Synthesis of Nano Alumina Reinforced Magnesium-Alloy Composites

*E.D.Francis
Reader, Department of Mechanical Engineering, Vegan's IIT, Visakhapatnam
.
N. Eswara Prasad
Scientist G, Director of Regional Centre for military, Airworthiness (Materials), MILAC- nbn, DRDO

Ch.Ratnam
S. Professor, Dept. of Mechanical Engineering, Andhra University, Visakhapatnam

P.Sundara Kumar
Associate Professor, Department of Civil Engineering, K.L.University, Guntur, A.P

V.Venkata Kumar
Research Scholar, Dept of Physics, Andhra University, Visakhapatnam, A.P
E- mail: princy.frany@gmail.com, pskumarphd@gmail.com

Abstract

The main aim of this paper was to determine the amount of Al2O3 reinforcement that results into best combination of strength. Magnesium nano composites containing 3.5, 7.0 and 14 volume percentage of Al2O3 were synthesized through powder metallurgy route. Resistance of furnace sintering technique (heating rate: 49°C/min).Micro structural characterization revealed reasonably uniform distribution of Al2O3 particulates in the matrix and the presence of nanopores. Thermo mechanical analysis revealed significant reduction in CTE of magnesium as a result of presence of alumina nano particulates. Mechanical characterization studies revealed that hardness, work of flexural strength of pure Mg increased when 3.5, 7.0 and 14 vol. % of nano alumina reinforcements are added into Mg matrix. Where as 14 vol.% is revealed that decrease the strength of hardness. The studies revealed that the best combination of mechanical properties is realized from the composite containing 7 volume percentage of alumina.

Key Words: *Sintering, SEM, CTE, Xrd, Magnesium, Nano-Alumina.*

1. Introduction

Magnesium based composites are increasingly getting attention of materials community as a result of their weight saving capabilities [1-3]. The main drawback of magnesium based materials is their low ductility, toughness and stiffness when compared to aluminum based materials. Recently, investigators have indicated that ductility and work of fracture of magnesium can be increased by using reinforcements such as Mo [4], Ti [5], and CNTs [6]. Most of the work on development of magnesium based materials is done on the samples processed using solidification route [7-10] or powder metallurgy route [11-14]. For magnesium matrix composites, conventional reinforcements such as SiC and Al2O3 in particulate form are commonly used. Results of literature search indicated that alumina in

particulate form in magnesium matrix has been investigated to a limited extent. Zaeffered et al. investigated the microstructure, tensile and compressive properties to correlate the mechanical anisotropy with texture of magnesium matrix reinforced with micron size alumina reinforcements of two different sizes up to 20 volume percent [14]. Koike et al. reported microstructure, room temperature tensile and compressive properties and creep of dispersion-strengthened-cast magnesium (DSC-Mg) containing 30 volume percent of submicron size Al2O3 particulates [9- 10]. Nano size alumina particles have been used as reinforcement only in the case of titanium matrix and not in magnesium matrix. The titanium composites were made using solidification processing and the amount of alumina was varied from 10 to 40 wt. %. Attempts were made in that study to establish

interrelationship between hardness and flexural strength [15]. The results of literature research reveal that no attempt is made to synthesize Mg/ Al2O3 formulations using energy efficient resistance of furnace assisted powder metallurgy route and where the alumina particulate length scale is in nanometers. Accordingly, the present study was aimed at synthesizing Mg/ Al2O3 composites using powder metallurgy route incorporating resistance of furnace sintering. The synthesized materials were characterized for physical, micro structural and mechanical properties. Particular emphasis was placed to study the effect of increasing presence of nano Al2O3 particulates in magnesium matrix. There have been used the powders given in table 1 technological characteristics of the powders used. Now powder metallurgical methods are widely used for fabrication a variety of materials. It is difficult to obtain sintering parts from mechanical Mg-Nano Al2O3 powder mixtures [16] due to the great oxidation tenders of Mg at the sintering; starting from these considerations we have studied the possibilities of obtaining Anti-Oxidization from the atmosphere during powders mixtures.

