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Hardness Characteristics of Tio_2/Al_2o_3 Reinforcement in Mg Composites

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ABSTRACT

The Mg based composites with the element addition of 3Zn, 0.5Mn and the reinforcement of ceramic particles are TiO_2 and Al_2O_3 are added to form an Mg-composites through powder metallurgy method. The examination is mainly concentrated on the effect of multi ceramic particles such as TiO_2 and Al_2O_3 inclusion in the composite matrix for the characteristics of hardness variation. Optical and scanning electron microscopy are used for the micro structural variation. X-ray diffraction is used to analysis the phases available in the matrix. Many intermetallic phase are formed during heat treatment process which reduces the porosity of the composites. The hard ceramic particles inclusion, hot extrusion and heat treatment/stress-relieving mechanism enhances the micro hardness of the composites.

Key words: *Hardness, Heat treatment, Microstructure, Mg based composite, Reinforcement*

INTRODUCTION

The composites are formed by two or more materials are combined together to enhanced the physical, chemical and mechanical properties of the composites over the individual materials. In general, the strength of composite material formation is depends with the reinforcement of metal/material into the composite matrix [1]. Now-a-days, composites are formed for using aerospace, sporting goods and military equipment. Magnesium (Mg) is one of the metal which is widely used in engineering applications next to iron and aluminum. Magnesium and magnesium based alloys/composites are having noticeable properties such as lightweight, high strength to weight ratio, better machinability, and excellent damping capacity. Therefore, magnesium composites are utilized in engineering structures which includes the above said applications. In contrast, these applications are limited in the field which requires high elastic modulus, hardness and electricity [2].

The characteristics of the Magnesium metal can be enhanced by the alloying/composite elements through heat treatment. The researchers suggested that the inclusion of ceramic particles is in the magnesium matrix could be improved the mechanical characteristics of

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magnesium alloys/composites [3]. The included/reinforced metal particles in the matrix forming different phases. The phase formation is depends with the nature of chemical reaction taken place between the reinforcement/inclusion and matrix material. The formed secondary phases could be improved the interfacing strength of the matrix material [4, 5]. In light weight material application, Mg alloys and their composites are exhibits satisfied mechanical properties.

Aluminum (Al) matrix composites are also used in light weight engineering applications. Many researchers are giving attention to Al matrix for utilizing in industry. Most of the research revealed that the Mg based composites are used in structural, biodegradable and biomaterials for human body Implants [2, 6-8]. Recently, Al_2O_3, CNTs, Y_2O_3 etc., are the reinforcement added to the Mg matrix to enhance its mechanical properties. In-general, the most of the studies are focusing the Mg composites based on their characteristics improvement through varying the grain size, weight fraction, morphology, reinforcement particles distribution and by altering the composites forming techniques [9–12]. Recent years, several alloys/composites were developed using titanium oxide (TiO_2) as the reinforcement in Mg matrix through powder metallurgy methods [3, 13, 14]. The incorporation of TiO_2 particles improved the strength of the composites than the pure form of Mg metal.

Hot extrusion is a process which improves the mechanical characteristics of the alloys/composites. A noticeable characteristics of the hot extrusion process is to alter the microstructure of the elements present in the composites. The microstructural alteration process tends to form a new intermetallic phases. This can be done through dynamic recrystallization and dynamic precipitation occurring during hot extrusion. Al_2O_3 is one of the alloying element in Mg alloy. This element is improved the physical and mechanical properties of the Mg alloy [15-17]. Similarly, the AZ81 composite reinforced with Alumina exhibits possible improvements in tensile and compressive characteristics when comparing AZ81 base alloy [18]. This work, prepared and studied the Mg-3Zn-0.5Mn based alloys and the alloy-based composites reinforced with TiO_2/Al_2O_3 particulates. The hardness characteristics of the prepared composites are investigated.

EXPERIMENTAL PROCEDURE

The composite elements are of purity 99.99% and 200 mesh is chosen for preparing an Mg-composites. The elements added to form an Mg-alloy is Mg, Zn, and Mn metal powders. Similarly, the ceramic reinforcements are TiO_2 and Al_2O_3 powders. The Zn and Mn metal powders added to the matrix by 3wt% and 0.5 wt%. Similarly, the reinforcement of TiO_2 is as

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0.1wt% and the Al_2O_3 added is varied by increasing wt%. The elements are mixed thoroughly according to their respective weight at 350 rpm for 1 hour to form a billet of sizes 50mm diameter and 30mm height using powder metallurgy technique. The compacting pressure used for this purpose is 690 Mpa. This compacting purpose a hydraulic power press is utilized. A 3:1 ball feed ratio carried out at100 rpm speed in ball milling process to form a homogeneous mixing and blending of the metal powders.

