탄소 섬유 보강이 IPN 기반 타원형 리프스프링 성능에 미치는 영향

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Impact of Carbon Fiber Reinforcement on the Performance of IPN-Based Elliptical Leaf Springs

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Abstract: In recent decades, composites have been touted as potential replacement material across various domains due to their higher weight-to-stiffness ratio compared to metallic materials. This research focuses on utilizing carbon fiber as the primary reinforcement in conjunction with different matrix blends such as epoxy with vinyl ester (EV), vinyl ester with polyurethane (VP), and epoxy with polyurethane (EP) to manufacture elliptical leaf springs. To validate the thermal and physical properties of the fabricated leaf spring, tests like differential scanning calorimetry (DSC), Thermogravimetric analysis (TG), load-deflection and cyclic loading (fatigue) analyses were conducted. Among these tests, combinations of carbon-reinforced epoxy with vinyl ester (CEV) demonstrated higher compression test results with a slight reduction in displacement. Similarly, carbon-reinforced vinyl ester with polyurethane (CVP) exhibited better performance in cyclic loading compared to carbon fiber-reinforced epoxy with polyurethane (CEP). All obtained results were thoroughly compared to determine the most suitable implementation in the automotive field.

Keywords: carbon, epoxy, vinyl ester, polyurethane, leaf spring, physical tests.

Introduction

Leaf spring normally considered as one of the main structure used towards most of the suspension units in all sector automobile vehicles. Typically it is called as the laminated spring, carriage spring sometimes which may be called as the elliptical spring or cart spring. Even it is applications are believed as the trustworthy in medieval times.¹⁻² As well, the main persistence of the leaf spring is to completely or partially arrest the vibrations and shocks while travelling on the uneven roads. Recent technologies adopted by most of the manufacturers are indeed in urge to find out the most versatile material that upkeep all the characteristics of the metallic materials. In that context,

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material science engineers are thriving themselves to find out the newer combinations like properly tailoring the metal alloys with various proportionate, along with that adopting the right technology in ceramics and polymeric materials.³⁻⁴ Nevertheless, the newer findings in their particular areas like aerospace and underwater transportation pipes are becoming as the pertinent factor in growing upcoming industrial sectors. Nowadays composite leaf springs are very wisely accepted in most of the automobile sectors as the proper replacement of the metallic leaf springs as it provides the superior damping and corrosion resistance than the metallic leaf springs.⁵⁻⁶ In the present scenario, the composite leaf springs are accepted and adopted in light weight vehicles, heavy tanker, cars and even in some of the vibrating absorbing machineries. Since it possesses the significant riding comfort ability, most of the automobile manufacturers are partially adopted the concept of utilizing the composite leaf springs in forthcoming manufactured vehicles.7-8 Additionally, all the springs irrespective of its material characteristics, primarily designed to absorb the vibrating forces and successively intend to give back the retrieve energy. Apart from that, its prime and predominant property is that, to absorb the lateral load, braking energy, driving force along with that absorbing the shocks during travelling in bumps. Most probably the ends are considered as the guiding member of the leaf spring towards defined path to absorb the energy. The common manufacturing methods of leaf springs are that, filament winding process, compression moulding process along with that pultrusion and hand lay-up processes are also used.9 The exact definition or explanation of the composite materials is that, combination of more than two materials formed with the purpose of extract the specific quality of the individual components. To exactly meet out the uniqueness of the composite materials, many researchers are repeatedly trying out to explicitly identify the newer materials with help of the advanced manufacturing technologies, upon adopting such an processes the unique chemical, physical and mechanical properties of the individual components are boldly extracted.¹⁰ The subdue property of the composite materials are that, high stiffness to weight ratio and strength to weight ratios, due to this advert properties it is widely considered as the best alterative material in most of the growing industrial sectors, particularly in the area of avionics where the weight reduction is considered as the most predominant factor. Furthermore, composite materials can be manufactured with least time frame with average man power; also their parts are easily produced without any heavy machining and surface cleaning operations.11 Besides, it also acts as the very good dampers and

