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LATVIAN JOURNAL OF PHYSICS
AND TECHNICAL SCIENCES
2024, N 6

DOI: 10.2478/lpts-2024-0046

MECHANICAL AND THERMAL PROPERTIES OF NANOCOMPOSITES REINFORCED WITH PAN NANOFIBRE MATS

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The integration of electrospun nanofibres into composite materials such as carbon fibre reinforced polymers (CFRP) and glass fibre reinforced polymers (GFRPs) has gained significant attention for enhancing their mechanical and thermal properties. This study focuses on the impact of incorporating polyacrylonitrile (PAN)-based nanofibres into an epoxy matrix to form multi-layered laminate composites. Our approach involved interleaving these orientated nanofibres within CFRP/GFRP matrices to investigate improvements in bulk material characteristics. The tensile and thermal properties of the resulting nanocomposites were thoroughly analysed. Thermogravimetric analysis (TGA) was employed to evaluate the thermal stability and degradation behaviour. In particular, nanocomposites exhibited an increase of 5–24 % in elastic modulus, aligning with predictions from analytical models. This research underscores the potential of PAN nanofibre mats for the development of lightweight, high-performance nanocomposites, offering a novel methodology for the enhancement of composite materials.

Keywords*: Mechanical properties, nanocomposite, PAN nanofibre mat, thermogravimetry (TGA).*

1. INTRODUCTION

The advent of electrospinning as a cost-effective method for producing nanofibres has ushered in a paradigm shift in material science research, particularly in the realm of carbon fibre reinforced polymers (CFRP) and Glass Fibre Reinforced Polymers (GFRP). Over the past decade, research has increasingly gravitated towards the integra-

tion of nanofibre mats into these composite materials. This growing interest is attributed to the compelling evidence that the interleaving of nanofibre mats significantly augments the mechanical and thermal attributes of CFRP/GFRP composites [1].

The strategic inclusion of nanofibre mats within laminated composites yields numerous benefits. A primary advantage is the marked improvement in delamination resistance [2], which is essential for the preservation of the structural integrity of composites. Furthermore, the impact tolerance of these materials shows substantial improvement, thereby bolstering their resilience against physical stresses and strains, a quality of paramount importance in applications subject to dynamic or unpredictable environmental forces.

Furthermore, the fracture and fatigue resistance of CFRP/GFRP composites is considerably reinforced through the introduction of nanofibre mats. This enhancement not only prolongs the useful life of the materials, but also reduces the frequency of necessary repairs or replacements. The interlaminar toughness and crack resistance also show significant improvements, contributing to the overall durability and reliability of these composite structures [3].

Another area of growing interest is the development of self-healing materials and nanofibrous materials with antibacterial properties with textiles [4]–[6]. Incorporation of nanofibre mats into composites is a gateway to pioneering materials capable of autonomously repairing damage, thereby extending their utility and functional lifespan.

Table 1 meticulously catalogues these enhancements, each improvement substantiated by pertinent references. This provides an exhaustive overview of the prevailing research in this domain, emphatically demonstrating the profound impact of nanofibre mats on the performance of composite materials. Table 1 also highlights the significance of integrating nanofibre mats, derived from various materials, into CFRP and GFRP. It elucidates how this integration materially enhances the mechanical and thermal properties of these composites, highlighting improvements in delamination resistance, impact and fracture tolerance, fatigue resistance, and interlaminar toughness and crack resistance.

