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COMPOSITES THEORY AND PRACTICE

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TENSILE STRENGTH OF TITANIUM/FIBRE REINFORCED POLYMERS AT DIFFERENT TEMPERATURE VALUES

The study describes the results of tensile strength tests of hybrid laminates composed of thin titanium layers and glass and carbon fibre reinforced polymer layers. The tests were conducted at –120, RT (23°C) and 85°C. The tests allowed the basic mechanical properties to be determined, including: tensile strength, Young's modulus and strain at failure. The tests proved that as the temperature decreases, the strength of titanium/glass fibre reinforced polymers increases by 21 to 26% depending on the configuration, while the strength of titanium/carbon fibre reinforced polymers decreases by 6 to 8%. The Young's modulus values for all the tested systems increase by 3 to 7% as the temperature drops. A different tendency was observed regarding the strain at failure which decreases by 1 to 11% as the temperature drops. The tensile strength test results for the increased temperature (85°C) differ only slightly from those obtained at room temperature. The macroscopic analysis of the failed specimens revealed the existence of characteristic, prevailing forms of failure, namely breaking fibres, matrix cracking, including delamination and permanent deformation of the titanium layers.

Keywords: **fibre metal laminates, tensile strength, temperature, titanium**

WYTRZYMAŁOŚĆ NA ROZCIