FORMATION OF MAGNESIUM METAL MATRIX COMPOSITES $AL_2O_{3\,p}$/AZ91D AND THEIR MECHANICAL PROPERTIES AFTER HEAT TREATMENT

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Abstract
This research chose nano-scaled Al_2O_3 granular materials as the reinforcement particle and used the modified disintegrated melt deposition (DMD) technique to integrate the particle $Al_2O_{3\,p}$ with AZ91D as the magnesium metal matrix composites (Mg MMCs). Subsequently, the melt soup was formed with rapid stirring, and the different heat treatment processes were used to improve the mechanical properties of the composites. With increase of the weight percentage of the particles added into AZ91D, the hardness was increased. But the hardness of the composite added with the particle is declined moderately under the process of air cooling and water cooling. With room temperature cooling condition, the ultimate tensile strength (UTS) of present $Al_2O_{3\,p}$/AZ91D MMCs is 189.55 MPa. With the water cooling condition, UTS of present MMCs increases to 191.86 MPa. However, with the air cooling condition, UTS of present MMCs increases up to 271.86 MPa. After T6 heat treatment, the hardness of 1wt.% $Al_2O_{3\,p}$ added MMCs is improved to 78.94 HV due to the aging effect and its UTS is 254.63 MPa under the condition of the 32hr aging treatment.

Keywords: metal-matrix composites, magnesium alloy, nano-scaled aluminum oxide pellet, heat treatment.

1 Introduction

Magnesium (Mg) alloys are gaining more recognition as a lightest structural material for light-weight applications, due to their low density and high stiffness-to-weight ratio. Even so, Mg alloys have not been used for critical performance applications because of their inferior mechanical properties, compared to other engineering materials. Hence, many researchers attempt to fabricate Mg-based metal-matrix composites (Mg MMCs) by varied methods to obtain light-weight materials with excellent mechanical properties [1-9].
Hassan and Gupta [10] applied disintegrated melt deposition (DMD) technique and hot-extrusion forming to prepare the composite of pure magnesium by adding reinforcement phase Al_2O_3 with the particle sizes of 50nm, 0.3μm, and 1μm and the content of vol. 1.1%. The result showed that the hardness and ductility would increase apparently when the reinforcement phase particle was 50nm, and the ductility of the reinforcement phase being 50nm was four times better than that of the reinforcement phase being 1μm. Hassan and Gupta [11] applied blend-press-sinter methodology to the process of producing pure magnesium by adding the composites with average 50nm Al_2O_{3p} and weight percentage 0.5%, 1.5%, and 2.5%. The findings showed

that the mechanical properties of the material, such as hardness, elastic modulus, yield strength, and ultimate strength, would be improved more apparently, with higher weight fraction of nano-scale reinforcement phase particles. Cao et al. [8] added AlN with 1wt% and the particle size 25nm into AZ91D melt soup, mixed them up with ultrasonic vibration, and further tested the tension in 25°C and 200°C afterwards. The findings demonstrated that the yield strength and tensile strength of AZ91D added with 1wt% AlN were enhanced in room temperature but the elongation decreased, while the mechanical properties were enhanced in 200°C.
Horng-Yu Wu et al. [12] extruded AZ91D in 350°C, further warming up to 415°C for 24 hours, and proceeded artificial aging after hardening. With different artificial aging, the ultimate strength continuously increased, while the elongation decreased gradually, with inverse relation. Jin-feng Huang et al. [13] prepared AZ91 with spray-forming and later with extrusion; then, three heat treatment processes, namely T4 heat treatment, T6 heat treatment, and specific heat treatment, were preceded individually. The findings showed that T4 heat treatment reached the lowest hardness in the first hour, while the maximum hardness of T6 heat treatment appeared at the aging of 12 hr. At last, the mechanical properties of the three heat treatment processes were also compared that the tensile strength and yield strength of T6 heat treatment that exhibiting the highest value but the lowest elongation, while the values of T4 heat treatment and specific heat treatments were close but the yield strength of the former was far lower than the latter. Cizek et al. [14] proceeded AZ91 alloy with heat treatment processes and further studied and compared with air cooling, water cooling, and furnace cooling. The results showed that the ultimate strength and elongation were increased with T4-air cooling, while the yield strength was largely increased with furnace cooling. Nonetheless, the mechanical properties in T4-water cooling and artificial aging were superior to it with air cooling and artificial aging, but without too much difference in hardness.
From previous studies, it was found that the MMCs using different size particles and different heat treatment can improved the mechanical properties. But the research of Mg MMCs added with nano-scale Al_2O_3 particle and treated with T-4 and T-6 is not adequate. This study selects nano-scale Al_2O_3 particle material as the reinforcement particle and, using the DMD-like process, integrates the reinforcement particles into AZ91D melt soup with spraying to form the melt soup by rapid solidification, and finally improves the properties of the Mg MMCs with various heat treatment processes so as to obtain the mechanical properties with hardness and tension tests as well as further discuss the factors.

