитів покращується в порівнянні з матрицею. При збільшенні вмісту TiO₂ від 0 до 12% мас. межа міцності при стисненні збільшується відповідно з 82 до 158 МПа, при цьому відповідна деформація зменшується. Зростання межі міцності при стисненні і твердості композитів Mg–TiO₂ обумовлено однорідною мікроструктурою TiO₂ в магнієвій матриці. Енергодисперсійна рентгенівська спектроскопія і скануюча електронна мікроскопія використовуються для вивчення морфології і розподілу зміцнюючого матеріалу в матриці. Однаковий розподіл часток TiO₂ було отримано по всій матриці в композитах Mg–TiO₂.

Ключові слова: композити на основі магнію, мікроструктура, механічні властивості, порошкова металургія.

Новые композиционные материалы с магниевой матрицей, упрочнённые частицами TiO₂ различных массовых долей (0, 4, 8, 12), изготавливались методом порошковой металлургии. Порошки, полученные в шаровой мельнице, уплотнялись и спекались для дальнейшего изучения механических свойств и характеристик. С увеличением массового процента частиц TiO₂ они демонстрируют постепенно возрастающую однородность их распределения в матрице. Прочность на сжатие композитов улучшается по сравнению с матрицей. При увеличении количества TiO₂ от 0 до 12% масс. предел прочности при сжатии увеличивается соответственно с 82 до 158 МПа, при этом соответствующая деформация уменьшается. Улучшение предела прочности при сжатии и твёрдости композитов Mg–TiO₂ обусловлено однородной микроструктурой TiO₂ в магниевой матрице. Энергодисперсионная рентгеновская спектроскопия и сканирующая электронная микроскопия использовались для изучения морфологии и распределения упрочняющего материала в матрице. Одинаковое распределение частиц TiO₂ было получено по всей матрице в композитах Mg–TiO₂.

Ключевые слова: композиты на основе магния, микроструктура, механические свойства, порошковая металлургия.

(Received February 13, 2019; in final version, February 12, 2020)

1. INTRODUCTION

Composite materials are promising essentially in response to the exceptional demands of technology due to rapidly advancing activities in aircrafts, aerospace and automotive industries [1]. These materials have low specific gravity that makes their properties, particularly superior in strength and modulus to many traditional engineering materials such as metals [2]. As a result of intensive studies into the fundamental nature of materials and better understanding of their structure property relationship, it has become possible to develop new composite materials with improved physical and mechanical properties. These new materials include high performance composites such as Polymer matrix composites, Ceramic matrix composites and Metal matrix composites [3].

MMCs have inducing a powerful concentration in topical times for possible applications. MMCs have very less weight, elevated strength, and stiffness and exhibit greater resistance to corrosion, oxidation and wear. For automotive application, the fatigue resistance is an essential property of Magnesium based metal matrix composites (Mg-MMC). Because their superior properties such as high strength to weight ratio, high hardness, superior wear and corrosive resistance, high temperature and thermal shock resistance, high specific modulus, high fatigue strength has been improved.

Magnesium has the potential to replace steel, aluminium alloy, and plastic-based materials. Initially, due to the high price, there were limited applications of magnesium alloy. But recently, the interest in magnesium alloys has increased because of the gradually decreasing cost of magnesium alloys. Magnesium alloys have better solidification characteristics over other cast metals such as copper and aluminium alloys [4]. The addition of reinforcement of magnesium and its alloys improves its strength, and stiffness. These materials have very low flexibility compared with other materials and hence limit its broad applications [5–8].

Magnesium is lighter in weight than aluminium and titanium [9]. Casting technologies are being inadequate for the manufacture of materials, since those materials have structural defects as explained elsewhere [10].

From the literature review, it is concluded that Mg composites with the intrinsic compensation of the metal casting process, capitulate to provide the product. A number of efforts have been made to explore the effects of reinforcements on the mechanical, the tribological and the microstructural behaviour of magnesium. Initially, reinforcements in the form of carbides (SiC, B₄C, ZrC, TiC), borides (ZrB₂, TiB₂), nitrides (TiN, ZrN, BN, AlN), and metals (Mo, Cu, Ti, Ni) were added to magnesium [11–15].