2. Experimental investigations

2.1 The Powders Mixing Process

(1) At first the dispersant dichloride methane, CH2Cl2 was distilled in order to obtain a pure solution without contamination. Dichloride methane is not aquiferous so that the Mg powders may not form oxide or Mg (OH) 2during wet mixing. However, it is toxic when heated so the heating and stirring must be held in a ventilation system. Subsequently, the Al2O3 particulates were poured into the dispersant and vibrated in ultrasonic bath

Table 1: Powders Morphology

Trade Mark	Particle size Dry size ASTM-B-214			Metallic Magnesium Gas-volumetry %	Shape
Magnesium (MGPC)	>500µm %	>63µm %	63µm %	97 Min	Spherical
	1Max	Balance	15 Max		

Nano Alumina (Al_2O_3)	>75µm %	0.05µm %	<45µm %	95 Min	Spherical
	5 Max	Balance	10 Max		

(2) The vibration proceeded for about 10 minutes to disperse the particles. After that the Mg powders were slowly poured into the solution which contained both CH2Cl2 and Nano Al_2O_3 and then vibrated together in the ultrasonic bath. After another 10 minutes, the whole solution was put on the stirring panel with a stirring magnet in it. The stirring remained about 30 minutes to mix the solute uniformly.

(3)After the stirring was done, the dispersant was removed by heating in a hot bath 600 C to vaporize the dispersant. At last, the powders were dried and degassed in a vacuum oven for 30 minutes. The temperature was set at about 700C. Finally, the dried and mixed powders were obtained Figure 1 and Figure 2.

The composition of nano and micro sized fraction that gives the best mechanical properties, we experimentally determined to be Mg-3.5 vol. % Al_2O_3 [18].The powders were compacted into a mold of 45x6x3 by cold uniaxial pressing at 30 ton/mm2.The compacted samples have been sintered in a resistance furnace at the temperature of 630°c for 2 hours in an atmosphere of high purity Argon 15L/Min. In these sintering conditions, established as adequate Oxidation i.e. any loss of Mg was completely prevented. After sintering, the maximum determined weight losses were under 10%.

As compact pressure increases, plastic deformation takes place in the contact zone between the particles starts to break. The porosity in the assembly decreases as particles are squeezed into the remaining free space. The cold welding that occurs during deformation at the inter particle contacts contributes to the strength of the component. Final compactnesses of 78-88%, depending on the compactness after pressing Mg powder. It is more evident in the case of the materials elaborated on the basis of Mg spherical powder. The greater the compacting pressure and the longer the sintering duration the more the compactness reduce.

Figure 1: Images of Pure Mg different stages of Consolidation and Mounting

Figure 2: Images of a Mg + 10Wt% Nano Al2O3 composite different stages of Consolidation

Flexural Strength on 3-Point Bend Strength

S.No	Specimen Code	a/w=0.30	a/w =0.45	a/w =0.45	a/w =0.60	No Crack
1	A(Pure Mg)	21.9	27.97	22.15	20.85	118.12
2	B(3.5Vol%)	36.90	71.81*	41.33	30.29	166.85
3	J(7.0 Vol%)	47.80	64.13*	45.32	25.81*	189.98
4	I(14.0Vol%)	83.60	40.74*	37.81*	30.10*	62.24

Batch 1: Pure Magnesium+ 3.5 Vol% Nano Alumina+ (1.5, 3.0, 6.0 Vol % Copper)

S.No	Specimen Code	a/w=0.30	a/w =0.45	a/w =0.45	a/w =0.60	No Crack
1	A(Pure Mg)	21.9	27.97	22.15	20.85	118.12
2	B(3.5Vol%)	36.90	71.81*	41.33	30.29	166.85
3	G(1.5Vol%)	27.60	36.07	23.89	11.59	228.02
4	H(3.0Vol%)	41.80	50.09	87.11*	39.39	230.32
5	C(6.0Vol%)	46.60	51.14	49.02	25.78*	213.17*

Batch 2: Pure Magnesium+ 3.5 Vol% Nano Alumina+(1.5,3.0,6.0 Vol % Nickel)