The billets are sintered at a temperature 400°C for one hour in argon environment. The secondary strengthening process is done on the billets through hot extrusion. The extrusion process is carried out the temperature of 400°C with an extrusion ratio of 5.4:1. The extrusion process is executed on the specially attached hydraulic power press at a speed of 0.2 to 0.5 mm/s. after forming rods, the rods are gone for heat treatment to relieve the stresses. The heat treatment process is carried out the temperature of 260°C for 20 minutes in argon environment using electronic muffle furnace. Harness characteristics is analyzed by as-extruded and heat treated composites. Also, analyzed by adding 1% and 2% alumina powder is in the matrix by un-altered the other elements.

The micro structural characteristic study is done through optical microscope and scanning electron microscope. The optical microscope used for this purpose is MVHD-1000 AP/MP, Shanghoui Dahens Optics and Fine Mechanics Co. Ltd. A digital micro hardness tester is attached to this machine. Before capturing the image, the samples are etched through etchant. The etchant consists of 1.5 grams of picric acid, 25 ml of ethanol, 10 ml of acetic acid and 10 ml distilled water followed by distilled water cleaning.

RESULTS AND DISCUSSION

The morphological changes could be occurred during sintering and hot extrusion process which can be represented in the figure 1. From the optical microscope, it is clear that the elements present in the matrix could be uniformly distributed in the composite matrix. Also, the alumina powder distribution is visible in the entire micrographs which is showed that the reinforcement is properly added into the alloy matrix. From the micrographs it is clear that the cavities, voids and micro pores are present in the composites. The cavities are reduced by the heat treatment process. There is a slight difference occurs between the increasing reinforcement content added into the matrix in terms of cavities as shown in the fig. 1 (b) and (c). The increasing reinforcements could be reduces the microspores and voids during extrusion.

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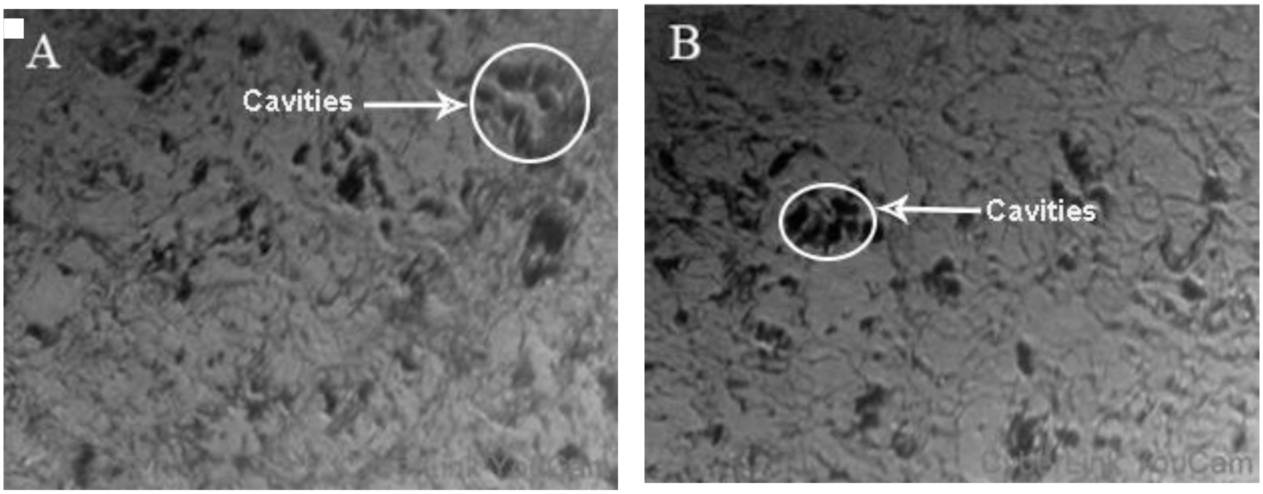