2 Experimental details

2.1 Materials preparation

The matrix used in this work is magnesium alloy AZ91D with 9.0% aluminium. Its chemical composition is shown in **Table 1**. Al_2O_3 particles with volume fraction of 0.1, 1, and 2 wt% within MMCs are used as the reinforcement phase. The commercially-available Al_2O_3 powder with a particle diameter about 20nm and the purity≧99.8% is added into AZ91D to form Mg-based metal-matrix composites.

Table 1 Chemical composition of AZ91D

Elements	Al	Zn	Mn	Si	Fe	Cu	Ni	Be	Mg
Wt %	9.0	0.69	0.20	0.05	0.001	0.005	0.001	0.001	Balance

The DMD-like process is used to fabricate the present Mg MMCs. Experimental setup is shown in **Fig. 1**. The alloy AZ91D was heated together with Al_2O_3 particles to 760°C under Argon gas atmosphere in a graphite crucible. This design prevented the Al_2O_3 particles from settling. The melt, with incorporated Al_2O_3 particles, was then released through a 15 mm diameter orifice at the base of the crucible and disintegrated by two jets of argon gas, oriented normal to the melt stream. The disintegrated droplets were then deposited onto a metallic mould following the disintegration. An ingot was obtained following the deposition stage. The Mg MMCs containing Al_2O_{3p} with different volume fraction of 0.1, 1, and 2 wt% are prepared for further mechanical thermal testing.

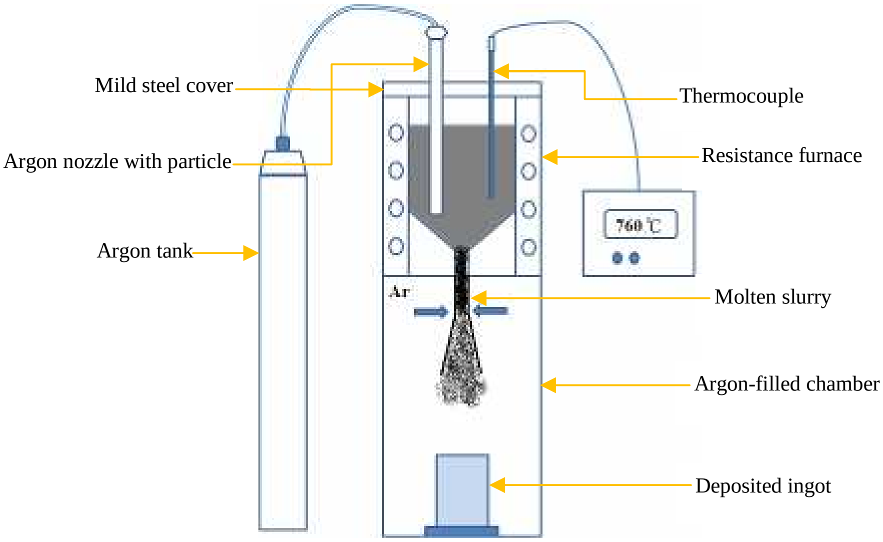


Fig.1 Setup configuration

With T4 heat treatment and T6 heat treatment respectively [15], the former is processed when the heat treatment furnace is heated to 260°C, the specimen is added into the furnace for 1 hour that the residual stress on the specimen is released, and then having slowly (in about 2 hours) warmed up to 415°C and continued for 16 hours, the specimen is cooled off with air cooling, water cooling, and furnace cooling; while the latter is processed with air cooling at room temperature after the T4 heat treatment, then after warming up to 170°C, the specimen is added into the furnace with the durations of 16, 24, 32, and 40 hours, and cools off the specimen with air cooling afterwards.