In the available literature, one cannot find reports on attempts to study the effects of primary processing techniques, on the microstructure and tribological properties of TiO_2 reinforced magnesium composites [16]. This research paper is to elaborate a novel approach to the synthesis of Mg-TiO_2 by ball milling and the effect of TiO_2 reinforcements on structural and mechanical properties.

2. EXPERIMENTAL DETAILS

Magnesium powder of 99.8% purity supplied by Kemphasol, Mumbai, India was used for the matrix material and TiO_2 (rutile) powders supplied by the Acechemie (India) were used as the reinforcement material.

The SEM image of the as received Mg powder and TiO_2 powders are shown in Fig. 1, *a-d* to understand the morphology of powders.

Fig. 1. SEM and EDS images of Mg (*a*, *b*) and TiO_2 powders (*c*, *d*).

Fig. 2. Punch and die (a). Compaction process setup (b).

The figure shows the absence of forged diffraction, which indicates the crystallographic purity of the material.

TiO₂ is blended with matrix material in the weight percentages to yield the different composites such as, Mg-0% wt. TiO₂, Mg-4% wt. TiO₂, Mg-8% wt. TiO₂, and Mg-12% wt. The required mass of Mg and TiO₂ were accurately weighed in an electronic weighing machine and blended in a ball mill for 2 hours to get uniform mixture of composite powders. The balls and powder weight ratio of 10:1 with the speed of 100 rpm.

The time period was 2 hours and the furnace cooling was done on the green samples. SEM was used to analyse the microstructure of the composites. EDS analysis was done to ensure the compositions.

The hardness measurements were done on the polished samples using a Brinell hardness tester. The specimens for compression tests were prepared as per ASTM E9 standard and the computerized UTM was used to determine the compressive strength of the composites at room temperature.

Fig. 3. Compacted Mg and Mg-4% wt. TiO₂ reinforced composite samples.

3. RESULTS AND DISCUSSION

3.1. Characterization of Composite Powders. SEM Investigations

SEM photograph of the as-prepared mixture of Mg and TiO_2 powder particles in various weight percentages are shown in Fig. 4, *a-c*. It can be seen that the TiO_2 particles distributed evenly within the Mg

Fig. 4. SEM images of Mg-TiO₂ composite powders (*a-f*).

matrix. An agglomeration of TiO₂ in the mixture is not found.

Figures 4, *a* and *b* show the SEM image of Mg-4% wt. TiO₂ mixed composite powders and it illustrate the uniform distribution of TiO₂ powder particles in Mg matrix. It can be also seen, that the uniform distribution of TiO₂ in Mg composite blends in Fig. 4, *b*. From Fig. 4, *c* and *d*, it can be also observed that the cold welding mechanism of matrix and reinforcement during the mechanical alloying. Figures 4, *e* and *f* show the presence of more weight percentages of TiO₂ particles in the matrix and this will enhance the mechanical properties of the composite samples. The uniform distribution of particles observed even though the weight percentage of the reinforcement is high (12% wt.).

Fig. 5. EDS analysis of Mg-TiO₂ composite powders: Mg-4% wt. TiO₂ (*a*); Mg-8% wt. TiO₂ (*b*); Mg-12% wt. TiO₂ (*c*).

3.2. Characterization of Composite Powders. EDS Investigations

Compositional analysis of the Mg and TiO_2 milled composite powders were analysed by energy dispersive analysis using EDS. The peaks corresponding to the Mg and TiO_2 are shown in Fig. 5 for the Mg and TiO_2 milled composite powders. The large number of peaks with highest intensity is pertained to Mg which confirms the major contents. The observed weight percentage of titanium-di-oxide content reveals that, it is well incorporated into Mg matrix. Figures 5, *a-c* show the EDS analysis of Mg-4% wt. TiO_2 , Mg-8% wt. TiO_2 , and Mg-12% wt. TiO_2 composites, respectively. From these results it is observed that the change in the intensity of peak for the increase in weight percentage of titanium dioxide content in the magnesium matrix.