prevalent to absorb all set of noises and high frequencies, due to this inherent properties leading customer goods and most of the sport utility products like tennis racquets and golf clubs are mass-produced with assistance of the composite materials. Normally the combination of fibers and matrix materials has been giving the fiber reinforced plastic materials (FRPs). In that manufacturing sequence, the commonly accepted fiber materials are glass, carbon, Kevlar and in some extend natural fibers are also playing the leading role in most of the industrial sectors, where the fibers significantly gives the maximum bearable strength to the composites.¹² Conversely, the matrix materials are expected to give the complete support structure to the composites and it protects the fiber materials from corrosion and chemical attacks. In that sequence, researchers are often try to use the fiber materials as glass fiber and carbon fibers to the maximum extend with different combinations of matrix materials.13 In recent days carbon fibers have attracted most of the researchers because of their better interfacial attachment with the matrix materials even though the cost is slighter higher than the price of glass fibers. Likewise, matrix material also plays the effective role in the formation of FRPs since it gives the structure to the FRPs. The commonly used matrix materials are vinyl ester, epoxy, polyester and polyurethane as it is frequently available from the open market. From the matrix (resin) perspective, every matrix has its own unique set of advantages and disadvantages while it is been used in customer point of view.14

In this specific research study, the concept of interpenetrating polymer networks (IPNs) has been initiated. The commonly available matrix materials vinylester, epoxy and polyurethane have been blended with each other; with some proportionate in the view to extract the specific quality of the individual resin. While blending the resin with each other, each component (resin) cure separately with its own, and both blend matrices have create firm entanglement with other. In this work, Carbon fibers have been chosen as the reinforcement material along with the various proportionate of epoxy with vinyl ester, epoxy with polyurethane and vinyl ester with polyurethane. However, to completely expose the physical characteristics of the blends, leaf springs have been fabricated with various proportionate of blends and their corresponding tests like compression tests and cyclic loading (fatigue) were performed in the intend to reduce the weight of the entire conventional (metallic) leaf springs as well as to extract the specific quality of the individual polymers.15

Experimental

Materials and Techniques. Materials: The prime component epoxy resin and their respective hardeners, as well another blending component vinyl ester resin and their corresponding hardener (MEKP) then another important blending agent polyurethane their respective curative agent (MOCA) were procured from sakthi fibers, Chennai. Their successive technical data's were given in the Table 1. In the same tactic, carbon fibers with an aerial density of 350 GSM was brought from another shop Jayantha fibre, Chennai as presented in the Figure 1.^{16,17}

Matrix Preparation: Initially, in the matrix preparation works, the combination of blends were justified as per given Table 2. During the initial process to form the combination of CVP, the vinyl ester with weight of 70% was taken and their corresponding hardener MEKP (Methyl Ethyl Ketone Peroxide -2%), promotor (Cobalt Naphthenate (CoNap) – 1.5%), catalyst (Dimethylaniline -1%) also was added into it.¹⁸ Similarly the polyurethane (30% wt.) and their corresponding curative agent MOCA ((Methylene-bis (ortho-chloroaniline) -7%) were also added and stirred thoroughly. Completion of above processes, the already hardener added resins were mixed and stirred thoroughly with help of the sonication device. As such processes done in the CVP, similarly in the CEP formation also, epoxy (70% wt) with their successive hardener (Triethylenetetramine (TETA) - 7%) and polyurethane (30% wt.) were added and stirred to form the CEP. At last the final combination of epoxy (70% wt.) and vinyl ester (30% wt.) were also blended and thoroughly sonicated to form the CEV.19,20

Mandrel Fabrication: The Figure 2 illustrates the mandrel setup, primarily which was utilized to form the elliptical spring; this mould was absolutely fabricated of wood.



Figure 1. Carbon fiber.

