

Fiber reinforced polymer composites: preparation, mechanical properties and thermal analysis

Konstantinos Karvanis, Ph.D.

Doctoral Thesis Summary



Tomas Bata University in Zlín

Faculty of Technology

Doctoral Thesis Summary

Vláknny vyztužené polymerní kompozity: příprava, mechanické vlastnosti a termická analýza

**Fiber reinforced polymer composites: preparation, mechanical
properties and thermal analysis**

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Key words: *FRP, polymer composite, thermal analysis, DMA, TMA, TGA, DSC, carbon fiber, glass fiber, basalt fiber, aramid fiber*

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SHRNUTÍ

V dnešní době se polymerní kompozity široce používají, proto je důležité podrobně zkoumat jejich vlastnosti do hloubky a za různých podmínek. Vlákem vyztužený polymer (FRP-Fiber-reinforced polymer) je kompozitní materiál s polymerním pojivem jako matricí a vlákny jako výtuznou fází. Jak je všeobecně známo, teplota má velký dopad na vlastnosti materiálů, zejména v případě vláknem vyztužených kompozitů, které mají polymerní matrici, protože polymery jsou relativně citlivé na vysoké teploty kvůli své viskoelastické povaze.

V této disertační práci byly pomocí různých výrobních technologií připraveny vláknem vyztužený kompozity s uhlíkovými, aramidovými, skleněnými a čedičovými vlákny. Byly zkoumány různé mechanické vlastnosti, jako je pevnost v tahu a ohybu, zejména byla pozornost věnována termické analýze vláknem vyztužených kompozitů prostřednictvím dynamické mechanické analýzy (DMA), termomechanické analýzy (TMA) a termogravimetrické analýzy (TGA).

Zejména v první experimentální části pojednání k disertační práce byly polymerní kompozity vyztužené skleněnými nebo uhlíkovými vlákny připraveny metodou vakuového lisování s použitím prepregových materiálů. Použitá vlákna jsou ve tkané formě s orientací 0° ($0^\circ/90^\circ$) nebo 45° ($-45^\circ/+45^\circ$) a v jednosměrné formě v podélném nebo příčném směru. V experimentální části tohoto pojednání byl zkoumán optimální typ vláken a jejich orientace prostřednictvím DMA a byla stanovena teplota skelného přechodu (T_g) kompozitů.

Ve druhé experimentální části pojednání disertační práce byly pomocí kombinované metody ruční laminace pod vakuovou fólií připraveny bazaltové vlákny vyztužené polymerní (BFRP) kompozity s epoxidovou matricí, pozůstávající s 20 vrstev a objemovým podílem vláken $V_f = 53,66\%$. Navíc pomocí DMA bylo prozkoumáno jejich viskoelastické chování v teplotním rozsahu $30 - 180^\circ\text{C}$ a frekvenčním rozsahu 1, 5 nebo 10 Hz, zatímco TMA byla zvolena na studium zkoušek creepového zotavení a napěťové relaxace. Kromě toho byla stanovena teplota skelného přechodu (T_g) kompozitů BFRP pomocí píku křivek $\tan\delta$, zatímco rozklad kompozitů BFRP a čedičových vláken ve vzduchu nebo v atmosféře dusíku byl zkoumán TGA. Také mechanické chování kompozitů BFRP bylo experimentálně zkoumáno v tahu a v tříbodovém ohybu.

Je třeba poznamenat, že v rámci zpracování následné disertační práce byly vyrobeny uhlíkové, aramidové nebo uhlík/aramidové hybridní vlákny vyztužené polymerní kompozity, které byly následně vytvrzeny při zvýšených teplotách a bylo zkoumáno jejich tepelné a mechanické chování. Tyto epoxidové kompozity po 7 dnech vytvrzování při pokojové teplotě byly dodatečně vytvrzované podle

specifického vytvrzovacího režimu. Vliv dodatečného vytvrzování byl zkoumán pomocí termické analýzy a tyto experimentálně naměřené výsledky budou prezentovány v rámci disertační práce.

SUMMARY

Nowadays, polymer matrix composites are broadly used, so their properties should be investigated in depth and under various conditions. Fiber-reinforced polymer (FRP) is a composite material which contains polymer as matrix and fibers as reinforcement phase. As it is broadly known, temperature exerts a high impact on materials' properties and especially in the case of the FRP composites which have polymer matrix; due to their viscoelastic nature, polymers are relative sensitive to high temperatures.

In this dissertation, FRP composites, with carbon, aramid, carbon/aramid hybrid, glass or basalt fibers as reinforcement phase were prepared through various fabrication methods. Their various mechanical properties, such as tensile and flexural strength were investigated whereas special attention was given in the thermal analysis of the FRP composites through Dynamic Mechanical Analysis (DMA), Thermomechanical Analysis (TMA), Thermogravimetric Analysis (TGA) experiments, and Differential Scanning Calorimetry (DSC).

In particular, in the first experimental part of this dissertation, glass or carbon fiber-reinforced polymer composites were prepared, through vacuum bag oven method, by using prepreg materials. The used fibers were in woven form, with orientations at 0° ($0^\circ/90^\circ$) or at 45° ($-45^\circ/+45^\circ$), and in unidirectional form, longitudinal or transverse direction. In the experimental study, the optimal fibers' type and orientation were investigated through DMA and the glass transition temperature (T_g) of the composites was determined.

In the second experimental part of the dissertation, basalt fiber-reinforced polymer (BFRP) composites with epoxy matrix, 20 layers, and volume fraction of fibers $\Phi_f = 53.66\%$, were fabricated through a hand lay-up compression molding combined method. Their viscoelastic behavior in the temperature range $30-180^\circ\text{C}$ and at 1, 5 or 10 Hz was explored by DMA whereas TMA took part in terms of creep recovery and stress-relaxation tests. Moreover, the T_g of the BFRP composites was determined through the peak of the loss modulus and $\tan\delta$ curves while the decomposition of the BFRP composites and basalt fibers, in air or nitrogen atmosphere, was explored through TGA. Also, the mechanical behavior of the BFRP composites was investigated by tensile and three-point bending experiments.

In the third experimental part of the dissertation, carbon, aramid or carbon/aramid hybrid fiber-reinforced polymer composites were fabricated; they were post-cured at elevated temperatures and their thermal and mechanical behavior was explored. After their 7 days curing at room temperature, these epoxy matrix composites were post-cured under specific heating-cooling rates. Extensive thermal analysis, and also exploration of the mechanical behavior of these composites is taken part, investigating in depth the effect of the post-cure, and the scientific results are presented in the dissertation.

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1. INTRODUCTION

Composites

Composites are materials which are composed of two or more phases which they remain separate and distinct in their final structure. The one phase, called matrix, is usually a low-density material whereas the other one, the reinforcement phase, gives to the composite the upgraded properties. Generally, the role of the matrix is to keep bonded the reinforcement phase and to protect it from harmful environment exposure. The great advantage of composites is that they combine the properties of their individual substances, so that the mechanical behavior of the final composite is better than the one of the initial materials. A broadly used type of composites is the polymer matrix composites (PMC).

FRP composites

FRP composites are widely used given their remarkable characteristics, such as high strength and stiffness to weight ratio, easiness in manufacture, low cost and anti-corrosion resistance. These composites consist of fibers, artificial or natural ones, embedded in a polymer matrix. The polymer matrix can be thermoset or thermoplastic polymer.

