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The Effects of Electroless Ni-P Coated SiC on the Properties of Magnesium Composite

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Reference

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ABSTRACT

Reuse of magnesium alloy is an essential approach to protecting the natural resources, and it eliminates the waste products dumped in landfills, water, and air. Therefore, the first time the scrap magnesium alloy materials are collected and fabricated, the newly formulated magnesium composites use different techniques, such as vacuum, squeeze, and stir casting. AZ91 alloy from the automobile scrap is used as matrix material, and silicon carbide (SiC) is used as reinforcement. Electroless nickel-phosphorous coating is applied to the ceramic particles to avoid unwanted chemical reactions during the casting process. The adhesion between the matrix and reinforcement is improved by masking the surface of the nonmetallic SiC particles. Magnesium composite is most commonly used in automotive and aerospace applications to reduce weight and improve the strength of the component. The magnesium composite is fabricated through four different methods, and the substrates are tested and analyzed for better results. The sample results taken from the composites are compared to the magnesium alloy obtained from the scrap stock. Comparatively, the substrate produced using squeeze casting shows a lower porosity level of 5.5 %, and it is clearly shown in optical images. Significant improvements in the mechanical properties, such as hardness and compression strength, are obtained, and the wear rate in the prepared composites is reduced to 28 % for the sample produced using squeeze casting instead of the vacuum and stir casting processes.

Keywords

scrap magnesium, electroless nickel-phosphorous, porosity, compression strength, wear

Introduction

Two or more constituent materials with significant physical or chemical properties are combined macroscopically to produce a material called composite materials. The formulated material properties are unique and different from the incorporated individual elements. Engineering composite materials are classified into two types: One type is particulate-reinforced composite and another type is fiber-reinforced composite. Furthermore, it is classified based on its matrix material, such as metal matrix composite, ceramic matrix composite, and polymer matrix composite. Magnesium can be used as a matrix material rather than aluminum because the density of magnesium metal is 1.740 g/cm^3, which is 2/3 of aluminum and 1/4 of steel. It is one of the lightest materials available for commercial purposes in engineering metals and is amply available on the planet.[1,2] Particulate reinforced metal matrix composites (PRMMCs) have a great potential to be applied in automobile and aerospace industries because of their high strength-to-weight ratio, specific tensile strength, and wear resistance.[3–5] The need for magnesium-based, high-strength materials has increased rapidly in most automotive industries. It is incorporated in US carmakers such as BMW (inlet manifold), Jaguar (instrument panel cross-car beam), GM (front console), Jaguar (seat frame), and Ford (front-end assembly).[6,7] The applications of magnesium used in various industries are shown in **Table 1**.

A recent industrial review revealed that there are 60 different types of components, from instrument panels to engine components, in which magnesium is used or is being developed for use. The use of magnesium in automobile parts is predicted to increase globally at an average rate of 15 % per year.[8,9] Because of this rapid increase in the use of magnesium, a lot of magnesium scrap is also produced every year. The innovation in this work is to reuse scrap magnesium AZ91 available in old automobiles to produce new magnesium composite. Based on the strength obtained from this study, the potential applications are derived. The selection of reinforcement has a major role in composite material because the strength and wear resistance of the composite material is based on its reinforcement. Silicon carbide (SiC) has good wettability and chemical bonding properties with magnesium. Thus, it is a commonly used reinforcement in the magnesium matrix. Several investigations are reviewed and reported[10] that the addition of SiC produces more strength and wear resistance to the composite. Several methods to fabricate PRMMCs are given in **Table 2**, and the problems in those methods are briefed.

TABLE 1
Applications of magnesium in industrial components

Industry Application	Components
Automotive	Wheel rim, cylinder head, steering wheel frame, engine block, front consoles, and hoods
Aerospace	Thrust reversers for the Boeings, engines, helicopter transmission casings, and intercontinental ballistic missiles
Medical	Orthopedic biomaterial and plate for bone strengthening
Electronic	Cameras, cell phones, laptops, and portable media device housings
Sports	Golf clubs, tennis rackets, handles of archery bows, bicycle frames, and chassis of in-line skates
Others	Spectacles, riflescopes, binoculars, chain saws, hand shears, hand drills, pneumatic nail guns

