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**CZECH SOCIETY FOR MECHANICS,
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and

**CZECH TECHNICAL UNIVERSITY IN PRAGUE,
FACULTY OF MECHANICAL ENGINEERING**

the international conference

POLYMER COMPOSITES 2023

**The conference is organized under the auspices
of the Mayor of Tábor Ing. Štěpán Pavlík.**

The aim of the Conference is to enable specialists from research and industry to present actual knowledge and trends in research and development, analyses, materials, technology, manufacturing and applications of composite materials.





SAERTEX®



Organization and program committee of Polymer Composites 2021 conference

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Detailed information:

<https://polymer-composites.cz/en/polymerni-kompozity-2023/>

ORGANIZATION HINTS

Date: May 23 – 24, 2023

Place:

LH Hotel Dvořák Tábor Congress & Wellness

Hradební 3037,

Tábor 390 01

tel.: 381 212 221

e-mail: recepce@lhdvoraktabor.cz

www: lhdvoraktabor.cz



Conference languages: Czech, Slovak and English

Accommodation:

Accommodation is not included in the participation fee. Accommodation is provided at the LH Hotel Dvořák in Tábor (lhdvoraktabor.cz). However, participants are asked to order the overnight directly in this hotel themselves with the use of promo code **PC2023**.

Participation fee:

- Conference proceedings
- Drinks and lunch during breaks
- Evening party

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POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



DEVELOPMENT AND PREPARATION OF THE PRODUCTION OF PLASTIC PARTS WITH THE HELP OF ADVANCED SIMULATIONS

VÝVOJ A PŘÍPRAVA VÝROBY PLASTOVÝCH DÍLŮ S POMOCÍ POKROČILÝCH SIMULACÍ

TOMÁŠ ČURDA¹, PETR SŮVA²

CAE simulations and optimization are now becoming one of the most important factors influencing the quality of plastic parts and the economics of their production. Today, almost every manufacturer is using these technologies in some way, but the critical benefit comes when they are applied to the entire development and production workflow. This does not necessarily mean an increase in the cost of CAE technologies.

CAE simulace a optimalizace se dnes stávají jedním z nejvýznamnějších faktorů ovlivňující kvalitu plastových dílů a ekonomiku jejich výroby. Dnes téměř každý výrobce tyto technologie nějakým způsobem používá, ale rozhodující benefit přichází až při nasazení na celé workflow vývoje a výroby. Nemusí to ale nutně znamenat nárůst vynaložených nákladů na CAE technologie.

KLÍČOVÁ SLOVA

CAE simulation, Optimization, Simulation-driven Design, Plastic injection molding, Mold flow analysis, Automated variant analysis and optimization, part quality, production, ...

ÚVOD

Plastové díly jsou naprostě běžnou součástí mnoha výrobků, od automobilů přes spotřební elektroniku po lékařská zařízení. Vývoj těchto dílů však není snadný, protože se musí brát v úvahu mnoho faktorů, jako jsou materiály, design, funkční požadavky a výrobní procesy.

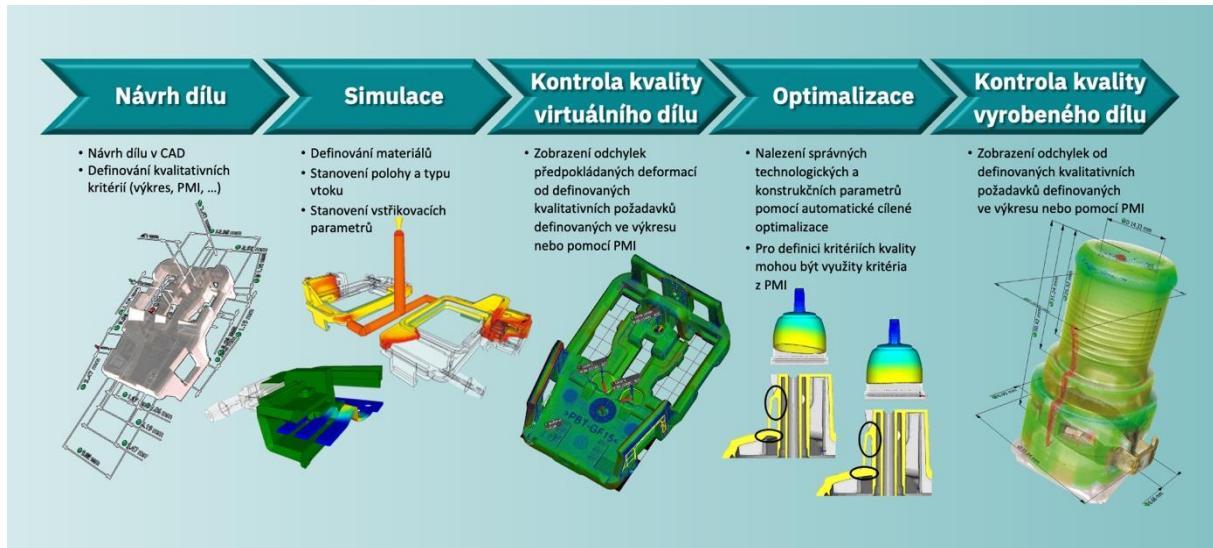
Protože simulace a optimalizace dnes neznamenají jen podpůrné procesy při konstruování výrobku a při přípravě jeho výroby (např. simulace procesu vstříkování do formy), budeme se v tomto článku věnovat celkovému workflow zajíšťující na výstupu výrobek optimalizovaný z pohledu technických parametrů, jakosti, cen a dnes i ekologické zátěže.

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V tomto článku se zaměříme na význam CAE simulací pro vývoj plastových dílů a jak mohou pomoci zlepšit jejich kvalitu, snížit náklady a urychlit výrobu.

Více k tomuto tématu můžeme diskutovat na stánku *Advanced Engineering s.r.o.* a *Plasty Gabriel s.r.o.* v rámci konference Kompozitní polymery 2023 a detailněji diskutovat funkcionality a přínosy níže zmiňovaných nástrojů od společností *Altair* a *Simcon* či zkušenosti z inženýrských projektů. Dobrou zprávou je, že nyní lze dále zmiňované technologie zastřešit jedinou licencí a významně ušetřit.



1. NÁVRH A KONSTRUKCE DÍLU

Jestliže v minulosti návrh dílu představoval proces, na jejímž konci ale zároveň v jakémse středobodu byl výrobní výkres, v lepším případě 3D geometrický model, dnes zaznamenáváme posun směrem ke komplexnějšímu digitálnímu modelu, chcete-li dvojčeti připravovaného výrobku. Ve fázi konceptu a konstrukce je asi nejvýznamnějším posunem přístup *Simulation-driven Design*, který konstruktérovi dává do ruky nástroje, které mu umožňují navrhovat díly rovnou na základě fyzikálních požadavků – např. silového namáhání. Jedná se o nástroje pro generativní design, topologické, topografické, tvarové, rozměrové a parametrické optimalizace. Takže požadovaný plastový díl bude mít s ohledem na požadavky provozu optimálně navržené proporce, rozměry, žebrování, výztuhy a podobně.

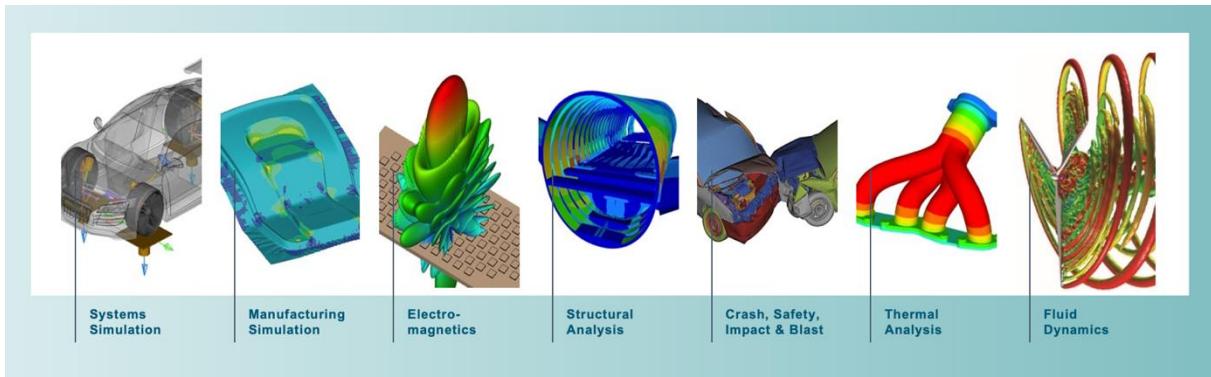
V raných stádiích vývoje je třeba rozhodnout i o výrobní technologii a jí od začátku přizpůsobovat konstrukční řešení. Zde se výše uvedený přístup vývoje za pomocí simulací naplňuje ve svém úplném názvu – *Simulation-driven Design for Manufacturing*. Ten doplňuje podporu konstrukce o ověřování výrobiteľnosti a provádí předběžné simulace výrobního procesu s cílem odhalit místa, ve kterých mohou vznikat výrobní vady. Pro oblast plastů je k dispozici podpora vstříkování plastů a PU pěny, a dále protlačování polymerů. Společnost Altair podporuje *Simulation-driven Design* produktovou rodinou *Altair Inspire*.



2. SIMULACE

Historicky vznikly počítačové simulace ve vývoji výrobků jako náhrada fyzických testů ve zkušebně v rámci ověřování. Dnes jsou simulace integrální součástí vývojového procesu počínaje koncepční fází popsanou v předchozí kapitole. Pokročilé simulace, zejména multi-fyzikální nebo vice-kriteriální optimalizace ale stále zůstávají doménou výpočtařů-specialistů, kteří se do vývoje v jednotlivých iteracích zapojují. Z pokročilých simulací týkajících se plastových dílů vzpomeňme např. virtuální pádové zkoušky (drop-testy) např. pro spotřební elektroniku, výpočty únavy a životnosti, termální či fluidní analýzy pro potrubní systémy nebo simulace EM polí při vývoji antén a jejich zakrytování. Protože řada plastových dílů je vystavena i významnému mechanickému namáhání, přicházejí v úvahu i strukturální analýzy kompozitních materiálů, kde vstupem jsou výsledky výrobní simulace určující orientaci výstužných vláken. Specifickou disciplínou je pak návrh vrstvených kompozitových struktur (laminátů), kde lze materiál modelovat na úrovni vláken a matrice, na úrovni jednotlivých vrstev a jako homogenizovaný na úrovni celého dílu. Funkcionalitu pro pokročilé multi-fyzikální simulace najeznete v prostředí *Altair HyperWorks* s řešiči *OptiStruct*, *Radius*, *AcuSolve*, *Feko* a dalšími.

Za předpokladu, že jsme v předchozích krocích zajistili podmínky vyrobitelnosti plastového dílu, lze „zmrazený“, design dílu použít pro komplexní a detailní analýzu technologických (výrobních) parametrů a kontrolu předpokládané kvality dílu. Program CADMOULD také ověří, případně navrhne, vhodnou polohu a počet vtoků i jejich správné dimenzování. Dále ukáže problematická místa z hlediska plnění, odvodu tepla z povrchu tvarové dutiny i předpokládané kvality dílu.



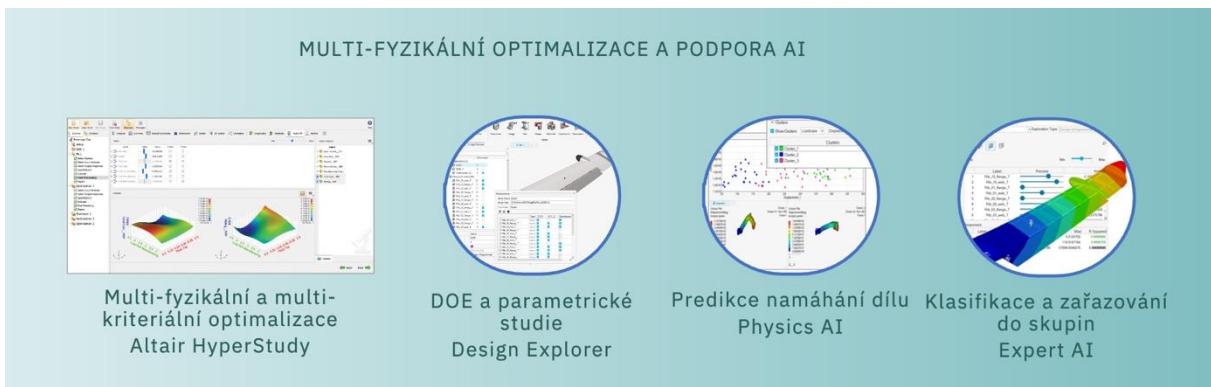
3. OPTIMALIZACE

U návrhu dílů vyvíjených pro hromadnou nebo velkosériovou výrobu se vyplatí konstrukční řešení co nejvíce optimalizovat – zajistit co nejvyšší kvalitu dílu, ale ušetřit na hmotnosti a výrobních nákladech. Přicházejí ke slovu pokročilejší varianty strukturálních optimalizací zmiňovaných u koncepčního vývoje (topologické, topografické, tvarové, rozměrové, parametrické) ale i další. Bez zajímavosti není, že s topologickými optimalizacemi se setkáváme již i u fluidních úloh (CFD). Specifické jsou opět laminátové kompozity, u kterých optimalizujeme tloušťky vrstev, směr vláken, materiály jednotlivých vrstev a pořadí vrstev, aby vyvíjený díl měl dostatečnou pevnost, ale i požadovanou tuhost. Tento typ optimalizací naleznete zejména v nástroji *Altair OptiStruct*.

Nejpokročilejšími optimalizacemi ve vývoji jsou multi-fyzikální a multi-kriteriální, kdy se snažíme najít ideální kompromis všech sledovaných parametrů – geometrických, fyzikálních i dalších. Anebo zjistit citlivost výstupů na jednotlivé vstupní parametry (DOE studie). Tyto výpočty fungují v režii nadřazeného řešiče (*Altair HyperStudy*), který podle pokročilých algoritmů vtipovává kombinace vstupních parametrů, volá pro jednotlivé fyziky příslušné řešiče a výsledky vyhodnocuje. Výhodou je, že lze zapojit různé řešiče různých výrobců. Takto sestavené optimalizační výpočty představují vysoký počet běhů a jsou tedy náročné na čas potažmo výpočetní výkon. Proto nově do algoritmů vstupují technologie strojového učení pro řádově rychlejší predikci výsledků.

Pozadu nezůstávají optimalizace výrobního procesu. Nejčastěji se provádí hledání nejlepších technologických parametrů s ohledem na požadovanou kvalitu dílu. Dále jsou prováděny optimalizace konstrukce dílu a formy za účelem dosažení stanovené kvality vyráběného dílu, spotřebu materiálu a energie a tím i cenu dílu. Pro tyto účely se využívá unikátní systém i od společnosti Simcon.

Po získání optimalizované konstrukce dílu z hlediska designu, mechanických a fyzikálních vlastností je nutné pověřit tento stav s ohledem na konstrukci formy, výrobu samotného dílu, a především na výslednou kvalitu vyrobeného dílu. Pomocí unikátního systému *VARIMOS* zjistíme potřebné nastavení technologických parametrů pro dosažení požadované kvality dílu a dodržení technologických a ekonomických požadavků.



4. KONSTRUKCE A VÝROBA FORMY

Pokud jsme v předchozích krocích vynechali návrh a kontrolu vtokového systému a jeho dimenzování, musíme jej navrhnout a ověřit nyní. Stejně jako temperační systém a ověření kvality dílu. Jestli nám výsledky simulací v programu *CADMOULD* ukáží nějaká problematická místa nebo přímo nedostatky v konstrukci dílu či formy, je vhodné všechny problémy opravit ještě před zahájením výroby formy. Ušetříte tím čas i náklady za pozdější úpravy formy. Pro snazší a rychlejší odstranění problémů se vyplatí použít systém *VARIMOS*, který v krátkém čase doporučí vhodné úpravy konstrukce dílu nebo formy a také nastavení technologických parametrů s ohledem na požadovanou kvalitu dílu a dodržení všemi definovaných technologických a ekonomických požadavků. Systém *VARIMOS* rychle nalezne technologické okno, ve kterém je možné vyrábět díl v požadované kvalitě. Dále ukáže citlivost technologických a konstrukčních parametrů a vnějších vlivů na kvalitu dílu a pomůže zajistit robustnost a stabilitu výrobního procesu.

Teplotní management formy je možné ověřit také pomocí CFD simulací, konkrétně pomocí *Altair HyperWorks CFD* se solverem *AcuSolve*, který umí, stejně jako *CADMOULD*, ukázat průběh a vliv ohřevu a chlazení formy na kvalitu dílu.

Po odstranění všech problémů v konstrukci dílu nebo formy, zajistění správného plnění dílu a požadované kvality dílu je možné přistoupit k výrobě vstřikovací formy. Přesnost vyrobené formy a tvarové dutiny můžeme zkontrolovat pomocí optického měření a vyhodnotiti měřicím softwarem od společnosti VG Studio MAX. S jeho pomocí a při využití CT technologie můžeme prověřit i vnitřní strukturu jednotlivých částí formy a provedení vtokového a temperačního systému.

Pokud chceme vyrábět malé množství dílů, není třeba vyrábět plnohodnotnou „železnou“ formu, ale je možné využít některou z rapid prototyping technologií (3D tisk dílů, technologii vakuového lití, ...) nebo 3D tisk tvarových vložek. Plastových i kovových. Pro simulaci aditivního výrobního procesu je vhodný *Altair Inspire Print3D*.



5. VZORKOVÁNÍ

Vzorkování slouží k ověření funkce formy, nalezení vhodných technologických parametrů a zajištění výroby dílu v požadované kvalitě. Pro zkrácení doby vzorkování a snížení nákladů na vzorkování je možné využít doporučených technologických parametrů získaných ze simulací nebo optimalizací. Některé vstřikolisy jsou propojeny se simulačním softwarem *CADMOLUD* a optimalizačním systémem *VARIMOS* a proto je možné získaná doporučená nastavení přenést do těchto vstřikolisů automaticky a zajistit jejich využití pro první zkoušení formy.

Jestliže chceme mít náklady a časovou náročnost vzorkování zcela pod kontrolou, využijeme možnosti a funkcí systému *VARIMOS Real*. Díky němu získáme přesný plán vzorkování a také jasně daný čas obsazenosti lisu.

Kvalita vyrobených dílů se dnes nejčastěji kontroluje pomocí optického měření. V případě dílu se zálistky se využívá CT technologie. Pro vyhodnocení změřené vnější i vnitřní kvality vyrobených dílů můžeme použít měřící software *VG STUDIO MAX*.

6. SÉRIOVÁ KONTROLA

Po schválení kvality vyrobených vzorků lze přejít k sériové výrobě. Její podoba a náročnost se odvíjí od požadavků na kontrolu kvality vyrobených dílů. Lze provádět nahodilou kontrolu kvality vyráběného dílu, ale je také možné mít 100% kontrolu nad sériovou výrobou. S využitím informací ze vstřikolisu a případně senzorů teploty a tlaku umístěných ve formě, je možné dosáhnout řízené a 100 % kontrolované sériové výroby. Po zapojení automatické kontroly kvality dílu je možná i 100% kontrola všech vyrobených dílů a jejich okamžité třídění podle výsledků změřené kvality. Vše záleží na požadavcích zákazníka a vybavení lisovny.



ZÁVĚR

Simulační a optimalizační technologie dnes významně přispívají ke zvyšování kvality plastových dílů při udržení nákladů v potřebných mezích. Daří se vylepšovat funkční a výkonové parametry výrobků, zajistit vyšší kvalitu dílu již při prvním vzorkování i minimalizovat počet korekčních smyček. Celkově pomohou zkrátit a zlevnit jak vývoj, tak výrobu, čímž si zajišťují návratnost investovaného úsilí a prostředků.

Na první pohled je celý proces návrhu a přípravy výroby komplikovaný, ale při využití vhodných a vzájemně provázaných nástrojů lze tento proces snadněji nasadit do praxe.

Správně nastavený a využívaný digitalizovaný proces vývoje a výroby plastového dílu je uživatelsky přívětivější a usnadňuje práci. Činnosti, které se během vývoje a výroby plastového běžně provádějí několikrát, jsou při využití digitalizovaného procesu prováděny pouze jednou. Z praxe víme, že doba vývoje dílu se mírně prodlouží. Také náklady na vývojovou fázi mírně vzrostou. Ale značně se sníží počet korekčních smyček vyrobené formy. Tím se celková doba vývoje a výroby plastového dílu významně zkrátí a také odpadnou vysoké náklady na korekční smyčky, resp. úpravy, formy. Pokud k této přínosům a výhodám přidáme ještě i dosaženou kratší dobu výrobního cyklu, nižší spotřebu materiálu a energie, tak celkové náklady na vývoj a výrobu plastového dílu budou výrazně nižší a produkt se na trh dostane mnohem dříve.

Protože zmiňovaní výrobci simulačních technologií Altair Engineering Inc. a Simcon Kunststofftechnische Software GmbH spolupracují v rámci tzv. *Altair Partner Alliance*, lze zmíněná řešení pořídit pod jednou licencí bez navýšení nákladů.

POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



TENSILE TEST OF COMPOSITES USING DIGITAL IMAGE CORRELATION

TAHOVÁ ZKOUŠKA KOMPOZITŮ S VYUŽITÍM DIGITÁLNÍ KORELACE OBRAZU

LUKÁŠ GREGOR¹

This paper deals with the measurement of mechanical properties of carbon fibre reinforced polymer samples using tensile testing and digital image correlation. This combination allows the determination of the mechanical properties and observation of the sample failure mechanism.

Tato práce se zabývá měřením mechanických vlastností uhlíkových kompozitů s využitím tahových zkoušek a digitální korelace obrazu. Tato kombinace umožňuje určení základních materiálových vlastností a sledování mechanismů poruchy vzorku.

KEYWORDS

DIC, CFRP, tensile test, composite, carbon fibre

INTRODUCTION

Carbon fibre reinforced polymers (CFRP) are widely used in many structural applications because of their high specific strength and stiffness. For lightweight and efficient structures, stress-strain analyses using finite element methods are often used, but there is a high risk of inaccuracy in simulations compared to reality. It is caused by high sensitivity to manufacturing technology, correct direction of fibres when they are laid, and incorrect mechanical properties of the input material model. This paper deals with the determination of mechanical characteristics using tensile testing and digital image correlation (DIC). This setup is used to measure the linear elastic constants of the T700G woven fabric and to evaluate the engineering constants of specified laminates. These data will be further used to improve and correct the material model of T700G lamina and finite element analysis (FEM). DIC is used as a convenient method for measuring surface strains and is an appropriate alternative to strain gauges as [1,2,3] confirms. Quasi-static tests are performed on universal testing machine (UTS) according to ASTM standards [4,5] with controlled head displacement. Since the strain rate has a minimal influence when the fibres dominate in the lamina, only one speed is used according to the standard [6].

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The effort to reduce potential measurement discrepancies leads to a focus on these areas of interest. Control of the entire production process, in particular: proper production technology, correct fibre orientation, prevention of sample damage during machining, bonding of glass fibre tabs to the clamping area, and accurate sample orientation during clamping.

SPECIMEN PREPARATION

Five sets of CFRP specimens were prepared. The materials used for the samples were carbon fibre weave (240 g/m^2 and 630 g/m^2) and biaxial tape ($+45^\circ / -45^\circ$, 300 g/m^2), both with a 60% volume of Toray T700G fibre and an epoxy resin system ER 38 by MEEM. The fibre and resin properties are in the Tab. 1. Samples fabrication was carried out using the manual lay-up technique and autoclave processing at 120°C and 6 bars. Specimens after autoclave processing are shown in Fig. 1. The geometry and other requirements are defined by the ASTM standard for the tensile test of polymer composites [4] and the samples were machined to the desired geometry with a diamond rotary blade. Glass fibre tabs were bonded to the ends of the samples to provide support and reduce the risk of sample damage by grip and therefore crack in an unacceptable area. The tabs can be seen in Fig. 5b). The overview and layup are in the Tab. 2.

Tab. 1 Selected mechanical properties of Toray 700G fibre and epoxy system ER 38

Mechanical properties of Toray fibre T700G		
Tensile strength	4900	MPa
Tensile modulus	240	GPa
Strain at failure	2.0	%
Mechanical properties of epoxy resin system ER 38		
Flexural strength	4900	MPa
flexural modulus	240	GPa

Tab. 2 Produced specimens

Specimen series	Areal density [g/m ²]	Layup	Specimen thickness [mm]
CF_600	630	0/90	1,3
CF1_200	245	(0/45)s	1,0
CF2_200	245	(0/45/0/45)s	2,0
SW_0	T - 630, Bi - 300	0(T)/(0/45/0/45/0)(Bi)/0(T)	3,1
SW_45	T - 630, Bi - 300	45(T)/(45/0/45/0/45)(Bi)/45(T)	3,1

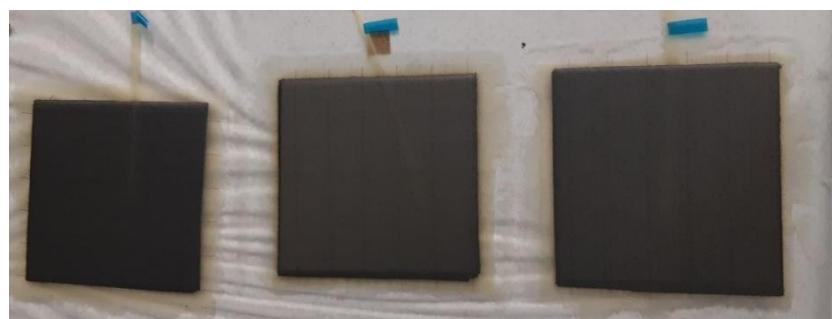


Fig. 1 Specimens before machining

Before testing, the samples were covered with a stochastic pattern. This was achieved by painting a white background and spraying black dots. Three commonly used paint bases were tested for pattern application. The suitability of these bases is in Tab. 3. The tests showed that visually there were no adhesion problems on the cleaned and degreased surface. However, in the tensile test, the hairspray (Fig. 2) proved unsuitable as its deformation was not telling and it fell off during the test. Acrylic-based (Fig. 3) or water-based (Fig. 4) paint performed well in the tests. The water-based paint performed best because it was the easiest to apply.

