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M.Sc. in Mechanical Engineering

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UNIVERSITY OF GAZİANTEP

GRADUATE SCHOOL OF

NATURAL & APPLIED SCIENCES

INVESTIGATION OF MECHANICAL PROPERTIES AND MICROSTRUCTURE OF Al MATRIX COMPOSITES REINFORCED BY NANO AND MICRO MgO PARTICLES

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This is to certify that we have read this thesis and that in our consensus opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science.

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Musa YILMAZ

ABSTRACT

INVESTIGATION OF MECHANICAL PROPERTIES AND MICROSTRUCTURE OF Al MATRIX COMPOSITES REINFORCED BY NANO AND MICRO MgO PARTICLES

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The main objective of this research is to investigate the mechanical, microstructural and physical properties of aluminum matrix composites reinforced with nano and micro MgO particles. In this study, different mass ratios of nano and micro MgO powders with average sizes of 40 nm and 400 µm and Mg were used. Taguchi experiment design method was used and 9 different casting conditions were determined according to L9 array for nano and micro sizes of MgO particles separately. Composites were fabricated at 3 different temperatures 700^{o}C, 800^{o}C and 900^{o}C by stir casting method. All samples were subjected to hardness, compression, density and porosity tests. Microstructures of composites were investigated. Microscopic observations of these composites reveal homogeneous distribution of MgO particles. According to the response tables of signal to noise ratio and ANOVA, it is observed that optimum level setting of casting parameters for compression strength of composites reinforced with nano sized MgO particles at 800^{o}C are 1% Mg and 0.5% MgO in weight and composites reinforced with micro sized MgO particles at 800^{o}C are 1% Mg and 1.5% MgO.

Keywords: Nano particle, MgO, Stir casting, Taguchi, Aluminum matrix composite

ÖZET

NANO VE MİKRO MgO PARÇACIKLARIYLA GÜÇLENDİRİLMİŞ Al MATRİSLİ KOMPOZİTLERİN MEKANİK ÖZELLİKLERİ VE MİKROYAPISININ ARAŞTIRILMASI

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Bu araştırmanın temel amacı, nano ve mikro MgO parçacıkları ile güçlendirilmiş alüminyum matrisli kompozitlerin mekanik, mikroyapı ve fiziksel özelliklerini incelemektir. Bu çalışmada, farklı ağırlık oranlarında ortalama boyutlar 40nm ve 400µm olan MgO tozları ve Mg kullanılmıştır. Taguchi deney tasarım yöntemi kullanılmış ve L9 dizisine göre, nano ve mikro MgO parçacıkları için ayrı ayrı olmak üzere 9 farklı döküm parametresi belirlenmiştir. Kompozitler, karıştırma döküm yöntemi ile 700^{o}C, 800^{o}C ve 900^{o}C'lik 3 farklı sıcaklıkta üretilmiştir. Tüm numunelere, sertlik, sıkıştırma, yoğunluk ve gözeneklilik gibi testler yapılmıştır. Kompozitlerin mikroyapıları incelenmiş ve MgO parçacıklarının homojen dağıldığı görülmüştür. Sinyal-gürültü oranı ve ANOVA tablolarına göre, nano ölçekli MgO katkılı kompozitlerin basma mukavemeti için uygun döküm parametresinin 800^{o}C'de, ağırlıkça % 1 Mg ve % 0,5 MgO olduğu, mikro ölçekli MgO katkılı kompozitler için ise 800^{o}C'de, ağırlıkça % 1 Mg ve % 1,5 MgO olduğu görülmüştür.

Anahtar Kelimeler: Nano parçacık, MgO, Karıştırma döküm, Taguchi, Alüminyum matrisli Kompozit

To My Parents

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LIST OF SYMBOLS/ABREVIATIONS

Al	Aluminum
ANOVA	Analysis of Variance
ASTM	American Society for Testing and Materials
Cr	Chromium
Cu	Copper
DOE	Design of Experiment
Fe	Iron
HCl	Hydrogen Chloride
Mg	Magnesium
MgO	Magnesium Oxide
Mn	Manganese
SiC	Silicon Carbide
S/N	Signal to noise ratio
Ti	Titanium
ρ	Density
CMC	Ceramic Matrix Composite
EDAX	Energy-dispersive X-ray Spectroscopy
MMC	Metal Matrix Composite
PM	Powder Metallurgy
PMC	Polymer Matrix Composite
SEM	Scanning Electron Microscope

CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

In recent years, rapid development of science and technology, modern production and manufacturing systems and methods give rise to increasing the need to new and improved materials. One of these new and improved materials is metal matrix composites (MMCs). Metal matrix composites attract attention due to their low coefficient of expansion, excellent dimensional stability, high specific strength and high hardness values as well as their low cost and wide range of applications.

Composite is a structural material that consists of two or more combined constituents that are combined at a macroscopic level and are not soluble in each other. One of the components forming the composite material is called the reinforcement phase and the other is called the matrix. Composite materials are classified according to the geometry of the reinforcement which are flake, particulate and fibers or according to the type of matrix such as polymer matrix composites (PMCs), metal matrix composites and ceramic matrix composites (CMCs) [1]. In metal matrix composites, aluminum and its alloys are most commonly used matrix material due to high specific strength, low density, high toughness, high thermal conductivity, excellent corrosion resistance and good machinability. Most of the ceramic materials, e.g. SiC, Al2O3, MgO and B4C, are extensively used to reinforce matrix materials [2].

Magnesium oxide (MgO) which has melting point of about 2800°C, is a refractory material. MgO has good properties such as the good thermal shock resistance, high melting point, high compression strength, low thermal conductivity and superior thermodynamic stability.

Metal matrix composites are produced by two different production methods as solid state and liquid state. Powder metallurgy (PM) is the most commonly used method in solid state production process. However, this method requires a high pressure for the production of large parts, and this enhances the cost [3]. Liquid state manufacturing processes are very attractive in terms of being simple, inexpensive and a rapid production technique. However, a significant disadvantage of this method is that the wettability of liquid aluminum, especially ceramic particles, is low [4]. Liquid state production techniques are generally classified into 6 categories: stir casting, squeeze casting, compocasting, spray deposition, pressure infiltration and in situ processing [5-7].

Various methods, which obtain most of the information in the shortest time with the least cost and labor, have been developed to design made experiments on process development studies in industry. The difficulties of some methods which can be accepted as classical for the design of experiments have caused new studies to be made in this respect. The Taguchi method, which was used especially for product development in 1980 and afterwards, is one of these studies. Experimental designs is made easier by using linear graphs and orthogonal array in the Taguchi method [8].

In this study, nano and micro-sized MgO particles with different weight ratios were used to reinforce the Al matrix, using stir casting method. Magnesium (Mg) in different weight ratios was added to the liquid matrix material in different casting temperatures. In order to optimize the production condition, the Taguchi experimental design method was used and the effects of different production conditions on microstructure and mechanical properties of nano and micro MgO particles reinforced in Al matrix composites were investigated.

1.2 ORGANIZATION OF THE THESIS

This thesis consists of six chapters, which are described with brief details of each chapter in this item and starts with introduction chapter.

In the first chapter of the thesis, introduction and layout of thesis are presented.

Chapter 2 consists of literature survey and contains a summary of previous studies and researches.

In Chapter 3, general knowledge on aluminum, magnesium oxide and metal matrix composites are presented. General properties of these materials are given in this chapter.

In Chapter 4, experimental set up, test devices used in measurement of mechanical and physical properties, test standards for specimen preparation and metallographic works were demonstrated.

Results and discussions are presented in the Chapter 5. Experimental results, graphs and microstructural pictures were given. Taguchi method is used to determine the optimum set level of process parameters. And also importance level of each casting parameter on the compression strength and hardness are presented.

Finally, conclusion on thesis is included in Chapter 6.

CHAPTER 2

LITERATURE SURVEY

2.1 INTRODUCTION

This chapter presents a brief survey of most relevant literature related to this thesis.

2.2 LITERATURE SURVEY

Geng et al. [9] produced Al-Mg / SiC composites containing 10% SiC by volume and 2.5%, 4.2% and 6.8% Mg by mass with semi-solid mixing method. Stirring process was carried out with the speed of 30 rpm at a temperature of 612-640^{o}C. Subsequently, the temperature was raised to 650°C and the stirring process was continued at this temperature with the speed of 250 rpm for 10 minutes. Finally, the mixture was poured into a mould preheated at 450°C, and a pressure of 100 MPa was applied by a compression machine. Distribution of SiC particles in the composites produced at 612°C was founded to be more homogeneous than at 650°C. The non-homogeneous distribution of composites produced at 650°C was related to lower viscosity and friction resistance of the melt, and thus the friction between reinforcement and matrix was decreased. It has been found that there was a better wettability between the matrix and the particle and an increase in the homogeneous distribution of the particles when the Mg ratio was increased. There was an increase in elastic modulus, yield and tensile strength and a decrease in the elongation when adding 10% SiC reinforcement. Particle and dislocation strengthening of the matrix induced by the addition of SiC particles.

Fathy et al. [10] investigated the effect of Mg addition on microstructure and mechanical properties of Al-Mg/10 wt. %Al_2O_3 composites. Crystallite size decreased from 44 to 26 nm and lattice strains increased from 0.22% to 0.32% with increasing the Mg content. They pointed out that the increase in Al_2O_3 content caused a decrease in the average particle size. With up to 15 wt.% Mg micro hardness increased from 120 to 230 HV caused by the increment of the Mg concentration and dislocation density as well as the decrease of the crystallite size.

Mazahery and Shabani [11] added nano SiC particles in the A356 alloy by casting method and investigated the effect of SiC on the mechanical and microstructure properties of these nano composites. As the proportion of SiC increased, grain size of composite decreased and porosity ratio of composite increased. While SiC ratio increased from 0.5% to 4.5%, hardness of nano composite increased. It was indicated that this increase was due to difficulty of dislocation movement. Furthermore, it was reported that the highest value of yield and ultimate tensile strength was obtained with 3.5% SiC nano particles. Nano SiC was well dispersed in microstructure and low porosity ratio was main parameter in increasing the strength.

Zhong et al. [12] added Al_2O_3, which has a diameter about 13µm, to Al-Mg (5083) matrix material at 600-640^oC. Then temperature was raised to 720°C and composites were obtained by compocasting (semi-solid casting) method. After these composites was re-melted again, it was poured again at 800^oC. It was detected that MgO which had about 5-20 nm in size, was as the main reaction product after casting and re-melting for 0.5 h and the MgO crystals were transformed into $MgAl_2O_4$ crystals with a diameter of 0.5-1 µm with increasing waiting time.

Valdez et al. [13] added 10% SiC by volume to the Al-Mg matrix material. Al-Mg alloy was poured at 700^oC and this casting was reheated to 470^oC. After reheating SiC particles were added to the matrix material and this mixture was stirred at 1150 rev/min for 15 minutes and then it was re-cast. They were determined that there was a homogenous structure and there was no agglomeration in this composite. They were reported that yield and tensile strength and ductility increase with increasing SiC ratio compared to Al-Mg alloy. The reason for this improvement was attributed to the solid solution strengthening and the distribution of SiC particles, which blocked the dislocation movement.

Contreras et al. [14] produced Al-Mg / TiC composites by infiltration technique. TiC particles with a diameter of 1.2µm and 1% and 20% by weight of Mg were used in this study. Wettability test was applied to composites. They founded that there was a dense interfacial bond in the materials and Al_4C_3, MgO and $AlMg_2O_4$ compounds in composites containing high Mg. They pointed out that increasing the crystal size, decreasing the density of the composites, increasing the porosity ratio and increasing the wettability at high Mg ratio. When the Mg ratio was high, they founded that the crystal size increased, the density of the composite decreased, the porosity ratio increased and the wettability increased.

Hemanth [15] fabricated composites by adding 3, 6, 9, 12 and 15 wt.% nano ZrO_2, which it has the size of 50-80 nm, in the aluminum alloy (LM13) with disintegrated melt deposition (DMD) method. When the LM13 material was at 720^{o}C, it was added at different ratios of nano ZrO_2 and stirring was carried out at a speed of 450 rev/min and it was applied hot extruded at 250°C. Nano composites showed homogeneous distribution, good matrix-strengthening interface composition, significant grain refining and minimum porosity. These are due to the stirring process and the good wettability property of the molten matrix to reinforcement material. He founded that the addition of up to 12% of the nano sized ZrO_2 particles enhanced the mechanical properties and when added more, it reduced these properties. Mechanical properties of LM13 such as hardness of 90 HV, yield strength of 124 MPa, tensile strength of 230 MPa and fracture toughness of 11.3 MPa have increased to 128 HV, 183 MPa, 262 MPa and 15.3 MPa, with addition of 12% ZrO_2 particles, respectively. The reason for these increases was based on the presence of the nano ZrO_2 particulates in the matrix, the decreasing grain size, the thermal expansion coefficient, the transmission of the applied force to the reinforcement, and the dislocation density.

Abdizadeh and Bagchesara [16] added 5%, 10% and 15% ZrO_2 (volume fraction) to the matrix A356 and stirred melt material with speed of 300 rev/min for 13 minutes and produced composites by via the stir casting method at 750, 850 and 950°C. They founded that the density of the composite decreased after the addition of 10% ZrO_2. They have stated that the cause of this decline was the increase in the rate of reinforcement, which was the cause of accumulation, and the increasing temperature. It has been reported that the hardness of the composites increases with the addition of

ZrO_2 and this increase in hardness is due to the density of dislocations occurring between the particle and the matrix interface during solidification.