TABLE 2
Casting defects based on the methods of production

Methods	Problems or Defects	References
Stir	Agglomeration of the reinforced particle, settling of reinforcing particle, and reaction between the materials	24
Vacuum	Impossible for complicated design components and unable to obtain consistent wall thickness	25
Powder metallurgy	Porosity increases with an increase in the void ratio in the composite. This leads to degradation in mechanical properties.	26
Pressure infiltration	if the applied pressure exceeds the strength of the reinforcement, it could get crushed and filled by the matrix alloy. This affects the mechanical property of the composite.	27
Squeeze casting	Accuracy of squeeze applying time is very important to get the uniform properties. Besides, it must be constant for each sample produced in the case of mass production. However, an experienced technician in production can avoid it.	28

In this research work, for the first time, the scrap magnesium material from car components is used as matrix material and electroless nickel-phosphorous (Ni-P)-coated SiC as reinforcement. To study the effect of the reuse of magnesium alloy, different manufacturing processes are selected and processed to get better properties. The produced composites for this study are represented as the following: (a) magnesium alloy, (b) vacuum casting, (c) squeeze casting, and (d) stir casting. The newly formulated composite materials are characterized and tested for the study of distribution, morphology, element identification, phase formation, the formation of blowholes, and resistance to compression and wear. From the critical literature survey, the control process parameters involved in the processes are studied keenly, and the parameters are selected by referring to the earlier researcher's work for the different casting processes involved in this work.

Experimental Procedure and Details

MATERIALS AND METHODS OF PREPARATION

The scrap AZ91 magnesium alloy (8.25 % aluminum, 0.63 % zinc, 0.22 % manganese, and balance magnesium, in wt. %) from the automobile waste was collected from the store and selected as a matrix material. SiC powder 90 µm in size was used as reinforcement material. The reinforcement size is verified using the scanning electron microscope (SEM) micrograph, as shown in **figure 1**. All the particles in the micrograph are measured using ImageJ processing software and verified with the average particle size of the reinforcement. The surface morphology of the substrates is done using the Carl Zeiss model EVO machine with energy dispersive X-ray analysis attachment.

The samples were prepared in the vacuum, squeeze, and stir casting processes to get the suitable method to reuse the magnesium alloy to produce composite material. The economical and commercially available methods are selected for the casting process. They are vacuum, squeeze, and stir casting processes. Before the casting process, the hard ceramic particles are coated using an electroless Ni-P coating process. The parameters required for electroless coating are taken from the previous paper published in our research group.[11,12] **Figure 2** shows the morphology and energy-dispersive X-ray spectroscopy (EDX) analysis of coated and uncoated SiC ceramic particles. The average particle size of the flakes is 80 µm, as shown in **figure 2*A*** and **2*B***. The coated substrate shows the nickel and phosphorous elements in the EDX analysis, as depicted in **figure 2*C*** and **2*D***. The parameters used in the coating process are a pH value of 8±0.2, duration of 15 min, and temperature of 50±2°C.[13]

FIG. 1

Particle size of the reinforcement purchased.

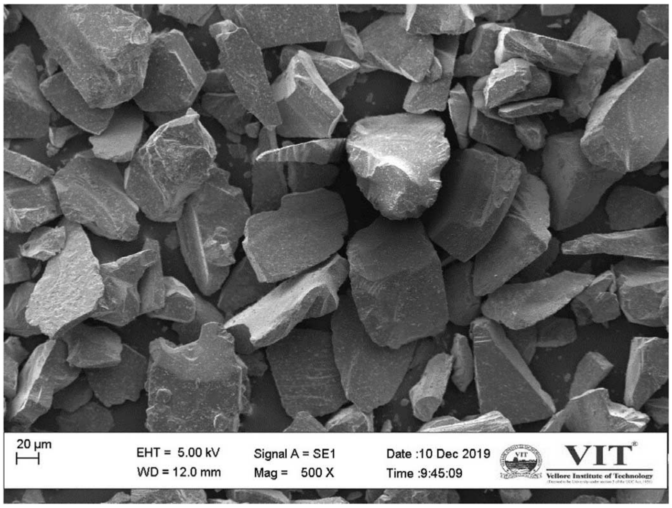
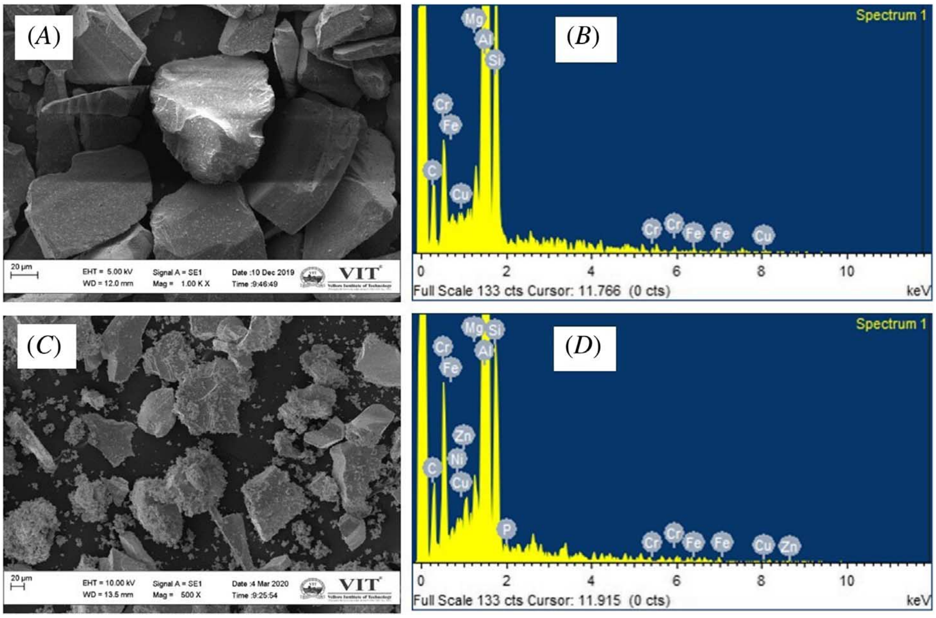


FIG. 2 Morphology of *(A)* uncoated SiC *(C)* coated SiC and EDX of *(B)* uncoated SiC *(D)* coated SiC.



The procedures and parameters involved in each casting process are given in the following sections.

Synthesis by the Vacuum Process

The AZ91 matrix containing 5 wt. % of SiC was produced by the vacuum casting process. The apparatus setup consists of three major components: an electrical heating system, an evacuation system, and a stirring system.[14]

The whole process is as follows:

1. Seven hundred thirty grams of scrap AZ91 are added to a steel crucible, which was preheated to a temperature of 800°C by an electric furnace. Argon gas with a pressure of 1 bar was supplied inside the furnace. After melting of the AZ91 alloy, the molten melt is stirred for 5 minutes at 525 RPM.
2. During stirring, 5 wt. % of preheated (300°C) coated SiC particles is added to molten AZ91 alloy.
3. After 5 minutes, stirring is stopped, and then the molten mixture is poured into the die by the bottom pouring method. The vacuum motor is maintained in the die with the aid of an electric motor. Then the molten mixture is allowed to solidify under vacuum conditions. The die and runway are also preheated to 250°C before the molten mixture is poured into the die.

Synthesis of the Stir Casting Process

In this process, the apparatus setup consists of two systems: the electrical heating system and a stirring system.

1. Seven hundred thirty grams of scrap AZ91 are added to a steel crucible, which was preheated to a temperature of 800°C by an electric furnace. Argon gas with a pressure of 1 bar was supplied inside the furnace. After melting of the AZ91 alloy, the molten melt is stirred for 5 minutes at 525 RPM.
2. During stirring, 5 wt. % of preheated (300°C) coated SiC particles is added to molten AZ91 alloy.
3. After 5 minutes, stirring is stopped, and then the molten mixture is poured into the die by the bottom pouring method.

Synthesis of the Squeeze Casting Process

In this method, the apparatus setup consists of three systems. They are electrical heating systems, hydraulic systems, and stirring systems.

1. Seven hundred thirty grams of scrap AZ91 are added to a steel crucible, which was preheated to a temperature of 800°C by an electric furnace. Argon gas with a pressure of 1 bar was supplied inside the furnace. After melting of the AZ91 alloy, the molten melt is stirred for 5 minutes at 525 RPM.
2. During stirring, 5 wt. % of preheated (300°C) coated SiC particles is added to molten AZ91 alloy.
3. After 5 minutes, stirring is stopped, and then the molten mixture is poured into the die by the bottom pouring method.
4. The molten mixture is solidified under squeeze pressure of 100 MPa, which is applied for 45 seconds.