Table 2. IPN – Blend by Means of	Reinforcement	Mixtures
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Codes	IPN - Blending Mixture	Fiber Reinforcement
CVP	Vinyl ester + Polyurethane (VP)	Carbon
CEP	Epoxy + Polyurethane (EP)	Carbon
CEV	Epoxy + Vinyl ester (EV)	Carbon



Figure 2. Wooden mould.

Production Technique of Composite Elliptical Spring: In order to make the elliptical lead spring, the mould which was made up of wood with fine surface finish were thoroughly cleaned with any kind of solvent. After completion the cleaning processes, the polyvinyl alcohol (PVA) was taken and applied over the cleaned surface and thin film to make sure the easy removing

Table 1. Methodical Description of Vinyl Ester, Epoxy and Polyurethane

	Vinyl ester	Epoxy	Polyurethane
Peak Exotherm (150 g@25°℃)	42	41	42-47
Contraction % (Volume)	0.29	0.32	0.27
Tensile strength	56 MPa	67 MPa	36
Elongation at break	0.9-1.2%	1-3%	1.4-2.1%
Flexural strength (MPa)	70-90	90-100	40-60
Compressive strength	70 MPa	80 MPa	62
Loss Tangent	0.051@50Hz	0.045@52 Hz	
HDT	86 °C	100 °C	45 ℃
Water absorption	0.19%	0.55%	0.10%
Co-efficient of expansion	45-55 ppm/°C	35-45 ppm/°C	55-75 ppm/°C



Figure 3. Fabricated EPC, VPC & EVC specimens.

of the fabricated spring. As noted in the Table 1, the respective premixed blends were applied over the wooden mandril as shown in the Figure 1. Likewise, the pre-cut carbon fiber with necessary dimensions were laid over the wetted surface, the same set of procedure of laying the fiber mat over the mandril was continued till the 5 layers of adhesion. This layer of mat laying gives the 3 mm of thickness of specimen. As soon as the laying processes were completed the entire specimens were kept in the open atmosphere for the period of 24 hours to get the polymerization of blends. After completing the drying process at ambient temperature, the entire specimens were removed out from the mandril and kept in the hot air oven for the period of 2 hours by retaining the temperature of 80 $^{\circ}$ C in order to get the complete polymeric condensation as shown in Figure 3.^{21,22}

Testing Methodology. DSC Analysis: The small segments of IPN laminates of various proportions of epoxy, vinyl ester and polyurethane had been tested by using differential scanning calorimetry. Similarly, the DSC plots were obtained by heating the samples between the ranges of 30 °C to 520 °C by maintaining the constant temperature rate of 10 °C/min, as well by retaining the inert gas flow of 100 mL/min. Further, the instruments used in this study were calibrated with indium, as well as fusion of heat and temperature calibration had been monitored during this each start-up of the instrument in order to maintain accurate results. Likewise, samples were precisely measured with the accurate of 0.01 mg and their subsequent corresponding ranges were 25-35 mg.²³

Load – Deflection Test Procedure: To do the compression test analysis, ASTM D2412 standard was followed. While doing the leaf spring analysis procedure, the specimen was kept in the universal testing machine (Instron 6500 - UTM, Instron, United



Figure 4. Compressive test set up - analysis.

States) such a way both the ends of the samples were firmly fixed as shown in the Figure 4. Finally, the lateral load was applied with the feed rate of 10 min/mm. the actual reason behind of that of finding this spring stiffness was that, to observe the adoptability of the samples during different loading circumstances. This test was intended to find out the left over strength also it was the prerequisite for low frequency impact loading test procedure.²⁴

Cyclic Loading Test: Once the load-deflection test was over, the specimens were subjected in order to find out the cyclic load parameters to find out the left over strength after load-deflection data's obtained. The fabricated carbon fiber reinforced interpenetrating polymer network (IPNs) samples were subjected with fluctuating loads (cycles) throughout the entire operation. The following states of subjected stresses were tension-compression, tension-tension along with compression-compression as well combining of all stresses. In fact, fatigue behaviour of specimen was completely depends upon variety of conventional parameters such as applied stress or strain, fluctuation with respect to applied load with respect to number of cycles.²⁵

