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Forging of magnesium oxide foam by powder metallurgy method

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ABSTRACT
Metallic foams have drawn significant attraction nowadays for their excellent physical, mechanical, thermal, electrical and acoustic properties with a lightweight structure. Magnesium oxide foams are widely used in automotive industries, biomedical applications, aerospace and other industries due to the properties of low density, higher strength to weight ratio, wear resistant, ductility, and adsorption. In this thesis report fabrication of magnesium oxide foam by space holder technique via powder metallurgy route is discussed. Here, ammonium hydrogen carbonate has been used as a space holder. After mixing the powders by different ratios by ball milling, uniaxial compaction of the mixture has been done in die punch under a pressure of 30 kN. The sintering is done at 1475°C for 6 hours. Through obtaining data after sintering the density and porosity have been measured and is seen to vary from 1.56 g/cc to 1.16 g/cc in density and 8.77% to 25.64% in porosity with the increase of space holder. The compressive stress is seen to vary from 197.6 MPa to 94.3 MPa with the decrease of the space holder.

Keywords— "magnesium oxide foam", "ball milling", "sintering", "density", "porosity"

1. Introduction

Metallic foams provide some interesting properties because of the combination of metallic structure with pores. It is now being used widely for various important sectors in the modern world of technology for its unique combination of properties like low density with high strength, increased ductility with damping abilities, etc. Scientists have found several ways of producing metal foam over time. The powder metallurgy method is a convenient and good process of fabricating the metal foam using space holders.

Songnan et al [2] synthesized porous magnesium oxide by a combustion method where he used $Mg(NO_3)_2$, ethylene glycol along deionized water as reactants. Here, he used magnesium nitrate by 1.28 g and added this to a solution of 4 mL which contained ethylene glycol and deionized water at a ratio of 1:1 by volume. The mixture then was stirred for about 30 minutes and then taken to the crucible. It was then calcined at 600°C for two hours in a muffle furnace. The characterization and adsorption test was done by X-ray diffraction, scanning electron microscope, nitrogen adsorption, infrared spectroscopy. It was seen from the results that the large BET surface area of as-prepared porous magnesium oxide was 203.8 m^2g^{-1} with a multiscale porous size. On the other hand, magnesium oxide when synthesized with other reactants had a BET surface area of 17.6 m^2g^{-1} with rod-shaped morphologies and 3.4 m^2g^{-1} with granular morphologies. Porous magnesium oxide shows better adsorption efficiency when compared to magnesium oxide with a low specific surface area. According to the results, the maximum adsorption capacity was approximately 1088 mg g^{-1} in removing Congo red from wastewater.

Morozov et al [1] fabricated porous magnesium oxide by thermal decomposition of the basic magnesium carbonate. He calcined the basic magnesium carbonate at 20-800°C and there observed an induction period in the initial stage of the decomposition process. The specific surface area was seen to vary in proportion with the decomposition degree when the decomposition degree was higher than 20% of the weight loss. The individual phase in the process of the calcination of magnesium oxide was found to be at a minimal temperature of 500°C. Morozov also stated that the MgO phase was seen to be structured further on increasing the calcination temperature and time resulting in the decrease of the specific surface area and basicity.

Yilong et al [3] fabricated magnesium foam using carbamide as a space holder through the powder metallurgy used 99.5% pure magnesium powder with 50~70μm particle size and 99.5% pure carbamide particle with a particle size of 1.1~1.2 mm. In the study he showed when porosity increases from 38.9% to 57.5%, elastic modulus decreases from 8.50 Gpa to 3.30 Gpa and yield stress decreases from 24.9 Mpa to 9.40 Mpa. Therefore he concluded that magnesium foam has good shock resistance and absorption property. Here the powders were mixed in different ratios and then uniaxially compressed by a hydraulic press with a pressure of 200 MPa for 1 minute. Then the green compaction samples were sintering in a furnace with argon atmosphere in two steps, firstly at 400°C for 1 hour and then at 630°C for 2 hours.

