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THE EFFECT OF THE ADDITION OF NANOTUBES UPON THE THERMO-PROTECTIVE PROPERTIES OF THE ARAMID-REINFORCED COMPOSITE

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Abstract

The aim of this article is to present findings concerning the thermo-protective research into ablative materials. The authors analysed the impact of the addition of carbon nanotubes upon the selected ablation properties, i.e. the ablation mass waste, average linear rate of ablation and the backside temperature of the specimens. The performed tests as well as the obtained findings allowed formulating a number of conclusions, which are useful in creating future composites.

Composition of individual test samples; ablation testing; average relative ablation mass loss, depending on the volume share of carbon nanotubes; average ablation rate, depending on the volume share of carbon nanotubes; temperature of the rear surface of the insulating sample, depending on the volume share of carbon nanotubes; temperature inside the composite, depending on the volume share of carbon nanotubes; comparison of the temperature of ablation surface, temperature inside the composite and temperature on the rear surface of the wall of carbon nanotubes after the exposition to a heat flux are presented in the article.

Keywords: *ablative materials, composites, thermal resistance, ablation properties*

1. Introduction

The use of modified plastics as ablative materials protecting against excessive temperature increase was connected with the middle of 20 century, directly with arms industry as well as aeronautical, rocket and space techniques. These materials can also be used in the design of passive fireproof protections for large cubature supporting elements in building structures, communication tunnels and for the protection of data stored in electronic, optical, and magnetic carriers. Moreover, polymeric ablative materials play a strategic role in the thermal protection system of rocket nozzles, space vehicles, and rocket combustion chambers [1, 3, 10]. Ablation is a self-regulating heat and mass transfer process, which, due to physical and chemical reactions, leads to irreversible structural and chemical changes of a material combined with heat absorption at the same time. The process is initiated and sustained by external sources of thermal energy (Fig. 1) [8]. Once an ablation surface interacts with a high-temperature heat stream, an ablation process is initiated. This is when the ablation surface, under the influence of temperature, undergoes internal structural changes, which protect lower layers and affect thermos-protective properties of a material. If an ablation shield has a multi-surface structure, the process takes place in cycles: the external layer is sometimes burned, falling off the larger part;

subsequently another layer undergoes ablation changes. Among the factors, which are not directly linked with the ablation layer, the cohesion of native material plays a tremendous role in its exploitation reliability, particularly in the case of thermal interactions. Despite numerous years of applying ablation materials, there is stillroom for fully determine the quality and quantity relationships between the type-phase composition and ablation properties, in the context of other exploitation features of composites used for thermos-protective shields [7, 9]. Polymer composites with fibre reinforcement in the form of glass fibres and carbon fibres have been quite thoroughly investigated, also in connection with various powder fillers [4-7, 12-13]. It is also true of composites with reinforcement in the form of carbon fibre as well as multilayer composites with hybrid reinforcement [11, 13].

The aim of this article is to present findings concerning the thermo-protective research into ablative materials. The authors analysed the impact of the addition of carbon nanotubes upon the selected ablation properties.

2. The course of experimental studies

In order to carry out the investigation, we prepared 20 samples with a diameter of 40 mm and the ultimate thickness of approximately 5 mm. Each series included 2 to 3 specimens. The reinforcement was 15 layers of aramid fabric of 220 g/m^2 in weight, which were laid out in the LH 145 epoxide resin matrix base, cured with H147 agent in room temperature. The composite materials (samples) were made by means of the wet method immediately in the specially prepared silicone moulds. The properties of the composite were modified by changing the volume share in the matrix of carbon nanotubes, which equalled as follows 0%, 1%, 2%, 3%, 4%, 5% and 6% (Tab. 1). The multi walled carbon nanotubes (MWCNT)) used in the investigation are 90% pure. They are 10-30 µm long and 10-30 nm in the external diameter. The ready samples underwent ablation testing on a special laboratory stand, which was designed and constructed in the laboratory of the Polish Air Force Academy.

