EFFECT ON PHYSICAL PROPERTIES OF EPOXY RESIN UNDER HYGROTHERMAL LOADING CONDITIONS

KHUSHMEET KUMAR, SUSHMA SINGH, JIMMY MEHTA

Abstract: In this paper the effect of Hygrothermal aging and moisture absorption on the micro-hardness of epoxy resin has been carried out experimentally. Hygrothermal aging on epoxy resin was conducted at temperatures of; room temperature, 45°C and 55°C in order to assess the susceptibility of material in wet conditions. Hygrothermal aging revealed that the immersion of material in the water results in the weight gain by the sample due to moisture. Moisture content leads to the swelling of the epoxy resin and results as the decrease in the micro-hardness of the material. Further the effect of loading conditions was also studied by preloading the samples. The preloading of the material also leads to the significant decrease in the micro-hardness of epoxy resin.

Keywords: Epoxy, Weight gain, Hygrothermal, Micro Hardness, Preloading.

Introduction: Composites are the materials which are formed by the combination of two or more materials, so that all the constituent material retains their properties but the properties of the composite may differ from the base materials [1]. Composites can be moulded to any shape which is required and they also have the high strength to weight ratio [2]. Composite materials were increasingly used in industry, military and aerospace applications. But when the fibre-reinforced composite material is exposed to humid and hot environment under loaded conditions then the material properties may change. These changes in material properties lead to the irreversible material degradation. The results of chemical changes and mechanical damage in general affect the overall material properties, e.g. elastic modulus, Hygrothermal expansion coefficients, diffusion coefficients [3].

Moisture content in the environment may damage the composite laminates such as debonding at fibre/matrix interface and results in cracking of composite material. The first observed damage mode is the matrix cracking. These cracks in the matrix material were not so dangerous individually, but when are exposed to surrounding moisture and thermal conditions a rapid adsorption of moisture may take place which lead to the accelerated material degradation. So the matrix material degradation is one of the major areas for investigation of their properties. Most investigations [4-12] have reported that composites based on the epoxy absorb significant amount of water which leads to degradation and dimension instability of the polymer material. These changes limit the material application in high tech industrial applications which demands high stability of materials under environmental conditions.

When a polymer is exposed to a small amount of heat over a long period of time, the cumulative effects can be considered equal to high temperature degradation under environmental conditions. The polymer had reached its thermal stability limits and begins to degrade; it was also known as thermal degradation or thermal aging [13-16]. Chemically and physically, the process of aging is irreversible with time. Taib et al. [17] experimentally studied the effect of home bleaching agent on the physical properties of acrylic moulds and found that 10% and 17% Carbamide peroxide has no different effect on surface hardness for an aging of 14 days. Koldrup [18] had experimentally investigated the effect of low ph beverages (coca cola) on soft dental materials and found that hardness of all the dental materials decreases when exposed to coca cola.

Based on the literature available it was found that physical properties of various polymers gets degraded when exposed to environmental conditions and may became unsuitable for some applications. This works had an attempt to investigate the effect of Hygrothermal conditions on the micro-hardness of the epoxy resin.

2. Experimentaion and Data Collection:

A. Setup

The setup consists of following main elements:

- Water Tank
- Heating Elements
- RTD Sensors
- Temperature Controllers
- Solid State Relays

A pictorial view of the setup is shown in the fig.1.



The experimental setup is being prepared in the lab using water storage and heating rods along with sensors. The aim of the experiment was to study the effects of environmental parameters moisture and heat (Hygrothermal) and percentage weight gain and micro-hardness were measured for the epoxy resin. The test matrix is as given in table I. Initially epoxy

resin samples were prepared and each sample was held in Hygrothermal conditions (i.e. some samples at room temperature, some at 45° C and some at 55° C) for pre-decided time periods with and without preloading and then tested for weight gain and micro-hardness

| Table I: Test Matrix Details | | | | | | | | |
|------------------------------|---------------------------|--|-----|-----|-------------|---------|--|--|
| Bath | Holding Time (days) | No. of samples pre stressed at%Load at failure (N) | | | | Total | | |
| Temperature (°C) | | Without load | 30% | 50% | 70 % | Samples | | |
| Room temperature | 3 | 1 | 1 | 1 | 1 | 4 | | |
| | 6 | 1 | 1 | 1 | 1 | 4 | | |
| | 9 | 1 | 1 | 1 | 1 | 4 | | |
| | 12 | 1 | 1 | 1 | 1 | 4 | | |
| 45°C | 3 | 1 | 1 | 1 | 1 | 4 | | |
| | 6 | 1 | 1 | 1 | 1 | 4 | | |
| | 9 | 1 | 1 | 1 | 1 | 4 | | |
| | 12 | 1 | 1 | 1 | 1 | 4 | | |
| 55°C | 3 | 1 | 1 | 1 | 1 | 4 | | |
| | 6 | 1 | 1 | 1 | 1 | 4 | | |
| | 9 | 1 | 1 | 1 | 1 | 4 | | |
| | 12 | 1 | 1 | 1 | 1 | 4 | | |

A. Procedure: After pre-decided immersion each sample was taken out and weight and then micro-hardness (VHN) of each sample was measured. The surface hardness of the different pile/interface materials was investigated by means of the Vickers hardness test. The Vickers hardness test has the flexibility to allow application of small loads, which is convenient for testing softer materials such as recycled plastic and FRP composites, and consists of pressing a standard diamond pyramid into the sample. The Vickers hardness number (HV) is related to the load applied and the area of the diamond pyramid indentation. Vickers hardness tests were performed in accordance with ASTM Standard E₃84-99 (ASTM 1999) [19].