2.2 Hardness tests and tension test

With a load of 10 kgf, Vickers macro-hardness measurements are carried out on the AZ91D and $Al_2O_{3\,p}$/AZ91D MMCs with a Matsuzawa (Model MV-1) hardness tester. Tension test of present MMCs was performed by Materials Test System of 5 tons with strain rate of 1mm/min. Specimen for test were prepared according to ASTM E8M-04 (see **Fig. 2**).

3 Results and discussion

3.1 Hardness

The Vickers hardness of the matrix material (AZ91D) is 62 HV, and its casted ingot is shown as **Fig. 3**. The hardness with different heat treatment processes, including without heat treatment, T4-air cooling, T4-water cooling, T4-furnace cooling and artificial aging with 16hr, 24hr, 32hr, 40hr, are presented in **Table 2** and **3**.

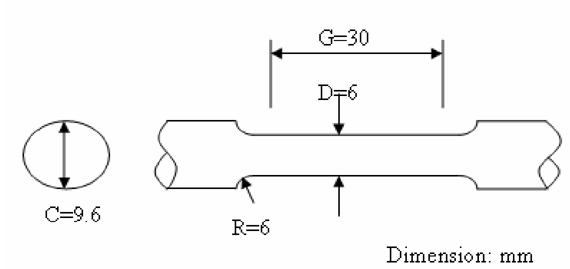


Fig.2 Size of specimen prepared according to ASTM E8M-04

Fig.3 Ingot of AZ91D

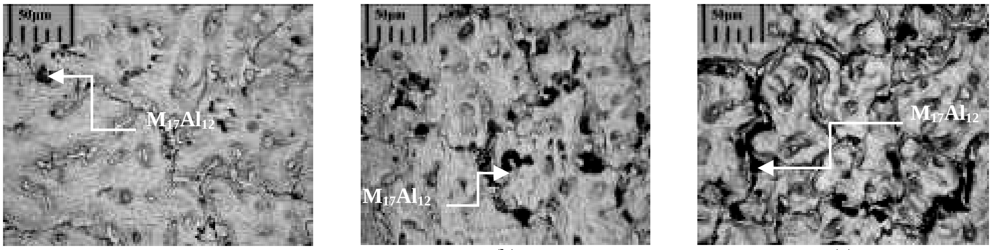
Table 2 The hardness with no heat treatment and T4 heat treatment

Process		Hardness(HV)
Without heat treatment	Al_2O_3 0.1wt%	63.28
	Al_2O_3 1wt%	67.68
	Al_2O_3 2wt%	65.11
T4-air cooling	Al_2O_3 0.1wt%	63.12
	Al_2O_3 1wt%	63.97
	Al_2O_3 2wt%	63.05
T4-water cooling	Al_2O_3 0.1wt%	63.28
	Al_2O_3 1wt%	66.42
	Al_2O_3 2wt%	66.11
T4-furnace cooling	Al_2O_3 0.1wt%	80.41
	Al_2O_3 1wt%	79.75
	Al_2O_3 2wt%	80.43

Table 3 Hardness in various aging with T6 heat treatment

Process		Hardness(HV)
T6-16hr	Al_2O_3 0.1wt%	62.63
	Al_2O_3 1wt%	65.26
	Al_2O_3 2wt%	69.99
T6-24hr	Al_2O_3 0.1wt%	64.83
	Al_2O_3 1wt%	68.56
	Al_2O_3 2wt%	70.39
T6-32hr	Al_2O_3 0.1wt%	68.11
	Al_2O_3 1wt%	73.20
	Al_2O_3 2wt%	75.44
T6-40hr	Al_2O_3 0.1wt%	75.44
	Al_2O_3 1wt%	77.24
	Al_2O_3 2wt%	78.94

