ICMIEE-PI-140137

Compressive Properties of Ceramic Microballoon Syntactic Foams

Zulzamri Salleh [1,2], *Md Mainul Islam* [2*] *and Jayantha Ananda Epaarachchi* [1]
[1]Centre of Excellence in Engineered Fibre Composites and School of Mechanical and Electrical Engineering,
Faculty of Health, Engineering and Sciences, University of Southern Queensland,
Toowoomba, Queensland 4350, AUSTRALIA
[2]Universiti Kuala Lumpur, Malaysian Institute of Marine Engineering Technology (MIMET),
32200, Lumut Perak, MALAYSIA

ABSTRACT
In this paper, fabrications of vinyl ester syntactic foams with different contents of ceramic microballoons or microspheres were prepared using the open mould method for compression specimens. The effects of the ceramic microballoons on the mechanical properties, particularly compressive properties were investigated. The results show that the syntactic foams with vinyl ester matrix possess strength behaviour varied with different contents of ceramic microballoons. Ceramic microballoons have an important effect on the stress-strain, mean plateau stress and strain energy absorption capacities of syntactic foams. The results also show that the content of ceramic microballoons in vinyl ester syntactic foams should be controlled in order to obtain a good combination of compressive strength and energy absorption capacities. The reasons are discussed here in details.

Keywords: Syntactic foams, ceramic microballoons, compressive properties, content.

1. Introduction

Ceramic microballoon or microspehere is defined as an inorganic nonmetallic polycrystal sphere or approximate sphere with the size of micron [1]. Ceramic microballoon is widely used in the medical industry, chemical and nuclear industry and also in the defence industry. From the literature review, ceramic microballoons are widely used combining with Aluminium matrix. It is not only limited use for aviation, but also used for civil industry and automotive industry [1]. This metallic foam is defined as metal-matrix syntactic foams (MMSFs) with the first production in 1990s [2]. The structural engineering is always desired for the better mechanical properties of material such the compressive behaviour of the products. For example stronger and lighter materials such as syntactic foams are better choice for both of the mechanical behaviours particularly for structural engineering. In the previous works, many researchers were interested on the properties of the foams particularly characteristic on the compressive strength and the absorbed energy. The investigation on the effects of the microballoon size on the compressive strength is very beneficial for future engineering [3]. From the research it was found that smaller microspheres ensure higher compressive strength because they contain fewer flaws in their microstructure than the larger ones. While Palmer et al. proved that larger microspheres contain more porosity in their walls and more flaws in their microstructure than the smaller ones [4]. From the literature review it was also found that the chemical reaction contributed detrimental effect on the load transfer during mechanical testing [5]. Balch et al. found that the microspheres have at least the same importance in the syntactic foams as the matrix material [6]. The fracture strength and the yield strength of the matrix determine the failure stress of the syntactic forms. Therefore, the investigation on mechanical properties, particularly compressive strength effect will be affected the quality of the microspheres and it is very important. Nevertheless, no report was published for ceramic microballoons mix with vinyl resin as matrix materials. In this study, the report will cover from the preparation of samples to the mechanical properties. The aim of this study is to investigate the distribution of the constituents in the ceramic microballoons with the different contents' effect on the vinyl ester resin as matrix material. All these information will be beneficial and acceptable for the production of ceramic microballoon of syntactic foams.

2. Materials and Methodology

Ceramic microballoons type SL75 is used in the difference weight percentages as 2.0wt.%, 4.0wt.%, 6.0wt%, 8.0wt.% and 10.0wt.% to fabricate these foams. E-spheres a ceramic bubble was supplied by Envirospheres Pty. Ltd (Australia) company are used in this study [7]. Their main parameters are provided by supplier is listed in Table 1.

Table 1 Properties of ceramic microballoon.

∇	ρ	T	Compressive Strength	Chemical Composition
45	0.40	1600-1800	45MPa	SiO_2-60% Al2O_3-38% TiO_2-2%

Fig. 1 shows the SEM photo for ceramic microballoon provided by the supplier [7]. Vinyl ester resin, supplied by Norox Australia Company, is used as the matrix material. This is diglicidyl ether of bisphenol A-based resin. An amine-based MEKP is used as a hardener.