TESTING OF PROPERTIES

Density and Porosity Measurement

The theoretical density of the sample is calculated using the rule of mixtures. The experimental density is calculated using Archimedes' principle. So, the experimental density was calculated using equation (1).

$$\rho_{\text{Experimental}} = \frac{w_a}{w_a - w_w} * \rho_w \tag{1}$$

where W_a is the mass of cylindrical sample in air, W_w is the mass in distilled water, and ρ_w is the density of distilled water. The porosity in the samples was determined by using equation (2).

$$P = 1 - \frac{\rho_{\text{Experimental}}}{\rho_{\text{Theoretical}}} * 100 \tag{2}$$

Wear Test

The sliding abrasive wear rate for magnesium composites fabricated by different production processes and magnesium alloy was tested under a 10-N load using a pin-on-disk apparatus. The samples are prepared as a pin with a 10-mm diameter and 30-mm height, and ASTM G99-17, *Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus*, was used. High-carbon, high-chromium steel is used as a counter-face disk with a hardness value of 62 HRC. After each test, the pins were cleaned with acetone and weighed using an electric balance to determine weight loss in an air-conditioned room maintained at 24°C. The specific wear rate of the samples is calculated using the formula as given in equation (3).

$$w = \frac{\Delta V}{L \times d} \tag{3}$$

where w is the specific wear rate, ΔV is the volume loss in mm^3, L is load in Newton, and d is the sliding distance in m.

Compression Test

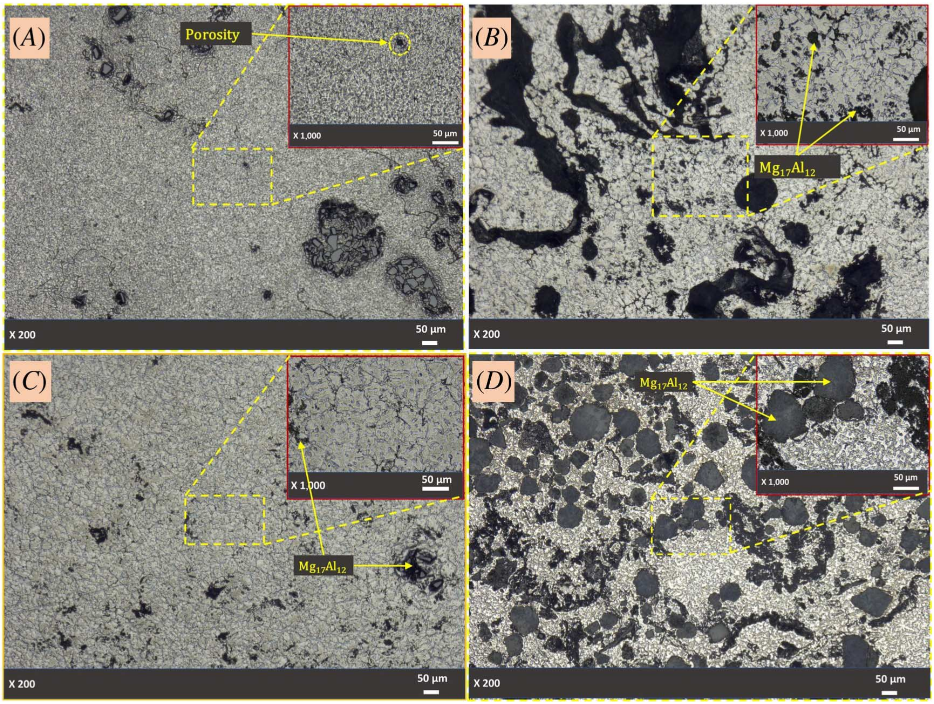
A compression test was carried out as per the procedures of ASTM E9, *Standard Test Methods of Compression Testing of Metallic Materials at Room Temperature*, using the computerized universal testing machine (UTM). The uniaxial compression test was performed on a cylinder sample of 13-mm diameter and 30-mm height with a crosshead speed of 1 mm/min using a 10-kN capacity UTM interfaced with a computer (model: 8801, Instron). The data from the compression test are obtained to prepare the stress-strain curve, and the maximum compression strength of the samples is determined.

Results and Discussion

CHARACTERIZATION OF PRODUCED COMPOSITES USING OPTICAL, SEM, EDX, AND X-RAY DIFFRACTION

The optical microscopic images for the four different composites at 200× magnification are shown in **figure 3**. In the pictures, dark regions are a mixture of both the reinforcement and the eutectic phase of silicon, and white

FIG. 3 Optical microscopic image of magnesium composites: (*A*) magnesium alloy, (*B*) vacuum casting, (*C*) squeeze casting, and (*D*) stir casting.



regions are the magnesium matrix. Figure 3*C* exhibited an almost non-dendrite shape at the grain boundaries because of the squeeze pressure, which resulted in finer dendrites and decreased dendrite arm spacing. Figure 3*B* and 3*D* shows that the microstructure has nearly uniform dispersion of the equilibrium intermetallic phase[15] in AZ91-SiC samples.