Tab. 3 Stochastic pattern basis

Stochastic pattern basis	result
hairspray (Fig.)	poor
acrylic paint (Fig.)	good
water-based paint (Fig.)	great



Fig. 2 Hairspray based pattern failure

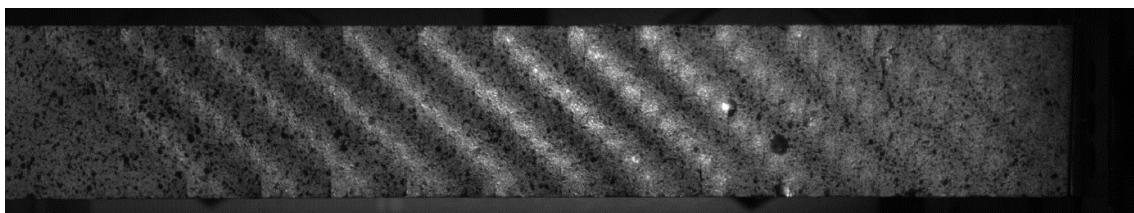


Fig. 3 Acrylic – based paint pattern

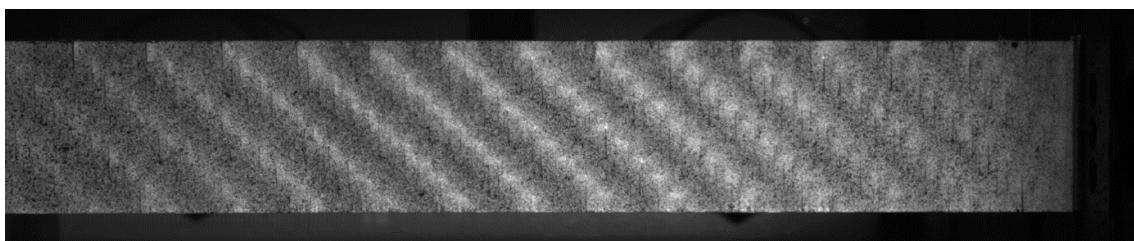


Fig. 4 Water – based paint pattern

DATA ACQUISITION AND EVALUATION

The tensile tests were carried out on Zwick-Roell Z100 UTS with TestXpert II software. An additional GOM Aramis optical system was connected and synchronised with the UTS. The strains were evaluated from the recorded deformation of the samples using DIC. As mentioned, this system is an alternative to conventional foil strain gauges with adequate accuracy [1,2,3]. It is fast and reliable solution allowing measuring in multiple axes simultaneously. The measurement setup is shown in Fig. 5a). Due to explosive rupture (Fig. 5b)) of some samples it was necessary to partially cover the UTS with a transparent plastic film, which does not affect the optical measurement.

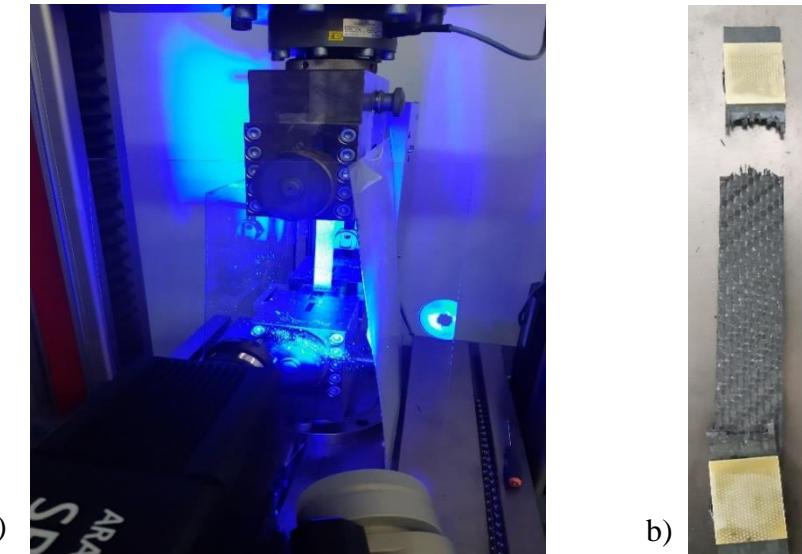


Fig. 5 a) Tensile testing setup with DIC camera b) Broken sample

RESULTS

The graph of the tensile test is in Fig. 6. The results derived from the Aramis software are in Tab. 4. Sample CF_600 damage development and technical strain increase up to failure is possible to see in Fig. 7. In the nonlinear region of the test it is clearly visible how the fibres slip and then appear LGM failure according to ASTM D3039 [4].

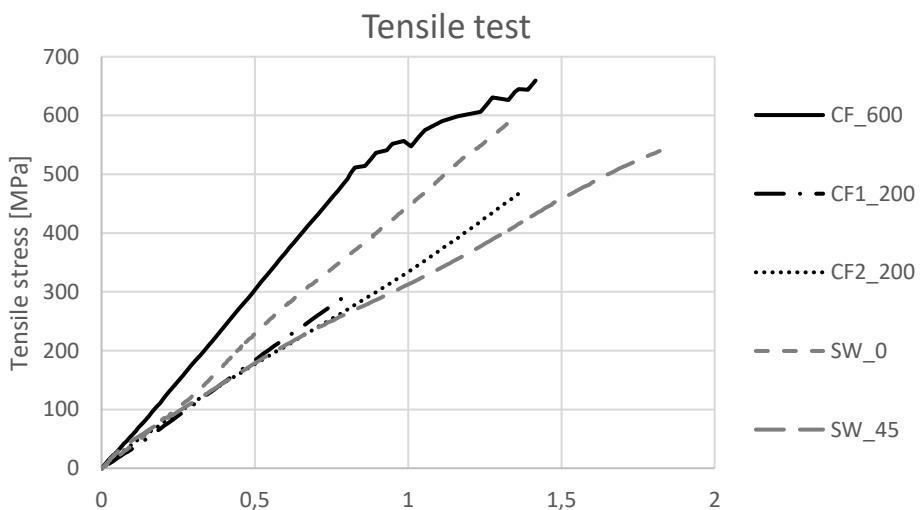


Fig. 6 Tensile test result

Tab. 4 Summary of measured properties

Specimen series	Areal density [g/m ²]	Layup	thickness [mm]	Tensile modulus [GPa]	Stress at break [MPa]	Strain at break [%]
CF_600	630	0/90	1,3	63,0	650	1,41
CF1_200	245	(0/45)s	1,0	37,7	285	0,84
CF2_200	245	(0/45/0/45)s	2,0	35,6	485	1,37
SW_0	T - 630, Bi - 300	0(T)/(0/45/0/45/0)(Bi)/0(T)	3,1	44,4	615	1,35
SW_45	T - 630, Bi - 300	45(T)/(45/0/45/0/45)(Bi)/45(T)	3,1	33,1	540	1,85

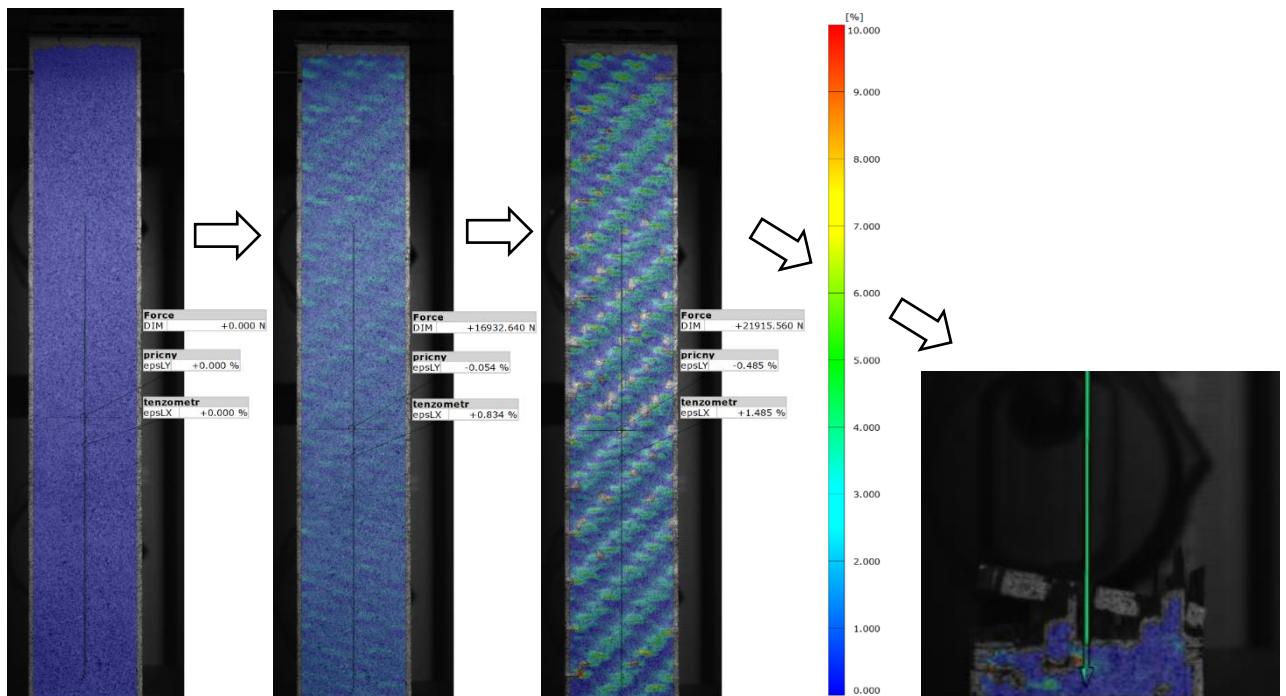


Fig. 7 Visualisation of technical strain ϵ in axial direction during damage development, CF_600 sample

The linear elastic data obtained from the measurement are specifically for the CF_600 lamina: Young's modulus in tension $E_L = E_T = 63$ GPa, ultimate tensile strength $\sigma_L = \sigma_T = 650$ MPa and poison ratio $v_{LT} = 0,064$. For a precise material model it is necessary also to obtain shear data - shear modulus G_{LT} and shear strength τ_{LT} . It can be achieved with the same method when the specimen for this lamina is angled at 45°.

CONCLUSION

The selected mechanical properties of woven fabric (T700 / ER 38) have been measured and the linear elastic properties of the samples were evaluated under tension until break. The DIC appears as an efficient tool for strain measurement, offering the possibility to evaluate data retrospectively in comparison with conventional extensometers. The data gained and future tasks are follows:

- Specific data gained for 0/90, $E_L = E_T = 63$ GPa, $\sigma_L = \sigma_T = 650$ MPa, $v_{LT} = 0,064$
- Engineering constants gained for selected laminate layups CF1, CF2, SW0, SW45
- Shear data G_{LT} , τ_{LT} , have to be measured to complete material model

Further work should focus on measuring the shear properties of woven fabric (T700/ER 38) and the complete elastic constants of biax fabric (T700/ER 38). The data and data obtained in this paper can then be used to validate the material model.

ACKNOWLEDGMENT

This research work was supported by the Ministry of Industry and Trade, Program OPPIK – Knowledge Transfer Partnership, with the grant " Design and strength analysis of a carbon frame for an electric motorcycle ", CZ.01.1.02/0.0/0.0/20_324/0023596.

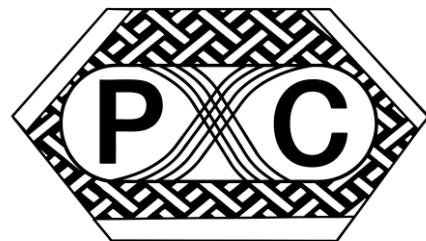
LITERATURE

1. Munoz, V., M. Perrin, M.L. Pastor, H. Welemane, A. Cantarel et al.. Determination of the elastic properties in CFRP composites: comparison of different approaches based on tensile tests and ultrasonic characterization. Advances in Aircraft and spacecraft science, 2014 vol. 2, pp. 249-260.
2. Tekieli, M., S. De Santis, G. De Felice, A. Kwiecien, F. Roscini. Application of Digital Image Correlation to composite reinforcements testing. Composites structures, 2017, pp. 670-688, ISSN 0263-8223
3. Shadmehri, F., S. Hoa, Digital Image Correlation applications in composite automated manufacturing, inspection, testing. Applied sciences, 2019 vol. 9, pp. 2719.
4. ASTM D3039/3039M-00. Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials. West Conshohocken: ASTM International, 2017, 13 p.
5. ASTM E 132 – 97. Standard Test Method for Poisson's Ratio at Room Temperature. West Conshohocken: ASTM International, 2017, 3 p.
6. Hou, J.P., C. Ruiz. Measurement of the properties of woven CFRP T300/914 at different strain rates. Composites science and technology [online]. Oxford: Elsevier, 2000, ISSN 0266-3538.

POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



OPTIMAL DESIGN AND TESTING OF A THERMOPLASTIC PRESSURIZED PASSENGER DOOR MANUFACTURED USING THERMOFORMING

OPTIMALIZACE NÁVRHU A ZKOUŠKY PŘETLAKOVANÝCH DVERÍ LETOUNU Z VYZTUŽENÉHO TERMOPLASTU

JOSEF KŘENA¹, ROMAN RŮŽEK², RADEK DOUBRAVA²

The present paper documents and discusses research work associated with a newly designed passenger door structure demonstrator. The composite structure was manufactured from carbon-fiber-reinforced thermoplastic. A composite frame with a variable cross-section was designed, optimized, and fabricated using thermoforming technology. FEM was applied for design and testing and special SW was used for process simulation. The demonstrator was put to the static and fatigue test.

KEYWORDS

Composites; thermoplastics; door; damage tolerance; static; fatigue; non-destructive testing; numerical analysis

1. INTRODUCTION

At present, aeronautical industry composite structures are still primarily thermosets; nevertheless, the share of high-performance thermoplastic composites (TPCs) is continuously increasing despite their high purchase prices [1]. They provide good material properties, such as fracture and impact resistance [2], formability, weldability, self-healing possibilities, and recyclability [3]. The main benefits of TPCs can be seen in their potential for repeated heating and molding, without negative influence on mechanical and physical properties. Traditionally, composite fibers within a layer have the same orientation, leading to constant stiffness properties. Recently, due to the development of advanced manufacturing technologies, such as automated laying processes (fiber placement, fiber patch placement, tow shearing), the fiber orientation of a layer can be continuously varied, together with varying stiffness properties. Another procedure is to align fibers in the direction of the principal stress with the aim of

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reducing stress concentrations and weight [4]. In [5], a three-step approach for variable-stiffness laminate design was applied to shear panels. The first step is to find the optimal stiffness distribution in terms of the lamination parameters [6]; the second step is to find the optimal manufacturable fiber angle distribution; and the third step is to retrieve the fiber paths [7]. All above mentioned methods have relatively significant disadvantages in terms of time and a costly manufacturing process. Additionally, utilization of these methods is limited by the requirements to exclude the fiber/tow shift and gaps or overlaps that are present during manufacturing. Thermoplastics, together with thermoforming, create possibilities to manufacture flat plates with predefined fiber/fabric layer orientations, and/or tailored blanks to achieve structural parts with varying stiffnesses and mechanical properties [8,9]. The thermoforming process enables the manufacture of a structural part with varying cross sections in a relatively very short amount of time. Thus, the thermoforming process appears to be a very promising manufacturing technology for TPCs. An additional significant impact of thermoplastic material exploitation is out-of-autoclave manufacturing process development (which can potentially save up to 75% on energy consumption compared with the autoclave process) as well as weight and waste reductions [10]. All these attributes lead to a significant reduction in eco-impact throughout the life cycle of a structure. These are the main reasons why this technology and type of material were used for the pressurised passenger door that was designed, optimized, manufactured, and verified in this work. Moreover, the above-discussed advanced technology is in accordance with composite roadmap developments within the Latécoère Innovation department. In compliance with airworthiness requirements, primary structures manufactured from composite materials must be designed and operated in compliance with the damage tolerance (DT) philosophy [11,12,13,14]. This approach allows, in certain cases, structure operations with an allowable size of flaw (damage). The experimental certification procedure of a structure, according to DT philosophy, requires conducting several relatively independent phases: mechanical loading (both static and fatigue), environmental loading, non-destructive inspection, simulation of impact damage, and residual static tests. Environmental effects could be included as a so-called knock-down factor, which includes not only the influence of humidity on structure properties, but also structure performance at different altitudes [15]. The present paper documents and discusses relevant procedures associated with a pressurised passenger door demonstrator, designed and manufactured from carbon-fiber-reinforced composite with thermoplastic resin. The work consisted of several phases: technology development, process optimization, numerical model development and verification, test campaign definition, loading system design and manufacturing, impact damage tests, mechanical loading, non-destructive inspection, deformation and strain measurements, and residual static strength evaluation. A new numerically verified manufacturing technology was developed where a composite frame with variable cross section was fabricated using thermoforming technology. The design was supported by an experimental campaign with the aim of verifying mechanical properties and comparing them to numerical simulations. The verification methodology was designed based on the damage tolerance philosophy; this means that structural properties were proofed under both static and fatigue loading conditions with the presence of impact damage. Main load is given by the inner overpressure in the fuselage which is about 60 kPa. Numerical simulations of finite element (FE) models were used to determine the load of the real structure, to optimize the design of critical parts, and to define the representative load distribution into the newly designed door panel. A linear analysis with linear contact between each part of the structure or the load system was used for numerical simulations. The Tsai–Wu and maximal stress criteria were used for damage analysis of the composite parts. Topological optimization was used for the design of metal hinges from the point of view of weight reduction. A FE model of the real test assembly

was also used for post-test analyses and results comparisons. The simplification of overpressure simulation for experimental verification of the door structure was designed and applied.

2. MATERIALS

The panel consisted of the skin, three Omega beams, two Z frames, and six stop fittings (locks) assembly. Individual parts of the structure were joined using bolts. The Z-frame and Omega profiles were fabricated using a hot forming method. All composite parts were fabricated of carbon material utilizing a polyphenylenesulfide (PPS) thermoplastic resin system which was supplied by Toray and specified as TC1100. The melting temperature of PPS is 280°C and that of Tg is 90°C. The reinforcement fabric was 5HS 3K T300 with an area weight of 285 g/m² and the weight rate of matrix was 43%. Basic properties are included in Tab. 1.

Tab. 1 Properties of one layer of the carbon fabric/PPS matrix material

Tensile strength	Tensile modulus	Compression strength	Compression modulus	Shear strength	Shear modulus
MPa	GPa	MPa	GPa	MPa	GPa
670	58	510	53	90	4,1

The Omega profiles had 10 plies of C fabric with the layup $[(0,90)/(-45)]_2/(0,90)]_s$. Z-frames had a layup of $[(0, 90)/(-45)]_3/(0/90)]_s$ with a thin glass layer on the surface. The skin was cut using water jet technology from a flat plate with a layup of $[(0,90)/(-45)]_5/(0,90)$. An overview of the used thermoplastic material is summarized in Tab. 2.

Tab. 2 Overview of composite materials used for door panel manufacturing.

Structure Element	Material	Resin System	Nominal thickness (mm)	Number of layers	Layup
Omega profile	TC1100	PPS	3,1	10	$[(0,90)/(\pm45)]_2/(0,90)]_s$
Z-profile	TC1100	PPS	4,34	14	$[(0,90)/(\pm45)]_3/(0/90)]_s$
Skin	TC1100	PPS	3,41	11	$[(0,90)/(\pm45)]_5/(0,90)$

Stop fittings were milled from titanium alloy (Ti-6Al-4V) annealed plate material (minimum ultimate strength of 900 MPa, yield strength of 830 MPa). Individual parts of the door panel were joined using bolts. The Omega profiles with the stop fittings were joined using NAS6604D16 steel bolts, the Omega profiles and Z-profiles were joined using ABS0114-4 titanium bolts, and the skin, Omega profiles, and Z-profiles were joined using EN6114V3-5 titanium bolts. The overall geometry of the door panel was: length (L) = 1250mm, width (W) = 950 mm and height (H) = 114 mm. The panel was manufactured as shown in Fig. 1.

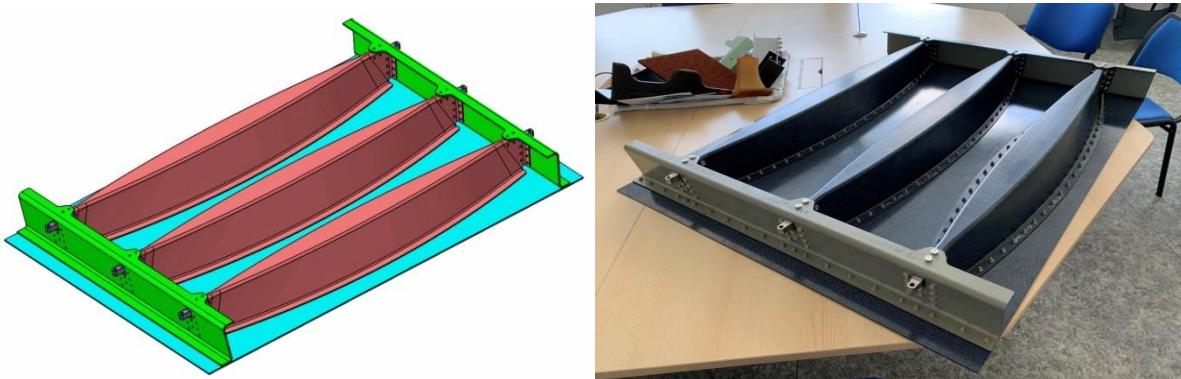


Fig. 1 Door panel with Omega beams and Z frame

3. STRUCTURE DESIGN AND MANUFACTURING

All elements of the structure were manufactured by the thermoforming process. The raw material is consolidated plate of required layup. The blank which was cut out from the plate is preheated in the oven and shaped in the suitable tool subsequently. The scheme of the process is displayed in Fig. 2.

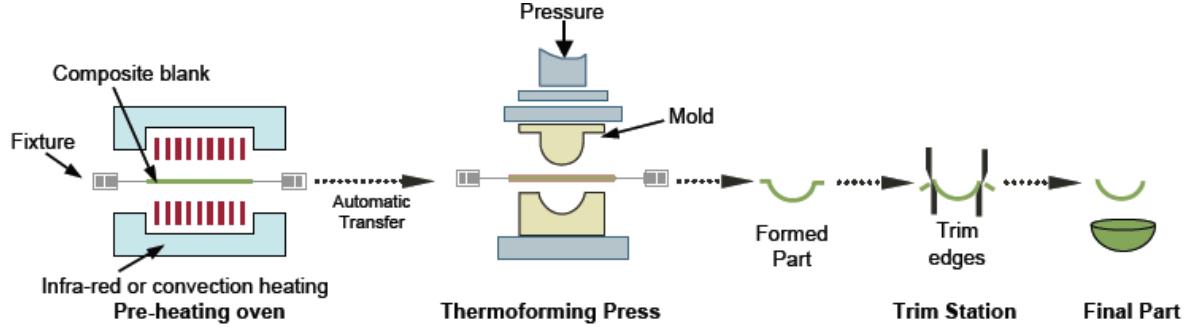


Fig. 2 Scheme of the thermoforming

The assembly consisted of three types of parts, where the Omega profile had the most complex shape of them. That shape was chosen due to the loading along that profile. There was maximal bending moment in the center of the profile, so that the maximal inertia of the cross section was located there, and the profile was wide. The opposite situation occurred on the edges of profile where the maximal shear force was located at the metal stop fitting. Here, we needed to distribute the large local force into the surrounding structure. This was why the Omega profile was designed with such a complex shape (Fig. 3). edges of profile where the maximal shear force was located at the metal stop fitting. Here, we needed to distribute the large local force into the surrounding structure. This was why the Omega profile was designed with such a complex shape (Fig. 3).

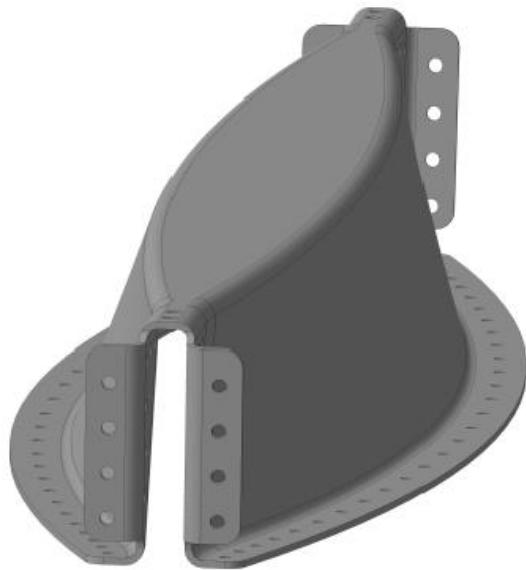


Fig. 3 Optimized Omega profile

The problem of non-developable shape thermoforming is a risk to the folded layers or the wrinkles created during forming. Two basic mechanisms can help to successfully form the complex shape of a laminate. The first is sliding between layers and the second is shear

deformation of the layer. Interlaminar sliding can be caused by folding of the laminate into the final shape of the part (Fig. 4).



Fig. 4 Interlaminar sliding caused by laminate folding

Planar shear deformation also enables fitting of the laminate to a final shape; however, shear stiffness strongly depends on the orientation of fibers relative to the load direction. It also induces sliding between layers with different fiber orientations (Fig. 5). This second mechanism is the key for forming non-developable parts.