Synthetic fibers are used in applications where remarkably properties as well as high thermal resistance are essential. Nevertheless, it should be mentioned that disadvantages, such as high cost and difficulty in recycling, have limited their usage. Broadly used types of FRP composites, are glass fiber-reinforced polymer (GFRP), carbon fiber-reinforced polymer (CFRP), aramid fiber-reinforced polymer (AFRP) and basalt fiber-reinforced polymer (BFRP) composites. The usual applications of Fiber Reinforced Polymers are in marine, aerospace, automotive and construction industries and also in ballistic armor [1].

Thermal analysis

Thermal analysis studies the effect of temperature on materials' properties in accordance with time. The thermal analysis sector involves techniques, such as the Dynamic Mechanical Analysis (DMA), Thermomechanical Analysis (TMA), Thermogravimetric analysis (TGA) and Differential Scanning Calorimetry (DSC). Notably, DMA offers the possibility of determining glass transition temperature (T_g).

Literature review

Lv et. al. [2] prepared carbon and aramid fibers reinforced polyimide composites and explored the friction and wear behaviour of them in simulated space irradiation environment and start-stop friction process.

By performing static and dynamic three point bending tests, Jia et. al. [3] studied the effect of temperature, in the range from – 100 °C to 100 °C, on the mechanical properties of unidirectional CFRP composites. Notably, the static three-point bending results revealed that CFRP composites had higher flexural strength and flexural strain at break at lower temperatures [3].

Alam et. al. [4] presented a review, including 336 references, on fatigue of carbon fibre reinforced plastics.

The possibility of using carbon fiber and Kevlar[®] fiber woven composites as materials for cryogenic tanks was explored by Islam et. al. [5]. In particular, by Vacuum Assisted Resin Transfer Molding (VARTM) process, carbon fiber and Kevlar[®] woven composites were fabricated; they were then investigated, with and without cryogenic exposure, by tensile, flexural, and short beam shear experiments. The results revealed that tensile strength, tensile chord modulus, flexural chord modulus and flexural strength of carbon and Kevlar[®] fiber composites had not been significantly influenced by cryogenic exposure [5].

Song [6] prepared six types of carbon/glass fiber hybrid composites and six types of carbon/aramid fiber hybrid composites by following VARTM technique and investigated their mechanical properties. In particular, in this study, the correlation between mechanical properties and pairing effects of lamination structures was explored [6].

A study on the effect of thickness on the vacuum infusion processing of aramid/epoxy composites for ballistic applications was conducted by Nunes et. al. [7]. In their work, composites with 5, 8, 13, 18 and 23 plain-weave fiber layers were fabricated and investigated through ultrasonic C-scan inspections, optical microscopy, density and constituent content analyses. Moreover, short beam and flexural tests were performed [7].

Costa et. al. [8] used DMA, DSC, and a rheometer in order to investigate cure kinetics and rheological behavior of a carbon/epoxy prepreg.

Hossain et. al. [9] presented a three-dimensional thermodynamically consistent framework which simulates polymeric materials during their curing. In particular, based on the extension of a one-dimension equation, equations of the three-dimensional finite strain curing framework were evolved. Furthermore, cure-dependent material parameter evolutions and some numerical examples were analysed [9].

A literature review concerning the machining of carbon fiber reinforced plastics/polymers has been presented by Che et. al. [10].

By using the VARTM technique and two types of fibers, namely plain weave carbon fabrics and plain weave aramid fabrics, Wang et. al. [11] produced 12 types of composites and investigated their tensile, flexural, interlaminar shear and damping properties.

The effect of extreme low temperature conditions on the dynamic mechanical properties of carbon fiber reinforced polymers (CFRPs) was investigated by Zaoutsos and Zilidou [12]. In detail, CFRPs were manufactured by the Vacuum Assisted Resin Infusion Molding technique; after their curing they were subjected in extreme low temperatures (-40 °C) for three different periods of time: 30, 45 and 60 days. The CFRPs were then explored by three point bending and DMA experiments. The results revealed that the three point bending strength, three point bending modulus and dynamic storage modulus of the CFRPs were decreased as the time of exposure at 40 °C was increased [12].

Hazer et. al. [13] produced carbon fiber reinforced with poly (lactic acid)/polycarbonate composites, with 5, 10, 15 and 30 % percentage of carbon fibers, and two compositions of poly (lactic acid)/polycarbonate (90/10 and 50/50) blends as matrix. Particularly, these composites were investigated by DSC, TGA, tensile test, DMA, limiting oxygen index, scanning electron microscope and cone calorimeter tests.

Joven et. al. [14] used a light radiation method in order to explore the thermal diffusivity and the thermal conductivity of carbon fiber-epoxy composites, which were made with prepregs of different weave fabrics: unidirectional, plain weave and eight-harness weave.

By the VARTM technique, Dong et. al. [15] fabricated carbon fiber/epoxy plain woven composites and they studied, experimentally and numerically, their temperature-dependent thermal expansion behaviors.

The contribution of aramid fibers on the mechanical behavior of a hybrid carbon-aramid-reinforced epoxy composite was explored by Pinchiera et. al. [16]. In particular, in this study, carbon and carbon-aramid fiber reinforced composites were explored by tensile, In-plane shear, out-of-plane compression, charpy and compact tension tests.

The influence of fiber orientation on tensile properties and low cycle fatigue of intraply carbon-Kevlar reinforced epoxy hybrid composite was explored by Hashim et. al. [17].

By implementing the hand lay-up method, Hossain et. al. [18] produced five different types of twill woven Carbon-Kevlar reinforced epoxy composites. The affection of the ply variation, from 1 to 5 plies, on the tensile strength, bending strength, tensile modulus, bending modulus, and impact strength of these composites were studied. Moreover, thermo gravimetric analysis was used for the evaluation of composites' thermal stability. The results revealed an increasing in the flexural strength of the composites, as the number of the plies was increased while the composite with the three plies achieved the highest tensile and impact strength [18].

Mahnken and Dammann presented a three-scale framework for fibre-reinforced-polymer curing [19].

Nikforooz et. al. [20] explored the effect of the temperature on the static tensile behavior of continuous glass fiber-reinforced thermoplastic laminates. Moreover, the T_g of the E-glass/polyamide composites was determined by DSC, DMA and TMA [20].

The influence of 10 cycles of fatigue (between 100-50000) and then accelerated ageing in tap and artificial seawater for 4, 20 and 40 days on the tensile properties of glass and Kevlar fibers reinforced epoxy composites was investigated by Menall et. al. [21].

Ni et. al. [22] inserted aramid non-woven fabric (ANF), with a thickness of 30 μm and a face density of 16 g/m^2 , into CFRP laminates as interlayers. They investigated the influence of ANF number and arrangement on the dynamic mechanical properties, interlaminar shear strength, flexural modulus, flexural strength, compression after impact, type I interlaminar fracture toughness, and type II interlaminar fracture toughness of the composites. Remarkably, it was found that the CFRP with 1ANF had a higher flexural strength than the control CFRP composite; in comparison with the control CFRP composite, the CFRP samples with 3 or 7 ANF had approximately the same or slightly lower flexural strength, respectively [22].

Cecen et. al. [23] studied temperature dependence of heat capacity and thermal conductivity of polyester and epoxy-based composites, reinforced with non-crimp stitched glass, carbon and aramid fabrics by heat-flux DSC [23].

Objectives of the thesis

First experimental part

The properties of PMC, with continuous fibers as reinforcement phase, depend to a great extent on the orientation of fibers. As an example, unidirectional FRP composites exhibit high tensile strength in the longitudinal fiber direction but contrastively low tensile strength in the transverse direction whereas the woven FRP composites present good properties in both directions of fibers. In the current knowledge of the author, a research study exploring the dynamic mechanical properties of glass fiber/epoxy composites which have unidirectional fibers in the longitudinal or transverse direction or woven at 0° or 45° has not been performed so far. It should be pointed that, due to the viscoelastic nature of the matrix of CFRP and GFRP composites, these need to be explored in a broad range of temperature, for their dynamic mechanical properties to be absolutely predictable.