Zhou et al. [17] coated to 0.2, 0.5, 0.8 and 1 wt% of nano TiC particles (50-100 nm) with nickel. By adding these particles to the melt Al-Cu matrix, they obtained the composites by casting. It was reported that compared with the unreinforced Al–Cu alloy, the tensile strength and elongation of the 0.8 wt. % nano-sized TiC/Al–Cu composite increased from 485 MPa and 6.6% to 573 MPa and 12.4%, respectively.

Abdizadeh et al. [18] used the A356 alloy as the matrix material and MgO (1.5%, 2.5% and 5% by volume) nanoparticles as reinforcement material and produced composites with via stir casting and powder metallurgy method. Different processing temperatures of 575, 600 and 625^{o}C for powder metallurgy and 800, 850 and 950^{o}C for stir casting were applied. It was founded that the porosity ratio was higher due to the pressure and sintering process in the powder metallurgy. In both casting and powder metallurgy methods, the composites were increased hardness by MgO addition. In both casting and powder metallurgy methods, the composites were increased hardness by MgO addition. It was stated that this increase in hardness was due to the density of hard MgO, the density of dislocations and the prevention of grain growth. They stated that the casting method has some advantages compared to powder metallurgy method such as better compression strength, more homogeneous structure, less porosity, more wettability between the matrix and the reinforcement.

Zhou and Xu [4] used two aluminum alloys (A536 and 6061) as matrix material and SiC with 10% and 20% volume fraction as reinforcement material. Gravity casting method was used. They preheated scraps of alloy 6061 and A356 at 450°C for 3 to 4 hours before melting, and SiC particles at 1100°C for 1 to 3 hours to make their surfaces oxidized. Matrix material was first melted and then it was cooled down just below the liquids to keep the slurry in a semi-solid state and preheated SiC particles were added into melted material. Stirring was performed for 20 minutes at an average stirring rate of 150-200 rpm and mixture was poured at 720°C. They determined that SiC showed a strong tendency to accumulate and composites had high porosity ratio. The interfaces between matrix and particles were investigated and interface reaction products such as Al_4C_3 were determined experimentally.

Baghchesara and Abdizadeh [19] fabricated composites by adding 1.5, 2.5 and 5 vol.% nano MgO, which it has the size of 70 nm, in the aluminum alloy (A356.1), which it has the size of 1 μm, with via powder method. These composites were sintered at different sintering temperatures such as 575, 600 and 625°C. They determined that some agglomeration was observed in samples with high content of MgO. The hardness of samples increased to maximum result of 44 BHN, with increasing MgO ratio of specimens. They found that the increase in temperature increased in density of composites and sintering temperature improved the compression strength of samples. Better compressive strength result was observed at 625°C and the maximum compression strength was found as 288 MPa at sample with 2.5 vol. % nano MgO.

Yar et al. [2] added MgO (by volume of 1.5%, 2.5 and 5) at a diameter of 50 nm to the melt matrix material (A356.1) and stirred at a speed of 420 rev/min for 10 minutes. Later, composite material was poured to steel mold at 800, 850 and 950°C. It was stated that the density of the composites decreased due to the increase of the reinforcement ratio. They thought that this could demonstrate the effect of low wettability and agglomeration at high ratio of reinforcement led to the formation of porosity. It is reported that, an increase in hardness was observed as the MgO ratio increased and compression strength decrease with 2.5% and 5% MgO addition at 850°C and 950°C. This reduction was based on the absorption of air during high casting temperature.

CHAPTER 3

METAL MATRIX COMPOSITES AND PROPERTIES OF ALUMINIUM AND MAGNESIUM OXIDE

3.1 INTRODUCTION

The aim of this chapter is to present general information on aluminum, magnesium oxide materials and metal matrix composites. Physical and chemical properties of aluminum and magnesium oxide are explained briefly.

3.2 ALUMINIUM

Aluminum is the third most widely found metal in the world. Aluminum has relatively good properties such as high specific strength, low density, good malleability, easy machining, excellent corrosion resistance and good thermal and electrical conductivity. Aluminum is very attractive metal in engineering applications, automotive and aerospace industry due to superior mechanical properties [20].

Al is a low density metal that can be strengthened by the addition of alloying elements. Al is nearly one-third of steel density; is malleable, ductile, and easily machined and cast; and has excellent corrosion resistance and durability due to the protective oxide layer [21].

The most important physical property of Al is low density (2.71 g/cm^3) which is superior to aluminum from other engineering materials. Al also has high thermal and electrical conductivity. Physical and chemical properties of aluminum are given in Table 3.1.

Table 3.1 Properties of Aluminum [22]

Property	Value
Atomic Weight	26.98 gr/mol
Melting Point	660.32^oC
Boiling Point	2467^oC
Density	2.71 g/cm^3
Electrical Resistivity	28.2 nΩ.m (20^oC)
Thermal Conductivity	237 W/(m.K)
Atomic Radius	125 pm (0.125 nm)
Coefficient of Thermal Expansion	21.50x10^{-6} $^oC^{-1}$

Aluminum alloys are widely used in automobile structures and aerospace vehicles due to their excellent strength, low-weight ratio, good corrosion resistance and easy machinability.

Structural components manufactured by aluminum, from automotive to aerospace, medical to military industry and transportation to the construction industry, have an important execution area in daily life.

Aluminum alloys are also used in architectural design, marine constructions, truck-trailer body panels and production of electrical wires.

3.3 MAGNESIUM OXIDE

Magnesium oxide is a white solid mineral and has an empirical formula of MgO. The MgO is composed of an ionic bond between oxygen and magnesium atoms. The majority of MgO fabricated is obtained from the processing of naturally occurring minerals like magnesium carbonate (magnesite), magnesium chloride rich brine, and seawater. The MgO is consumed as refractory magnesia. The primary use of the refractory mineral is in furnace linings in the iron and steel industries.

Magnesium oxide is used as a basic refractory material for lining crucibles, principal ingredient in construction materials, optical material, electrical heating industry, animal feeding industry, magnesium supplement. Physical and chemical properties of magnesium oxide are given in Table 3.2.

Table 3.2 Properties of Magnesium Oxide

Property	Value
Atomic Weight	40.304 gr/mol
Melting Point	2800^{o}C
Boiling Point	3600^{o}C
Density	3,58 g/cm^{3}
Thermal Conductivity	45-60 W/(m.K)
Coefficient of Thermal Expansion	14.45x10^{-6} ^{o}C^{-1}

3.4 METAL MATRIX COMPOSITES

In recent days, metal composite materials have been used many different application especially in engineering, automobile, aerospace industry. Metal matrix composites (MMCs) are fabricated by means of unification of two or more different materials, which are form of reinforced particles or continuous-discontinuous fibers by dissolving and dispersion into a molten metal or partially solidified metals as a new material [23].

These advanced and new materials introduce many possibilities for modern material science and advanced manufacturing technologies. The characteristic of MMCs provide more low-economic productions and quick solutions for manufacturing. In engineering application, cost of production, production time and higher mechanical properties are very important factors for solutions in engineering problems. Combination of different materials presents enormous mechanical properties in advanced manufacturing technologies. The composite materials have better and superior specification than each component material.

MMCs have many superior properties such as;

- low density,
- mechanical compatibility,
- chemical compatibility,
- thermal stability,
- high Young's modulus,

- high compression and tensile strength,
- good machinability,

Metal matrix composites can be classified in various ways. One classification is the consideration of type and contribution of reinforcement components in particle, layer, fibers and penetration composite materials (Figure 3.1). Fiber composite materials can be further classified into continuous fiber composite materials (multi- and monofilament) and short fibers or, rather, whisker composite materials shown in Figure 3.2 [24].

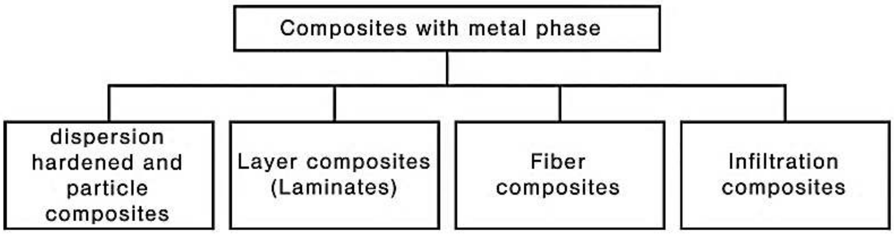


Figure 3.1 Classification of Composite Materials with Metal Matrixes[25]

Main type and contribution of reinforcement components in MMC;

- layer types
- fiber types
- particle types

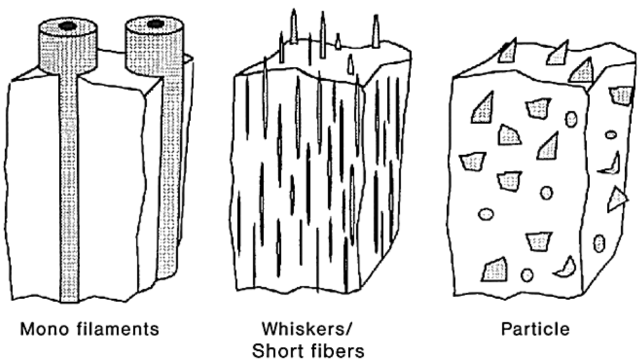


Figure 3.2 Types of Metal Matrix Composites[26]

3.4.1 Fabrication Methods of Metal Matrix Composites

Metal matrix composites are produced by using different kind of production methods. The processes are classified;

- Liquid State Fabrication
- Solid State Fabrication

3.4.1.1 Liquid State Fabrication

In liquid state fabrication, materials are melted and dispersed by mechanical stirring into a molten matrix metal, following by solidification. In order to get high level of mechanical properties of the composite, good interfacial bonding (wetting) between the dispersed phase and the liquid matrix must be provided.

The methods of liquid state fabrication of Metal Matrix Composites:

- Stir casting
- Squeeze casting
- Compocasting
- Spray deposition
- Pressure infiltration
- In situ processing

3.4.1.2 Solid State Fabrication

In solid state production of MMC, MMC are formed as a result of bonding of matrix metal and dispersed phase due to mutual diffusion occurring between them in solid state at elevated temperature and under high pressure. Powder blending and consolidation is a commonly used method for the preparation of discontinuously reinforced MMCs [19]. In this process, powders of the metallic matrix and reinforcement are first blended and fed into a mold of the desired shape. Blending can be carried out dry or in liquid suspension. Pressure is then applied to further compact the powder.

The methods of solid state fabrication of Metal Matrix Composites:

- Powder Metallurgy
- Diffusion Bonding

3.4.2 Matrix Materials Selection

Wide range of metals can be used for the matrix of the composites whereas lightweight and compatibility with the ceramic reinforcements restricts choice of matrix metal. The common material candidates are Aluminum, Magnesium, Titanium, Copper, Zinc, Nickel and Lithium (Table 3.3).

Table 3.3 The Typical Reinforcements Used in Metal-Matrix Composites [27].

Reinforcement	Matrix
Boron, fiber (including coated)	Aluminum, titanium
Graphite fiber	Aluminum, magnesium, copper
Magnesium oxide	Aluminum, magnesium
Carbon nanotube	Aluminum, magnesium
Silicon carbide	Aluminum, titanium
Alumina-silica	Aluminum

Another restriction is that chemical reactions should not take place between matrix and reinforcement. Such a reaction deteriorates reinforcement-matrix interface properties hence the mechanical properties are reduced[28].

The reinforced metal-matrix composites have enormous importance in aerospace, automotive, electronics and military industry. The reinforced metal-matrix composites had superior properties and reduced cost when compared with pure matrices materials [27].

3.4.3 Aluminum Matrix Composites (AMCs)

Aluminum is the most popular matrix for the metal matrix composites (MMCs). The Al alloys are quite attractive due to their low density, their capability to be strengthened by precipitation, their good corrosion resistance, high thermal and electrical conductivity, and their high damping capacity. Aluminum matrix composites (AMCs) have been widely studied since the 1920s and are now used in sporting goods, electronic packaging, armors and automotive industries. They offer a large variety of mechanical properties depending on the chemical composition of the Al-matrix. They are usually reinforced by Al_2O_3, SiC, MgO, SiO_2, B, BN, B_4C, AlN may also be considered. The aluminum matrices are in general Al-Si, Al-Cu, 2xxx or 6xxx alloys.

The major advantages of AMCs compared to unreinforced materials are as follows [20]:

- Greater strength
- Improved stiffness
- Reduced density(weight)
- Improved high temperature properties
- Controlled thermal expansion coefficient
- Good thermal and electrical conductivity
- Enhanced and tailored electrical performance
- Improved abrasion and wear resistance
- Control of mass (especially in reciprocating applications)
- Improved damping capabilities.

CHAPTER 4

EXPERIMENTAL PROCEDURE AND SETUP

4.1 INTRODUCTION

In this chapter, the experimental work and the procedure that was followed during the casting operation as well as the equipment and their properties are described in detail.

4.2 MATERIALS

Materials used in the current study are 99.95% pure aluminum (Figure 4.1), 99.95% pure magnesium oxide powder, 99.90% pure magnesium, drossing of (Aluflux enviro) and deoxiding tablets (Degasal Enviro DM200). The magnesium oxide powders are nano and micro sized and the average particles size are 40 nanometers and 400 micrometers, respectively. The chemical compositions of the materials which were used in this study are given in Table 4.1 and 4.2.