S.No	Specimen Code	a/w=0.30	a/w =0.45	a/w =0.45	a/w =0.60	No Crack
1	A(Pure Mg)	21.9	22.15	22.15	20.85	118.12
2	B(3.5Vol%)	36.90	41.33	41.33	30.29	166.85
3	E(1.5Vol%)	94.70	64.44	57.14	33.07	181.35
4	F(3.0Vol%)	62.40*	26.98*	36.22*	53.22*	70.22*
5	D(6.0Vol%)	135.30	57.68	52.17	47.81	258.71

Re Work

Batch No	a/w=0.30	a/w =0.45	a/w =0.60	No Crack
B_1	B	B,I,J	A,I,J	I
B_2	-	B,H	C	C
B_3	F	F,D	F	F

Batch 3: Pure Magnesium+ (3.5, 7.0, and 14.0 Vol% Nano Alumina)

3. Result and Discussion

3.1 Macrostructure

The results of macro structural characterization on the compacted and composite samples did not reveal presence of any macro defects. The outer surfaces were smooth and free of circumferential cracks.

3.2 Density Measurements

The density and porosity measurements conducted on the magnesium and its composite samples are listed in Table 2 the amount of porosity level in all samples remained below 1% indicating the near net shape forming capability of the processing methodology adopted in this study.

Table 2 Results of density, porosity and grain morphology determinations.

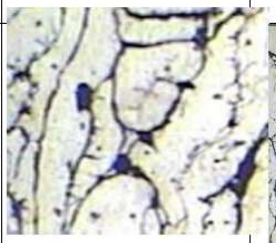
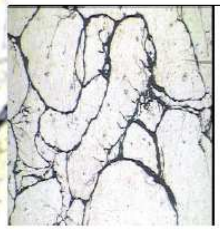
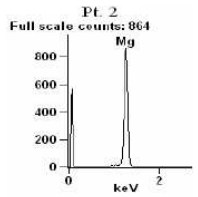
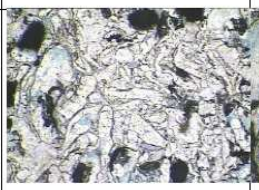
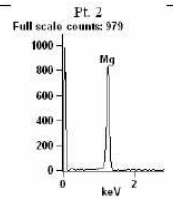
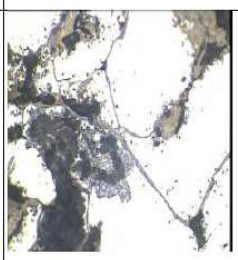
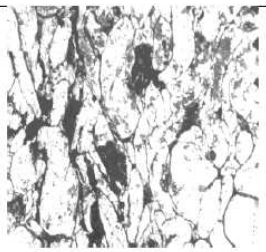
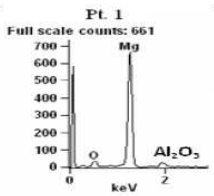
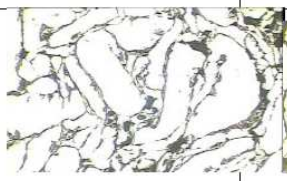
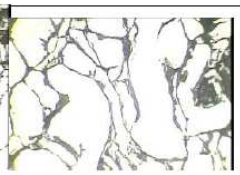
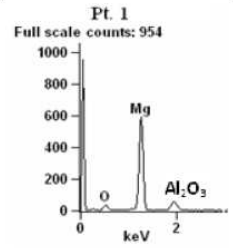
Material	Reinforcement		Theoretical Density (g/cm3)	Experimental Density (g/cm3)	Porosity (vol.%)	Grain characteristics	
	wt.%	vol.%				Size (μm)	Aspect ratio*
Mg	-	-	1.740	1.73±0.2	0.09	6 ± 1	1.6 ± 0.1
Mg/ Al_2O_3	10	03.5	1.8379	1.75±0.2	0.11	8 ± 1	3.7 ± 0.1
Mg/ Al_2O_3	20	07.0	1.9476	1.68±0.2	0.13	10 ±1	4.7 ± 0.1
Mg/ Al_2O_3	40	14.0	2.2106	1.58±02	0.28	5 ±1	2.5 ± 0.1