In this study, a comparison between the viscoelastic properties of GFRP composites, which contain unidirectional fibers, in longitudinal or transverse direction, and woven fibers at 0° or 45° took place. Specifically, by using prepreg materials and vacuum bag method, GFRP and CFRP composites were fabricated, and their dynamic mechanical properties were investigated, in order to determine the optimal fibers' type and orientation at elevated temperatures. Moreover, the T_g of these composites was determined by two different methods: peak of loss modulus and $\tan\delta$ curves.

Second experimental part

The natural origin fibers, like the basalt, when they are embedded in a polymer matrix composite, in percentage over 50%, provide to it biodegradable characteristics. The latter is nowadays becoming more and more important due to environmental concerns. Furthermore, in order to gain a better understanding of FRP composite, due to its viscoelastic matrix, several thermal analysis techniques should be employed, in order to study and characterize it. Based on the literature review, a research study on BFRP composites, with volume fraction of fibers (Φ_f) over 50%, with adequate experimental results, both on mechanical and thermal behavior of these compounds, appears to be lacking. In the present study, BFRP composites, consisting of 46.4% epoxy and 53.6% of basalt fibers, in 20 layers, were successfully prepared by a hand lay-up compression molding combined technique and by DMA; their storage modulus, loss modulus, $\tan\delta$, and T_g were determined. TMA creep-recovery and TMA stress-relaxation tests were also performed whereas decomposition of basalt fibers and BFRP composites as well as weight fraction of the basalt fibers and epoxy matrix were determined by TGA, in air and N_2 atmosphere, respectively. Moreover, the mechanical behavior of BFRP composites was explored following tension and three-point bending experiments.

Third experimental study

The post-cure process is a cheap method for improving thermal behaviour of cold-cured polymer matrix composites. Moreover, due to their viscoelastic nature, post-cured FRP composites need to be explored by many thermal analysis techniques, so as valid conclusions on their thermal resistance to be drawn. In terms of performance, carbon and aramid fibers are ranked top on synthetic fibers' classification and they are used in high demanding applications. It needs to be noted that in case the properties of FRP composites are compared, it is essential these composites to have same volume fraction of fibers (Φ_f), and to consist of reinforcement phase which has the same weave, weight, and thickness. For this purpose, in this study CFRP, AFRP and (aramid/carbon fiber reinforced polymer) ACFRP composites were prepared by using 161 g/m² twill 2/2 aramid fibers fabric and 160 g/m² twill 2/2 carbon fibers fabric. Similarity of both fabrics, in terms of weight, weave and thickness, allows for a direct comparison between the properties of the composites, so that valid conclusions regarding advantages of each composite structure to be drawn.

Contribution of the thesis to science and practice

In this dissertation, various types of fibers are combined with epoxy matrices so as the positive and negatives of each composite structure combination to be determined. Special attention is given on thermal analysis of FRP composites, with the target to be potential composite materials for applications in aerospace industry. Also, as the mechanical behavior of FRP composites is always a critical factor, various mechanical properties of them are determined.

2. FIRST EXPERIMENTAL PART: STUDY ON DYNAMIC MECHANICAL ANALYSIS OF GLASS OR CARBON FIBER/EPOXY COMPOSITES

This study has been presented in the following journal:

KARVANIS, Konstantinos, Soňa RUSNÁKOVÁ, Milan ŽALUDEK and Alexander ČAPKA. Preparation and Dynamic Mechanical Analysis of Glass or carbon Fiber/Polymer Composite. International Conference on Smart Engineering Materials (ICSEM 2018), *IOP Publishing IOP Conf. Series: Materials Science and Engineering* [online], 2018, 362, 012005, DOI:10.1088/1757-899X/362/1/012005.

In this study, by using prepreg materials and vacuum bag oven technique, GFRP and CFRP composites were prepared while their dynamic mechanic properties, such as the storage modulus, loss modulus, $\tan\delta$ and glass transition temperature were determined by DMA.

Materials

During the preparation of GFRP and CFRP composites, the prepreps were placed in the moulds in various layers numbers, so as the thickness of the compounds to be variable. In detail, the carbon and glass fibers were in unidirectional or woven fabric type. The glass fiber epoxy prepreps, both the unidirectional and woven types, were fabricated by the Delta-Preg S.p.A. by the resin DT 806R while the carbon fiber prepreps were prepared by Impregnatex Compositi S.r.l. by using the resin IMP 503Z. The unidirectional prepreg were placed in longitudinal or transverse direction and the woven prepreg were oriented at 0° (fibers at $0^\circ/90^\circ$) or at 45° (fibers at $-45^\circ/+45^\circ$). The GFRP and CFRP composites were produced by vacuum bag oven method.

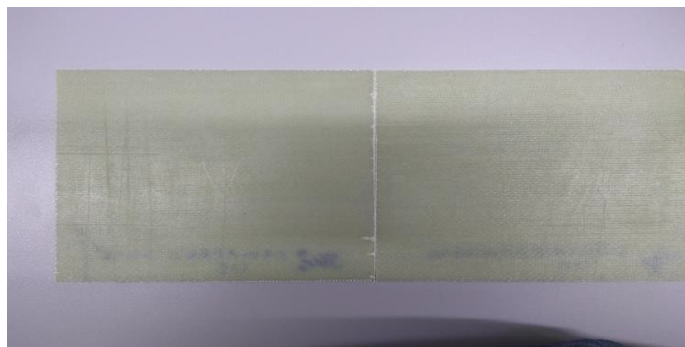


Figure 2.1: GFRP composite plates

Results

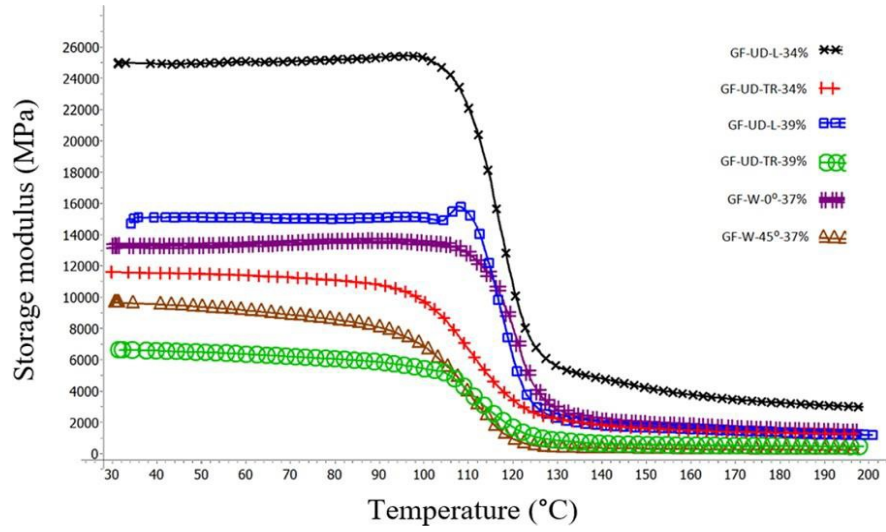


Figure 2.2: Storage modulus of the GFRP composites as a function of the temperature in the range 30-200 °C

