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Microwave field effects on internal stresses in additively manufactured polymer composites

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Abstract. Studies have been conducted on the influence of microwave electromagnetic fields on the magnitude of internal stresses in cured carbon fiber, glass fiber, and organic plastics, as well as unidirectional composites obtained by FDM technology from PEEK thermoplastic reinforced with continuous carbon fiber. A reduction in internal stresses as a result of microwave exposure was established, averaging 9%, 6.5%, 6%, and 5.4% for carbon fiber, glass fiber, organic plastics, and unidirectional reinforced PEEK, respectively. The reduction in internal stresses is small in value and is a concomitant effect; however, in the context of a certain increase in the safety factor and, consequently, the reliability of products made from polymer composite materials, it can be considered as another argument for the practical application of microwave technologies in the production of PCM products.

1 Introduction

Currently, polymer composite materials (PCMs) reinforced with continuous fibers or fabrics based on them are widely used in various industries [1,2]. This is due to the high specific strength of PCMs compared to metals and alloys, as well as the ability to form properties predetermined by operating conditions and functional purpose of products simultaneously with obtaining their shape [3-4]. Despite the increased use of super-structural thermoplastic polymers as binders, a significant volume is still retained by thermosetting polymers, particularly epoxy binders. These materials are characterized by low shrinkage and sufficient thermal stability in relation to most fillers, and also provide higher impregnation quality compared to thermoplastics [5-6].

At the same time, PCMs, including those based on epoxy binders, have significant drawbacks: pronounced anisotropy of properties, low impact resistance compared to metals, low thermal conductivity, and increased creep. Shrinkage accompanying the curing process leads to the formation of microdefects in the material volume and the formation of residual stresses due to significantly different thermophysical characteristics of the components, which results in reduced strength and stiffness [5-6]. To reduce the disadvantages of PCMs, methods of chemical and physical modification of components are applied at the preliminary stage of manufacturing and during the curing process. In this context, the modification of

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cured PCMs based on epoxy binders that have undergone a complete technological cycle becomes important [7-8]. This solution does not require changes to established technologies for synthesizing materials from commercially produced components and eliminates the possibility of subsequent technological operations influencing the obtained modification result. Consequently, the development of research in this direction is relevant for both science and practice.

2 Problem statement

One of the promising directions for physical modification of polymer composite materials (PCM) products in Russia and abroad is treatment in a super-high-frequency (SHF) electromagnetic field [9-11]. However, the majority of conducted research and technical solutions relate to separate processing of components and processes occurring during the curing stage of PCM. The impact of SHF electromagnetic and other fields on the change in physical and mechanical properties of PCM in the cured state is mainly considered as a destructive natural or technogenic factor.

At Yuri Gagarin State Technical University of Saratov, experimental studies were conducted from 2015 to 2024 on the influence of SHF treatment at a radiation frequency of 2450 MHz on the strength characteristics of cured carbon, glass, and organic plastics under static and dynamic loading. In particular, it was shown that SHF exposure with an energy flux density of (17-18)x10^4 μW/cm^2 for 2 minutes on fully formed samples of carbon fiber-reinforced PCM contributes to an increase in interlaminar shear stress by (16-18)% and elastic modulus by 14-20% [12]. It was demonstrated that the greatest effect of SHF exposure is observed at a fixed integral temperature over the sample surface in the range of (60-80)°C, which corresponds to data reported in scientific literature [13].

The possibility of obtaining certain effects from SHF exposure on cured PCM has also been confirmed by foreign researchers [14,15]. However, the influence of SHF electromagnetic field on the physical and mechanical properties of continuous fiber-reinforced PCM formed using additive technologies, which are currently one of the trends in the development of digital production, remains poorly studied.

The scientific literature also insufficiently covers the effect of SHF exposure on internal stresses in PCM. It is noted that internal stresses caused by thermal shrinkage of PCM during curing, considering the sharply different thermal expansion coefficients of the filler and binder, often reach 60% of the tensile strength [16]. Consequently, reducing internal stresses during PCM formation is one of the reserves for improving the functional characteristics and reliability of products made from them.

In this regard, it is important to note that when heating polymer composite materials, including those with epoxy binders, effects of internal stress relaxation are observed, which contributes to a significant leveling of composite "aging" and even a certain return to the initial state - "rejuvenation" [17-20].