Tab. 1. Composition of individual test samples

Number of sample	Reinforcement		Matrix		manner of sample hardening	time of hardening
	aramid fabric (number of layers)	carbon nanotubes [%]	epoxy resin	hardener		
0.1	15	0	LH 145	H 147	Wet with no pressure	7 days
0.2					Wet with no pressure	7 days
1.1		1			Wet with no pressure	7 days
1.2					Wet with no pressure	7 days
1.3					Wet with no pressure	7 days
2.1		2			Wet with no pressure	7 days
2.2					Wet with no pressure	7 days
2.3					Wet with no pressure	7 days
3.1		3			Wet with no pressure	7 days
3.2					Wet with no pressure	7 days
3.3					Wet with no pressure	7 days
4.1		4			Wet with no pressure	7 days
4.2					Wet with no pressure	7 days
4.3					Wet with no pressure	7 days
5.1		5			Wet with no pressure	7 days
5.2					Wet with no pressure	7 days
5.3					Wet with no pressure	7 days
6.1		5			Wet with no pressure	7 days
6.2					Wet with no pressure	7 days
6.3					Wet with no pressure	7 days

16 test samples were subjected to ablation testing by fixing them successively in fire resistant plasterboard pieces on an adjusted laboratory stand and then exposing to a high temperature heat flux for a period of τ = 150 seconds. The heat source was obtained from the combustion of the gas mixture of liquid propane – butane, producing the flame temperature of 1100°C.