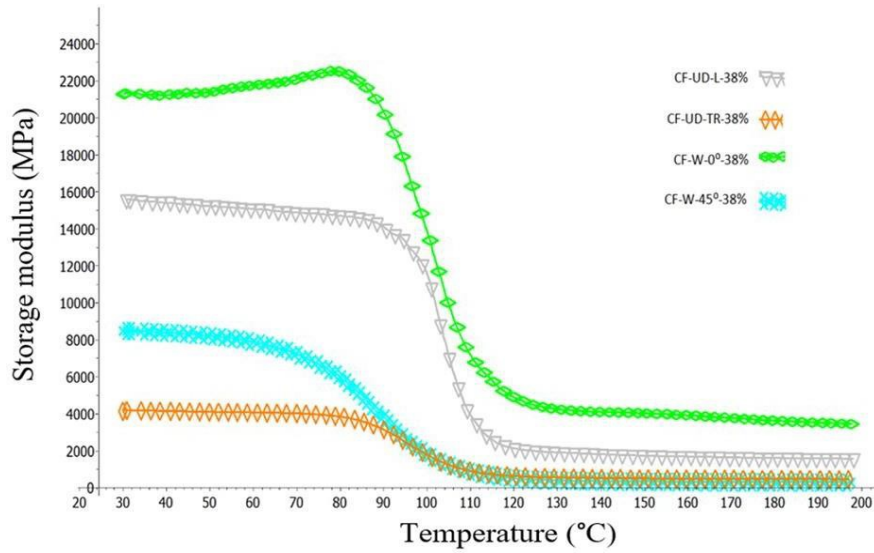


Figure 2.3: Storage modulus of the CFRP composites as a function of the temperature in the range 30-200 °C

Glass transition temperature (T_g)

Table 2.1 T_g of the GFRP and CFRP composites

	T_g values obtained by loss modulus peak (°C)	T_g values obtained by $\tan\delta$ peak (°C)
GF-UD-L-34%	116.91	120.54
GF-UD-TR-34%	111.69	119.85
GF-UD-L-39%	117.47	121.27
GF-UD-TR-39%	111.68	121.83
GF-W-0°-37%	120.79	123.66
GF-W-45°-37%	110.46	120.01
CF-UD-L-38%	103.08	107.54
CF-UD-TR-38%	96.27	104,96
CF-W-0°-38%	100.97	106.56
CF-W-45°-38%	88.93	101.42

Conclusions regarding the GFRP and CFRP composites

The overall conclusions of this research study are the following:

1. The principal research finding of this study is that when placed in the longitudinal direction, the glass fibers contribute towards to epoxy matrix composite exhibiting much higher storage modulus and loss modulus than if they are placed in the transverse direction.
2. As the volume fraction of the glass fibers in an epoxy matrix composite increases, the composites exhibit higher storage and loss modulus.
3. In DMA experiments under three-point bending configuration, the low thickness of CFRP composites is a restrictive factor.
4. The vacuum bag oven method is characterized as a cheap and efficient method for fabrication of GFRP and CFRP composites. In particular, by this method, the increased cost of autoclave is avoided while the produced composites display remarkably dynamic mechanical properties.
5. The GFRP and CFRP composites exhibited remarkably high T_g , fact which classifies them as potential materials in high demanding applications, such as in aircraft industry, and generally in cases thermal resistance is a critical factor.

3. EXPERIMENTAL PART 2: STUDY ON PREPARATION, THERMAL ANALYSIS, AND MECHANICAL PROPERTIES OF BASALT FIBER/EPOXY COMPOSITES

This research study has been published in the journal *Polymers*:

KARVANIS, Konstantinos, Soňa RUSNÁKOVÁ, Ondřej KREJČÍ and Milan ŽALUDEK. Preparation, Thermal Analysis, and Mechanical Properties of Basalt Fiber/Epoxy Composites. *Polymers* [online], 2020, vol. 12, iss. 8. 1785. Available from: <https://doi.org/10.3390/polym12081785>

General information about this study

In this study, BFRP composites, with epoxy matrix and twill 2/2 weave basalt fibers fabric, in 20 layers, were prepared by a hand lay-up compression molding combined technique. In the experimental part, DMA was exhibited in the range 30-180 °C and at 1, 5 or 10 Hz and based on corresponding temperatures of the peak of loss modulus and $\tan\delta$ curves, the T_g of these composites was determined. Moreover, TMA was performed in the modes of creep recovery and stress-relaxation experiments whereas thermal decomposition of the basalt fibers and BFRP composites in air and nitrogen atmosphere, respectively was investigated by TGA experiments. Based on TGA experiments, it was calculated that $\Phi_f = 53.66\%$. Also, tensile and flexural characteristics of the BFRP composites were determined by tension and three-point bending experiments.

Materials

The epoxy matrix of the BFRP composites, is a mixture of the Epoxy resin Epidian[®] 652 CIECH Sarzyna S.A (Cieszyn, Poland) and the hardener TFF CIECH Sarzyna S.A (Cieszyn, Poland), in a mixing ratio of 100:27 parts per weight and the reinforcement phase is a fabric of basalt fibers 235 g/m², in twill 2/2 weave, supplied by Havel Composites (Cieszyn, Poland) (Figure 3.3). It needs to be noted that the specific weight of the used basalt fibers is 2.67 g/cm³. The steps followed for the preparation of the BFRP composites are described below. By the hand lay-up technique, a laminate, composed of 20 layers of polymer fibers, was prepared. In the next step, this laminate was placed in a compression machine, where it was pressed under 20 MPa for 24 h, at a laboratory temperature of 24 °C. Then, the composite plate was left for curing at room temperature for a week, and finally specimens were cut in the desired dimensions by water jet and mechanical cutting.

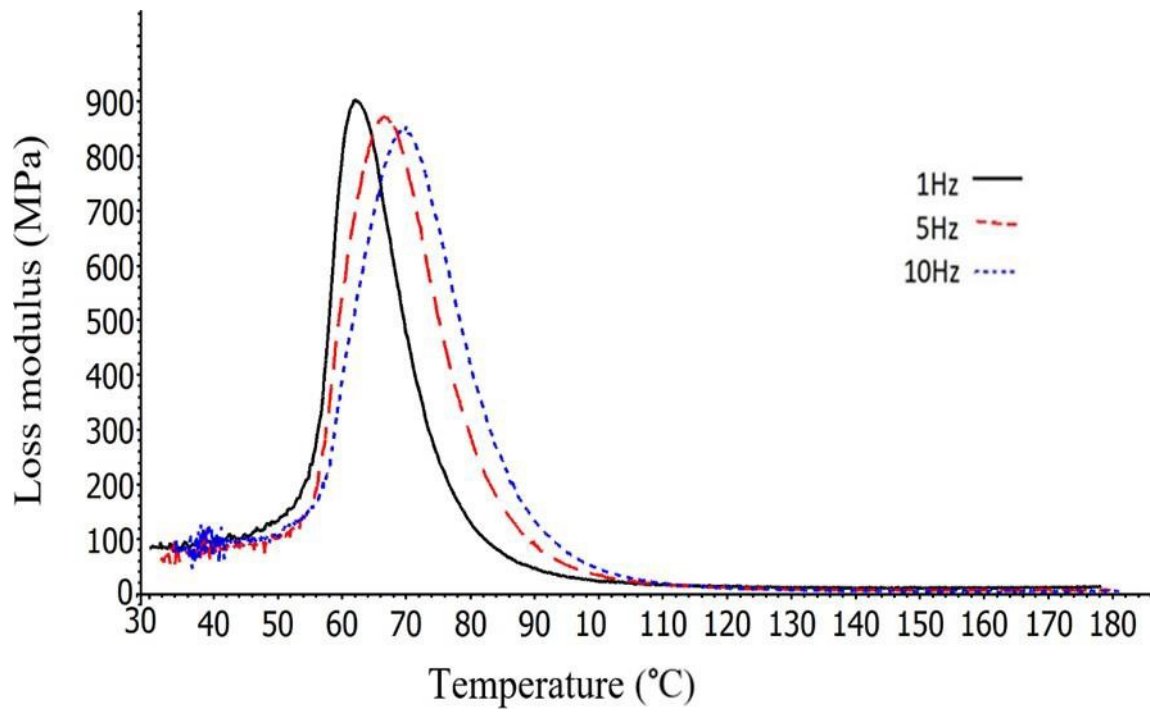


Figure 3.1: Loss modulus of the BFRP composites, as a function of the temperature, at 1, 5 and 10 Hz