Consequently, with the purpose of justify the cyclic behaviour of carbon fiber reinforced variety of IPN blend elliptical springs, loading and unloading cycling tests were performed as shown in the Figure 5 (Instron 8800 Series - Dynamic and Fatigue Testing System, Instron, United States). While doing so, half million cycles at the rate of 1 Hz frequency was adopted as per the ASTM E606 standard.^{26,27}



Figure 5. Specimen loaded in fatigue (cyclic) stress.

Result and Discussion

Differential Scanning Calorimetry Analysis. The cure behaviour against the temperature of various IPN formulations was shown in the Figure 6. All set of curves illustrated in the figure shows that, two kind of normal deviations such as crosslinking and decomposition in the DSC curves. Irrespective of IPN formulations, common crosslinking was largely noticed in between the range of 80 $^{\circ}$ C to 240 $^{\circ}$ C. Along with that, matrix decomposition also observed in between the ranges of 330 $^{\circ}$ C and 410 $^{\circ}$ C in all kind of IPN blends. In the view to calculate the thermic reactions in normal composite materials, manipulating area under the DSC curves were common method also it was believed as the tedious process. Additionally, the existence of cross linking towards the side of exothermic reactions across the transition temperature moderately describes the heat with standing capacity of the composite laminates. Also it was seriously looked that, an endothermic reactions concurrently causes the massive weight loss of the samples in due respect of compositional changes during the heating processes. As well, endothermic dip was the cause of decomposition reaction of matrix blends. Moreover, it was seen that, only carbon fibers were remain in the DSC pans as well noticed that, no trace of matrix traces in the pan. From the visualisation in the pan, carbon fibers were completely disintegrated. Furthermore, a large temperature range as well, had been noticed between the final end of the exothermic as well as the start of the endothermic reactions. Also it was noticed that, in between the temperature range of 210 $^{\circ}$ C to 310 $^{\circ}$ C, the heat dissipation was linearly proportional towards temperature, similarly, it was the evident of specific heat capacity of full cross lined polymers. By accurately adjusting the imaginary line between the glass transition temperatures towards the left and extending the same line towards right up to the decomposition temperature, it could be easy way to calculate the reactions in another way. Similarly, the degree of cross-linking might be easy to determine the specific heat of the corresponding reactions in such a way it could also be inferred with data provided by most of the suppliers and many authors doing research in this particular arena. The profiles which were mentioned in the various DSC curves predominately demonstrated the uniformity of the fabrication processes of different IPN laminates. In all the samples, beyond the specific limit of 590 °C, the kept samples were completely degraded and became as the residual matter. From the graph it was clearly understood that, the exothermic hump at 112, 115, and 119 °C for Vinyl ester + polyurethane, Epoxy + polyurethane and Epoxy + vinyl ester respectively.



Figure 6. Differential scanning calorimetry curves of IPN blends.

Simultaneously, the endothermic curve as well was noticed at an on average of 410 $^\circ\! C$ on all blends.^^28

Thermogravimetric Analysis (TGA). The Figure 7 illustrates the various blends of thermogravimetric analysis. In the bar chart of TGA, it was clearly understood that, the blend combination of epoxy and vinyl ester possess the higher TG value as compared with remaining two set of blends. Their corresponding obtained value was that nearly 121 °C, the higher in value of TG shows that, presence of hard segment presence had played the pivotal role in sustaining such as higher TG value. As well, hard segments interlocking mechanism holds or perhaps needs higher temperature to de-bond the interlocking mechanism of the blends. In the contrary, the EP (Epoxy with polyurethane) samples had showed the slight lesser value than the EV (Epoxy with vinyl ester), their subsequent obtained value was 114 °C, the reason behind that of lesser value was that presence of soft segment presence in the samples, though the hard segment presence of epoxy holds the interlocking mechanism against the heat, the soft segment of polyurethane loosens the locking chains and comes early choking. Likewise the VE (vinyl ester with polyurethane) as well shows the same trend as such value received from the EP, the presence of soft segment in the VE comes with rapid loosing of interlocking mecha-



Figure 7. Thermogravimetric depiction of EV/EP/VP of IPN blends.



