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Synthesis, Characterization and mechanical behavior of AZ91E/Ni coated Al_2O_3 (p) reinforced composites fabricated by Semi Solid Stir Casting

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Abstract— The developments of any composite always depend on the strong wettability of reinforcement phases in matrix. One way of improving the wettability is to apply a layer of coating on reinforcement particles. The present study aims at the development of Ni coating on nano Al_2O_3 ceramic particles and to reinforce it in AZ91E magnesium matrix material. The Electroless plating method has been employed to coat the particles and Semi Solid Stir Casting technique was used to prepare the composites. Several weight fractions of reinforcement are considered to analyze the behavior of the fabricated composites. FESEM and XRD analysis has been carried out to investigate the distribution of particles and phase characteristics of the proposed material. The physical and mechanical behavior of the material was examined through density measurements, hardness, Elastic modulus, ductility and tensile strength calculations. The metal coating on reinforcement aids to promote metal – metal bonding interface reactions which resulted in improved properties of the composite.

Keywords— AZ 91 E Mg alloys, semi solid stir casting, mechanical properties, FESEM,XRD

I. INTRODUCTION

Development of the usage of alternate materials in automotive industries has been significantly increased over the past few decades. The researchers proved Magnesium as a substitute to aluminum in many parts of an automobile [1-3] because of its low density, good strength with an additional advantage of recyclability. However, pure magnesium is rarely used because of high reactivity and poor corrosion resistance and AZ91 is widely used magnesium alloy having good mechanical and thermal properties [4]. The ceramic particles have high hardness, strength and melting point. SiC,Tio_2 and Al_2O_3 reinforcements are mostly used as reinforcement Magnesium composites [6-7]. Al_2O_3 particles can exist in various crystalline phases and produces the most stable hexagonal alpha phase at elevated temperatures, which will be useful in structural applications. Al_2O_3 particles provides good strength as well as enhanced ductility in magnesium composites [8]. the composites prepared with Al_2O_3 ceramic reinforced Mg MMCs have high potential in structural applications in terms of improved mechanical properties [9].

Semi Solid Stir Casting was widely used to prepare the composite [17]. The ceramic reinforcements are added to molten metal in between liquidus and solidus zone. This gives good adhesion of ceramics over the molten liquid metal.the preparation of these composites in Semi Solid Stir Casting needs a special attention as it contains ceramic and metal interactions. Wettability is a key factor in deciding the properties of the composite. The ceramics always provide low wettabilty in liquid matrix material.

Nickel, known for its excellent corrosion resistance, which can be used as an alternative for Wear and fatigue resistance. A few methods like co-precipitation method, sol-gel method, Electroplating, Heterogeneous precipitation method [12] were employed earlier to prepare the coated powder. The electro less plating method is another preference with an advantage of simple operation and capability of mass production [13].

The Nano particles are proved to be an effective strengthening phase when reinforced in MMCs [14]. Leon et.al. [10] prepared Ni coated Al_2O_3, SiC particles of varying sizes at micro level using Electroless and observed uniform coating over the surface. But, the nickel coating on these Ultra fine particles was very limited [15, 16]. The characterization of these powders are also mainly focused on coating thickness and nature of coating.., the work focusses on the fabrication of AZ91E Magnesium based composites using semi solid stir casting method after the surface modification methods (Ni Coating) applied to the ceramic reinforcements (Nano Sized Al_2O_3 particles) via electroless plating technique. The preparation, metallurgy, Physical as well as Mechanical responses of these cast composites with changes in weight fractions are presented in later sections.

II. EXPERIMENTATION

A. Nickel coated alumina powders

The 99% pure α-Al_2O_3 powders with an average particle size 50 nm, supplied by M/s.United Nano Tech Products Limited (UNTPL), Howrah, were used in this experiment. Electroless plating technique is used to coat Ni on the surface of these particles. The powders are prepared with the best experimental conditions mentioned by Sameer et.al [13]. The process depends upon the catalytic reduction of a metallic ion in an aqueous solution containing a reducing agent. In this experiment, Nickel Chloride Hexahydrate ($NiCl_2.6H_2O$) is used as a source of Nickel ions while Sodium hypophosphite

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($NaH_2PO_2.H_2O$) is used as the reducing agent. At first, the powders are pre-treated with various chemicals like acetone, $Sncl_2$ and $Pdcl_2$ to clean the surface and to make the powder ready for attracting the nickel. Then these powders are immersed in an electrolytic bath to attract the nickel. The temperature, pH are maintained constant throughout the operation.