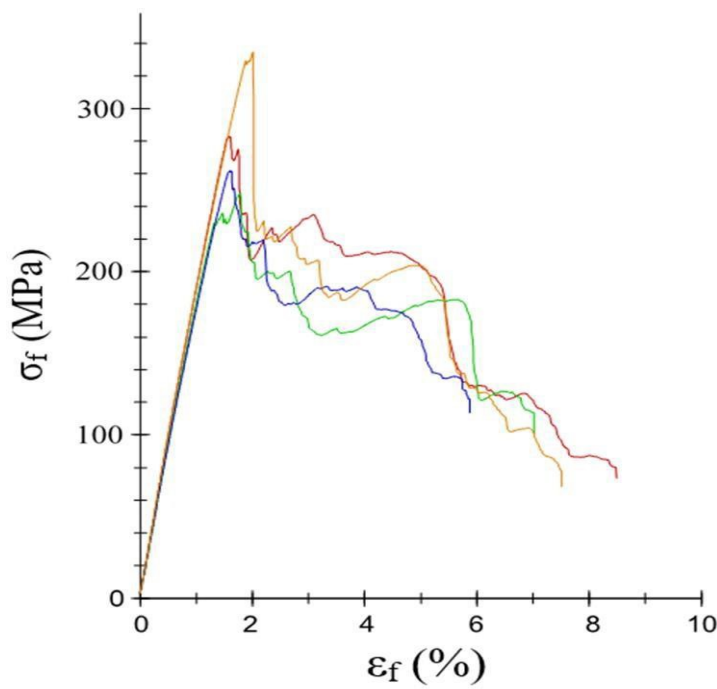


Figure 3.2: The four σ_f - ϵ_f curves of the BFRP composites

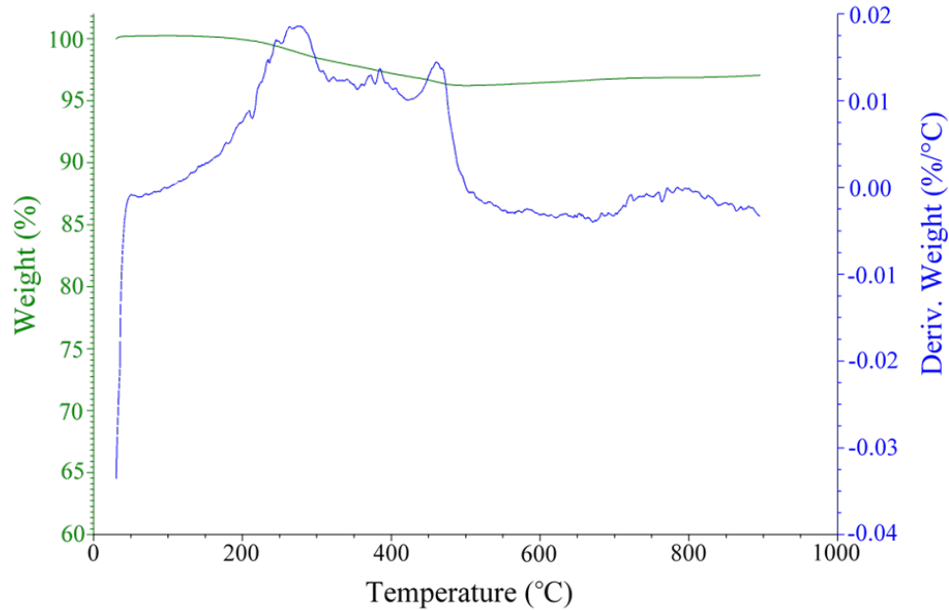


Figure 3.3: TGA results showing the weight (%) variation and weight loss rate (DTG) of the basalt fibers* in air atmosphere

Conclusions regarding the BFRP composites

Based on DMA results, it was revealed that as frequency is increased the BFRP composites achieve higher T_g and that T_g based on $\tan\delta$ is higher than T_g acquired from the loss modulus curves.

One of the significant outputs of this research is the thermal resistance of basalt fibers, as these were not thermally influenced, in oxygen or N_2 atmosphere, up to the final 900 °C of TGA experiments. This characteristic of the basalt fibers can be attributed to their origin from the earthquake lave.

Generally speaking, the BFRP composites exhibited high tensile and flexural strength, verifying a very good interfacial bond formed between basalt fibers-epoxy; the FRP composites demonstrate a low mechanical behavior in the case of a poor bond between matrix-fibers. In addition, the BFRP composite plate has a stable thickness through its entire structure (+0.1 mm). So, the novel hand lay-up compression molding method is characterized as a highly efficient and reliable technique for the preparation of FRP composites with significantly high Φ_f .

4. EXPERIMENTAL PART 3: PREPARATION, THERMAL ANALYSIS AND VARIOUS MECHANICAL PROPERTIES OF POST-CURED CARBON, ARAMID OR CARBON/ARAMID HYBRID FIBER EPOXY MATRIX COMPOSITES

The research about the AFRP composites has been published in the following journal:

KARVANIS, Konstantinos, Soňa RUSNÁKOVÁ, Ondřej KREJCI and Alena KALEDOVÁ. Thermal analysis of post-cured aramid fiber/epoxy composites. Reviews on advanced materials, 2021, volume 60. DOI: 10.1515/rams-2021-0036

Purpose of the research study and general information

In this research work, applying the vacuum infusion process (VIP), eight layered CFRP, ACFRP and AFRP composites, with twill 2/2 weave fibers fabric and epoxy matrix, were prepared and then post-cured under specific heating/cooling rates. The important point is that both fiber fabrics, carbon and aramid, have the same weave, thickness and dry weight; in this sense, the properties of composites can be compared and the advantages of each composite structure are revealed. The results are presented in two result parts: in the first part, the AFRP composites are investigated in depth with various thermal analysis techniques, so as the effect of the post-cure process on them to be deeply explored; in the second part, a comparison between thermal behavior and mechanical properties of the CFRP and ACFRP composites is taking part.

Materials and preparation method

For the preparation of the CFRP, AFRP and ACFRP composites, carbon and aramid fiber fabrics, both produced by the company C. Cramer & Co, were used. These have the same characteristics, namely 160 sqm weight, twill 2/2 weave and thickness around 0.36 mm, In particular, the carbon fabric is the product code Style 442, composed of the fibers Pyrofil™ TR30S T-Size 3K 200tex (Mitsubishi Chemical), while the aramid fabric is the product code Style 502 (C. Cramer & Co) with fibers Kevlar49 T965 (1580dtex) (DuPont). The epoxy matrix is a mixture, parts by weight 100:30, of the epoxy Biresin® CR80 (Sika®) and the hardener Biresin® CH80-2 (Sika®) which was gently stirred until homogeneity was achieved. The laminate structure of the hybrid ACFRP composite is composed of four aramid fiber fabric layers in the middle and of two carbon fiber fabric layers in up and down outer sides, respectively. The CFRP, AFRP and ACFRP composites' were produced by Vacuum Injection Process.

