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Wet lay-up technique for manufacturing of advanced laminated composites

Abstract

Incorporation of nanofillers into laminated composites is expected to produce a material with improved performance for many potential industrial applications. The focus of this work is to produce advanced composites by simple wet lay-up technique. The impact of adding three different nanofillers (multi-wall carbon nanotubes (MWCNT), silicon carbide (SiC) and aluminum oxide (Al₂O₃),) on the thermal and mechanical stability of woven Kevlar KM2Plus/epoxy composites was investigated. Silicon carbide and aluminum oxide were added at different weight percentages up to 3wt%, whereas the addition of multi-wall carbon nanotubes was limited to 1wt%. The mechanical behavior was evaluated through three-point bending tests. The thermal stability was examined using differential scanning calorimetry (DSC) and thermo-gravimetric analysis (TGA) techniques. The results revealed that simple wet lay-up technique is a recommended alternative method for production of thermostable composites with improved thermomechanical performance. The improvement in thermomechanical stability increases with the nanofiller content. However, MWCNT composites showed an optimum content value of 0.5wt % after which the mechanical performance starts to degrade due to agglomeration.

Keywords

Wet lay-up, Kevlar KM2Plus, Nanofillers, Composites, Thermo-mechanical properties

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1. Introduction

Nanomaterials are well known for their enhanced chemical activity, improved surface effects and exceptional physical properties and utilized in numerous commercial and engineering applications. Nanoparticle reinforced materials have drawn the attention of researchers due to their superior impact, mechanical, thermal and electrical characteristics. Low concentration of nanoparticle incorporation into polymers can produce composites with enhanced mechanical (strength and stiffness) and thermal properties without compromising density, toughness [1–5].

Kumar et al. [6] used aluminum oxide (Al_2O_3) nanofillers in urea-formaldehyde resin for the preparation of medium density fiber boards. Alumina fillers are found to be much efficient in improving the crosslinking densities with the resin material. These nanomaterials helped to increase the heat transfer throughout the matrix during the hot pressing. The internal bond strength of the composite increased by 33% after nanoparticle loading. Derradji et al. [7] investigated the influence of adding SiC to the phthalonitrile resin. The high transfer load of SiC filler improved the mechanical performance of the epoxy matrix such as flexural strength and microhardness. Flexural strength and modulus has improved by 66% and 55% respectively, by the incorporation of 20% of SiC into the epoxy matrix. The toughness of the composite rose from 0.27 MPa to 0.35 MPa by the incorporation of 15% SiC to the epoxy matrix. Friedrich et al. [8] Poornima et al. [9] have also investigated the effect of SiC on the mechanical properties of epoxy composites. Recently, many scientists and industries are focusing on carbon nanotubes (CNTs) due to their high aspect ratio and exceptional thermo-mechanical, physical and electrical properties. Furthermore, CNTs are most effective fillers used as reinforcement in fabrication of high strength and lightweight polymer composites [10]. Ciecierska et al. [11] reported the effect of CNT on the properties of epoxy matrix such as thermal conductivity, thermal stability and cross linking density. The results show that, the carbon nanotubes are good thermal conductors as they are evenly distributing the heat all over the matrix. Hence the thermal diffusivity is increases with the weight percentage of CNT. Kia Yang et al. [12] reported 9% increase in the bending modulus of the epoxy matrix by adding 0.6wt% MWCNT. Jiaoxia

Zhang et al. [13] discussed the presence of Van der Waals forces and unsaturated dangling bonds which act as interlocking between CNT and the epoxy matrix. This helps in the even desperation of CNT's and effective transfer of stress all over the matrix. They have reported an enhancement of 9% in the flexural strength by adding 1% MWCNT into epoxy matrix. Some researchers reported negative impact on addition of MWCNT into the epoxies. Dan Guo et al. [14] reported that, there is a reduction in value of micro hardness when the MWCNT content in the epoxy is less than 1%. Also there is reduction in flexural strength for the sample containing 2% nanotubes. This reduction is due to the presents of micro voids in the matrix, also structural non-homogeneity and weak interaction between nanotubes and the epoxy matrix. Similar research works were performed by zeiler et al. [15] and Reichert et al. [16] on hybrid composites.