B. Specimen preparation

AZ91E alloy was purchased from M/s.Exclusive Magnesium Pvt. Ltd., Hyderabad with the following chemical composition given in Table I.

Table I: Chemical Composition of AZ91E alloy

Alloy	% wt
Al	8.93
Zn	0.86
Mn	0.28
Cu	<0.001
Si	0.13
Fe	<0.001
Mg	Remaining

The casting process was similar to the procedures earlier reported [17]. Mild steel crucible, flux (20% KCl, 50% $MgCl_2$, 15% MgO, 15% CaF_2, wt%) and a high purity (99.98%) argon gas (IOLAR-1) supplied by Linde India Limited, Jamshedpur was used in the preparation of the composite. The raw materials, moulds, flux are pre-heated to avoid the casting defects. AZ91E was introduced into mild steel crucible when the furnace temperature reaches 250ºC. Then temperature was raised to 700ºC and kept for 15 minutes to make the melt homogenous. The melt was slowly cooled down to 590ºC to introduce the reinforcement particles as the liquidus and the solidus temperature of the AZ91 alloy are found to be 596ºC and 468ºC [17]. After cleaning the surface of the melt, the preheated Nickel coated nano Al_2O_3 particulates were wrapped in an aluminum foil and added the melt. The mixture was stirred using a four blade mechanical stirrer with a rotation speed of 450 rpm for 3 minutes. The ingots of 15mm diameter with 150 mm length are obtained from casting. The procedure was repeated for all corresponding weight fractions of reinforcements to get the samples.

C. Density Measurement

The density of Ni coated Al_2O_3 particles reinforced with AZ 91E and pure AZ 91 E specimens are calculated experimentally using Archimedes principle with distilled water as immersion fluid. The weights are measured by using an electronic balance. The average value of 5 density readings was noted for each sample.

D. Microstructural examination

For Metallographic studies, the sample was taken from the middle of ingot and Microstructural characterization was carried out for AZ 91 E alloy as well as for reinforced composites. The samples were polished using an automatic polisher to produce mirror like surface on the samples. Particle distribution and presence of elements were examined using Field Emission Scanning Electron Microscope (FESEM) attached with EDS (TESCAN-MIRA coupled with QUANTAX 200). The phase analysis also carried out using X-Ray diffractometer (SMART Lab, Rigaku, Japan) with a scan speed of 3 deg/min between a scanning range of 20-90 degrees.

E. Mechanical properties evaluation

Mechanical Properties of the composite with different weight fractions of reinforcements were evaluated in terms of Micro hardness, Macro Hardness and Tensile properties. Hardness studies were carried out to study the effect of reinforcement and its quantity on hardness. The Micro hardness was measured with 100gf load on the samples using Digital Vickers hardness tester supplied by M/s.Daksh Quality Systems (bearing the Model No: HVD 200 MP). Macro hardness was evaluated for a load of 10kg with a loading period of 15 sec using FIE M-50 Machine. The hardness values were measured by taking an average of 8 readings. Tensile testing was done as per ASTM B 557 Standard on UTN 40, FIE machine. Three readings for each composition were noted for tensile testing.

III. RESULTS AND DISCUSSIONS

A. Density of cast composites

The experimental densities values of different weight fractions are shown in Table 2. It is observed that the density values increased with increased in the wt% of reinforcements. Pure az 91 E mg alloys has low density compare to other composites .The increased density may be due to the presence agglomeration of reinforcements because of higher percentage of reinforcements and may be due to gas molecules entrapment. The similar trend was reported in case of $AZ91E/Al_2O_3$ as well as in AZ91D/SiC Composites [18].