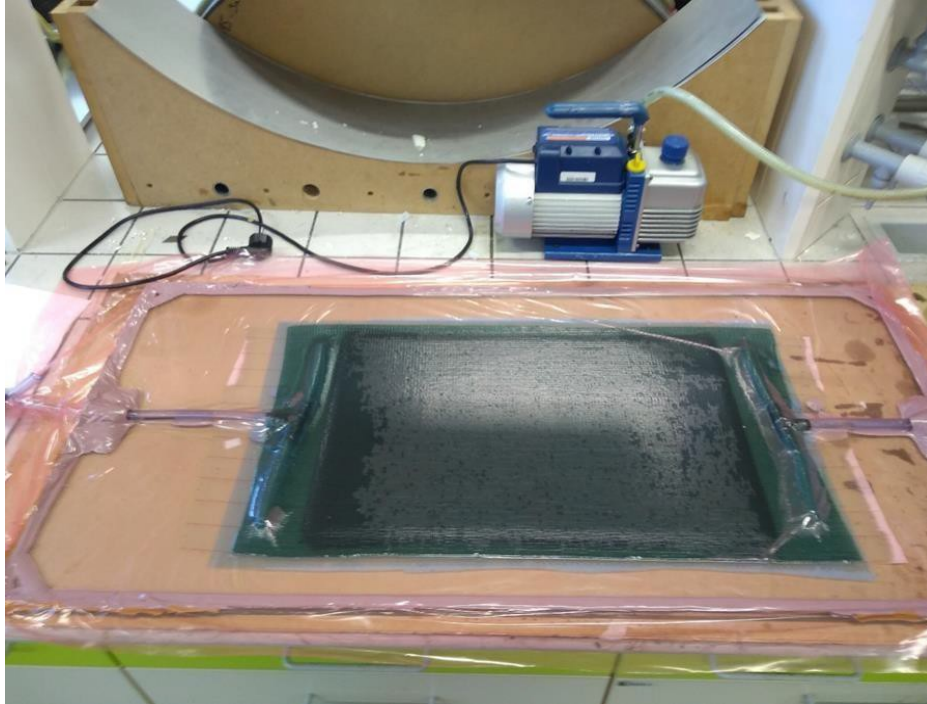


Figure 4.1: Preparation of the CFRP composite with the Vacuum Infusion Process. The photo was taken hours after the resin infusion.

Results

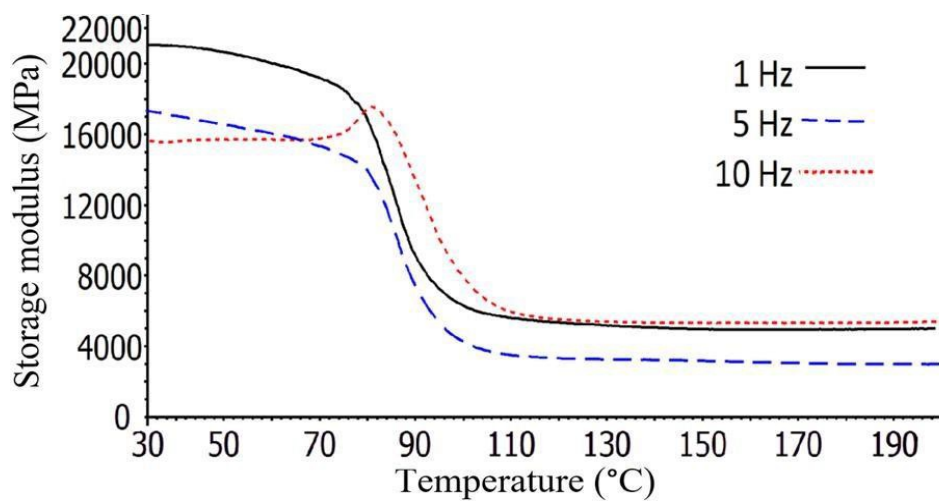


Figure 4.2: Storage modulus of the AFRP composite in the range 30-200 °C, at 1, 5 and 10 Hz

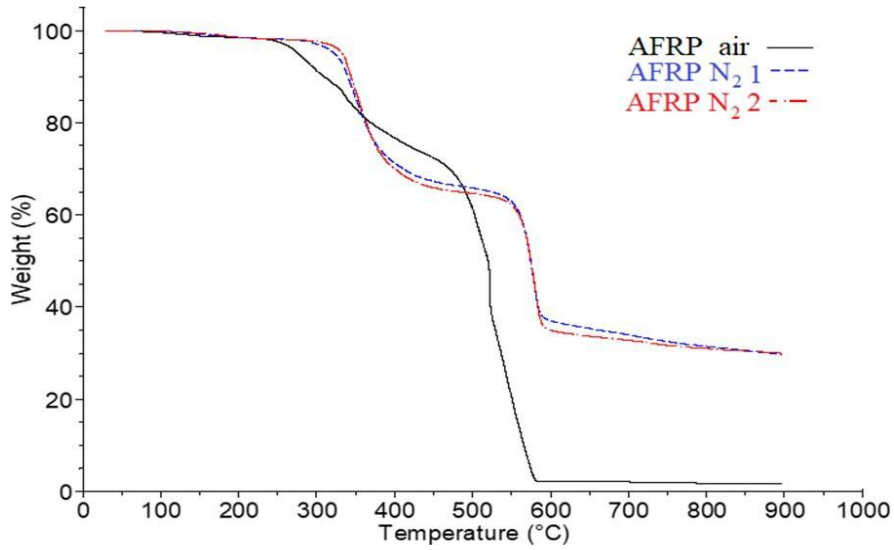


Figure 4.3: TGA results showing the weight of the AFRP composites as a function of the temperature, in air and N₂ atmosphere, respectively

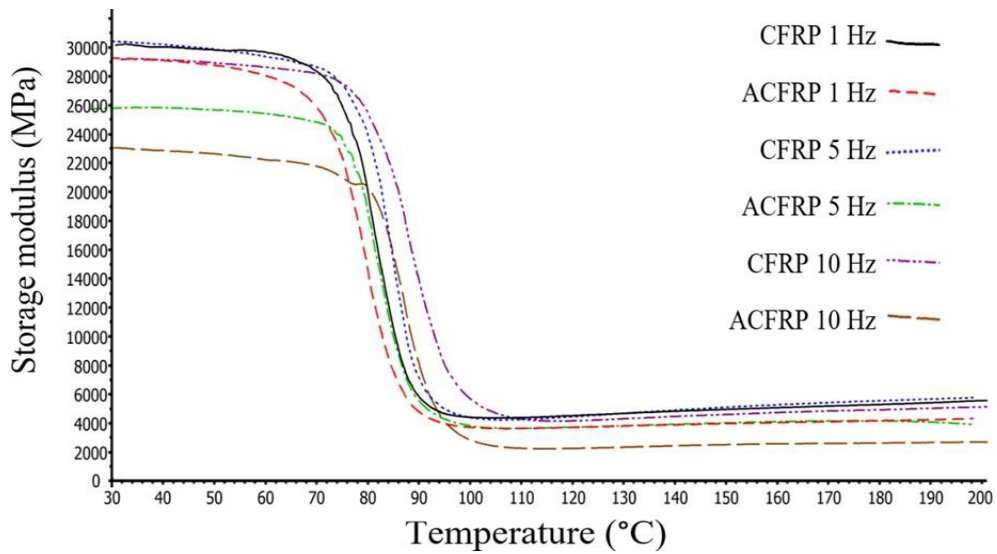


Figure 4.4: storage modulus (E') versus temperature of the CFRP and ACFRP composites under 1, 5 and 10 Hz

