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An Experimental Investigation of Glass Transition Temperature of Composite Materials Using Bending Test

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Abstract: In this study, the bending test is used to investigate the glass transition temperature for epoxy reinforced with three types of fibers, fiberglass, Kevlar and synthetic wool, these materials have a wide used in many application which they are used composite materials. The glass transition temperature can be measured at the point of inflection for the curve of variation of the deflection and temperature. The results show that, the glass transition temperature is affected by the type of the reinforcement of the composites. On the other hand, the glass transition temperature of the wool composite is higher than the other.

Key words: Glass transition, composite materials, bending test.

1. Introduction

The glass transition of a polymer matrix composite is a temperature-induced change in the matrix material from the glassy to the rubbery state during heating or from a rubber to a glass during cooling. A change in matrix stiffness of two to three orders of magnitude occurs during the glass transition, due to the onset or freezing out of long range molecular mobility of the polymer chains. The temperature at which the glass transition occurs is a function of the molecular architecture and crosslink density of the polymer chains, but it is also dependent on the heating or cooling rate used in the measurement, and on test frequency if a dynamic mechanical technique is employed. In addition to the change in stiffness, the glass transition is marked by a change in the heat capacity and the coefficient of thermal expansion of the material, and so has at least some characteristics of a second order thermodynamic transition. The glass transition is frequently characterized by a glass transition temperature (T_g), but since the transition often occurs over a broad temperature range, the use of a single temperature to characterize it may give rise to some confusion. The experimental technique used to obtain the T_g must be described in detail, especially temperature scanning rate and frequency used. The method by which T_g is calculated from the data must also be clearly stated. Reported T_g may reflect onset of the glass transition or midpoint temperature depending on the data reduction method [1].

Upon exposure to high humidity environments, polymer matrices will absorb environmental moisture and be plasticized by it. One effect of this plasticization is the depression of T_g, frequently by a significant amount. A highly cross linked resin (one based for instance on a tetra functional epoxide such as TGMDA (tetraglycidyl methylene dianiline)) may have a high initial T_g, but it may be depressed more strongly than that in a less highly cross linked system. Measurement of the T_g in a composite material plasticized by absorbed moisture poses some difficult experimental challenges. Heating the test specimen as required by the measurement will drive off at least some of the

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absorbed moisture, thereby affecting the measured properties.

Due to the decrease in matrix stiffness, that occurs at the glass transition and to the low strength of these polymer matrices in the rubbery state, the matrix can no longer function effectively to transfer load to the fibers or suppress fiber buckling above the glass transition. T_g is, therefore, frequently used to define the upper use temperature of a composite material, although the time-dependent properties of the material such as creep compliance may be more sensitive to temperature within the glass transition range than are the quasi-static mechanical properties. A safety margin of 50 °F (28 °C) between the T_g and the MOL (material operational limit) has been proposed for epoxy matrix composites [2]. This approach is useful for initially estimating the MOL, or for verifying a previously chosen MOL. However, since glass transition frequently occurs over a temperature range, and the measured value of T_g is highly dependent on method, supplemental mechanical property tests should be considered, particularly for new material systems.

Since the heat capacity of a composite material changes at the glass transition, DSC (differential scanning calorimetry) may be used to determine T_g. The glass transition is detected as a shift in the heat flow versus temperature curve. Many calorimeters are supplied with software which may be used to calculated T_g. Various studies were carried out on calculate T_g by using DSC technique [3-5].

TMA (thermo mechanical analysis) is another way to calculate T_g. Thermo mechanical techniques such as expansion, flexure, or penetration TMA may also be used to determine T_g. In expansion TMA, the coefficient of thermal expansion α is measured as a function of temperature. As noted above, α undergoes a change during the glass transition, and T_g is determined by the point of intersection of lines fit to the thermal expansion data above and below the glass transition range. Illustrates the specimen geometries and data reduction methods used for various TMA techniques. In flexural TMA, a rectangular specimen is loaded in bending and the dimensional change is measured as a function of temperature. Flexural TMA measurement of T_g is similar to HDT (heat distortion temperature) measurement, since in both cases the specimens are loaded in flexure. An HDT specimen may be a full-size flexural test specimen, and is loaded in three-point bending or as a cantilever beam. Displacement is measured as a function of temperature, and the HDT is the temperature at which the displacement reaches some predetermined value. Use of a full-size specimen minimizes moisture loss during the HDT test. DMA (dynamic mechanical analysis) is the most common method of characterizing the glass transition of organic matrix composites. There are several types of DMA which have been used with composites, including TPA (torsion pendulum analysis) and other resonant techniques, and forced oscillation measurements in tension, torsion and shear. These forced measurements are made using a number of DMA instruments, manufactured by DuPont, Perkin Elmer, Polymer Laboratory, Rheumatics, TA Instruments and others [6]. Kaj, et al. [1] studied the glass transition temperature (T_g) for two lattices with different styrene/butadiene compositions, was determined by the thermal SPM (scanning probe microscopy) probe resonance frequency method. The results were compared with the T_g values obtained by DSC, DMA, PR (process rheometer) and TMA measurements. The T_g values detected by the thermal SPM method agreed well with the T_g values, obtained by DSC and calculated by the Fox-Flory equation. DMA, on the other hand, showed a significantly higher T_g value for both lattices than those obtained from theoretical calculations.