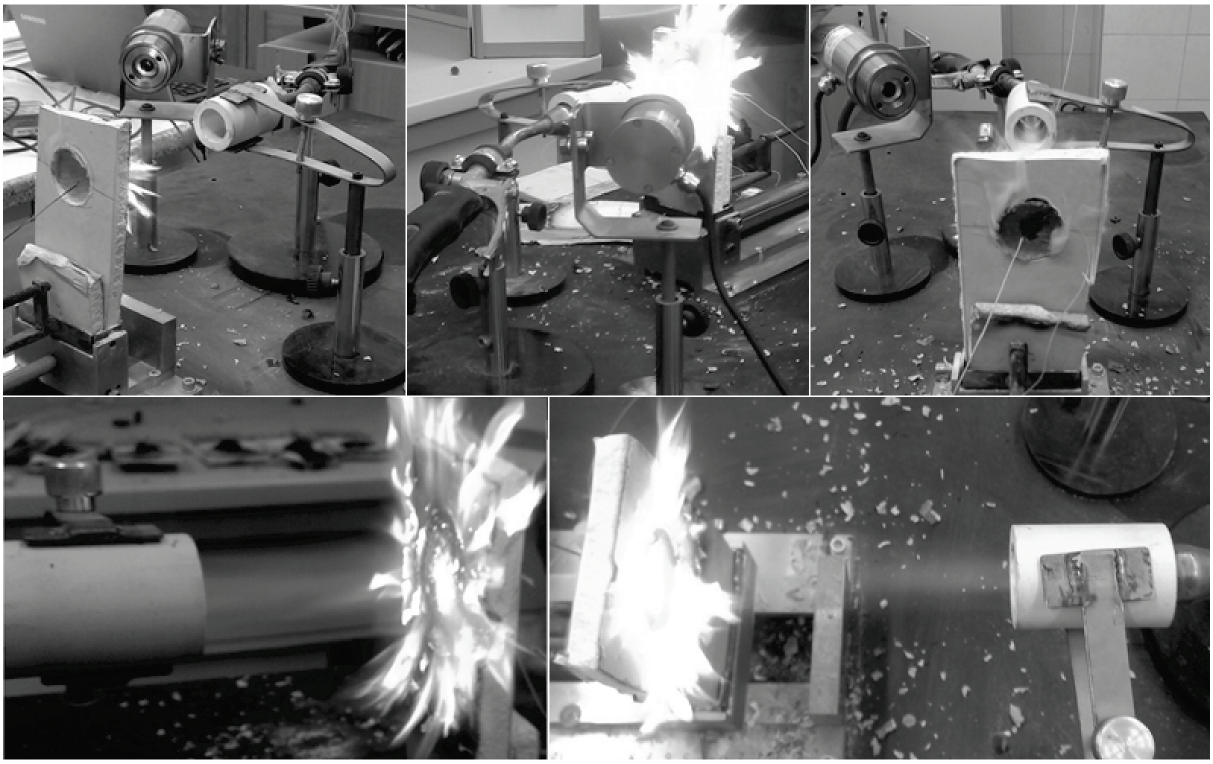

Fig. 1. Ablation testing

The flame was stabilized in the burner nozzle and also by means of an ablation gun – a fire resistant cylinder of an external diameter of 41 mm, with the hole diameter of 21 mm and the length of 60 mm, which reduces the flow rate of the exhaust gas streams. Thus, the temperature of the ablation surface t_{pa} over the whole surface of the sample reaches almost the same level. The burner together with the ablation gun was set at a distance of 95 mm away from the ablation surface of the sample. In order to measure the temperature of the ablation surface t_{pa} we used the optical pyrometer OPTCTL2MH1CF3 manufactured by Optris with an integrated dual-focus laser, which determines the exact position and the size of the measurement field size. In addition, we measured the temperature of the back side of the wall of the test sample t_s and the temperature inside the composite t_{ins} (at a depth of approx. 2 mm) using the thermocouple J type (Fe-CuNi) and K (NiCr -NiAl), which had previously been placed in the composites, as well as using the contact method (Fig. 1).

3. Findings

The main parameters of the evaluation of the ablation thermo-protective properties of the examined composites were adopted as follows: relative ablative mass loss U_a, rate of ablation v_a, temperature of the rear surface of the wall t_s, and the temperature inside the composite t_{int}. Additionally, we calculated the density of the composites prior to and after conducting the experimental studies (Fig. 2).

On the basis of the obtained graph, it was found that the density of the composites decreased by approximately 50% for all types of the test samples.

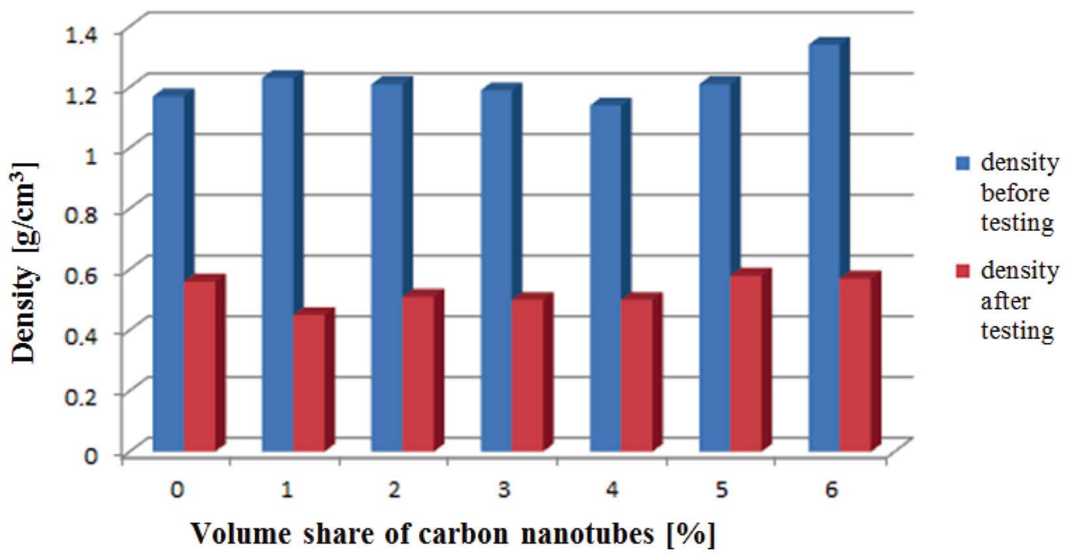

Fig. 2. Comparison of density of the composite samples before and after ablation testing