* Corresponding author. Tel.: +6174631 1338
E-mail address: Mainul.Islam@usq.edu.au

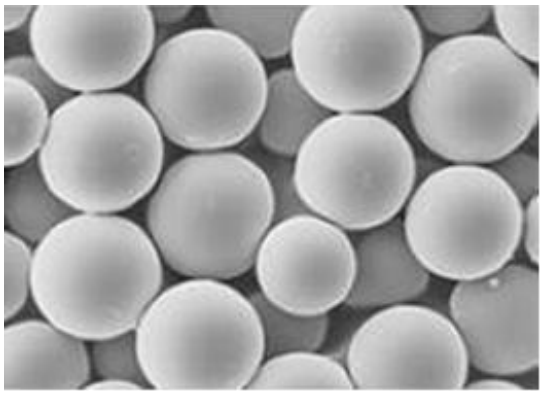

Fig.1 SEM photo for ceramic microballoons [7]

The synthesis method consists of mixing measured quantities of ceramic microballoons in the vinyl ester resin and mixing them until a slurry of uniform viscosity is obtained with the intermittent mixing approximately 4 ~ 5 minutes. The hardener is added to this slurry and gently mixed using a stir magnetic bar at stir machine until completely mixing also approximately 4 ~ 5 minutes. Fig. 2 shows that the slurry was mixed with stir magnetic bar during the process of syntactic foams.

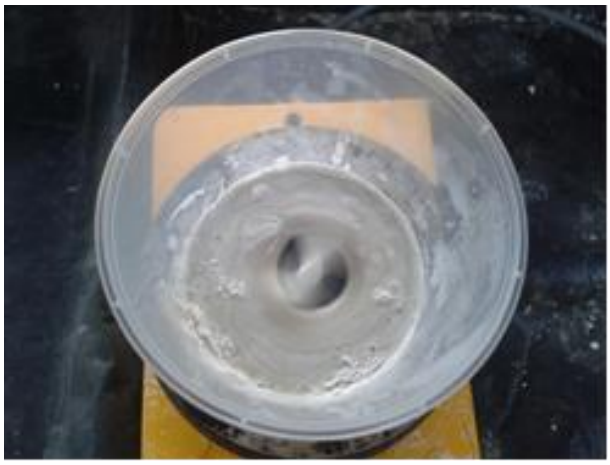

Fig.2 The magnetic stir bar for mixing process

The mixture is then cast in PVC pipe moulds with the diameter 19mm and length 42mm. The overview of one of the specimen photos shown at Fig. 3 after re-moulded process and next is sent to the curing process. This size is following the ASTM D-695 for compression test of syntactic foams [8]. The specimens cure in the mould for 24 h at room temperature and re-moulded on next day for post-cured at 80°C for 4 h in the oven. After post cure all the specimens need to trim by using the grinder machine on the top and bottom surfaces. This process is done before proceeding to compression testing because both surfaces need the flatness surface in order to avoid the erosion phenomena which is will affect the compression values. The compression values also can be affected when the specimens were not located properly between upper jaw jig and lower jaw jig. Hence, the specimens must be centred on the lower jaw jig surface and make sure it is not too tight or too loose. Otherwise the starting point will have negative values, and then the graph will not start from the zero point. Selection for the data is important while plot the graph for stress versus strain during the data analysis process.

Fig.3 Overview one of specimen syntactic foam for compression test

Compression test is carried out using MTS test systems show at Fig. 4. The test is carried out at the constant deformation rate of 2 mm/min. The compression tests are carried out on specimens following the ASTM-D695 dimensions. Fig. 5 shows the enlarged photo for one of the specimen was tested during compression test at MTS machine. From the output result, there are two parameters has been selected for data analysis such as load and crosshead displacement to develop the stress–strain curves. Then for next step it is necessary to re-calculate the actual values of stress and strain by using the area formula for cylindrical shape of each sample. This is a very important procedure need to be taken because the raw data for the stress and strain from the machine sometime inaccurately.

