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The Influence of Metal Reinforcement upon the Ablative Properties of Multi-Layered Composites

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ABSTRACT

Fibre-metal laminates combine both the properties of metal and composite materials, reinforced with fibres; first such laminates already appeared at the end of the 70s. Alongside with the emergence of spacecraft, in which the external plating heats up to temperatures even exceeding 3000°C, there was a demand for new materials, with increased thermal resistance. Moreover, high thermal resistance is also required in the construction of different protective casings for sensitive equipment, e.g. flight data recorders. In order to protect a spacecraft from massive amounts of heat This article presents research findings, aimed at determining the influence of including metal reinforcement in the form of steel sheet upon the thermal resistance of a multi-layered polymer composite. The samples were exposed to a mixture of hot gases at a temperature of over 900°C for a period of approximately 150 s. The most important parameter determined on the basis of experimental research was the temperature of the rear surface of the sample. It was observed, that the addition of metal reinforcement causes stabilization of the temperature of the back wall of the sample and a decrease in temperature of the rear wall surface of the insulating sample by approximately 2-fold increase in the ablative mass loss.

Keywords: layered composite, ablative properties, thermal protective.

INTRODUCTION

Today, sandwich composites are widely used in the construction of civil and military aircraft [1, 2]. One of these types of materials is the material for which the idea appeared in the late 1970s and consisted of combining two materials – aluminum alloys and fiber composites – into one hybrid composite to avoid most of the failures that these materials seemed to have separately. In this way, thanks to Delft University of Technology (DUT) there emerged new fibre-metal laminates (FML), which consisted of metal and polymer composite layers, placed alternately. FML combine both the properties of a metal and a composite material, reinforced with fibres.

The high tempo of development of hybrid composites, reinforced with metal, was pressured by the industrial demand, particularly by aviation industry and later by spacecraft industry for modern materials with high mechanical properties and

low mass and at the same time low exploitation costs [3, 4]. The first ever manufactured fibremetal laminate was ARALL (Aramid Reinforced Aluminium Laminate). It consists of thin, alternately laid, high-endurance layers of aluminum alloy (0.2 - 0.4 mm) and layers of aramid fibre. The next step was to create a much stiffer material by incorporating carbon fiber instead of aramid fiber (CARALL) - this material is now used in various constructions [5]. In 1990, there was another attempt to enhance ARALL, due to the employment of highly-resistant glass fibres in place of aramid fibres. This led to the creation of GLARE (Glass Reinforced Aluminium Laminate. Application of this type of material in aircraft constructions largely contributes to lowering its mass, especially in cases where fatigue tolerance and failure resistance constitute a major constructive aspect [6]. It must be noted that the existing Federal Aviation Regulations (FAR) do not refer to the requirements for the fuselage

skin plating construction and tests of its thermal resistance. They refer to the materials used inside the cabin, such as seat cladding, acoustic isolation and baggage hatch cladding. Therefore, all the previous tests of thermal resistance of fibre-metal composites which could be employed as fuselage skin plating, were conducted in accordance with the regulations for the baggage hatch cladding. The thermal resistance test of the baggage hatch cladding (FAR 25.855) assumes the influence of a flame of 930°C (the flame should produce a heat stream of 91 kW/m² in value). These requirements do not correspond with the conditions during a massive fuel fire, which may happen during a plane catastrophy (temperature of 1150°C and heat stream equaling 160 kW/m²). In table 1 there are test findings conducted by Boeing company on three GLARE material types, with the flame temperature of $1100 + 25^{\circ}C$.

The test results enable to compare GLARE characteristics with the traditional materials, such as aluminium. In case of $1.5 \div 2.0$ mm thick aluminium (compared to GLARE thickness used to conduct the experiment), the time needed for complete burning down equals about 90 sec, whereas in case of GLARE, except the damage of the layers exposed to flame, there was no flame penetration [7]. Together with the development of spacecraft, which enter the atmospheric layers of the Earth, where the external plating of the spacecraft heats up to over 3000°C, within a short time span, there was a demand on new materials with increased thermal resistance. Also during the start-up of a rocket engine on solid fuel, the insulation between the casing and the fuel is exposed to the temperature of over 2000°C and the pressure of over 7 MPa [8, 9]. High thermal resistance is also required in the construction of protective casings of flight recorders. Detailed requirements which pertain to the protection of the recorded data, introduced by FAA (Federal Aviation Administration), currently in force, are included in documents TSO C123a and C124a. The military standard MIL-STD-2124A, in this respect, are parallel with the civilian ones [10]. The protective casing of the flight data recorder or its memory element, should secure the information about the flight parameters and working of the aircraft equipment, even during the influence of a flame at the temperature of 1100°C within 60 min. Since 1990, all the flight data recorders must meet an additional requirement, which specifies the resistance of thermal shields during the influx of a heat