3.3 Micro structural Characterization

The results of micro structural characterization revealed uniform distribution of nanosized Al2O3 particulates in the case of Mg/3.5% Al_2O_3 (Figure 3b) and fairly uniform distribution of nanosized Al2O3 particulates with a few clusters in the case of Mg/7% Al_2O_3 (Figure 3c) samples. Nanopores were observed in all the composite samples. The results of grain size and samples are shown in Table 2 Near-equiaxed grain morphology was observed for both monolithic and reinforced samples. The identity of the Al_2O_3particulates was confirmed through TEM point analysis (Table 3). The results of grain size and aspect ratio of

the samples are shown in Table 2. Near-equiaxed grain morphology was observed for both monolithic and reinforced samples

Table 3. TEM analysis showing the presence of Al2O3 particulates in the case of: (a) Mg (b)Mg/3.5 Al_2O_3 and (c) Mg/7.0 Al_2O_3 (d) Mg/ 14.0 Al_2O_3

Figure	Specimen Code	100X	200X	
a	Mg			
b	Mg/ 3.5Al_2O_3			
c	Mg/ 7.0 Al_2O_3			
d	Mg/ 14.0Al_2O_3			

3.4 Coefficient of Thermal Expansion

The results of coefficient of thermal expansion (CTE) measurements obtained from monolithic and composite samples are shown in Table 3. The results revealed: (a) reduction in CTE of magnesium as a result of presence of $Al_2O_3$and (b) marginal reduction in average CTE with an increase in Al2O3 amount from 3.5 vol.% to 14 vol.%.

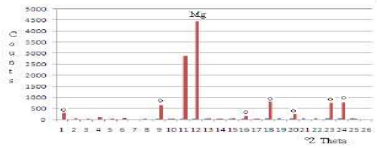
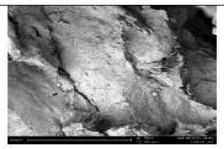
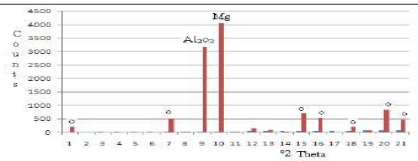
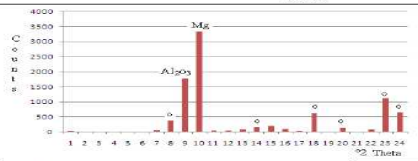
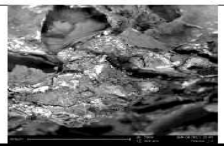
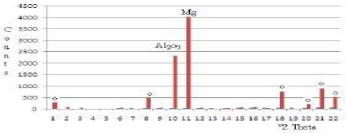
Table 4: Experimental Values of CTE, Micro hardness and Flexural Strength

Material	CTE(x10-6)/oC	Microhardness(HV)	Bend Strength (σy=) Mpa
Mg	38.25	37 ± 2.0	118.1131± 2
Mg/3.5% Al2O3	35.91	47 ± 2.0	167.2246± 2
Mg/7.0% Al2O3	32.74	52 ± 2.0	190.5344± 2
Mg/14.0% Al2O3	36.05	40 ± 2.0	120.1340± 2

3.5 Fractography

Flexural fracture surfaces of Mg and Mg/ Al_2O_3 samples are shown in Figure 3 Fracture surface of Mg samples indicates the presence of cleavage steps and microscopically rough features. Fracture studies conducted on the composites samples revealed mixed mode failure showing more evidences of matrix plastic deformation. XRD analysis also showed the appearance of Mg and Al_2O_3 phases in nanocomposite samples (Table 3). In addition, increasing presence of intermetallic clusters with increasing amount of Alumina particulates in nanocomposites was observed from the micrographs.