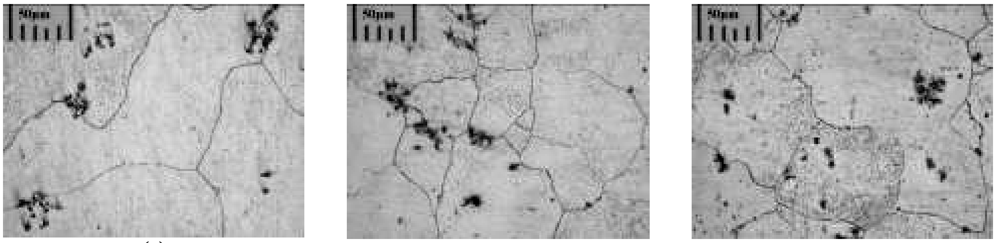
From the tables, the hardness of Mg MMCs with Al_2O_{3p} is larger than that of the matrix materials. The enhancement of mechanical properties is caused by the reinforcement phase making the number of heterogeneous nucleations increases so that the speed of nucleations increases; and, the reinforcement phase also hinders the movement of the matrix and retards the growth of crystalline grains when the melt soup is solidified so that grain refinement is resulted. Discussing from the microscope (see **Fig. 4**), it is found that β phase will increase with the increase of Al_2O_{3p} weight proportion; besides, the excellent mechanical properties of the reinforcement phase have also increase the hardness and strength of the composite materials.



(a) (b) (c)

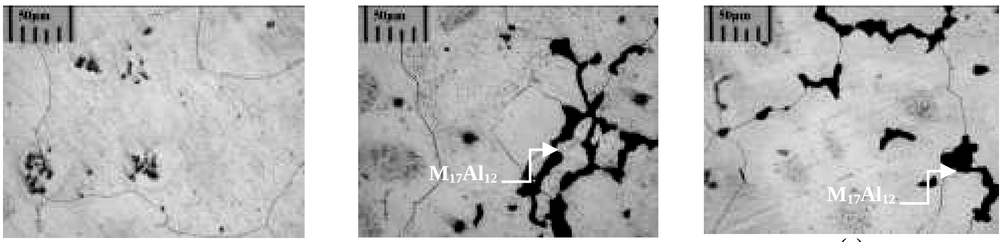
Fig.4 Microstructure of Al_2O_{3p} /AZ91D (a) 0.1wt%, (b) 1wt%, (c) 2wt%, 200 X

The optimal hardness appears at MMCs with adding 1wt% Al_2O_{3p}, while the hardness of that with adding 2wt% particles decreases, presuming that it is affected by the agglomeration of present Al_2O_3 particles. Nevertheless, the hardness after water cooling being larger than it with air cooling is supposed that the specimen is taken out of the water before it is completely cooled off so that the specimen presents different temperatures between surface and the inner part. As the temperature inside the specimen is too high in the process of hardening, dendritic structure is therefore formed. Besides, when the solute starts diffusing, it would accumulate to the dendritic arms (β phase) to generate an area with high condensation and concentration that further affects the enhancement of hardness after water cooling (see **Fig. 6**).



(a) (b) (c)

Fig.5 Microstructure of Al_2O_{3p} /AZ91D with T4-air cooling (a) 0.1wt%, (b) 1wt%, (c) 2wt%, 200 X



(a) (b) (c)

Fig.6 Microstructure of Al_2O_{3p} /AZ91D with T4-water cooling (a) 0.1wt%, (b) 1wt%, (c) 2wt%, 200 X

Regarding T6 heat treatment, a large difference appears at aging of 16hr and 24hr, presuming that the casted ingot selected in the two aging is close to columnar area, where the nucleations is less so that the particle size of the crystal grains is enlarged but the hardness decreased. The hardness is enhanced with the increase of aging time that the maximum 78.94 HV appears at MMCs adding 2wt% of particles, but still less than the hardness 80.43 HV in T4-furnace cooling, showing that the aging peak does not appear.