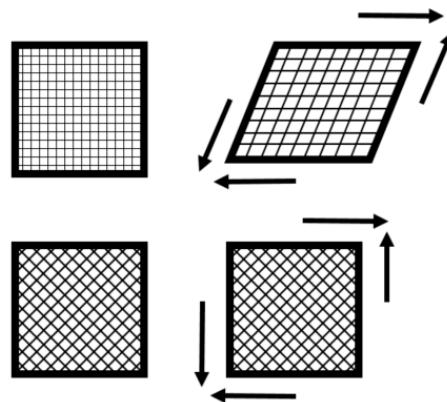


Fig. 5 Shear deformation of the layers with different fiber orientations

All described and more mechanisms are included in the special SW AniForm [16], which was used for simulations of the thermoforming process. It enables prediction, not only of wrinkles, but also reorientation of fibers, stress in layers, etc. The complex shape of the Omega beam was created on the basis of loading, as described above. However, slight modification using simulations was needed for it to be feasible to produce the beams without wrinkles and folding due to the non-developable shape. Two extracted steps in the forming simulation are displayed in Fig. 6. The first step (a) shows us the shape of the blank at the beginning of forming, and the second step (b) is a fully shaped part with a spectrum displaying shear deformations.

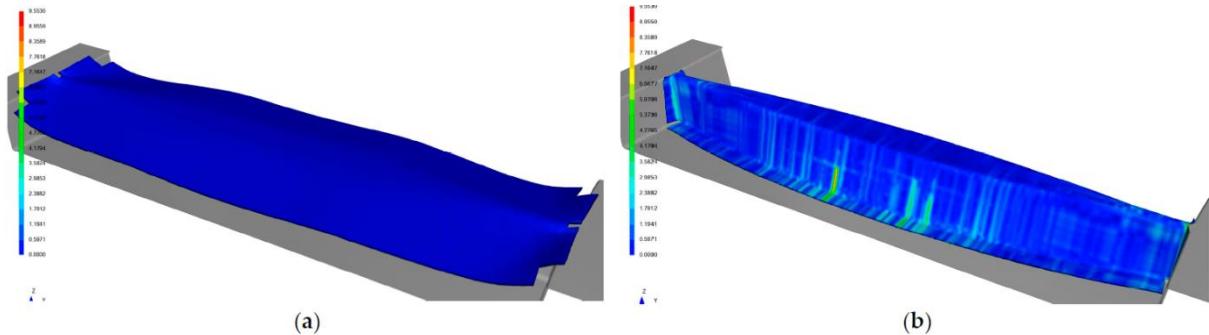


Fig. 6 Contour maps of shear deformation at the beginning of forming (a) and in the final shape of stamping (b)

The verified shape of the part was the baseline for designing the stamping tool, which consisted of male and female metal parts is shown in Fig. 7.



Fig. 7 Stamping tool for omega beam

Stamping was performed after preheating of the blank up to a process temperature of about 330 °C. The manufacture of the part confirmed the correct prediction as a good quality of profiles was achieved. Trimming of the contour was performed using a NC machine and assembly was carried out with the help of titanium mechanical fasteners (Hi-Lock), excluding the joining of titanium stop fittings where titanium screws were used.

4. TESTING OF THE PANEL

The main load acting on the door structure was overpressure. For simulations of real pressure loads on the physical test, several variants, as rubber bags, foam boards, special inserts, and using existing fasteners points, were considered. Finally, the continuous pressure load was transformed into the resultant force and the continuous load was introduced into the door structure using a special whiffle tree loading system. The overpressure load was distributed (pressure simulation) into the door panel through 24 individual points. A scheme of the whiffle tree system is shown in Fig. 8 panel.

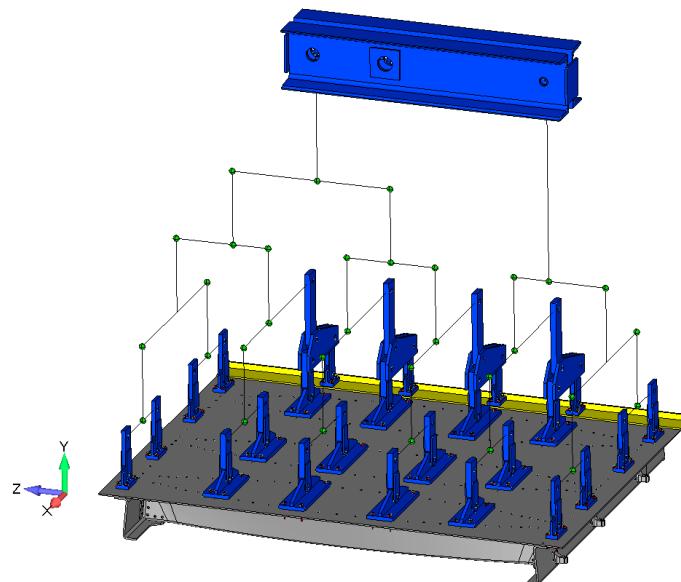


Fig. 7 CAD model of the whiffle tree system for load distribution into the door panel

The experimental procedures complied with airworthiness regulations included the following separate procedures:

- Barely visible impact damage (BVID) creation by energy up to 45 J;
- Static loading up to LL;
- Fatigue loading;
- Static loading up to LL;
- Static loading up to UL followed by continuous loading to failure;
- Static loading

During the experimental work, the following systems were used for data acquisition and measurements:

- Deformation optical measurement system;
- Strain gage measurement system;
- LVDT measurement system (displacement of given point).

5. RESULTS

Load vs. displacement dependence up to door panel failure was measured using LVDT sensors placed in the middle of the panel span (Fig. 8) and strain measurements in the highly loaded regions of the panel (surrounding of fittings) up to failure using resistance strain gauges. Panel failure occurred at 214.01 kN in an area of the stop fitting connection with the Omega and Z-profiles.

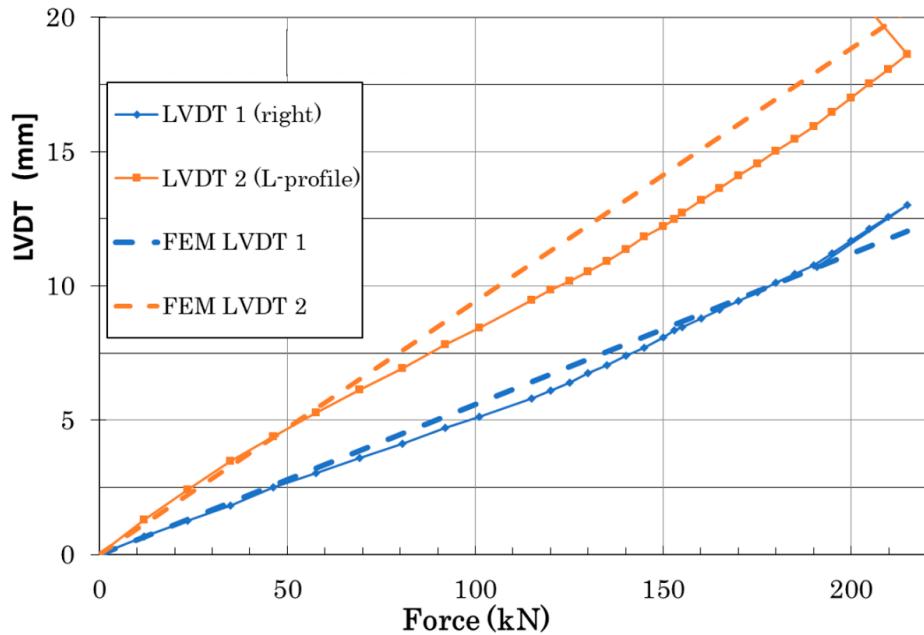


Fig. 8 Force vs. LVDT displacement comparison of experimental data and numerical prediction

The door panel sustained all required procedures and loading without structural failure. No damage propagation was observed. Real structural strength was about 25% higher compared with the ultimate (UL) structure strength.

6. CONCLUSIONS

A new thermoplastic carbon composite panel door design, its manufacture, numerical analyses, and experimental verification according to the damage tolerance philosophy is presented. Thermoforming technology was used for the composite beam with variable cross section manufacturing. The variable shape of the Omega profile was optimized and manufactured without any imperfections (typically the occurrence of wrinkles in the thermoforming process is very dangerous). No impact damage propagation was observed in the composite parts during mechanical tests (180,000 fatigue cycles under a load level close to the limit load) despite visible impact damage being introduced into the structure. No significant difference between numerical simulations and the experimentally measured total deformation was observed. Repeated deformation measurements over a time interval during fatigue showed changes and a nonlinear structure behavior. This can be attributed to the relaxation of the thermoplastics or by the behavior of mechanical fasteners. The stiffness degradation of the thermoplastic composites needs future additional detailed analysis. As there was an expectation that no growth of impact damage would be observed, this leads to the conclusion that the composite structure was overdesigned. Accordingly, in general, future work should be focused on improving the design philosophy to achieve weight and costs saving. Two ways to achieve this seem to be suitable: higher utilization of material mechanical properties (decrease of knock-down factors) and improving damage tolerance design (change the non-growth design criterion using the slow growth criterion, for example).

ACKNOWLEDGMENT

This research was funded by the Ministry of Industry and Trade of the Czech Republic (MPO), program TRIO, grant number FV30033 “Structural and technological development of primary aircraft parts made by composite with thermoplastic matrix” and with the help of institutional funding of the MPO in the DKRV01 program dedicated to the development of research organizations.

LITERATURE

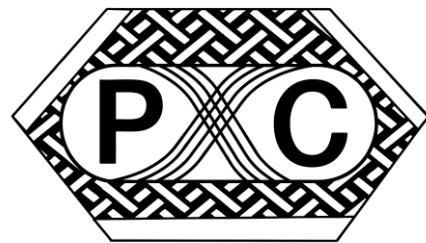
1. Dale, B. Thermosets vs. Thermoplastics: Is the Battle Over? 2015. Available online: www.compositesword.com (accessed on 10 February 2020).
2. Hron, R.; Kadlec, M.; R° užek, R. Effect of the Test Procedure and Thermoplastic Composite Resin Type on the Curved Beam Strength. *Materials* 2021, 14, 352.
3. Schinner, G.; Brandt, J.; Richter, H. Recycling Carbon-fiber-reinforced Thermoplastic Composites. *J. Thermoplast. Compos. Mater.* 1996, 9, 239–245.
4. Crothers, P.J.; Drechsler, K.; Feltin, D.; Herszberg, I.; Kruckenberg, T. Tailored fiber placement to minimise stress concentrations. *Compos. Part A Appl. Sci. Manuf.* 1997, 28, 619–625.
5. Peeters, D.M.J.; Irisarri, F.X.; Groenendijk, C.; Růžek, R. Optimal design, manufacturing and testing of non-conventional laminates. *Compos. Struct.* 2019, 210, 29–40.
6. IJsselmuiden, S.T. Optimal Design of Variable Stiffness Composite Structures using Lamination Parameters. Ph.D. Thesis, Delft University of Technology, Delft, The Netherlands, 2011. Available online: <https://agris.fao.org/agris-search/search.do?recordID=AV2012066516> (accessed on 26 September 2021).
7. Blom, A.W.; Abdalla, M.M.; Gürdal, Z. Optimization of course locations in fiber-placed panels for general fiber angle distributions. *Compos. Sci. Technol.* 2010, 70, 564–570.
8. Růžek, R.; Šedek, J.; Kadlec, M.; Kucharský, P. Mechanical behavior of thermoplastic rib under loading representing real structure conditions. In Proceedings of the EAN

- 2016—54th International Conference on Experimental Stress Analysis, Srní, Czech Republic, 30 May–2 June 2016. EID: 2-s2.0-84986296610.
- 9. Růžek, R.; Kadlec, M.; Petrusová, L. Effect of fatigue loading rate on lifespan and temperature of tailored blank C/PPS thermoplastic composite. *Int. J. Fatigue* 2018, 113, 253–263.
 - 10. Miraglia, A.; Felline, F.; Rocco, C.; Ceresa, A.; Raganato, U.; Zavala-Arredondo, M.; Flipo, B.; Liaptsis, G.; Riise, J.; Cui, X.; et al. TOD—Thermoplastics on Doors: Development of full scale innovative composites doors, surrounds and sub-structure for Regional Aircraft Fuselage barrel on-ground demonstrators. Innovative overall manufacturing and assembly approach and preliminary results obtained at the early stage of the project. *IOP Conf. Ser. Mater. Sci. Eng.* 2021, 1024, 012023.
 - 11. CMH 17—Composite Material Handbook; SAE International: Amsterdam, The Netherlands, 2002; Volume T6.
 - 12. FAA Code of Federal Regulations (CFR): Aeronautics and Space. 2007. Available online: <http://www.access.gpo.gov/cgi-bin/cfrassemble.cgi?title=200714> (accessed on 26 September 2021).
 - 13. FAA Advisory Circular 20-107A: Composite Aircraft Structures; Federal Aviation Administration, US Department of Transportation: Washington, DC, USA, 1984.
 - 14. FAA Advisory Circular 21-26: Quality Control for the Manufacture of Composite Materials; Federal Aviation Administration, US Department of Transportation: Washington, DC, USA, 1989.
 - 15. Certification Specifications for Large Aeroplanes CS-25 Amendment 2; EASA: Cologne, Germany, 2006.
 - 16. SW Aniform Manual. Available online: www.aniform.com (accessed on 26 September 2021).

POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



PRECISION AND DIMENSIONAL STABILITY OF BONDED JOINTS OF CFRP PARTS

PŘESNOST A ROZMĚROVÁ STABILITA LEPENÝCH SPOJŮ KOMPOZITNÍCH SOUČÁSTÍ

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In this paper, a precision bonding technology that could enable the use of CFRP in optical applications is presented. 15 technological samples were tested under mechanical and climatic conditions based on ISO 9022-1. The total runout of the key dimensions was evaluated after production and after passing the tests. The results suggest that for accurate bonding, the stresses introduced during adhesive curing must be eliminated by mechanical or thermal treatment.

KEYWORDS

Carbon fiber, composite materials, sports optics, precision bonding

INTRODUCTION

The popularity and use of fibre reinforced composite materials have been growing year by year. The demand for composite materials tripled between 2010 and 2020 [1]. Despite that the application of CFRPs in the production of sports optics or other optical instruments is still very rare. Among the obstacles that hinder the adoption of composite materials in optical devices, the limited manufacturing precision of composite parts is the major one [2]. In some cases, limited impact resistance and the absence of plastic deformation are the main limitations [3]. In other cases, it may be necessary to consider outgassing parameters, which for composites is currently a complex topic with a limited state of knowledge [4]. If these problems can be eliminated, composite materials could bring several benefits to optical devices. This paper builds on previous research on this topic [2] [5]. The accuracy and thermal stability of tubular bonded joints of metal and CFRP adherents was studied. Only a few studies are focusing on the

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use of CFRP in industries similar to sport optics. The research goal is to demonstrate that the use of CFRP can be beneficial for optical devices as well and that all limitations can be eliminated by selecting an appropriate manufacturing method.

SUBJECT OF RESEARCH

The main goal of this study was to investigate the accuracy and thermal stability of tubular bonded joints of metal and CFRP adherents. For our research purposes, a sports optics device, primarily a binocular or spotting scope, was selected as a reference instrument. The testing conditions are established by ISO 14490-6:2014 and ISO 14490-7:2016 for testing of optical parameters and by ISO 9022-1:2016, ISO 9022-2:2015, ISO 9022-3:2015 and ISO 9022-8:2015 for testing of mechanical properties and climatic resistance. The choice of a particular reference device does not limit future adoption to this a relatively narrow set of devices. The insights gained are likely to overlap with other types of optical devices. Winding technology was chosen for the technological samples, mainly for the wide range of commercially wound cylindrical tubes available.

DESIGN OF TEST SAMPLES

The technological sample (Fig. 1) consists of a wound CFRP tube made of [90°/0°/±30°] layers with a length of 90 mm, an inner diameter of 50 mm, and a wall thickness of 1.5 mm. The CFRP tube is bonded to anodized EN_AW-6061-T651 aluminium alloy sleeves, which represent the interface for mounting optical assemblies, e.g. objective and eyepiece of the Keplerian telescope. The 1 mm wide gluing gap allows for compensation of manufacturing inaccuracies of the carbon components. The mutual relative position of the two sleeves according to the required tolerances is ensured by precise gluing or turning after bonding.

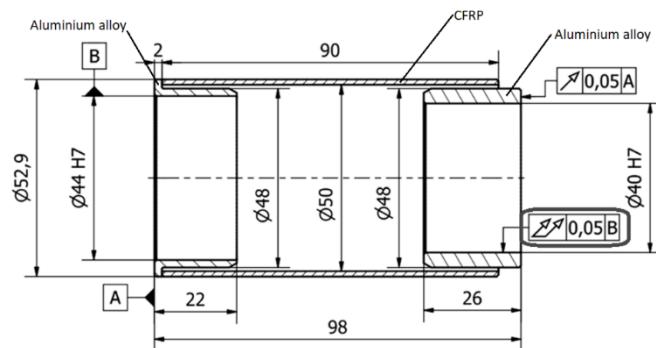


Fig. 1 Geometry of a technological sample

An assembly fixture was designed for bonding the technological sample with the required precision. (Fig. 2).

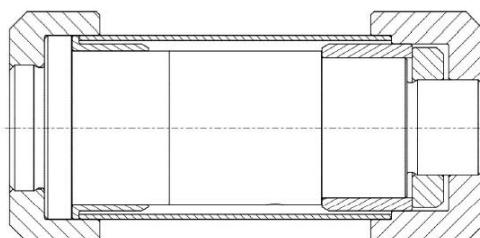


Fig. 2 An assembly fixture

Total runout of the inner diameters of both sleeves was chosen as a critical parameter with the maximal allowed value of 0.05 mm (Fig. 1). These requirements must be achieved after

production and must also be met after a set of mechanical and climatic tests, which are described in the next chapter.

DESIGN OF EXPERIMENT

To guarantee a logical structure of the tests/experiments, it was necessary to establish a methodology and schedule to be followed during the experiments. The processes were divided into three stages:

- **Stage 1 = Design and preparation of technological samples**

The first stage was focused on material selection and design of the sample and assembly fixture. After that, it was necessary to select the adhesives. Then, the samples were produced by turning metal parts, shortening carbon tubes and gluing the samples using the assembly fixture.

- **Stage 2 = Verification of dimensions and geometric tolerances**

The inspection of the pieces was carried out using CMM Zeiss Contura G2, Formline Roundscan 555, and a set of OK/NOK gauges.

- **Stage 3 = Verification of the stability of a bonded joint under operating conditions**

Samples with unsatisfactory tolerances were fixed by turning and tested again according to the methodology of the stage 2. After that, climatic resistance tests based on ISO 9022-2:2015 were realized using the climatic chamber Vötsch VT3 7030 S2. Firstly, the technological sample was placed into a climatic chamber tempered to $+70 \pm 2$ °C for 16 hours. Then, the same process was repeated under temperatures of -40 ± 3 °C. The last climatic resistance test was focused on thermal shock testing when the technological sample was exposed to thermal shocks in temperatures – 25 °C / +40 °C. After all climatic resistance tests, the dimensions and geometric tolerances of the samples were verified again using the same equipment as in the stage 2. The final set of mechanical resistance tests according to ISO 9022-3:2015 included a shock test, a bump test using TIRAshock 4110, and a sinusoidal vibration test using Derritron TW 6000. After all mechanical resistance tests, the dimensions and geometric tolerances of the samples had to be again verified according to the methodology of the stage 2.

SELECTION OF ADHESIVES

Epoxy adhesives are the most common adhesives for composite materials [3]. Therefore, individual types of epoxy adhesives (Tab. 1) were also selected for our experiments based on the following criteria:

- the selected adhesive is suitable for bonding metals and thermoplastics,
- it has high environmental resistance (-40 °C to +70 °C),
- and low shrinkage (however, most manufacturers do not specify this parameter).

Tab. 1 Selected adhesives and their properties [6] [7] [8] [9] [10]

Adhesive type	Working time [min]	Clamping time [min]	Cure time [days]	Viscosity at room temperature [Pa.s]	Density [g/cm ³]	Shear strength [MPa]	Volumetric shrinkage [%]
DP110	9-15	20	2	65,0	1,12	17	not specified
DP125	25	150	1	52,5	1,13	13,4	not specified
DP190	90	480	7	86,3	1,31	11,7	not specified
Spabond 340 LV	45	1060	28	264	1,12	29	1,91-1,94
Eprosin Flex	70	1440	7	15	1,55	15,9	not specified

RESULTS AND DISCUSSION

According to the prescribed methodology and schedule, 15 samples were produced. The required accuracy of 0.05 mm was achieved in 7 out of 15 (46.6 %) samples (Fig. 3).

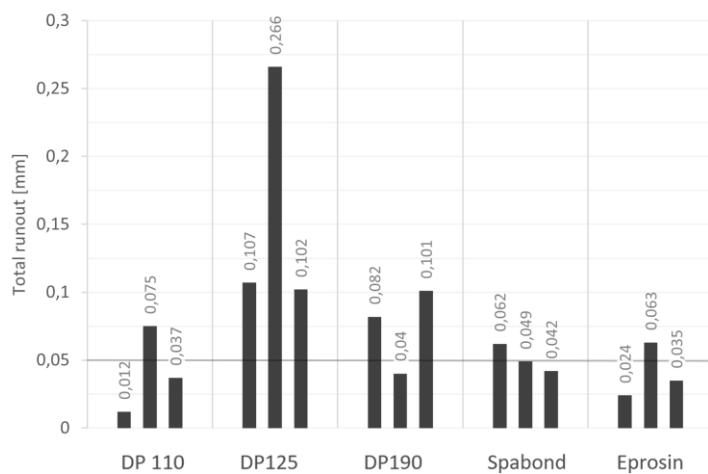


Fig. 3 Values of total runout

Based on the experiments and measurements performed, it can be concluded that:

- 46.6 % of the samples have the total runout deviation below the required value 0.05 mm
- The non-compliant samples were likely to deform due to stress introduced by shrinkage of the adhesive and/or uneven filling of the adhesive joint (presence of bubbles), to be verified by a CT scan.
- The most favorable results were obtained with DP 110, Eprosin, and Spabond and therefore, they appear to be suitable adhesives.
- Increasing the accuracy of the assembly fixture and metallic counterparts would result in higher accuracy of the resulting samples.

Stage 3 followed. The non-compliant samples from the previous stage had to be calibrated by turning to ensure their required accuracy before environmental testing. As Fig. 4 presents, the total runout of all the samples was satisfactory after calibration (see the highlighted part of columns compared to previous values in the light colour).

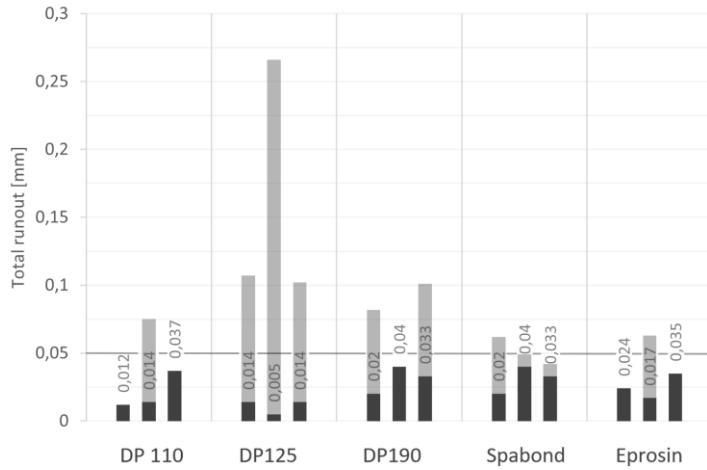


Fig. 4 Values of total runout after calibration

Then all the samples went through the climatic and mechanic tests. From the data presented in the graph in Fig. 5 the relationship between the machining (calibration) of the samples and the total runout deviation during the tests can be seen.

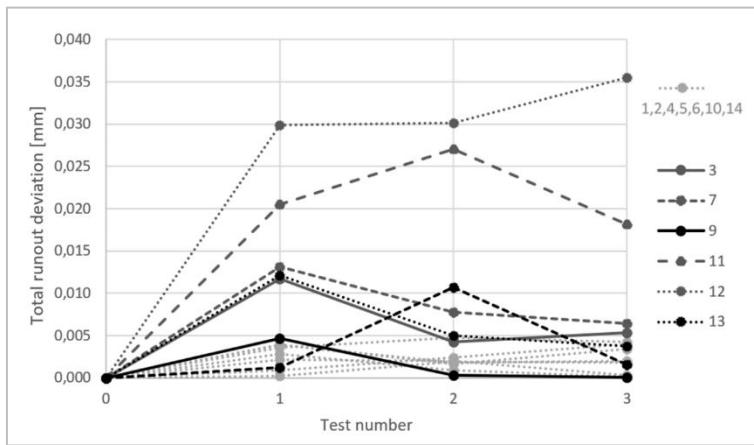


Fig. 5 Deviation of total runout during the environmental tests

A total of 7 samples underwent calibration turning because of unsatisfactory precision after bonding (1,2,4,5,6,10,14 – plotted in light grey). None of the calibrated samples exceeded the deviation of total runout more than by 0.01 mm. For 7 of the 8 calibrated samples (87,5 %) the deviation of total runout did not even exceed 0,005 mm. On the other hand, a total of 7 samples did not require calibration turning because their precision was satisfactory after bonding. 6 of the 7 (85 %) non-calibrated samples exceeded the 0.01 mm tolerance at least once during environmental testing. 2 out of 7 (28,5 %) non-calibrated samples exceeded the deviation of 0,025 mm at least once during environmental testing. From the above, a link between not calibrating and a tendency to reach a higher maximum deviation value of total runout is evident.

CONCLUSION

Technological samples consisting of a CFRP tube into which aluminium alloy sleeves were bonded using an assembly fixture. 46.6 % of the samples achieved the required precision after bonding. The failed samples were calibrated by turning. During the stability tests of the bonded joint, which consisted of two climate resistance tests and one mechanical resistance test, the following findings were observed:

- For 7 out of 8 calibrated samples (87.5 %), the deviation did not exceed 0.005 mm.
- In 6 out of 7 (85 %) of the noncalibrated samples, the deviation of 0.01 mm was exceeded at least once during the environmental tests, which is probably due to shrinkage stress in the bonded joint that was partially released during turning in the calibrated samples.