Table 4. Representative fractrographs and Micrographs showing: (a) brittle failure in pure Mg, (b) intergranular crack propagation in the case of Mg/3.5% Al_2O_3 and (c) some dimple like features in the case of Mg/7.0% Al_2O_3. (d) Some dimple like features in the case of Mg/14.0% l_2O_3

Figure	Specimen Code	SEM- Observation	Xrd- Observation
a	Mg		
b	Mg/3.5Vol%		
c	Mg/7.0Vol%	----------	
d	Mg/14.0Vol%		

4. Discussion

4.3.1 Synthesis of Mg and Mg/Al2O3 Nanocomposites

Synthesis of monolithic and Mg/ Al_2O_3 nanocomposites has been successfully accomplished using powder metallurgy route resistance of furnace sintering technique. The density of both monolithic and composite samples was greater than 99% indicating the appropriateness of the processing steps and parameters. In particular, the results revealed that energy efficient resistance sintering can be used. It may be noted that normal sintering time (heating-holding-cooling) for a 45x6x3 billet is about 2 hours at 6300c.

4.3.2 Microstructure

The studies conducted on the samples revealed near equiaxed grain shape (Table 2). Grain size measurements of the nanocomposite samples revealed a reduction in average matrix grain size with increasing volume percentage of nanosized Al_2O_3 particulates. The reduction in grain size, however, was marginal and within the standard deviation of each other. The distribution of Al2O3 particulates was relatively more uniform at low volume percentages (3.5 vol.%) and the presence of clusters was evident at high volume percentages (14.0 vol.%). The results thus suggest that the blending parameters need to be further optimized for high volume percentage of Al_2O_3 particulates. The results are, however, encouraging considering the difference in size of Mg powder (60-300μm) and Al2O3 particulates (30-50nm). Microstructural characterization further revealed the presence of almost equiaxed nanopores (see Figure 3). The pores were not necessarily located with particulate clusters. The volume fraction of porosity was also limited and indicate the appropriateness of blending, resistance sintering. [17, 18].

4.3.3 Microhardness

Microhardness measurements revealed that the addition of alumina nanoparticulates in magnesium matrix shows a noticeable improvement in average microhardness of magnesium. This can be generally attributed to: (a) presence and fairly well distribution of harder and stronger second phases in magnesium matrix, (b) an increase in resistance to localized matrix deformation due to the presence of Al2O3 and (c) reduction in matrix grain size.

4.3.4 Coefficient of Thermal Expansion

The reduction in average coefficient of thermal expansion (CTE) of composite samples can be attributed to the presence of nano Al_2O_3 reinforcements which has lower CTE value (7.6x10-6K-1) [19] when compared to pure Mg (28.9x10-6K-1) [20]. The results suggest the existence of good microstructural conditions (distribution and interfacial integrity) in terms of presence of Al2O3 particulates. The results obtained in this study thus reveal that good thermal stability can be obtained by the addition of nanosized Al2O3 particulate reinforcement to the magnesium matrix. Variation in CTE with the change in amount of Al2O3 particulates from 3.5 to 14.0 vol.% was marginal and can be attributed to the relatively poor distribution of Al2O3 particulates in the case of Mg/14.0 vol.% Al_2O_3 (Table 4).

4.3.5 Fracture Behavior

The fracture surface observed in Mg sample indicates presence of cleavage steps and a visible microscopic rough surface (Figure 3a). This can be attributed to HCP structure of magnesium with limited slip systems and hence ductility [21]. With the increasing presence

of Al_2O_3 particulates, intergranular cracks (Figure 3b) and dimple like features (Figure 3c) were observed. The presence of limited intergranular cracks is associated with the increase in ductility consistent with the previous findings [22]. The presence of dimple like features may be attributed to the formation of tiny voids in the Mg/ Al_2O_3 interfacial zone under fracture mode of deformation and their subsequent coalescence.

The incorporation of ductile metals inside a hard ceramic matrix increases its toughness. The probably toughening mechanism is crack bridging due to the presence of a homogeneous ductile metal network in the microstructure, as observed in Table 4. Further work is continuing i

5. Conclusion

1. Conventional solid state powder metallurgy technique incorporating rapid resistance sintering. can be successfully used to synthesize near dense Mg composites containing nano Al_2O_3 particulates.