Table 4.1 Chemical Compositions of Aluminum (wt. %)

Al	Cu	Fe	Mg	Mn	Si	Zn	Other
99.95	0.005	0.015	0.001	0.005	0.02	0.001	0.002

Table 4.2 Chemical Compositions of Magnesium (wt. %)

Al	Cu	Fe	Mg	Mn	Si	Zn	Other
0.01	0.005	0.005	99.9	0.01	0.05	0.01	0.009

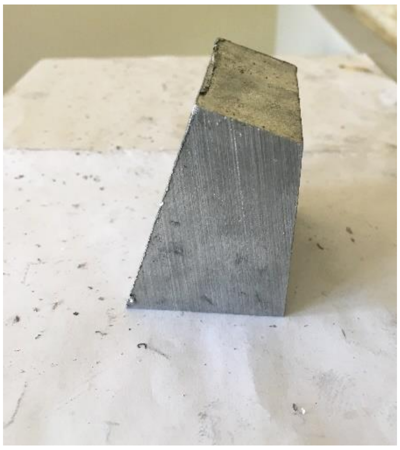


Figure 4.1 Pure aluminum

4.3 EXPERIMENTAL METHOD

In this study, fabrication parameters were established and Taguchi experimental design method was used in order to optimize fabrication condition. These parameters are given in Table 4.3. Subsequently, a series of experiments were conducted. In these experiments, stir casting method was used to produce nano and micro MgO particles reinforced in Al matrix composites.

Table 4.3 Parameters and Corresponding Levels

Parameters	Level 1	Level 2	Level 3
Temperature (^{o}C)	700	800	900
Magnesium (wt %)	1	3	5
Magnesium Oxide (wt %)	0.5	1	1.5

The procedures followed during the experiment are as follows;

- Firstly, MgO particles were pre-heated at 300°C for 1 hour in order to increase the wettability properties of these particles. The particles are wrapped with aluminum foil to prevent oxidation.

- After the preheating, the MgO particles and the aluminum pieces were placed in the crucible and the aluminum pieces were melted in resistance furnace (Figure 4.2), equipped with a mechanical stirring system.

- The drossing (Aluflux enviro) and deoxiding tablets were added into the crucible after the aluminum pieces melted. Subsequently, magnesium was added and the furnace cover was closed.

- The use of argon gas was started with the closure of the furnace cover to prevent oxidation and stirring was carried out at rate of 300-350 rpm for 5 minutes. The stirring rate was adapted, according to the result of literature and previous works [29].

- Steel mold was used for casting of samples. The casting was performed at three different temperatures (700, 800 and 900^{o}C). After the pouring, samples were allowed to cool at room temperature. Finally, samples fabricated in eighteen various conditions (nine samples with nano particles and nine samples with micro particles) were prepared for subsequent mechanical, physical and microstructural analyses.

Figure 4.2 Resistance furnace

4.4 DESIGN OF EXPERIMENT (DOE)

Design of Experiment (DOE) is a powerful statistical technique introduced by R. A. Fisher. DOE is used to design the experimental run layout, in order to study the effect of level change in the process parameters on the output performance. It is known that any change in the input parameters affect the output functional performance. It is important to know that all factors do not effect on the performance in the same manner.

The aim of a carefully planned design of experiment is to find the effect of process parameters on the output product and also to find which of the selected parameters has greatest effect on the output performance and then to determine the best set level for these parameters to obtain a high quality performance products. Minitab software had been used for design of experiment in this study. Minitab program offers these four different methods for DOE; factorial, response surface, mixture and Taguchi.

Within this study Taguchi method was used to design of experiment layout. Taguchi has developed an approach which is based on the orthogonal array experiments, which offers much reduced variance for the experiment with an optimum set of process control factors [30]. Orthogonal array (OA) offers a set of well distributed factor levels to give a minimum experimental run.

Figure 4.3 shows the Taguchi orthogonal array chart for the selection of a proper array's name for the experiment. In the given chart, the intersection of the column and row related to the number of parameters and number of their levels, represents the appropriate array name. Taguchi approach gives an orthogonal array L9 for each particle size and this array was selected based on three casting parameters with three levels.

		Number of parameters																													
		2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
Number of level	2	L4	L4	L8	L8	L8	L8	L12	L12	L12	L12	L16	L16	L16	L16	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32
	3	L9	L9	L9	L18	L18	L18	L18	L27	L27	L27	L27	L27	L36	L36	L36	L36	L36	L36	L36	L36	L36	L36								
	4	L16	L16	L16	L16	L32	L32	L32	L32	L32																					
	5	L25	L25	L25	L25	L25	L50	L50	L50	L50	L50	L50																			

Figure 4.3 Orthogonal Array Selection Chart

Factors and their levels were interfered into the Taguchi method in MINITAB program to get the experimental run layout. Table 4.4 represents the design of experiment orthogonal array layout which will be used to produce the samples according to the mentioned factors and their levels in this study for each particle size.

Table 4.4 Taguchi Orthogonal Array Design L9 for Process Parameters and Corresponding Levels for Particle Size.

Sample no	**Sample Name**	**Temperature (^oC)**	**Mg (wt %)**	**MgO (wt %)**	
1	N1	700	1	0.5	**Nano particle**
2	N2	700	3	1	
3	N3	700	5	1.5	
4	N4	800	1	1	
5	N5	800	3	1.5	
6	N6	800	5	0.5	
7	N7	900	1	1.5	
8	N8	900	3	0.5	
9	N9	900	5	1	
10	M1	700	1	0.5	**Micro particle**
11	M2	700	3	1	
12	M3	700	5	1.5	
13	M4	800	1	1	
14	M5	800	3	1.5	
15	M6	800	5	0.5	
16	M7	900	1	1.5	
17	M8	900	3	0.5	
18	M9	900	5	1	

The aim of this investigation is to study the effect of different levels of casting parameters on the mechanical and microstructural properties, in order to determine the optimum set of casting parameters level to obtain a high quality performance of the composite materials. The optimum casting factors level combination can be determined by analysis of the signal to noise (S/N) ratio for each experimental run. The level of importance of the casting factors are determined by using (ANOVA).

4.4.1 Signal to Noise Ratio (S/N)

The Taguchi approach, highly recommends analyzing data by using S/N ratio that offers two main advantages; it gives a guidance to select the optimum level of design parameters which is based on the minimum variation around the target value and also it provides an objective comparison of two sets of experimental data according to deviation of the average value from target value. S/N ratio is a logarithmic function of desired result which serve as an objective function for optimization of the process parameter level and it is a technique helps in data analysis and prediction of optimum level [31]. Taguchi method, identifies the S/N ratio as the ratio of "Signal" representing desirable value, like mean of output characteristics and the "noise" representing the undesirable value i.e., squared deviation of the output characteristics. The signal to noise ratio is used to measure the quality performance. There are three status of performance characteristic in the analysis of the signal-to-noise (S/N) ratio: smaller-the-better, bigger-the- better and nominal-the-better [30]. These categories can be explained as follows:

a) Smaller-the-Better: this kind of quality characteristic is used when it is required to decrease the resultant value of the output like weld defects, vibration value of the machines, wear in machine components and required force for metal forming. For this type of quality characteristic the minimum value the higher quality achieved. The signal to noise ratio in this case can be formulated as follows:

$$\mathrm{S/N} = -10\log_{10}(1/\mathrm{n}\sum_{\mathrm{i}=1}^{\mathrm{n}}(\mathrm{yi})^2) \qquad \mathbf{(4.1)}$$

b) Nominal-the-Better: in this kind of quality characteristic the nominal or small amount of variability around the nominal value is required. This category of quality is used when it is needed to measure the dimensions like diameter, thickness, width and length. The signal to noise ratio for this condition can be determined by the following equation:

$$S/N = 10\log_{10}(y'^2/s^2) \qquad (\mathbf{4.2})$$

c) Larger-the-Better: this condition for quality characteristic is needed when it is required to increase the response value like in measuring the strength of the joint or material and efficiency of the machine, the maximum value of S/N ratio the higher quality is obtained. The S/N ratio for this category can be measured by the following formula:

$$S/N = -10\log_{10}(1/n \sum_{i=1}^{n}(1/y^2)) \qquad (\mathbf{4.3})$$

Where:

y = Response Value

y'= Mean of the Response Value

s = Standard Deviation

n = Number of Trails for Given Experiment

4.4.2 Analysis of Variance (ANOVA)

Taguchi method cannot judge and determine effect of individual factors on entire process, while the importance level and percentage contribution of individual factors can be very well determined by using ANOVA [32]. Analysis of variance (ANOVA) is a statistical tool which is used to determine the difference or similarity between two or more groups of data. Analysis of variance formally helps to evaluate the significance of all main factors by comparing the mean square versus an estimate of the experimental error at a specific level of confidence. The goal of experimentation is to find the possible methods to reduce the deviation of the required quality as much as possible. This can be reached by identifying those parameters which play a significant role on the performance characteristic [33, 34]. In this study (ANOVA) was used to indicate which casting process parameters is statistically.

4.5 MECHANICAL TESTS

Compression test and hardness test were applied to the samples to compare the effect of the parameters on their mechanical properties after producing composite samples.

4.5.1 Hardness Test

Hardness is the property of a material that enables it to resist plastic deformation, usually by penetration. The usual method to achieve a hardness value is to measure the depth or area of an indentation left by an indenter of a specific shape, with a specific force applied for a specific time. There are large varieties of methods used for determining the hardness of materials, but three popular standard test methods are applied for expressing the relationship between hardness and the size of the impression, these are Brinell, Vickers, and Rockwell hardness test.

In this study, Hardness tests were performed by using MATSUZAWA DXT-3 Universal Hardness Tester Device (Figure 4.4). Hardness of the composites was measured using The Rockwell 15T hardness method. Hardness test values were evaluated by averaging the results of 10 successive measurements (Figure 4.5) in all samples.

Figure 4.4 Hardness Test Device

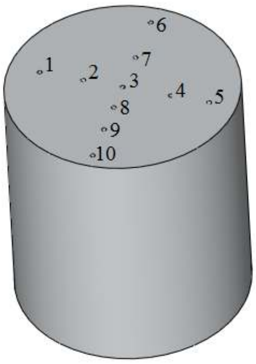


Figure 4.5 Hardness measurement points on the sample

4.5.2 Compression Test

The samples prepared according to ASTM E9-09 standards were subjected to compression test at room temperature. The compression test was applied up to 50 kN load and at a speed of 1 mm/min and compression yield strength, compression strength and maximum strain were obtained. The samples were machined by lathe with a diameter of 10 mm and length 20 mm. (Figure 4.6). Compression test of the samples were carried out by using Shimadzu AGX universal testing device having a capacity of 30 tones (Figure 4.7).

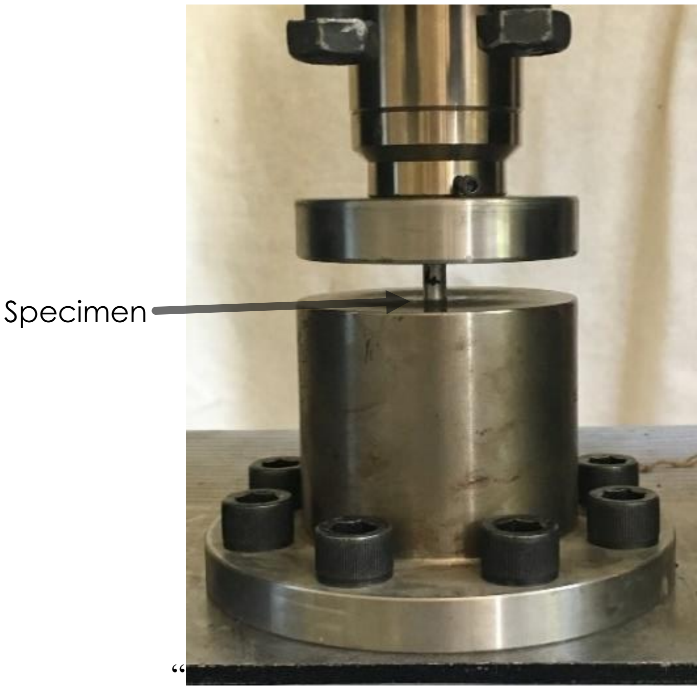


Figure 4.6 Compression Test Specimen and Test Apparat

Figure 4.7 Compression Test Device

4.6 MICROSTRUCTURE EXAMINATION

In this study, specimens firstly were cut with a height of 20 mm. The samples were ground by 320, 600 and 1000 mesh SiC papers and polished by 3μm diamond suspension in the Metkon Forcipol 2V Grinding and Polishing Machine shown in Figure 4.8.

Figure 4.8 Grinding and Polishing Machine

Later, all samples were chemically etched with Keller's reagent (2 ml HF + 3 ml HCl + 5 ml HNO_3 + 190 ml Distilled water) for 30 seconds. The microstructure pictures were captured by Nikon MA-100 Optical Microscope shown in Figure 4.9.

Figure 4.9 Optical Microscope

Zeiss EVO 50 Scanning electron microscope, shown in Figure 4.10, was used to get image of MgO particles in aluminum matrices.

Figure 4.10 Scanning electron microscope and Control Unite

4.7 PHYSICAL TEST

4.7.1 Density Measurement

In this work, the bulk density of the samples was determined by Archimedes' method. The bulk density can be measured by the following formula:

$$\rho_b = \frac{m_1}{m_1 - m_2} \times \rho_w \qquad \textbf{(4.4)}$$

Where:

ρ_b = Bulk Density

ρ_w= Density of Water

m_1 = Mass of Sample in the air

m_2 = Mass of Sample in the water

The mass of the samples were determined using a precision scale, shown in Figure 4.11.