Based on these findings, it is evident that one of the main topics for further research will be thermal deformation in the bonded joint and its elimination. One of the possible causes of these deformations may be shrinkage stress. From the obtained results it is evident that it is important to deal with the problem further and it is likely that the potential findings will be used in practice.

ACKNOWLEDGMENTS

This work has been supported by the project No. FV 22 – 10 funded by The Ministry of Education, Youth and Sports (MEYS, MŠMT in Czech) support.

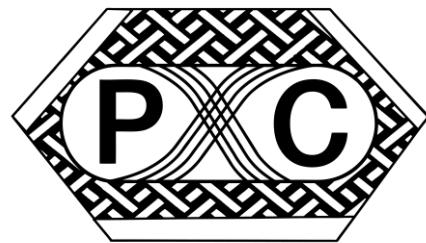
LITERATURE

1. QIAO, Yao, Lisa FRING, Madhusudhan PALLAKA a Kevin SIMMONS. A review of the fabrication methods and mechanical behavior of continuous thermoplastic polymer fiber–thermoplastic polymer matrix composites. *Polymer Composites*. ISSN 0272-8397. Dostupné z: doi:10.1002/pc.27139
2. KUPČÁK, Radim a Jan ZOUHAR. Application of composite materials in sports optics. *Manufacturing Technology*. 2020, 20(2), 200-209. ISSN 12132489. Dostupné z: doi:10.21062/mft.2020.038
3. CAMANHO, P a Stephen HALLETT, ed. *Composite Joints and Connections: Principles, Modelling and Testing*. Woodhead Publishing, 2016. ISBN 9781845699901.
4. PASTORE, R., A. DELFINI, M. ALBANO, A. VRICELLA, M. MARCHETTI, F. SANTONI a F. PIERGENTILI. Outgassing effect in polymeric composites exposed to space environment thermal-vacuum conditions. *Acta Astronautica*. 2020, 170, 466-471. ISSN 00945765. Dostupné z: doi:10.1016/j.actaastro.2020.02.019
5. KUPČÁK, Radim, Jan ZOUHAR a Lukáš GREGOR. Precision Bonding of CFRP Parts with Application in Sport Optics. *Polymer Composites*. Tábor, 2021.
6. 3M Scotch-Weld TM Epoxy Adhesive DP110 Translucent and Gray [online]. In: . 3M Industrial Adhesives and Tapes Division, 2019 [cit. 2023-01-08].
7. 3M Scotch-Weld TM Epoxy Adhesives DP125 Translucent and Gray [online]. In: . 3M Industrial Adhesives and Tapes Division, 2009 [cit. 2023-01-08].
8. 3M Scotch-Weld TM Epoxy Adhesives DP190 Translucent and Grey [online]. In: . 3M Industrial Adhesives and Tapes Division, 2016 [cit. 2023-01-08].
9. SPABOND™ 340LV HT HIGH TG STRUCTURAL EPOXY ADHESIVE: Full General Datasheet [online]. In: . Gurit, 2015 [cit. 2023-01-08].
10. Eprosin Flex: Technical datasheet [online]. In: . Stachema, Divize průmyslová lepidla, 2021 [cit. 2023-01-08].

POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



MORPHOLOGY AND PERFORMANCE OF NATURAL FIBRE-REINFORCED AND BIOBASED-FILLED POLYMER MATERIALS

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The present contribution gives short insight into the long-time successful co-operation between German and Nepal groups dealing with biodegradable/biorebased and natural fiber-reinforced/biorebased-filled polymer materials. The first example is dealing with the composting behavior and the properties of PBAT/nano-cellulose composites and the second example with the morphology and the properties of biobased polymer blend filled with biogenic nano-hydroxyapatite.

KEYWORDS

Nanocomposites, PBAT, PCL/PLLA/gelatin blend, nanocrystalline cellulose, nano-hydroxyapatite, bone tissue engineering, biocompatibility, biodegradability, composting

INTRODUCTION

Unmanaged dumping of the polymeric waste is causing environmental problems. Natural nanofibers from plant sources such as nanocrystalline cellulose (NCC) [1–3] – extracted by different thermo-chemical and mechanical methods [4] – can enhance the biodegradability of the polymer composites. Incorporation of such nanofibers in biodegradable polymer results in good biocompatibility and are reliable for various high-tech applications such as biomedical applications, implants materials, tissue engineering, filtration device, permeable membrane, packaging materials, cell culture, paints, screen coatings, low load-bearing materials, furniture and many more [5–8]. Although there are some works done to prepare NCC-fabricated degradable polymer nanocomposites [2, 9–11] more detailed information concerning the degradation behavior and properties of NCC-fabricated poly (butylene adipate-co-terephthalate) (PBAT) nanocomposites are needed. In this work, NCC is extracted from agricultural waste, the wheat stalk, by thermochemical treatments and melt mixed with PBAT to prepare nanocomposites with various concentrations of the NCC (0 – 10 %). The composites are studied for their spectroscopic, morphology, thermogravimetric analysis and mechanical

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deformation behavior (for detailed information about the preparation of NCC and the composites as well the used characterization techniques see [12]). The novelty of this work is the study of degradation susceptibility for the composites by measuring its water absorption with absorption kinetics, contact angle measurements for water droplets i.e. wettability and validating soil composting behavior by micrographs of the composted specimens.

Recently, composites of biopolymers and bioceramics have attracted increasing attention in the scientific community for their use as scaffolds in bone tissue engineering (BTE). Polycaprolactone (PCL), poly L-lactic acid (PLLA), and gelatin are one of the most studied biopolymers because of their nontoxicity, excellent biocompatibility, and biodegradable nature [13, 14]. Furthermore, the US Food and Drug Administration (FDA) has approved PCL and PLLA for clinical use in humans [15–17]. For decades, the scaffolds of these biopolymers have been used in various biomedical applications despite their weak mechanical properties [18–20]. Therefore, there have been numerous studies to enhance the mechanical properties of these scaffolds by incorporating different filler materials where the bioactive and biocompatible nature nano-hydroxyapatite (nano-HAp) is extensively used as a filler material to enhance the mechanical properties and osteoconductivity of scaffolds for BTE applications [13]. As this ceramic has a chemical composition similar to the inorganic component of natural bone minerals, biopolymer-based scaffolds filled with nano-HAp have received a keen interest in BTE [13, 21–24]. In the past decades, several studies have attempted to explain the mechanical behavior of HAp-blended electrospun binary composite scaffolds of PCL/PLLA, PCL/gelatin, and PLLA/gelatin [25–28]. However, the micromechanical behaviour of nano-HAp- and vitamin D3-incorporated ternary blends of PCL/PLLA/gelatin scaffold has been rarely studied. Hence, in this work, we aim to fabricate non-woven fibrous scaffolds with different volume fraction percentages (0 – 12 %) of nano-HAp/TFE suspension blended into ternary composite mixtures of PCL/PLLA/GEL by electrospinning technique and to study the effect of filler addition on the micromechanical behaviour after tensile test of the scaffold fibers (for detailed information about the preparation of nano-HAp and the nanocomposites as well the used characterization techniques see [29–31]).

PBAT/NCC NANOCOMPOSITES FOR PACKAGING MATERIALS

Hydrophilic properties such as water absorption capacity and wettability of the materials increase with the NCC content in the PBAT/NCC nanocomposites thereby enhancing their biodegradability. The soil composting is induced by the morphological transformation due to bacterial and fungal attacks. Moreover, this composting adds more fragility to the nanocomposites prepared with increased NCC content.

After 2 month composting higher biodegradability of the nanocomposite compared to the neat PBAT can clearly detected on the fracture surfaces (Fig. 1a). The NCC agglomerates were found to be disappeared to form big voids. A closer look into Fig. 1b observed many oval spiny microbes on fracture surfaces. It clearly shows that the specimens with high NCC weight fraction get much affected by the microbes on soil composting.

Underlying structure–properties correlations in PBAT/NCC nanocomposites regarding theirs structural, mechanical, thermal and surface properties are concluded as follows. The structural characterization of the nanocomposites by spectroscopic, microscopic and diffraction techniques reveals that the nanocomposites comprise the compatible and phase-segregated constituents which keep their identity in the nanocomposite materials. The morphology of the fillers and matrix remains unchanged. Analysis by Fourier transform infrared (FTIR) spectroscopy showed that the NCC phase is homogeneously dispersed in PBAT matrix inside the nanocomposites and both phases are held together by physical interactions. The analysis by

scanning electron microscopy (SEM) also manifested the homogeneously dispersed NCC in PBAT matrix.

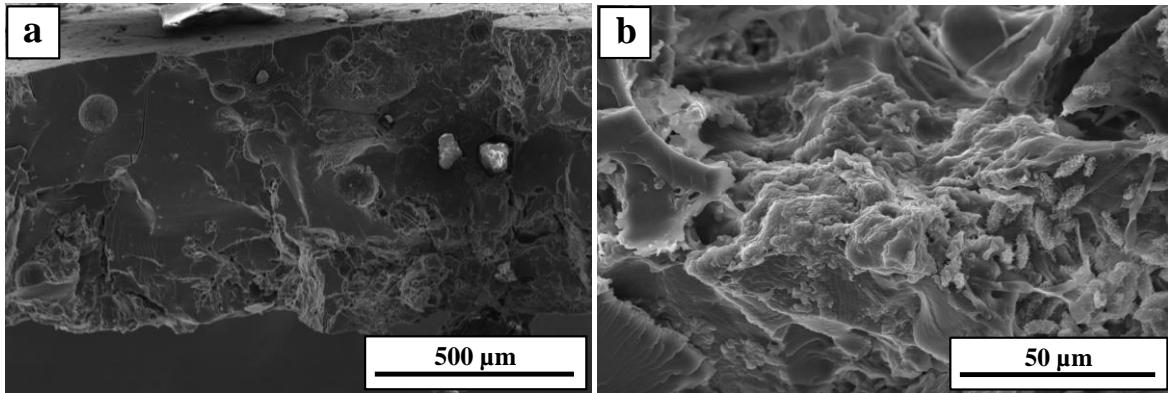


Fig. 1 Micrographs showing fracture surface of 2 months soil compost: N-10 (a) low resolution (500 μm) and (b) high resolution (50 μm)

The PBAT/NCC nanocomposites are thermally stable within their application ranges, showing two stages of degradations for NCC at 250 °C and for PBAT at 430 °C, separately. This means that the investigated nanocomposites are found to be stable within their desired application temperature as packaging materials.

Tab. 1 Values of the mechanical parameters of PBAT and different nanocomposites

NCC content (%)	Tensile test		Recording microindentation test		
	Tensile strength (MPa)*	Strain at break (%)*	Martens hardness (N/mm ²)	Indentation modulus (MPa)	Elastic–plastic work of deformation ratio
0	16.01	835	6.86±0.11	118±1	3.07±0.24
1	11.93	810	6.97±0.21	119±3	2.98±0.09
2	7.49	398	4.53±1.60	73±29	3.62±1.23
5	6.25	345	6.17±0.51	103±10	2.77±0.37
10	9.98	624	5.99±0.65	104±10	2.47±0.41

*Mean standard deviation of tensile strength: 2.45 MPa, mean standard deviation of strain at break: 226 %.

Applying the uniaxial tensile test (Tab. 1) the tensile properties of the nanocomposites are found to degrade in terms of strain at break, tensile strength and tensile modulus. By using the recording microindentation test (Tab. 1), the initial increase in the elastic work relative to plastic work during indentation correlates well with the decrease in the hardness values, attributable to the softening effect found by uniaxial tensile testing. However, the reinforcing effect dominated at higher filler content leading to an increase in indentation modulus and hardness, and a decrease in the work of elastic deformation.

NANO-HAP-BLENDED NANOCOMPOSITES FOR BONE TISSUE ENGINEERING

For bone tissue engineering, nano-HAp is a widely used bioceramic filler in polymer fibrous scaffolds that changes the morphology and micromechanical behaviour of fibers. In this study, different volume fraction percentages of nano-HAp and vitamin D3 were incorporated into electrospun polymer blend fibrous scaffolds comprising PCL, PLLA and gelatin. FTIR, SEM, transmission electron microscopy (TEM), and uniaxial tensile testing were used to characterize the chemical, morphological, and micromechanical properties of the scaffolds.

The FTIR analysis showed the presence of functional groups of fillers and polymers and their molecular interactions in the fibers. In the microscopic analysis, the fillers were found to affect the fiber morphology (including diameter, phase separation, and texture; see Tab. 2 and inserts in Fig. 2) and the response of the fibers under tensile deformation. The addition of 3 %

by volume of filler suspension resulted in maximum diameter of 4.34 μm , and the addition of 6 % to 12 % filler led to an increase in tensile strength from 0.7 MPa to 5.6 MPa and strain at break from 2 % to 37 % for the composite fibers compared to neat fibers (Fig. 2 and Tab. 2). The crazing behaviour of the fiber is more noticeable at a low filler content (0 %), whereas thin-layer yielding becomes predominant at a higher filler content (12 %) [30, 31]. Thus, the study shows that scaffold fibers undergo brittle-to-ductile transitions as the filler percentage in the polymer blend increases from 0 to 12 %. This transition in microdeformation mechanism was observed in the microphase-separated electrospun fibrous scaffolds but not in the macrophase-separated solution-cast films which are very brittle independently of the nano-HAp content (Tab. 2).

In cell-line experiments high concentration of osteoblasts has been further found on electrospun mats containing nano-HAp (Fig. 3) indicating highly improved biocompatibility.

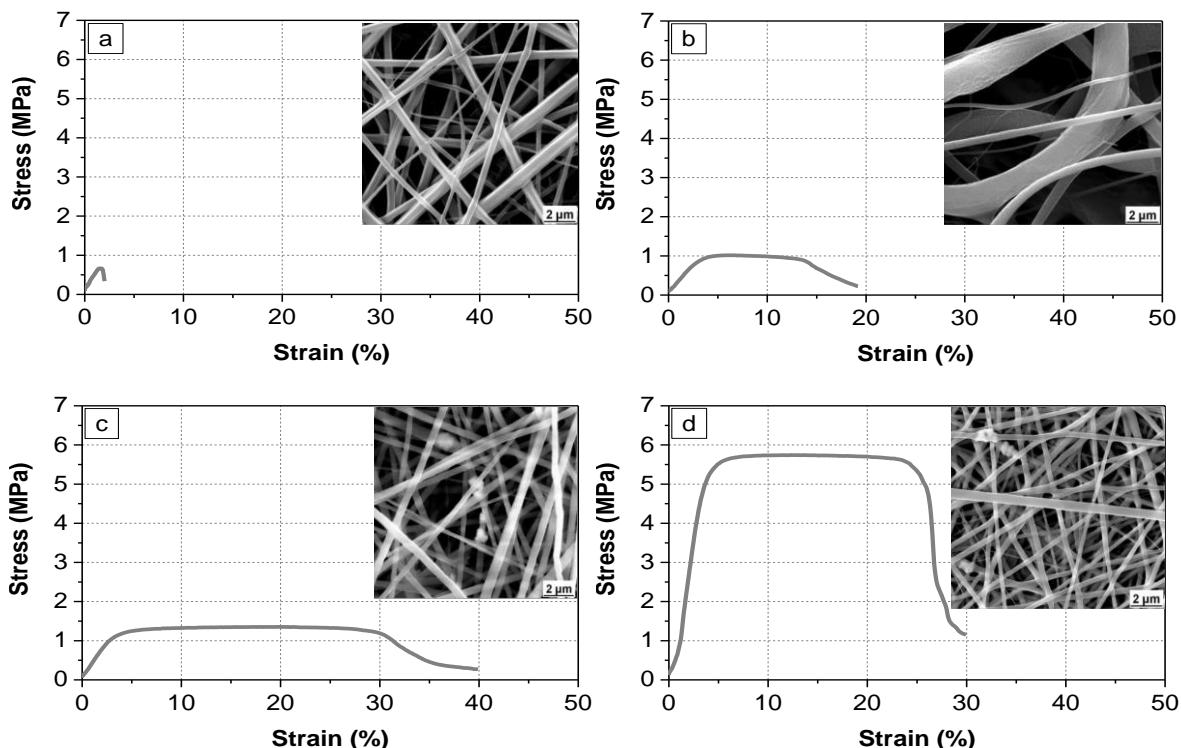


Fig. 2 Stress–strain curves of fibrous scaffolds as a function of nano-HAp fraction and related SEM images: 0 % (a), 2 % (b), 6 % (c) and 12 % (d) nano-HAp

CONCLUSION

Structural, mechanical, thermal and surface properties of nanocomposite polymer materials were studied comprising biodegradable aromatic–aliphatic PBAT reinforced with wheat stalk-based bio-NCC. The NCC phase was found to be homogeneously dispersed inside in the polymer matrix even at high volume fractions. The tensile properties of the nanocomposites degraded in terms of strain at break and tensile strength. At higher filler content, the reinforcing effect dominated leading to an increase in indentation modulus and hardness. The wettability and the water absorption capacity of the materials increased with NCC content thereby enhancing the biodegradability of the composites.

Tab. 2 Average fiber diameter of electrospun fibrous scaffolds and mechanical properties of the scaffolds and solutions cast films

Volume of nano-HAp suspension in polymer blends (% v/v)	Electrospun fibrous scaffolds			Solutions cast films	
	Average fiber diameter (μm)	Mechanical properties			
		Elastic modulus (MPa)	Tensile strength (MPa)	Strain at break (%)	
0	0.17	52 \pm 16	0.68 \pm 0.28	2.18 \pm 0.67	1.81
2	1.84	50 \pm 41	1.08 \pm 0.31	22.6 \pm 8.5	—
3	4.34	31 \pm 18	0.90 \pm 0.31	17.7 \pm 4.7	1.25
6	0.58	41 \pm 7	1.46 \pm 0.16	36.9 \pm 6.3	1.54
12	0.48	199 \pm 40	5.62 \pm 0.98	27.3 \pm 6.1	0.50

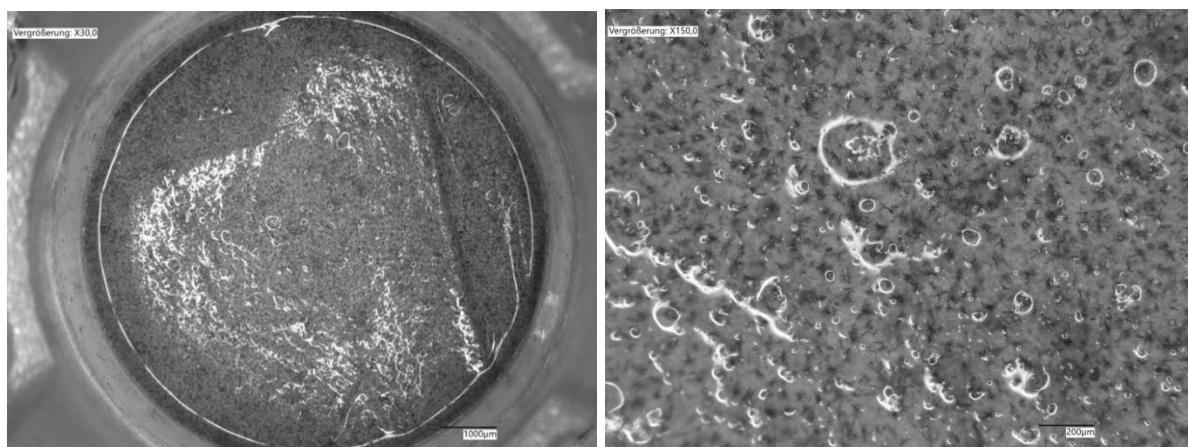


Fig. 3 High concentration of cells (osteoblasts) on electrospun mats with 6 % nano-HAp

Non-woven mat-like scaffolds for bone regeneration were fabricated using a biobased polymer blend composed of PCL, PLLA and gelatin by electrospinning which were partly modified using vitamin D3. The blend was reinforced with 0 – 12 % biobased nano-HAp – extracted from ostrich bones – to improve both biocompatibility and mechanical performance. Both elastic modulus, tensile strength and strain at break were highly increased by incorporation of nano-HAp. Thus, filler addition improved the fiber's microdeformation behaviour corresponding to a brittle-to-ductile transition, i.e. a transition from highly localized crazing to non-localized thin-layer yielding. Additionally, electrospun nano-HAp-filled blends were characterized in cell line experiments using osteoblasts confirming excellent biocompatibility.

ACKNOWLEDGEMENT

JG sincerely acknowledges the Indian National Science Academy (INSA) – Nepal Academy of Science and Technology (NAST) collaboration for providing financial support for her 3 months research stay in the Indian Institute of Guwahati (IITG) Assam, India. KPM thanks German Academic Exchange Service for financial support during his stay in Germany (grant 57507869). RL and SH acknowledge German Research Foundation for funding of the research project MI 358/37-3. RA thanks the University Grants Commission (UGC) for the grant CRG73/74-ST-02. Furthermore, the authors wish to thank Dr. Andre Wutzler (PSM) for conducting the FTIR investigations. The cell line experiments were kindly done by Dr. Andrea Sowislok (Universitätsklinikum Essen, Germany).

LITERATURE

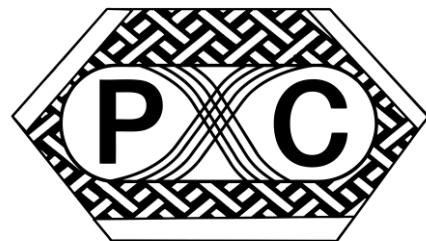
- Hasan M, Lai TK, Gopakumar DA, Jawaid M, Owolabi FAT, Mistar EM, Alfatah T, Noriman NZ, Haafiz MKM, Abdul KHPS. J Polym Environ 27 (2019) 1602

2. Giri J, Lach R, Grellmann W, Susan MABH, Saiter JM, Henning S, Katiyar V, Adhikari R. *J Appl Polym Sci* 136 (2019) 48149
3. Giri J, Adhikari R. In: Katiyar V, Kumar A, Mulchandani N (eds). *Advances in Sustainable Polymers, Materials Horizons: From Nature to Nanomaterials*, Springer: Singapore (2020), 289
4. Giri J, Adhikari R. *BIBECHANA* 9 (2013) 81
5. Mariano M, El KN, Dufresne A. *J Polym Sci Part B: Polym Phys* 52 (2014) 791
6. Siro I, Plackett D. *Cellulose* 17 (2010) 459
7. Baghban MH, Mahjoub R. *Appl Sci* 10 (2020) 357
8. Tavares TD, Antunes JC, Ferreira F, Felgueira HP. *Biomolecules* 10 (2020) 148
9. Kaboorani A, Riedl B, Blanchet P, Fellin M, Hosseinaei O, Wang S. *Eur Polym J* 48 (2012) 1829
10. Yousefian H, Rodrigue D. *Polym Compos* 37 (2014) 1473
11. Mukherjee T, Tobin MJ, Puskar L, Sani MA, Kao N, Gupta RK, Pannirselvam M, Quazi N, Bhattacharya S. *Cellulose* 24 (2017) 1717
12. Giri J, Lach R, Henning S, Grellmann W, Bhasney SM, Katiyar V, Saiter JM, Adhikari R. *Polym Bull* (2022)
13. Shuai C, Yang W, Feng P. *Bioact Mater* 6 (2020) 490
14. Scaffaro R, Lopresti F, Botta L. *Eur Polym J* 96 (2017) 266
15. Remya KR, Joseph J, Mani S, John A, Varma HK, Ramesh P. *J Biomed Nanotechnol* 9 (2013) 1483
16. Su Z, Li J, Ouyang Z, Arras MM, Wei G, Jandt KD. *RSC Adv* 4 (2014) 14833
17. Zhang Y, Liu X, Zeng L, Zhang J, Zuo J, Zou J, Ding J, Chen X. *Adv Funct Mater* 29 (2019) 1903279
18. Ba Linh NT, Min YK, Lee BT. *J Biomater Sci: Polym Ed* 24 (2013) 520
19. Nithya R, Sundaram NM. *Int J Nanomedicine* 10 (2015) 119, Supplement 1
20. Shitole AA, Raut PW, Sharma N, Giram P, Khandwekar AP, Garnaik B. *J Mater Sci: Mater Med* 30 (2019) 1–17
21. Abdal-hay A, Abbasi N, Gwiazda M, Hamlet S, Ivanovski S. *Eur Polym J* 105 (2018) 257
22. Kim CH, Khil MS, Kim HY, Lee HU, Jahng KY. *J Biomed Mater Res Part B: Appl Biomater* 78 (2006) 283
23. Ni P, Bi H, Zhao G, Han Y, Wickramaratne MN, Dai H, Wang X. *Colloids Surf B: Biointerfaces* 173 (2019) 171
24. Yang X, Li Y, He W, Huang Q, Zhang R, Feng Q. *J Biomed Mater Res: Part A* 106 (2018) 2863
25. Andersson RL, Ström V, Gedde UW, Mallon PE, Hedenqvist MS, Olsson RT. *Sci Rep* 4 (2014) 6335
26. Hossan MJ, Gafur MA, Karim MM, Rana AA. *Bangladesh J Sci Ind Res* 50 (2015) 15
27. Sánchez-Arévalo FM, Muñoz-Ramírez LD, Álvarez-Camacho M, Rivera-Torres F, Maciel-Cerda A, Montiel-Campos R, Vera-Graziano R. *J Mater Sci* 52 (2017) 3353
28. Selezneva M, Swolfs Y, Katalagarianakis A, Ichikawa T, Hirano N, Taketa I, Karak T, Verpoest I, Gorbatikh L. *Compos Part A: Appl Sci Manuf* 109 (2018) 20–30
29. Malla KP, Regmi S, Nepal A, Bhattarai S, Yadav RJ, Sakurai S, Adhikari R. *Int J Biomater* 2020 (2020) 1690178
30. Malla KP, Henning S, Lach R, Jennissen H, Michler GH, Beiner M, Yadav RJ, Adhikari R. *Macromol Symp* 403 (2022) 2200079
31. Malla KP, Henning S, Lach R, Jennissen H, Michler G, Beiner M, Adhikari R. *Procedia Struct Integr* 43 (2023) 71

POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



NEW GENERATION SANDWICH PANEL FOR BALLISTIC RESISTANCE FB1-FB4

SENDVIČOVÝ PANEL NOVÉ GENERACE PRO BALISTICKOU ODOLNOST FB1-FB4

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Civil and infrastructure security as well as army strategical fortifications has always been a priority, nowadays even more due to the current geopolitical situation. A plethora of ballistic protection objects have been developed, from shield jacket to high resistance panels. The Czech Technical University in Prague (ČVUT) has commercialized an ultra-high-performance concrete (UHPC) for a whole range of projectiles from civilian FB4 standard ČSN EN 1522 to NATO Stanag 2280 level A4. Those composite panels show an outstanding ballistic resistance, even though a certain thickness is required to avoid any harmful concrete fragment generation called scabbing effect. To tackle this issue and increase the ballistic performance of the current UHPC, a next generation multilayer sandwich has been designed by the help of a finite element model. Prototypes for FB1 to FB4 protection optimized for civil engineering consisted in two polymeric layers glued on a 30 mm UHPC core by a structural epoxy adhesive. These polymeric panels were a combination of a 3 mm fibre reinforced composite, and a 5 mm filled polyurethane elastomer. The latter material acted as a first barrier against projectiles and as a flame retardant for the fibre reinforced composite which slows down the projectile enough to avoid back stripping. The elastomer showed a constant damping from -35 °C to 50 °C and fair fire retardancy. The fibre reinforced composite has been designed from a twill wave E-glass fabric impregnated with an epoxy-amine resin having a glass transition temperature of 80 °C. The whole sandwich structure has been certified by VTÚ (Vojenský technický ústav) for the FB1-FB4 ballistic resistance range. In addition, it resulted in weight reduction of 29 wt. % compared to the 5cm sole UHPC required to fulfill the standard FB4.