Sharma et al [12] fabricated magnesium alloy specimen through powder metallurgy route using pure magnesium powder (99.9% pure) with a particle size of 120μm. He used zinc and aluminium powder along with this Mg powder to form Mg alloy foam to attain better corrosion resistant properties from magnesium for biomedical uses. Here in this study, several factors like compaction pressure, sintering temperature and sintering time were observed on how they impact the porosity of the foam. At first magnesium powder along with zinc and aluminium powders were mixed in the high energy ball mill for different time durations. The size of the particle decreases and a better homogeneous

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mixture is obtained with the increase in the ball milling time duration. Later the mixture was compacted in a compaction press of 1.5 ton. After compaction, the samples were sintered in a muffle furnace with an argon atmosphere with a variety of temperatures of 450°C and 500°C along with the time duration varying from 30 to 60 minutes. Yang et al [4] fabricated Mg-Al alloy foam with closed cell structure by powder metallurgy route and investigated the mechanical properties and advantages it offers for alloying with aluminium. Pure magnesium and aluminium powders were mixed with $CaCO_3$ where $CaCO_3$ was used as the blowing agent. He first mixed the two powders with the blowing agent by blending in the mixture for 72 hours. The mixture was then cold-pressed and formed into tablets of 25 mm diameter and 6-7 mm thickness. The dense semi-finished objects were subjected to sintering. Those were wrapped up in Al paper and immersed in the sand under a temperature of 415°C for 3 hours. After that, the foam with the interconnected structure was subjected to hot pressing and was given uniaxial compression at 350°C for yielding foamable precursor. This precursor was then inserted into a cylindrical steel mould that was preheated to a temperature of 590-650°C. The precursor then was in progress to be foam when it was heated for a certain time around 90-200 seconds.

Zou and Li [5] fabricated the magnesium foam using camphene as the space holder and followed the powder metallurgy method. Magnesium powder of high purity was mixed in a beaker with high purity camphene at a temperature of 60°C for 5 minutes. The mixture of camphene and magnesium were then laid open to solidify at a temperature of 20-25°C. This solidified mixture was then compressed in a pellet mould under a pressure of 500 MPa for 5 minutes. The compacted samples were then put under medium vacuum for 7 days to sublimate properly. Long time ensured the complete sublimation of camphene and hence left a porous structure of the magnesium. This structure was then sintered by two steps and was sintered at 250°C for 2 hours to ensure any camphene left is then sublimated. After that, the samples were again sintered at 630°C for 6 hours which turned the compacted magnesium into porous magnesium foam. Foams of different porosities were produced and were tested. He stated that with the increase of porosity the compressive strength and yield strength were seen to decrease.

2. Methodology

2.1 Methodology of the study

1. In this study, magnesium oxide foam has been produced by the powder metallurgy method. The process is discussed below:
2. In this method, at first pure magnesium oxide powder and ammonium bicarbonate as space holder material has been mixed thoroughly by ball milling. High energy ball milling is necessary for having a homogeneous mixture.
3. Mixture of magnesium oxide powder and ammonium bicarbonate is subjected to compaction by using a die punch assembly. Maximum 30 kN pressure is required to obtain sufficient green strength of the mixture. The die is a (50 mm×50 mm) square die with a hole of 15 mm through it. The plunger size is in the cylindrical shape of 70 mm length and 15 mm dia. Then the magnesium oxide powder and ammonium bicarbonate mixture have been poured gradually into the die hole. At the bottom of the die hole, there would have been a coin of 5 mm thickness and 15 mm dia. There would be a coin of the same dimension present in the upper surface of the mixture. The purpose of using coin at the top and bottom surface is to give a uniform contact area. Then the plunger has been pushed through the die hole. By this uniaxial compression, a dense product of magnesium oxide powder with a space holder in it has been obtained.
4. Then the dense product has been sintered at 1475°C in a Furnace. At this temperature ammonium bicarbonate in the dense product has been decomposed and would leave pores in the product. As the amount of pores increases with the amount of space holder, so samples of different porosities has been made. The samples are cylindrical.
5. After making magnesium oxide foam by powder metallurgy method compressive stress test has been done on the sample. The test has been performed in the universal testing machine (UTM).

2.2 Materials and equipments

1. The materials and equipment required for this process are listed below:
2. Magnesium oxide powder
3. Space holder (ammonium bicarbonate)
4. Ball milling machine
5. Die-punch
6. Hydraulic press
7. Furnace
8. Universal testing machine (UTM)

2.2.1 Magnesium Oxide Powder

500 gm of magnesium oxide powder has been brought so that 6 foam samples can be formed for safety.