Results and Discussion:

A. Percentage weight gain

The variation of percentage weight gain (wt) can be measured as:

$$W_T = \frac{W_t - W_0}{W_0} \times 100$$
(1)

W_t -is the total weight after time t

W_o-is the reference dry weight of the specimen before immersion in medium.

Percentage weight gain by different samples immersed in water at different temperatures with respect to time is shown in fig. 2.

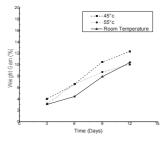


Fig. 2:Percentage weight gain in samples at different temperature with respect to time

The fig. 2 shows that with increase in time weight of the samples increases, this increase is due to the the increase in the moisture content present in the sample. Weight gain percentage initially increases with a steep slope and then this slope decreases at the saturation in the sample reached.

B. Hardness: The Vickers hardness test or the 136 degree diamond pyramid hardness test is a microindentation method. The indenter produces a square indentation, the diagonals of which are measured. Vickers's Hardness Number (VHN) can be found directly from the Vickers's Hardness Testing machine. In that machine first of all an indent was made on the surface of sample by applying force of 200 g with the help of indenter for dwell time of 20s. Average of 3 readings in each sample at different places was taken. A screenshot

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of the micro-hardness test is shown in fig.3.



Fig. 3: VHN directly found from QUANTIMET micro hardness analysis software

The average VHN for different samples immersed in water at different temperatures with respect to time is shown in Table II and the results were represented in the fig.4 –fig. 6.

| Table II: V | ickers's Hardr | ness Number o | f epoxy matrix | with respect t | o time | | |
|---------------------|---------------------------|-----------------|----------------|----------------|----------------|--|--|
| Bath | Holding Time (days) | VHN | | | | | |
| Temperature (°C) | | Without load | 30% loading | 50% loading | 70% loading | | |
| Room temperature | 0 | 19.64 | 17.98 | 18.98 | 19.61 | | |
| | 3 | 17.39 | 16.26 | 15.24 | 14.52 | | |
| | 6 | 14.84 | 12.04 | 11.95 | 11.45 | | |
| | 9 | 12.43 | 10.69 | 11.26 | 11.81 | | |
| | 12 | 11.08 | 10.09 | 10.69 | 10.46 | | |
| 45°C | 0 | 18.91 | 17.34 | 18.30 | 18.94 | | |
| | 3 | 13.04 | 15.68 | 14.50 | 16.77 | | |
| | 6 | 11.23 | 11.68 | 11.65 | 14.31 | | |
| | 9 | 11.39 | 10.31 | 10.86 | 11.29 | | |
| | 12 | 14.16 | 12.10 | 12.32 | 14.31 | | |
| ₅₅ ℃ | 0 | 17.90 | 16.39 | 17.29 | 17.87 | | |
| | 3 | 15.45 | 14.44 | 13.70 | 12.96 | | |
| | 6 | 12.12 | 11.04 | 11.01 | 10.94 | | |
| | 9 | 11.40 | 09.74 | 10.02 | 10.76 | | |
| | 12 | 10.11 | 09.70 | 09.45 | 09.21 | | |

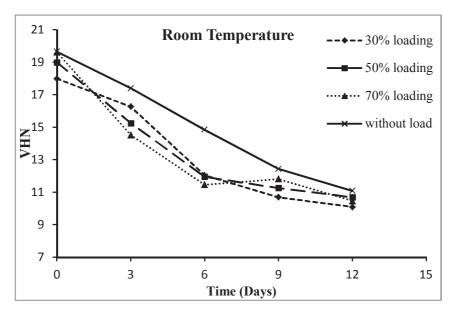


Fig:4 VHN of diffrent preloaded samples with respect to time at Room temperature

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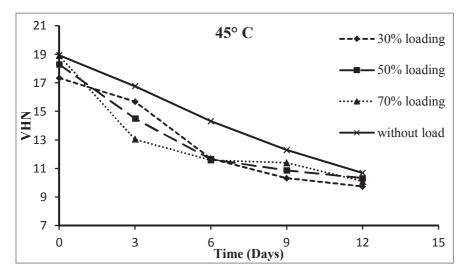