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KEYWORDS

Sandwich panel, Elastomer, Fibre reinforced composite, Ultra-high-performance concrete, Ballistic resistance

INTRODUCTION

With the development and research in the field of stable protective and defense structures and objects, the use of high-performance cement composites (UHPC) is increasingly coming to the fore recently as a very suitable solution for ballistic protection (1) (2). In addition to their main function, UHPC also meet economic requirements and are thus often deployed while maintaining a relatively slim and subtle design (3). Despite all the efforts in the development of these materials, it is difficult to suppress the formation of the front and rear craters, and with the impact of the projectile, dangerous secondary fragments can be generated which may be very injurious in the immediate vicinity (sometimes more damaging than the effect of the bullet itself) (4). The solution is to connect the UHPC carrier layer with advanced polymer composite layers of suitable composition into a sandwich panel with high ballistic resistance, which stops the projectile without any release of secondary fragments into its surroundings.

Compared to available existing protective structures, this sandwich structure has several advantages: versatility of use, flexibility in structure and formulation, the possibility of using renewable raw materials and nanomaterials, as well as rapid transfer to production. Contemporary non-metallic armors are based on fiberglass, aramide, special concrete, polycarbonate, or ceramic/elastomer sandwiches, (5) (6) etc. The implementation of an additional composite polymer layer on concrete, according to available information, has not been used yet. This additional layer should combine the high energy dissipation and flexibility of an elastomer with the high impact resistance provided by an aramid/epoxy or glass/ epoxy composite. The primary function of the polymer layer is to prevent the formation and release of concrete fragments after being hit by a projectile. In the case of a ballistic resistance up to FB4, the following structure has been chosen: 30 mm UHPC, 3 mm glass/epoxy composite and 5 mm elastomer. In this work we will focus on the panels design and the added value of the polymeric layer to UHPC.

MATERIAL DESCRIPTION

Each layer of the sandwich panels is detailed in Fig. 1 (not at scale) and Tab. 1, Tab. 2 and Tab. 3. The preparation of a ballistic panel occurs as follow:

- 1) Preparation of the epoxy/glass composite
- 2) Casting of the elastomer on the epoxy glass composite
- 3) Penetration of UHPC with 100% solid primer (CHS Epoxy 474 primer)
- 4) Gluing of the polymeric layer (elastomer + composite) on the UHPC

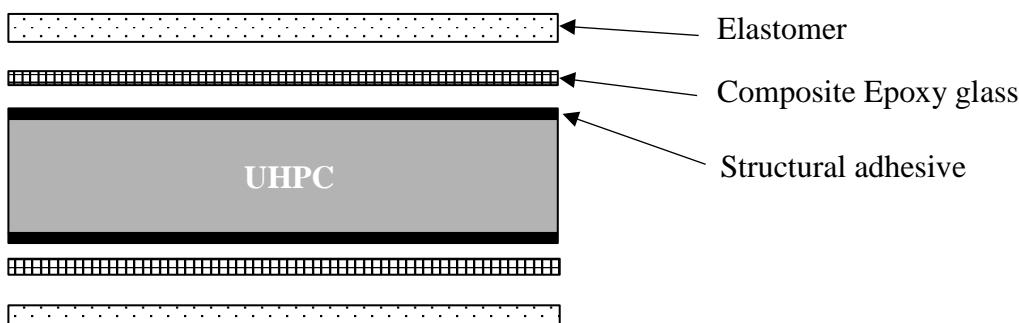


Fig. 1 Structure of a new generation ballistic panel

Tab. 1 Characteristics of the elastomer layer

Characteristics	Filled two component PU elastomer
Viscosity 25 °C (Brookfield CAP 2000+)	13.305 ± 0.182 Pa·s
Density 23 °C (ISO 2811-1)	1.182 g/cm ³
Weight ratio (comp. A + comp.B)	100 + 13
Filling rate	30 wt. %
Curing	24 h/23 °C + 3 h/80 °C
Preparation	casting

Tab. 2 Characteristics of the epoxy structural adhesive

Characteristics	Filled two component PU elastomer
Viscosity 25 °C (Brookfield CAP 2000+)	Thixotropic
Density 23 °C (ISO 2811-1)	1.55 g/cm ³
Weight ratio (comp. A + comp.B)	100 + 40
Preparation	hand spreading

Tab. 3 Characteristics of the epoxy glass composite

Glass fabric	INTERGLAS™ 92125, 280 g/m ² , twill 2/2
Number of plies	13
Binder	E582-T0542 (100+32)
Curing	1 h/RT + 6 h/45 °C + 2 h/120 °C
Composite thickness	3.1 ± 0,1 mm
Composite density	1.82 g/cm ³
Surface weight fabric	5.6 kg/m ²
Fiber weight/volume content	61 ± 2 wt. % /44 ± 2 vol. %.
Preparation	hand lay-up

RESULTS

Design (numeric finite element model)

The impact of a projectile into a sandwich composite composed of an elastomer, a glass fabric reinforced composite (GFRC) and a core (UHPC) of high-quality fiber-reinforced concrete was simulated. The elastomer layers constituted the surface on both sides of the sandwich panel. The GFRC were below them, glued to the UHPC core. UHPC, GFRC and elastomer thicknesses were kept constant in all simulations. The thickness of the core was 30 mm, the thickness of the elastomer was 5 mm, the thickness of the epoxy composite was 3 mm. The properties of the contact between the elastomer and the GFRC were unknown, so its damage was not considered and it was created using finite element node sharing. The projectile under consideration was of the .357 Magnum type. According to EN 1522:1998, the initial velocity of the projectile was considered to be 430 m/s. The Simulation of ballistic resistance of sandwich panels was performed with Ansys mechanical software. The damages induced by such of projectile are shown in Fig. 2.

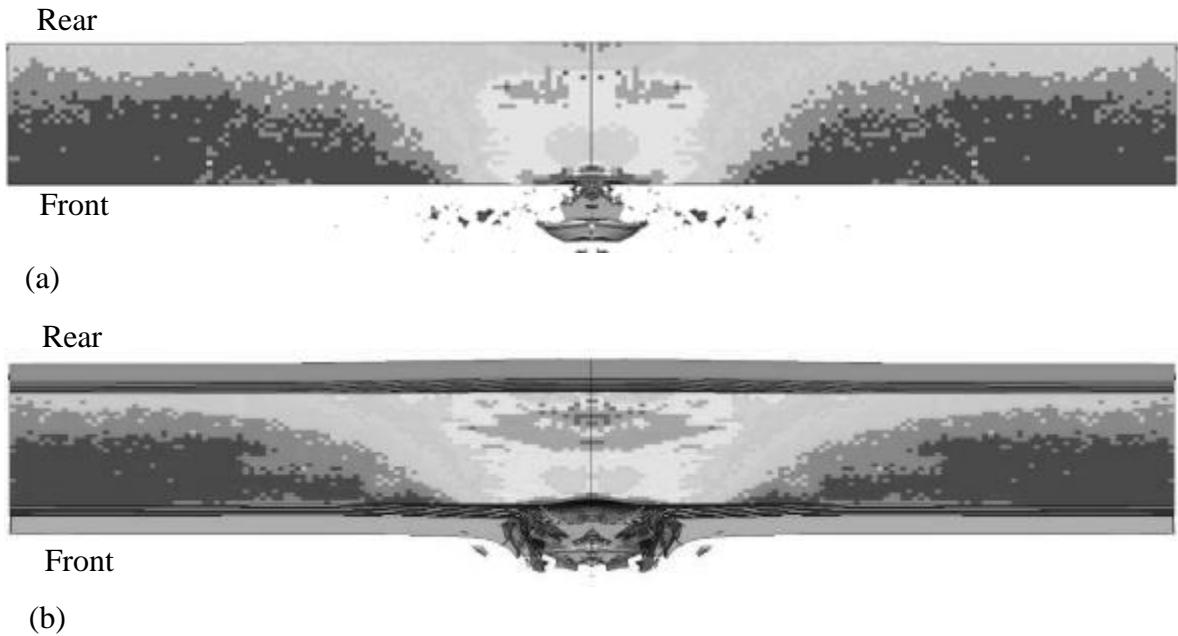


Fig. 2 Simulation of damaged induced by a 357 magnum projectile
 (a) Sole 3mm UHPC
 (b) Sandwich panel 30 mm UHPC, 3 mm GFRC, 5 mm elastomer

As can be seen, without the polymeric layer, a 30 mm thick UHPC would probably not pass the FB4 certification as scabbing would occur. Significant damages are seen even in the case of the upgraded UHPC, but the those are more spread in the core thickness. At the same time, the thickness of the panel cannot be too high, in order to avoid oversizing and thereby increasing of weight and price. Material properties of the polymeric layer (elastomer, epoxy glass composite and adhesive) were measured in the SYNPO lab department; the details are given in the next chapter.

Polymeric layer properties

The polymeric layer protecting layer for the FB1-FB4 ballistic resistance was composed of a 3 mm epoxy composite and a 5 mm elastomer. The whole layer is glued on the UHPC surface. Tab. 4, Tab. 5 and Tab. 6 details the properties used for modelling.

Tab. 4 Properties of the 3 mm GFRC

FVC (tested panel) [%]	42.2 ± 1.2
T_g (tested panel) DMA (ISO 6721-7), ONSET G' [°C]	74
Tensile (ISO 527-4)	E [GPa]
	σ^{\max} [MPa]
	ϵ [%]
Flexural (ISO 178)*	M [GPa]
	σ^{fM} [MPa]
	ϵ^{fM} [%]
Interlaminar Shear Strength (ILS) [MPa]**	42.5 ± 0.9

*Characterization will be completed with ISO 14125 standard for more precise results

**Values are given for orientation purposes due to an invalid rupture type

Tab. 5 Properties of the 5 mm elastomeric material

Tensile (ASTM 1708 mikro) 100 mm/min; 23 °C	σ^{\max} [MPa]	3.52 ± 0.25
	ε [%]	121 ± 10
Adhesion (epoxy substrate)	σ^{\max} [MPa]	2.36 ± 0.34
Tear resistance (ISO 34-1)	Strength A [kN/m]	1.7 ± 0.09
	Median peak [N]	3.29 ± 0.19

Tab. 6 Properties of the structural epoxy adhesive

T_g (tested panel) DMA (ISO 6721-7), ONSET G' [°C]	65
Density 23 °C (ISO 2811-1)	1.4 g/cm ³
Shear strength (ČSN EN 1465)	19.3 ± 1.2 MPa
Maximal strength T peel [N/cm] (ISO 11339)	8.45 ± 0.93

Ballistic test results

Ballistic panels for FB1-FB4 resistance certification were submitted to VTÚ (Vojenský technický ústav). Two standard types of munitions were used for testing .357, MAG and .44 Rem MAG. Fig. 3 shows the damages encountered by the panels. As can be seen, neither front nor rear fragments were ejected from the samples validating the certification acc. ČSN EN 1522.

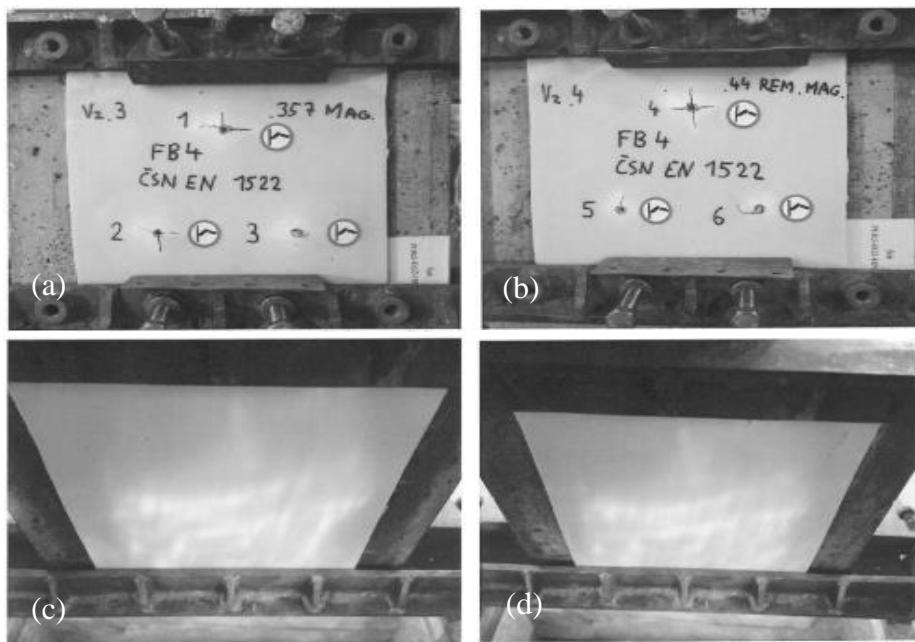


Fig. 3 Appearance of panels after shooting (a) 357, Magnum front (b) .44 Rem Magnum front
(c) 357, Magnum rear (d) 44 Rem Magnum rear

Price and weight comparison

The impact of the polymeric layer addition was evaluated considering the weight saving and inevitable price increase. The new generation panel with a 30 mm UHPC core was compared to a sole 50 mm UHPC withstandng the FB4 ballistic resistance. Fig. presents the comparison per a one-meter square panel. As can be observed, there was a significant weight reduction of almost 30 wt. %. This together with an increased security due to the elimination of the scabbing effect (primary goal of the UHPC upgrade), makes the new generation sandwich panel efficient for ballistic purposes. A rough estimation of costs was calculated. A significant

increase of price was expected, nevertheless it can be reduced by further optimizing the formulation of the polymeric layers.

CONCLUSION

The new generation of ballistic panels based on UHPC were designed to prove resistance up to level FB4, with complete suppression of scabbing and reduction of panel requisite weight.

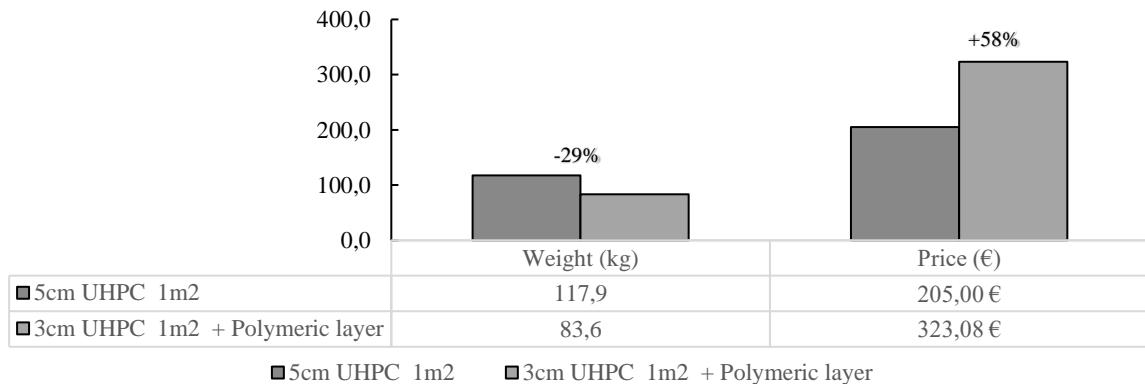


Fig. 4 Price and weight comparison of UHPC and new generation ballistic panel withstanding the FB4 resistance

Next, the panels design will be upgraded for higher ballistic resistance such as FB5 to FB7 and finally Stanag A1 to A4. Accelerated aging will be performed on panels to complete the FB1- FB4 resistance certification.

ACKNOWLEDGMENT

This work has been supported the Technological Agency of Czech Republic (TACR) within the project program TREND3, number FW03010141.

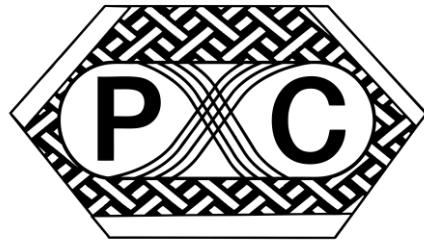
LITERATURE

1. Multi-Functional High-perfomance Cement Based Composite. Bažantová Z. et al. 2016, Key Eng. Mater, 53-56.
2. Development of a new Ultra High Performance Fiber Reinforced Cementitious Composite (UHPFRC) for impact and blast protection of structures. Nicolaides D. et al. 2015, Constr. Build. Mater., 667-674.
3. Resistance of slim UHPFRC targets to projectile impact using in-service bullets. Sovják R. et al. 2015, Int. J. Impact Eng., Tom 76, 166-177.
4. Experimental procedure for determination of the energy dissipation capacity of ultra-high performance fiber-reinforced concrete under localized impact loading. Konrád P., Sovják, R. 2019, Int. J. Prot. Struct., 251-265.
5. Advanced preped ballistic composites for military helmets. Dimeski dimko a Vineta Srebrenkoska. 2014. 6th International Scientific Conference on Defensive Technologies.
6. Optimizing viscoelastic properties of rubber compounds for ballistic applications. Karl Janis, Franziska Kirsch, Norbert Faderl, Leonard Perko a Teresa Fras. 2020, Applied science (Switzerland), 10, 1-15.

POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



VISCOELASTIC MATERIAL MODELS OF POLYMER COMPOSITES

VISKOELASTICKÉ MATERIÁLOVÉ MODELY POLYMERNÍCH KOMPOZITŮ

ANTONÍN POTĚŠIL¹, PETR HORNÍK², MILAN ŠTÍPEK³, ANNA LUCIOVÁ⁴

The demands of industrial development require the use of CAE approaches to predict and evaluate the time response of structures containing hybrid structures composed of structural polymer composites. This paper discusses options that account for the viscoelastic behaviour of materials as a function of time or frequency under operationally standard loading and temperature conditions, typically in the temperature range of 23 to 120 °C.

Požadavky průmyslového vývoje vyžadují aplikovat CAE přístupy, které by umožnily predikovat a ocenit časovou odezvu konstrukcí obsahujících hybridní struktury složené z konstrukčních polymerních kompozitů. Příspěvek diskutuje možnosti, které respektují časové nebo frekvenční viskoelastické chování materiálů za provozně standardních zatežovacích a teplotních podmínek, zpravidla v intervalu teplot 23 až 120 °C.

KEYWORDS

Viscoelasticity, CAE numerical simulations, Finite Element Method

INTRODUCTION

In the current practice of predictive CAE simulations, the stresses of structural composite material structures and their assemblies are usually evaluated and assessed according to the strain and stress levels in an elastic anisotropic/ortho-tropic continuum or an elasto-plastic isotropic continuum. It is evident that it is not possible to make an educated guess, by means of the mentioned material models, as to what states of stress and strain occur in these materials under the assumption of their viscoelastic response induced mainly by the operating temperature, or under the normatively prescribed thermal-climatic conditions. A requirement and necessity for informed CAE predictions is the knowledge of viscoelastic material models, including failure criteria for structural composites from longer-term temperature-influenced perspectives.

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1. RHEOLOGICAL MANIFESTATIONS OF MATERIALS DURING LOADING AND UNLOADING

The rheological manifestations of substances are associated, among other things, with phenomena known as viscoelasticity/relaxation/creep. These phenomena are spontaneous material processes activated by external energy supply and subsequently by transport thermodynamic phenomena occurring both in the material itself and in contact with the surrounding environment, with varying intensities depending on the environmental conditions [1, 2], see basic diagrams in Fig. 1.

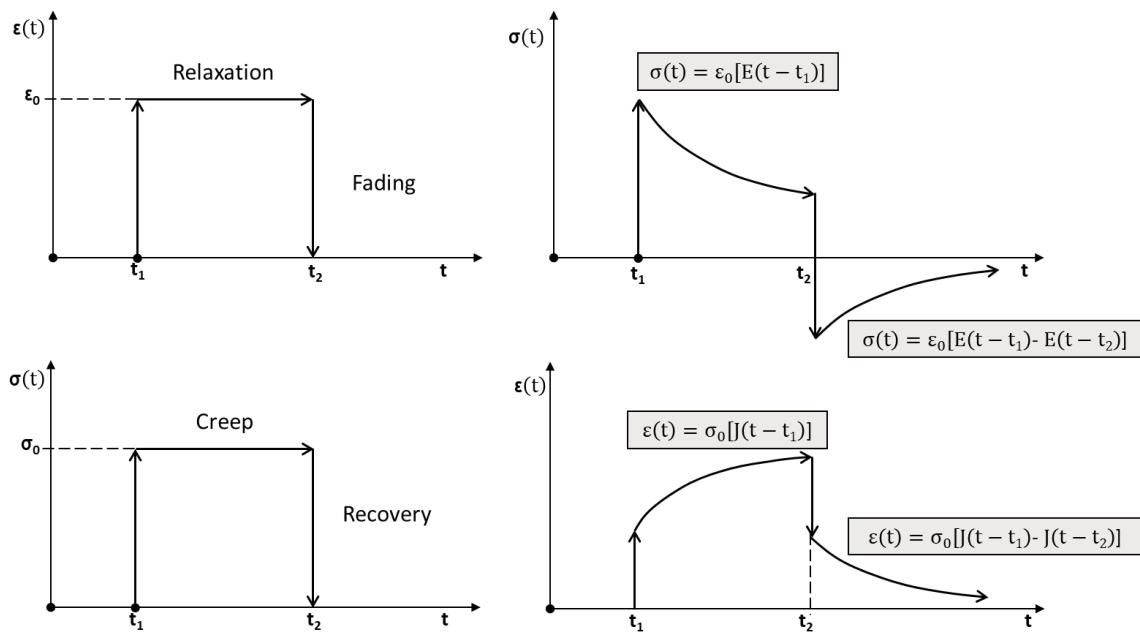


Fig. 1 Viskoelasticita – relaxace/creep

2. VISCOELASTIC MODELS OF MATERIALS

Basic rheological models of substances, see Fig. 2, cannot describe the real response of materials used in engineering practice due to their simplicity.

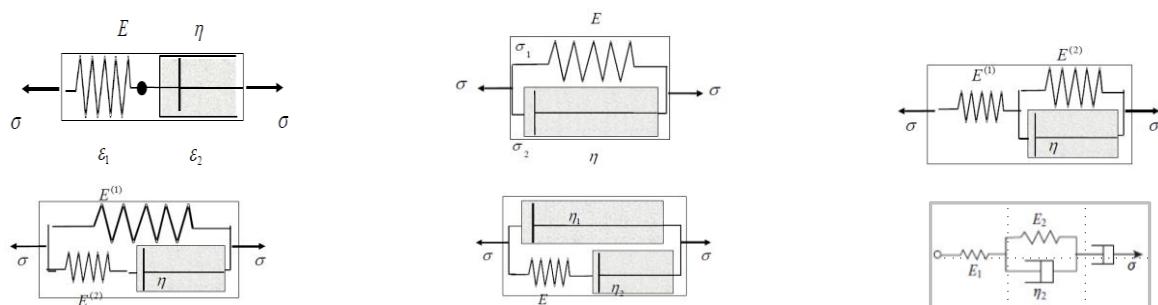


Fig. 2 Examples of "two", "three" and "four" parametric viscoelastic models

Therefore, these models are combined in an attempt to approximate reality. This broad topic has become part of the educational field referred to as "materials engineering". The basis of the field is physical experimentation, followed by analytical and numerical methods, which only together can satisfactorily identify the behaviour of the material in question under operating conditions (generally climatic conditions, operating temperature range, strain rates, long-term service, environmental aggressiveness, radiation, etc.).

For a more comprehensive description of the viscoelastic response of materials under service conditions, especially polymer composites, generalized viscoelastic models are used. Initial examples are given in Fig. 3.