Table 1. Enhancements in CFRP/GFRP Composites through Nanofibre Mat Interleaving

Application of nanofibre mats in CFRP/GFRP composites	References
Improving the delamination resistance	[7]–[11]
Enhancing the low impact tolerance	[7], [12]–[15]
Augmenting the fracture resistance	[12], [16]–[20]
Enhancing the fatigue resistance	[2], [8], [13], [16]
Improvement in thermal properties	[21]–[26]
Enhancement of mechanical properties	[22]–[31]
Improvement in interlaminar toughness	[32]–[39]
Development of self-healing composite materials	[40], [41]

Table 1 encapsulates the diverse applications of nanofibre mats in reinforcing CFRP and GFRP composites, as delineated in the literature. Each category of improvement is supported by a range of references, illustrating the breadth of research and corroborating the multifaceted enhancements attributed to the inclusion of nanofibre mats. This compilation provides a valuable resource for understanding the scope and depth of current advances in this field.

Notwithstanding these advances, inves-

tigations into the tensile strength and thermal properties of nanofibre interleaved composite laminates remain relatively unexplored. Although notable enhancements in tensile strength and thermal degradation resistance are observed with nanofibre mat interleaving, corroborative evidence for similar improvements in tensile strength for interleaved composites is limited. This underscores the need for more comprehensive research into the mechanical and thermal behaviours of nanofibre interleaved composite laminates, particularly in order to determine if this methodology can effectively mitigate one of the fundamental limitations of laminated composites.

The present research focuses on the influence of polyacrylonitrile (PAN) nanofibre mats on the mechanical and thermal properties of epoxy composites. Through the interleaving of various layers of nanofibre mats to fabricate layered structures, this study endeavours to discern their impact on the mechanical and thermal properties of nanocomposites.

2. MATERIALS AND METHODOLOGY

This study delves into the impact of polyacrylonitrile (PAN) nanofibre layers on laminated nanocomposite materials, employing a methodology that integrates the fabrication of electrospun nanofibres, mechanical testing, and thermal analysis.

In the fabrication of electrospun nanofibres, the materials used included PAN powder, with an average molecular weight of 150,000 and a CAS number of 25014-41-9, and N,N-dimethylformamide (DMF), a solvent with a purity of at least 99.8 % and a CAS number of 68-12-2. These materials were obtained from Sigma-Aldrich, Merck KGaA, Darmstadt, Germany. Additionally, the liquid resin epoxy and hardener, identified by the CAS number 964-67-8, were obtained from the Latwood Master razosanas komercfirma Ltd in Latvia. The PAN nanofibre mat was conducted in a manner similar to that previously described in the literature [42]–[45] and employed the hand lay-up method, as previously utilised by the author [25] for reinforced polyamide 6 reinforced nanocomposites. A detailed flow chart of the nanocomposite fabrication process is presented in Fig. 1.

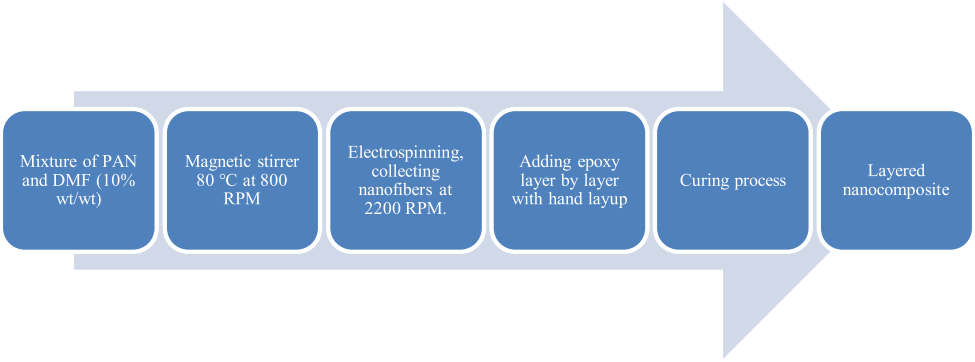


Fig. 1. Comprehensive schematic illustrating the fabrication process of the layered nanocomposite.