Figure 4.11 Precision Scale

The theoretical density of samples was established by using mixture law. The theoretical density can be measured by the following formula:

$$\rho_c \quad = \rho_{Al} * m_{Al} + \rho_{MgO} * m_{MgO} + \rho_{Mg} * m_{Mg} \qquad (\mathbf{4.5})$$

Where:

ρ_c = Density of composites

ρ_{Al} = Density of aluminum

ρ_{MgO} = Density of Magnesium Oxide

ρ_{Mg} = Density of Magnesium

m_{Al}= Mass percent of Aluminum

m_{MgO}= Mass percent of Magnesium Oxide

m_{Mg}= Mass percent of Magnesium

4.7.2 Porosity Measurement

The percent porosity of samples was calculated from the measurement density and theoretical density using Eq. (4.6)

$$\text{Porosity}(\%) = \left(\frac{\rho_t - \rho_{exp}}{\rho_{exp}}\right) * 100 \qquad (\mathbf{4.6})$$

Where:

ρ_{exp} = Measured density (Bulk density)

ρ_t = Theoretical density

CHAPTER 5

RESULTS AND DISCUSSION

5.1 INTRODUCTION

In this chapter, mechanical and physical test results and microstructural examinations are presented and discussed.

5.2 MECHANICAL TEST RESULTS

Mechanical properties of a material are those properties that involve a reaction to an applied load. The most common properties considered are strength and hardness. General range of usefulness of a material are determined according to these properties [35].

In this study, compression and hardness tests were carried out as mechanical tests and the results are given in detail in this section.

5.2.1 Compression Test Results

In this study, the samples which are prepared according to the Taguchi's layout are tested. The higher the compression strength of composite, the higher the mechanical performance of composite.

The S/N ratio for each experimental run is calculated according to the larger the better formula as given in Equation 5.1. Table 5.1 and 5.2 show the compression yield strength, compression strength and maximum strain of Al-Mg/MgO composites and their signal to noise ratio. Figure 5.1 and Figure 5.2 also show comparison of compression yield strength and compression strength of the composites.

$$S/N = -10\log_{10}\left(1/n\sum_{i=1}^{n}(1/y^2)\right) \qquad \mathbf{(5.1)}$$

Table 5.1 Compression Yield Strength, Compression Strength and Maximum Strain of Al-Mg/MgO Composites (particle size of MgO is 40nm)

Sample Name	Temp. (^oC)	Mg (%)	MgO (%)	Compression Yield Strength (N/mm^2)	S/N Ratio	Compression Strength (N/mm^2)	S/N Ratio	Max. Strain (%)
N1	700	1	0.5	133.11	42.484	636.63	56.08	71.61
N2	700	3	1	110.77	40.889	630.36	55.99	59.37
N3	700	5	1.5	129.48	42.244	621.31	55.87	53.23
N4	800	1	1	111.76	40.966	653.16	56.30	62.36
N5	800	3	1.5	147.61	43.382	611.92	55.73	57.09
N6	800	5	0.5	111.55	40.949	**673.15**	56.56	62.91
N7	900	1	1.5	106.00	40.506	608.93	55.69	64.78
N8	900	3	0.5	**153.11**	43.700	627.78	55.96	58.80
N9	900	5	1	110.44	40.863	597.30	55.52	56.63

Table 5.2 Compression Yield Strength, Compression Strength and Maximum Strain of Al-Mg/MgO Composites (particle size of MgO is 400 μm)

Sample Name	Temp. (^oC)	Mg (%)	MgO (%)	Compression Yield Strength (N/mm^2)	S/N Ratio	Compression Strength (N/mm^2)	S/N Ratio	Max. Strain (%)
M1	700	1	0.5	106.47	40.544	606.00	55.649	65.12
M2	700	3	1	95.68	39.617	583.02	55.314	53.48
M3	700	5	1.5	112.07	40.990	600.26	55.567	53.29
M4	800	1	1	103.42	40.292	624.08	55.905	64.47
M5	800	3	1.5	107.54	40.631	**646.40**	56.210	58.32
M6	800	5	0.5	103.24	40.277	597.20	55.522	50.43
M7	900	1	1.5	105.82	40.491	597.22	55.523	64.09
M8	900	3	0.5	**129.75**	42.262	582.99	55.313	54.59
M9	900	5	1	125.50	41.973	605.97	55.649	54.93

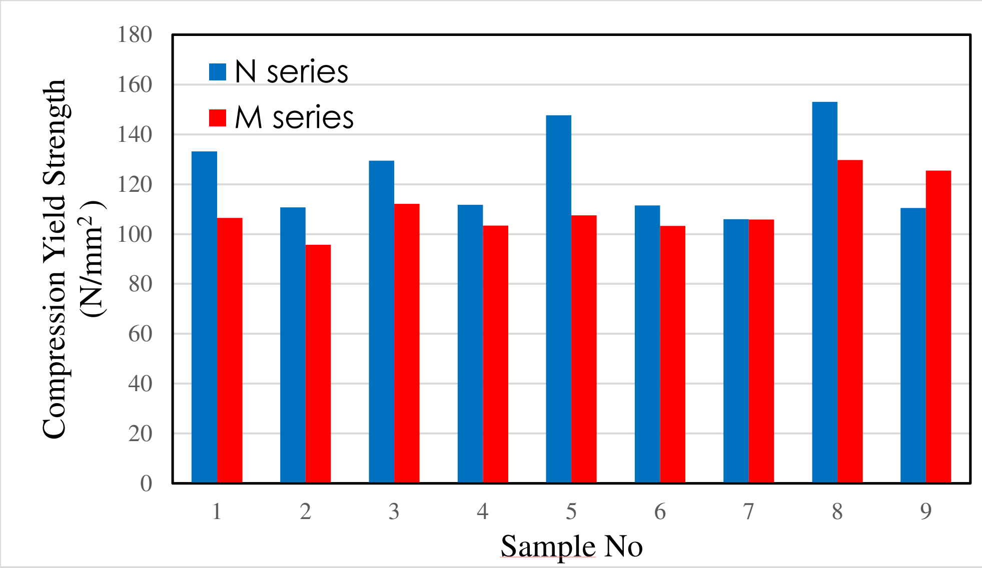


Figure 5.1 Comparison of Compression Yield Strength of the Composites

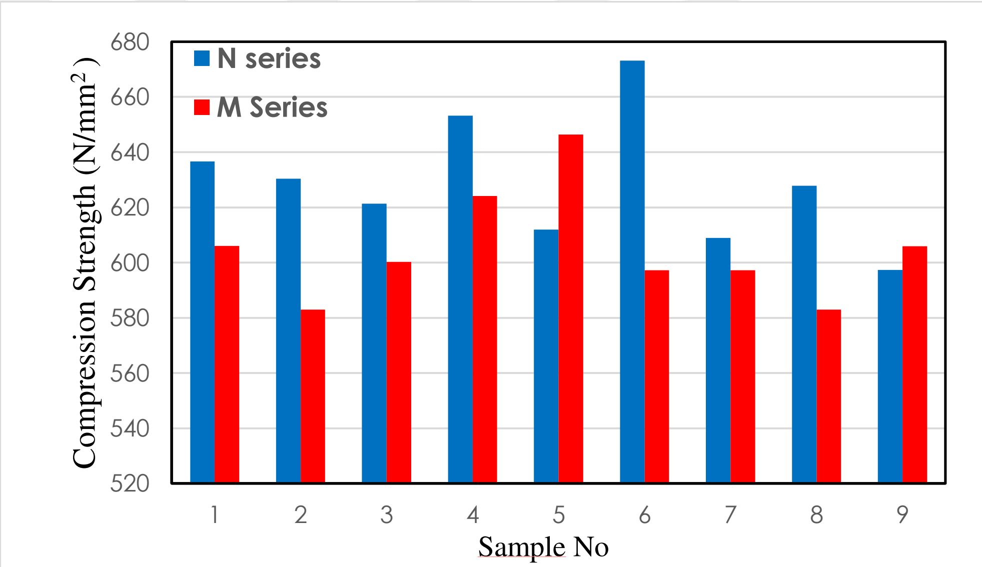


Figure 5.2 Comparison of Compression Strength of the Composites

Figures 5.3, 5.4, 5.5 and 5.6 present the main effects plots for compression yield strength and compression strength of Al-Mg/MgO composites.

These figures demonstrate how strength is changing according to the changes in parameters. MINITAB software was used to show the effect of each casting process parameter level on the compression strength value and compression yield strength value.

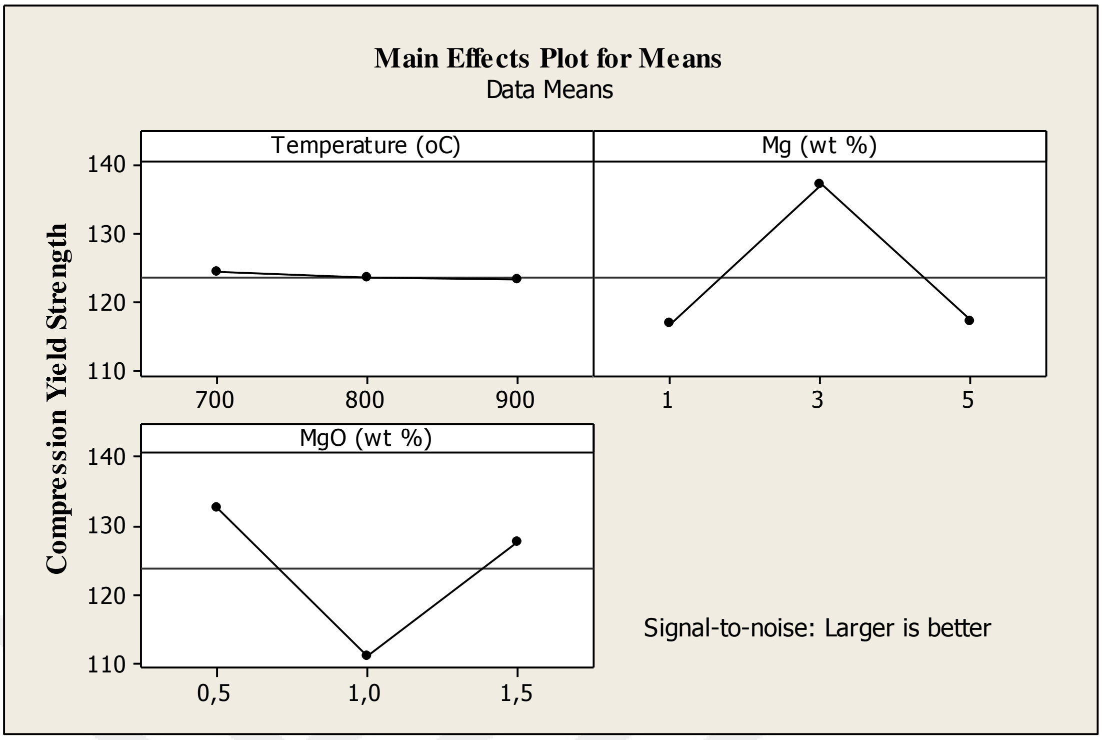


Figure 5.3 Main Effect Plot for Compression Yield Strength of Al-Mg Composites (particle size of MgO is 40nm)

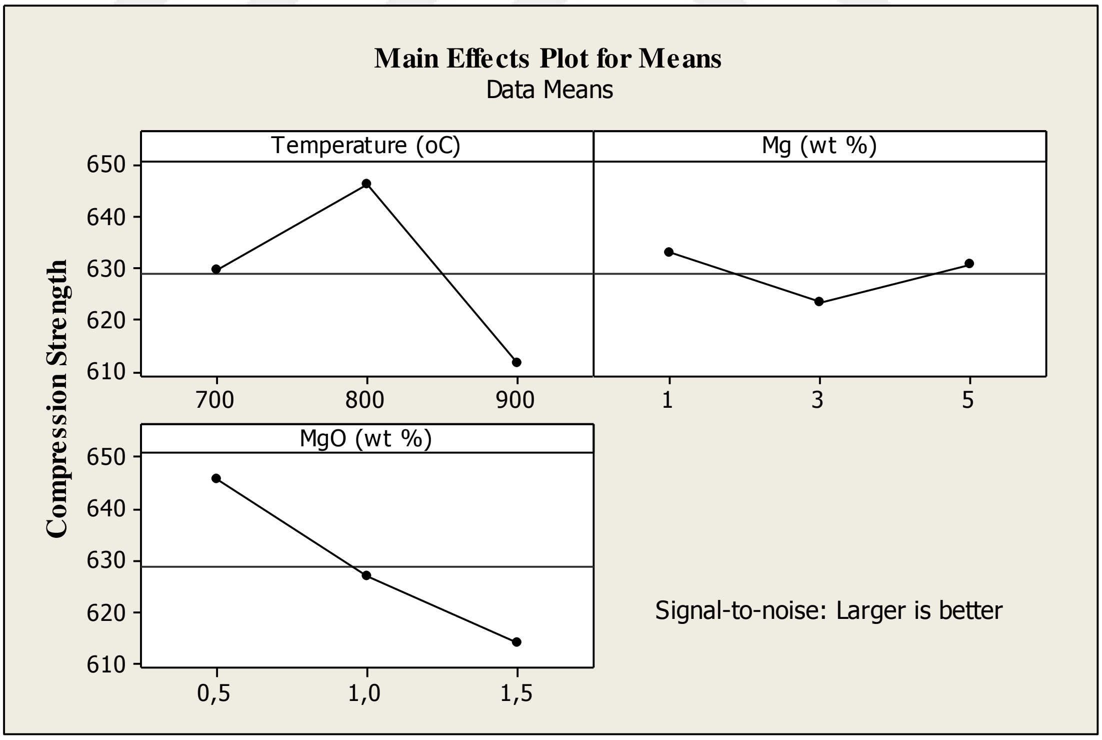


Figure 5.4 Main Effect Plot for Compression Strength of Al-Mg Composites (particle size of MgO is 40nm)