The SEM analysis for the squeeze cast sample is shown in the figure 4 at a magnification condition of X250. The distribution of the reinforcement material is seen clearly in the micrograph. Furthermore, the gray area is related to the α phase, while the dark area is the β phase ($Mg_{17}Al_{12}$) distribution in AZ91-SiC samples. It is noticed that the reinforcement particles are located near the inter-dendritic zone, as shown in the magnified image.

The EDS analysis done for the sample produced by a squeeze casting process is shown in figure 5. The elements are identified and represented in different colors to show the distribution nature. The uniform distribution of reinforcement particles should be the possible reason for the elevated property. It can observe that the peak of magnesium is reached above the 12 cps/eV of amplitude. EDX mapping identifies the presence of magnesium, aluminum, silicon, and oxygen phases, and it also can show the different elemental phases that present in the matrix. The patterns provide a clear vision of the effects of the reinforcement material on the matrix, which has the potential to develop and enhance the mechanical properties of the composites. The evidence of porosity level is shown in EDX analysis.

X-ray diffraction (XRD) analysis was performed on the AZ91 + SiC sample fabricated by squeeze casting process by exposing CuKα radiation (λ = 1.5406 nm) on the sample with a scan speed of 2 deg./min on a diffractometer. XRD analysis is used to identify any new phases that might occur in the production process. Figure 6 shows the X-ray diffraction pattern of the squeeze cast composite. Bragg's angle and interplanar distance of magnesium and SiC were obtained and compared with standard values to verify the presence of magnesium and SiC in the sample. In diffractograms (fig. 6), the characteristic peak intensity value of the magnesium matrix is

FIG. 4 SEM image of squeeze cast magnesium composite.

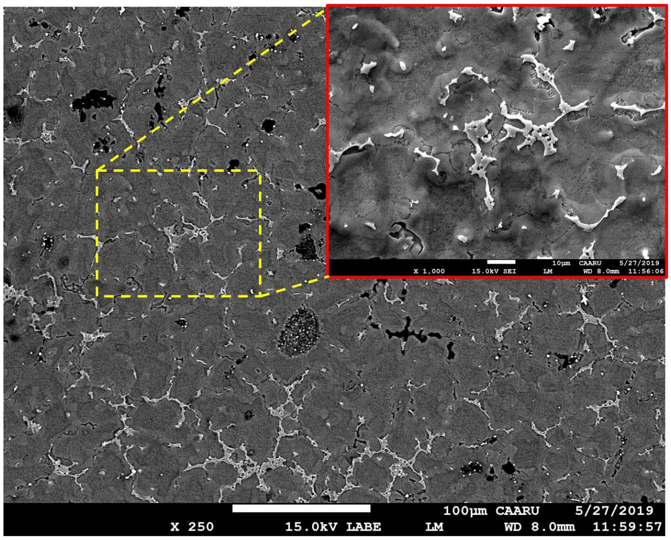
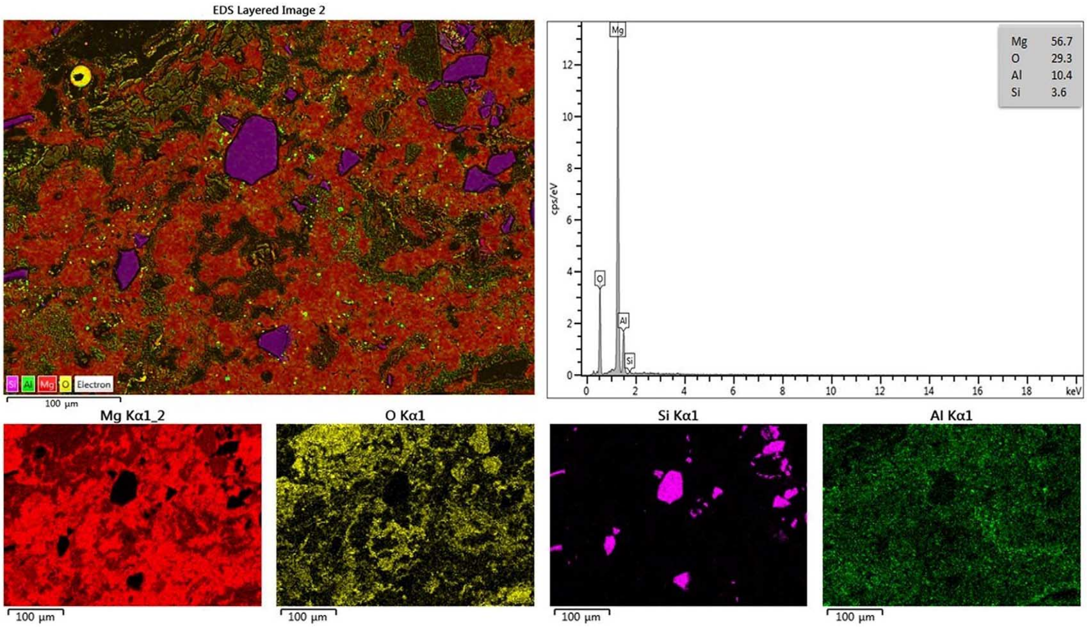