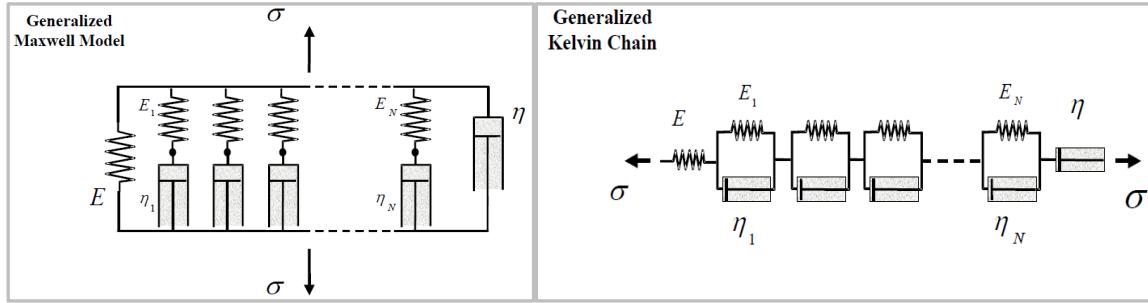


Fig. 3

The mathematical description of their behaviour is based on Boltzmann's superposition principle, which considers the loading effects on the viscoelastic medium to be independent and additive, and the response of the medium to these effects to be linear. It can be applied to both creep and relaxation processes. This results in so-called hereditary integrals, i.e.:

Hereditary Integral for Strain

$$\boldsymbol{\varepsilon}(t) = \int_0^t j(t-\tau) \frac{\partial \sigma(\tau)}{\partial \tau} d\tau \quad \text{where } j(t) \text{ is the creep function (compliance).}$$

Hereditary Integral for Stress

$$\boldsymbol{\sigma}(t) = \int_0^t g(t-\tau) \frac{\partial \varepsilon(\tau)}{\partial \tau} d\tau \quad \text{where } g(t) \text{ is the relaxation function (modulus).}$$

Often the above relations are given in forms that can be obtained by integrating them per partes. Valid

$$\boldsymbol{\varepsilon}(t) = j(0) \boldsymbol{\sigma}(t) + \int_0^t \boldsymbol{\sigma}(t-\tau) \frac{\partial j(\tau)}{\partial \tau} d\tau$$

$$\boldsymbol{\sigma}(t) = g(0) \boldsymbol{\varepsilon}(t) + \int_0^t \boldsymbol{\varepsilon}(t-\tau) \frac{\partial g(\tau)}{\partial \tau} d\tau$$

where $j(0)$ and $g(0)$ are the initial values of the creep compliance function and the relaxation modulus.

From the preceding, it is evident that the creep and relaxation behaviour of materials are related and, therefore, that there is a correlation between the material functions of creep compliance $j(t)$ and relaxation modulus $g(t)$. This can be interpreted in integral forms, which result from the use of the Laplace transform. Valid

$$j(0)g(t) + \int_0^t g(t-\tau) \frac{\partial j(\tau)}{\partial \tau} d\tau = 1 \quad \text{and/or} \quad g(0)j(t) + \int_0^t j(t-\tau) \frac{\partial g(\tau)}{\partial \tau} d\tau = 1$$

$$\int_0^t g(\tau) j(t-\tau) d\tau = t \quad \text{and/or} \quad \int_0^t j(\tau) g(t-\tau) d\tau = t$$

3. VISCOELASTIC MATERIAL MODELS IMPLEMENTED IN CAE/FEM SW PRODUCTS

The formulation for these models is based on the above and the parameters contained in them are usually attributed to the behaviour of materials in the solid state, the so-called "solids".

The basis is the *Prony series*, which is based on generalized models composed of many Maxwell groups

$$G(t) = G_\infty + \sum_{i=1}^N G_i \exp\left(-\frac{t}{\tau_i}\right),$$

where G_∞ is the long term "equilibrium" modulus that corresponds to "complete" relaxation (in infinitely long time), τ_i a G_i are the parameters of the Prony series and t is time.

If we introduce the instantaneous so-called "dynamic" modulus G_0 , which represents the stiffness of the material immediately after loading (at time $t = 0$)

$$G_0 = G(t = 0) = G_\infty + \sum_{i=1}^N G_i$$

we get the Prony series in the equivalent form

$$G(t) = G_0 - \sum_{i=1}^N G_i \left[1 - \exp\left(-\frac{t}{\tau_i}\right) \right]$$

Note that the parameters of the Prony series G_i and τ_i are determined on the basis of suitable experiments (tension, pressure, shear, volumetric or biaxial tests) and by processing the measured data through numerical regression procedures into the desired form (fitting curves, e.g. least squares method).

Predicting the viscoelastic response of polymer composite products requires the implementation of relaxation or creep experiments depending on temperature and time or frequency, see next.

In certain cases, the so-called Time-Temperature Superposition Principle can be used to shorten the experimental measurement time and determine the relaxation modulus or creep compliance as a function of time in the form of a so-called master curve, represented mathematically by a Prony series, together with the Williams-Landel-Ferry empirical relationship. For polymeric materials, it is necessary to distinguish between materials exhibiting thermo-rheological simplicity or complexity under operational loading. In the former case, the time-temperature superposition principle (master curve) can be used, but not in the latter. The reason for this is the differences in their molecular structure, which consists in the environmental mobility of the "structural elements" of the respective material, especially as a function of temperature [3].

The above approximate relationships of experimental creep and relaxation measurements in the time domain are usually implemented in sw tools (ANSYS, Abaqus, MSC.MARC, etc.) in one of the following normalized (dimensionless) forms:

$$\alpha_\infty^G = \frac{G_\infty}{G_0} \quad \text{and/or} \quad \alpha_i^G = \frac{G_i}{G_0}$$

$$G(t) = G_0 \left[\alpha_\infty^G + \sum_{i=1}^N \alpha_i^G \exp\left(-\frac{t}{\tau_i}\right) \right] \quad \text{and/or} \quad G(t) = G_0 \left[1 - \sum_{i=1}^N \alpha_i^G \exp\left(1 - \frac{t}{\tau_i}\right) \right],$$

where τ_i is a user-selected so-called relaxation time spectrum in graphical interpretations usually displayed in logarithmic time decades.

4. MATHEMATICAL REPRESENTATIONS OF THE VISCOELASTIC MODEL IN FREQUENCY FORM

We also mention that the viscoelastic behaviour of the material can be identified by dynamic vibration experiments, the so-called DMTA (Dynamic Mechanical Temperature Analysis) method. In these experiments, the material is subjected to small strain vibrations at different modes of excitation. The measurements result in real $G'(\omega)$ and imaginary $G''(\omega)$ components of the complex moduli $G(\omega)$, which are functions of the frequency ω . From the theoretical background, it follows that the following relationship holds between the Prony series parameters introduced above in the time domain and the frequency representation of the linear viscoelastic response of materials

$$G'(\omega) = G_0 \left[1 - \sum_{i=1}^N \left(\alpha_i^G - \frac{\alpha_i^G (\tau_i^G \omega)^2}{1 + (\tau_i^G \omega)^2} \right) \right] \quad G''(\omega) = G_0 \sum_{i=1}^N \left(\frac{\alpha_i^G \tau_i^G \omega}{1 + (\tau_i^G \omega)^2} \right)$$

For the complex module $G(\omega)$ and the loss factor „ $\text{tg}\delta(\omega)$ “ valid

$$G(\omega) = \sqrt{G'^2(\omega) + G''^2(\omega)} \quad \text{tg}\delta(\omega) = \frac{G''(\omega)}{G'(\omega)}$$

The preceding shows that if we determine the value of G_0 and the normalized parameters α_i from experimental data by regression methods through the above mathematical relations (in time and frequency, respectively), we can convert the material dependencies to each other. However, this is subject to the validity of superposition principles (Boltzmann + TTSP) and for thermo-rheologically simple materials.

5. PHYSICAL EXPERIMENT – THE CONDITION AND BASIS FOR UNDERSTANDING THE VISCOELASTIC RESPONSE OF MATERIALS

To identify the time response of solid materials to external loading effects, it is necessary to be aware of a number of implications arising from the theory of solid continuum mechanics. The basic point is that for a homogeneous isotropic material, the strain and stress of a viscoelastic body, as well as that of an elastic body, can be divided into two components that characterize the change in shape of the elementary volume of the material (stress and strain deviators) and the change of the elementary volume of the material and where relations hold between the quantities K , G , E and μ

$$K(t) = \frac{2G(t)(1+\mu(t))}{3(1-\mu(t))} = \frac{G(t)E(t)}{3(3G(t)-E(t))} = \frac{E(t)}{3(1-2\mu(t))}$$

To identify the viscoelastic properties of the materials, experimental techniques based on the following experimentally verified assumptions are used:

1. the material behaves identically in shear and volumetric deformation

$$\mu(t) = \mu = \text{konst.} < 0.5 \quad \frac{E(t)}{G(t)} = \frac{E}{G},$$

2. the material is elastically compressible

$$K(t) = K = \text{konst.} \quad \frac{E(t)}{E} = \frac{(1-2\mu(t))}{(1-2\mu)},$$

3. the material is incompressible $K(t) \rightarrow \infty$, $E(t) = 3G(t)$, $\mu(t) = \text{konst.} = 0.5$.

6. NUMERICAL SIMULATION OF DMTA TEST IN 3-POINT BEAM BENDING MODE

For the purpose of CAE simulations of hybrid structures, LENAM has developed a methodology to identify the viscoelastic properties of polymer composites using the DMTA experimental method in combination with numerical simulations [4].

Fig. 4 shows a comparison of DMTA experimental results and FE simulations in the temperature range 40–120°C.

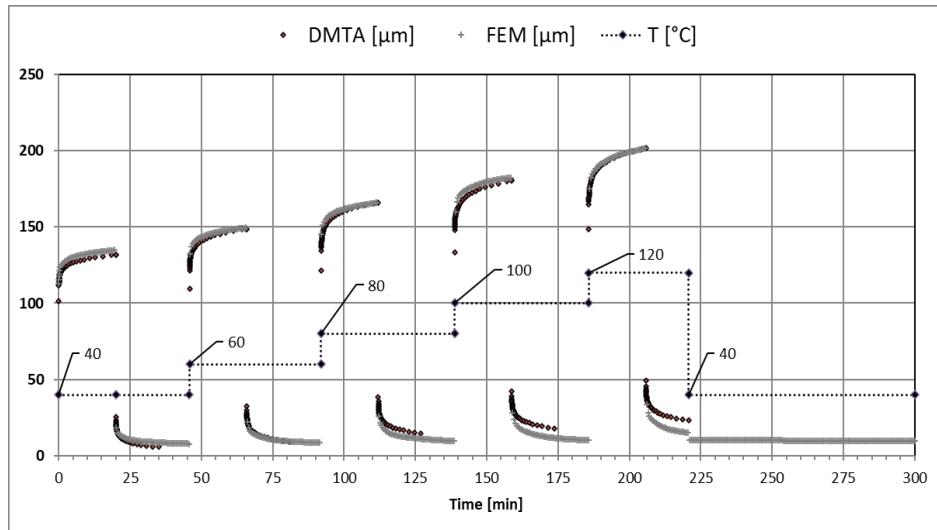


Fig. 4 Comparison of the results of DMTA experiments and FEM simulations

CONCLUSION

The developed methodology allowed to verify the correctness of the determination of the Prony series parameters entered into the material models of the respective parts/components/objects by calculations of the analyzed hybrid structural assemblies.

With the support of CAE modelling and FE simulations of physical experiments such as DMTA, the realistic behaviour of polymer composite materials in their viscoelastic response modes to service loads can be competently estimated and their material models can be developed.

By deploying modern experimental techniques in conjunction with computational matrices, both analytical and numerical, in the pre-development conceptual stages of product development, gaps between the desired goal and its outcome can be bridged and appropriate measures can be discussed and proposed, thereby minimizing product development errors early on.

ACKNOWLEDGMENT

This work was carried out within the TAČR project TM03000010 and partially supported by the Student Grant Programme of the Technical University of Liberec through project no. SGS-2023-3378.

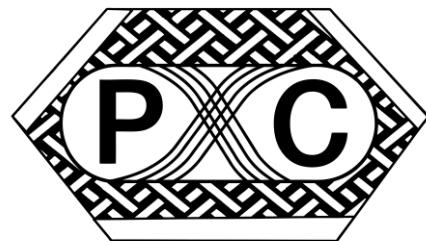
LITERATURE

1. Sobotka Z.: Rheology of materials and structures. Academia Praha, 1981.
2. Maršík F.: Thermodynamics of a continuum, Academia Praha, 1999. ISBN 80-200-0758-X.
3. Deshpande A.P.: <https://www.youtube.com/watch?v=YG53m6roxR8>.
4. LENAM, s.r.o.: Archive of technical reports, 1993–2023.

POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



BENEFITS OF COMPOSITE MATERIALS IN MACHINE TOOL STRUCTURAL PARTS

PŘÍNOSY VLÁKNOVÝCH KOMPOZITŮ V KONSTRUKCI NOSNÝCH DÍLŮ VÝROBNÍCH STROJŮ

MILAN RŮŽIČKA¹, VIKTOR KULÍŠEK², PETR KOLÁŘ³,
JAN SMOLÍK⁴, ONDŘEJ UHER⁵

This paper describes application of carbon fiber composites in machine tools, where the main advantage is not the strength, but high stiffness and lower weight compared to steel or cast-iron structures. Three practical examples are described. The first one on the rotary parts where the steel drive shafts have been replaced by a long high modulus carbon fiber composite shaft. The second on a full composite Z-axis glide structure and the third on a hybrid ductile-cast iron Z-axis glide, which is reinforced by inner composite tube.

KEYWORDS

Machine tools; fibre composites; light-weight design; dynamic stiffness

ÚVOD

Vláknové kompozity jsou již široce používanou skupinou materiálů, která nachází uplatnění v různých průmyslových aplikacích. Jejich vyšší uplatnění v oblasti výrobních strojů je dosud poměrně vzácné, představuje velké technické výzvy i slibné potenciální přínosy. Na komponenty pohybových os výrobních strojů jsou totiž kladený enormně vysoké nároky z hlediska jejich statické i dynamické tuhosti. Tradiční materiálová řešení z litin nebo ocelí vedou k silnostennému charakteru komponent o značné hmotnosti, což omezuje dynamické

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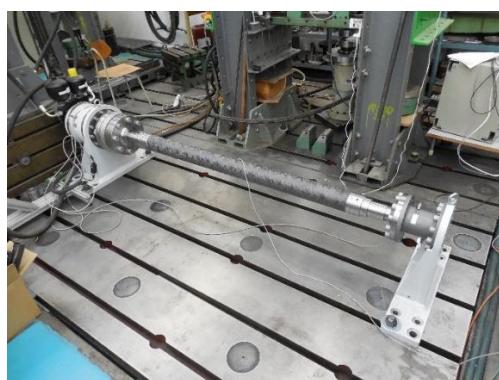
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chování pohybových os (dosažitelné rychlosti a zrychlení lineárních posuvů, kritické otáčky rotačních komponent). Možné odlehčení pomocí vláknových kompozitů by vedlo ke zvýšení produktivity výroby výrobního stroje, v případě zvýšení dynamické tuhosti i ke zvýšení přesnosti a jakosti při obrábění výrobků. Požadavek vysoké tuhosti však představuje odklon od tradičních tenkostěnných kompozitních konstrukcí k silnostěnným strukturám, ve kterých jsou namísto vysoko-pevnostních vláken (s modulem pružnosti $E_f \sim 230$ GPa) aplikována (ultra)-vysoko-modulová vlákna ($E_f \sim 640\text{--}780\text{--}960$ GPa). Z důvodu ortotropního chování kompozitu však nelze jednoznačně konstatovat, že aplikace uhlíkových kompozitů do komponent výrobních strojů bude vždy přínosná. Zejména víceosové namáhání – kombinace ohybu, krutu a příčného smyku a technologické i konstrukční požadavky na výslednou skladbu kompozitu a připojovací rozhraní mohou devalvovat teoretické úvahy o jeho přínosu. Mezi další nevýhody patří vysoká cena kompozitního materiálu, zejména vysokomodulových vláken a návazných technologií připojování. Přehled kompozitních aplikací v různých komponentách výrobních strojů je uveden například v [Mohring].

KOMPOZITNÍ MATERIÁLY V ROTAČNÍCH KOMPONENTÁCH

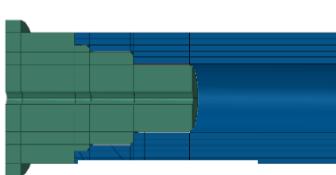
Velký potenciál mají vláknové kompozity v konstrukci komponent s rotační osou, kdy její výrazné odlehčení při zachování nebo zvýšení její statické a dynamické tuhosti přináší snížení spotřeby energie pro pohony a vyšší dynamické parametry sestavy. To lze využít zejména u nástrojů pro obrábění, hřidel vysokootáckových vřeten, náhonových hřidel, případně dalších aplikací. Využití ultra-vysoko-modulových uhlíkových vláken umožnuje dosáhnout výrazně vyšších vlastních frekvencí a kritických otáček než u tradičních ocelových hřidel. Díky tomu lze např. nahradit sestavu kratších ocelových hřidel a spojek jednou dlouhou kompozitní hřidelí, která kromě zjednodušené montáže přináší i další úspory z hlediska redukce počtu ložisek, spojek a výrobních a montážních nákladů. Příkladem je náhonová hřídel (Obr. 1) pro obráběcí stroj s průměrem 65 mm, délou 2000 mm a schopností dlouhodobé zátěže na $M_k=3000$ Nm.



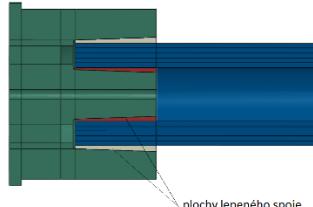
Obr. 1 Kompozitní náhonová hřídel během zkoušek pevnosti spojů

Kritickou oblastí výše zmíněných konstrukcí bývá zejména tvorba spojovacích rozhraní. U náhonové hřídele (Obr. 1) to představuje tvorbu spoje mezi kompozitní částí hřídele a ocelovými koncovkami, obsahujícími připojovací prvky pro spojení do sestavy (plochy pro ložiska, drážky pro pera apod.). Jednou z možností je použití lepených spojů mezi kompozitní a ocelovou částí. Návrh lepeného spoje obvykle limituje využitelnou pevnost a životnost sestavy hřídele. Na Obr. 2 až Obr. 4 jsou zobrazeny 3 modelové verze lepeného spoje, které jsou tvořeny lepenou válcovou plochou, lepeným kuželovým spojem, nebo kombinovaným lepeným a tvarovým spojem mezi kompozitní hřidelí a ocelovou koncovkou. Experimentální porovnání pevnosti pro zkušební vzorky průměru 42 mm je zobrazeno na Obr. 5, kdy uspořádání zkoušky odpovídá konfigurace z Obr. . Různá konstrukce spoje vede k drobným

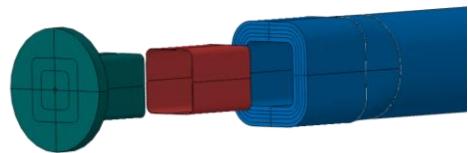
rozdílům v torzní tuhosti celkové sestavy hřídele. Zejména ale ovlivňuje pevnost hřídele i chování hřídele po iniciaci poruchy spoje, jak demonstrují křivky deformačního diagramu.



Obr. 2 Válcový lepený spoj



Obr. 3 Oboustranný kuželový lepený spoj



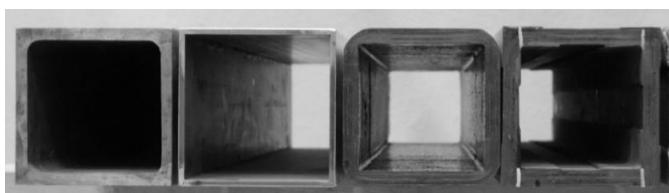
Obr. 4 Lepený a tvarový spoj



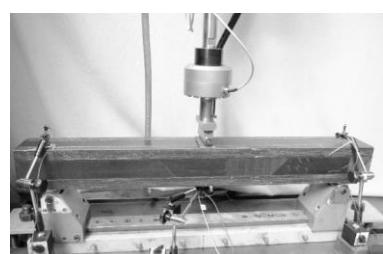
Obr. 5 Porovnání pevnosti a tuhosti hřídelů modelových spojů při torzním namáhání

KOMPOZITNÍ MATERIÁLY VE SMYKADLECH A DALŠÍCH NOSNÍCÍCH

Dalším potenciálním uplatněním kompozitů ve výrobních strojích je odlehčení, nebo využitzení komponent, které zajišťují lineární pohyby dílů do směru pohybových os. Jedná se o smykadla, příčníky, ramena robotických manipulátorů a jiné strukturální nosníky. Společným požadavkem těchto aplikací je důraz na vysokou statickou tuhost – minimalizaci deformací pro kombinované namáhání ohýb – krut – příčný smyk. Experimentální porovnání klíčových vlastností je pro vzorky modelových nosníků o rozměrech $120 \times 120 \times 1200$ uvedeno na Obr. 6 a v Tab. 1. Vzorek z šedé litiny, ocelového svařence je zde porovnán s dvěma typy kompozitních vzorků, které byly vyrobeny z ultra-vysoko-modulových vláken modulu pružnosti $E_f = 780$ GPa. Rozdíl mezi vzorky Kompozit1 a Kompozit2 spočíval v tom, že vzorek Kompozit1 byl vyroben jako integrální vzorek technologií navijení a kládení vláken, zatímco Kompozit2 byl vyroben lepením z předvyrobených desek a jako takový v sobě obsahoval další poddajnost rohových spojů.



Obr. 6 Modelové vzorky nosných struktur (vlevo) a zkoušky statické tuhosti (vpravo)



Tab. 1 Porovnání geometrie, modálních parametrů a tuhosti nosníků 120×120×1200

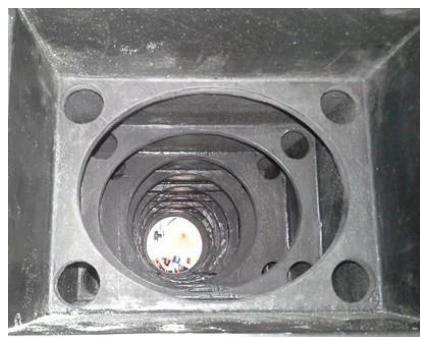
	Litina	Ocel	Kompozit1	Kompozit2
Hmotnost [kg]	43,4	23,2	11,2	11,5
Tloušťka stěny [mm]	10	5	13	14
1. frekvence [Hz]	493	390	915	762
Tlumení 1. ohyb. tvaru ζ_1 [%]	2,1	0,3	1,6	1,3
Průměrné tlumení ζ_{pr} [%]	0,45	0,38	1,37	0,7
Ohybová tuhost EJ [N.mm ²]	1,52e12	1,57e12	2,22e12	1,61e12
Statická tuhost F/y [N/mm]	55 503	34 068	46 903	34 123

Porovnání hodnot v Tab. 1demonstruje, že při stejné ohybové tuhosti lze dosáhnout výrazné hmotností redukce, zvýšení vlastních frekvencí i tlumení u kompozitních vzorků vůči tradičním konstrukčním kovům. Nutností však bylo použít vyšší tloušťky stěny u kompozitu oproti ocelovému i litinovému vzorku stejného vnějšího průřezu. Je to způsobeno vlastnostmi anizotropního materiálu. To pro kombinované namáhání (zde ohyb + příčný smyk, viz Obr. 6) vede k nutnosti použít vrstvy s orientací 0° i vrstvy s orientací ±45°, čímž dochází k snižování efektivního modulu pružnosti v podélné ose, zvyšuje se však modul smykový. Porovnání vzorků Kompozit 1 a Kompozit 2 ukazuje, že celonavíjený nosník má výrazně vyšší tuhost než nosník spojovaný.

Výhody a nevýhody celokompozitních řešení ukazuje studie kompozitních smykalidel obráběcího stroje. Zkušební kompozitní těleso rozměrů 275×230×1100 je zobrazeno na Obr. 7 během zkoušky statické tuhosti. Těleso bylo vytvořeno z lepených kompozitních desek s žebrovou vnitřní výztuhou. Ke kompozitu bylo uloženo referenční ocelové těleso pro měření průhybů nosníku při jeho zatížení ve vertikálním směru (viz Obr. 7). Na Obr. 8 je ukázáno žebrování vnitřního prostoru smykadla, které bylo u kompozitu tvořeno vlepenými kompozitními deskami, u oceli svařenými výztužemi. Porovnání geometrie a statické tuhosti je uvedeno v Tab. 2.



Obr. 7 Smykadlo při zkoušce tuhosti



Obr. 9 Žebrování kompozitního smykadla

Tab. 2 Porovnání hmotnosti, geometrie a statické tuhosti smykařských obráběcích strojů

Těleso	Hmotnost [kg]	Tloušťka stěny/žebra [mm]	Khorizon [N/um]	Kvertikal [N/um]
Referenční ocelové sm.	112	10/8	55	64
Deskové kompozitní sm.	45	19/11	26	58

I přes dvojnásobné zvýšení tloušťky stěny bylo analogicky k základním vzorkům dosaženo u kompozitního řešení výrazného snížení hmotnosti a podobné statické tuhosti při zatížení ve svislém směru, kde působí kombinace ohyb a smyk. Problém se ukazuje při zatížení ve vodorovném na podélnou osu příčném směru, kdy se navíc vyvouzí krut. Kombinované namáhání a poddajnost struktury (lepené desky, lepené žebrování) pro tento případ vedlo k dosažení přibližně k poloviční tuhosti s referenční ocelovou variantou. Jistěže existují způsoby, jak finální výrobek řešit lépe a zvýšit jeho statickou tuhost, například použitím celonavíjeného profilu místo lepených desek. Nicméně na výsledcích zkušebních smykařských lze demonstrovat, že celokompozitní řešení je vhodné do komponent pohybových os s požadavky na velké zrychlování a co nejvyšší redukci pohybových hmot. Vhodnou aplikací mohou být například příčníky strojů pro rychlé řezání laserem a další.