Table 4.1 T_g of the CFRP and ACFRP composites (based on DMA)

	T_g obtained by $\tan\delta$ peak (°C)	T_g obtained by loss modulus peak (°C)
CFRP 1 Hz	82.52	86.10
ACFRP 1 Hz	79.96	84.54
CFRP 5 Hz	84.45	87.81
ACFRP 5 Hz	82.84	85.84
CFRP 10 Hz	89.38	93.72
ACFRP 10 Hz	87.08	92.65

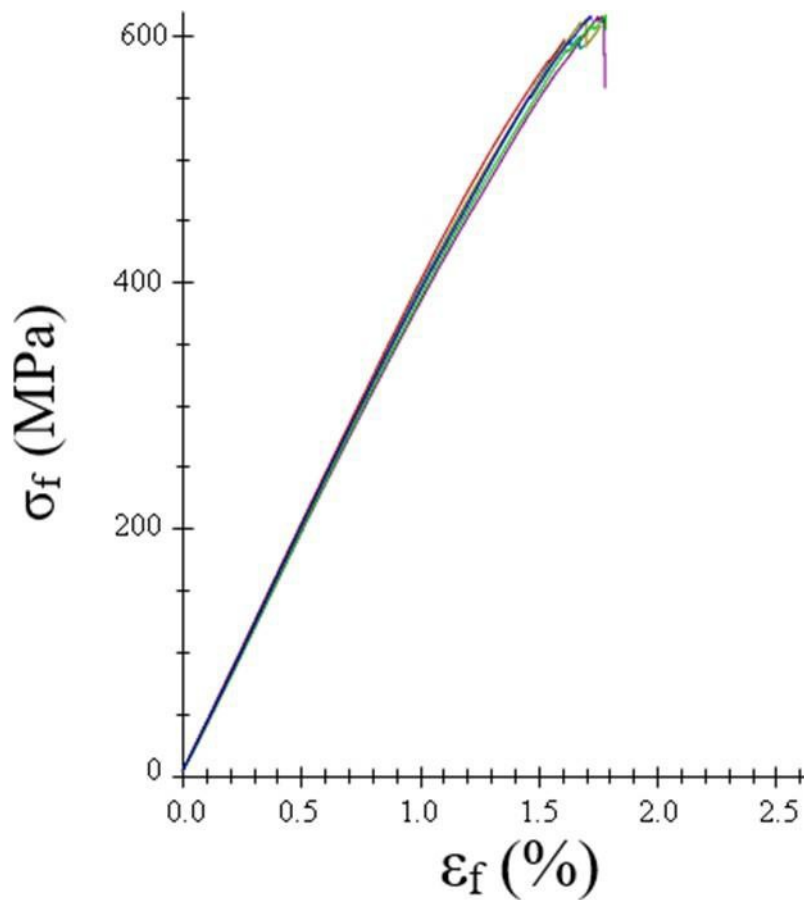


Figure 4.5: σ_f - ε_f diagram of the six three-point bending experiments on the ACFRP composite

Conclusions regarding the CFRP, AFRP and ACFRP composites

In all cases of the TGA experiments, it was found that thermal degradation in an oxidative environment, (i.e. in air atmosphere) is much more harmful than the

one due to pyrolysis (in N₂). In the TGA experiments, the very good thermal resistance of both carbon and aramid fibers was revealed. This characteristic together with the relatively high T_g of the post-cured CFRP, AFRP and ACFRP composites, in the range 80-95 °C, depending on the frequency and determination method, classify these composites as potential materials in applications where resistance in high temperatures is of great interest.

Based on the TMA creep-recovery experiments, it was revealed that the recovery of the AFRP composite structure took place immediately after the force release and that in all cases the final (plastic) deformation in the AFRP composite structure was relatively small. Relating this aspect with the overall results from the DMA experiments, it is concluded that, under the T_g, elastic behavior prevails entirely over viscous one in the AFRP composites' structure. Additionally, the creep-recovery behavior of the AFRP composite is the same at 25-50 °C, confirming hence the composite structure ability to work in this temperature range.

The exceptionally low densities of both carbon and aramid fibers, 1.77 g/m³ and 1.44 g/m³ respectively, result in a composite structure with low weight, making thus the composite highly attractive in a broad range of applications, especially in airplane industry where, nowadays, the efforts for fuel savings are of significant importance.

The results showed that a steady difference, under all frequencies and determination methods, between T_g temperatures of the CFRP and ACFRP composites; in all cases, the former composite achieved higher T_g. In terms of mechanical behavior, flexural strength, and hardness shore D, the CFRP composite demonstrated higher values than the ACFRP one. Moreover, the carbon fibers exhibited significant high thermal resistance in exposure at remarkably high temperatures, such as in 450, 460, and 470 °C in oxygen atmosphere; exceptionally, they lost approximately 5% of their weight after exposure in these temperatures for 60 minutes. According to the exhibited experiments, the overall results reveal that the hybrid CFRP composite did not manage to achieve the performance of the CFRP composite.

5. CONCLUSIONS OF THE DISSERTATION

In this dissertation synthetic fibers, namely glass, carbon and aramid as well as natural origin basalt fibers were embedded in various epoxy matrices. For the production of the composites were both used expensive production methods and materials, such as prepreg processing and more economic ways, like the hand lay-up compression moulding combined method and Vacuum Infusion Process. The purpose of the dissertation is to explore FRP composites, targeted as potential

materials in a broad range of applications, from automobile parts to aircraft applications. The experimental part of this dissertation is divided in three parts.

In the first part, the GFRP and CFRP composites verified their high quality, as these achieved significant high T_g . In detail, the GFRP composites exhibited T_g in the range 111-123 °C, depending on the type of fabric/orientation of fibers and as well as on the method followed for the T_g determination: peak of $\tan\delta$ or loss modulus curves. Remarkably, DMA revealed that, in terms of storage and loss modulus, the placement of unidirectional glass fibers in the longitudinal direction of GFRP composites is much more effective than when placed in the transverse one. Moreover, the GFRP composites with slightly higher Φ_f , exhibited higher storage and loss modulus; this points out the positive effect of glass fibers in the composite structure. Based on the DMA results, the GFRP and CFRP composites are classified as optimal materials for the production of body parts of luxury cars, marine boats along with low-speed small airplanes. However, it should be mentioned that the applications of composites produced by prepreg materials need to justify their significantly high cost.

In the second experimental part, an eco-friendly reinforcement phase, basalt fibers were embedded in an epoxy matrix, in a high $\Phi_f = 53.66\%$, applying the hand lay-up compression molding method. In terms of tensile and flexural properties, the cold-cured BFRP composites displayed very good mechanical behavior, which verifies a very good interaction between basalt fibers/epoxy matrix, despite the high Φ_f . In FRP composites industry, the introduction of fibers in a polymer matrix in a high Φ_f is always a major concern, as in many cases, high fiber volume tends to form an inappropriate interfacial bond between them and the matrix; this, in turn, results in poor overall performance of the composite. Exceptionally, the hand lay-up compression molding method is qualified as highly effective in preparation of high Φ_f FRP composites. Moreover, the basalt fibers showed excellent thermal resistance, as they were not thermally affected up to the final 900 °C of the TGA experiments, pointing out their highly usefulness in applications where thermal resistance is essential. As an example, if basalt fibers are combined with a high thermal resistance polymer matrix in high Φ_f and the formed composite is applied as structural material in the body of an airplane, in case of fire, basalt fibers will continue to offer structural support up to very high temperatures, important behavior in airplane industry. Moreover, the potential applications of basalt fibers could be in the surrounding sites of exhaust systems of cars, airplanes, and helicopters. In addition, the relative low price of basalt fibers, compared especially with the price of carbon and aramid fibers, combined with the inexpensive composites' fabrication method categorize the BFRP composites as optimal materials in low-cost applications, such as body parts of cars, motorcycles, and boats. Significantly, the natural origin of basalt fibers is another positive advantage. Specifically, these fibers will not pose the

difficult problem of recycling, after their end-cycle use, like artificial fibers, such as carbon and aramid; this is a very important factor nowadays, due to the continuously increased use of FRP composites.

