THERMOMECHANICAL ANALYSIS OF COMPOSITE MATERIALS WITH PLASMA TREATMENT RECYCLED CARBON FIBERS

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ABSTRACT: In present work are analyzed the changes thermomechanical properties of epoxy composites filled with 2,5wt% recycled short carbon fibers. Main objective was to study the influence of plasma treatment of fibers to melting point and thermal stability of composites.

Key Words: recycled carbon fibers, epoxy composites, plasma treatment, thermomechanical analysis

1. INTRODUCTION

It is known, that the chemical, physical and mechanical properties of the resulting polymers can be changed with additives [1]. If the polymer is filled with a carbon filler, are the properties of this composite changes. Once is the surface of the fibers modified by plasma, the adhesion between the filler and the matrix will improve, and thus improve the mechanical properties of the composite. In our study, we want to find out whether the thermal properties are changes.

2. EXPERIMENT

For experimental analysis were prepared epoxy specimens made from Bisphenol A-based low viscosity epoxy resin LR 285, and cyclic-alifatic polyamine curing agent H 508 (mixing ratio 100:40 by weight)[2]. Specimen were filled with short recycled carbon fibers in the 2,5wt% concentrations. The used fillers were Carbiso Milled Carbon Fibre with average diameter about 7 μm, and average length 100 μm [3].
It was prepared 8 different plasma intensities of samples. Specific functionalization of fibers was prepared by microwave plasma treatment, a gas mixture of oxygen and hydrogen was used for this purpose.

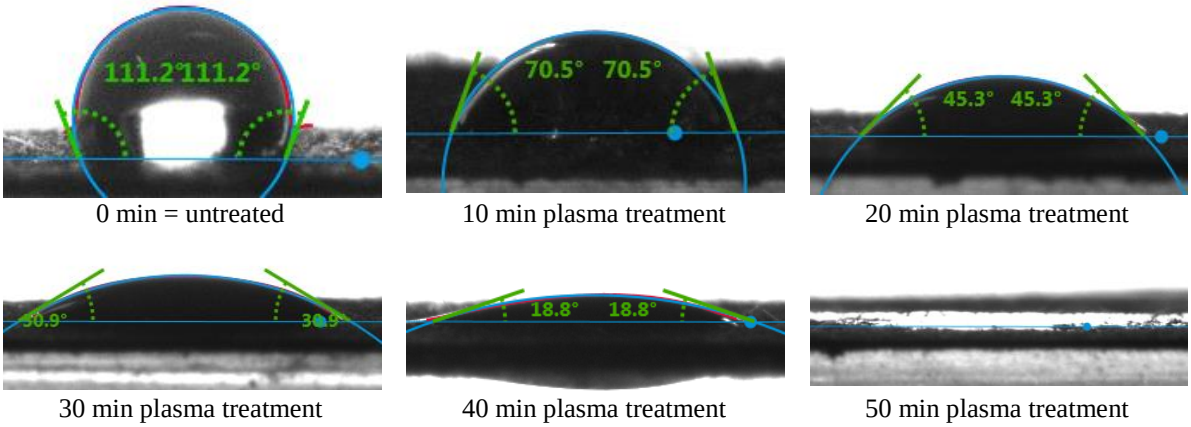
Table 1. List of testing composites samples

Sample	Plasma treatment time [min]
Pe	0
P0	0
P10	10
P20	20
P30	30
P40	40
P50	50
P60	60
P70	70
P80	80

*Notice: Sample **Pe** its sample without carbon fibres, just matrix´.*

2.1. Plasma treatment of carbon fibres

The Carbiso fibres were processed in the low-pressure laboratory plasma system (LA 400 SurfaceTreat). Plasma is generated by microwave source with a power of 1 kW, which is placed on the top of 64 l chamber. Working pressure 100 Pa was secured by rotary vacuum pump with pump speed 40 m^3/h and Roots pump with pump speed 240 m^3/h. Pressure measurement was done using Pirani gauge, regulation of pressure was secured by butterfly valve. The treatment chamber was filled with working gasses – oxygen 200 sccm and helium 50 sccm during the plasma treatment. This mixture was chosen due to oxygen functionalization of the material surface, which is observed after hydrophilisation of the surface. Fibers were placed in the mixing unit (pot) designed for processing of powder materials. This device is optional content of the LA400 system. The batch size of 5 g was treated for 10-80 min.