Fig. 5:VHN of diffrent preloaded samples with respect to time at 45°Ctemperature

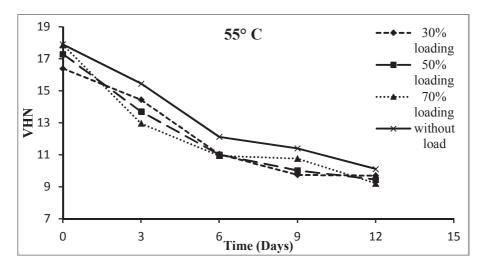


FIG:6 VHN of diffrent preloaded samples with respect to time at 55°C temperature

Above graphs fig. 4 to fig 6 are showing trend of change in Vickers's Hardness Number with respect to time. It is observed from the graphs that the VHN is constantly decreasing with increasing time period. Change in VHN in different preloaded samples was also significant with respect to time. The reason for the decrease in VHN with respect to time may be the Hygrothermal environment. The epoxy immerged in the water at elevated temperatures may get soften due to temperature and moisture. Pores of epoxy will loosen which gives way to moisture due to which the

sandwich structure getting softer day by day and hardness decreases.

Fig. 7 shows the relation of percent decrease in VHN and percentage weight gain for each loading samples at room temperature, 45°C and 55°C. Graph shows that for every percent of loading samples with increase in time percentage weight gain increases whereas VHN decreases. Due to increse in weight gain epoxy becomes softer which leads decrease in VHN.

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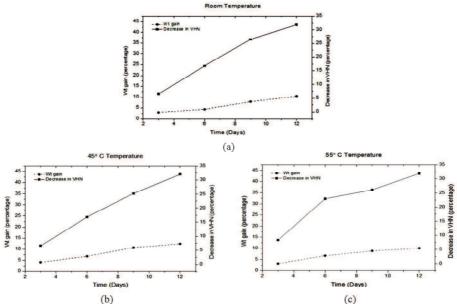


Fig. 7: Relation of percentage decrease in VHN and percentage weight gain with time

Conclusion: The percentage weight gain showed an increasing trend with time at the starting and when saturation reached this increase is very small. Moisture content leads to the swelling of the epoxy

surface and hence the decrease in micro-hardness. It was also concluded that the decrease in the micro-hardness for the preloaded samples is significantly more as compared to the unloaded sample.

References:

- 1. www.wikipedia.com/Composite material.html
- 2. www.structsource.com/pdf/composite.pdf
- 3. www.structsource.com/pdf/composite.pdf
- 4. Springer, G.S. (ED.), Environmental effect on composite materials, v₃, technical pub. Co., Lancaster, PA, 1988.
- 5. J. Zhou and J.P. Lucas, journal of thermoplastic composite materials v9(4), pp 316, 1996.
- 6. J.P. Lucas and J. Zhou, journal of materials, pp 37-40, 1993.
- 7. J.P. Lucas and B.C. Odergard, Advances in thermoplastic composites, STP1044, Newaz G., ed. Pp 231, 1989.
- 8. C.S. Wang and G.C. Chang, the effect of moisture absorption on composite laminates, composite structures, v2,pp 260, 1983.
- 9. O.K. Joshi, the effect of moisture on the shear properties of carbon fiber composites, composite structures, v 14, pp 196, 1983.
- 10. E.G. Wolf, sample journal, v 29 (3), pp 11, 1984.
- C. Deneve and J. L. Shanahan, polymer, v 34, pp 5099, 1993.
- 11. M.J. Adamson, journal of materials Science, v 15, pp 1736, 1980.
- 12. A.B. Strong, Effects of thermal changes on

- polymers, Plastic Materials and Processing, Prentice Hall: USA. pp. 90-96, 2000.
- 13. J. Decelle, N. Huet, and V. Bellenger, Oxidation induced shrinkage for thermally aged epoxy networks, Polymer Degradation and Stability, v 81(2) pp 239-248, 2003.
- 14. F. Awaja, B. Arhatari, K. Wiesauer, E. Leiss and D. Stifter, An investigation of the accelerated thermal degradation of different epoxy resin composites using X-ray microcomputed tomography and optical coherence tomography, Polymer Degradation and Stability, 94(10), p p 1814-1824, 2009.
- 15. V. Bellenger, J. Decelle, and N. Huet, Ageing of a carbon epoxy composite for aeronautic applications, Composites Part B: Engineering, 36(3), pp. 189-194, 2005.
- 16. F.M. Taib, Z.A. Ghani and D. Mohamad, Effect of home bleaching agents on the hardness and surface roughness of resin composites, Arch Orofac Science, v8(1), pp 34-40, 2013.
- 17. www.google.com/url?sa=t&rct =j&q=&esrc= s&source=web&cd =6&cad=rja & ved =oCE 4Q FjAF&url=http%3A%2F %2Fwww.uib.no%2Food% 2FForskning %2 FDiv _ pres % 2F

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- 18. Dentsply_Anne_Linn_o4.pdf&ei= Wd_MUrufIY rrAee64 HgDQ &usg= AF Qj CNF sPBjFTk eseOt7pm q7KLnOXgVBQA
- **19.** www.fhwa.dot.gov/publications/research/infrastr ucture/structures/04043/04043.pdf

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Assistant Professor, Assistant Professor, Mechanical Engineering Department, Baddi University, Baddi (H.P.)-173205, khush2k3@yahoo.com sushma.me@gmail.com, jimmymehta.chd@gmail.com

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