Fig. 1: Magnesium oxide Powder

2.2.2 Space Holder

For this magnesium oxide foam, fabrication ammonium bicarbonate has been used as a space holder. 500 gm of space holder ammonium bicarbonate is needed because different ratios of the mixture of magnesium oxide powder and ammonium bicarbonate would be used in this experiment.

Fig. 2: Ammonium bicarbonate .

2.2.3 Ball Milling Machine

A ball milling machine has been managed to mix the space holder and magnesium oxide powder thoroughly.

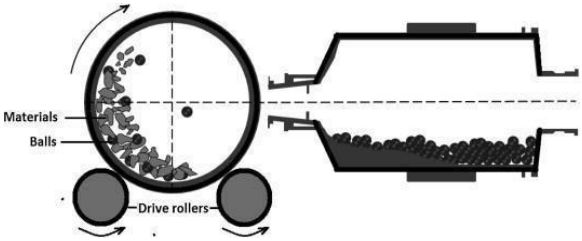

Fig. 3: Schematic Diagram of a Ball Milling Machine

2.2.4 Die-Punch

Stainless steel was used as the material of the die. A 50 mm×50 mm square block of stainless steel was taken, and then a 15 mm hole was drilled in the center of the block. The inner surface of the drilled hole has been made smooth by polishing. A plunger was also made of stainless steel of diameter 15 mm and a length of 70 mm. The plunger would be cylindrical.

Fig. 4: Die punch

2.2.5 Hydraulic Press

A hydraulic press machine has been required to compress the mixture of the powders in a die punch assembly for forming a compact solid cylindrical shape of the powders and for giving the powders green strength.

Fig. 5: Hydraulic press machine

2.2.6 Furnace

An inert furnace has been needed to sinter the dense object so that the space holder may decompose and porous magnesium oxide foam may remain. A temperature of 1450°C is needed. Figure 3.6 shows the diagram of a furnace.

2.2.7 Universal Testing Machine

A universal testing machine has been used to measure the compressive strength of the magnesium oxide foam.

Fig. 7: Universal Testing Machine.

3. Production of Magnesium Oxide Foam

Fabrication of magnesium oxide foam can be done in several methods as suggested by material scientists and researchers. As mentioned earlier, in this thesis project the method of foam fabrication via powder metallurgy route is followed. In this chapter, the steps of foam fabrication will be discussed elaborately with each dimension and parameters required for the fabrication and analysis.

3.1 Mixing of magnesium oxide powder and space holder :

The two powders were measured and mixed in different ratios for observing and analyzing the difference in porosities and properties for the difference in composition. 75 wt% of magnesium oxide powder was weighed on an electronic balance followed by 25 wt % of ammonium bicarbonate and then they were mixed. Similarly, mixtures of 65 wt% of magnesium oxide and 35 wt %of ammonium bicarbonate, 55 wt% of magnesium oxide and 45 wt% of ammonium bicarbonate were measured and mixed.

3.2 Ball milling of the powders :

Ball milling was done for obtaining a homogeneous mixture of the magnesium oxide powder and the ammonium bicarbonate.

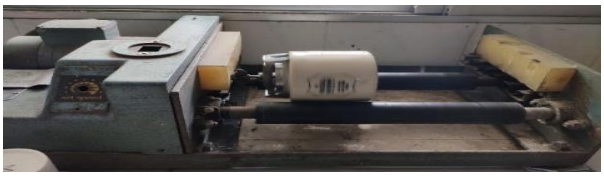

Fig. 8: Ball milling of magnesium oxide powder and space holder

3.3 Compaction of powder mixture :

Compaction was done to consolidate the powder mixture of magnesium oxide and space holder. Now for the compaction, 6 gram of mixed powder with a weight ratio of 75:25 was taken. A stainless steel coin of 15 mm diameter was placed at the lower side of the die cavity to prevent the powders leaving from the cavity. The powder mixture was poured into the cavity and the upper part of the cavity was then sealed by another coin similar to the previous one. After that, the die was placed in between the jaws of the hydraulic press and then the pressure of about 30 kN was applied gradually. The compaction was done at 30 kN of pressure for 5 minutes. As result, a dense solid product of cylindrical shape was obtained from the die cavity. So, compaction of the mixture of magnesium oxide powder and ammonium bicarbonate of weight ratio 75:25 was

completed. Similarly, the dense solid structures of weight ratios 65:35 and 55:45 were prepared following the exact procedures.