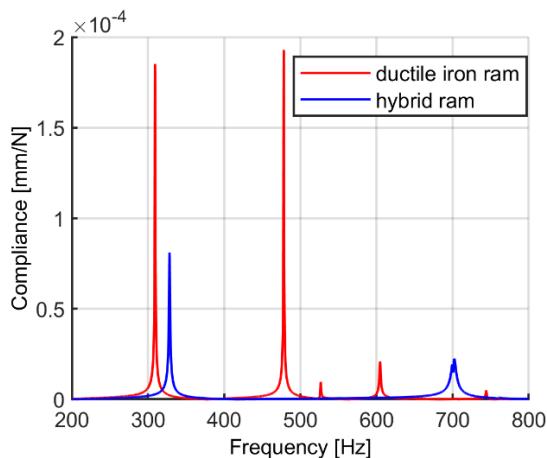
Jako vhodná se také ukazuje kombinace kompozitů z ultra-vysoko-modulových vláken s ocelí / litinou pro tvorbu tzv. hybridních struktur. Kovové části lze pak využít pro spojovací rozhraní a také jako nosnou část, která pomůže se statickou tuhostí při kombinovaném namáhání. Kompozitní část lze navíc optimalizovat pro dosažení vhodné tuhosti a hmotnosti celku. Příkladem je hybridní smykařské obráběcího stroje s vnějším pláštěm z tvárné litiny a vnitřní kompozitní výztuží (Obr. 9). Na tělesu rozměru 420×400×2500 znamenalo vlepení kompozitní výztuže hmotnostní přírůstek menší než 10 % původní hmotnosti. Vyztužení lehkou a tuhou kompozitní výztuží pak vedlo k výraznému zvýšení dynamické tuhosti samotného tělesa (viz Obr. 11), které bylo naměřeno při modální analýze smykařských (Obr. 10). Je zde patrný výrazný přínos hybridní struktury z hlediska zvýšení dynamické tuhosti i vlastních frekvencí. Přínos je nutný ale vždy posuzovat z hlediska sestavy celého stroje, kdy například zvýšení tlumení samotného tělesa se v sestavě výrobního stroje nemusí projevit, viz [Kulisek]. Na Obr. 12 je výpočtově demonstrován efekt zvýšení tuhosti smykařského na dynamické tuhosti a frekvencích sestavy obráběcích strojů.



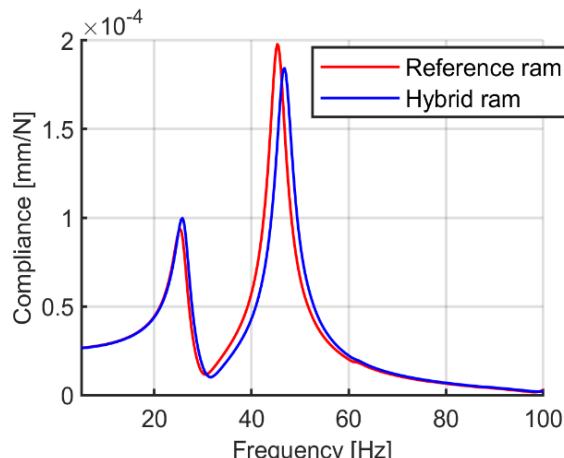
Obr. 9 Hybridní smykařské obráběcího stroje s vnějším pláštěm z tvárné litiny a kompozitní výztuží.



Obr. 10 Hybridní smykařské obráběcího stroje při modální analýze



Obr. 11 Dynamická poddajnost hybridního/referenčního smykadla (zkouška)



Obr. 12 Dynamická poddajnost sestavy obráběcího stroje s hybridním/referenčním smykadlem (MKP).

ZÁVĚRY

Vláknové kompozity mají velký potenciál pro zvýšení užitných vlastností výrobních strojů. Nabízejí výrazné odlehčení komponent pohybových os při zachování vysoké statické tuhosti. Jedná se ale o specifické uplatnění kompozitů, ve kterém se zejména jedná o silnostěnné díly s kompozity z ultra-vysoko-modulových vláken. Aplikace kompozitů však vyžaduje řešení řady dalších otázek, např. spojovací rozhraní, optimalizovanou tuhost pro všechny módy namáhání včetně posouzení finanční efektivity aplikace. Příspěvek ukazuje využitelný potenciál kompozitů jak v rotačních tělesech, tak v nosných dílcích pohybových os výrobních strojů.

PODĚKOVÁNÍ

V této práci byly shrnuty výsledky projektů TE01020075 Centrum kompetence – Strojírenská výrobní technika a TA02010543, které byly podporovány Technologickou agenturou České republiky.

LITERATURA

1. Mohring, H. C. Composites in Production Machines. Procedia CIRP, 2017, Vol. 66, pp 2-9. ISSN 2212-8271
2. Kono, D., et al. A Machine Tool Motorized Spindle with Hybrid Structure of Steel and Carbon Fiber Composite. In: CIRP Annals - Manufacturing Technology. Vol. 68, 2019, pp. 389-392. ISSN: 1726-0604.
3. Kulisek, V., et al. On Passive Damping in Machine Tool Hybrid Structural Parts. International Journal of Advanced Manufacturing Technology, 2021, Vol. 114, pp 1925-1952. ISSN 1433-3015

POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



FLAX MULTIAXIAL FABRICS – WAY FOR REDUCING OF THE PRODUCT CARBON FOOTPRINT

LNĚNÉ MULTIAXIÁLNÍ TKANINY – CESTA KE SNÍŽENÍ UHLÍKOVÉ STOPY VÝROBKU

MAREK ŠČERBA¹

KEYWORDS

Multiaxial fabrics, flax, natural fiber composite

INTRODUCTION

One idea to reduce the product carbon footprint is to reduce over-engineering - by using natural fiber fabrics. In cooperation with Terre de Lin, one of the world's biggest suppliers of linen, we developed this new product series for our customers: the new flax fiber NCFs. Made by SAERTEX in France. Maybe they will not replace glass fiber fabrics. But they are one way to reduce the CO₂-footprint. For example, of interior parts in boats, for skis or boards and those composite parts in transportation that are o.k. with the mechanical properties of flax fibers and good damping characteristics are a benefit.

Our main goals are following:

Improve product offer at a competitive price in order to complement STX non-crimp fabric product range. This will not replace glass fiber applications but applications where glass is overdesigned. In combination with glass/carbon this can reach semi-structural to structural applications.

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ÚVOD

Jedním z nápadů, jak snížit uhlíkovou stopu produktu, je omezit mechanickou předimenzovanost klasických výztuží – pomocí tkanin z přírodních vláken. Ve spolupráci s Terre de Lin, jedním z největších světových dodavatelů lněných vláken, jsme pro naše zákazníky vyvinuli tuto novou produktovou řadu: nové multiaxiální tkaniny na bázi lněného vlákna. Vyrobeno ve společnosti SAERTEX France. Možná nenahradí tkaniny ze skelných vláken. Ale jsou jedním ze způsobů, jak snížit uhlíkovou stopu. Například interiérové díly v lodích, pro lyže nebo snowboardová či kite prkna a ty kompozitní díly v dopravě, které uspokojí uživatele s mechanickými vlastnostmi lněných vláken a jejich dobrými tlumícími vlastnostmi, které jsou jejich velkým přínosem.

Naše hlavní cíle jsou následující:

Vylepšit nabídku produktů za konkurenceschopnou cenu, abychom doplnili sortiment našich multiaxiálních tkanin. To nenahradí aplikace ze sklovýztuží, ale aplikace, kde je sklo předimenzované. V kombinaci se sklem či uhlíkem můžeme dosáhnout až polostrukturálních až strukturálních aplikací.

POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



INFLUENCE OF THE PREPARATION TECHNOLOGY AND PLASMA TREATMENT ON THE STRENGTH OF POLYETHYLENE WITH GLASS FIBERS

OVLIVNĚNÍ PEVNOSTI POLYETHYLENU SE SKLENĚNÝMI VLÁKNY TECHNOLOGIÍ PŘÍPRAVY A PLAZMOVOU ÚPRAVOU

JAN SEZEMSKÝ¹, ZDEŇKA JENÍKOVÁ², TAŤANA VACKOVÁ³

The paper deals with the preparation and testing of a composite material based on a non-polar thermoplastic matrix reinforced with short glass fibers. This material was prepared by various manufacturing technologies, namely rotomolding, compression molding and FDM 3D printing. Pre-treatment of the polyethylene matrix with plasma was included in the preparation process to improve adhesion between phases and as a result the properties of the final material.

KEYWORDS

Polyethylene, glass fibers, plasma, rotomolding, compression molding, 3D printing

ÚVOD

Kompozitní materiály na bázi polymerů jsou v současnosti nedílnou součástí řady technických aplikací, a to díky snadné zpracovatelnosti mnoha výrobními technologiemi nebo nízké měrné hmotnosti v kombinaci s dostatečnými mechanickými vlastnostmi. Těch je ale dosaženo za předpokladu dobré adheze matrice k vláknům. Lepšímu spojení může dopomoci v průběhu přípravy vnější tlak, jako je tomu např. v případě vstřikování nebo lisování. V technologii, kde není vyvýjen tak vysoký tlak (3D tisk), nebo celý proces probíhá za atmosférického tlaku (rotomolding), je v případě nedostatečné adheze nutno zařadit předúpravu vláken nebo matrice. U nepolárních polymerů je možností např. chemická funkcionalizace, roubování nebo plazmová modifikace povrchu. Ověření vlivu plazmové úpravy polyethylenové matrice na pevnost různě připraveného kompozitu je předmětem tohoto příspěvku.

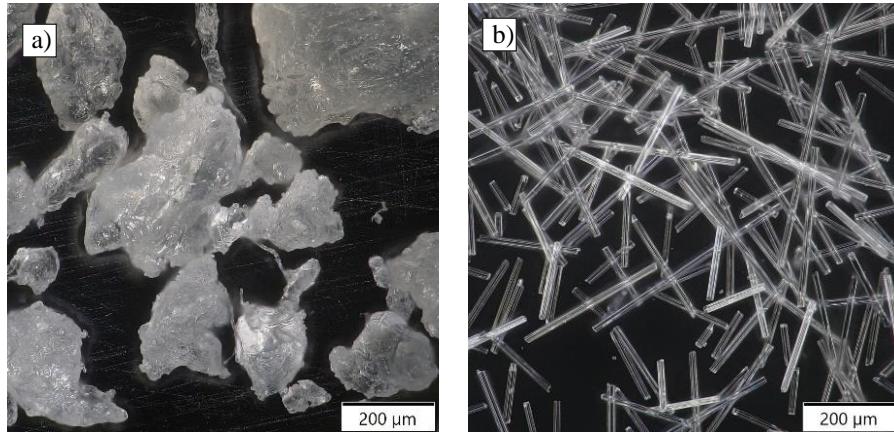
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POUŽITÉ MATERIÁLY

Jako matriční materiál byl použit polyethylen (PE) Dowlex 2629.10UE, který byl ve výchozím stavu dodán v podobě prášku (Obr. 1a). Použitý polyethylen je nízkohustotním typem a má tyto základní vlastnosti: hustota $0,935 \text{ g}\cdot\text{cm}^{-3}$, teplota tání 124°C , pevnost v tahu $17,5 \text{ MPa}$, MFI $4 \text{ g}/10 \text{ min}$ [1]. Matrice byla vyztužena krátkými skleněnými vlákny (GF) Lanxess MF 7982 (Obr. 1b) o průměru $14 \mu\text{m}$, s průměrnou délkou $150 \mu\text{m}$ a objemovou hmotností $0,750 \text{ g}\cdot\text{cm}^{-3}$ [2].



Obr. 1 Výchozí suroviny a) polyethylenový prášek; b) skleněná vlákna

POPIS PLAZMOVÉ ÚPRAVY

Modifikace polyethylenového prášku byla provedena na nízkotlakém plazmovém laboratorním zařízení LA 400. Plazma bylo generováno pomocí mikrovlnného zdroje v kyslíku při konstantním průtoku 300 sccm . Vakuum o tlaku 100 Pa bylo vytvořeno a udržováno dvoustupňovou rotační vývěrou Pfeiffer Vacuum Duo 35. Pro dosažení rovnoměrné úpravy PE částic byl prášek v aparatuře umístěn do míchačky s konstantními otáčkami 40 ot./min . V jednom plazmovém modifikačním cyklu bylo dohromady naváženo a upravováno 250 gramů surového materiálu. Celková doba plazmové úpravy byla 10 minut .

TECHNOLOGIE PŘÍPRAVY

Proces přípravy kompozitních vzorků ve všech případech začal navážením a smícháním 10 hm. \% skleněných vláken s polyethylenovým práškem. Výchozím polotovarem pro rotomolding a lisování byla právě tato směs, pro 3D tisk byl připravován filament.

Rotomolding (RM)

Příprava vzorků technologií rotomolding probíhala na laboratorním zařízení zkonstruovaném firmou Železný, s.r.o. Jedná se o stroj typu "rock and roll", který umožňuje kívání pece jako celku v jednom směru a otáčení formy umístěné ve vnitřním prostoru pece. Forma se pak otáčí ve směru kolmém na směr kívání. Nastavená maximální teplota v peci byla 250°C , PIAT, která určuje maximální dosaženou teplotu uvnitř formy byla 210°C . Chlazení formy probíhalo proudem vzduchu vháněného dovnitř pece.

Lisování

Lisování vzorků proběhlo na hydraulickém lisu HOLZMANN WP45H. Tento lis byl upraven pro zpracování a přípravu vzorků z termoplastů. Čelisti stroje byly nahrazeny dvěma elektricky vyhřívanými deskami, které bylo možno chladit vodou. Mezi desky se vložila měděná forma s připravenou kompozitní směsí, která se následně zahřála na 160°C . Vzorky byly poté lisovány za tlaku $1,25 \text{ MPa}$ po dobu 20 minut .

3D tisk (FDM)

Proces přípravy kompozitního filamentu pro tisk vzorků byl následující. Směs PE prášku a vláken se nejprve nechala spéct v peci při atmosférickém tlaku a teplotě 180 °C po dobu 30 minut. Slinuté desky byly poté rozdrceny v laboratorní dřtičce. Kompozitní filament byl připraven z rozdrcených pelet pomocí extrudéru Noztek Pro. Extrudování probíhalo při teplotě 180 °C. Kvůli abrazivním účinkům skleněných vláken byla použita ocelová tryska o průměru 1,75 mm. Nakonec byly kompozitní vzorky vytiskány na tiskárně Prusa i3 MK3S+ s ocelovým tiskovým plátem a s hladkým oboustranným PEI povrchem. Soubory GCode byly generovány v programu PrusaSlicer 2.3.3., kde byly nastaveny tyto tiskové parametry: teplota trysky 240 °C, teplota podložky 90 °C, průměr trysky 0,8 mm, rychlosť tisku 60 mm·s⁻¹, výška vrstvy 0,3 mm. Zkušební tělesa byla tištěna se 100% přímočarou výplní, v podélném směru.

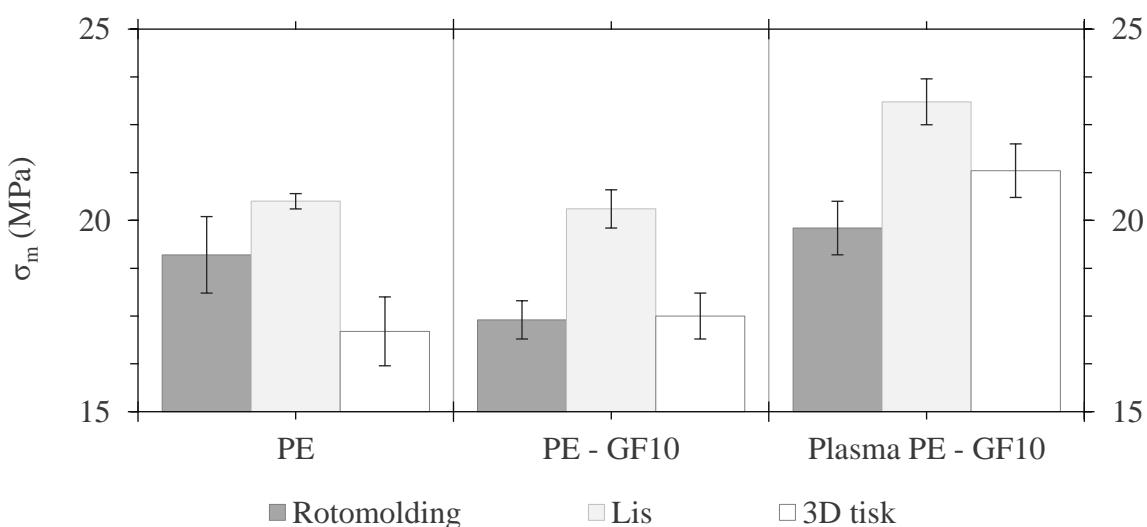
TESTOVACÍ METODY

Připravený kompozitní materiál byl charakterizován z hlediska mechanických vlastností a mikrostruktury. Pevnost v tahu byla měřena na zkušebních tělesech s rozměry dle normy ASTM D638 (Typ IV). Ze vzorků připravovaných rotomoldingem a lisováním byla zkušební tělesa obrobena na CNC frézce, pro tisk byla přímo definována vygenerovaným GCodem. Samotné testování probíhalo na univerzálním stroji MTS Exceed E42.

Rozložení vláken v matrici bylo vyhodnoceno pomocí světelného mikroskopu Nikon Eclipse ME600 na preparátech z tenkých fólií. Pro pozorování morfologie vnitřního povrchu byl použit skenovací elektronový mikroskop JEOL JSM-7600F. Studované vzorky byly získány mrazovým lámáním v tekutém dusíku. Lomové plochy byly pokryty vrstvou Cu pomocí vakuového naprašovacího zařízení HVM Flexicoat 3.

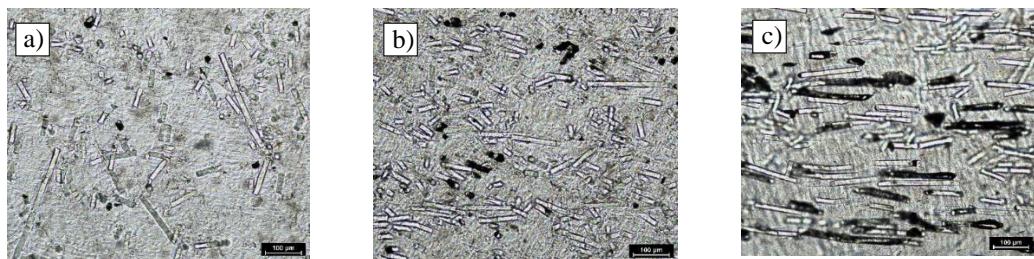
VÝSLEDKY

Ze srovnání na obrázku 2 je patrné, že nejvyšší hodnoty pevnosti nevyztuženého PE dosahují lisované vzorky. Během lisování dojde vlivem vnějšího tlaku k dokonalejšímu slinutí částic prášku bez defektů, jako jsou bubliny, jejichž přítomnost je typická u vzorků připravovaných technologií rotomolding [3], nebo prázdných míst – voidů, které se formují mezi jednotlivými vrstvami při FDM tisku [4]. Tyto nehomogenity pak ve výsledku negativně ovlivňují celkovou pevnost materiálu.



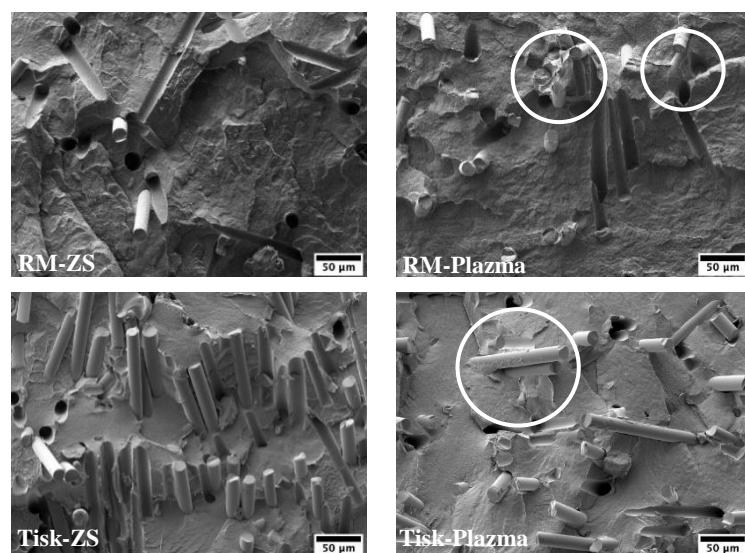
Obr. 2 Srovnání pevnosti kompozitu použitím odlišné technologie a plazmové úpravy

Dále z výsledku plyne, že přidání skleněných vláken do čisté polyethylenové matrice zcela nepřináší žádoucí využívající efekt. U vzorků připravovaných lisováním došlo k poklesu pevnosti z průměrných 19,1 MPa na 17,4 MPa. Hodnoty pevností lisovaných a tištěných těles zůstaly na hodnotách výchozího matričního materiálu. Na snímcích pořízených světelnou mikroskopí lze pozorovat, že u vzorků připravovaných v rotomoldingu (Obr. 3a) jsou skleněná vlákna v polyethylenu rozmístěna náhodně. To je dán charakterem technologie, kdy se v první fázi pracovního cyklu materiál pouze volně pohybuje uvnitř formy a vlákna zůstanou náhodně uložena v místech natavení polyethylenového prášku. Působením tlaku při lisování (Obr. 3b) a vlivem pohybu vláken při extruzi a skrz trysku během tisku (Obr. 3c) zaujmají GF určitou preferovanou orientaci. Proto by v některých místech mohlo do jisté míry dojít k přenosu napětí na vlákna, a tudíž u lisovaných a tištěných vzorků se nemusel projevit pokles pevnosti jako u rotomoldingu. V tomto případě by neusporelá vlákna mohla fungovat jako další defekty snižující nosný průřez.



Obr. 3 Rozložení vláken vzorků z a) rotomoldingu; b) lisu; c) 3D tisku

Nakonec pozitivně se ve všech případech na zlepšení pevnosti kompozitu podílela předúprava matrice plazmatem v porovnání s neupraveným stavem. Toto zjištění podporují snímky pořízené SEM. Odtud byla patrná místa lepší interakce mezi plazmovaným PE a GF, která se projevila pokrytím vláken matricí. Při plazmové úpravě se na upravovaný povrch navíc váží nové funkční skupiny a zvyšuje se jeho polarita [5]. Tím by se mohly ustanovit silnější intermolekulární interakce mezi polárními molekulami SiO₂ a novými skupinami na povrchu PE. Zlepšení adheze mezi těmito dvěma fázemi může zabránit vytahování skleněných vláken z matice a tím zlepšit přenos napětí z matice na vlákna. Adheze mezi GF a neupraveným PE je nedostatečná a GF jsou vylomená nebo dokonce chybí (viz Obr. 4).

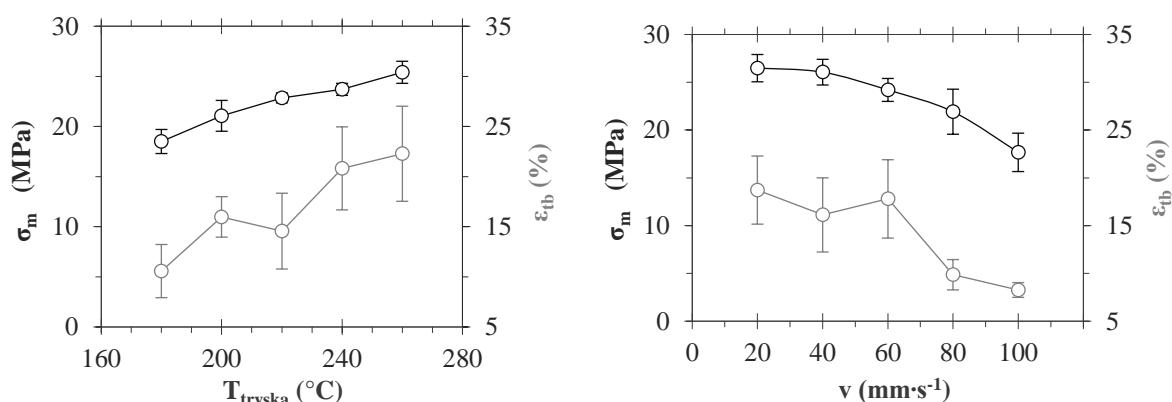


Obr. 4 Snímky vzorků z RM a tisku z PE v základním stavu ZS a po plazmové úpravě

OPTIMALIZACE 3D TISKU

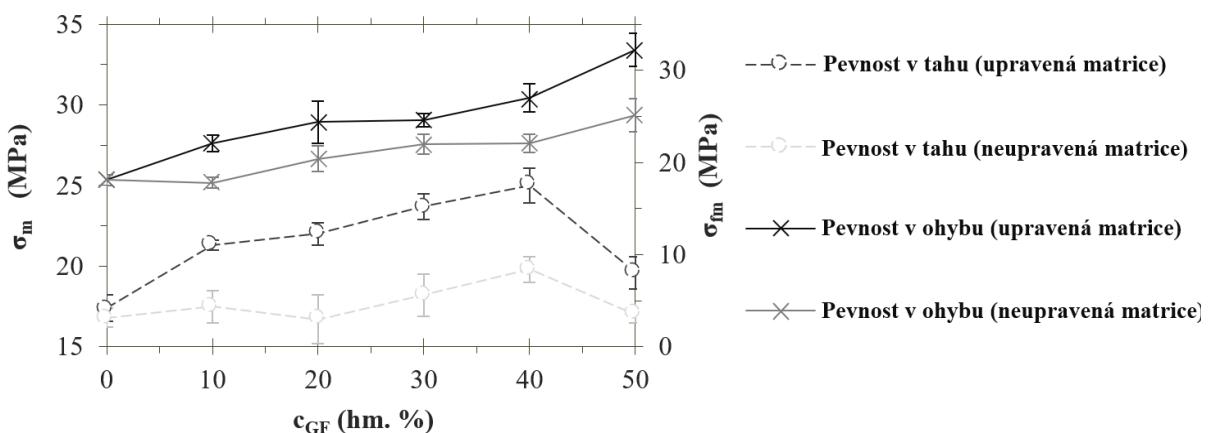
Při 3D tisku mají zvolené tiskové parametry zásadní vliv na mechanické vlastnosti. Z důvodu nedostatku publikací zabývající se kompozitním tiskem nepolárních polyolefinů byla navíc provedena optimalizace teploty a rychlosti tisku. Z výsledků vynesených na Obr. 5 je vidět postupný nárůst pevnosti z průměrných 18,5 MPa až na 25,4 MPa v rozmezí teplot od 180 do 260 °C. Při nižších teplotách tisku může docházet k nedostatečnému spojování jednotlivých vrstev, a jakožto nově vzniklé poruchy se budou podílet na snížení mechanických vlastností.