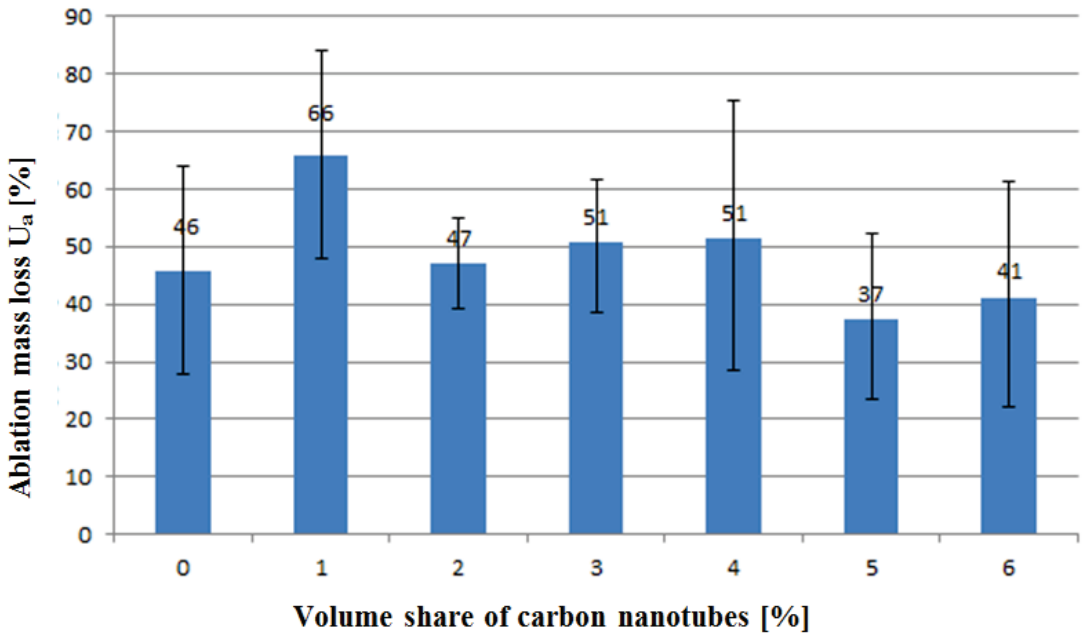

Fig. 3. Average relative ablation mass loss U_a [%], depending on the volume share of carbon nanotubes after a period of 150 seconds of the exposition to a heat flux

Based on the obtained results, it was found that the mass of the test samples after the ablation tests decreased by approximately 60%. The addition of carbon nanotubes of 5% and 6% volume share lowers the ablation mass loss by approximately 10%, while the addition of 1% of carbon nanotubes increases the ablation mass loss by approximately 15% (Fig. 3).

Another determined parameter was the linear rate of ablation, i.e. the depth where the ablation front was shifted. The composite of 1% volume share of carbon nanotubes was characterised by the lowest rate of ablation (15 µm), whereas the highest (22 µm) was the composite of 5% share (Fig. 4).

The lowest temperature on the backside of the wall (150°C) was achieved by the composite made from: 15 layers of aramid fibres and epoxy resin matrix base LH145 as well as the hardener H147, without carbon nanotubes. The highest temperature of the rear surface of the wall (223°C) was achieved in the composite built with 15 layers of aramid fibre and the epoxy resin LH145 as well as the hardener H147 containing 1% of carbon nanotubes (Fig. 5).