The aim of the research was to study the relaxation of internal stresses in samples of carbon, glass, and organic plastics, as well as carbon fiber reinforced plastic obtained by the FDM (Fused Deposition Modeling) additive technology after exposure to SHF electromagnetic field.

3 Materials and methods

To evaluate the internal stresses in the samples, the methodology described in [21] was employed. This methodology is based on the calculation of stresses using the thermal expansion coefficient of the investigated material, utilizing the corresponding dependence:

$$\sigma = E\,\alpha\,(T - T_0), \tag{1}$$

$$\alpha = \delta / (h{*}T), \tag{2}$$

where E is the elastic modulus of the material in MPa, α is the coefficient of thermal expansion in K^{-1}, δ is the deformation in mm, h is the sample thickness in mm, T is the test temperature in K, T_0 is the initial temperature (ambient) in K.

Samples were fabricated from 500x500x5 mm plates of carbon fiber and fiberglass composites produced by LLC "Eurocomplekt" in Kaluga, and organic plastic from JSC TsVM "Armokom" in Khotkovo, Moscow region. Additionally, samples were produced using FDM technology from unidirectional super-structural thermoplastic polyetheretherketone (PEEK) reinforced with continuous carbon fiber, with dimensions of 150x80x(1.55-1.6) mm.

According to [1], the following elastic modulus values were adopted for calculations: carbon fiber composite E = 120-130 GPa, fiberglass E = 40-69 GPa, organic plastic E = 80-140 GPa. Due to the lack of reference data on the physical and mechanical properties of composites obtained by additive methods, for the unidirectional PEEK sample, E = 30 GPa was adopted according to [20], assuming 30% carbon fiber filling, as currently, denser filling by 3D printing methods is not feasible.

Samples in the form of plane-parallel plates with transverse dimensions of 75x10 mm were cut from the plates and divided into two groups - control and experimental (subjected to microwave exposure).

The second group of samples was processed using a special microwave technological installation assembled based on the "Zhuk-2-02" emitter (LLC NPP "AgroEcoTech", Obninsk, Kaluga region) with a horn-type emitter. The electromagnetic field frequency was 2450 MHz, with a magnetron power consumption of 1200 W. The equipment was modernized by installing a three-coordinate table with a positioning accuracy of 0.1 mm and a polypropylene PP panel measuring 400x500 mm for placing samples of processed materials.

Microwave processing was carried out at the following energy flux density values: EFD = $(17\text{-}18)\times10^{-4}$ μW/cm^2 (carbon fiber composite and unidirectional carbon fiber reinforced PEEK) and EFD = $(40\text{-}50)\times10^{-4}$ μW/cm^2 (fiberglass and organic plastic). The modes were selected in accordance with, which showed that under these conditions, a heating temperature of (70-80)°C is achieved for polymer composite materials (PCM) reinforced with carbon fiber, and (40-60)°C for other PCMs, ensuring maximum improvement in physical and mechanical properties. The microwave exposure time was recorded upon reaching the specified surface temperature, determined by a FLIR E-40 thermal imager.

After microwave exposure, plates with transverse dimensions of 5x5 mm were cut from the experimental samples, with sample thicknesses of 1.5-2.5 mm achieved by splitting the plates. Control samples were also brought to similar dimensions. A total of 5 control and experimental samples were prepared for all investigated materials.

The elongation of samples during heating was determined using an experimental setup assembled according to recommendations in [20]. The setup was equipped with a precision inductive micro-displacement sensor Pepperl&Fuchs 3RG4012-3KB00-PF Artikel 123301, connected to a computer with LabView software (IP "Mayorov", Orel). The test scheme is presented in Fig. 1. The sample was placed in a cylindrical case made of D16T alloy and covered with a KU-1 quartz glass disk. The case was fixed on a D16T plate with built-in electric heater and temperature control thermocouple. A KU-1 rod with a diameter of 5 mm and length of 150 mm was inserted into the case opening until it touched the sample. A steel tip was attached to the upper end of the rod for interaction with the sensor. Using a micrometer screw, the sensor was brought to the tip surface until a gap of 1 mm was obtained. The heater regulator was set to a temperature of 70°C to avoid thermal damage to the rod and adhesive securing the tip. After turning on the heater, the sample elongation was recorded by the movement of the cursor (line) on the computer monitor screen. The test time was set to 5 minutes for stabilization of heating and changes in sample dimensions.