Fig. 9: Compaction of powder mixture in a hydraulic press

3.4 Sintering :

Sintering is the process in which the solid mass of material is compacted and formed using pressure or heat. During sintering the ammonium bicarbonate was decomposed leaving a porous structure of the magnesium oxide behind that is magnesium oxide foam. The sintering was done at an electric furnace at 1475°C for 6 hours.

Fig 10: Sintering in a furnace

Figure 11,12 and 13 show the finished magnesium oxide foams of different weight ratios after sintering in the furnace.

Fig 11: Magnesium oxide foam of wt% 55-45 of MgO and NH_4HCO_3

Fig 12: Magnesium oxide foam of wt% 65-35 of MgO and NH_4HCO_3

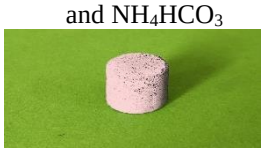

Fig 13: Magnesium oxide foam of wt% 75-25 of MgO and NH_4HCO_3

2.7 Determining the compressive stress of magnesium oxide foam :

Samples of magnesium oxide foam of different weight ratios were taken to test the compressive stress. The compressive stress was measured by a Universal Testing Machine.

4. Data Collection and Analysis

After preparing the magnesium oxide foam by powder metallurgy method, the foams are still subjected to the tests like porosity test and compressive stress test. Required data will be collected and measured for assessing the foams produced in this project. In this chapter, the required data collection method is discussed and the data are stated and showed in tables.

4.1 Data collection method

Magnesium oxide foam was obtained by mixing it with a space holder, compacting and sintering in the furnace. Here, for measuring the density, height and diameter of the foam were measured before and after sintering by using slide callipers and mass was measured using an electronic balance. Hence, density before and after sintering was measured from this data. The porosity of the respective foam was calculated from the difference in densities before and after sintering. Besides, porosity was calculated compared to the original MgO density (3.58 g/cc). The density and porosity can be calculated from the following equations.

Density, $\rho = 4m/\pi d^2 h$ ……..(1)

Where m is the mass of the foam,

d is the diameter of the foam,

h is the height of the foam.

Porosity = $(\rho - \rho_o/\rho) \times 100$ …… (2)

Where ρ is the density before sintering and ρ_o is the density after sintering

4.2 Data collection

Different dimensions of the foam before and after sintering were measured for analysis. They are stated below in table 1

Table 1: Different dimensions of foam with calculated density and porosity

Serial No.	MgO Powder: Space Holder	Before Sintering				After Sintering				Porosity (%)	Porosity compared to the original density of MgO (%)
		Mass (m) gram	Height (h) cm	Dia (d) cm	Density (ρ) =g/cc	Mass (m) gram	Height (h) cm	Dia (d) cm	Density (ρ) =g/cc		
1	75:25	5.97	1.98	1.5	1.71	4.59	1.81	1.44	1.56	8.77	56.42
2	65:35	6.04	2.03	1.5	1.68	4.01	1.84	1.42	1.38	17.86	61.45
3	55:45	5.95	2.16	1.5	1.56	3.45	1.98	1.38	1.16	25.64	67.60

6. Results and Discussions

Magnesium oxide foam was fabricated following the necessary steps and the porosity was calculated by measuring several parameters of the fabricated foam. The compressive stress was also measured by using a Universal testing machine. In this chapter, the acquired data and results of the tests will be analyzed and discussed elaborately.

6.1 Summary of the result

The porosity and compressive stress of magnesium oxide foam are shown below in table 2.2 and table 2.3 respectively.

Table 2: Density and porosity of the magnesium oxide foams of different ratios

Serial no.	Magnesium oxide powder: Space holder	Density (g/cc)	Porosity % (After sintering)	Porosity % compared to original density of MgO
1	75:25	1.56	8.77	56.42
2	65:35	1.38	17.86	61.45
3	55:45	1.16	25.64	67.60

Table .3: Compressive stress of magnesium oxide foams of different ratios

Serial no.	Magnesium oxide powder: Space holder	Compressive stress (MPa)
1	75:25	197.6
2	65:35	122.1
3	55:45	94.3

Pore structure analysis has been carried out by the Image J software. By using the software the mean diameter of the pore and pore to pore distance were measured. The mean diameter of a sample was measured by analyzing the 8-10 pores and the average distance among the pores were measured by analyzing 20distances .