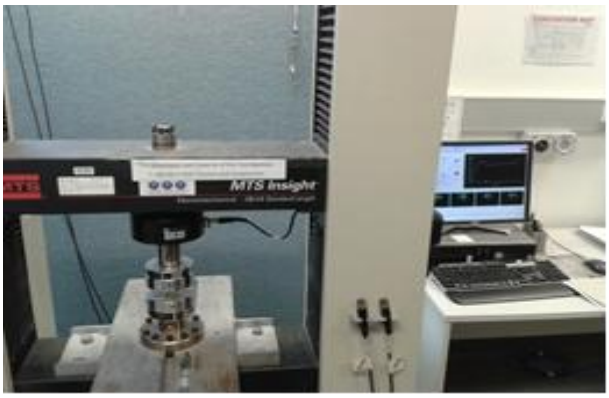

Fig.4 MTS Insight machine for compression testing

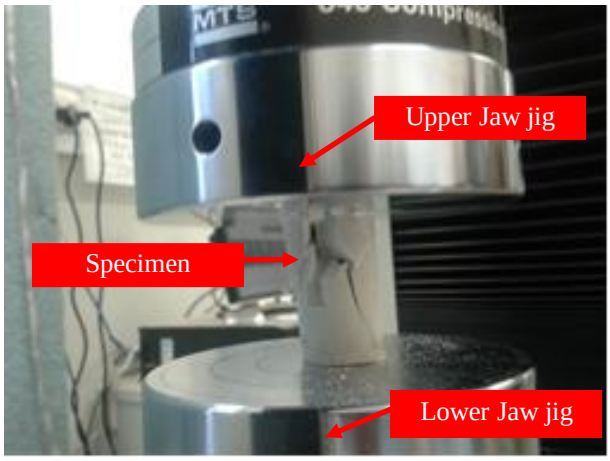

Fig.5 MTS Specimen in-progress for compression test

3. Results ad Discussion

The representative compressive engineering stress-engineering strain curve for all types of vinyl ester/ceramic microballoons are presented in Fig.6. From the stress–strain profiles both neat resin and syntactic foams show the same consisting of a linear elastic region followed by a strain softening region that is characterized by a slight drop in stress. When the compression is continued further, then the stress starts rising again. The increase in stress is faster and significantly higher in the case of neat resin, whereas for syntactic foams it depends on the type and volume fraction of microballoons [8]. The compressive modulus values are measured as the slope of the initial linear region of the stress–strain graphs and are presented in Fig. 6. This initial linear deformation region (I) is where stress, increased linearly to the first peak (from this gradient line it can be defined the yield strength or Young's Modulus), followed by a plastic plateau stage (II) where stress slightly increased as the strain increasing, then a densification stage (III) where stress raises sharply with strain increasing slightly. It can be observed that all syntactic foam compositions show a stress plateau, which is a typical feature of most types of syntactic foams[11].The compressive modulus values at stage (I) is measured as the slope of the initial linear region of the stress–strain graphs shown in Fig. 7. The results show that the compressive modulus of syntactic foams increases with increasing the contents of microballoons and only composition with 2wt% shows the lower modulus behaviour. It is also seen that the modulus decreases as the volume fraction of the same type of microballoons increases. Several syntactic foam compositions show compressive modulus values comparable to that of the neat resin, however, the specific moduli for most composites are 10–47% higher than the neat resin tested at the same compression rate, see Fig. 6. Hence, for compressive loading conditions, syntactic foams can lead to a significant advantage over the neat resin in terms of weight saving. Compressive strength of composites is defined as the first peak in the Engineering stress–Engineering strain curves at stage (I).

Fig. 7 shows that the strength values for several foam compositions are comparable to each other's.

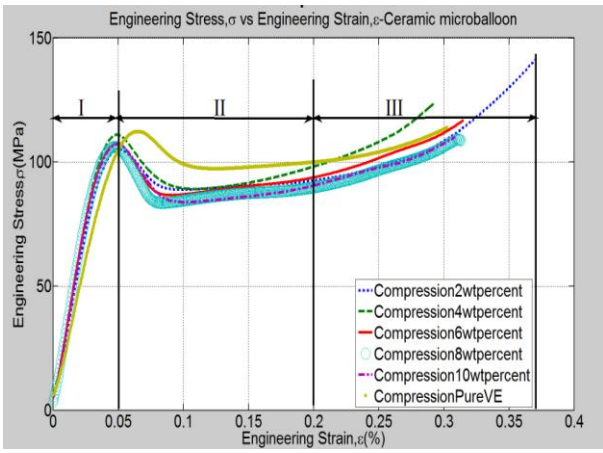

Fig.6 Engineering stress–Engineering strain curves.

The results also indicate that the compressive strength can be tailored over a wide range by selecting microballoons SL75 by using them in different weight percentages. The yield strengths of the foams with ceramic microballoon contents of 2.0wt.%, 4.0wt.%, 6.0wt.%, 8.0 wt.% and 10.0wt.% are about 102, 115, 105, 103 and 104 MPa, respectively.

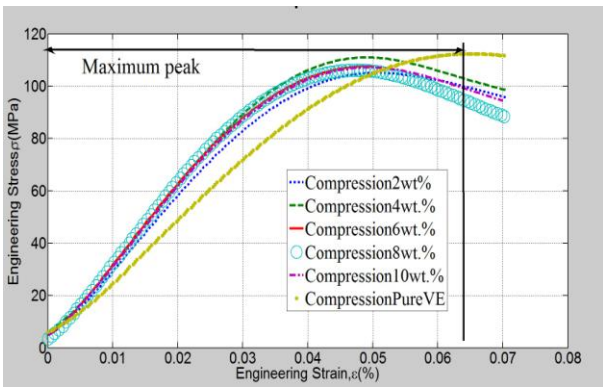

Fig.7 Graph for initial linear deformation region (I).