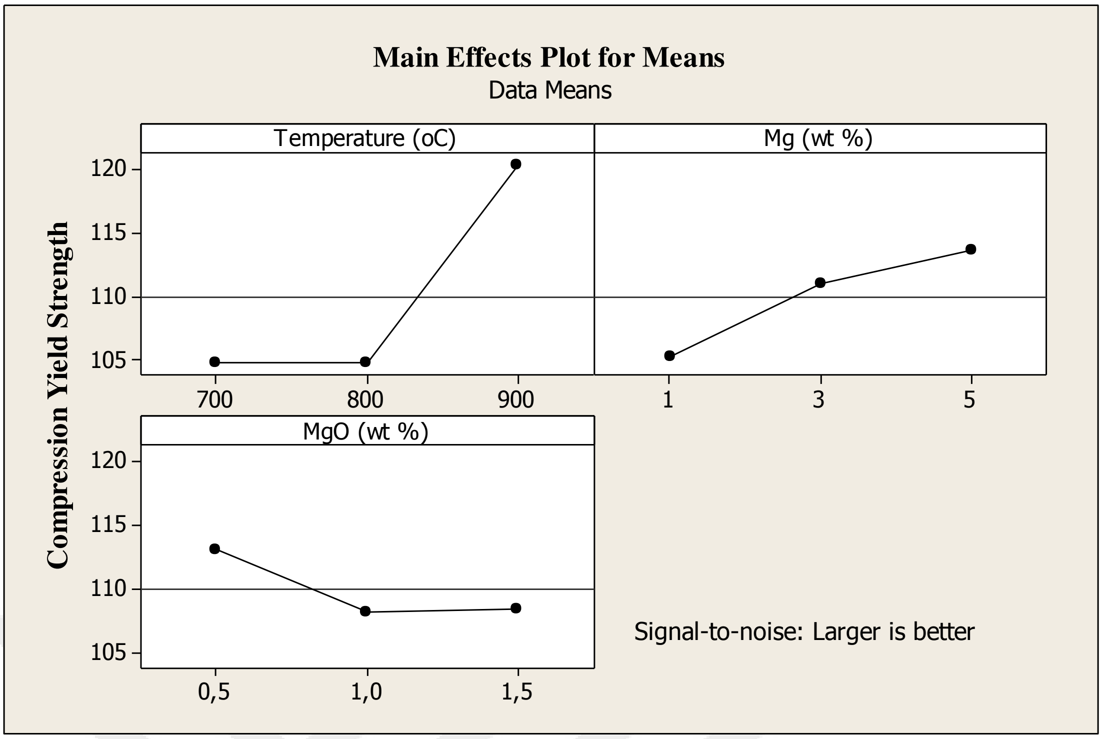


Figure 5.5 Main Effect Plot for Compression Yield Strength of Al-Mg Composites (particle size of MgO is 400 μm)

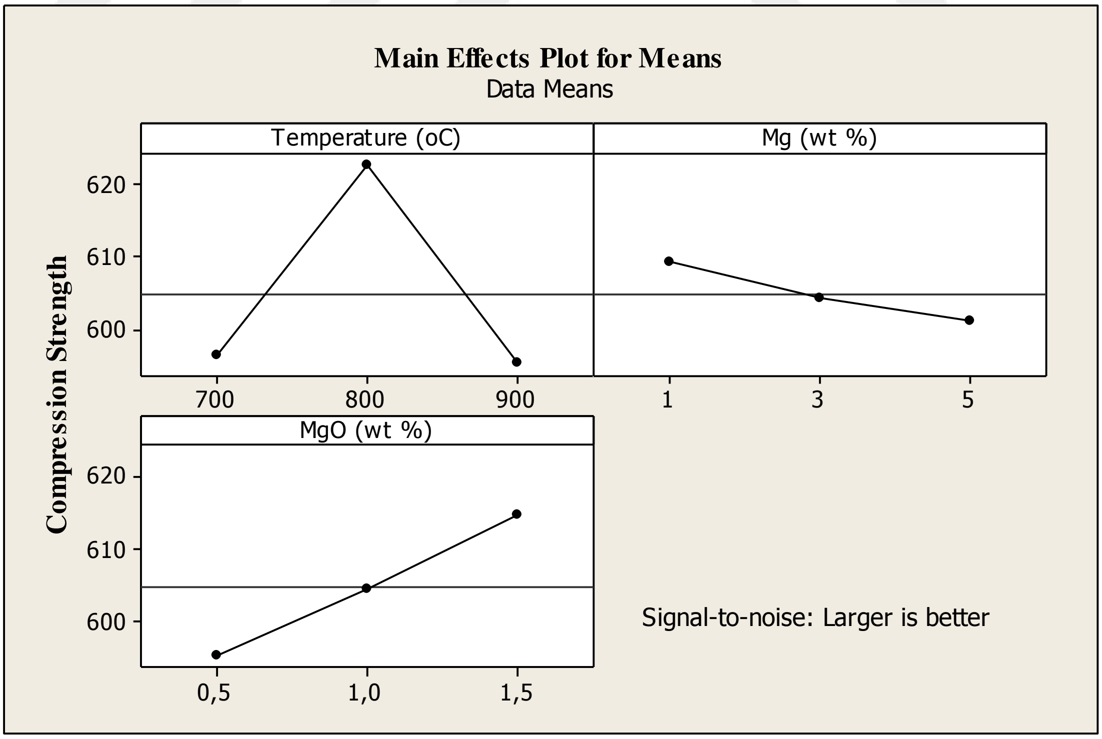


Figure 5.6 Main Effect Plot for Compression Strength of Al-Mg Composites (particle size of MgO is 400 μm)

Compression strength is an essential test for evaluation of the mechanical properties. In case of compression strength, larger the better option was selected for calculation of the signal to noise ratio. Hence, the optimum level setting of casting factors is the level that shows the maximum value of signal to noise ratio.

From Figures 5.3 it can be clearly seen that, addition values of MgO and Mg are playing an important role in strength while temperature is not. On the other hand in Figures 5.4, temperature effect is suddenly decreasing from 800^{o}C to 900^{o}C. Another conclusion can be drawn from these two figures that parameters are effecting in different scales in both compression yield and compression strength values for nano size of MgO particles.

As far as micro sized MgO particles are concerned, Al-Mg composites are presenting different results. For example, addition of Mg and MgO particles are not so effective parameters for yield strength while it is not in compression strength. Because, it is seen that micro sized addition of MgO is increasing the compression strength considerably. Another contrary result is seen for temperature. The best result for yield strength was obtained at 900^{o}C, while the best result for compression strength was obtained at 800^{o}C.

The rank of each casting parameter which is shown in Table 5.3 was calculated by the following procedure. Casting temperature for nano composite is taken as an example:

$$\text{S/N ratio for each level in a specific parameter} = \frac{1}{n}\sum_{1}^{n}\frac{S}{N} \qquad \mathbf{(5.2)}$$

Where:

n = number of runs in that level

S/N = signal to noise ratios in that level

$S/N_{(CT=700)} = 1/3*(56.08+55.99+55.87) = 55.98$

$S/N_{(CT=800)} = 1/3*(56.30+55.73+56.56) = 56.20$

$S/N_{(CT=900)} = 1/3*(55.69+55.96+55.52) = 55.72$

Delta is the difference between the maximum value and minimum value of the signal to noise ratio in a specific casting parameter. Casting temperature for nano composite is taken as an example.

Delta = 56.20 – 55.72 = 0.48

Same procedure was applied for the rest of the parameters for micro composite. The rank is determined according to the value of delta for each parameter. The higher the rank for a factor, the higher the influence of the parameter on the specimen performance. This is due to the same change in controllable factors causes a higher effect on the output characteristic being measured than the other factors.

By referring to Table 4.3 for related levels, following ranking tables are showing the contribution of parameters on Compression Yield Strength and Compression Strength values for Al-Mg composites.

Table 5.3 Response Value of S/N ratio for Compression Yield Strength

Particle Size	**Casting Parameter**	**Signal to Noise ratio (dB)**			**Delta**	**Rank**
		Level 1	**Level 2**	**Level 3**		
Nano Particle	Casting Temperature	41.87	41.76	41.69	0.18	**3**
	wt. % Mg	41.32	42.66	41.35	1.34	**2**
	wt. % MgO	42.38	40.91	42.04	1.47	**1**
Micro Particle	Casting Temperature	40.38	40.40	41.58	1.19	**1**
	wt. % Mg	40.44	40.84	41.08	0.64	**2**
	wt. % MgO	41.03	40.63	40.70	0.40	**3**

Table 5.4 Response Value of S/N ratio for Compression Strength

Particle Size	Casting Parameter	Signal to Noise ratio (dB)			Delta	Rank
		Level 1	Level 2	Level 3		
Nano Particle	Casting Temperature	55.980	56.200	55.720	0.480	**1**
	wt. % Mg	56.020	55.890	55.980	0.130	**3**
	wt. % MgO	56.200	55.940	55.760	0.440	**2**
Micro Particle	Casting Temperature	55.510	55.879	55.495	0.384	**1**
	wt. % Mg	55.69	55.61	55.58	0.11	**3**
	wt. % MgO	55.49	55.62	55.77	0.28	**2**

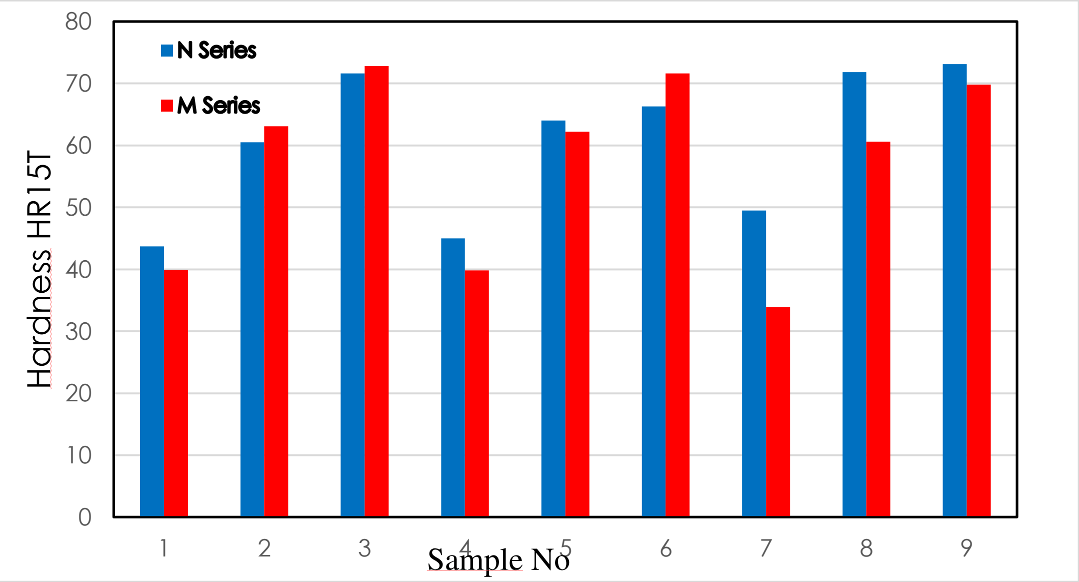
The rank of each casting process parameter for compression yield strength and compression strength are shown in Table 5.3 and 5.4, respectively. In table 5.3, it shows that the casting temperature has greater effect on the response characteristic for compression yield strength test result of micro composite. In nano composites, the biggest effect is MgO percentage according to the compression yield strength test result. Accordingly, Table 5.4 shows that the casting temperature has greater effect on the response characteristic followed by wt. % MgO and wt. % Mg for compression test result.

5.2.2 Hardness Test Results

Rockwell hardness tests were conducted on the composites to assess the hardness value of the samples. Hardness test values were evaluated by averaging the results of 10 successive measurements in all samples. The S/N ratio of the larger is better was chosen. Table 5.5 shows the hardness test results of composites and its signal to noise ratio and Figure 5.7 shows comparison of hardness of composites.

Table 5.5 Hardness Test Result of Composites

Sample Name	Temperature (^{o}C)	Mg (wt %)	MgO (wt %)	Hardness	S/N ratio
N1	700	1	0.5	43.7	32.80
N2	700	3	1	60.5	35.63
N3	700	5	1.5	71.6	37.10
N4	800	1	1	45.0	33.07
N5	800	3	1.5	64.0	36.12
N6	800	5	0.5	66.3	36.44
N7	900	1	1.5	49.5	33.88
N8	900	3	0.5	71.8	37.12
N9	900	5	1	**73.1**	37.28
M1	700	1	0.5	39.9	32.02
M2	700	3	1	63.1	36.00
M3	700	5	1.5	**72.8**	37.24
M4	800	1	1	39.8	31.99
M5	800	3	1.5	62.2	35.88
M6	800	5	0.5	71.6	37.10
M7	900	1	1.5	33.9	30.61
M8	900	3	0.5	60.6	35.65
M9	900	5	1	69.8	36.88



Figures 5.7 Comparison of Hardness of Composites

The main effect plot hardness test for nano and micro added MgO composites are demonstrated in Figures 5.8 and 5.9 respectively. MINITAB software was used to show the effect of each casting parameter level on the hardness test value.