3.2 Tensile strength

The ultimate strength and elongation rate of the matrix AZ91D are 158 MPa and 3.6%, respectively. **Table 4** and **5** present the comparisons of the ultimate strength and elongation rate between no heat treatment, T4 heat treatment, and T6 heat treatment, while the stress-strain diagrams of no heat treatment, T4 heat treatment, and T6 heat treatment are shown as **Figs. 7-10** and **Figs. 11-14**, respectively. In the process of no heat treatment, when the weight fraction is added from 0.1% to 1% with the elongation increases 7.3%, and the weight fraction added from 1% to 2% with the elongation increases 18.7%, the elongation of the latter is about 2.5 times more than the former. The ultimate strength after heat treatments presents the highest with air cooling, up to 271Mpa with the elongation rate 13.8%, and yield strength 141.5 MPa. Since reinforcement phase can absorb heat from the matrix and enhance the cool-off of the interface between the matrix and the reinforcement phase, effectively preventing the solute from diffusing in the interface, which can largely increase the elongation rate. The ultimate strength with water cooling is the lowest, as the quench on the surface of the specimen is over, but not the inside, so that the surface has been homogeneous but the inside appears dendritic structure that results in the mechanical properties between the surface and the inside of the ingot being weakened.

Table 4 The ultimate tensile strength (UTS) and elongation with no heat treatment and T4 heat treatment

	Adding rate (wt %)	UTS (MPa)	Elongation (%)
Without heat treatment	0.1	160.42	6.7
	1	192.96	7.2
	2	189.55	8.5
T4-air cooling	0.1	183.94	7.2
	1	271.86	11.5
	2	271.08	13.8
T4-water cooling	0.1	196.86	7.2
	1	150.91	3.8
	2	191.86	4.6
T4-furnace cooling	0.1	223.55	3.8
	1	162.25	3.5
	2	224.53	3.3

According to Table 5, the tensile strength is in between 170-250MPa, which is obviously lowered than it with T4-air cooling (see **Fig. 5**), as the dissolving lamellar β phase ($Mg_{17}Al_{12}$) affects the tensile strength and the increase of β phase ($Mg_{17}Al_{12}$) reinforcing the hardness but reducing the ductility. The maximal tensile strength 254.63.MPa appears at aging of 32hr and will decrease with the time prolonged. Comparing aging of 40hr with T4-furnace cooling, the hardness is similar but the tensile strength and elongation of the latter is far less than the former. It is discussed that furnace cooling start dissolving at slightly lower than 415°C; but, as the temperature is too high, the atoms diffuse very fast so that the hardness peak is reached fast, then it transforms into long-time over-aging which results in the softening of the matrix material. The softening is correlated with the growth of crystalline grains that when crystalline grains grow to certain size in a unit area, small particles would be gradually dissolved and big particles gradually grow so that there are few big crystalline grains left.

Table 5 The ultimate tensile strength (UTS) and elongation with T6 heat treatment

	Adding rate (wt %)	UTS (MPa)	Elongation (%)
T6-16hr	0.1	161.03	4.3
	1	176.26	2.4
	2	139.46	2.2
T6-24hr	0.1	231.11	4.7
	1	236.23	4.4
	2	251.46	6.7
T6-32hr	0.1	227.21	7.3
	1	254.63	4.7
	2	247.72	4.9
T6-40hr	0.1	238.54	4.3
	1	246.34	4.6
	2	240.86	2.8