For mechanical testing, the tensile properties of the nanocomposites were evaluated using a Mecmesin Multi-Test 2.5-i tensile testing machine, equipped with a 25/250-N sensor, provided by PPT Group UK Ltd., Mecmesin, UK. The consistency and reliability of the results were ensured by conditioning the samples at room temperature according to ISO 139:1973 "Standard Environments for Conditioning and Testing," which stipulates a temperature of 21 ± 1 °C, a relative air humidity of 60 % and atmospheric pressure of 760 mm Hg. The sample dimensions adhered to the ASTM D882-18 standard, measuring 50 mm × 10 mm (length × width), with four measurements taken to accurately determine the tensile properties. The thickness of the nanofibre mats was precisely measured using a digital micrometre (range: 0–25 mm; Digimatic micrometre, MDC-25PX, code No. 293-240-30, serial No. 71912410, Mitutoyo, Japan). For the tensile testing, specimens were prepared both parallel to the nanofibre direction, facilitating testing in longitudinal directions. A specific paper template was used for sample preparation, and the samples were mounted on the tensile testing machine with double-sided thin scotch tape, followed by careful removal of the sides of the paper template. Figure 2 illustrates the tensile test specimen.



Fig. 2. Depiction of post-tensile nanocomposite samples.

The methodology further encompasses a systematic approach to classifying the nanocomposite specimens, which were composed of varying layers of PAN nanofibre mats interleaved within the epoxy matrix. These specimens were designated with specific labels: "CL1", "CL2", and "CL3". Each label corresponds to a distinct composite structure, with "CL1" representing a composite containing a single layer of nanofibre mat, "CL2" for a composite with two layers, and "CL3" for those comprising three layers of nanofibre mats. The initialism "CL" in these designations stands for "Composite Layer", followed by a numeral denoting the number of nanofibre layers incorporated within the composite. This classification system facilitated a structured and precise evaluation of the mechanical and thermal properties of the nanocomposites, allowing for a clear comparison between the varying compositions and structures.

For statistical significance the Halpin-Tsai model was applied to the PAN nanofibre-reinforced epoxy composites, with its predictions compared with experimental data to validate its effectiveness in estimating the mechanical properties of these composites.

$$E_c = \frac{E_m(1 + \zeta\eta V_f)}{(1 - \eta V_f)}, \quad (1)$$

where

E_c – the effective modulus of the composite material, MPa.

E_m – the modulus of the matrix material, MPa.

V_f – the volume fraction of the fibres.

η – the efficiency parameter, which is a function of the aspect ratio of the fibres.

ζ – the reinforcement factor, which relates the modulus of the fibre to the modulus of the matrix.

The Halpin-Tsai model, useful for composites with a high aspect ratio of reinforcement fibres, assumes perfect bonding between fibres and matrix, treating the composite as a homogenised material.

Thermal properties were evaluated by thermogravimetric analysis (TGA), using a TG 209 F1 Libra® thermomicrobalance from NETZSCH, Germany. Samples, weighing between 5 and 6 mg, were placed in Al_2O_3 crucibles and subjected to a temperature range of 20 °C to 800 °C at a heating rate of 10 °C/min, under an inert nitrogen atmosphere with a flow rate of 30 ml/min. Precise weight measurements of the samples were carried out using a laboratory scale from KERN&Sohn GmbH, Germany. The TGA provided insightful data on the thermal stability and decomposition characteristics of the composite materials, with a focus on identifying specific temperatures at which significant mass loss occurred, thus indicating the thermal degradation points of the composite material.

3. RESULTS AND DISCUSSION

The results obtained from the tensile testing of PAN nanofibre mats, pure epoxy, and nanocomposites with varying layers of PAN nanofibre are instrumental in understanding the mechanical behaviour of these materials.

Figure 3 shows the tensile strength and elongation properties of PAN nanofibre mats. The data indicate that the PAN nanofibre mat exhibits a maximum tensile strength of approximately 8.5 ± 0.5 MPa and an elastic modulus of around 920 ± 15 MPa. Furthermore, the material demonstrates an elongation at break of 5.4 ± 0.1 %, suggesting a degree of flexibility inherent in the PAN nanofibre mats.