For the hardness test results, larger the better option has been selected for calculation of the signal to noise ratio. Hence, the optimum level setting of casting factors is the level that shows the maximum value of signal to noise ratio.

From Figures 5.8 and 5.9, it can be concluded that the optimum level setting of casting temperature, wt. % Mg and wt. % MgO according to hardness test of nano added MgO composites are 900^{o}C, 5 wt. %, 1.5 wt. % and for micro added MgO composites are 700^{o}C, 5 wt. %, 0.5-1wt. % respectively.

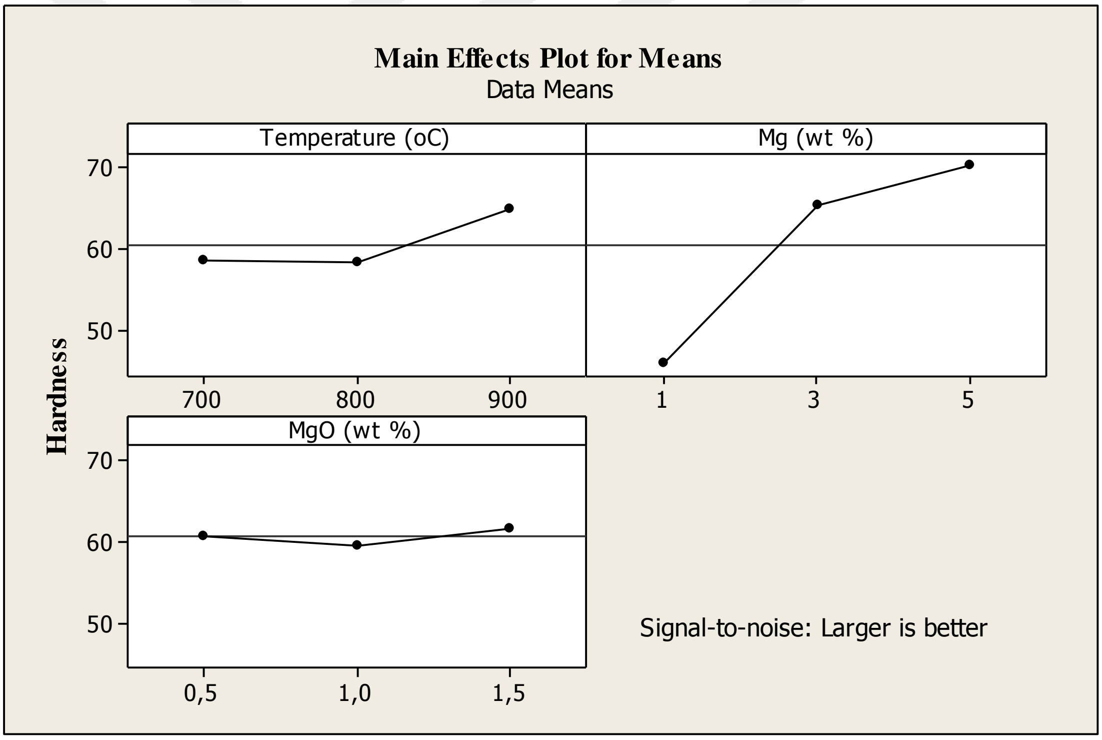


Figure 5.8 Main Effect Plot for Hardness Test of Nano Composites

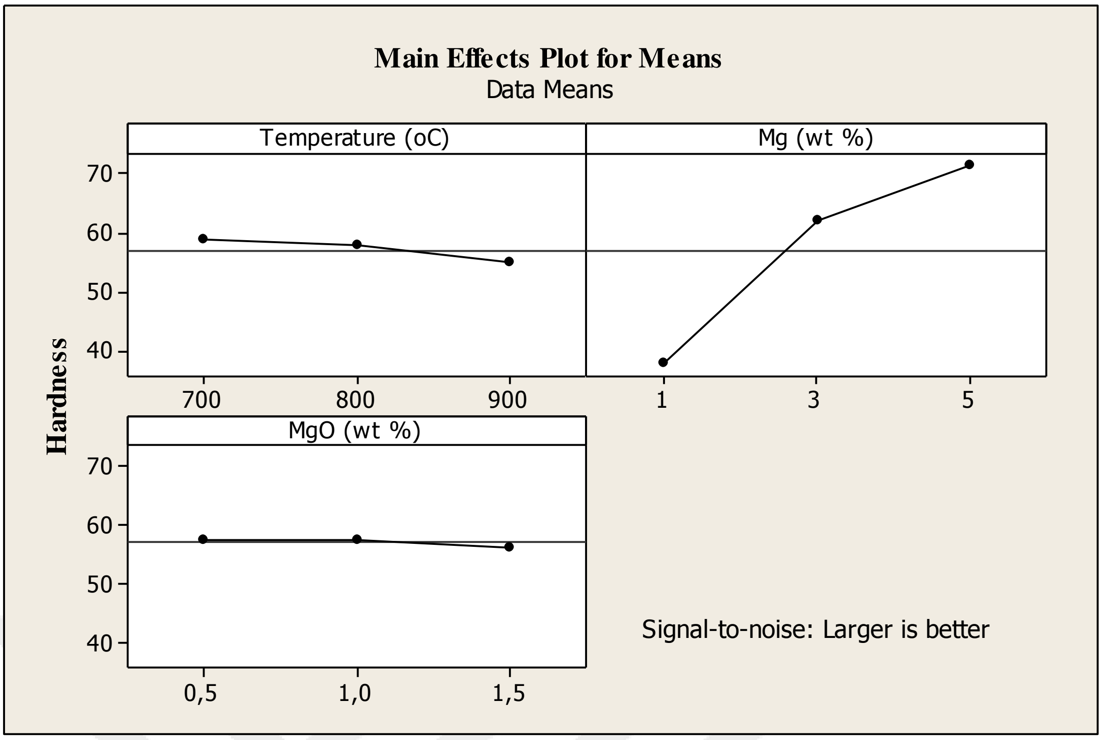


Figure 5.9 Main Effect Plot for Hardness Test of Micro Composites

The rank of each casting parameter is shown in Table 5.6. The rank is determined according to the value of delta for each parameter. The higher the rank for a factor, the higher the influence of the parameter on the hardness test results. This is due to the same change in controllable factors causes a higher effect on the output characteristic being measured than the other factors.

Table 5.6 Response Value of S/N ratio for Hardness Test

Particle Size	**Casting Parameter**	**Signal to Noise ratio (dB)**			**Delta**	**Rank**
		Level 1	**Level 2**	**Level 3**		
Nano Particle	Casting Temperature	35.18	35.21	36.09	0.92	**2**
	wt. % Mg	33.25	36.29	36.94	3.69	**1**
	wt. % MgO	35.45	35.33	35.70	0.37	**3**
Micro Particle	Casting Temperature	35.09	34.99	34.38	0.71	**2**
	wt. % Mg	31.54	35.84	37.07	5.53	**1**
	wt. % MgO	34.92	34.96	34.58	0.38	**3**

Accordingly, Table 5.6 shows that the % Mg has greater effect on the response characteristic followed by casting temperature and wt. % MgO.

5.3 PHYSICAL TEST RESULTS

5.3.1 Density Measurement Results

In this work, the bulk density and the theoretical density of the samples, which are prepared according to the Taguchi's layout, was determined by Archimedes' method and mixture law, respectively. These densities are given in Table 5.7.

Table 5.7 The Bulk and Theoretical Densities of Composites

Sample Name	Temperature (^oC)	Mg (wt %)	MgO (wt %)	Bulk Density (g/cm^3)	Theoretical Density (g/cm^3)
N1	700	1	0.5	2.6920	2.6961
N2	700	3	1	2.6261	2.6816
N3	700	5	1.5	2.6397	2.6672
N4	800	1	1	2.5956	2.7006
N5	800	3	1.5	2.6581	2.6861
N6	800	5	0.5	2.5831	2.6580
N7	900	1	1.5	2.6941	2.7050
N8	900	3	0.5	2.6877	2.6770
N9	900	5	1	2.5812	2.6626
M1	700	1	0.5	2.6802	2.6961
M2	700	3	1	2.6458	2.6816
M3	700	5	1.5	2.6247	2.6672
M4	800	1	1	2.6839	2.7006
M5	800	3	1.5	2.6194	2.6861
M6	800	5	0.5	2.6269	2.6580
M7	900	1	1.5	2.6695	2.7050
M8	900	3	0.5	2.6410	2.6770
M9	900	5	1	2.6161	2.6626

5.3.2 Porosity Measurement Results

The percent porosity of samples which are prepared according to the Taguchi's layout was calculated from the measurement density and theoretical density using Eq. (4.6).

The lower porosity the higher material performance, so the S/N ratio of the smaller is better was chosen. Table 5.8 shows porosity content of composites and its signal to noise ratio.

Table 5.8 Porosity Content of Composites

Sample Name	Temperature (^oC)	Mg (wt %)	MgO (wt %)	Porosity (%)	S/N ratio
N1	700	1	0.5	0.154	16.22
N2	700	3	1	2.113	-6.50
N3	700	5	1.5	1.042	-0.36
N4	800	1	1	4.046	-12.14
N5	800	3	1.5	1.055	-0.46
N6	800	5	0.5	2.898	-9.24
N7	900	1	1.5	0.405	7.85
N8	900	3	0.5	0.102	19.85
N9	900	5	1	3.152	-9.97
M1	700	1	0.5	0.595	4.51
M2	700	3	1	1.351	-2.61
M3	700	5	1.5	1.619	-4.19
M4	800	1	1	0.620	4.16
M5	800	3	1.5	2.548	-8.12
M6	800	5	0.5	1.183	-1.46
M7	900	1	1.5	1.331	-2.48
M8	900	3	0.5	1.365	-2.71
M9	900	5	1	1.776	-4.99

The main effect plot for porosity content of nano and micro composites are presented in Figures 5.10 and 5.11 respectively. MINITAB software was used to show the effect of each casting parameter level on the porosity content.

For the porosity test results, smaller the better option has been selected for calculation of the signal to noise ratio. Hence, the optimum level setting of casting factors is the level that shows the minimum value of signal to noise ratio.

From Figures 5.10 and 5.11, it can be concluded that the optimum level setting of casting temperature, wt. % Mg and wt. % MgO according to porosity test of nano composites are (800^{o}C, 5 wt. %, 1 wt. %) and for micro composites are (900^{o}C, 3 wt. %, 1.5 wt.%) respectively.