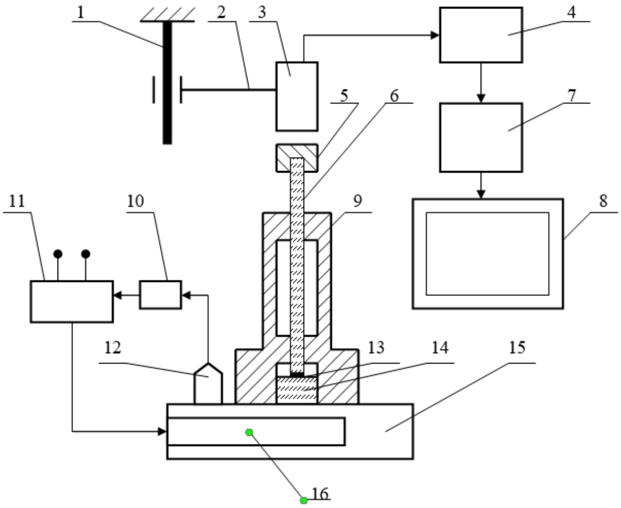
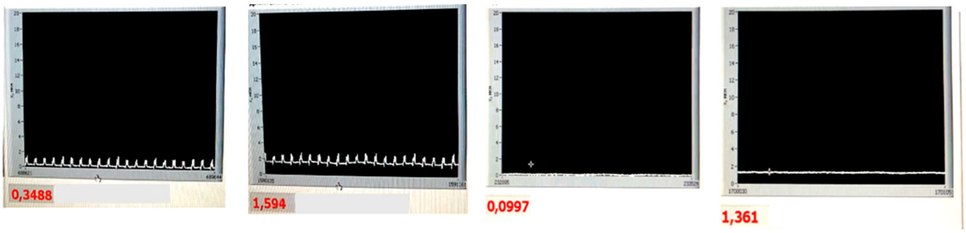


Fig. 1. Schematic diagram for determining the thermal expansion of PCM samples: 1 - Stand with displacement mechanism; 2 - Movable bracket; 3 - Micro-displacement sensor; 4 - Analog-to-digital converter; 5 - Steel tip; 6 - KU-1 glass rod; 7 – Computer; 8 – Monitor; 9 – Case; 10 – Controller; 11 - Heater power supply; 12 - Temperature sensor; 13 – Sample; 14 - KU-1 plate; 15 - Heating plate; 16 - Electric heater.

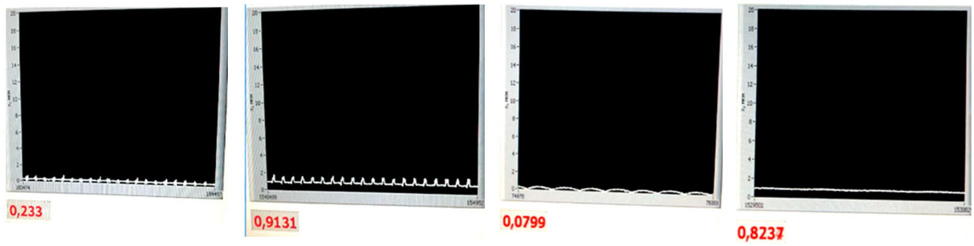
4 Results and discussion

The obtained results are presented in Figures 2-6 and Tables 1 and 2. Figures 2-5 depict photographs of typical readings indicating the change in position of the nozzle on the quartz glass rod, caused by the elongation of the polymer composite material (PCM) sample during the heating process. The absolute value of elongation is shown in micrometers (μm).



a b c d

Fig. 2 Typical photos of the setup screen, illustrating the absolute increase in thickness of the control (top row) and experimental (bottom row) carbon fiber samples when heated to 500C. (a) and (c) – initial stage of heating, (b) and (d) – after 5 minutes.



a b c d

Fug. 3. Typical photos of the setup screen, illustrating the absolute increase in thickness of the control (top row) and experimental (bottom row) fiberglass samples when heated to 500C. (a) and (c) – initial stage of heating, (b) and (d) – after 5 minutes.

