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Synthesis and characterization of hollow glass microspheres reinforced magnesium alloy matrix syntactic foam

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ABSTRACT

In the present scenario, a considerable importance is being given to find newer materials for marine applications. An endeavour has been made to synthesis hollow glass microspheres reinforced magnesium matrix (AZ91D/HGM) syntactic foams and analyze its mechanical properties for such applications in the present study. The 40 μm size reinforcement particles have been added with matrix under dissimilar mass fractions of 15%, 20% and 23% to investigate the interfacial reaction, compressive properties, hardness, density, porosity, corrosion resistance. From the experimental investigation, it has been inferred that the density of foams and corrosion rate have been considerably reduced owing to increase in reinforcement mass fraction. It has also been observed that the developed syntactic foam has produced higher compressive strength due to its cushion effect and higher plateau stress. From the porosity phase analysis, it has been found that the porosity has been increased with increasing mass fraction percentage. From the composition structural analysis, $Mg_{17}Al_{12}$ has been observed as main interfacial reaction between the microsphere and matrix alloy.

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1. Introduction

Owing to its low density, the research attention on metal foams has been increasing for the past several decades. In metal foams, the base metal has gas porosity within the structure to reduce the structural weight. Metal foams are the materials which displays a distinctive combination of physical and mechanical properties due to their light weight, high specific stiffness, strength to weight ratio and energy absorbing capabilities. The performance of existing foams has not been promising due to strong variation in the cell structure. Most commercially available cellular metals do not achieve the mechanical properties which have been predicted from the scaling relations. This can be partially attributed to morphological defects in the structure such as missing cell walls and wiggles in the cell wall. These defects must be eliminated to enhance the properties of the synthesis foams with desired amount of orientation and porosity size. Despite of this enhancement, one critical limitation of the foam is their low strength and modulus. Porosity can be incorporated in the foam structure as form of hollow particles. The presence of hollow particles instead of gas porosity can provide a closed cell structure to these foams called syntactic foams. These foams are a class of particulate composites that have been fabricated by distribute hollow microspheres in matrix material [1,2].

The conventional foaming operation tends to create foams having more random cellular arrangement in the matrix material. Since the syntactic foams have not been produced by conventional foaming operations, the composition and densities of syntactic foams are predictable. Syntactic foams have better mechanical and thermal properties as those of conventional polymeric foams [3]. Various research works have been found to investigate the effect of fly ash cenospheres on mechanical properties of AZ91D/ZC63 magnesium alloy. It has been reported that weight percentage of reinforcement has indirectly proportional nature to density of syntactic foams [4,5].

Light weight materials with high specific strength are essential in marine applications. Mostly buoyancy materials for such applications are developed by introducing a light weight material in the matrix. The metal matrix syntactic foams can be made of

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aluminium, steel, titanium and magnesium alloys. However most of the researches have been focussed on aluminium matrix alloy only. Magnesium is the lowest density of any structural material with high specific strength [6]. It can also be proved that magnesium metal foams have the potential to serve as structural material for regular light weight applications compared with aluminium syntactic foams [7]. AZ91D is most widely used magnesium alloy due to its excellent corrosion resistance, castability and mechanical properties [8].

From the literature survey, it has been observed that only very little attention has been given to studies on magnesium matrix syntactic foams in comparison with aluminium matrix syntactic foams. The major difficulty on synthesising magnesium matrix syntactic foams is high chemical reactive ability of magnesium. It is also tedious to synthesis magnesium based syntactic foam owing to large difference in the density of matrix and reinforcement. Hence an endeavour has been made to introduce new series of magnesium alloy based light weight syntactic foam reinforced with hollow glass microspheres (HGM) for enhancing the mechanical property in the present study.

2. Experimental methodology

Since the magnesium is light density material, AZ91D magnesium alloy has been considered as matrix material in the present study. The chemical composition of AZ91D magnesium alloy is shown in Table 1. The reinforcement used for the syntactic foam is hollow glass microspheres (HGM) of SiO_2, B_2O_3, CaO, MgO and Na^+ salts with particle size and density of 45 μm and 0.37 g/cc respectively which is commercially available from 3 M technologies [9]. The density of reinforcement is lower than that water which is highly essential property for subsea applications. Nevertheless it has high compressive strength, high energy absorption and low water absorption. It has been found the reinforcement has been effectively mixed with matrix material at the maximum level of 23% [4,5]. Hence, the reinforcement has been added with matrix alloy under various mass fractions such as 15%, 20% and 23% in the present study.