FIG. 5 The elements' EDX analysis of the layered magnesium composites.



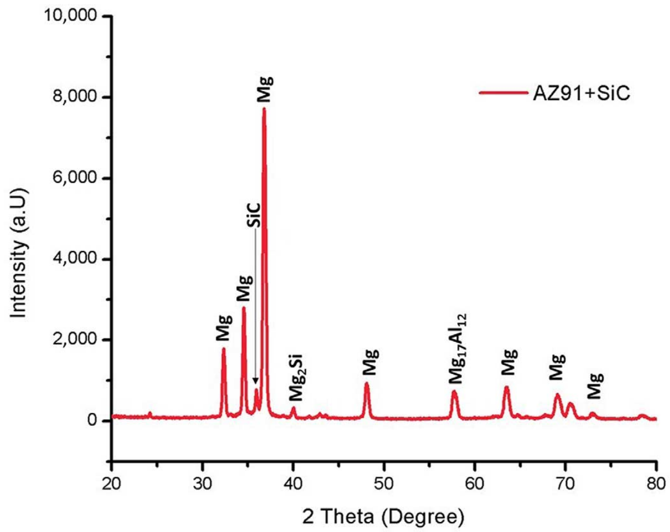
observed at an angle of 38.71°and 37.3° for SiC.[16] It also shows the presence of intermetallic phases Mg_2Si and $Mg_{17}Al_{12}$ in the sample; this may be because of the reaction of magnesium with SiC and aluminum.[17]

$$2\text{Mg} + \text{Si} \rightarrow \text{Mg2Si}\,(\text{Intermetallic Phase}) \tag{4}$$

$$\text{Mg17Al12}\,(\gamma\ \text{intermetallic phase}) \tag{5}$$

FIG. 6

XRD analysis of magnesium composites.

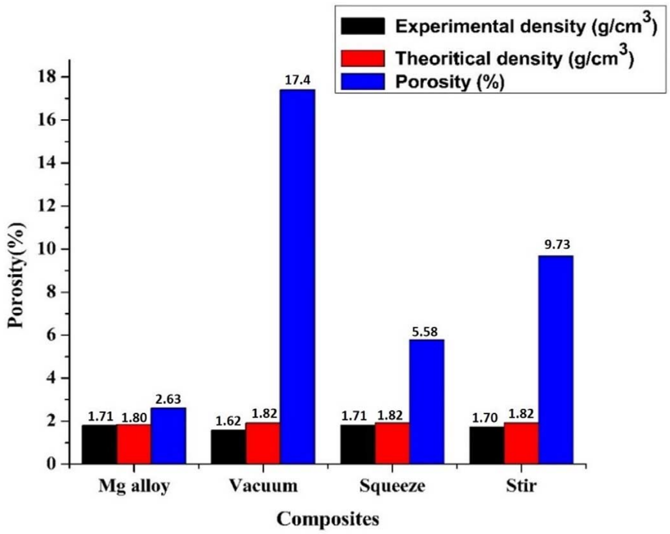


DENSITY AND POROSITY ANALYSIS

Figure 7 shows the variation of density and porosity of the samples. It is depicted that The magnesium alloy without any reinforcement has a much lower porosity than 5 % of the SiC reinforced AZ91 magnesium alloy. The porosity of vacuum casting and stir casting is 300 % and 167 % higher than the porosity of squeeze casting, respectively. In composites, the reinforcement materials are treated as foreign elements, but in the alloy, no such elements are introduced, and hence, a very low porosity is obtained. Among composites, squeeze casting has very little porosity as mentioned earlier. This is achieved by applying squeeze pressure on the sample during fabrication. The cooling rate is increased by applying pressure to the sample, and the gap between the dendrites is forced to move together.[18] Thus, the porosity of the sample is reduced in the sample produced by the squeeze

FIG. 7

Porosity percentage of magnesium composites.



casting process. As a result, the reuse of magnesium alloy from the scrap does not affect the porosity in composites; it is at a controlled level of around 5.5 %.