Kevlar fabrics are aramid fibers having high-strength and high-modulus. The properties of Kevlar include high toughness, low weight, high flexibility and high specific strength. They have also superior impact-resistance-to-weight properties and mechanical properties [17]. Therefore, Kevlar fibers are widely used in the manufacturing of advanced composites [18,19]. Composite materials using various types of Kevlar have been widely used in several engineering applications. Kevlar is commercially available in various forms, providing manufacturing ease to fit wide range of applications. One of the most recently developed Kevlar is 767 aramid or Kevlar®, KM2Plus 600 denier. It has a broad range of potential applications due to its superior properties.

The bisphenol-A (epichlorohydrin) based epoxy material have a glass transition temperature of $\approx 60^\circ\text{C}$. It increases with isothermal curing temperature. Curing reaction starts $\approx 100^\circ\text{C}$ and curing time is dependent on the heating rate [20]. Since the material is a thermoset polymer it undergoes direct decomposition at higher temperatures. Li et al. [21] reported the decomposition of cured epoxy starts at 320°C . Ahmad et al. [22] have shown that the major volatile products during the decomposition of the epoxy are CO_2 , H_2O , CO, aliphatic hydrocarbons and some aromatic compounds etc. Jing Li et al. [23] have reported that the Kevlar fiber decomposes after 500°C .

Earlier studies on thermo-mechanical characterization of laminated Kevlar KM2Plus composites, to the best of authors' knowledge, have not been reported.

Thus, in the current study, influence of adding various weight percentages of SiC, Al₂O₃ and MWCNT nanofillers to woven KevlarKM2Plus/epoxy resin composites on the thermo-mechanical properties are investigated. Sonication technique is implemented to disperse nanofillers uniformly in the epoxy resin matrix. Wet layup method is used to fabricate the composite samples. Mechanical performance of nanofiller incorporated Kevlar/epoxy composite samples were evaluated by three point bending test. Moreover, the thermal properties of composite samples were analyzed using both TGA and DSC techniques in order to examine the impact of adding nanofillers to the thermal stability of composite samples prepared using wet lay-up technique.

2. Materials and methods

Kevlar KM2Plus: 767 aramid or Kevlar®, KM2Plus 600, a registered trademark of DuPont, was used for this study. It was purchased from JPS Composite Materials Corporation (Anderson South Carolina).

Epoxy Resin and Hardener: ARALDITE® AY105, a medium viscosity unmodified epoxy resin based on bisphenol-A, was used in this study. Araldite®, a registered trademark of Huntsman LLC (The Woodlands, Texas), was used to bound the layers of the Kevlar fabrics together. Low viscosity cycloaliphatic polyamine - Hardener HY 2962—Aradur 42—from Huntsman LLC (The Woodlands, Texas), was used in this work.

Nanofillers: Silicon carbide, SiC, with 15.3 µm particle size, aluminium oxide (Al₂O₃) with 50 µm particle size from PANADYNE Company (Warminster, Pennsylvania, USA) and multi-wall carbon nanotubes (MWCNT) from Nanolab (Newton, Massachusetts, USA) with a purity more than 85 wt%, were used in this work. SiC and Al₂O₃ nanofillers were added to the epoxy resin with three weight percentages of 1 wt%, 2 wt% and 3 wt%. MWCNT nanofillers were added with weight percentages of 0.25 wt%, 0.5 wt% and 1 wt%. The composite samples higher than these weight percentages were difficult to fabricate due to the increasing viscosity of the epoxy/composite solution and the increasing sedimentation rate of the nanofiller [24].