Figure 8. (a) and (b) Load – deflection & stiffness (without cyclic loading) of pristine leaf spring CEV/CEP/CVP.

nism, as the evident for the same, obtained value was 109 $^\circ C$, this was quite less than all set of blends.^29

Load - Deflection Test Analysis. The Figure 8(a) demonstrates the load deflection curve of the carbon fiber reinforced various proportionate of interpenetrating polymer network leaf spring. The foremost purpose of this assessment was that to find out the left over strength after the leaf spring subjected with the different types of loading. Throughout the entire test, it was detected that the CEV specimens had shown the better characteristics in terms of load bearing capacity, whereas the deflection found on various loading was very less as compared with another set of specimens, for the load of 100 N the obtained value of CEV was nearly 6 mm, that value kept increases with respect to various type of loading. The observed value of the corresponding loading of 200 N was nearly 14 mm. As such increasing trend observed in the case of 100 N and 200 N, all the loads had shown the same increase in the trend of deflection. Whereas the CEP specimens had shown the deflection value was quite higher than the CEV, the obtained values were 8, 16, 29, 43, 54 mm for 100, 200, 300, 400, and 500 N respectively. The increase in the deflection value clearly evident with the influence of IPNs, the blend of polyurethane shows the key role in such a way to increase the deflection value of the lead spring specimens. However, the soft segment presence in the polyurethane had completely enhances the spring back effect of the specimens as well successively reduces the brittleness characteristics of the epoxy, as it possess the hard segment presence. As such values observed for the CEP, the CVP specimen also shows the same like trend. To evident that, the obtained values were 9, 17, 31, 44, 55 mm for successive loads of 100, 200, 300, 400, and 500 N respectively. Nevertheless, it was understood that as much as addition or loading of the polyurethane into the base matrix it increases the elongation or displacement in considerable way. From the curve also it was unmistakably understood that, as long as load increases their equivalent deflection value also increases irrespective of IPNs.³⁰

Similarly the Figure 8(b) also states the stiffness value of the carbon fiber reinforced IPN leaf spring with various combinations of blends. In this curve, it was evident that the stiffness value was completely deterministic with respect to the addition of polyurethane and other kind of blends into the IPNs. The obtained values had shown the direction correlation with the load displacement curves.

Cyclic Load Analysis. The main objective of subjecting the IPN leaf spring specimens towards cyclic load test was that to find out influence of prestressed effect in view of deflection aspects. While conducting this tests, the samples were exposed with the cyclic loads for the specific periods of 2760, 6540, and 9430 cycles and their corrponding graphs are presented in Figure 9(a-c).

After cyclic load or fatigue test was over, the specimens again subjected for load deflection test in order to find out the threshold limit of the speicmens to suitably opt the respective IPNs for the specified purpose. As the Figure mentioned in 9(a), (b), (c) based on the cyclic loads, speicmens had showed the conclusive set of variables with respect to the applied load. In that context, the 9(a) specimens had showed the respective values for 2760 cycles, it was observed that, CEV specimens showed the deflection value of 42, 48, and 50 mm for CEV, CEP, and CVP specimens for the load of 500 N. Similarly, the specimens subjected with the 6540 cycles had showed the elongation value as 38, 44, and 48 mm for CEV, CEP and CVP respectively. The obtained value for this particular cyclic process was quite less as compared with the 2760 cycles. It shows that due to the continuous fatigue loading on the specimens, the thermoset polymers used as the matrix materials were continuously coming across the thermal strain in the specimens, due to which the matrix materials were completely undergoing or squeezing themselves