Rychlosť tisku měla opačný vliv na hodnoty pevnosti a tažnosti, jak je znázorněno na obrázku. Vyšší rychlosť způsobila trhání kladeného filamentu a nesprávnou tvorbu vrstev. Z těchto dat byly s ohledem na dobu tisknutí určeny optimální tiskové parametry, které pro teplotu tisku ležely v rozmezí 220 až 260 °C a pro tiskovou rychlosť mezi 40 až 60 mm·s⁻¹.



Obr. 5 Vliv tiskových parametrů na mechanické vlastnosti kompozitu

Vliv množství skleněných vláken a plazmové úpravy polyethylenové matrice je ilustrován na Obr. 6. Odtud lze usoudit, jak s narůstajícím obsahem skleněných vláken roste i pevnost v tahu, a to až do 40 hm. % GF. Při této koncentraci bylo dosaženo nejvyšší průměrné pevnosti v tahu o hodnotě 25 MPa. Tento nárůst byl 43% oproti nevyzkušenému materiálu. Při vyšších množstvích je matrice vláknové přesycena a kompozit vykazoval pokles pevnosti. Pevnost v ohybu tento pokles nevykazoval, což může být vysvětleno tím, že se jedná o kombinované namáhání, kdy kromě tahu figuruje i tlak. Vyšších pevností bylo dosaženo zařazením plazmové modifikace matrice, kdy jsou patrné vyšší hodnoty plazmovaných vzorků. Pevnosti vzorků s neupraveným PE kolísaly kolem hodnot neplněného základního materiálu.



Obr. 6 Vliv množství vláken a plazmové úpravy na pevnosti tištěných vzorků

ZÁVĚR

Bylo ověřeno, že výsledné hodnoty pevnosti polyethylenu využitěho skleněnými vlákny závisí na použití výrobní technologie. Nicméně využitím matrice v základním stavu nedocházelo k požadovanému využívání efektu, kdy pevnosti kompozitních vzorků byly horší, případně prakticky odpovídaly hodnotám čistého matričního materiálu. Zařazením plazmové modifikace polyethylenu došlo k částečnému zlepšení adheze mezi vlákny, což se v konečném důsledku projevilo i do celkové pevnosti kompozitu.

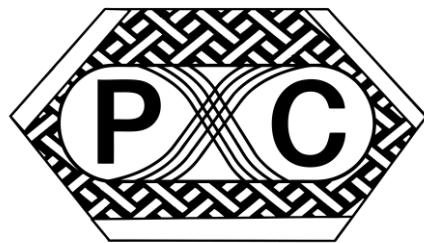
LITERATURA

1. THE DOW CHEMICAL COMPANY. Dowlex 2629.10UE: Materiálový list, 2013
2. LANXESS. Milled Fiber MF7982: Materiálový list, 2020
3. CRAWFORD, R. J. a James L. THRONE. Rotational molding technology. Norwich, N.Y.: Plastics Design Library/William Andrew Pub., c2002. ISBN 1-884207-85-5.
4. REDWOOD, Ben, Filemon SCHÖFFER a Brian GARRET. *The 3D printing handbook: technologies, design and applications*. Amsterdam: 3D Hubs, 2018. ISBN 9082748509.
5. ŠOURKOVÁ, Hana a Petr ŠPATENKA. Plasma Activation of Polyethylene Powder. *Polymers*. 2020, **12**(9). ISSN 2073-4360.

POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



RETEX – PRESENTATION OF THE COMPANY'S POSSIBILITIES FOR THE COMPOSITE INDUSTRY

RETEX – PŘEDSTAVENÍ MOŽNOSTI FIRMY PRO KOMPOZITNÍ PRŮMYSL

DAVID VČELIŠ¹

Presented paper deals with presentation of RETEX company and its possibilities for the composite industry.

KEYWORDS

RETEX, company, introduction

ÚVOD

Společnost RETEX a.s. působí v oblasti výroby a zušlechťování netkaných textilií již 73let.

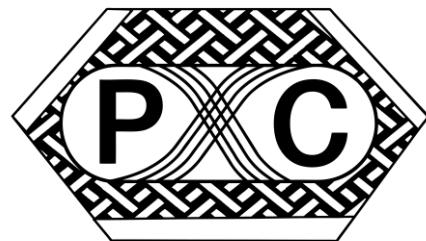
Pod záštitou systémových certifikátů řady ISO 9001, 14001, 45001, 50001 a IATF 16949 jsou řízeny dva výrobní závody. Hlavní v Moravském Krumlově a druhý ve Stráži nad Nisou. Technologický park obou závodů pokrývá oblasti recyklace, výrobu netkaných textilií a jejich následné zušlechťování na chemických linkách a výrobu kompozitů (tedy spojování různých druhů materiálu do jednoho výrobku). Naše silná tradice a zkušenosti v recyklaci textilu nám přináší možnosti obohatovat trh o řešení cirkulární ekonomiky. Působíme v různých segmentech trhu jako automotive, stavebnictví, železniční průmysl a také přinášíme řešení, které napomáhají v boji proti klimatickým změnám. Zpracovávané vstupní materiály jsou jak z na syntetické, tak i na přírodní bázi. Díky širokým výrobním možnostem a inovativnímu přístupu vývojového oddělení, rádi přijímáme výzvy z různých odvětví a připravujeme pro ně řešení na míru. Záleží nám na udržitelnosti a životním prostředí, proto jsme v mnoha segmentech trhu schopni nabídnout řešení na principech oběhového hospodářství. Společnost tedy pro kompozitní průmysl nenabízí pouze výrobu kompozitních výrobků samotných (zejména lamináty netkaných textilií), ale i řešení pro jejich následnou recyklaci a znovupoužití. Dále konvenčními textilními technologiemi, jsme schopni připravit multiaxialní vlákenné výztuže, pro následné zpracování s plastickým pojivem. Vlákenná výztuž pak dodává výslednému kompozitu lepší mechanické vlastnosti, než by měla matrice samotná.

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POLYMER COMPOSITES

2023

Tábor, May 24th–25th 2023
CZECH REPUBLIC



DEVELOPMENT OF NEW COMPOSITE MATERIALS WITH THERMOPLASTIC MATRIX AND CONTINUOUS FIBER REINFORCEMENT USING INFUSION TECHNOLOGY FOR AEROSPACE APPLICATIONS

VÝVOJ NOVÝCH KOMPOZITNÍCH MATERIÁLŮ NA BÁZI TERMOPLASTICKÉ MATRICE A KONTINUÁLNÍ VÝZTUŽE S POUŽITÍM INFUZNÍ TECHNOLOGIE PRO APLIKACE V LETECTVÍ A KOSMONAUTICE

JANA ZEMANOVÁ¹, BOHUSLAV CABRNOCH², MICHAL KRÁL³

This work is devoted to the development of new composite materials with a thermoplastic matrix and continuous reinforcement using the infusion technology, commonly used in thermosets, for aerospace applications. The principle of production technology and an overview of thermoplastics suitable for these purposes are presented here. Then, the preliminary performed experiment, the results of material tests and analysis are described. Finally, future potential research as an opportunity to create new advanced composite thermoplastic materials is identified and discussed.

KEYWORDS

Thermoplastic Composites, Continuous Fiber Reinforcement, Infusion, Aerospace

INTRODUCTION

Fiber reinforced polymer composites are increasingly popular alternatives to metal alloys in aerospace industry, and thanks to their excellent strength-to-weight ratio, they are some of the best structural materials ever. The polymer matrix is usually formed by a thermoset resin. The market size of thermoplastic composites is estimated to be about \$10 billion smaller than that of thermosets [1, 2], and they differ from them in that they soften and melt under the influence of heat and solidify again upon cooling. The fibers play an important role in improving the mechanical properties of the polymer, this function of reinforcement is well fulfilled by either inorganic (glass, carbon) or organic (polymer) fibers.

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Although thermoplastic materials in general have many advantages compared to thermosets, like easy storage, faster processing, environmental friendliness, medical harmlessness, lower flammability, good toughness and impact resistance, good resistance to corrosion and solvents, possibility of thermal welding and bonding, and recyclability at the end of their life cycle, the obstacle to their more frequent use are properties related to processing, like high melt viscosity and high melting point. This combination means high processing pressures and temperatures that limit production technology, and some composite structures are too complicated, or even impossible due to their geometry, to produce other than by infusion.

As a progressive solution in this case, polymerization in situ is offered, which provides similar processing conditions as in the case of thermoset resins. Infusion of the composite requires resins with viscosity less than 1 Pa·s to ensure good fiber impregnation, therefore, thermoplastic polymer melts having a high molecular weight are unusable for this technology. However, by polymerizing monomers directly in the tool together with fiber reinforcement, the desired viscosity can be achieved at a temperature from (20 to 250) °C. [3–5]

This research project is focused on the polymerization process, manufacturing into the final product by infusion technologies commonly used for thermosets, modification of composite material to improve its properties, or possibilities of its recycling. Efforts to understand, adapt and improve individual processes could help not only save production costs, but also make structural composites of large and complex dimensions sustainable even into the distant future for the more favorable ecological and health aspects of thermoplastic materials.

INFUSION TECHNOLOGY

The production of composite parts by infusion is a very flexible out-of-autoclave method (however, an autoclave can also be used), where a liquid system is infused into the fiber reinforcement, thereby impregnating it. Subsequently, the composite is allowed to cure at room or elevated temperature, depending on the polymer matrix used. The production of composites with a thermoplastic matrix by infusion technologies is feasible with the help of monomer/oligomer polymerization in situ where the system has almost a water-like viscosity.

There are three main infusion technologies worth introducing when it comes to the infusion of thermoplastic materials. The first is resin transfer moulding (RTM), an infusion method of vacuum-pressure impregnation of the fibrous reinforcement semi-finished product with monomer and its polymerization in the mould cavity. This process is more suitable for resins with a shorter process window, and for small to medium sized parts. On the contrary, vacuum assisted resin transfer moulding (VARTM) is a method applicable also for large structures, since the monomer is infused through a plane into the reinforcement enclosed between the bottom tool (flat or structured) and a flexible membrane under vacuum. By eliminating the upper mould and requiring only technological layers instead, this method is cheaper than RTM. And finally, resin film infusion (RFI) is a technology of infusing a thermoplastic polymer or oligomer film into the reinforcement. Such a film can be applied to dry reinforcement, or it can be sandwiched with it. This eliminates the need for a dosing equipment, which can reduce the total working time. [6–10]

SELECTION OF THERMOPLASTIC MATRIX

The viscosity, moisture absorption, process temperature, process window, service temperature and mechanical properties were selected as key parameters for choosing a suitable thermoplastic matrix for infusion technology. Based on the selected parameters, three acceptable groups of thermoplastics that theoretically meet the requirements were studied, i.e. cyclic polyesters, anionic polyamides, and acrylate copolymers. [9]

Polyesters are semi-crystalline thermoplastic polymers containing ester functional groups (-COOR). The polymerization of cyclic polyester takes place by opening the cyclic oligomers with a suitable initiator, which then spontaneously combine to form the resulting thermoplastic. However, this process does not produce exothermic polymerization, so large macrocyclic molecules are formed, and due to continuous ring expansion, the molecular weight is higher than that of a linear polymer produced by step polymerization. [9, 11, 12]

Aliphatic polyamides (PA) are linear semi-crystalline thermoplastic polymers containing an amide group (-CONH) in the main chain. By default, polyamides are produced by hydrolytic polycondensation, however PA 6 and PA 12 can be produced by relatively quick anionic polymerization of lactams at elevated temperature. Due to their chemical structure, the monomers tend to absorb the surrounding atmospheric moisture, so that partial or no polymerization can occur, which subsequently affects the mechanical properties. Therefore, it is advisable to keep used tanks and preparations that come into contact with lactams in a vacuum or treat them with N₂. It is also advisable to heat infusion tubes or hoses. [4, 5, 9, 13]

Acrylate polymers containing acrylic acid (CH₂=COOH) are amorphous linear thermoplastics that can be processed by radical polymerization already at room temperature (or higher) and viscosity from 0.1 Pa·s. Acrylate resins may contain methacrylate monomers such as methyl methacrylate or butyl methacrylate, and acrylate copolymer chains. The reaction takes place in the presence of a thermal initiator. The variability of the available resins consists in copolymerization, e.g. vinyl acetate can be added to the acrylate monomers, which acts as a crosslinker to improve the mechanical properties of the polymer. [3, 14]

PRELIMINARY EXPERIMENT

Based on the selected properties, Elijum®188 XO acrylic resin and Noviper BP 50 dibenzoyl peroxide initiator were selected to manufacture a thermoplastic composite specimen at room temperature with a theoretical viscosity of 0.1 Pa·s, a processing window of (60–90) minutes, a moisture absorption of 0.55 hm. %, a glass transition temperature of 102 °C, a tensile strength of 52.9 MPa, and a Young's modulus of 3 GPa. [15] The reinforcement function is performed by ten layers of Aeroglass 280 glass fabric, as it is often used with a thermoset matrix for the production of composites in Czech Aerospace Research Centre based in Prague.

A 500×500 mm sample was prepared using VARTM technology. The impregnation proceeded well, similar to thermoset resin, however tiny air bubbles could be seen accumulating under the vacuum bag film. Subsequently, the laminate was left for 24 hours at room temperature under vacuum, while the monomer in situ changed by reaction into a polymer under spontaneous heating and increasing viscosity. The composite specimen taken out after the given time from under vacuum was post-cured in an oven at 80 °C for 2 hours in accordance with the manufacturer's technical data sheet. The average measured thickness of the specimen containing ten layers of fabric was 2.4 mm.

In the next phase of the experimental part, the basic available tests such as differential scanning calorimetry (DSC) to study the thermal properties of the polymer, dynamic mechanical analysis (DMA), in-plane shear strength (IPS) and interlaminar shear strength (ILSS) to evaluate the mechanical properties of the composite, and finally, scanning electron microscopy (SEM) in order to assess the failure mechanism of the tested composite for ILSS were carried out on the manufactured composite specimen, see Fig. 1 for results.

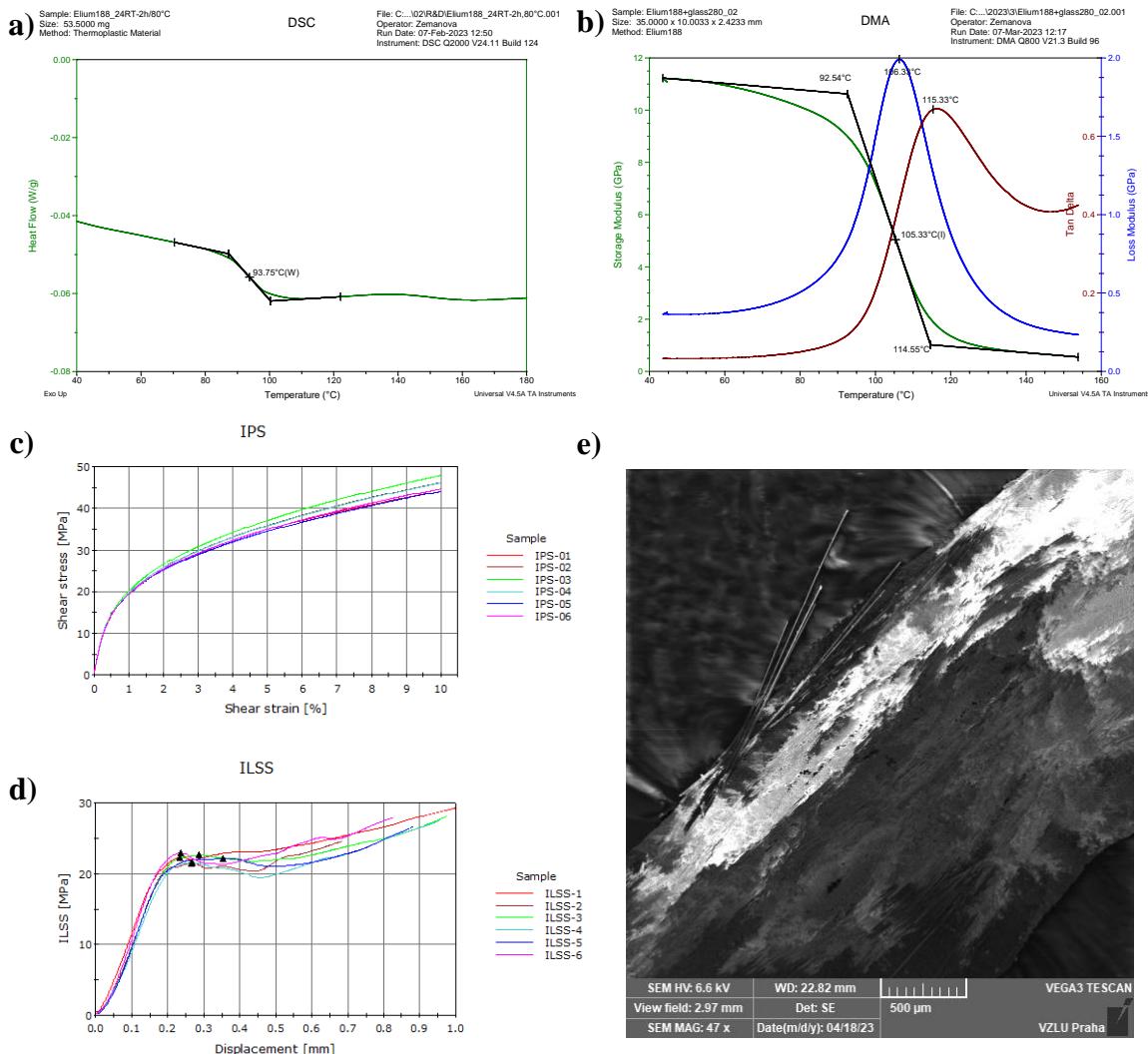


Fig. 1 Material tests and analysis results: a) DSC, b) DMA, c) IPS, d) ILSS, e) SEM.

The main result values from graphs 1 a) to d) are shown in the table below for comparison with the available internal data of conventional Epoxy LH288/Aeroglass 280 composite. As can be seen, the loss modulus, representing the viscous part of the polymer material, is higher for the Elijum® based composite. At this temperature, the polymer undergoes a maximum change in the mobility of the polymer chains and loses mechanical energy by being converted into heat. On the other hand, the in-plane shear strength and interlaminar shear strength are higher for the epoxy-based composite. The cause could be insufficient adhesion of the fiber to the thermoplastic matrix, which can be confirmed using scanning electron microscope.

Tab. 1 Test results comparison of Elijum®188 XO/Aeroglass 280 and available internal results of Epoxy LH288/Aeroglass 280 composites.

Test/Analysis	<i>Elijum®188 XO/AG280</i>	<i>Epoxy LH288/AG280</i>
DSC glass trans. temp. T_g [°C]	90.42 (pure Elijum®)	n/a
DMA loss modulus E'' [°C]	106.33	90.28
IPS [MPa]	35.55	43.53
ILSS [MPa]	22.21	48.00

In the SEM image (Fig. 1e)), it is quite clear that there is no matrix left on the pulled fiber, so it can be deduced that the failure of the composite occurred at the fiber/matrix interface as a result of the already mentioned insufficient adhesion of the fibers to the matrix. However, as is usually the case, it is advisable to do more research and testing on this topic. In every way, the main goal should be to achieve results of epoxy composite, or better, so that the thermoplastic composite can be used for current applications in the aerospace industry.

Additionally, cuttings will be prepared from the remains of the porous part of the preliminary composite specimen, and the quality of the impregnation of the reinforcement and the content of voids in the polymer matrix will be studied using optical microscopy. Although, just by looking at the specimen after the infusion ended, it was possible to conclude that appropriate corrective actions will need to be implemented in this specific case.

FURTHER POTENTIAL RESEARCH

As this is just beginning a long-term research project, attention might be focused on a more specific area of interest over time. There are certainly more opportunities where to aim. Considering the first results, and their comparison with epoxy resin as a representative of thermosets, it is suggested to carry out more different tests, the results of which could lead not only to adjust the preparation of the material and the infusion technology itself, but also to modify the individual material phases of the composite (matrix and fibers) to enhance the mechanical, thermal, electromagnetic, or other properties of the final composite.

Even the ongoing continuous search for a suitable material for the thermoplastic matrix and continuous reinforcement is beneficial and may lead to finding the golden path, not to mention the possibility of copolymerization and the creation of a new unique matrix.

Another option is to make a self-reinforced polymer (SRP) composite that has both matrix and fabric from the same polymer material that has the ability to spin. One of the benefits of SRP composites is their ability to achieve excellent fiber-matrix interfaces and full recyclability. And as is obvious, the resulting self-reinforced composite has higher stiffness and strength than the pure polymer. SRP composites might have the potential to be used for radiotransparent radomes to antenna of radar stations. [16, 17]

Furthermore, it is necessary to consider improving the surface properties of the composite such as surface energy, roughness and cleanliness affecting adhesion before painting and bonding, or applying coatings ensuring the resistance to environmental influences such as temperature, fire, ultraviolet radiation, or humidity, depending on the specific application.

Last but not least, nowadays it is desirable to study the recycling of thermoplastic composites, either by a physical method, which is more common and approachable, or by a more challenging chemical depolymerization.

CONCLUSION

The preliminary experiment showed that although the manufacturing of thermoplastic composites by infusion technology, originally intended for thermosets, is feasible without any major problems, the acrylate-based composite does not yet have the shear strength to achieve the typical epoxy composite used in aerospace applications.

Future research and experiments shall ensure the necessary properties according to the given application, and thus allow a wider use of thermoplastic composite materials with undeniable advantages even for large and complex structures, and where the institution's equipment is not capable to create such high pressures and temperatures as are required for the traditional production of thermoplastic composites.

LITERATURE

1. Thermoset Composites Market Size, Share & Trends Analysis Report. BrandEssence® Market Research and Consulting. Volume 300. London, UK. 2022.
2. Thermoplastic Composites Market – Growth, Trends, Covid-19 Impact, and Forecasts (2023 - 2028). Mordor Intelligence. Hyderabad, Telangana, India. 2023.
3. N. Pantelelis, E. Bistekos, R. Emmerich, P. Gerard, A. Zoller, R. R. Gallardo. Compression RTM of reactive thermoplastic composites using microwaves and cure monitoring. Procedia CIRP. 2019. Volume 85. Pages 249-254. ISSN 2212-8271.
4. C. Ó Brádaigh, Reactive Thermoplastic Composites - A Potential Game Changer? SAMPE Masterclass. NCC. Bristol, UK. 2013.
5. M. Bitterlich, M. Ehleben, A. Wollny, P. Desbois, J. Renkl, S. Schmidhuber. Tailored to Reactive Polyamide 6. Kunststoffe international. Munich, Germany. 2014. Volume 104. Pages 47–51.
6. A. Ausperger. Zpracování kompozitů. Technologie zpracování plastů. Střední odborné učiliště Svitavy. Svitavy. 2016. ISBN 978-80-88058-77-9.
7. F. Martaus. Infuzní technologie výroby kompozitů a jejich simulace v MKP. Seminář Aplikace moderních SW pro výpočet kompozitů. Výzkumný a zkušební letecký ústav. Praha. 2009.
8. J. Summerscales, T. J. Searle. Review: Low Pressure (Vacuum Infusion) Techniques for Moulding Large Composite Structures. Journal of Materials: Design & Applications. University of Plymouth, School of Engineering. Plymouth, UK. 2004.
9. Y. Qin, J. Summerscales, J. Graham-Jones, M. Meng, R. Pemberton. Monomer Selection for In Situ Polymerization Infusion Manufacture of Natural-Fiber Reinforced Thermoplastic-Matrix Marine Composites. Polymers. University of Plymouth, Faculty of Science and Engineering. Plymouth, UK. 2020.
10. R. Januš. Experimentální studie vakuové technologie a její možnost pro výrobu profilovaného dílu. Diplomová práce. Univerzita Tomáše Bati ve Zlíně, Fakulta technologická. Zlín. 2015.
11. Y. Zhang. Blends of cyclic poly (butylene terephthalate)/multiwalled carbon nanotube nanocomposites prepared by in situ polymerization. Diploma thesis. University of Birmingham, School of Metallurgy and Materials. Birmingham, UK. 2014.
12. D. Hanušová. Krystalizace polymerních směsí obsahujících polybutylentereftalát (PBT). Bakalářská práce. Univerzita Tomáše Bati ve Zlíně, Fakulta technologická. Zlín. 2019.
13. P. Rosso, K. Friedrich, A. Wollny, R. M. L Haupt. A Novel Polyamide 12 Polymerization System and its Use for a LCM-process to Produce CFRP. Journal of Thermoplastic Composite Materials. 2005. Volume 18.
14. M. S. Chebil, G. Bouaoulo, P. Gerard, S. EL Euch, H. Issard, E. Richaud. Oxidation and unzipping in ELIUM resin: Kinetic model for mass loss. Polymer Degradation and Stability. 2021. Volume 186. Pages 1–13.
15. ELIUM® 188 XO. Technical Data Sheet. Arkema Innovative Chemistry. France. 2023.
16. R. A. M. Santos, L. Gorbatikh, Y. Swolfs. Commercial self-reinforced composites: A comparative study. Composites Part B: Engineering. 2021. Volume 223. ISSN 1359-8368.
17. L. M. Morgan, B. M. Weager, C. M. Hare, G. R. Bishop, G. M. Smith. Self Reinforced Polymer Composites: Coming of Age. ICCM International Conferences on Composite Materials. Chesterfield, UK. 2009.



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