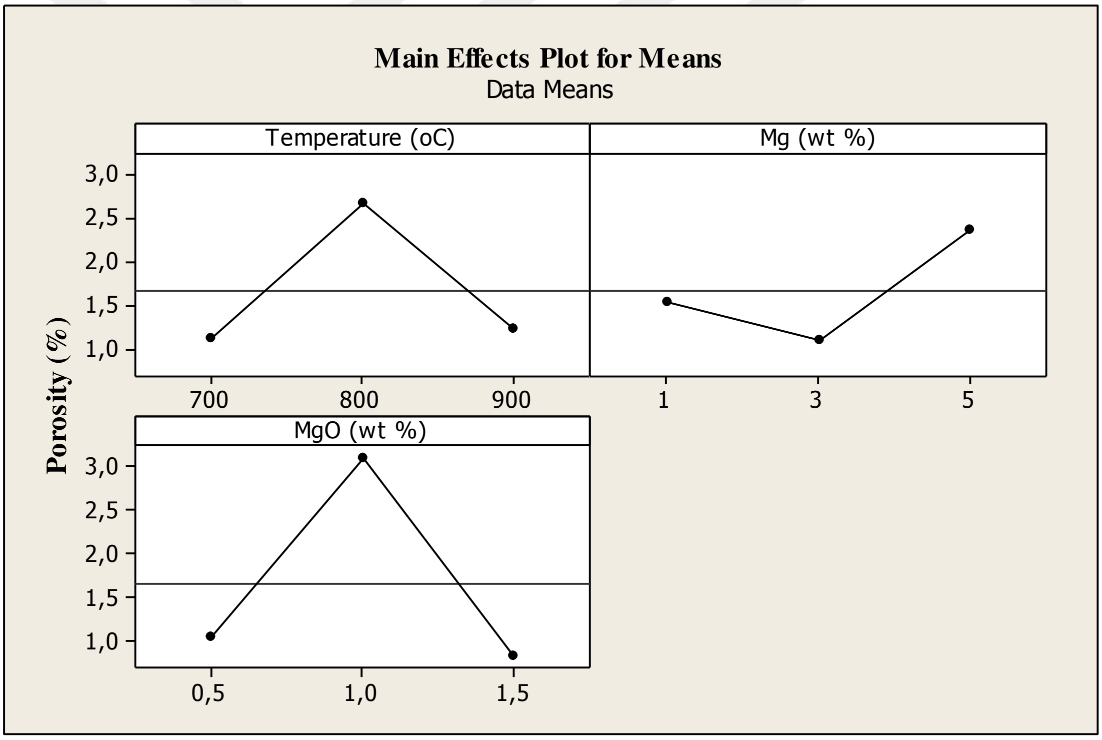


Figure 5.10 Main Effect Plot for Porosity Content of Nano Composites

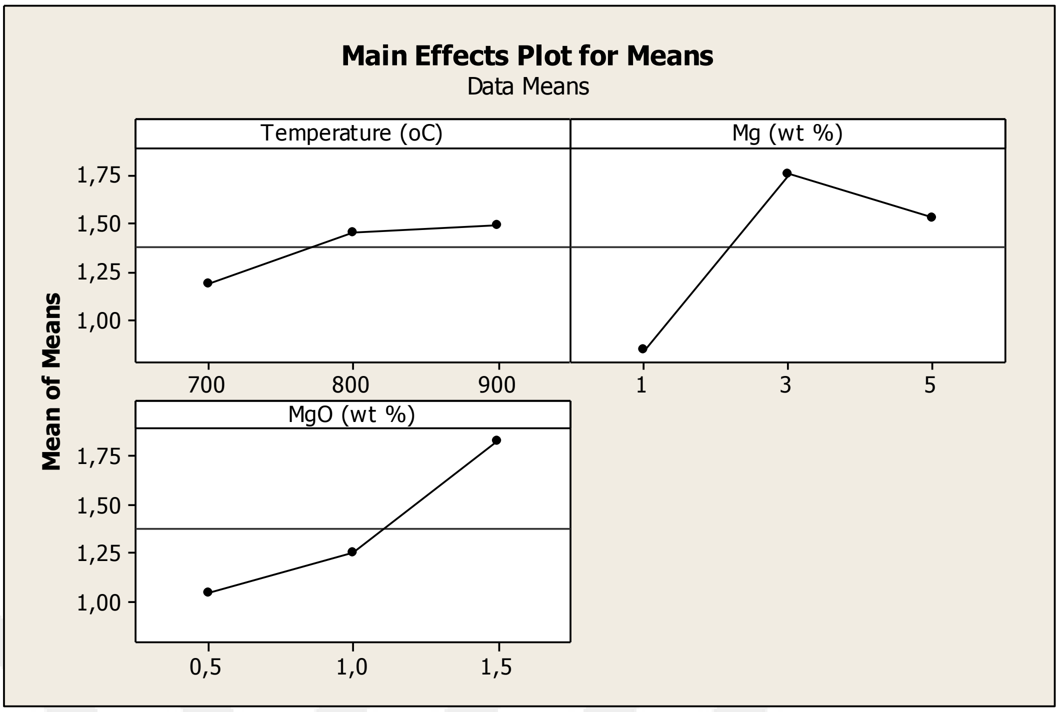


Figure 5.11 Main Effect Plot for Porosity Content of Micro Composites

5.4 MICROSTRUCTURE RESULTS

Optical microscopy observations have been carried out on the composites. Polished and etched samples photographed to investigate their microstructures. Optical photographs are taken in 10x, 20x and 50x scales from optic microscope equipped with camera. The optical photograph of sample 3 is depicted in Figure 5.12.

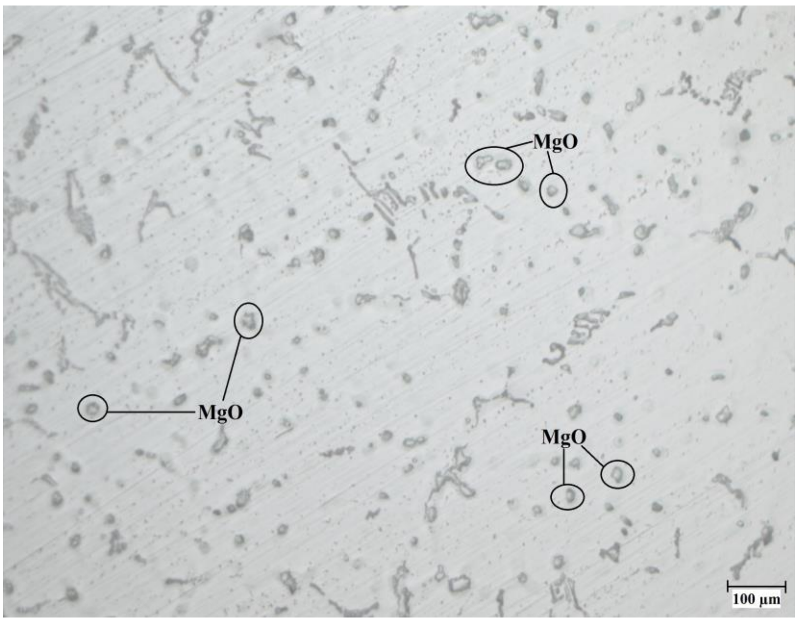


Figure 5.12 Microstructure of Sample 3 at 50x

The MgO particles are evidenced by the metallographic study of samples. The light gray region represents the matrix, the dark gray particles as MgO. In general, it has been observed that the particles have a homogeneous distribution. Figure 5.13 demonstrates the microstructure of sample 4.

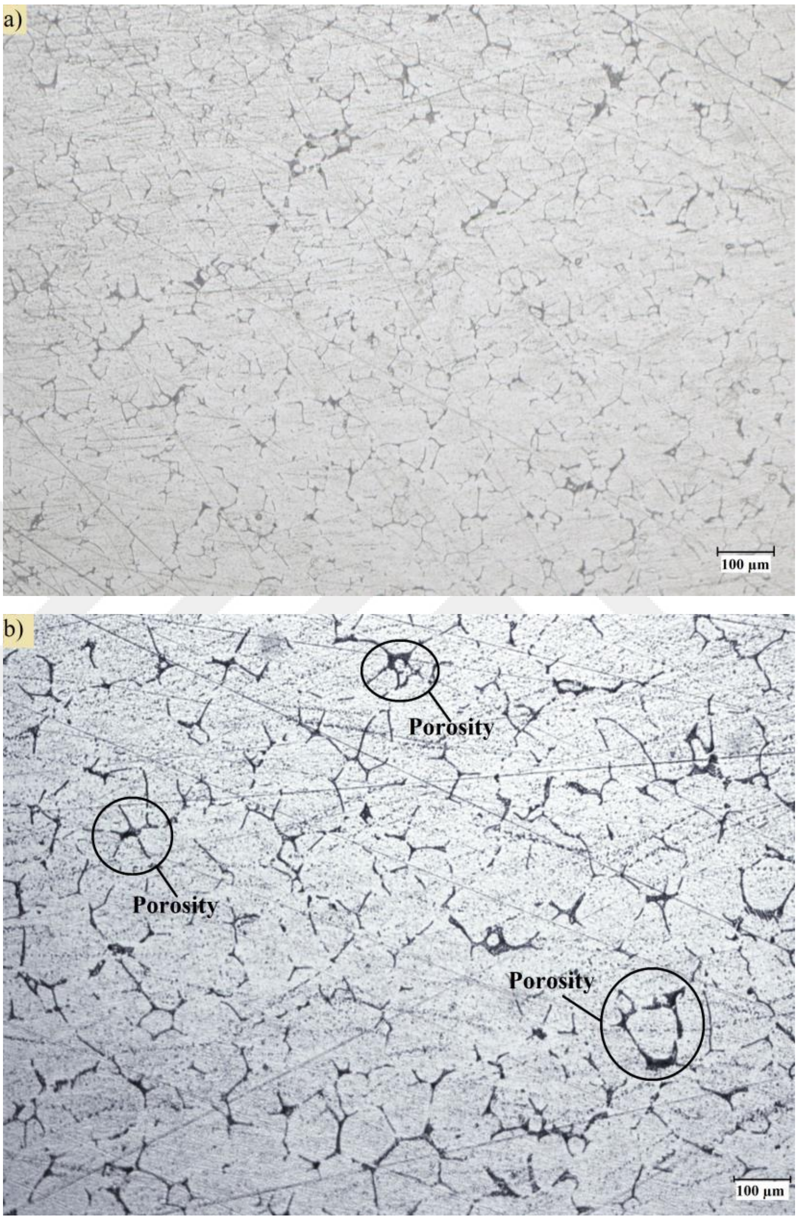


Figure 5.13 Microstructure of Sample 4

a) 10x b) 20x

As it can be seen in Figure 5.13, in some areas there are porosities in the structure. Figure 5.14 reveal the microstructure of sample 7 fabricated at 900°C. Equiaxed grains are seen in sample 7.

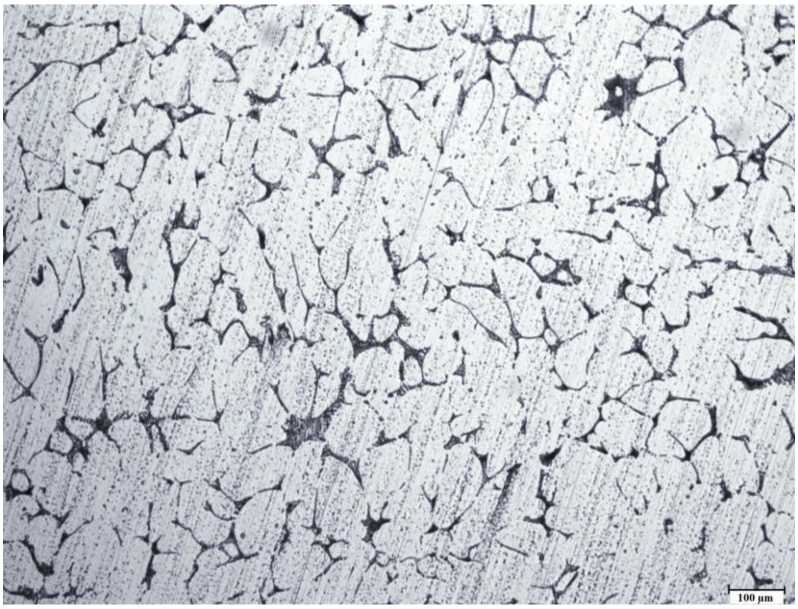


Figure 5.14 Microstructure of Sample 7 at 20x

In order to display detailed shapes, characterization and reinforcement in aluminum matrices, Scanning Electron Microscopy (SEM) analysis was conducted. Dark Al matrix, bright particles of MgO can be clearly observed in the image taken from the scanning electron microscope (Figure 5.15). SEM observations of sample 2 reveal homogeneous distribution of MgO particles in the matrix as shown in Figure 5.15.

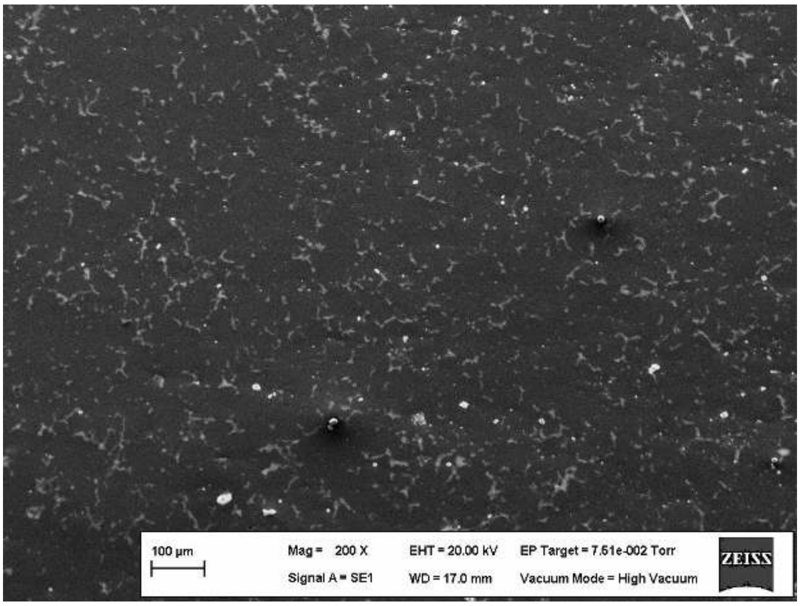


Figure 5.15 SEM micrograph of Sample 2

SEM images of Sample 9 and Sample 16 are illustrated in Figure 5.16. Casting samples manufactured at 900°C shows MgO agglomerations.