a b c d

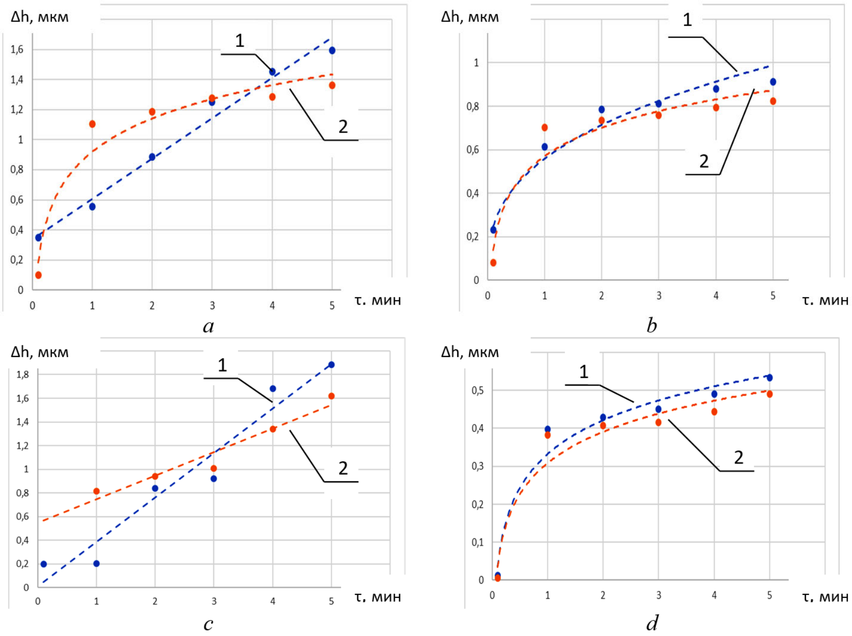
Fig. 4. Typical photos of the setup screen, illustrating the absolute increase in the thickness of the control (top row) and experimental (bottom row) organoplastic samples when heated to 500C. (a) and (c) – initial stage of heating, (b) and (d) – after 5 minutes.

a b c d

Fig. 5. Typical photos of the setup screen, illustrating the absolute increase in the thickness of the control (upper row) and experimental (lower row) samples of superstructural thermoplastic PEEK, reinforced with continuous carbon fiber during 3D printing, when heated to 500C. (a) and (c) - the initial stage of heating, (b) and (d) - after 5 minutes.

In certain instances, a serrated pattern was observed on the monitor line indicating the position of the tip, caused by the sensor's detection of external vibrations from the operating computer cooling system. It can be noted that the maximum elongation is characteristic of organoplastic, while the minimum elongation is observed in reinforced thermoplastic PEEK formed by additive manufacturing. Furthermore, analysis of Figures 2-5 reveals that all experimental polymer composite material (PCM) samples exhibit reduced elongation upon heating.

Analysis of the graphs depicting the change in elongation of PCM samples over heating time (Figure 6) indicates that all materials demonstrate rapid initial elongation followed by a deceleration, as well as decreased elongation for the experimental samples. The relationships for carbon fiber and glass fiber composites, as well as unidirectional carbon fiber reinforced PEEK, are accurately approximated by logarithmic functions (Table 1). Linear dependencies are characteristic of organoplastic materials.



a *b*

c *d*

Fig. 6. Kinetics of thermal expansion of control (1) and experimental (2) samples of carbon (a), glass (b), organoplastics (c) and thermoplastic PEEK reinforced with continuous carbon fiber in the process of 3D printing (d).

The notably pronounced excess elongation of experimental samples compared to control samples in the first half of the heating period and its subsequent decrease is noteworthy. This phenomenon may be attributed to the increase in the number of contact points between the matrix and fiber, which is particularly evident in carbon fiber reinforced plastics. Possessing higher thermal conductivity coefficients, the fibers in carbon fiber and organic fiber reinforced plastics facilitate rapid propagation of heat flow throughout the volume of the polymer composite material and intensive heating of the matrix through a greater number of contact surfaces, resulting in an increase in the overall elongation of these materials.

Table 1. Approximating functions of absolute expansion of control and experimental samples when heated to 700C and held for 5 minutes.