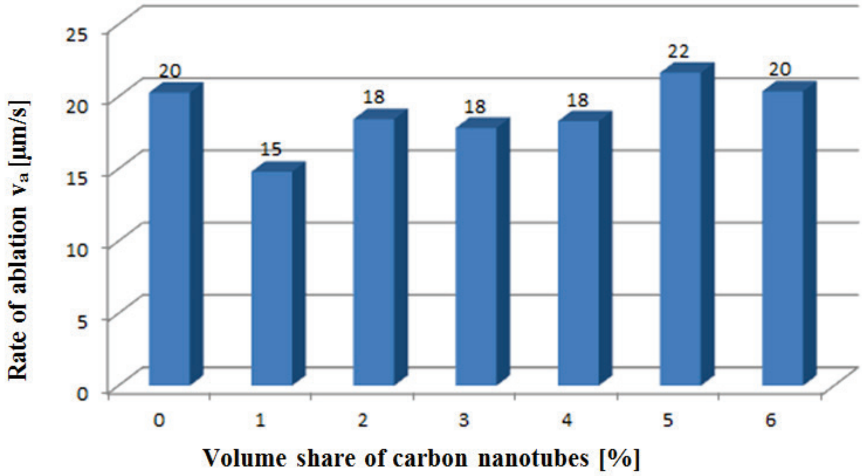

Fig. 4. Average ablation rate v_a [μm], depending on the volume share of carbon nanotubes after a period of 150 seconds of the exposition to a heat flux

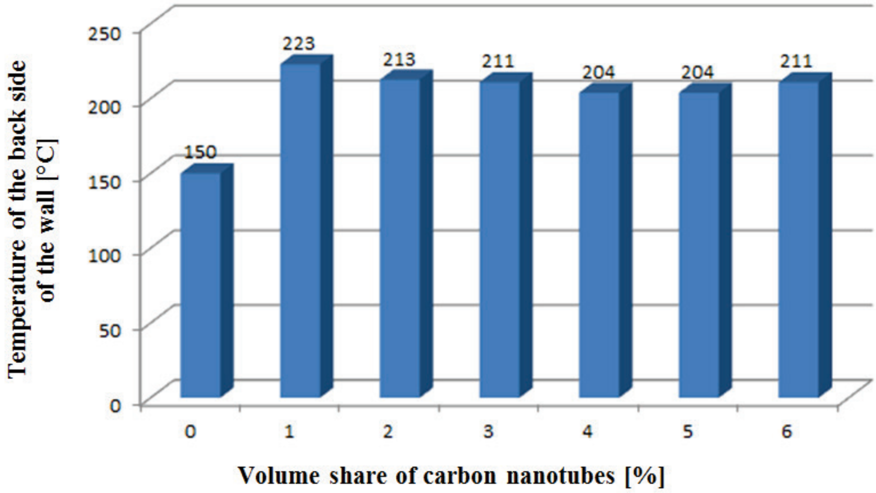

Fig. 5. Temperature of the rear surface of the insulating sample t_s [°C], depending on the volume share of carbon nanotubes after a period of 150 seconds of the exposition to a heat flux

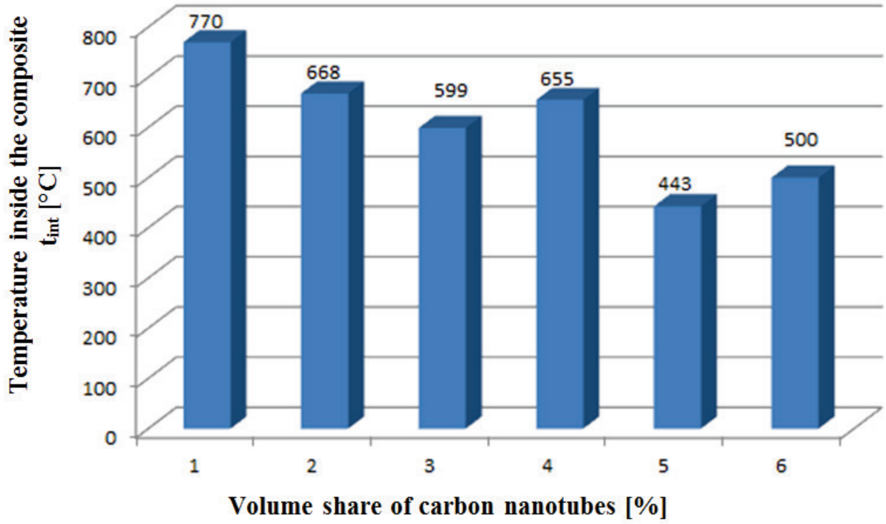

Fig. 2. Temperature inside the composite t_{int} [°C], depending on the volume share of carbon nanotubes after a period of 150 seconds of the exposition to a heat flux

The highest temperature between layer 7 and 8 of the aramid fibre t_{int} (770°C) was achieved by a composite with 1% volume share of carbon nanotubes, whereas the lowest one of 443°C was achieved by the composite of 5% volume share of carbon nanotubes. The composites with 6% volume share of carbon nanotubes also achieved a lower temperature inside than in the case of the other ones (Fig. 6).