3.3. Properties of Mg- TiO_2 Composites

3.3.1. Effect of TiO_2 on the Density and Porosity of Mg Matrix Composite

Figure 6 shows the effect of TiO_2 addition on the density of Mg composite. The density of Mg without the addition of TiO_2 is only about 1.69 g/cm^3 . The density increases with increase of doping amount from 4 to 12% wt. of TiO_2 . The reason for the increase in density is due to the addition of high density reinforcement in the low density matrix materials. Proper mixing of matrix and rein-

Fig. 6. Effect of TiO_2 addition on the density of Mg matrix composite.

forcement and sintering of the samples ensure the increase in density of the composite samples than the pure matrix material.

Irrespective of the particle size, the porosity of the composite is observed to be increasing with the increase in the weight percentage of the addition of TiO₂ particles in Mg matrix is shown in Fig. 7. The proper milling parameters make the TiO₂ particles to disperse uniformly in the magnesium matrix. However the different density of matrix and reinforcement particles leads to increased porosity with the increase in weight percentage of reinforcement [17]. The size of the TiO₂ particle is less than the matrix particle size. Hence the reinforcement particles occupy the pores and enhancing the density of the composite for increasing weight percentage of the TiO₂ particles.

3.3.2. Effect of TiO₂ on the Hardness of Mg Matrix Composite

Figure 8 shows the hardness of Mg matrix reinforced with different weight fractions of TiO₂. It can be seen that the hardness of Mg-TiO₂ increases with an increase in the amount of TiO₂, maximum hardness was 74 BHN and it resulted from the sample containing 12% wt. of TiO₂ particles. This may be due to incorporation of TiO₂ particles, which mired the movement of matrix dislocation in the composite leading to increased strength and hardness. Also the hardness of TiO₂ powders is more if compare with the hardness of Mg matrix material. The addition of a hard nature of TiO₂ into the

Fig. 7. Effect of TiO₂ addition on the porosity of Mg composite.

soft Mg matrix increases the hardness of the composite. This hard TiO_2 particles resist the ball indentation during the testing.

3.3.3. Effect of TiO_2 on the Compressive Strength of Mg Matrix Composite

Micron sized particles are normally used to improve the metal ten-

Fig. 8. Effect of TiO_2 addition on the hardness of Mg composite.

Fig. 9. Stress-strain graphs of Mg based composite.

sile properties. However, the ductility of the composite declines significantly with an increase in weight fractions of TiO₂.

The stress–strain behaviour of all samples has been tested is shown in Fig. 9. The Figure shows that the ductility decreases with further increase in reinforcement weight percentage. It is apparent that there are two factors influencing the strength of the composite: (1) the reinforcing particles, increasing the strength of the material by simply load transfer, which depends on the integrity at the particle/matrix interface and dislocation motion; (2) the inhibition of plastic relaxation at the particle/matrix interface [18]. In this study, the uniform distribution of TiO₂ particles in Mg matrix leads by the lower air gaps between grains resulting from the low degree of porosity using powder metallurgy route. The low degree of porosity is causing the great enhancement in compressive strength. The strong multi-directional grain refinement and thermal stress also play a great role in improving the strength of the Mg/TiO₂ composites. The grain refined strengthening effect of the TiO₂ particles is improved with the increases in weight fraction due to they act as the heterogeneous nucleation catalyst for the matrix [19, 20]. The weakening factors of the mechanical properties might be the reason for this including the porosity.