For the mean plateau stresses of these foams at stage (II) (shown in Fig. 8 are around 95, 124, 90, 87 and 89 MPa. This indicates that SL75 ceramic microballoons have a significant effect on the foams yield strength, mean plateau strength and densification stress under compression state. The yield strength for the specimen 4.0wt.% have the maximum stress is about 115MPa. Similar phenomenon appears on mean plateau stress of the foams as having the higher stress of 124MPa. All of these results could be effected on higher energy absorption capacity of the foams containing different contents of SL75 ceramic microballoon (as shown in Fig. 10) and the energy absorption capacity decreased with the strain increasing like other foams [7,12]. Therefore, for densification strain, SL75 ceramic microballoons enhance the densification strain of the foams. It is interesting that there is slightly reduction in

the energy while variety of the foams increased with different contents of SL75 ceramic microballoon.

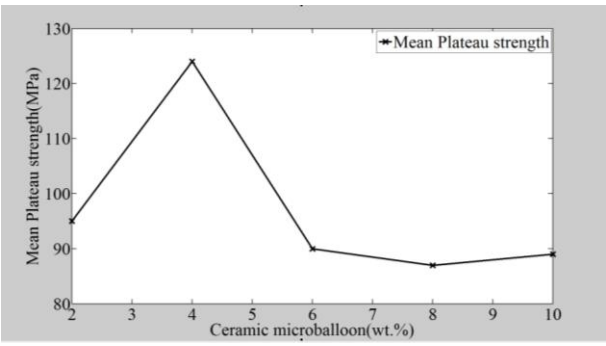

Fig.8 Graph for mean plateau region strength (II).

Fracture features of five types of syntactic foams are compared in Fig. 9. It is observed that the foam specimens containing 2wt.% microballoons deform with longer time to fracture as well as have higher stress behaviour at 141MPa compared other specimens. Although the specimens with 4wt.% have maximum compressive strength in the earlier stage (I) but it did not sustained at the end because it fractured around 124MPa compared with specimen 2wt.%. The failure features of these specimens are similar to those presented earlier [31], which include initiation of shear cracks in the specimen and formation of fragments from the side walls. Inclusion of higher weight percentage of stiff ceramic microballoons in relatively ductile matrix results in increased brittleness of the composite.

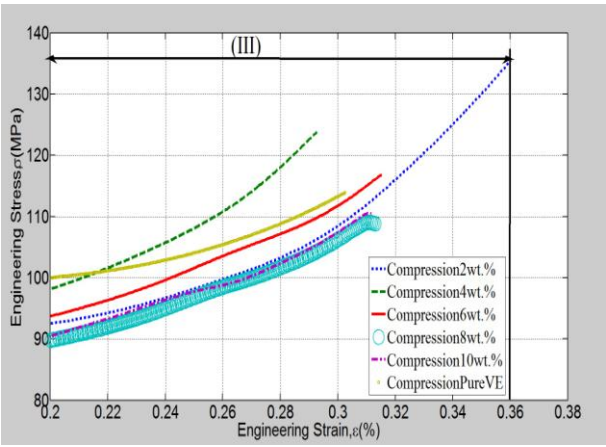

Fig.9 Graph for fractured feature region (III).

An area under the graph of the engineering stress-Engineering strain can be defined as energy absorption capacity [9]. Numerically it can also be determined by using the trapezoidal integration method and can be plotted by using the Matlab software. Energy absorption capacity (E) is an important aspect to evaluate the properties of metal foams. It is also known as energy absorption and can be calculated till the end of plateau region (II) i.e., where the stress value starts to increase again [10]. Fig. 10 shows the graph of energy absorption capacity vs microballoon contents. The energy absorption capacity (E) of closed-cell vinyl ester ceramic syntactic foams was calculated according to Eq. (1):

$$E = \int_0^{\varepsilon} \sigma d\varepsilon \qquad (1)$$

Where σ is called as engineering stress and ε is called as engineering strain which is varied from zero to maximum strain values. The energy shows decreases after adding more weight contents of ceramic microballoons starting with initial content of 2wt. %, initially starting from 40J then decrease slowly, almost four times to 32.3J. This trend was observed and reported that it was similar for all the ceramic material systems [11]. This result also supports that the ductility of the specimens is more, hence the internal structure become weak and has difficulty to combine with matrix vinyl ester resin.