2. The increasing presence of nanosize Al_2O_3 particulates leads to an increase flexural strength and work of fracture. Coefficient of thermal expansion showed reverse trend indicating an increase in thermal stability.

References

[1] A. Evans, C.S. Marchi and A. Mortensen, Metal Matrix Composites in Industry: An Introduction and a Survey, Boston, Kluwer Academic Publishers, 2003.

[2] I.A. Ibrahim, F.A. Mohamed and E.J. Lavernia, J. Material Science, 26 (1991) 1137-1156.

[3] W.L.E. Wong and M. Gupta, Advanced Engineering Material, 7 (2005) 250-256.

[4] P. Pérez, G. Garcés and P. Adevanced Composite Science Technology, 64 (2004) 145-151.

[5] S.F. Hassan and M. Gupta, Material Science Technology, 20 (2004) 1383-1388.

[6] C.S. Goh, J. Wei, L.C. Lee and M. Gupta, Nanotechnology, 17 (2006) 7-12.

[7] A. Luo, Canad. Metaigy. Quarterly, 35 (1996) 375-383.

[8] B.Q. Han and D.C. Dunand, Material Science Engineering A, 277 (2000) 297-304.

[9] J. Koike, T. Kobayashi, T. Mukai, H. Watanabe, M. Suzuki, K. Maruyama and K. Higashi, Acta Mater., 51 (2003) 2055-2065.

[10] K.U. Kainer and F. Bush, Magnesium Alloys and Technologies, Cambridge, Weinheim, Wiley-VCH, 2003, p. 2.

[11].Kiparisov, s.s. and perel`man,V.E.,Advances in sintering,(ed) Kollar,D. and Ristic, M.M.,Elsevier soience Publishers,Amsterdam 1982,340.

[12]Wei, S.C. “Mechanical Properties and Microstructure of Magnesium Alloy from Severe Plastic Deformation”, National University of Singapore. 2007

[13]Gupta, M. Lai, M.O. and Caravans, D. J. Synthesis, microstructure and properties characterization of disintegrated melt deposited Mg/SiC composites, Mater. Sci. 35, pp.2155-2165. 2000.

[14] Yi, S.B Zaefferer, S.Brokmeier, H.G. Mechanical behaviour and microstructural evolution of magnesium alloy AZ31 in tension at different temperatures, Mater. Sci. Eng. A, 424, pp.275-281. 2006.

[15] F. Thummler and R. Oberacker, An Introduction to Powder Metallurgy, London, Institute of Materials, 1993. [19] R. Morrell, Handbook of properties of technical & engineering ceramics. London, HMSO, 1985.

[16] A. Buch, Pure metals properties: A scientific-technical handbook, Materials Park, Ohio, ASM International, London, Freund Publishing House, 1999.

[17] P.O. Kettunen, V.T. Kuokkala, Plastic deformation and strain hardening, Enfield, N. H, Trans Tech, 2002.

[18] L.H. Dai, Z. Ling and Y.L. Bai, Composite Science Technology, 61 (2001) 1057-1063.

[19] T.W. Clyne and P.J. Withers, An Introduction to Metal Matrix Composites, New York, NY, USA, Cambridge University Press, 1993.

[20] N. Chawla and K.K. Chawla, Metal Matrix Composites, New York, Springer Science + Business Media Inc., 2006, p.197.

[21] R.E. Reed-Hill and R. Abbaschian, Physical metallurgy principles, 3rd ed., Boston, PWS-Kent Pub., 1992.

[22] Partridge, P.G. and Ward-Close, C.M. Processing of advanced continuous fiber composites: Current practice and potential developments, Introduction of. Materials Review. 38, pp.1-24. 1993.

Author

E.D.FRANCIS received his M.Tech degree from Andhra University, India in 2004, Pursuing Ph.D in Andhra University (Thesis to be submitted) He is currently working as a Associate Professor in Vignan's IIT. He is having overall teaching experience of 09 years and 2 years Industrial experience including professional colleges and Industrial. His major research Interests are in Nano technology, CAD/CAM (Mechanical Engineering).

.