Table 4 : Mean diameter of pore and pore to pore distance of foams of different ratios

Magnesium oxide powder: Space holder	Mean diameter of the pore (mm)	Pore to pore distance (mm)
75:25	0.581	1.015
65:35	0.708	0.720
55:45	0.862	0.402

6.3 Discussion on result : In this project, magnesium oxide foam has been fabricated by powder metallurgy method and the properties of the foam depends on few factors like the properties of the base metal powder, the density and porosity of the foam, the topology of the pore and the compressive stress of the foam. These factors affect the properties of the foam.

The characteristics and properties of the foam can be varied by a significant range by controlling and varying the parameters mentioned above. Here in this magnesium oxide foam fabrication process, the porosity of the foam is seen to vary from 8.77% to 25.64% with the increasing amount of space holder powder while by comparing to the original density of magnesium oxide the porosity ranged from 56.42% to 67.60%. The density of the foam is measured to vary from 1.56 g/cc to 1.16 g/cc with the increase of the space holder. The compressive stress of the foam was tested by a UTM and is seen to vary from 197.6 MPa to 94.3 MPa with the increase of the space holder. Hence we see that more amount of space holder results in higher porosity but less density and compressive stress. Higher porous foams are entitled to lower compressive strength.

Here the pore diameter and pore to pore distances are observed to vary with the variation of the porosities of the foam. The mean diameter of the pore is seen to vary in the range of 0.581 mm to 0.862 mm with the increase of the space holder. The mean diameter of the pore of foam with 75:25 wt% is 0.581 mm and for the foams of 65:35 wt% and 55:45 wt% are 0.708 mm and 0.862 mm respectively. With the increased amount of the space holder, the porosity has increased and as a result, the pore became interconnected and larger in diameter. Similarly, the pore to pore distance of the foams was seen to decrease with the increase of porosity. The average distance among the pores of the foam of 75:25 wt% is 1.015 mm while for the foams of 65:35 wt% and 55:45 wt% are 0.720 mm and 0.402 mm respectively. It is clear that with more space holder causing greater porosity, the pores increased in numbers, hence the distance among the pores decreased. In short, it is observed that with the increasing amount of the space holders and porosity, the mean diameter of the pore of foams is increasing and the pore to pore distance is decreasing.

6. Conclusion

Magnesium oxide foam via the powder metallurgy method has been fabricated in this project thesis. Ammonium bicarbonate was used as a space holder. Pure magnesium oxide powder and space holder powder were measured in different ratios and mixed in the ball milling machine for obtaining a homogeneous mixture. Uniaxial compression of the mixture was then done and later the dense objects of different weight ratios were sintered in a furnace at 1475°C for 6 hours. Hence the magnesium oxide foam was fabricated. Density and porosity after the sintering were calculated and analyzed accordingly. For the ratio of 75:25 wt%, the density and

porosity were 1.56 g/cc and 8.77% respectively. The density decreased for the ratios 65:35 wt% and 55:45 wt% up to 1.38 g/cc and 1.16 g/cc. The porosity of the foams of ratios 65:35 wt% and 55:45 wt% increased to 17.86% and 25.64% respectively. The compressive stress for ratio 75:25 wt% was 197.6 MPa and has been seen to decrease up to 122.1 MPa and 94.3 MPa for the ratios 65:35 wt% and 55:45 wt%. Pore size control and uniform distribution of pore were seen to be difficult to obtain. The mean diameter of pore for the ratio of 75:25 wt% was 0.581 mm and increased further to 0.708 mm and 0.862 mm for the ratios 65:35 wt% and 55:45 wt%. Pore to pore distance has been measured at 1.015 mm for 75:25 wt% and decreased for the ratios 65:35 wt% and 55:45 wt% to 0.720 mm and 0.402 mm respectively.

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NOMENCLATURE

m : Mass , gm
h Height , cm
d : Diameter , cm
ρ : Density g/cc
c : Compressive stress, Mpa