Gracia-Fernández, et al. [7] studied the measurements of the glass transition temperature obtained by DMA and TMDSC (temperature modulated differential scanning calorimetry) are reported to be different. This was mainly attributed to the frequency effect. To obtain comparable

measurements, experimental conditions were studied. Although some experimental issues had to be fixed, it was possible to apply the same frequency and temperature using quasi-isothermal conditions in both instruments. The method was tested with three different polymer samples. It was then found that, when choosing a suitable response parameter, i.e., the phase angle, the dynamic glass transition temperature values obtained by both methods are practically the same. Mei and Chung [8] used the DC electrical resistance measurement to investigate the glass transition and melting behavior of carbon fiber reinforced nylon-6 composite. The electrical resistance exhibited temperature dependencies that were attributed to the matrix molecular movements associated with structural transitions. The electrical resistance was affected by the degree of crystallinity and the thermal oxidative degradation, which were governed by the thermal history. The resistance results are consistent with DSC results. The resistance is more sensitive to the glass transition than DSC.

2. Experimental Work

2.1 The Bending Test Device

The test rig used in this research is shown in Fig. 1. The main parts of the test rig are: 1—the main frame, 2—the fixture parts to support the specimens, 3—the dial gage to measure the deflection, 4—variac device to generate the heat, 5—oven surrounding the specimens, 6—hook to hold the loads, 7—thermocouples to measure the specimen temperature.

2.2 Specimen Preparation

The specimens of composite material in this study are mixture of two materials, epoxy (matrix) and glass fibers, Kevlar and wool which are acting as (reinforcement). The epoxy chosen in this study is a viscous liquid in room temperature and become a rigid state by adding the hardener. Fig. 2 shows the steps of specimen preparation. The hardener (KETANOX B182) which is a viscous diaphanous liquid added to the epoxy by a weight ratio of 0.02 of epoxy weight.

2.3 Preparation of Apparatus

(1) Apparatus

The apparatus shall be arranged so that the deflection of the midpoint of the specimen can be measured on a scale calibrated in 0.01 mm.

(2) Conditioning

Conditioning: Condition the test specimens at 23 ± 2 °C and 50 ± 5% relative humidity for not less than 40 h prior to test in accordance with procedure A of practice D618, for those tests where conditioning is required.

(3) Procedure

The test specimen is placed in the apparatus with its 13-mm dimension vertical:

- The temperature measuring device is placed, so that it extends to within 3.2 mm of the specimen but does not touch it;
- The load is adjusted;
- Five minutes after applying the load, adjust the scale of the gage so that the needle points to zero and start the heating. This waiting period may be omitted when testing materials that show no appreciable creep during the initial 5 min. The 5 min, waiting period is provided to compensate partially for the creep exhibited by several materials at room temperature when subjected to the prescribed fiber stress. That part of the creep which occurs in the initial 5 min. is usually a large fraction of that which occurs in the first 30 min;
- Record the temperature of the medium (at 0.010 in.) as the deflection temperature at which the bar has deflected 0.25 mm the specified fiber stress.