Figure 9. (a), (b), (c) illustrates the deflection value after periodic cyclic loads.

as part of their complete polymerization due to this effect the matrix materials were stiffened themselves along with the carbon fiber reinforcement and lessens the elongation. Moreover, the same effect was again evident with the specimens which subjected with the (Figure 9(c)) 9430 cycles.²⁴⁻²⁵ The specimens subjected with the 9430 cycles were shown comparatively very less deflection value; the obtained values were 32, 40, and 42 mm for CEV, CEP and CVP respectively. This deflection value was nearly 6% lesser than the value which was obtained in the sequence of 6540 cycles. This entire method processes had showed the specimens which were subjected with so long increment value in the cyclic process were showed the lessened deflection value

irrespective of IPN blend. It seems that the cyclic loading emphasizes or holds the thermal strain in the specimens thus the way it leads to the complete polymeric condensation process and significantly increases the stiffness value in considerable way.³¹

Scanning Electron Microscopic Analysis (SEM). The fractured surfaces of the CEV, CEP, and CVP had been examined with help of the scanning electronic microscopic apparatus and their images were presented in the Figures 10(a), (b), (c). The common failures mechanisms were seen across fracture images like de-bonding, fiber scissoring, fiber pull-out, void and



Figure 10. (a), (b), (c) fractographic images of fractured surfaces of various proportionate of IPN blends reinforced with carbon fiber.

matrix cracking. The Figure 10(a) represents the CEV specimens, as per the load versus deflection curve, it holds the higher level of load with less deflection because of the hard segment presence on both the individual matrixes. In the picture, common delamination between the fiber and matrix was seen as well as small amount of fiber scissoring was visualized across the fractured surface. It shows matrix holds the fiber with better interfacial strength before the fracture happens in the specimen. Similarly, in the Figure 10(b), fiber scissoring were commonly seen across the fractured surface. It shows that, though the hard segment presence of polyurethane epoxy holds the fiber rigidly; soft segment presence of polyurethane induces the early failure because of its elastic nature.³²⁻³⁴

As such delamination seen in the CEV, the CEP also evidences the delamination across the fractured surface. Likewise 10(c), also shows the fractured images of CVP, though the hard segment of vinyl ester gives resistance against the load and deflection, the soft segment presence in the polyurethane could not withstand against the static load, even though it possess the better spring back effect.

Conclusions

The carbon fiber reinforced interpenetrating polymer network (IPNs) elliptical spring specimens were given the following affirmations out of the entire experimental works.

1. The combination of variety of blends like epoxy (E), vinyl ester (V) and polyurethane (P) were eventually utilized in the field of elliptical spring manufacturing along with the reinforcement of carbon fiber (C).

2. The IPN blend composition's decomposition was observed in between the range of 310 °C to 410 °C in all set of IPN blends respective of blend ratios. Similarly the EV had shown the higher T_g value as 121 °C higher than the remaining compositions like EP (114 °C) and VP (109 °C) T_g values.

3. While doing the load deflection tests, the results exhibit that, CEV specimens had showed excellent strength with standing capacity as compared with other combinations, but it fails without giving much elongation at peak loads. Though CEP and CVP specimens had not shown equal strength to the CEV specimens, still elongation at break was consecutively better than the CEV specimens. The reason behind that of this property was, possessing the property of shape memory property of polyurethane. The soft segment presence of PU properly entangles with hard segment of vinyl ester and epoxy and gives the spring back effect during the entire study. 4. Another interesting finding was that, the cyclic load (fatigue) proportionately increases or holds the thermal strain on the specimens. This stored thermal strain adversely squeezes the thermoset blend and increases the stiffness factor of the specimens in marginal level. It seems that the effect of squeezing diligently increases the interfacial strength between the fiber and matrix, also leads to the complete polymeric condensation process to the predominant level.

Conflict of Interest: The authors declare that there is no conflict of interest.

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