Table 1. Test findings of GLARE thermal resistance

Type of fibre-metal laminate	Temperature of the side unexposed to flame (°C)			
GLARE 3-2/1	After 5 min: 220			
GLARE 3-3/2	After 5 min: 160 After 10 min: 215			
GLARE 4–2/1	After 5 min: 220			

stream, at the temperature of $t = 260^{\circ}$ C and density $q(t) = 134 \text{ kW/m}^2$ by 10 hours. Increasing the surface area of the external casing, which is under the influence of a heat stream, in 1990 doubled the amount of heat that have to be absorbed by the recorder's casing. Other requirements included in C124a and ED 112 norms extended the time of the flame influence upon the recorder's casing by 60 min., which also doubled the amount of heat that should be absorbed by the casing, without damaging it. The stronger requirements have caused a four-times higher increase in the resistance requirements for the recorder's casing to a high-temperature heat stream [11, 12]. Use of traditional thermal protection materials would mean an increase in the volume of the thermal protective flight data recorder's casing by approximately 750%, which would considerably raise the mass of the whole recorder. A solution for the flight data recorder's casing seems to be the adoption of a protective layer, made with ablative materials. These materials have been in long use in aviation, rocket and space technology as well as anti-fire protection. For this reason, currently research is being made into such materials with powder additives [13–17], and fibre reinforcement [18–21] or fillings in the form of plates, aerogels or phase change materials [22-26]. An interesting paper on the use of Kevlar 49 fibres was presented by Alagar et al. [27] and Minkook et al. [28], who used aramid fibres in combination with a phenolic foam-filled honeycomb.

The ablative materials which are used in the construction of thermal shields in spacecraft, which enters the atmosphere, are a classic example of such materials. In order to protect the spacecraft against massive amounts of heat produced due to friction in the atmosphere, the ablative materials degrade through endothermal reactions (Fig. 1), while absorbing heat and blocking its transfer inside the craft [29]. The protective casings of the flight recorder are manufactured from ablative polymer composites and characterize with increased thermal protective properties (Fig. 2) and could decrease mass much more efficiently.



Fig. 1. Physical model of ablation [1]

After exceeding the temperature of 200 +/-°C, there begins thermal decomposition of the ablative material base. During this decomposition, the external stream of thermal energy is used for several endothermal processes and chemical reactions:

- depolarization of organic components of the composite,
- melting, vaporization and sublimation of easily melting powder fillers of the composite,
- reaction between components of the composite and depolarization products.

Moulding ablative thermal protective properties consists in searching for materials of high specific heat $c_p(t)$ and high density ρ and also low thermal conductivity $\lambda(t)$ (i.e. low thermal diffusivity value a(t)) [30].

Casings made with ablative polymer composites are characterized with heightened thermal protective properties. So far scientists tried to manufacture and research thermal resistance only ablative material not to taking into account the influence of the metallic material so there are lack of articles included that kind of research.

Bearing in mind the potential employment of polymer ablative composites for securing aircraft flight instruments, it was conducted research aimed at determining the influence of metal reinforcement, in the form of steel sheet, upon thermal resistance of a multi-layered composite. During the research it was used polymer ablative composite, whose composition was adopted on the basis of the analysis and comparison of the ablative properties of thermal protective parameters, which had previously been examined in the light of their usage for the manufacture of the thermal universal casing of flight data recorders.

MATERIALS AND METHOD

For the sake of the research, whose aim was to determine the influence of metal reinforcement, it was used polymer ablative composite, consisting of 14 layers of aramid fibre of 230 g/m² basis weight, 6 layers of carbon fibre of 160 g/m² basis weight, and one or two layers of steel sheet, S235, 2 mm thick. As matrix, was used Epidian 52 epoxide resin, cured in room temperature by TFF hardener, manufactured by Z. Ch. Organika S.A. in Nowa Sarzyna. The ablative properties of the resin composition were modified with a layered aluminosilicate, Bentonite Special Extra, which contained 75% calcium montmorillonite MMT (Zebiec Mining-Metal Plant located in Zebiec). The composition of the composite was adopted on the basis of an analysis and comparison of the ablative thermal protective properties of the composite that was designed to build a thermal cover of the universal protective casing of a flight