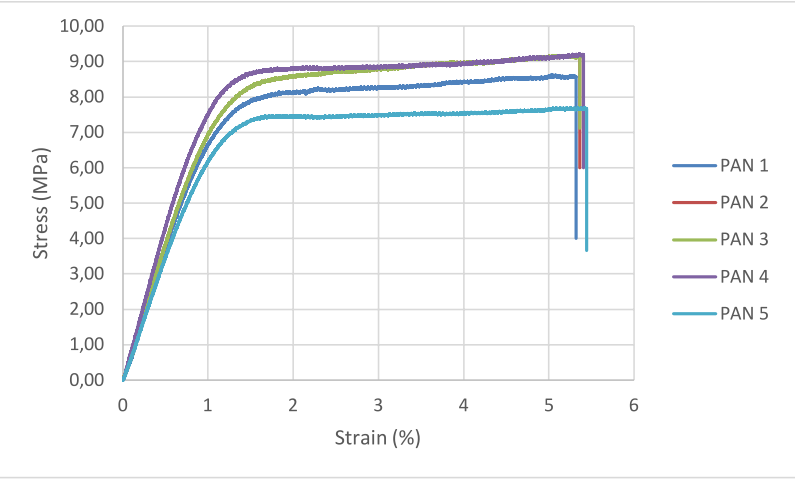


Fig. 3. Results of PAN nanofibre mats.

Figure 4 shifts the results of the focus to the tensile test for pure epoxy. The results reveal that pure epoxy has a higher maximum tensile strength of 12.8 ± 2 MPa compared to the PAN nanofibre mats, although with a lower elastic modulus of 580 ± 12 MPa. The elongation at break for pure epoxy is recorded at 5.1 ± 0.2 %, which is

closely aligned with the values observed for the PAN nanofibre mats. In particular, sample Ep 1 was excluded from the analysis due to measurement error.

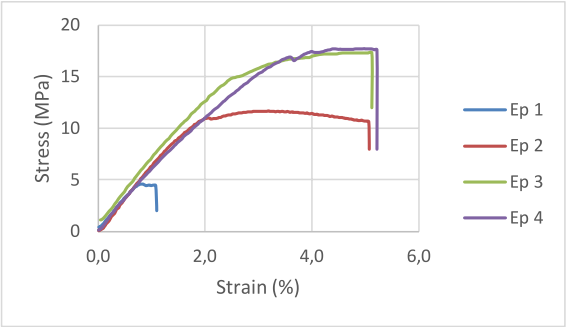


Fig. 4. Results of pure epoxy.

The tensile properties of nanocomposites with different layers of PAN nanofibre are detailed in Figs. 5–7. The single layer nanocomposite, as shown in Figure 5, exhibits a maximum tensile strength of 12.5 ± 2 MPa and an elastic modulus of 610 ± 15 MPa, in conjunction with an elongation at break of 2.1 ± 0.2 %. On the contrary, the two-layer nanocomposite shown in Fig. 6 shows an increase in tensile strength to 16 ± 3 MPa and elastic modulus to 650 ± 18 MPa, with an elongation at break of 3 ± 0.3 %. The three-layer nanocomposite, presented in Fig. 7, further enhances these properties, demonstrating a maximum tensile strength of 18 ± 2 MPa, an elastic modulus of 720 ± 12 MPa, and an elongation at break of 3 ± 0.5 %.

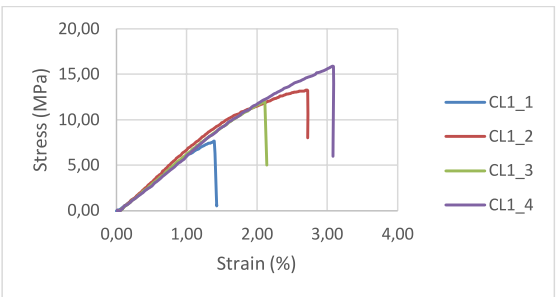


Fig. 5. Tensile test results for single-layer PAN nanofibre-epoxy composite.