Fig. 1 shows the complete procedure of the wet lay-up technique. All stages (a-f) followed in the preparation of control sample (using solution A) and composite sample (using solution B) is presented. The control sample was prepared from ten woven layers of Kevlar KM2Plus stacked one on the top of the other at

0° angle. Hardener was added to the epoxy resin to bind the Kevlar piles, ensure and accelerate the curing process. The resin-hardener weight ratio was 5:1. The first Kevlar layer was placed on an aluminum plate, where the resin was distributed evenly over the Kevlar ply using a regular painting brush. Once the resin was spread, another layer was placed on the top of the first Kevlar pile at 0° angle. Then a wiper and a metallic roller were used to assure that the second ply is bounded to the first one and the air bubbles were eliminated completely from the resin and from the cavities between the two Kevlar layers. The same process was repeated for the rest of the layers. Once the layup of the specimen was completed, it was placed inside the hot press machine (Carver Inc., Indiana, USA) for curing under force of 4200 lb (18.68 KN)), temperature of 175 °C and time of 15 min.

In the preparation of the nanofiller incorporated samples, the same procedure applied in the preparing the control specimen was followed. Different nanofillers with different weight percentages were mixed to the epoxy resin. Sonication process (QSonica Q700) was applied for 15min to ensure the proper mixing and achieve homogeneous dispersion of the nanofillers within the resin. During the sonication process, heat is produced, where the temperature reached up to 60 °C. This makes the resin less viscous and ensures proper mixing and homogeneous dispersion of the nanofiller within the resin. The hardener HY 2962 — Aradur 42 was poured into the resin-nanofiller mixture and then stirred properly to ensure accelerated curing.

The following 4 groups of composite samples were prepared:

Sample group	Properties
1	10 layers of Kevlar/resin (without nanofillers)
2	10 layers of Kevlar/resin (with 1 wt%, 2 wt% and 3 wt% SiC) composites
3	10 layers of Kevlar/resin (with 1 wt%, 2 wt% and 3 wt% Al ₂ O ₃) composites
4	10 layers of Kevlar/resin (with 0.25 wt%, 0.5 wt% and 1 wt% MWCNT) composites

2.1. Microstructure analysis

Light microscopic images were taken using Olympus BX43 upright Microscope. SEM images were taken using QUANTA 250 FEG.

Fig. 1. Sol A with epoxy and hardener only and sol B with epoxy hardener with nanofiller content. a-f represents different steps in the Kevlar/epoxy/composite preparation.

2.2. Mechanical tests

a. Flexural test

Flexural modulus of elasticity and flexural strength were obtained from the three point bending test using Material Testing System (MTS), model number 20/MH. Flexural strength of the composites is dependent on both tensile and compressive strengths, which vary directly with the interlaminar shear strength. However, the flexural modulus represents the resistance of the bending deformation. Five specimens from each case were tested and the average values were recorded. The load continued up to the maximum value and beyond. The specimen was loaded slowly under a constant overhead speed of 5 mm/min. Flexural modulus (E_{bend}) and flexural strength (σ_{fM}) were calculated according to ASTM D790-10 standard using equations (1) and (2) [11,25]:

$$E_{\text{bend}} = \frac{L^3 \times m}{4 \times b \times d^3} \quad (1)$$

$$\sigma_{\text{fM}} = \frac{3 \times P_{\text{max}} \times L}{2 \times b \times d^2} \quad (2)$$

where L , b and d (mm) are the support span, specimen width and specimen depth respectively. m is the slope of the initial straight-line portion of the load-deflection curve, (N/mm). P_{max} is the peak load (N).