Table II. Density values of AZ91E alloy and processed composites

Material	Designation	Experimental density(g cm^{-3})
AZ91E	Sample P	1.806
AZ91E+1wt% Nickel Coated Al_2O_3	Sample Q	1.825
AZ91E+2wt% Nickel Coated Al_2O_3	Sample S	1.842

ISSN No: - 2456 – 2165

AZ91E+3wt% Nickel Coated Al_2O_3	Sample U	1.856

B. Microscopy Results

Fig.I represents the microstructure of cast composites observed by FESEM of different samples as mentioned. . In Fig.5 the Ni coated alumina particles were uniformly distributed in the matrix region and the non uniformity increases by increasing the reinforcement percentage (above 2 percentage weight fraction). The increase in the reinforcement percentage causes the agglomeration of particles due to higher surface energy in Sample U and can be seen in. The EDS analysis of sample U is shown in Fig.6. The presence of nickel along with Magnesium, Aluminum and Nickel can be observed in the selected region. It was further noticed from Fig 6 (d) that nickel is uniformly identified in the matrix confirming that the Ni coated particles are uniformly distributed in the matrix of Sample S. X- Ray Diffractions of composite sample U was shown in figure 7 . The peaks obtained by X- ray diffraction composite sample are compared with JCPDS (Joint Committee for Powder Diffraction Standards) data (Table 3). The presence of nickel in the XRD for Sample U can be clearly observed between 42-45 degrees. [19]. The XRD analysis results confirm the presence of Ni and Al_2O_3 particles in the prepared material Sample S along with preliminary tests.