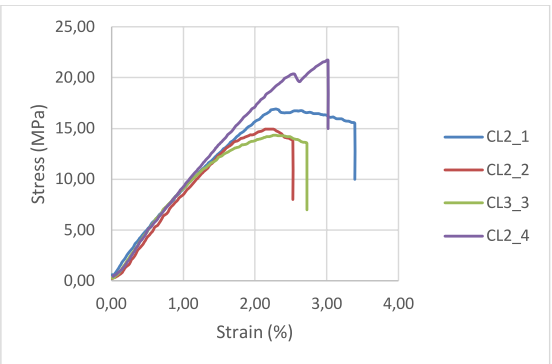


Fig. 6. Results of tensile testing on a two-layer PAN nanofibre-epoxy composite.

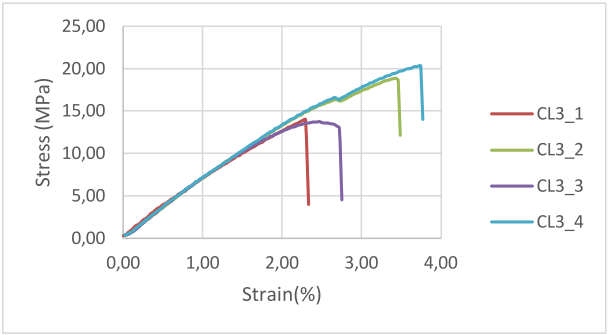


Fig. 7. Results of tensile properties for three-layer PAN nanofibre-epoxy composite.

Table 2 offers a concise summary of the mechanical properties of all the materials tested, including PAN nanofibre mats, pure epoxy, and various layered nanocomposites. Table 1 enables an efficient comparison across different materials, underscoring the incremental improvements in tensile strength and elastic modulus brought about by the addition of PAN nanofibre layers, while moderately affecting their elongation at break.

Table 2. Summary of Mechanical Properties: PAN Nanofibre Mats, Pure Epoxy, and Layered Nanocomposites

Samples	Thickness, t (μm)	Tensile strength σ at break (MPa)	Young's modulus, E (MPa)	Elongation at break, ε at break (%)
PAN	74 ± 10	8.5 ± 0.5	920 ± 15	5.4 ± 0.1
EP	154 ± 7	12.8 ± 2	580 ± 12	5.1 ± 0.2
CL1	200 ± 11	12.5 ± 2	610 ± 15	2.1 ± 1
CL2	270 ± 15	16 ± 3	650 ± 18	3 ± 0.3
CL3	335 ± 18	18 ± 2	720 ± 12	3 ± 0.5

The findings of these tests and analyses highlight that the incorporation of PAN nanofibre mats into epoxy composites significantly improves their mechanical properties, particularly in terms of strength and stiffness. This enhancement in material properties with the addition of nanofibre layers illuminates the potential of nanofibre reinforcement in the development of advanced material applications.

The study also incorporates the application of the Halpin-Tsai micromechanical model to calculate the elastic modulus of the nanocomposites. This model uses specific experimental values, including the modulus of the epoxy matrix (E_E) recorded at 580 MPa and the modulus of the PAN nanofibre mat (E_{PAN}) recorded at 920 MPa.

Upon integrating these values into the model, the resultant calculations for the elastic modulus of the nanocomposites with varying layers of PAN nanofibre are as follows:

- for the composite with one layer of PAN nanofibre mat (E_{CL1}), the calculated elastic modulus is 610 MPa;
- for the two-layer composite (E_{CL2}), the modulus increases to 635 MPa;
- the three-layer composite (E_{CL3}) exhibits a further increase in the elastic modulus, reaching 670 MPa.