Material	**Sample type**	
	Control	**Experimantal**
Carbon fiber	$y = 0{,}2689x + 0{,}337$	$y = 0{,}3197\ln(x) + 0{,}9194$
Fiberglass	$y = 0{,}5585x^{0{,}3546}$	$y = 0{,}1882\ln(x) + 0{,}5706$
Organoplast	$y = 0{,}3765x + 0{,}0076$	$y = 0{,}2x + 0{,}5451$
PEEK, reinforced with continuous carbon fiber	$y = 0{,}1284\ln(x) + 0{,}3324$	$y = 0{,}1186\ln(x) + 0{,}3085$

Subsequently, the significantly lower coefficient of thermal expansion of the reinforcing fibers compared to the binder, due to a greater number of contact surfaces, inhibits the expansion of the matrix, which reduces the overall elongation of samples of all polymer composite materials (PCMs) in the second half of the heating cycle.

The results of calculations using equation (1) for internal stresses in control and experimental samples of the studied PCMs (Table 2) demonstrate a reduction in this parameter as a result of microwave exposure, on average, for carbon fiber, glass fiber, organic plastics, and unidirectional reinforced PEEK by 9%, 6.5%, 6%, and 5.4%, respectively.

Table 2. Change in internal stresses in PCM after exposure to a microwave electromagnetic field in the cured state.

Material	**Carbon fiber**		**Fiberglass**		**Organoplastic**		**PEEK reinforced with continuous carbon fiber**	
Type	Contr.	Exper.	Contr.	Exper.	Contr.	Exper.	Contr.	Exper.
δ, mkm	1.59	1.36	0.91	0.82	1.89	1.62	0.53	0.49
h, mm	1.6	1.5	2.0	1.93	2.6	2.37	1.6	1.57
$\alpha*10^6$, K^{-1}	3.077	2.807	1.409	1.315	2.25	2.116	1.026	0.966
σ, MPa	11.54	10.53	2.3	2.15	7.4	6.98	0.92	0.87
$\Delta = \sigma_{оп}/ \sigma_{к}$	-	0.91	-	0.935	-	0.94	-	0.946

It should be noted that while the obtained results demonstrate a tendency towards reduction of internal stresses in the experimental samples, they are nevertheless significantly lower than the effects obtained in terms of increasing the strength and stiffness of PCM samples [12]. The effect of stress reduction for carbon fiber reinforced plastic is almost 2 times less compared to the effect of its strengthening, for organoplastic - 1.5 times, and for unidirectional PEEK the effect is generally insignificant. Some correspondence between the effects of strengthening and reduction of internal stresses is observed for fiberglass. Evidently, the determining factor for improving the physical and mechanical properties of cured PCMs through their treatment in a microwave electromagnetic field can be considered the mechanism we proposed, which consists in the temporary transition of the binder to a plastic state as a result of heating, making it possible to redistribute it with the formation of a greater number of points of physical and mechanical contact with fibers under the action of the wave component of microwave radiation. The accompanying reduction in internal stresses makes a generally insignificant contribution to increasing the strength and stiffness of materials; however, in terms of some increase in the safety factor and, consequently, the reliability of PCM products, it can be considered another argument for the practical application of microwave technologies in the production of polymer composite materials. To clarify the significance of the microwave exposure effect on reducing internal stresses in PCMs formed on the basis of super-structural thermoplastics using additive technologies, additional research is advisable.

5 Conclusion

Internal stresses in polymer composite materials are a factor that reduces the strength margin of products made from them. It has been established that microwave electromagnetic field treatment of cured carbon fiber, glass fiber, and organic plastics, as well as polyetheretherketone (PEEK) reinforced with continuous carbon fiber and formed using FDM technology, contributes to a reduction in internal stresses by 9%, 6.5%, 6%, and 5.4%, respectively.

The effect of stress reduction for carbon fiber-reinforced plastic is almost 2 times less compared to its strengthening effect, for organic plastic - 1.5 times less, and for unidirectional PEEK, the effect is insignificant. To clarify the significance of the microwave exposure effect on reducing internal stresses in polymer composite materials formed on the basis of super-engineering thermoplastics such as PEEK using additive technologies, it is advisable to continue research.

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