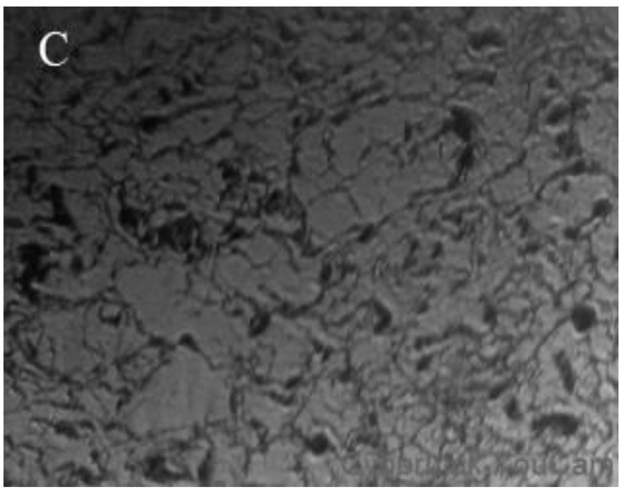

Fig.1. Microstructures of Mg composite
(a) as-extruded with 1%Al_2O_3 reinforcement
(b) heat treated with 1%Al_2O_3 reinforcement
(c) with 2%Al_2O_3 reinforcement

The higher deformation could be occurred on the composite matrix due to extrusion process. This deformation caused a rearrangement of reinforcement leads to a uniform distribution of both the reinforcement into the alloy matrix. Also, the rearrangement/uniform distribution of the reinforcement occurring during hot extrusion which could be filled voids and pores present in the composite matrix. The filled voids and pores are leads to increase the bonding strength of the matrix due to interfacial reaction taken place between the reinforcement and the alloy matrix [19]. The formed grains are evident for the recrystallization taken place during sintering and hot extrusion process. The stretched grains are marked in the fig. 2(C) is evident for the grain deformation phenomena.

The grains formed are near equiaxed for after heat treatment samples than the as extruded samples as shown in the fig. 2. The grain refinement is takes place to increase the sizes of the grains. The formed grains are higher in size for the heat treated Mg-composite samples than the as – extruded samples. The grain growth is mainly due to the ability of reinforcement TiO_2 and Al_2O_3 particles to nucleate the Mg-matrix grains during recrystallization process. Also, these

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particles are restricted the grain growth of the composites elements causing grain pileup. The pileup of grains are increases its size. The increasing sizes of the grains are reducing the number of grain boundaries. The reduction in grain boundaries enhanced the interfacial reaction between the matrix grains and reinforcement particles which leads to increase the bonding strength of the composites.

