



EFFECT OF VOLUME FRACTION EPOXY-HOLLOW GLASS MICROSPHERES AND CURING TEMPERATURE VARIATION ON COMPRESSIVE PROPERTIES OF COMPOSITES

Sutikno, Wajan Berata, Wahyu Wijanarko and Indra Sidharta

Mechanical Engineering Department - Faculty of Industrial Technology Institut Teknologi Sepuluh Nopember, Surabaya, Indonesia

E-Mail: sutikno@me.its.ac.id

ABSTRACT

Composite has growth rapidly in industrial applications. This research deals with epoxy-hollow glass microsphere composites since researches on it are very limited. Epoxy-hollow glass microspheres composites has advantages such as providing a light weight, low thermal conductivity and high compressive strength. This research focused on the volume fraction of epoxy-hollow glass microspheres, the curing temperature and its effect on the compressive strength of the composites. Compressive test specimen dimensions are produced according to ASTM D695, by varied hollow glass microspheres volume fraction of 0% to 30% and curing at room temperature and 90 °C. This research showed that 15% volume fraction of hollow glass microspheres and curing temperature of 90 °C gained the maximum compressive strength of the composites at 128.95 MPa. The maximum compressive strength is obtained due to high percentage crystallinity on composites matrix that occurred in adequate volume fraction of hollow glass microspheres and curing temperature.

Keywords: hollow glass microspheres, composites, curing temperature, compressive properties.

INTRODUCTION

In 2005, Soo-Jin Park and Fan-Long Jin [1] conducted research on effect of the volume fraction of hollow glass microspheres (HGM) in surface energy, thermal properties and electrical properties composite. In the research, curing temperature of 110 °C and hollow glass microspheres volume fraction of 0% to 2% were adopted. In 2011, C. Swentha and Ravi kumar [2] carried out research on effect of volume fraction hollow glass microspheres of 0% to 60% and epoxy matrix on the compressive strength at curing temperature of room temperature. This research investigates effect of variations of curing temperature and volume fraction hollow glass microspheres on compressive properties of composites with epoxy matrix. Volume fraction of hollow glass microspheres is varied from 0% to 30% and Curing temperature at room temperature and temperature of 90°C.

MATERIAL AND METHOD

Material

HGM is a hollow glass with thin-walled made of borosilicate glass containing sodium. Hollow glass microspheres has a high compressive strength, high strength-to-density ratio, grain size of about 18 microns, and have isostatic strength of 28,000 psi with a density of 0.6 g/cc on the type of hollow glass microspheres iM30K [3]. Hollow glass microspheres are often used as filler in lightweight composite materials such as syntactic foam and lightweight concrete. With low density properties, low thermal conductivity, and resistance to high compressive stress the hollow glass microspheres widely used on the

hull and the equipment used in deep-water drilling equipment.

Method

Epoxy-hollow glass microspheres composites material specimens are produced by mixing epoxy resin and hollow glass microspheres. Volume fraction of hollow glass microspheres is varied from 0% to 30%. The curing temperature are at room temperature and temperature of 90 °C for 24 hours. The compressive specimens are then tested by using compressive testing machine. The specimens are also investigated by Differential Scanning Calorimetry (DSC) and Scanning Electron Microscope (SEM).

RESULT AND DISCUSSIONS

Effect of Volume Fraction Hollow Glass Microspheres on Compressive Strength of Composites

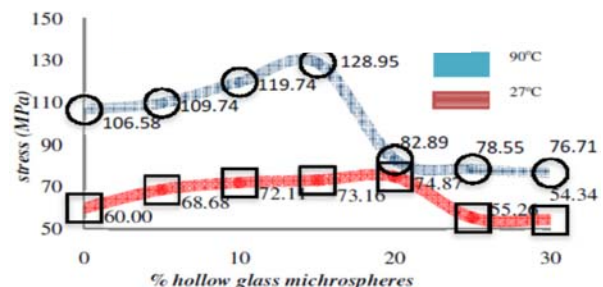


Figure-1. Compressive strength of composites cured at temperature 90 °C and room temperature.



Figure-1 shows that compressive stress of composite epoxy-hollow glass microspheres cured at temperature of 90 °C increased as the increasing of volume fraction hollow glass microspheres of 5-15%, and the highest compressive stress is 128.95 MPa in the volume fraction hollow glass microspheres of 15%. For volume fraction of hollow glass microspheres more than 20%, the compressive strength tend to be constants.

Effect of Volume Fraction Hollow Glass Microspheres on Compressive Strength of Composite

Figure-2 depicted the density of composite as a function of volume fraction hollow glass microspheres. The magnitude of the density tends to decrease as increasing of volume fraction hollow glass microspheres. Volume fraction hollow glass microspheres 30% has density of 0.94 g/ml. The density of pure epoxy resin is 1.08 g/ml.

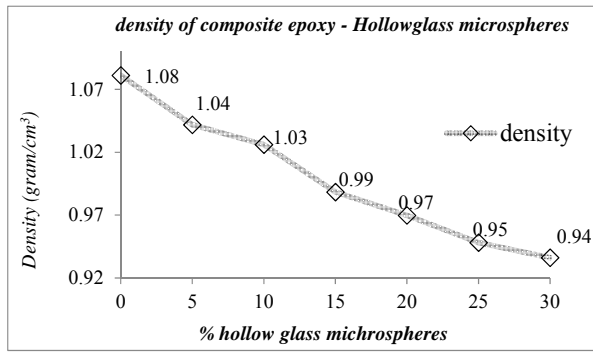


Figure-2. Curve density composite with volume fraction of hollow glass microspheres.

Deformation Behaviour



Figure-3. Fracture pattern of pure epoxy cured at temperature of 90 °C.



Figure-4. Fracture pattern of pure epoxy cured at temperature of 25 °C.

Result of the compressive test of pure epoxy resin shows that columnar fracture patterns appear as shown in Figure-3 and Figure-4. There are no differences between fracture pattern in the specimen cured at room temperature 25 °C and specimen cured at temperatures 90 °C.



Figure-5. Fracture pattern of epoxy-hollow glass microspheres 15% cured at temperature of 90 °C.



Figure-6. Fracture pattern of epoxy-hollow glass microspheres 15% cured at temperature of 25 °C.



Fracture pattern of Epoxy-hollow glass microspheres 15% composites cured at room temperature and temperature of 90 °C are shown in figure-5 and figure-6. Both specimens yield columnar fracture patterns.

Differential Scanning Calorimetry

DSC testing is conducted to determine value of the transition temperature (T_g) and crystallinity of pure epoxy, hollow glass microspheres, and volume fraction composites. The value of T_g is indicated by the peak value of the DSC curve. Specimens tested by using DSC start from the room temperature of 25 °C to 450 °C with increment temperature of 10 °C/min.

From the DSC test results, presented in Figure-7 and figure-8 shows that the transition temperature (T_g) of epoxy composites-hollow glass microspheres 15% cured at temperature of 25 °C is 113.4 °C and epoxy composites-hollow glass microspheres 15% at cured temperature of 90 °C has a transition temperature (T_g) of 117.36 °C.

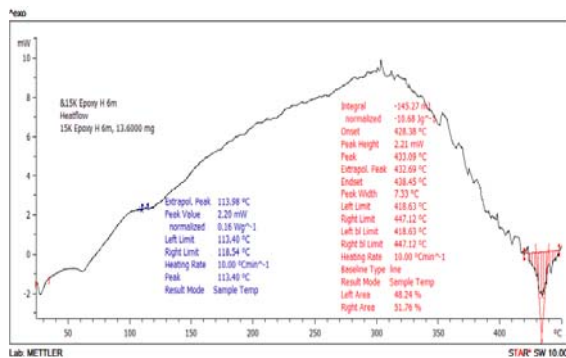


Figure-7. DSC curve of epoxy-hollow glass microspheres 15% cured at temperature of 90 °C.

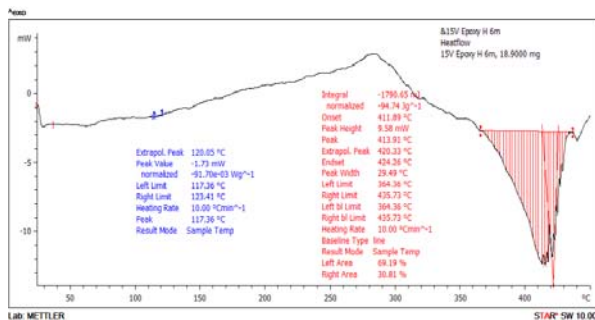


Figure-8. DSC curve of epoxy-hollow glass microspheres 15% cured at temperature of 25 °C.

Percentage of crystallinity contained in composite can be obtained through the calculation enthalpy of endothermic reaction and heat of fusion epoxy resin. Epoxy-hollow glass microspheres 15% cured at temperature of 25 °C has enthalpy of 10.68 J/g and epoxy-HGM 15% cured at temperature 90 °C has enthalpy of 94.74 J/g.