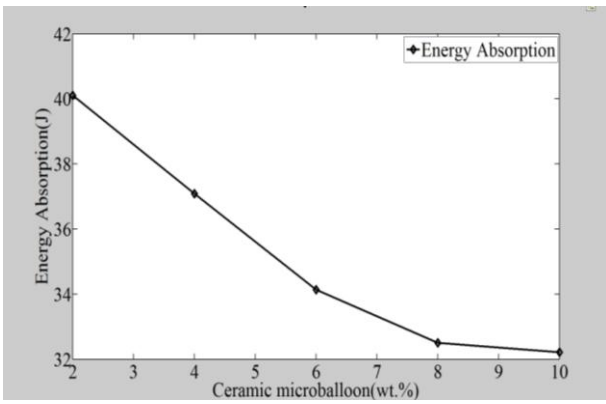

Fig.10 Graph for energy absorption capacity.

4. Conclusion

The effects of SL75 ceramic microballoons on the compressive properties of vinyl ester syntactic foams were studied and the results were summarised as follows:

- The distribution of ceramic microballoons particularly of 4wt.% possesses the highest compressive stress.
- The addition of ceramic microballoons prominently improved the mean plateau stress specifically, for specimens with 4 wt.% while for both behaviours of densification strain and energy absorption capacity was led by specimens with 2 wt.%.
- Hence, by using the small amount of ceramic microballoons it might also reduce the porosity contain in the specimens, and additional checking is needed to study on this matter.

It is clearly shown that to achieve the optimum stress value, the contents of ceramic microballoons in the syntactic foams also should be limited because to ensure that it is not difficult to mix with vinyl ester resin.

5. Acknowledgment
The authors wish to thank Universiti Kuala Lumpur Malaysia and MARA for providing scholarship to first author on doing this work.

NOMENCLATURE

∇ : Mean particle size, µm
ρ : Bulk density, g/m^3
T : Melting point, ^{o}C
σ : Engineering Stress, MPa
wt.% : Weight percentage
ASTM: American Standard Testing Method
E : Energy Absorption, kJ
PVC : Polyvinyl Chloride
SEM : Scanning Microscope Machine
ε : Engineering Strain, %

REFERENCES

[1] Cheng X., Liu P., LI X., Shui A., and Zeng L., Preparation of Beauxite Ceramic Microsphere, *Journal of Materials Science*, Vol. 42(13), pp 4047-4054, (2006).

[2] G.H. Wu. Z.Y. Dou, D.L. Sun, L.T. Jiang, B.S. Ding and B.F. He, Compression behaviours of cenosphere–pure aluminum syntactic foams, *Scripta Materialia*, Vol.56, pp 221–224, (2007).

[3] Imre N. O., and Kornél M, Microballoon of Metal-matrix composite reinforced by ceramic microballoon, *Physics of Fluids*, Vol. 16, pp 11180-1126, (2004).

[4] R. A. Palmer, K. Gao, T. M. Doan, L. Green and G. Cavallaro, Pressure infiltrated syntactic foams-Process development and mechanical properties, *Materials Science and Engineering A*, Vol.(464), pp 85–92, (2007).

[5] D. K. Balch and D. C. Dunand, Load partitioning in aluminum syntactic foams containing ceramic microspheres, *Acta Materialia* Vol.(54), pp 1501–1511, (2006).

[6] D.K. Balch, J. G. O'Dwyer, G. R. Davis, C. M. Cady, G. T. Gray III, and D. C. Dunand, Plasticity and damage in aluminum syntactic foams deformed under dynamic and quasi-static conditions, *Materials Science and Engineering A* Vol.(391), pp 408–417(2005).

[7] Product specification Envirospheres Pty. Ltd, view 14 Nov 2014, <http://www.envirospheres.com.au/products.asp>

[8] N.Gupta, R.Ye, M and Porfiri, Comparison of tensile and compressive characteristics of vinyl ester/glass microballoon syntactic foams, *Composites: Part B*, Vol.(41), pp 236–245, (2010).

[9] C. Swetha and R. Kumar, Quasi-static uni-axial compression behaviour of hollow glass microspheres/epoxy based syntactic foams, *Materials and Design*, Vol.(32), pp 4152–4163, (2011).

[10] Periasamy C,Jhaver R and Tippur HV., Quasi-static and dynamic compression response of a light weight interpenetrating phase composite foam. *Material Science Engineering A*, Vol.(527), pp 2845–56, (2010).

[11] Xing C.X, Xiaowei C., Zan Z., Xu C., Weimin Z., Bo L. and Boyoung H., Compressive properties of closed-cell aluminum foams with different contents of ceramic microspheres. *Materials and Design* Vol.(56), pp 353-358, (2014).