Fig. 2. Thermal resistance of selected thermal protective materials

Variant	Composition of laminates including the metal layer	
Variant no. 0	Without metal reinforcement (only ablative polymer composites)	
Variant no. 1	1 One internal, middle layer as metal reinforcement	
Variant no. 2	Two external layers as metal reinforcement	
Variant no. 3	It no. 3 One-sided external layer as metal reinforcement on the rear surface of the isolation sample wall	
Variant no. 4 One-sided external layer as metal reinforcement on the surface of the wall, exposed to flame		9.1±0.4

Table 2. Laying metal reinforcement in composite

recorder [13, 31–33]. In order to conduct the research, were prepared control panels measuring 150×200 mm, in the form of a laminate. It were prepared three laminates, which differed in the order of placing the metal reinforcement layers (Table 2) and in the Fig. 3. Moreover, to compare influence of metal, the ablative composites without metal layers were tested (variant no. 0, Tab. 2)

In case of the laminate with external metal layer, were prepared a double number of samples (variants no. 3 and 4) so that the metal reinforcement could constitute the wall surface that was exposed to a flame, as well as the rear surface of the sample.

The laminate's plates were used for cutting out samples 30×35 mm in order to examine the ablative thermal protective properties. The laminate was cut by means of a water stream on WaterJet DARDi machine.

DISCUSSION OF THE RESULTS

The ablation tests were conducted on a special test stand at Dęblin Polish Air Force University (fig. 4), with the following assumptions:

- time of trial $\tau = 150$ sec;
- the thermophysical characteristics of the materials are constant;

- the heat stream does not change during the ablation test;
- the ablation surface is an isothermal surface of the ablation front;
- we disregarded the heat transfer between the environment and the external surface;
- it was accepted that heat transfer is mainly through conduction without degradation of the ablation part.

The simplest energy balance equation can be written as [18, 34]

$$q_{K} = q_{e} - q_{rw} - q_{bL} - q_{w} \alpha ty = s_{l}(t)$$
(1)

where: $q_{K} = -K \frac{\partial T}{\partial y}$ – thermal energy transferred from the environment to the ablative surface by conduction;

> $q_e = h(T_e - T_0)$ – thermal energy transferred from the environment to the ablation surface by convection,

> $q_{rw} = \varepsilon \sigma T_w^4$ – thermal energy radiated by the ablative surface;

 $q_w = C_p I(T_w - T_p)$ – thermal energy released from the surface heated to the temperature T_w , to the environment by convection;

 $q_{bL} = \gamma_{bL} GI^{-1}(q_e - q_w)$ – transpiration cooling.



Fig. 3. Picture of variants of the prepared fibre-metal composite



Fig. 4. Stand for testing ablative thermal protective properties: 1 - sample, 2 - ablating gun, 3 - pyrometer, 4 - temperature measuring instrument, 5 - thermocouple on the back of the sample

Because composite is separated from flame by metallic layer it is exposed to high temperature but there are not heated directly by flame. To describe mathematical model illustrating the thermal decomposition and ablation of the composite material it can be used the following transient partial differential heat conduction equation:

$$\rho C \frac{\partial T}{\partial t} \left(K \frac{\partial T}{\partial y} \right) + C_g \dot{m_g} \frac{\partial T}{\partial y} + \left(h_g - \bar{h} \right) \frac{\partial \rho}{\partial t} \quad (2)$$

where: $(h_g - \bar{h}) = \Delta H_p; (h_g - \bar{h}) \frac{\partial \rho}{\partial t} =$ = $Q_p(T); \bar{h} = \frac{\rho_1 h_1 - \rho_3 h_3}{\rho_1 - \rho_3}.$

$$\rho C \frac{\partial T}{\partial t} = \frac{K}{\left(L_0 - s_1(t)\right)^2} \frac{\partial^2 T}{\partial \xi^2} + \frac{1}{\left(L_0 - s_1(t)\right)^2} \left(\frac{\partial T}{\partial \xi}\right)^2 \frac{\partial K}{\partial T} + \frac{\dot{m}_g C_g + \rho C \left(\frac{\partial s_1(t)}{\partial t}\right) (1 - \xi)}{(L_0 - s_1(t))} \frac{\partial T}{\partial \xi} + Q_p(T)$$
(3)

The ablation equation involves three zones and two moving boundaries (Combustion products moving boundary and rear surface of the insulating boundary). Thermal conductivity, K, specific heat, C, and density, ρ , all change by thermal degradation of composite, and are necessary to solve this equation.