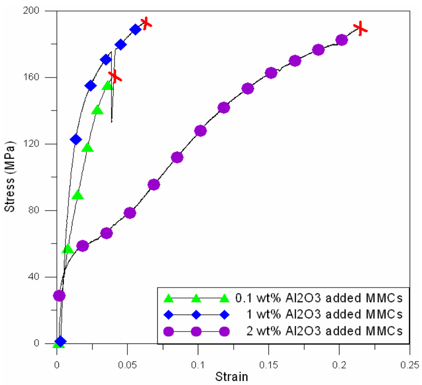


Fig.7 Stress-strain curve of specimen without HT

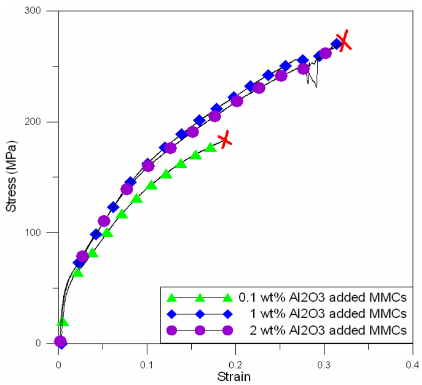


Fig.8 Stress-strain curve of specimen with T4-air cooling

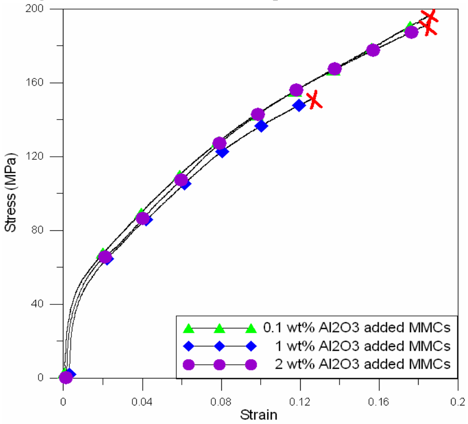


Fig.9 Stress-strain curve of specimen with T4-water cooling

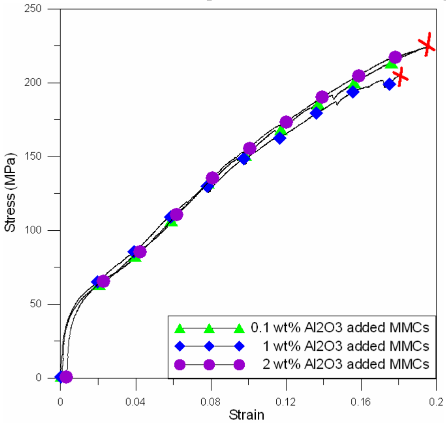


Fig.10 Stress-strain curve of specimen with T4-furnace cooling

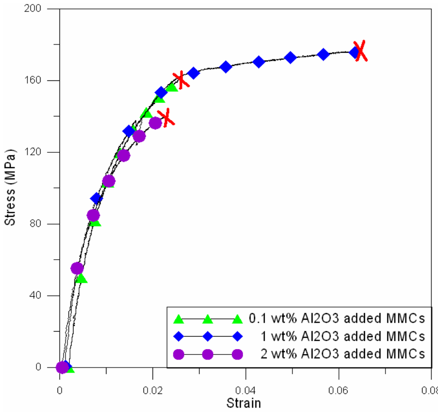


Fig.11 Stress-strain curve of specimen with T6-16hr

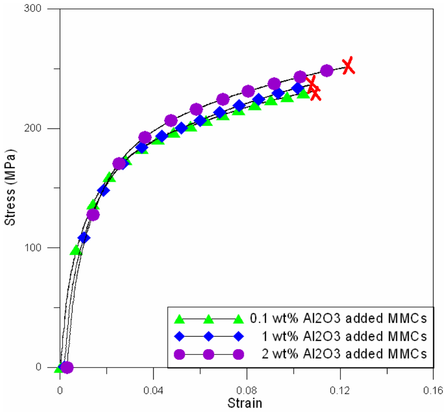


Fig.12 Stress-strain curve of specimen with T6-24hr

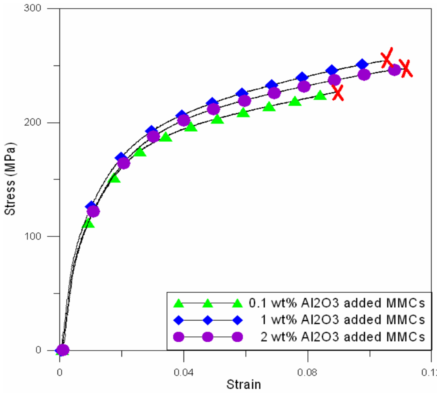


Fig.13 Stress-strain curve of specimen with T6-32hr

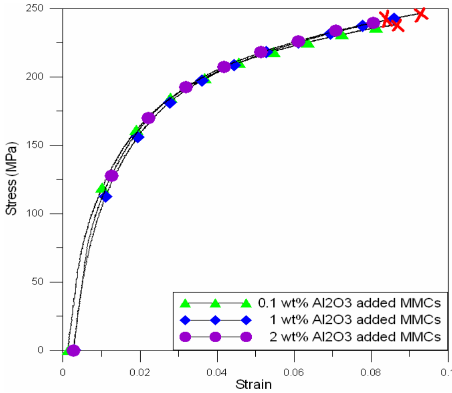


Fig.14 Stress-strain curve of specimen with T6-40hr

4 Conclusion

In addition to comparing the effects of adding $Al_2O_{3p}$with different weight fraction, this study also focuses on heat treatments to observe the difference of cooling speed and mechanical properties with different aging time. The results are concluded as follows:

1) In no heat treatment, the hardness and tensile strength will increase with 0.1wt% and 1wt% reinforcement phase, while the reinforcement phase will decrease with 2wt%.
2) T4 heat treatment cannot be slowly cooled off (furnace cooling), as it would result in early softening so that the mechanical properties become worse than in T6 heat treatment.
3) With T4 heat treatment, the highest elongation appears at MMCs with adding 2wt% particles with the value13.8% that is larger than the matrix material of 3.6% and the elongation of MMCs increases 5.3% in comparison with it being 8.5% of MMCs with adding 2wt% particles in no heat treatment.

4) The highest ultimate strength of MMCs with reinforcement particles in no heat treatment is 192.96 MPa, while it is 271.86 MPa in T4 heat treatment and 254.63 MPa in T6 heat treatment.