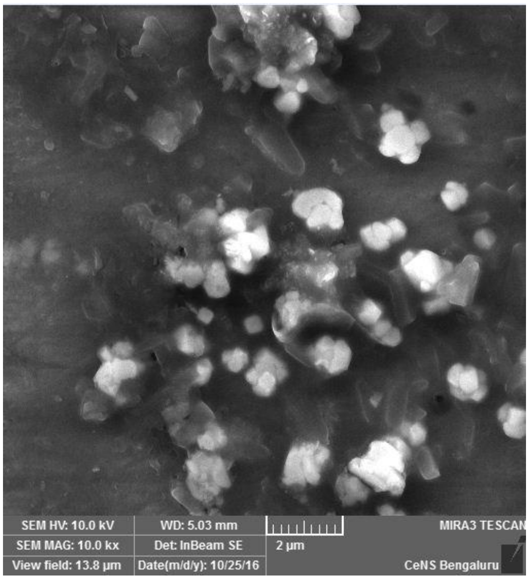

Fig I. FESEM images of Sample U which has aggloermations

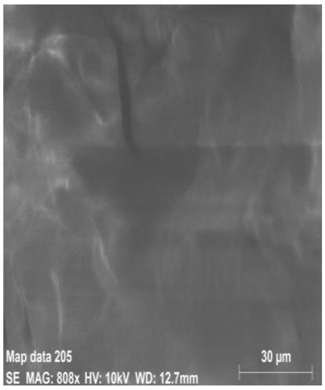

Fig II. SEM and Elemental Analysis

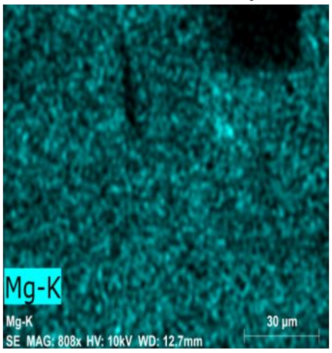

Fig III. SEM and Elemental Analysis

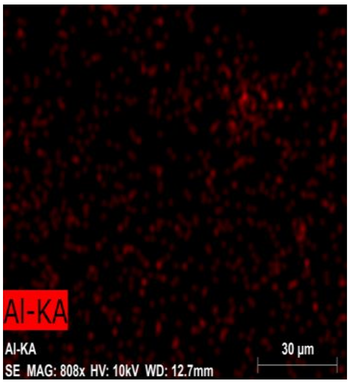

Fig.IV. Elemental mapping of Sample S

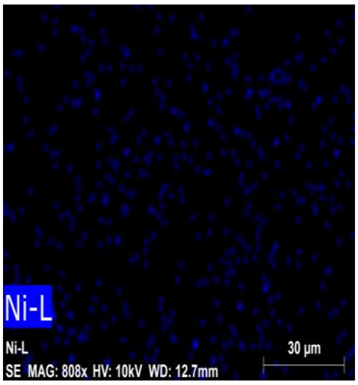

Fig V. Elemental mapping of Sample S

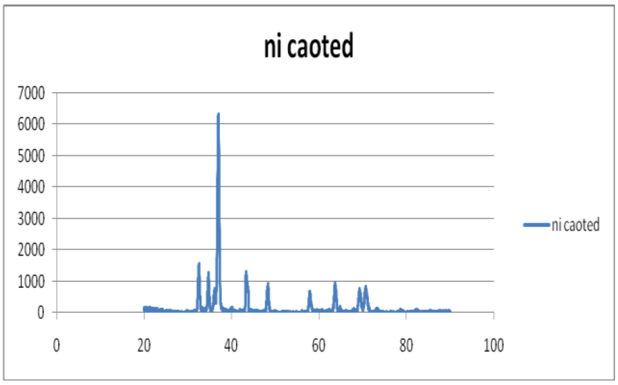

Fig VI. XRD of Sample S

Table III: JCPDS data

S. No.	Element	JCPDS File No.	2 theta	Intensity	h k l
1	Corundum Al_2O_3	75-0787	25.794	692	0 1 2
2			35.471	999	1 0 4
3			38.091	472	1 1 0
4			43.732	956	1 1 3
5			53.026	473	0 2 4
6			58.049	930	1 1 6
7			67.139	354	2 1 4
8			68.632	534	3 0 0
9			77.715	151	1 0 10
10	Magnesium Mg	65-3365	32.185	242	1 0 0
11			34.398	268	0 0 2
12			36.619	999	1 0 1
13			47.820	142	1 0 2
14			57.384	148	1 1 0
15			63.072	153	1 0 3
16			68.646	149	1 1 2
17			70.024	104	2 0 1
18	Nickel Ni	04-0850	44.505	100	1 1 1
19			51.844	42	2 0 0
20			76.366	21	2 2 0

C. Mechanical Properties:

Hardness:

The hardness values of both macro and micro for different samples were shown in [fig 8 fig 9]. The micro hardness of sample P is 64.94 VHN while Sample S is 87.94 VHN similarly the macro hardness of Sample P and S are 80.87 VHN and 113.89 VHN. From the graph it can be seen that the maximum Hardness was observed in Sample S both at macro and micro levels. The hardness values are increased with increase in percentage of reinforcement up to 2% of weight fraction and then suddenly decreases. The observed increased hardness is due to the uniform distribution of reinforced ceramic particles help to increase strengthening mechanism and also restrict the matrix deformation The Nickel coating helps to provide strong bonding between ceramic and matrix phases. After 2% of weight fraction, the increase in reinforcement resulted in decrease of hardness in case of sample U. This is due to formation of clusters and non uniform distribution of particles. It may also be noted that hardness state obtained here is similar to the findings reported for ceramic reinforced magnesium matrices [17, 18] but with increased values