These results demonstrate a progressive increase in the stiffness of the com-

posite material with the incorporation of additional layers of PAN nanofibre mats. The calculated values reflect the enhanced mechanical properties of the nanocomposites, confirming the reinforcement efficacy of the PAN nanofibres. This progression aligns with the predictions of the Halpin-Tsai model, highlighting its applicability in estimating the mechanical properties of fibre-reinforced composites.

Figure 8 presents the results of thermogravimetric analysis (TGA) for pure epoxy. The analysis elucidates that the maximum degradation temperature of pure epoxy is recorded at 373.5 °C. This temperature is indicative of the point at which the most significant thermal decomposition occurs, providing an insight into the thermal stability of the epoxy.

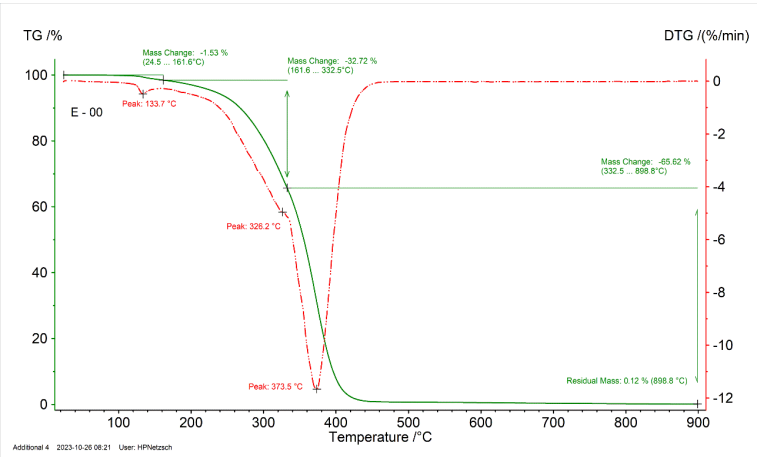


Fig. 8. TGA and DTG graphs of pure epoxy.

Proceeding with the TGA studies, Fig. 9 examines the thermal degradation of the single-layer reinforced nanocomposite. In particular, the peak degradation of this composite was observed at a slightly reduced temperature of 370.5 °C. This marginal decrease, in comparison with pure epoxy, suggests that the incorporation of a single nanofibre layer subtly influences the thermal stability of the composite.

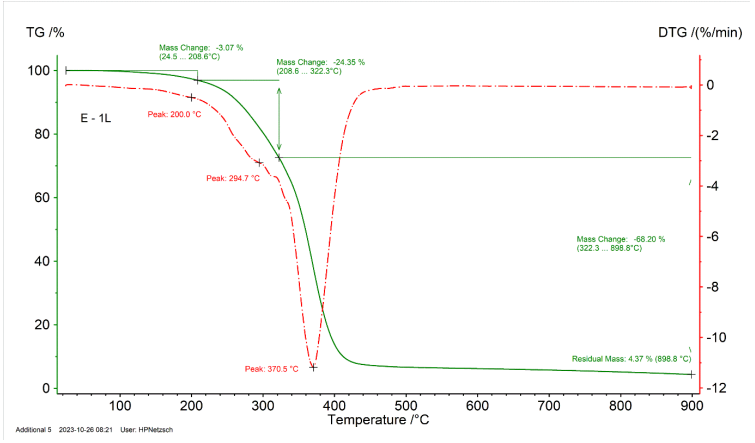


Fig. 9. TGA and DTG graph of the PAN nanofibre reinforced composite.

In the context of the double layer reinforced nanocomposite, as demonstrated in Fig. 10, the maximum degradation temperature was found to be 369.4 °C. This further decline in the peak degradation temperature with the addition of another layer of nanofibres underscores a more pronounced impact on the thermal properties of the composite.