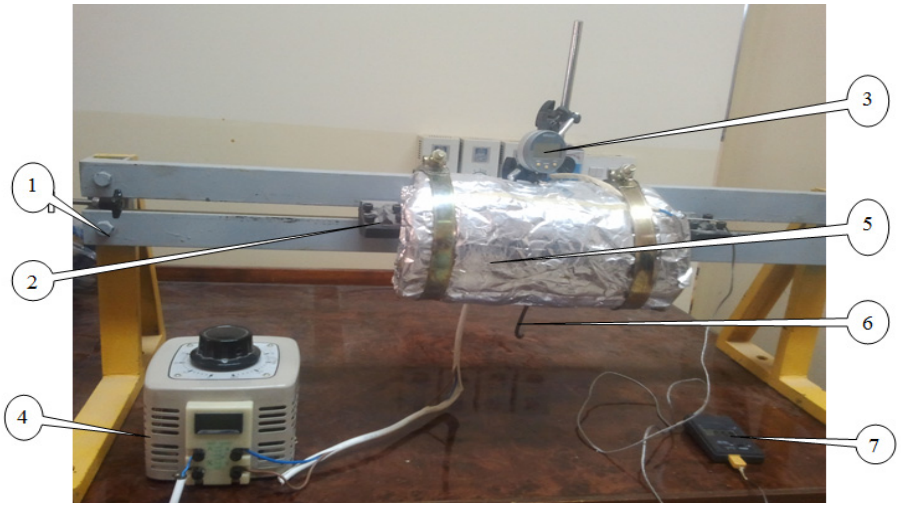

Fig. 1 Bending test rig.

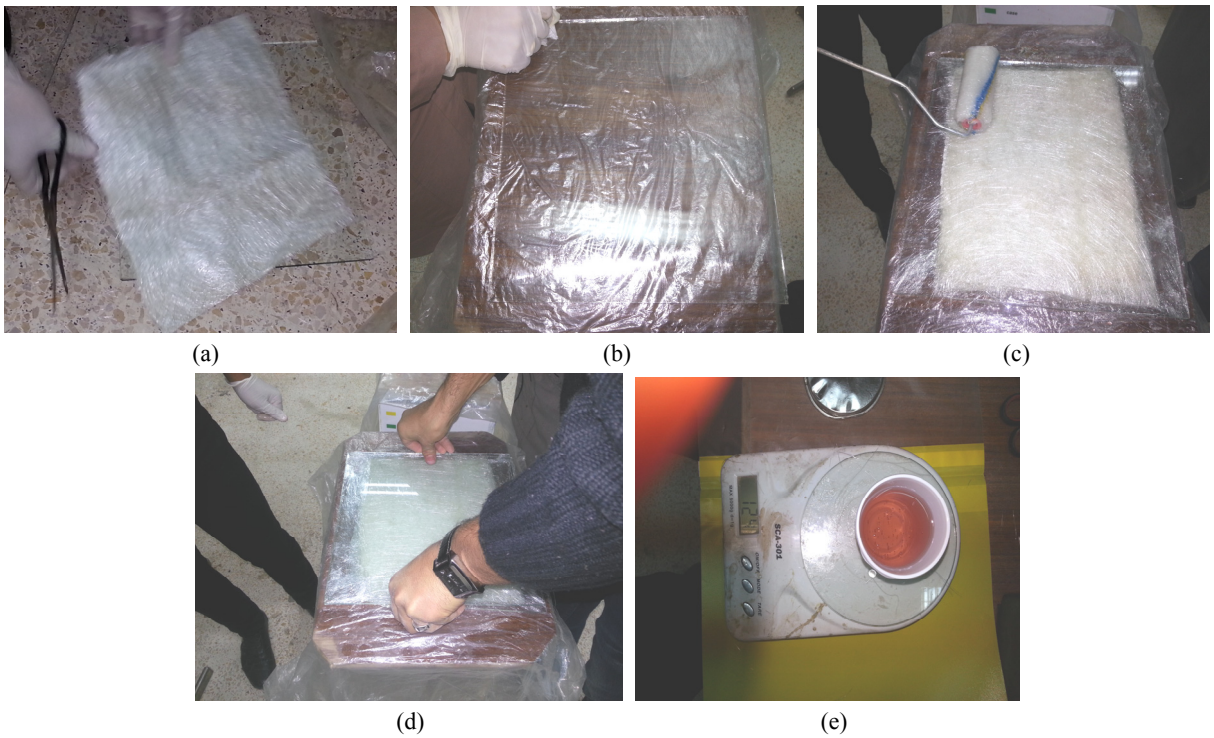

Fig. 2 The steps of samples preparation.

3. Results and Discussion

The experimental data of bending test under various temperatures for different types of fibers are shown in Figs. 3-5. In flexural test, a rectangular specimen is loaded in bending and displacement is measured as a function of temperature, the glass transition temperature (T_g), is the temperature at which the amorphous phase of the polymer is converted between rubbery and glassy states.

Fig. 3 shows the variations of deflection with temperature for (synthetic wool). It can be seen that, the glass transition temperature for the wool is nearly 70 °C.

Fig. 4 shows the variations of deflection with temperature for (fiberglass composite). It can be seen that, the glass transition temperature is nearly 50 °C.