Analysis Microstructure on Deformation Behaviour

Analysis of fracture pattern on compressive test shows that the failure mechanism of the composite are almost the same on each specimen, the presence of matrix cracking, and debonding between the epoxy and hollow glass microspheres.

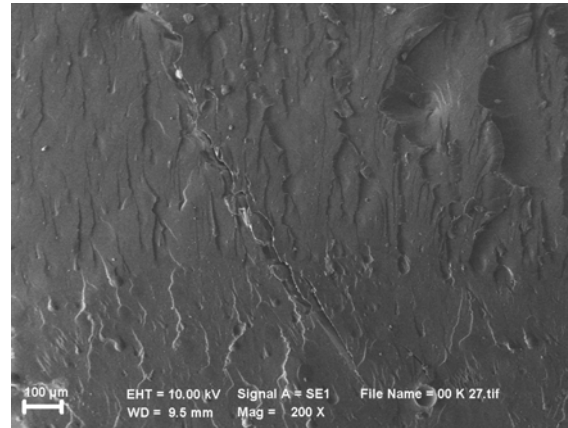


Figure-9. SEM result pure epoxy.

Pure epoxy fracture pattern obtained from Scanning Electron Microscope is shown in figure-9. There are cracks initiated and quickly spread without presence of reinforcements. So compressive load will be charged on strength of the epoxy resin itself.

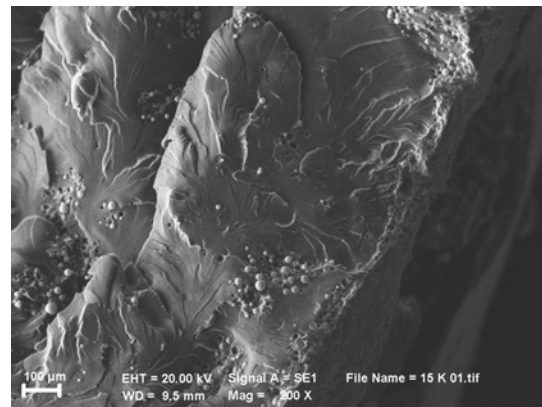


Figure-10. SEM result of volume fraction of 15% hollow glass microspheres cured at room temperature of 25 °C.

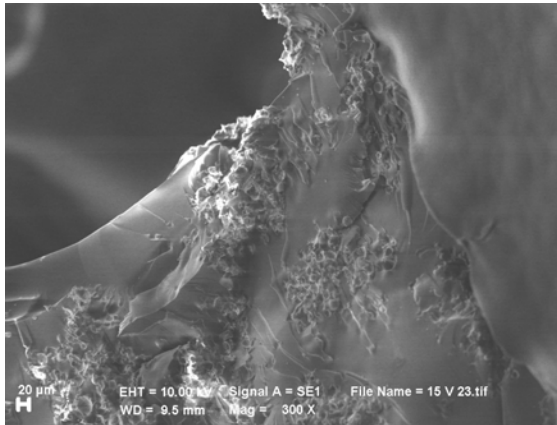


Figure-11. SEM result of volume fraction of 15% hollow glass microspheres cured at temperature 90 °C.

As depicted on figure-10 and figure-11, volume fraction 15% hollow glass microspheres showed that initial cracks spread from the matrix to hollow glass microspheres, and there are a lot of strong bonding between the epoxy with hollow glass microspheres.

Analysis DSC result

From DSC results seen that increasing volume fraction hollow glass microspheres in epoxy matrix is increase the temperature transition composite. Seen from figure-7 for the composite with volume fraction 15% hollow glass microspheres has transition temperature (T_g) is 117.46 °C and for volume fraction 20% hollow glass microspheres has transition temperature (T_g) is higher at 127.98 °C. This occurs because epoxy has T_g is 75.27 °C combined with hollow glass microspheres has higher T_g is 167.92 °C, so T_g of composite is mixture of both. This means that composite material can work at a higher temperature conditions than pure epoxy. T_g is indicated on curve of heat is absorbed (endothermic), because to change glass becomes viscous liquid phase requires more heat. This is appropriate with researched did Soo jin park [1]. But for the addition hollow glass microspheres in volume fraction will also affect the reaction endothermic (melting) because structure of hollow glass microspheres will be absorbing heat and heat prohibitive when curing composites. Heat transfer between the epoxy and hardener will be hindered by hollow glass microspheres and crystallinity matrix epoxy will decrease so DSC result curves endothermic reactions (melting) are small.