COMPRESSION ANALYSIS

Compression properties of scrap magnesium alloy and composites fabricated from different production processes are shown in figure 8. It is observed that magnesium alloy (AZ91) reinforced with SiC and fabricated by squeeze casting process shows a maximum compression strength of 318 MPa and a low compression strength for the composite fabricated by vacuum and stir casting. The compression strength can be accredited predominantly to the following factors: grain refinement, evenly distributed hard particles called reinforcement, compatibility between the matrix and reinforcement material, and the Orowan strengthening mechanism.[19] The addition of SiC particles to Scrap magnesium alloy improves the compression stress of the composite, as shown in figure 9. But this trend occurs only when fabrication is done by the squeeze casting process because the porosity of the

FIG. 8

Compression strength of fabricated magnesium composite.

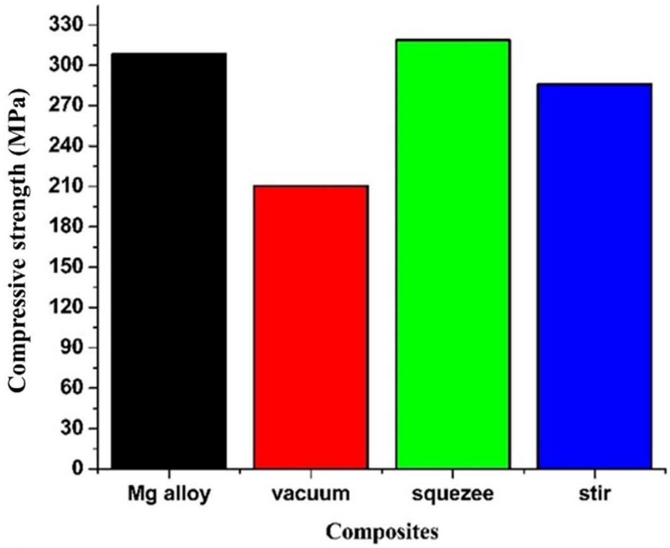
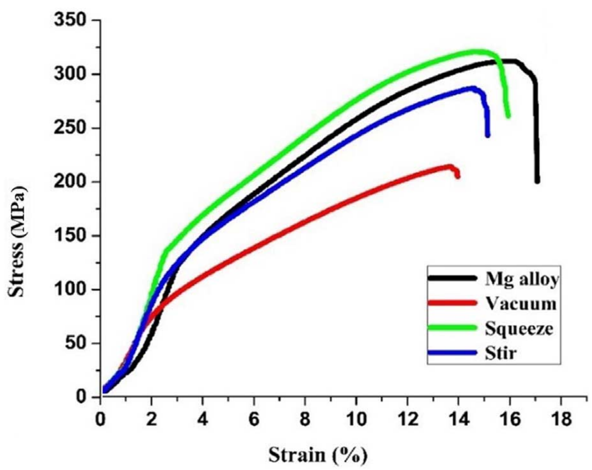


FIG. 9

Stress-strain graph of fabricated magnesium composites.



composite produced is lower compared to the composites produced using vacuum and stir casting. From the literature, it is witnessed that the SiC is the most compatible material for magnesium alloy to add as reinforcement. As a consequence, the compressive strength of the magnesium composite is elevated.[20]

SPECIFIC WEAR RATE

Figure 10 shows the specific wear rate of magnesium alloy and SiC-reinforced magnesium composite fabricated by different production processes. The specific wear rate of scrap magnesium alloy is improved with the addition of SiC. It is concluded that the specific wear rates of SiC-reinforced magnesium composite produced by vacuum,

FIG. 10 Specific wear rate of fabricated magnesium composites.