2.3. Thermal tests

a. Thermal gravimetric analysis (TGA)

Thermal Gravimetric Analysis (TGA) analysis of the epoxy resin, hardener and composite samples were carried out using Thermo-gravimetric Analyzer TGA Q50 V20.10 Build 36 Model (TA Instruments, Water LLC - New Castle, Delaware). Nitrogen atmosphere was maintained with a sample purge flow of 60 ml/min. All composite samples are heated up to 800 °C with a heating rate of 10 °C/min. TGA was conducted to investigate the thermal stability of control and nanocomposite samples [26–29].

b. Differential scanning calorimetry (DSC)

Differential Scanning Calorimetry (DSC) analysis of the epoxy resin, hardener and composite samples

were performed using Differential Scanning Calorimeter (DSC) DSC-60 (Shimadzu Corporation – Analytical & Measuring Instruments Division – Kyoto – Japan. Nitrogen atmosphere was maintained with a sample purge flow of 50 ml/min. All composite samples are heated up to 600° with a heating rate of 10 °C/min. Glass transition temperature, curing temperature and decomposition can be obtained from DSC data.

3. Results and discussion

a. Microstructure

The prepared Kevlar/epoxy composite sheets were examined using a light microscope (Fig. 2) and SEM (Fig. 3) of control and 0.5%MWCNT composite sheets. We can observe that epoxy is spread uniformly all along the Kevlar layers and there are very few defective points on the surface.

The distribution of MWCNT is uniform all along the surface of Kevlar, and they occupy on the voids between the woven Kevlar fibers. MWCNT forms a

crosslinked network around the Kevlar layers which makes them mechanically strong when compared with the control sample.

b. Flexural test

The representatives of control and composite sample were obtained from the MTS machine data. It is observed from load - deflection curves that all curves have the same trend regardless the nanofiller type and content. Fig. 4a represents the load-deflection curves for control sample and Al₂O₃ composite samples. These curves comprise of three distinct regions. In the first region, there is a linear relationship between the load and the deflection. In the 2nd stage, the curves deviate from their linearity and the relationship becomes nonlinear with a decreasing slop till the curves reach a plateau, in the third stage. No full separation was observed and the test was terminated after certain deflection before the sample touches the base of the fixture. The five specimen's curves are almost overlapping. This repeatability in the results illustrates the effectiveness of the wet lay-

Fig. 2. Microscopic images (2× magnification) of control and 0.5%MWCNT composite sheets.

Fig. 3. SEM images of control (80× magnification) and 0.5%MWCNT (100× magnification) composites.

Fig. 4. a. Load –Deflection curves for control and Al_2O_3 composite sample. b. cross-sectional view after 3 point bending test

up technique in producing composites. Also, from the cross sectional view from Fig. 4b, it is observed that there is no delamination of the Kevlar layers even after the maximum load is applied.

The average slope of the linear part of the load - deflection graph was obtained and hence E_{bend} and σ_{fM} were calculated from equations (1) and (2) respectively.

The control sample (Kevlar/epoxy) has achieved E_{bend} of ≈ 9 GPa and $\sigma_{fM} = 141$ MPa. The effect of adding nanofillers on E_{bend} and σ_{fM} of the Kevlar/epoxy composites is demonstrated in Fig. 5a and b, respectively. It is evident that the control sample has the lowest values if compared to that of nanofiller incorporated composites. Moreover, for Sic and Al_2O_3 samples, increase in the nanofiller weight percentage has significantly increased the flexural properties of the composite samples. This can be interpreted as, when the nanofiller content increases, the heat distribution and load sharing in the epoxy matrix increases, thus enhances the flexural strength of the composites [7,11]. Also the large surface area of the nanofillers and their

roughness improves the flexural modulus by enhancing the interaction between Kevlar layers and epoxy matrix. In addition, it improves the chemical interaction between the nanofillers and the matrix. In the previous work on Epoxy/MWCNT composites, the reported flexural modulus enhancement is up to 10% by addition of MWCNT into the epoxies [12,13]. In this work, Kevlar KM2Plus samples reinforced with 0.5% MWCNT recorded the highest flexural modulus among all the samples (33.3% above the control sample). This improvement may be due to the bridging of CNT between the epoxy and the woven Kevlar sheets. The dangling bonds in CNT's with the epoxy matrix make them stronger when compared to other fillers resulting in the highest σ_{fM} and E_{bend} among all samples [12,13]. It is also noticed that the flexural properties reduces at carbon nanotube content of 1%. This is likely due to carbon nanotubes agglomeration. This local agglomeration creates local high stress points which could be failure sources [13].