The temperature of the surface exposed to the heat flux (Fig. 5) was measured using a pyrometer Optris CTlaser 3M.

The main parameter for the suitability assessment of the produced multi-layered polymer composites for the manufacture of thermal protective casings was the maximum temperature of the rear surface of the sample t_s [14].

In addition, based on the analyzes presented in [35–37], was calculated the ablative mass loss (U_a) for each sample and the speed of ablation (v_a) . The calculations ignored steel in the sample composition. For the sake of comparison, in the Table 3 were put the results of the multi-layered



Fig. 5. Average temperature on the surface exposed on flame

composite, of the same structure, however without metal reinforcement (variant no. 0 sample).

On the chart of an average temperature layout of the rear surface, for all laminate variants, it can be observed that the curves for each variant are similar (Fig. 6). Differences emerge only for variant no. 0. On the chart of the temperature of the sample's rear surface, there is only a slight difference in temperatures towards the end of the exposition to the heat stream. Fig. 7 lists all the obtained parameters.

All the four sample variants showed a very similar average ablative loss of mass U_a , approximately 34%, as well as similar speed of ablation. Whereas ablative mass loss is basically a term for thermal stability parameter, it seemed that layer of metal caused more extensive changes in the area of the polymer composite in the form of physical transformations and chemical reactions of the native material. This may be due to the secondary heat flux, the source of which has become the metal layer.

Due to the presence of two layers of steel, variant 2 had the largest initial mass. The mass of the remaining samples constituted merely 57% of variant 2 samples' mass. The presence of steel had favourable effect on the decrease in temperature of the rear wall sample, compared with the samples with metal reinforcement, laid inside the sample (variant no. 1). It caused both a decrease in the final temperature as well as its stabilization during the test and stabilization at a constant level of about 40 °C already after 60 s. As in the case ablative loss of mass, the reason may be the heat given off by the metal, which as a material with better thermal conductivity, after accumulating thermal energy, became a secondary source of heat radiated towards the ablation material.

It is planned to carry out to conduct numerical research using experimental research findings as was done in publication [38] where a simplified numerical analysis of the problem was carried out for a similarly conducted experiment (details can be found in paper no. 38) and an exemplary diagram is provided below Fig. 8. The authors do not analyze these results in detail (this was described in paper no. 38) but only presented as a potential addition to further work in the field of numerical studies of FML composites.

CONCLUSIONS

The samples in which the steel layers were symmetrically arranged outside or in the middle, allowed to maintain the lowest temperature on the rear wall, however, due to the need to minimize the weight, the use of two steel layers in the light of the obtained results turns out to be pointless.

Sample variant	Ablative loss of weight without steel $U_a(\%)$	Speed of ablation without steel v _a (µm/s)	Maximum temperature of the rear surface of the insulating wall $t_{\rm smax}$ (°C)
0	17.0	12.6	75
1	35.8	19.2	37
2	31.5	19.0	38
3	35.5	19.0	39
4	35.0	16.9	40

Table 3. Ablation properties of examined materials



Fig. 6. Average temperature layout on the rear surface of the sample wall during tests



Fig. 7. Ablative thermal protective properties of examined materials



Fig. 8. Numerical and experimental results t [38]

The addition of metal reinforcement causes a decrease in temperature of the rear wall surface of the sample by approximately 45%.

The use of a steel layer causes stabilization of the temperature of the back wall of the sample after some time and maintaining this temperature at the same level until the end of the test.

The use of a metallic layer causes an approximately 2-fold increase in the ablative mass loss which is the smallest for the variant in which the outer layers were metal reinforcement which is due to the lack of direct impact of the heat flux on the composite part of heat transfer is mainly through conduction without degradation of the ablation part.

A slight increase in the ablation rate correlation with the increased loss of ablation mass, may indicate an acceleration of the composite degradation process and a prospective shortening of the thermal protection time.

The obtained characteristic properties of individual variants of the laminate may change in the case of testing elements with larger dimensions. In order to examine the test results, tests should be performed using ready-made structural elements, e.g. an aircraft recorder housing with a thermal shield made of an ablative composite, what is planned in the further part of the scientific research as well as placing thermocouples on the metal surface in order to check its use as an ablation barrier of the composite. In addition, it is planned to determine more temperature readings between different layers of the composite for the purpose of a broader analysis of the internal temperature distribution (not only the back surface).

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