Figure 10 shows the compressive strength of Mg and TiO₂ reinforced Mg matrix composites. The increase of compressive strength of the composite at room temperature along with an increase in the TiO₂ particles from 4 to 12% wt. has been noted. The measured

Fig. 10. Effect of TiO₂ addition on the compressive strength of Mg composite.

maximum compressive strength of the composite sample was 160 MPa at 12% wt. of TiO_2 . According to the experimental results, a significant enhancement in strength is noted initially when the TiO_2 particulate is added; however, the further increase in the amount of TiO_2 particles leads to an improvement in strength values.

4. CONCLUSION

Mg- TiO_2 composites were effectively manufactured through ball milling and powder metallurgy method and effects of TiO_2 on the properties and microstructure were studied. The following conclusions were drawn from the above experimental investigations; SEM images of ball milled Mg matrix composite powders show the homogenous distribution of the TiO_2 in the matrix. The inclusion of TiO_2 in magnesium matrix increases the density and increase the porosity. The EDS patterns of the milled composite powders confirm the occurrence of the Mg matrix and TiO_2 reinforcement phase. SEM analysis of milled composite powders evident the uniform distribution of reinforcement in the matrix. The improved hardness and compressive strength were obtained for the addition of 12% wt. of TiO_2 in Mg matrix for sintered samples.

REFERENCES

1. M. Dinesh, and R. Ravindran, *Metallofiz. Noveishie Tekhnol.*, **39**, No. 11: 1497 (2017).
2. M. Ravichandran, A. Naveen Sait, and V. Anandakrishnan, *Mater. Res.*, **17**, No. 6: 1489 (2014).
3. V. Mohanavel, K. Rajan, and M. Ravichandran, *J. Mater. Res.*, **31**, No. 24: 3824 (2016).
4. A. A. Luo and K. Sadayappan, *Am. Foundry Soc.* (Schaumburg: IL: 2011), p. 29.
5. K. U. Kainer and F. Buch, *Magnesium Alloys and Technology* (Germany, Weinheim: Wiley-VCH: 2003), p. 1.
6. D. J. Lloyd, *Int. Mater. Rev.*, **39**: 1 (1994).
7. G. E. Dieter, *Mechanical Metallurgy* (USA. New York: McGraw-Hill: 1986).
8. Y. N. Wang and J. C. Huang, *Mater. Chem. Phys.*, **81**: 11 (2003).
9. S. Uganda, M. Gupta, and S. K. Sinha, *Compos. Struct.*, **72**, No. 2: 266 (2006).
10. S. Sankaranarayanan, S. Jayalakshmi, G. Manoj, and A. S. Hamouda, *Metals*, **2**, No. 3: 274 (2012).
11. X. J. Wang, K. Wu, H. F. Zhang, W. X. Huang, H. Chang, W. M. Gan, M. Y. Zheng, and D. L. Peng, *Mater. Sci. Eng. A*, **465**: 78 (2007).
12. B. L. Mordike and T. Ebert, *Mater. Sci. Eng. A*, **302**: 37 (2001).
13. S. F. Hassan, K. F. Ho, and M. Gupta, *J. Mater. Tech.*, **17**, No. 4: 224 (2002).

14. W. L. E. Wong and M. Gupta, *Compos. Sci. Technol.*, **67**, Nos. 7–8: 1541 (2007).
15. K. S. Tun and M. Gupta, *Compos. Sci. Technol.*, **67**, No. 13: 2657 (2007).
16. M. Ravichandran, A. Naveen Sait, and V. Anandakrishnan, *J. Sandwich Struct. Mater.*, **17**, No. 3: 278 (2015).
17. S. Aravindan, P. V. Rao, and K. Ponappa, *J. Magnesium Alloys*, **3**: 52 (2015).
18. A. Mazahery and M. O. Shabani, *J. King Saud Univ. Eng. Sci.*, **25**: 41 (2013).
19. A. E. Nassar and E. E. Nassar, *J. King Saud Univ. Eng. Sci.*, **29**, No. 3: 295 (2017).
20. D. Muthukrishnan, A. N. Balaji, and G. R. Raghav, *Metallofiz. Noveishie Tekhnol.*, **40**, No. 3: 397 (2018).