Fig. 5 shows the variations of deflection with

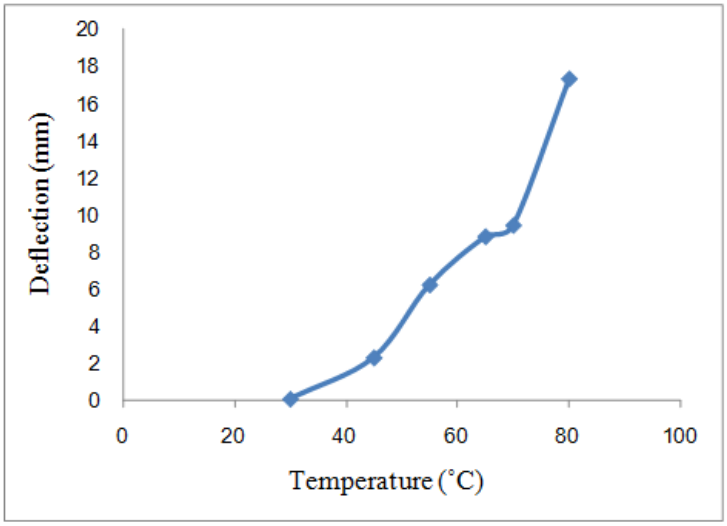

Fig. 3 The variations of deflection with temperature for synthetic wool composite.

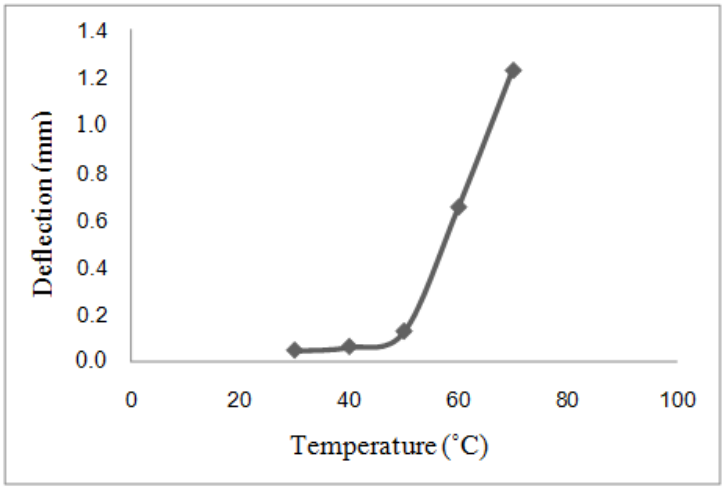

Fig. 4 The variations of deflection with temperature for fiberglass composite.

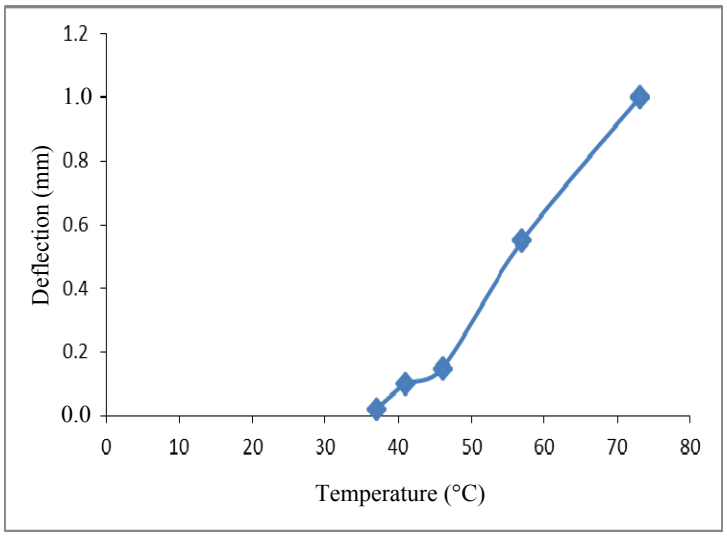

Fig. 5 The variations of deflection with temperature for Kevlar fibers composite.

temperature for (Kevlar fiber composite). It can be seen that, the glass transition temperature is nearly 46 °C.

The experimental results showed that the value of T_g for Kevlar fibers composite is less than that the values of the fiber glass composite and synthetic wool composite, this because the Kevlar fibers has higher mechanical properties, also have very heat resistant in high temperature compared with fiber glass and synthetic wool, This makes the Kevlar fibers play a role in maintaining the constituents and microstructure of composite when the heat is applied.

4. Conclusions

Based on the results, the glass transition temperature is affected by the type of the reinforcement fibers and the deflection of the Kevlar fibers composite is less than that of the fiber glass composite and wool fibers composites. In addition, the glass transition temperature of the wool composite is higher than the other.

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