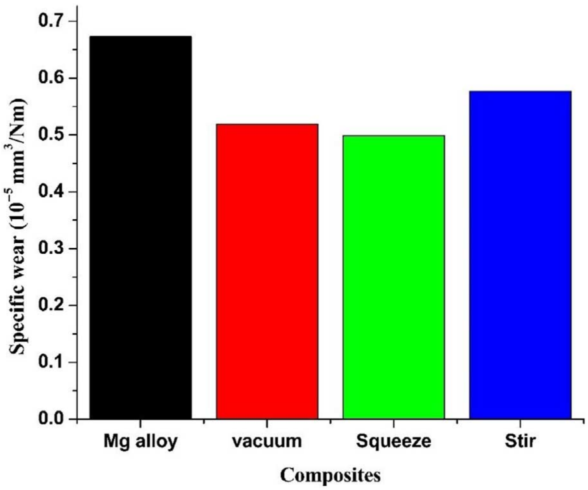
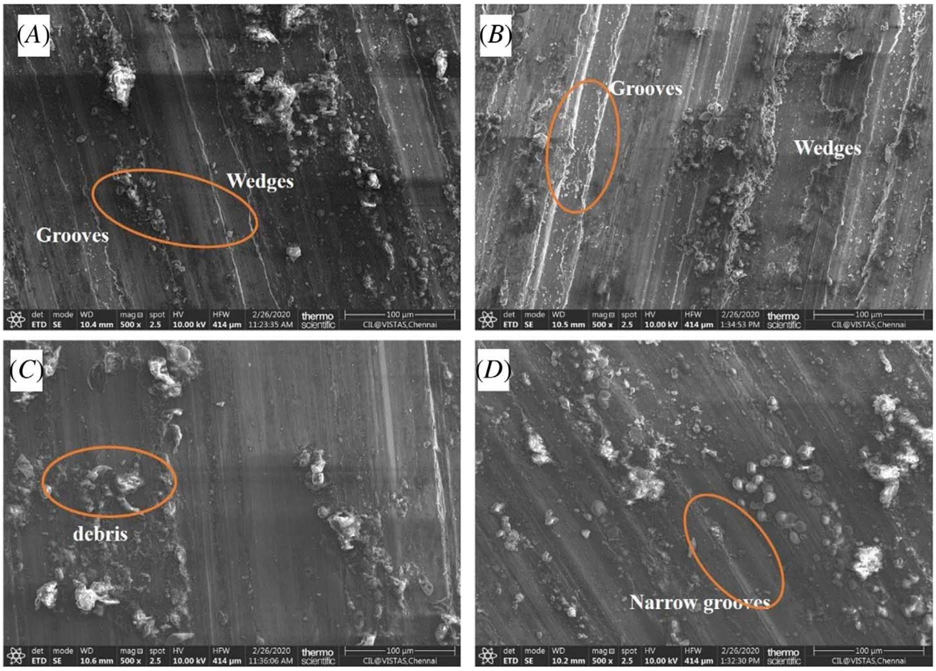


FIG. 11 SEM micrograph of the worn-out surface *(A)* Mg alloy *(B)* Vacuum *(C)* Squeeze *(D)* Stir.



squeeze, and stir casting are 22 %, 25 %, and 15 % lower, respectively, than unreinforced scrap magnesium alloy. Hence, the incorporation of the hard particle in the matrix resulted in improved compression strength of the composite. The composite fabricated by the squeeze casting process has a lower wear rate compared to other techniques. The SiC particles in the matrix of the composite withstand the load, so the contact area between the substrate and the counterpart surface is reduced, the friction between the pin and disk is reduced, and material removal from the pin is reduced. Undoubtedly, this must be the main reason for the improvement in wear resistance.[21]

The study of the worn-out surface was done using a field emission SEM (FESEM) test to study the nature of wear that took place during the wear study. Substrates produced through other casting methods, which are shown in **figure 11*A*** and **11*B***, show the highly deformed area and Peak to valleys in the worn-out surface. Only debris is visible, and the line of demarcation is observed for the substrate produced using the squeeze casting process, which means the wear rate is very low in this case, as shown in **figure 11*C***. Only narrow grooves are visible for the substrate produced by the stir casting process; hence, less amount of wear takes place, as shown in **figure 11*D***.[22,23] Hence, as seen from the FESEM micrograph, the lowest wear rate is achieved in the squeeze cast substrate.

Conclusions

In the research work, scrap magnesium alloy is productively reused to yield a new electroless, Ni-P reinforcement-coated magnesium composite, and the best processing method was found among vacuum, squeeze, and stir castings.

- Electroless Ni-P coating on the reinforcement shows an even distribution of ceramic particles, and less porosity is achieved in the composites, which are cast by the squeeze casting process.
- As evident, the optical and SEM morphological observations are provided.
- The samples fabricated using the squeeze casting process produced higher compressive strength and hardness than the samples produced by other processes, such as vacuum and stir casting.
- The porosity percentage is very low for squeeze cast substrate compared to other composites.
- The wetting between the matrix and reinforcement is improved after Ni-P coating and the masking of the ceramic surface.
- The wear rate is comparatively low in the case of squeeze cast substrate and it is witnessed in the FESEM micrograph.

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