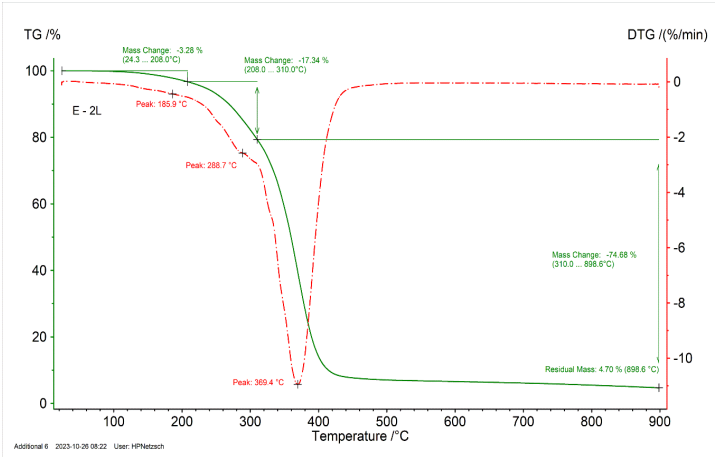


Fig. 10. TGA and DTG graphs of double-layer PAN nanofibre reinforced composite.

Extending this thermal analysis to the reinforced three-layer nanocomposite in Fig. 11, the degradation peak occurs at a lower temperature of 364.2 °C. This consistent reduction in the degradation temperature with each additional layer of nanofibres highlights the significant effect of nanofibre reinforcement on the thermal behaviour of the composites.

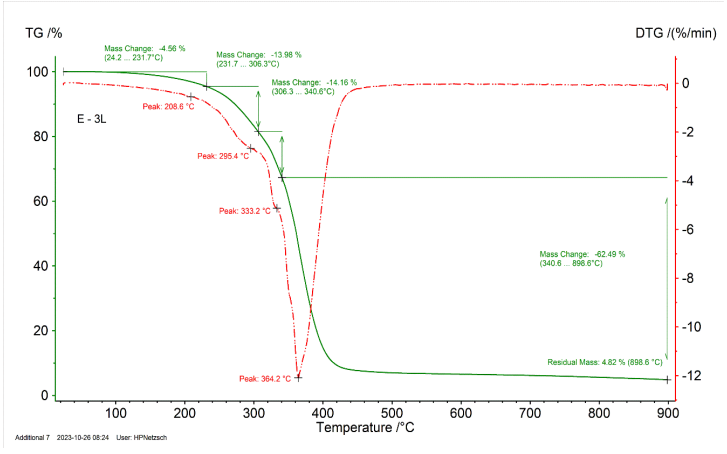


Fig. 11. TGA and DTG graph of three-layer PAN nanofibre reinforced composite.

Figure 12 offers a comparative TGA analysis of all nanocomposites alongside pure epoxy. This juxtaposition provides a lucid visual depiction of the impact of nanofibre layer addition on the thermal degradation behaviour of epoxy composites. It facilitates a straightforward comparison between the thermal stability of the pure

epoxy and that of the nanocomposites, accentuating the role of nanofibre reinforcement in modifying the thermal response.

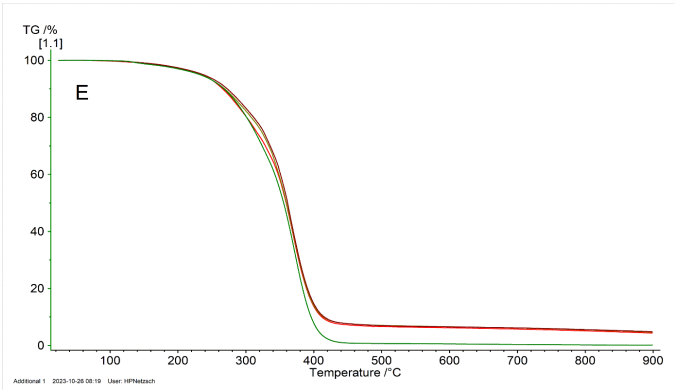


Fig. 12. Comparative thermogravimetric analysis: Nanocomposites versus pure epoxy (highlighted in green).

