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Preparation and comparative evolution of mechanical behavior of Fe and Fe_2O_3 foams and their polymer composites

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ABSTRACT

The open cell foams with reticulated structure are cellular materials which is gaining interest in the recent past not only because of scientific interest but also because of their technological importance. By the virtue of their inherent properties derived out of their unique structure, made them promising and rather indispensable materials for wide range of applications. Some of the examples are structural parts, aerospace components, filters, energy absorbing, insulation, catalysis, heat exchangers and energy absorption. In the present investigation, highly porous reticulated open cell iron oxide (Fe_2O_3) and iron (Fe) foams are successfully fabricated by polymeric sponge replication process. In order to explore the possibility to enhance the energy absorption capability of this cellular materials, Fe_2O_3 and Fe foams prepared in this study are encapsulated and infiltrated with epoxy polymers which have exhibited an enhancement of weight by 1.45–1.90 times as compare to bare foams. These composite specimens are being metallic/ceramic skeletons with second phase as epoxy polymer on encapsulation/infiltration are expected to fail under stresses with unique mechanisms. This substantially enhances the energy absorption though extend of weight increase is not significant. The bare and composite foam samples were evaluated for quasistatic compression and dynamic impact behavior under identical conditions. On compression, a significant enhancement in energy absorption of 42 and 90 times for Fe_2O_3 and a less prominent enhancement of 1.3 and 1.95 times in case Fe foams are observed on encapsulation and infiltration respectively. Under dynamic impact an enhancement in energy absorption of 4 and 11 times in case of Fe_2O_3 and for Fe foam 6.3 and 20.6 times are observed on encapsulation and infiltration respectively. It is evident that energy absorption property is a strong function of material of fabrication, epoxy phase and stress type and conditions. The current study demonstrates an effective way to enhance the mechanical properties of ceramic (Fe_2O_3) and metallic (Fe) cellular foams through epoxy polymer encapsulation/infiltration process though infiltration have exhibited significant increase in both cases.

1. Introduction

Cellular structured materials with unique mechanical properties in combination with lower relative densities in comparison to their solid counterparts are extensively used for emerging applications [1,2]. Cellular materials mainly classified in two major groups based on their configuration and arrangement of cells such as honeycombs and foams. Honeycombs are characterized by two-dimensional regular array of cells with the shape of square, triangular or hexagonal and foam consists of three dimensional array of network of struts that interconnects with the outer surface to the inner body [3,4]. Cellular structures can be made of wide range of materials such as polymers, metal, ceramics, and composites. Basically the mechanical properties of cellular structure materials depends on the relative density of foam, material of fabrication, second phase and other cellular properties like cell size, cell density and cell geometry [5].

Among the various available fabrication methods to produce open cell foams, the polymeric sponge impregnation technique is a widely practiced and is regarded as a versatile technique because of its inherent advantages of attaining the engineered porosity and pore structures [6]. Relative density of the foam is the fundamental property that plays a dominant role in the specific mechanical properties [7,8]. Cellular solid structures with engineered

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properties are having wide range of application in commercial, military and structural applications such as sacrificial crash boxes [9], acoustic insulation and thermal applications [10–12]. Open cell iron foam is most attractive cellular structure for structural applications, such as light weight structures, sandwich foam panels in aircraft engine etc. [13–15]. However, there it is beneficial if it is possible to enhance the mechanical properties especially the energy absorption properties with these light weight structures [16–18]. Seismic first fabricated metallic foam in 1948 [19], followed by many researchers followed different techniques for fabrication of metallic foams such as thermal decomposition [20], chemical vapor deposition [21], and investment casting [22].

Aluminum alloy-based foams are widely used as the core materials for in energy sandwich panels and absorption application [23]. Iron foam having several advantages when compared to aluminum alloys, such as high strength and stiffness, high energy absorption property, high melting point and low cost [24]. In the present study Fe_2O_3 foams are fabricated by polymeric sponge replication process.

In order to achieve uniform and defect free foams a homogeneous and stable slip of Fe_2O_3 with desirable viscosity was prepared using organic surface modifiers and binders. The foams are sintered at 1125 °C and further reduced to Fe foam under hydrogen atmosphere at 1300 °C. A systematic study has been undertaken to explore the effect of encapsulation and infiltration with epoxy polymers within the cell on enhancement of energy absorption capabilities. Light weight being a prime advantage which is compromised on encapsulation to an extend of 1.45 times and 1.9 times by infiltration of epoxy materials, the advantage of substantial enhancement can be obtained due to the mechanism operative in a composite failure. Investigation in this direction is limited and hence, the study was undertaken. Study also carried out evaluation of bare foams and encapsulated and infiltrated composites which are subjected to compression strength and Charpy impact test to calculate specific compressive strength (SCS) and specific impact energy (SIE) respectively. Fractographic studies on these samples were also carried out and attempts were also made also to propose a mechanism for significant energy absorption properties demonstrated by these specimens up on encapsulation and infiltration.