Stir casting technique has been used for synthesising magnesium matrix syntactic foams due to its low processing cost and high production rate [10]. While preparing magnesium matrix syntactic foam by stir casting method, the various factors have to considered such as difficulty on achieving uniform distribution of the reinforcement, wettability, porosity and chemical reaction of reinforcement with matrix alloy [11]. Daoud et al. noted that the pouring temperature has a significant effect on particle distribution on cenospheres in ZC63 alloy. It has found that the optimum temperature for casting should be approximately 685 °C [12]. The AZ91D magnesium alloy in crucible has been melted at 685 °C and stirred using high shear impeller. The mixture of SF_6 gas with CO_2 is commonly used as cover gas to protect the molten magnesium from burning and excess oxidation with flow rate between 20 and 40 L per minute [13]. The desired mass fraction of HGM particles have been preheated at 450 °C and incorporated manually into the melted magnesium matrix. The optimal diameter of the stirrer is the size at which the solid particles are fluidized in both the central and marginal part at the same speed. It has been found that the stirrer diameter should be equal to 0.4D and the blade width should equal to 0.1–0.2D; where D is the diameter of the vessel [14].

Table 1
Chemical composition of AZ91D magnesium alloy.

Elements	Al	Mn	Zn	Si	Cu	Ni	Mg
% composition	8.3	0.13	0.35	0.5	0.5	0.03	Remaining

Since the random distribution of reinforcement particles in the matrix alloy may cause the different hardness values at dissimilar positions of the specimens, the stirring speed and stirring time has to be chosen in optimal levels. The values of stirring speed and time has to be chosen in order to achieve an enhanced homogeneous distribution of reinforcement in the magnesium matrix syntactic foams by avoiding clustering of reinforcement particles. In the present study, the stirring speeds have been taken as 400,500 and 600 rpm and the stirring time consider as 5, 10 and 15 min.

The distribution of HGM particles in magnesium matrix alloy has been investigated using scanning electron microscope (SEM) analysis. A stainless steel die has been designed and fabricated for pouring AZ91D/HGM based foams with the volume of 141.37 cc. The die has been coated with sulphur powder to prevent oxidation and placed near the spruce to minimize the interaction between the molten foam and the atmosphere during gravity pouring. The molten syntactic foam has been allowed to cool inside the die at ambient conditions.

Rockwell F-scale hardness testing machining has been utilized to compute the hardness of AZ91D alloy and magnesium matrix syntactic foams under as-cast conditions. The values have been measured in the 15T scale with a steel ball indenter of 1/16-inch diameter under 15 kg applied load. The average of five measurements near the center of 1.8 × 1.8 × 1.8 cm cube has been considered as the hardness of the specimen [15]. The compressive testing on syntactic foam behavior has been performed under room temperature using specimens with height of 20 mm and diameter of 10 mm. The specimens have been compressed using computer controlled universal testing machine (UTM) as per ASTM E9 standard at the cross head speed of 0.5 mm/min. The density of the syntactic foam has been determined using Archimedes method with ethanol as suspending medium. In the present study, the optical microscope has been utilized to investigate the microstructure features and the distribution of HGM in magnesium matrix alloy. The compositional spot analysis has been carried out in and around the HGM using energy dispersive X-ray analysis (EDX) available in scanning electron microscope (SEM). The X-ray diffraction analysis (XRD) has been performed to analyze the phases in matrix alloy and reinforcement. In the present study, the percentage of porosity has been analyzed as per ASTME562 & E1245. The corrosion test has been performed as per ASTM B117-11 salt spray test. This test has provided a controlled corrosive environment which has been developed to produce relative corrosion resistance for the specimen exposed in a given test chamber. The specimens of various mass percentages have been polished to 1200 grade silicon carbide over one face. The specimens have been weighed and the unpolished areas have been marked. The specimens have been tested in salt spray chamber with exposed surface of each specimen inclined at 25° to the vertical. Cyclic spray condition has been used with on and off periods of 1.5 h each. The test temperature has been maintained at 25° and fresh 3.5 wt% salt spray has been used during wetting cycle. The process has been repeated for 48 h [16].

3. Results and discussion

In the present study, an endeavour has been made to synthesis magnesium matrix (AZ91D/HGM) with various mass fractions of HGM as reinforcement. The experimental investigation on analyzing the mechanical properties such as compressive properties, hardness, density, porosity, micro structure, interfacial reaction and corrosion resistance of syntactic foam are discussed in the present section.

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