5) The hardness of the original matrix is 62 HV. After T6-40hr with 0.1wt%, the hardness of Mg MMCs reaches 75.44 HV. With adding 2wt% of particle, the hardness of MMCs becomes 78.94 HV.

References

[1] P.K. Rogathi: *Cast metal–matrix composites*. ASM handbook-castings, Vol. 15, 1992, p. 840-854.
[2] R.A. Saravanan, M.K. Surappa: Materials Science and Engineering A, Vol. 276, 2000, p. 108-116.
[3] Y. Cai, M. J. Tan, G. J. Shen, and H. Q. Su: Materials Science and Engineering A, Vol. 282, 2000, p. 232-239.
[4] K. F. Ho, M. Gupta, and T. S. Srivatsan: Materials Science and Engineering A, Vol. 369, 2004, p. 302-308.
[5] H. Lianxi, W.Erde: Materials Science and Engineering A, Vol. 278, 2000, p. 267-271.
[6] A.H. Wang, T.M. Yue: Composite Science Technology, Vol. 61, 2001, p. 1549-1554.
[7] C.Y.H. Lim, S.C. Lim, M. Gupta: Wear, Vol. 255, 2003, p. 629-637.
[8] G. Cao, H. Choi, J. Oportus, H. Konishi, X. Li: Materials Science and Engineering A, Vol. 494, 2008, p. 127-131.
[9] S. Ugandhar, M. Gupta, S.K. Sinha: Composite Structures, Vol. 72, 2006, p. 266-272.
[10] S.F. Hassan, M. Gupta: Journal of Alloys and Compounds, Vol. 419, 2006, p. 84-90.
[11] S.F. Hassan, M. Gupta: Materials Science and Engineering A, Vol. 392, 2005, p. 163-168.
[12] Horng-Yu Wu, et al: Journal of Materials Processing Technology, Vol. 209, 2009, p. 4194-4200.
[13] Jin-Feng Huang, Hong-Yan Yu, Yong-Bing Li, Hua Cui, Jian-Ping He, Ji-Shan Zhang: Materials and Design, Vol. 30, 2009, p. 440-444.
[14] L. Čížek, M. Greger, L. Pawlica, L.A. Dobrzánski, T. Tánski: Journal of Materials Processing Technology, Vol. 157-158, 2004, p. 466-471.
[15] M.M. Avedesian and H. Baker: *Magnesium and Magnesium Alloys*, ASM Speciality Handbook, 1999.