0 min = untreated | 10 min plasma treatment | 20 min plasma treatment

30 min plasma treatment | 40 min plasma treatment | 50 min plasma treatment

Figure 1. Examples of measurement of contact angle for evaluation of plasme treatment effect

The apparent contact angle value is measured only due to surface roughness and it shows only trend of plasma treatment effect. It can be observed from the measured values, that Carbiso plasma treatment has significant influence on change of hydrophobic character. Non-treated fibers show hydrophobic behaviour, plasma treated fibers then show hydrophilic behaviour. Increased hydrophilicity of the fibers is probably caused by binding of oxygen groups on the fibers surface together with surface roughness caused also by plasma treatment.

2.2. Thermomechanical analyses of composites

Specimens of composites were tested by thermal method of measurement:DMA (Dynamical-mechanical analyses), DSC (Differential scanning calorimetry) and TMA (Thermo-mechanical analyses). Results of measurement you can see on table 2.
The DMA method was used to measure the three-point bend, whereby the complex modulus of elasticity and the loss coefficient were determined. The complex modulus of elasticity has decreased, so plasma has an effect on mechanical properties, unfortunately negative.
The DSC method was used to determine the glass transition temperature and sample degradation temperature. The plasma treatment had no effect on the glass temperature. The values were very similar and their difference was not statistically significant. The effect of plasma was evident at the degradation temperature. Here we can say that temperature resistance is better. The more the sample is polished, the higher the degradation temperature.
The TMA method measures the coefficient of thermal expansion. The coefficient of thermal expansion was calculated. Using this method, it was found that samples tend to shrink rather than increase. In some cases the reduction was very significant. Which is not desirable. Thus, plasma treatment is also not suitable.

Table 2. Results of measurement of thermal analyses

Method of thermal analyses	DSC		TMA	DMA	
Sample	T_g [°C]	T_{deg} [°C]	λ [K^{-1}]	E [GPa]	tgδ [-]
Pe	60,0	205,8	$-7{,}50.10^{-6}$	1,312	0,277
P0	62,4	218,5	$8{,}56.10^{-6}$	1,850	0,227
P10	59,9	214,7	$-5{,}04.10^{-5}$	1,603	0,204
P20	61,7	206,8	$-2{,}19.10^{-5}$	1,474	0,234
P30	61,7	225,7	$-3{,}55.10^{-5}$	1,349	0,294
P40	63,4	232,5	$-5{,}97.10^{-5}$	1,375	0,267
P50	62,0	234,9	$-6{,}56.10^{-5}$	1,378	0,277
P60	64,6	230,5	$2{,}53.10^{-7}$	1,386	0,263
P70	62,3	231,1	$-4{,}56.10^{-5}$	1,398	0,255
P80	65,7	229,3	$-1{,}05.10^{-5}$	1,325	0,259

T_g [°C] ... temperature of glass transition; T_{deg} [°C] ... temperature of degradation; λ [K^{-1}] **...** coeficient of thermal expansion; E [GPa] … module of elasticity, tgδ [-] ... loss coeficient

3. CONCLUSION

The aim of this work was to verify the effect of plasma carbonization on the resulting thermal properties of composite materials made from them. And to find out whether composite materials will have better mechanical and thermal properties through plasma treatment. Samples were tested, DMA - dynamic-mechanical analysis, DSC - differential scanning calorimetry and TMA thermomechanical analysis.

We made a new composite where two-dimensional carbon fibers acted as bridges, whereby the formation of a strong three-dimensional mesh structure in the epoxy matrix was attributed to better surface compatibility from carbon fibers in the matrix. [4] The use of plasma carbon surface modification results in an improvement in the thermal stability of the composites produced. Other preconditions for improving thermomechanical properties have not been confirmed.

Acknowledgements

Authors are grateful for the financial support from the Student research project 2019 No. 21308 supported by Czech Ministry of Education, and by Faculty of Textile, Technical University of Liberec.

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