There is difference between the results of percentage crystallinity composite that used temperature curing at room temperature and curing at temperature 90°C with same volume fraction hollow glass microspheres. It is because by using a higher temperature curing, epoxy molecules to be more active has forming bonds with other molecules. Crystallinity percentage obtained from the enthalpy of endothermic reaction divided by the heat of

fusions epoxy resin. Heat of fusion is the enthalpy required to form 100% cristalinity on epoxy resins.

With increasing cristalinity on matrix composites will increase the strength of matrix so that the stiffness and strength of the matrix that surrounds hollow glass microspheres will also be greater and strength. This makes composite will have ability to withstand higher compressive loads. Bonding matrix which has a larger cristalinity will have mechanical properties and physical properties as well as. With well mechanical properties of epoxy matrix, there will be difference compressive strength up to 40% increase in compressive strength occurred when compared with composite curing at room temperature because it has fewer cristalinity.

Analysis Result of Compressive Strength

Compressive strength of the composites with volume fraction hollow glass microspheres up to 15% increase compressive strength. That is due to hollow glass microspheres covered with resin epoxy matrix so that the optimum thickness when compressive test hollow glass microspheres cannot move or shift cause compressive load. it is because the stiffness in thickness of the epoxy matrix that surrounds the hollow glass microspheres can withstand hollow glass microspheres cannot move, hollow glass microspheres will deformed to withstand compressive load and because hollow glass microspheres has high crush strength so need a high load for deform composition of the epoxy-hollow glass microspheres composites.

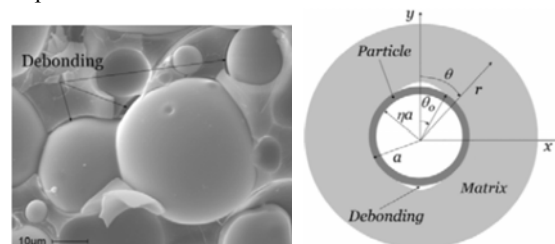


Figure-12. Composition of epoxy-hollow glass microspheres composites.

Seen in figure-12 because more hollow glass microspheres composites fill volume of the composite so matrix surrounds hollow glass microspheres will be thinner. This resulted in the stiffness matrix that holds hollow glass microspheres will decrease and when the compressive load will be much hollow glass microspheres getting loose from matrix without any deformation due to compressive load, so that the compressive strength of the composite will decrease with increasing volume fraction of hollow glass microspheres addition to the composite after optimal volume fraction hollow glass microspheres in composite is 15 %.



CONCLUSIONS

The increase of hollow glass microspheres volume fraction in the epoxy-hollow glass microspheres composites will increase compressive strength. The maximum compressive strength of 128.95 MPa obtained on epoxy-hollow glass microspheres composites for hollow glass microspheres volume fraction of 15%. The curing temperature contributes for increasing crystallinity percentage on epoxy matrix composites and improving compressive strength.

REFERENCES

- [1] Soo-Jin Park. 2005. "Preparation and physical properties of hollow glass microspheres-reinforced epoxy matrix resins". Korea
- [2] Swentha. C, Kumar Ravi. 2011. "Quasi-static uni-axial compression behaviour of hollow glass microspheres/epoxy based syntactic foams". Department metallurgical. India
- [3] Baris yalcin, "Polymer composite with hollow glass microspheres: processing, properties and applications", 3M company, 2012.
- [4] Andrew S. D'Souza, E. Stephen. Innovative High Strength Glass Microspheres for Extruded and Injection Molded Plastics. 3M Deutschland GmbH, Neuss, Germany.
- [5] Yung K.C., Zhu B.L., Yue T.M, Xie C.S, Preparation and Properties of Hollow Glass Microsphere-filled Epoxy-Matrix Composites, Composite Science and Technology 69 (2009) 260-264.
- [6] P. Wong, "Measurement of Mechanical, Electrical and Thermal Properties of Glass Powder Reinforced Epoxy Composites and Modelling", University of Southern Queensland, 2012.
- [7] Alexander Trofimov, Dr. Lev. Pleshkov, Haslen Back., Hollow Glass Microsphere for High Strength Composite Cores, Alchemie Technology 50 (2007) 44-46, 48-50.
- [8] Nie Shihua, 2004, "A micromechanical model for effective elastic properties of particulate composite with imperfect interfacial bond." University at Buffalo, USA
- [9] Schwartz, M.M., 1984. "Composite Material Handbook", McGraw Hill; Inc. New York.
- [10] Park, Metals. Composites. ASM International Handbook Vol. 1. 1987
- [11] Baris yalcin, "Polymer composite with hollow glass microspheres: processing, properties and applications", 3M company, 2012.