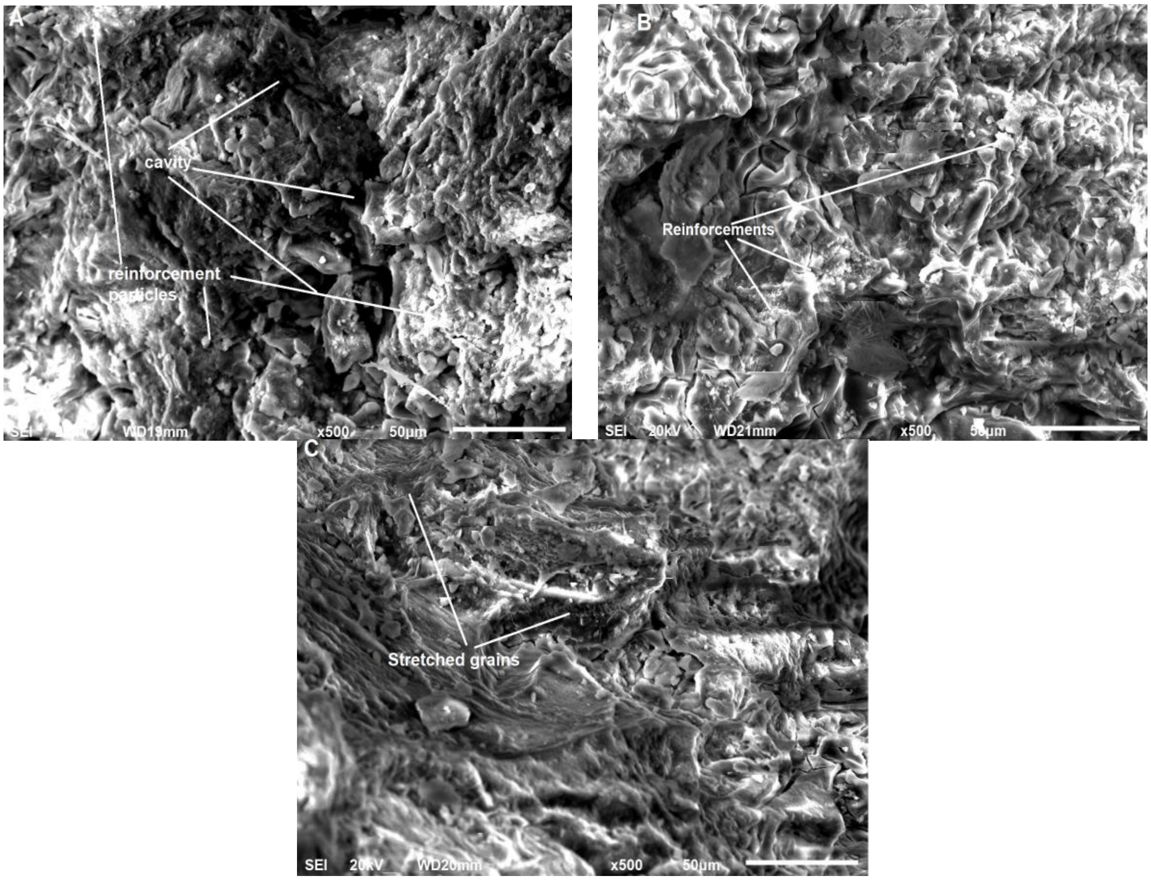

Fig.2 SEM of Mg composite (A) as-extruded Mg- composite with 2% Al_2O3 (B) heat treated Mg-composite with 1% Al_2O_3 (C) heat treated Mg-composite with 2% Al_2O_3

When comparing the figures. 2 (A), (B) and (C), it is clear that the heat treatment process decreases the cavities formed during the composite formation through powder metallurgy method. The reducing order of cavities are visible in the figures 2(C) and (B) respectively by increasing reinforcement particles. In-contrast, higher cavities are visible in fig. 2 (A). In-general, Archimedes principle is the easiest method to find the porosity and density of the composites [12]. In this work, the same method is used to calculate porosity and density of the composites. The distilled water is used as an immersion fluid for the porosity measurement. A digital type physical balance is used to measure the weight of the samples. The obtained density and porosity of the composites are depicted in the table.1.

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Table 1: Density and porosity of Mg-composites

Specimen	Condition	Density (g/cm^3)	Porosity (%)
Mg-Composite without Al_2O_3	As-extruded	1.7246	37
Mg-Composite without Al_2O_3	Heat treated	1.7334	35
Mg-composite with 1% Al_2O_3 reinforcement	As-extruded	1.7602	34
Mg-composite with 1% Al_2O_3 reinforcement	Heat treated	1.7695	32
Mg-composite with 2% Al_2O_3 reinforcement	As-extruded	1.7921	32
Mg-composite with 2% Al_2O_3 reinforcement	Heat treated	1.8012	29

The density and porosity measurement reveals that, a noticeable reduction in the porosity occurs by increasing density of the composites. The addition of alumina powder added into the Mg-matrix could increases the density of the composites. In addition, the heat treatment/stress relieving process could further increases the density of the composites for all the cases as shown in the table 1. The decreasing porosity is due to the release of entrapped gases present inside the composite during composite formation and by filled the voids and pores during heat treatment process. During heat treatment, oxidation of Mg might to be happened is the other reason for reducing porosity of the composites.

Table 2: Micro hardness value of Mg-composites

Materials	Condition	Average micro hardness (VHV)
Pure Mg	Pure form	48
Mg-3Zn-0.5Mn/0.1TiO_2	As-extruded	69
Mg-3Zn-0.5Mn/0.1TiO_2	Heat treated	72
Mg-3Zn-0.5Mn/0.1TiO_2/1Al_2O_3	As-extruded	76
Mg-3Zn-0.5Mn/0.1TiO_2/1Al_2O_3	Heat treated	81
Mg-3Zn-0.5Mn/0.1TiO_2/2Al_2O_3	As-extruded	84
Mg-3Zn-0.5Mn/0.1TiO_2/2Al_2O_3	Heat treated	92

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The micro hardness of pure Mg and the prepared Mg-composites are measured through the digital vickers micro-hardness (VHV) tester using a pyramidal diamond indenter. The specification of the micro-hardness test is 136° included angle, 25gf indenting load and 20s as dwell time. The obtained results are depicted in the table 2. The pure Mg exhibits lower micro hardness when compared to the Mg-composites and the increasing quantity of reinforcement addition further increases the hardness value of the composites. Moreover, the heat treated composite samples are exhibits a highest hardness. The TiO_2 particle reinforcement increases the hardness of the composites but the quantity added into the matrix is very low as compared to the other matrix element. Therefore, the heat treatment carried out on the Mg/ TiO_2 composites are not much effected to increase the hardness of the composites when compared to Al_2O_3 added composites.