The thermogravimetric analyses conducted are pivotal in delineating the thermal properties of the nanocomposite materials. The gradual decrease in the peak degradation temperature with the incremental addition of nanofibre layers clearly manifests the influence of nanofibres reinforcement on the thermal behaviour of epoxy composites.

These analyses are instrumental in optimising the design of nanofibre-reinforced composites for specific applications where thermal properties are crucial. The findings emphasise the necessity of comprehending the interactions between the matrix material and the reinforcing nanofibres, since these interactions are determinants of the composite's overall performance and applicability in various scenarios.

Incorporation of the Halpin-Tsai model into our analysis provides a more nuanced understanding of the mechanical enhancements brought about by the inclusion of PAN nanofibre mats in epoxy composites. The predictions of the model, corroborated by our experimental findings, underscore the significant role these nanofibres play in increasing the stiffness of the composites. Notably, the observed progressive increase in elastic modulus with the addition of more nanofibre layers is consistent with theoretical expectations and is experimentally validated, affirming the reinforcing efficacy of the PAN nanofibres.

Tensile strength tests further reinforce the notion that PAN nanofibre mats significantly bolster the mechanical attributes of epoxy composites. The evident increase in tensile strength and elastic modulus with each additional nanofibre layer underscores the effectiveness of this reinforcement strategy, which is also noted in this research [3] the research on composite laminates enhanced with electrospun thermoplastic nanofibres is still very limited and a thorough understanding of the toughening mechanism is still missing. This article provides thorough insights into the micromechanisms that lead to the interlaminar toughening of carbon/epoxy composite laminates interleaved with electrospun polyamide nanofibrous veils. The main mechanism leading to a higher interlaminar fracture toughness, both under Mode I and Mode II loading conditions, was the bridging of (micro. However, this improvement in strength comes with a concomitant decrease in ductility, as evidenced by the

reduced elongation at break. This trade-off is a common feature in composite materials, where the infusion of reinforcing elements typically enhances strength at the expense of flexibility.

Thermogravimetric analysis (TGA) reveals an intriguing aspect of the thermal properties of the nanocomposites. While pure epoxy exhibits a higher thermal degradation temperature, the addition of PAN nanofibres induces a reduction in this temperature. This phenomenon suggests a possible alteration in the thermal degradation behaviour of the epoxy matrix upon introduction of the nanofibres. The consistent reduction in the maximum degradation temperature with each layer of nanofibres could imply a cumulative effect of nanofibre reinforcement on the thermal stability of the composites. This observation warrants a deeper investigation into the thermal interaction mechanisms between the epoxy matrix and PAN nanofibres.

4. CONCLUSIONS

This study provides a comprehensive analysis of the mechanical and thermal properties of PAN nanofibre-reinforced epoxy composites, enriched by the application of the Halpin-Tsai model. The progressive increase in stiffness with the addition of more nanofibre layers, as predicted by the model and confirmed by experimental results, highlights the effectiveness of nanofibre reinforcement in improving the mechanical properties (5–24 %) of composites.

The findings of tensile testing and thermogravimetric analyses demonstrate the potential of these composites in applications requiring enhanced mechanical strength and modified thermal properties. The gradual decrease in the maximum degradation temperature with increased nanofibre layers, though warranting further investigation, provides valuable insights into the thermal behaviour of these materials.

In conclusion, the study contributes significantly to the field of composite materials, offering vital information on the interplay between mechanical strength, flexibility, and thermal stability in nanofibre-reinforced composites. Future research should focus on a deeper exploration of the interfacial interactions between the matrix and the reinforcing fibres and the scalability of these composites for practical applications.

ACKNOWLEDGEMENTS

The research has been supported by the Doctoral Grant Programme of Riga Technical University.

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