Next, in order to illustrate the obtained findings, all the measured temperatures for a selected test sample were compared and presented in Fig. 7.

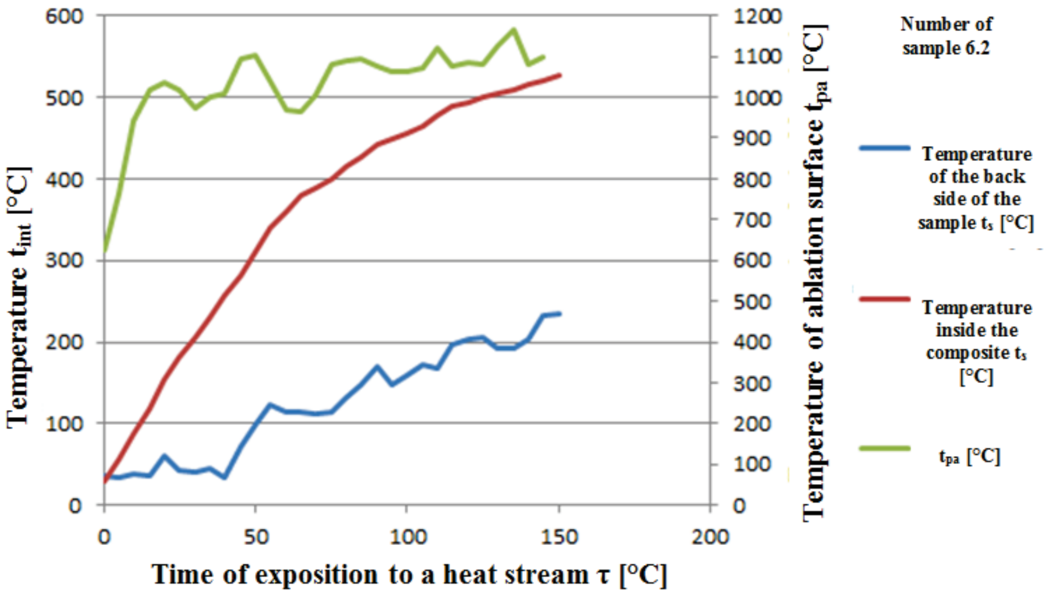

Fig. 7. Comparison of the temperature of ablation surface t_{pa} [°C], temperature inside the composite t_{int} [°C] and temperature on the rear surface of the wall t_s[°C] in sample no.2 of 6% volume share of carbon nanotubes after a period of 150 seconds of the exposition to a heat flux

The graph above (Fig. 7) indicates that the temperature of the ablation surface was approximately 1100°C, the highest temperature inside the test sample reached the value of approximately 500°C, whereas the maximum temperature on the rear surface of the wall of the composite sample equalled approximately 211°C. Basing on the measured data, it was found that on the backside of the sample wall, there was a temperature reduction of around 80%, while inside the composite it was by approximately 55%.

3. Conclusion

1. Repeatable results of research in conditions similar to an aircraft accident has been assured because construction of designed a stand for thermal resistance research of manufacturing FDR's shields meets requirements of thermal loads according to TSO-C124b and ED-112;
2. Designed and built a stand gives an much more opportunity to recognize possibility of using tested shield to improve thermal resistance of FDR's than the requirements of ED 112;
3. The universal protective shield combined with steel housing recorder allows to satisfy the FAA criteria of thermal resistance of flight data recorders, therefore it can be used as additional thermo-protection;
4. Based on conducted research, it seemed that universal protective shield combined with steel housing recorder achieved favourable parameters:
   - about 100°C lower the average final temperature of the rear surface,
   - above 55% smaller, the relative change in mass U_a.

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