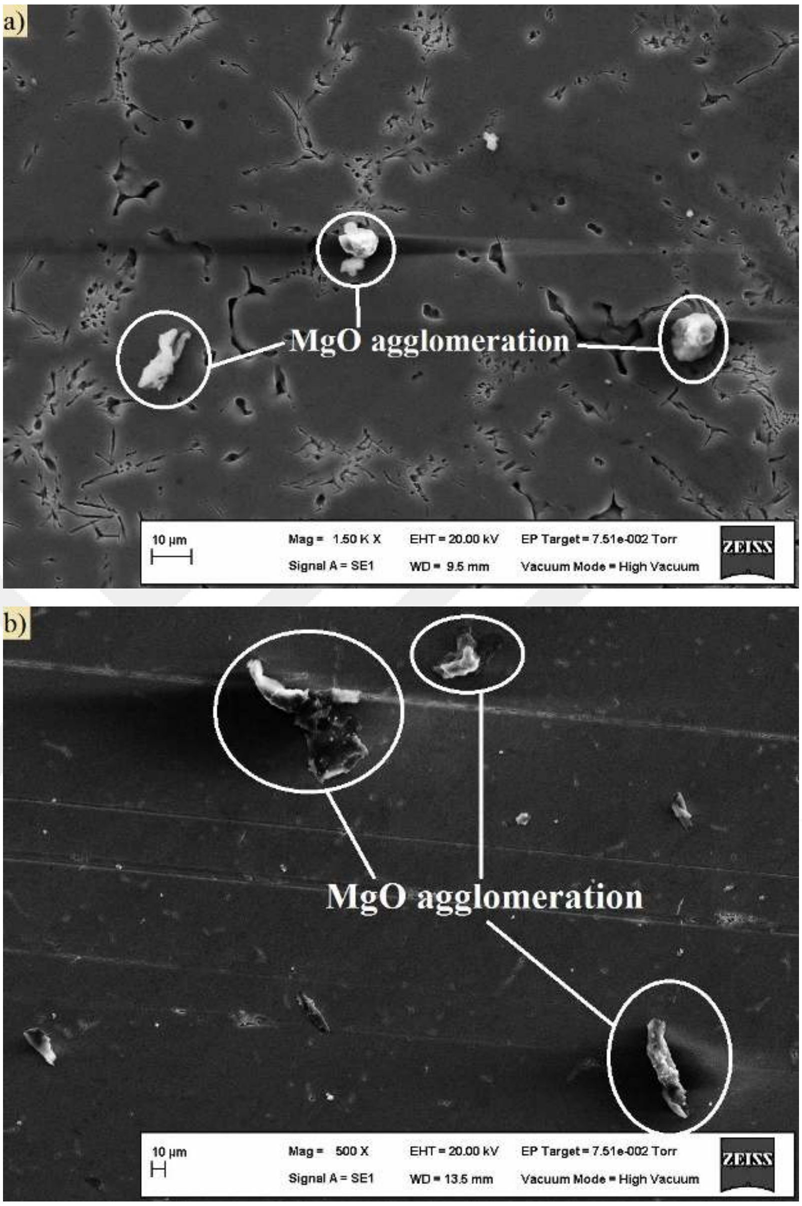
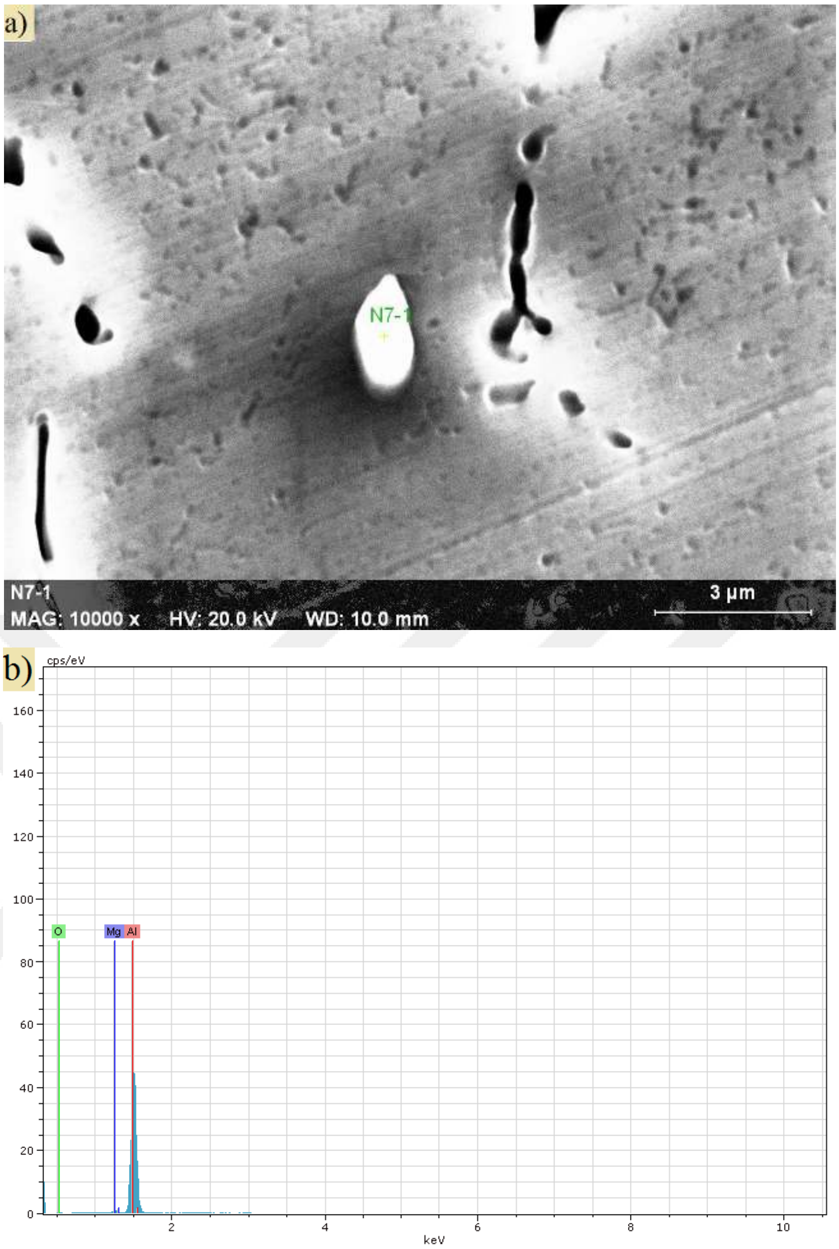


Figure 5.16 SEM micrograph of

a) Sample 9

b) Sample 16

EDAX analysis has been done in the scanning electron microscope and the result show that expected, there are Mg, O and Al elements. SEM and EDAX images of Sample 7 represent in Figure 5.17.

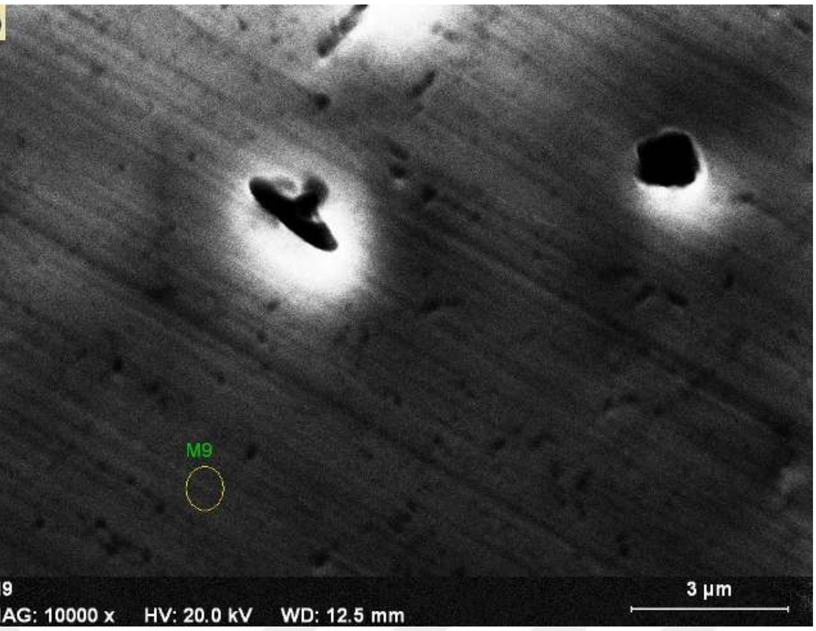


Element	C norm wt. %	C Atom at.%	C Error %
Al	**92.40**	88.33	3.0
Mg	**1.06**	1.13	0.1
O	**6.54**	10.54	1.1

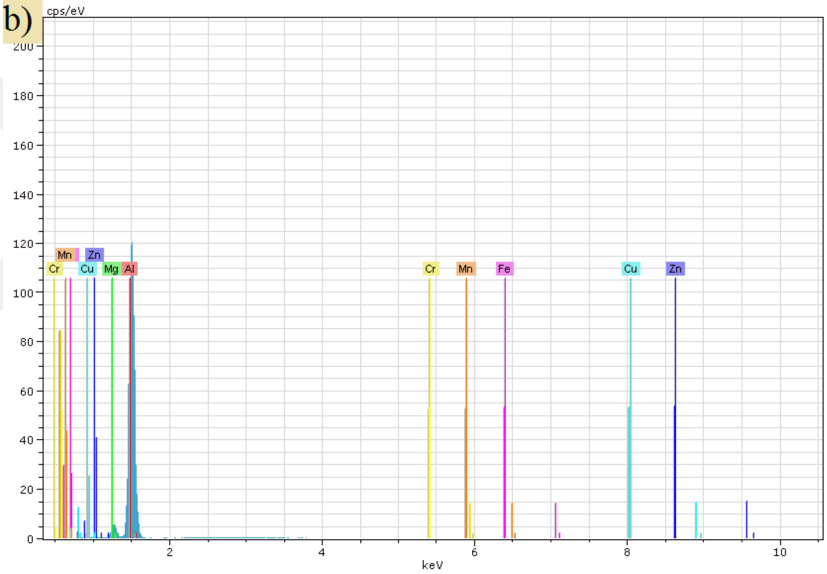
Figure 5.17 a) SEM and b) EDAX images of Sample 7

Figure 5.18 demonstrates the SEM and EDAX images of Sample 18 which is fabricated at 900^{o}C.

a)



b)



Element	C norm wt. %	C Atom at.%	C Error %
Al	95.74	95.84	5.3
Mg	3.40	3.78	0.2
Zn	0.35	0.14	0.1
Cu	0.21	0.09	0.0
Fe	0.16	0.08	0.0
Cr	0.07	0.04	0.0
Mn	0.07	0.04	0.0

Figure 5.18 a) SEM and b) EDAX images of Sample 18

5.5 DISCUSSION

The effects of particle size of MgO and Mg content on compression strength for each casting temperature were investigated. As it can be seen in design of experiment, 9 samples containing nano sized MgO and 9 samples containing micro sized MgO were obtained. Compression yield strength, compression strength and maximum strain of composites were depicted in Table 5.1 and Table 5.2.

Analysis of the tabulated values highlights that highest compressive strength is obtained for sample 6 (N6: 800°C, 5 wt. %, 0.5 wt. %). Under the same conditions for M series of experiments, compression strength is considerably low. As a general understanding, only two measurement of compressive strength of N series is lower compared with M series. This may be due to high porosity content, MgO agglomeration and also air absorption during high temperature casting, caused the decreasing of compressive strength. The rest of the measurements for compressive strength, totally 16 over 18, have higher values that is why it can be said that compressive strength of the N series composites produced in the same condition were higher than the M series composites.

Overall remarking points are:

- Magnesium is known to strengthen element for Aluminum alloys through solute solution strengthening [36-41]. As a primary alloying element for aluminum alloys, magnesium creates point defects within the lattice and act as a barrier to dislocation motion.

- Increasing the size of MgO particles in a constant temperature condition reduces the particle distance which enhances dislocation density that increases the amount of stress for dislocation [19, 42]. This is consistent with the result of this thesis that compression strength is improved by the implementation of MgO particles with aluminum.

- Different thermal expansion coefficient of Al (21.50×10^{-6} $^{o}C^{-1}$) and MgO (14.45×10^{-6} $^{o}C^{-1}$) results in stress concentration in the Al–MgO interface [19, 43]. This is another fact of validation of compression test results obtained in this thesis.

MgO particles are considered as a group of ceramics. Due to this fact, addition of MgO particles into aluminum enhances the hardness properties. All hardness values had been presented in Table 5.5. It is very obvious that increment in Mg amount directly increasing the hardness values for all temperature ranges. The highest hardness values for N and M series of composites were measured as 73.1 and 72.8, respectively. Another fact is hardness values of the nano composites are mostly higher than micro composites. This can be attributed to solid solution of Mg element and the presence of MgO particles.

Sample 6 and sample 15 have the lowest theoretical density. By increasing the content of reinforcement, the measured densities of specimens were increased but it decreased with the addition of Mg (for sample 1 and sample 2). It was observed that the measured density increased from sample 2 to sample 3. This may demonstrate the effect of low wettability and agglomeration at high content of reinforcement led to the formation of porosity. The lowest measured density values of nano and micro composites were observed for sample 9 and sample 18, respectively. This is due to increasing temperature. It is well known that temperature plays an important role to increase the wettability of particles[2, 18].

The minimum and maximum porosity percentages were observed for sample 8 and sample 4 for nano composites and sample 10 and sample 14 for micro composites, respectively. The increasing MgO content and casting temperature rise the porosity percentage but the addition of Mg decreases it. This may be due to increasing agglomeration by casting composites well above the melting point [2, 18].

Microstructural analysis was carried out for samples. From the analysis of captured pictures it is clear that dark regions are aluminum and bright particles are MgO.

Despite the highly concentrated agglomerations and porosities in some areas of composites, in general, particle distribution was observed to be homogeneous. In other words, fine nano and micro-sized MgO particles were well embedded in the aluminum-magnesium matrix and just a partial agglomeration in composites with high content of MgO can be detected. It is well known that the aggregation of MgO nanoparticles is more probable in casting composites with different casting temperatures show similar microstructural appearances.

CHAPTER 6

CONCLUSIONS

6.1 INTRODUCTION

This chapter is related with the conclusions drawn from this study on the metal matrix composite reinforced with nano and micro MgO.

6.2 CONCLUSIONS

In this thesis, Al-Mg composites reinforced with nano and micro MgO particles were successfully fabricated by stir casting method. Effects of nano and micro-sized MgO particles, Mg and casting temperature on mechanical and physical properties and microstructure of metal matrix composite were investigated.

Three different casting temperatures, three different MgO and Mg contents by weight were selected and Taguchi method was used as a design of experiment. According to L9 array, totally 18 composites reinforced with both nano and micro particles were fabricated.

Mechanical tests such as compression and hardness test and physical test such as density measurement and porosity were tabulated. Microstructural examinations were executed to understand the implementation of MgO particles and the relation with the mechanical properties.

As a result of this study;

- By using the stir casting process, MgO-reinforced aluminum composites were successfully obtained.

- Particles were homogeneously dispersed despite some agglomeration exists in some areas of few samples.

- From the response tables of signal to noise ratio and ANOVA which are associated with the compression strength, it is noticed that the casting temperature has a significant role in casting process and followed by content of MgO and content of Mg respectively.

- It is drawn that the hardness value is increased with the increment of Mg content.

- According to Rockwell 15T, highest hardness value is obtained as 73.1 on the sample N9.

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