In the third experimental part, CFRP, AFRP and ACFRP composites were fabricated, following the VIP process. Remarkably, both carbon and aramid fiber fabrics demonstrated the same waive, weight and thickness, so the composite properties were compared with accuracy. Moreover, in this study, an effort was made to improve thermal behavior of the composites, with a relatively low cost. In particular, after curing at 23 °C for seven days, the composites were exposed to post-curing in an oven, under specific heating-cooling rates, following the resin producer suggestions. Various thermal analysis techniques were performed on the composites, so as to determine the positives and negatives of each composite structure. Notably, by TGA, an approach for determining the composite Φ_f was followed. Both the CFRP and ACFRP composites had a similar Φ_f and hence, the results section of this study was divided in two parts: thermal analysis of the AFRP composite and comparison of the CFRP and ACFRP composite properties.

Regarding the results section on the AFRP composites, their T_g was found to be in the range of 85-95 °C. T_g appears more a transition than a specific temperature whereas the T_g of the composites was specified by various ways, DMA and DSC, and under various frequencies, justifying thus such a fluctuation. Specifically, the T_g of the AFRP composites was relatively high for epoxy matrix composites, which indicates that the low-cost post-cure process has significantly improved the thermal behavior of the composites. Moreover, aramid fibers showed very good thermal resistance in TGA under N_2 atmosphere; they were very slightly thermally influenced under 60 minutes exposure in the temperature range of 350-390 °C. Also, the aramid fiber low density of 1.44 g/cm³ results in a composite with very low weight, a factor which nowadays is very important in all transport industries due to environmental concerns; low weight will eliminate the overall weight of a vehicle, resulting hence in lower fuel consumption. Moreover, the experimentally determined T_g values are in agreement with those provided by the resin producer datasheets. It is concluded that the AFRP composites are fully and appropriate cured while the cure-post-cure process, specifically the particular heating-cooling rates which were followed are highly effective for this kind of materials.

In the second results section, a comparison between the CFRP and ACFRP composites was realized. The overall results show that the CFRP appears to have a better mechanical behavior than the ACFRP composite.

6. RELATING THE SCIENTIFIC RESULTS WITH PRACTICAL APPLICATIONS

- Regarding the first experimental part, the GFRP and CFRP composites exhibited remarkably high T_g , fact which classifies them as potential materials in high demanding applications, such as in aircraft industry, and generally wherever thermal resistance is a critical factor.
- The relatively high T_g of the post-cure AFRP composite, which is in the range of 85-95 °C and depending on the frequency and determination method, classifies these composites as potential materials in applications where resistance in high temperatures is of great interest.
- The low density of aramid fibers, 1.44 g/cm³, results in a composite with very low weight, a very important factor in all transport industries nowadays due to environmental concerns; the low weight is expected to eliminate the overall weight of a vehicle, resulting hence in lower fuel consumption.
- The excellent thermal resistance of basalt fibers, both in air and N atmosphere, makes their introduction in high performance polymer matrix, like bismaleimide or phenolic, essential, in order for a FRP composite with excellent overall thermal behavior to be formed. For sure, this composite could be target as potential material for body parts of airplanes. Moreover, the potential applications of basalt fibers could be in the surrounding sites of exhaust systems of cars, airplanes, and helicopters.
- Exceptionally, the hand lay-up compression molding method is qualified as highly effective in preparation of FRP composites with high Φ_f . Moreover, the method's low cost and simplicity classifies it as suitable for both low cost uses, such as every day and high demanding applications, for example marine and automotive industries.
- Future research on a hybrid high performance polymer matrix composite, consisted of alternately layers of carbon-aramid fiber fabrics and its comparison with pure AFRP, CFRP composites, where all of them have reinforcement phases with the same weight, weave and thickness appears an optimistic idea.

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LIST OF SYMBOLS, ACRONYMS AND ABBREVIATIONS

FRP	Fiber-reinforced polymer
DMA	Dynamic Mechanical Analysis
TMA	Thermomechanical Analysis
TGA	Thermogravimetric Analysis
DSC	Differential Scanning Calorimetry
T _g	Glass transition temperature
BFRP	Basalt fiber-reinforced polymer
Φ _f	Volume fraction of fibers
PMC	Polymer matrix composites
GFRP	Glass fiber-reinforced polymer
CFRP	Carbon fiber-reinforced polymer
AFRP	Aramid fiber-reinforced polymer
VARTM	Vacuum Assisted Resin Transfer Molding
CFRPs	Carbon fiber reinforced polymers
ANF	Aramid non-woven fabric
%	percentage in 100
GF-UD-L-34%	Unidirectional longitudinal glass fiber-reinforced polymer composite with 34% matrix
GF-UD-TR-34%	Unidirectional transverse glass fiber-reinforced polymer composite with 34% matrix
GF-UD-L-39%	Unidirectional longitudinal glass fiber-reinforced polymer composite with 39% matrix
GF-UD-TR-39%	Unidirectional transverse glass fiber-reinforced polymer composite with 39% matrix
GF-W-0°-37%	Woven at 0° (fibers at 0°/90°) glass fiber-reinforced polymer composite with 37% matrix
GF-W-45°-37%	Woven at 45° (fibers at -45°/+45°) glass fiber-reinforced polymer composite with 37% matrix
CF-UD-L-38%	Unidirectional longitudinal carbon fiber-reinforced polymer composite with 38% matrix
CF-UD-TR-38%	Unidirectional transverse carbon fiber-reinforced polymer composite with 38% matrix
CF-W-0°-38%	Woven at 0° (fibers at 0°/90°) carbon fiber-reinforced polymer composite with 38% matrix
CF-W-45°-38%	Woven at 45° (fibers at -45°/+45°) carbon fiber-reinforced polymer composite with 38% matrix
E'	Elastic (storage) modulus

°	Degrees
Hz	Hertz
N	Newton
°C	Celsius
min	Minute
$\tan\delta$	Loss factor
g/m^2	Grammar per square meter
g/cm^3	Grammar per cubic centimeter
MPa	Megapascal
h	Hour
mm	Millimeter
K/min	Kelvin per minute
σ_{fmax}	Maximum flexural stress
δ	Deformation
σ_f	Flexural stress
ε_f	Flexural strain
σ	Tensile stress
F	Force
σ_{max}	Maximum stress
E	Young modulus
VIP	Vacuum infusion process

APPENDICES



LIST OF AUTHORS' PUBLICATIONS

1. **Karvanis Konstantinos**, Rusnáková Soňa, Krejčí Ondřej, Žaludek Milan. Preparation, Thermal Analysis, and Mechanical Properties of Basalt Fiber/Epoxy Composites. *Polymers*. 2020, 12(8):1785. Available from: <https://doi.org/10.3390/polym12081785>
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