The addition of Al_2O_3 particles into the composite matrix implies higher hardness than the TiO_2 particles inclusion. This could be due to the temperature used for processing the composites because the melting temperature of the titanium is higher than elements present in the composites. Generally, the addition of hard ceramic particles into the composite matrix could increases the hardness of the composites. In-contrast, the extrusion/heat treatment temperature plays a major role for the formation of composites. The hot extrusion process contributes a dynamic recrystallization and precipitation of grains to enhance the hardness of the composites.

The grains of the Mg- composites are evaluated through X-ray diffraction (XRD) using a Siemens X-ray diffractometer with Cu-Kα radiation. Phases formed in the composites are obtained from the diffractograms. The scanning angles (2θ) with a step size of 0.02° per 2 s is used for this purpose. The obtained XRD is depicted in the fig .3. The mentioned figure revealed the available phases of the composites. Some of the intermetallic phases such as MgO, Mn_2O_3, Mg_2Zn_3, Ti_2O_3 etc., are shown in the XRD along with the primary phases Mg, TiO_2, Al_2O_3. These formed intermetallic phases are possible to fill the micro voids and contributing hardness improvement. The multi particles reinforcement in to magnesium matrix composites is one of the suitable method to enhance the hardness value of the Mg-composites.

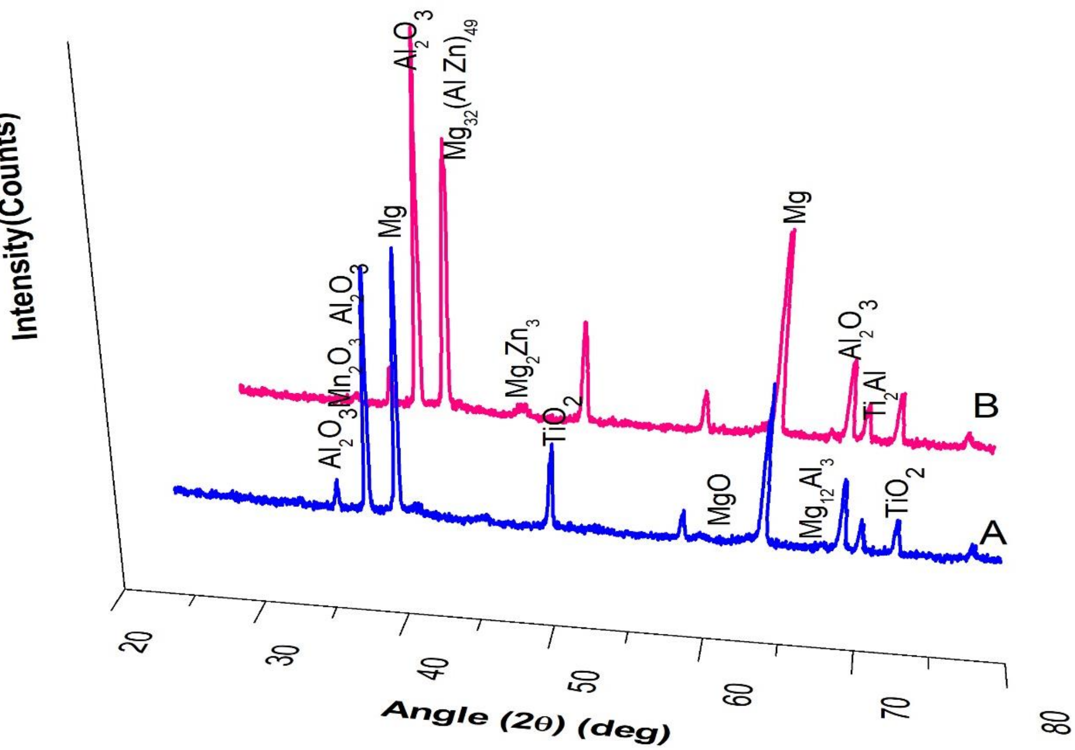

Fig. 3. X-ray diffraction of heat treated Mg-composites (A) 1%Al_2O_3 and (B) 2%Al_2O_3

CONCLUSIONS

- ✓ The uniformly distributed reinforced phase into the Mg matrix is to increases the surface integrity between both the phases leads to improve the strength of the composite
- ✓ Hot extrusion and heat treatment process are enhances the harness characteristics of the composites through dynamic recrystallization and grain refinement
- ✓ The refined grains are increases the area of grain boundary through reinforcement of TiO_2 and Al_2O_3 ceramic particles which improved the hardness of the composite
- ✓ The reinforced TiO_2 and Al_2O_3 particles are arrest the dislocation movement leads to occurs an grains pileup yields increases the strength of the composites
- ✓ Forming an intermetallic phases during extrusion and heat treatment are also a reason for the increasing hardness.

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