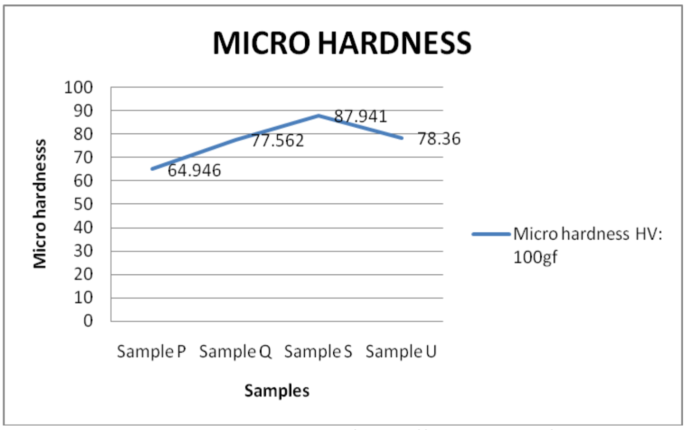

Fig VII. Micro hardness values of the different weight fractions

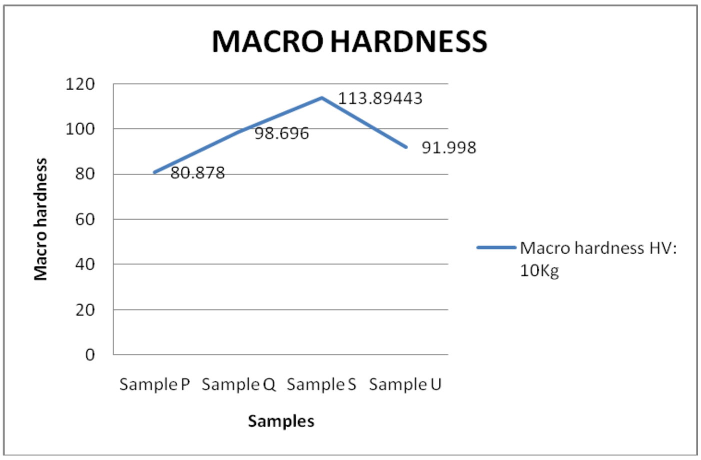

Fig VIII. Macro hardness values of different fractions

Tensile Behavior :

The Yield strength of the AZ91 E is 96.4 MPa and yield strength is found to be increased with an increase in percentage of reinforcement from table 4. This increase in yield strength has a similarity with the results obtained by researchers, who have reported that the yield strength of the particle reinforced composites is highly dependent on the volume fraction of the reinforcement [18]. Yield strength of AZ91E/ Al_2O_3 Composite was 150Mpa [10] and for the sample U mentioned here is 159.3 MPa. This may be because of Ni coating on Al_2O_3 particles provide strong interactions with the matrix material so that more load can be transferred to the reinforcement.

The tensile strength of casted Sample P is 162 MPa while for 2% reinforcement it was 221 MPa and decreased to 203 MPa [23] for Sample U. It is clear that tensile strength of composites are greater when compared to cast AZ91.With an Increase in Al_2O_3 content the agglomerations are increased so that the defects are induced around the Al_2O_3 particles due to the difference in the thermal expansion coefficients of AZ91 and Al_2O_3.This might result in de bonding of the interface and decrease in UTS of the composites.

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The Elastic modulus of Sample P is 43.45 Gpa while the Sample U has 53.33 Gpa (Table 4). The increment in Young's Modulus is due to the dispersion of the fine and hard reinforcement particles (Ni Coated Alumina) in the matrix (AZ 91 magnesium alloy) drastically blocks the motion of the dislocations because of strong bonding with the matrix and strengthens the magnesium alloy composite.

The ductility was measured in terms of percentage of Elongation and can be affected by reinforcement content and matrix alloy. The addition of nickel (known for good ductility) as a coating layer on the particles increases the ductility of base material. Srivatsan et.al [19] has also observed increase in ductility than the base metal in case of hybrid composites prepared with AZ31 as matrix, Ni and Al_2O_3 as reinforcements. The ductility values were decreased with an increase in reinforcement because of void nucleation in progress with increased amount of reinforcement [18,22].

Table IV. Mechanical behavior of Cast Specimens

Samples	Elastic Modulus (GPa)	Ultimate Tensile Strength (MPa)	Yield Strength (MPa)	% of Elongation (mm)
Sample P	43.45	162.77	96.4	3.08
Sample Q	48.20	191.97	129.56	3.56
Sample S	51.02	221.09	155.05	3.15
Sample U	53.33	203.03	159.3	2.07

IV. CONCLUSIONS

The surface coatings at nano level was done by Electroless plating technique and these particles as reinforcements in a Mg alloy composite was successfully developed with Semi Solid stir casting technique. The density, hardness and tensile behavior was evaluated. Weight percentages are altered and the effects of NI coating on behavior of proposed composites are analyzed. The micro structural studies revealed the uniform distribution of the nano particles is observed at 2% reinforcement. There has been an improvement of 40.08% in Macro hardness, 35.40% in micro hardness, 60.80% of yield Strength, 35.80% of Tensile Strength and 2.56% of ductility observed. The prepared composites simultaneously enhanced both Strength and Ductility. Hardness, Yield and Tensile strengths of the composites were found to increase with increased nano Al_2O_3 up to 2% weight fraction and, then decreased with further addition of nano particles.

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