2. Experimental procedure

Fe_2O_3 foams were fabricated by polymeric sponge replication process. In this process Fe_2O_3 powder (99.9% purity, Alfa Aesar) were made into an aqueous slip with deionized water. Methylcellulose as a binder and Darvan 821 A as a dispersant were added and ball milled for 4 h with alumina grinding balls to achieve 55 wt% solid loading. Slurry was characterized for their viscosity using a rheometer (Anton Paar, Austria). Polyurethane foam (PUF) with 10 PPI (Pores Per Linear Inch) used as the template and were cut with shrinkage allowance into $25 \times 25 \times 25\,mm^3$ sample suitable for compression test and $70 \times 25 \times 25\,mm^3$ rectangular samples suitable for impact. Coating of Fe_2O_3 slurry on the PUF was carried out by immersing the PUF in the homogeneous Fe_2O_3 slurry. Excess slurry was removed by gently blowing compressed air without affecting the coating on PUF. PUF foam was dried and repeated the process till close to 10 times of initial weight of PUF is achieved. Finally, PUF was coated with a fine spray of the slurry to eliminate defects based on visible inspection. Slurry coated PUF was finally dried at 50 °C for 2 h in a hot air oven.

Dried foams were subjected to organic burning out at 550 °C with low heating rate of 1 °C/min followed by heating upto 1125 °C for 1 h in air furnace. Heating schedule followed was based on TG/DTA analysis (STA 2500, Netzsch, Germany) of the PUF and final sintering temperature was arrived at based on the dilatometric study. Sintered Fe_2O_3 samples were reduced in Fe foam in hydrogen atmosphere at 1300 °C for 1 h Fe_2O_3 and Fe foam samples were characterized for their density through Archemedics principle (ASTM 396) and also for the cellular properties using scanning electron microscope. Foams were also characterized for their phases by XRD (XPert Powder, PAN analytical, Netherland).

Fe_2O_3 and Fe foam samples were encapsulated, and vacuum infiltrated with epoxy polymer to make the composite specimens. Both the foams were placed within a specially designed mould. The epoxy polymer formulation was mixed thoroughly without generating air bubbles and poured the solution in the mould containing the foams under vacuum. Foams with dimensions of $25 \times 25 \times 25\,mm^3$ and $70 \times 25 \times 25\,mm^3$ were infiltrated. After infiltration, the vacuum infiltrated Fe_2O_3 and Fe foam was placed in the oven at 70 °C for one and half an hour for effective polymerization. For encapsulation both the foams were first wrapped in a 10 μm thick aluminium foil in order to avoid polymer infiltration into the cells. Foam samples were immersed in the epoxy polymer solution and polymerized under the above conditions.

Static compression test on the bare, encapsulated and infiltrated foam samples were carried out using a Universal Testing Machine (Tensile Testing Machine-30 T, Heico, India) at a strain rate of $0.003\,s^{-1}$ at room temperature. Charpy impact test (AIT-300N2011-224, Hi-tech India Equipment's) was also performed on the bare, encapsulated and infiltrated foam samples.

3. Results and discussions

A flowchart of the polymeric sponge replication processing of foam is shown in Fig. 1 and viscosity vs. shear rate plot is shown in Fig. 2. Viscosity of the slurry is very critical not only to achieve percentage loading on the foam but also to achieve a uniform coating on the foam. The slip has exhibited a pseudoptotic behavior in the lower shear rate. A shear thinning behavior is preferred for retaining the slip on the polymer studs after coating and thixotropic over a period to adhere uniformly while excess slip is removed. This can be attributed to the optimized binder (2 wt% methylcellulose) and dispersant (Darvan 821 A, 1 wt%) concentration added to the slip formulation and ball milling process.

TG/DTA plot of the PUF is shown in Fig. 3. It is evident from the TG plot that weight loss is started at around 260 °C proceeds through major thermal exo events indicated by two broad DTA peaks leading to the complete loss in the range of 550–600 °C. This corresponds to the thermal degradation of urethane structure which is the inherent chemistry of polymeric foams [25]. As decomposition of the polymer occurs through formation of mainly gaseous products, hence a lower heating rate is preferred to avoid sudden release of gaseous products creating stresses in the Fe_2O_3 coatings forming minor cracks. However, a longer soaking time will ensure to reach an equilibrium to ensure close to complete loss of organic formulations. An optimum heating schedule followed in the current study is depicted in Fig. 4.

As the infiltration and encapsulation is by similar process, a typical Fe_2O_3 and Fe foams are presented in Fig. 5 (a) and (d). It is evident from the figure that there is no air bubble or defects on the encapsulated and infiltrated samples. Sintered densities and cellular properties of Fe_2O_3 and Fe foam samples are shown in Table 1 and XRD plots showing the phase pure pattern are shown in Fig. 6. It is evident from the XRD pattern (Fig. 6(b)) that reduction of Fe_2O_3 to Fe is completed and no evidence for the presence of Fe_2O_3 is observed.