Valenca et al. [30] have studied the flexural properties of Kevlar/epoxy composites. Maximum strength

Fig. 5. a. Flexural modulus of elasticity of all composite samples. b. Flexural strength of all composite samples.

of 61.59 (± 2.93) MPa and flexural modulus of 5.68 (± 0.93) GPa have been reported. The results achieved in the current work, for Kevlar KM2Plus ($\sigma_{fM} = 141$ MPa and $E_{bend} = 9$ GPa), are much higher than their results. Similar studies are carried out by Iman et al., [7] in woven Kevlar AK502/epoxy laminated composites are also in agreement with the results of the current work. They showed that the bending stiffness of the composites reinforced with 0.5 wt% MWCNT has improved up to 15% at room temperature. Also, they have achieved 13% improvement by the incorporation of 0.3 wt% MWCNTs at low temperature. This enhancement is due to the interlocking of MWCNT between the Kevlar fibers and epoxy matrix. Therefore, their observation consolidates the results reported in this study, where the addition of nanofillers has significantly increased both the modulus of elasticity (up to 36% above the control sample) and the flexural strength (up to 50% above the control sample). Similarly, the conclusions drawn by Gojny et al. [31] are in agreement with the results of this work, where strength and strain to failure were improved by incorporating 0.1 wt% nanotube to the epoxy matrix.

c. Thermo-gravimetric analysis (TGA)

Thermo-gravimetric analysis (TGA) was performed for the matrix (epoxy resin and hardener) and all the Kevlar composite samples to trace the degradation temperatures and thermal stability of the composites [32].

TGA curves for control and composites have the same trend regardless the type and content of the nanofiller. Fig. 6 shows the TGA curve for the matrix, control sample and Kevlar/epoxy composite enhanced with 0.5 wt% MWCNT samples, respectively, as representative curves along with TGA curves for different MWCNT concentrations (0.25%, 0.5% and 1%).

The matrix TGA curve shows one step/degradation stage, where the initial degradation temperature (T_i) is about 315 °C. At this temperature, the epoxy material starts decomposition and almost all the matrix material is decomposed at 450 °C. The same trend can be seen in the work of Fan-longjin and Soo-jin Park [33]. The TGA curves of the control sample and all composites samples have two stages of degradation. Table 1 Shows the 1st and 2nd degradation temperatures of all composite samples. The first initiation temperature of degradation (T_i) of all composites varies within 315 °C–320 °C. This range of variation is limited and comparable with the T_i (315 °C), of the neat matrix. The second stage of degradation temperature of the all composite samples varies from 532 °C to 537 °C, which represents the T_i of degradation of the Kevlar. It has also been reported that all the Kevlar materials have a decomposition temperature in range 520°C–565 °C [34]. Jinchun Fan et al. [35] have shown that when MWCNT are added to the Kevlar there was only a shift of 10 °C (from 565 °C to 575 °C) in the decomposition temperature. This value aligned with the value obtained in this study for the used new

Fig. 6. TGA curves for matrix, control and MWCNT composite samples.

Table 1

1st and 2nd degradation temperatures obtained from TGA data and glass transition temperature values from DSC curves for all composites.

Composite sample	1 st Degradation temperature (°C)	2 nd degradation temperature (°C)	Glass transition temperature (°C)
Matrix	320	-	40.2
Control	315	532	60.2
1% SiC	317	533	61.3
2% SiC	316	537	61.2
3% SiC	318	535	60.8
1% Al ₂ O ₃	316	533	61.2
2% Al ₂ O ₃	320	532	60.5
3% Al ₂ O ₃	318	537	60.4
0.25% MWCNT	317	533	60.6
0.5% MWCNT	320	534	60.4
1% MWCNT	319	535	60.3

Kevlar type. Hence, the addition of the nanofillers increases T_i of the composites minutely and improves the thermal stability of the samples as well.

d. Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) for the matrix (and all the Kevlar/epoxy laminated composites) were conducted to determine the glass transition temperature (T_g), curing temperature (T_c), decomposition temperature (T_d) [36–39]. Fig. 7 presents representative DSC curves for the epoxy matrix, control sample and 0.5 wt% MWCNT reinforced Kevlar/epoxy composite samples. The DSC curves for the prepared control and composites samples are almost with the identical trend. The investigated

composites constituents experience direct decomposition without melting. Hence, the crystallinity for this type of composites will be impossible to be defined. Therefore, thermal stability of the matrix is determined by the thermal stability of all composites. The T_g characterizes the range of temperatures over which this glass transition takes place. The T_g temperature is determined for all the samples, where the results were zoomed at the expected region of T_g . Fig. 7 demonstrates the glass transition and decomposition temperatures of the matrix, control sample and the 0.5 wt % MWCNT composite as representative results. The initial T_g for the matrix and composite samples is $\approx 40^\circ\text{C}$. It is the point at which the material starts to change its phase (point a). The T_g may also be taken at the midpoint between the initial and final temperatures ($\approx 60^\circ\text{C}$) for all composite samples (point b and c). The glass transition temperature of all composite samples are listed in Table 1. It is evident from the curves of neat epoxy and the composites that, after crosslinking of the epoxy (after hot pressing in elevated temperatures), the T_g shifts to higher values. This agrees with the results of Behrouz et al. In addition, the variations between all composites values are negligible. This shows that the T_g of the composites samples are independent of nanofiller concentration. The observed slight variation in the T_g for all the samples might be due to the curing process or the change of heat flow due to the inclusion of the fillers. For the matrix sample, there is one peak at 100°C , which corresponds to the curing of the epoxy resin. Since the hot pressing temperature is beyond 100°C , all the Kevlar/epoxy composite samples are already

Fig. 7. DSC curves for matrix, control and 0.5 wt% MWCNT composite samples.

cured and thus the corresponding peaks are not appeared on the DSC curves. The DSC test has continued to enable the decomposition of the sample to be observed. When temperature exceeded 320 °C, there is a large peak for the matrix sample and small peaks observed for all the other Kevlar/epoxy composites. These peaks correspond to the decomposition of epoxy material. Almost all the material is decomposed at ≈ 375 °C (points d, e and f). The small differences in peak temperatures values depend on the epoxy content in the sample. These results agree closely with the data provided in the TGA analysis, where the first stage of decomposition is obtained at a temperature of 315 °C.

4. Conclusions

Wet lay-up technique is employed successfully for the production of Kevlar KM2Plus/epoxy laminated composites reinforced with different nanofillers. Uniform dispersion of nanofiller in the epoxy matrix is achieved through the sonication process. When compared with the Kevlar KM2Plus/epoxy control sample, the addition of nanofillers has significantly enhanced the mechanical performance of the Kevlar/epoxy composites. The findings of this research have also been compared with the results of Kevlar/epoxy composites available in the literature. The obtained strength values showed an increasing trend with nanofiller percentage. The composite sample with 0.5 wt% MWCNT showed the best mechanical properties. However there is a reduction in the strength for the 1 wt % MWCNT due to agglomeration. The inclusion of the nanofillers has enhanced the thermal stability of the composites. Both T_d and T_g are

increased minutely. The results prove that, the wet lay-up technique is an alternative recommended technique in producing good quality and advanced composite sheets with highly consistent results.

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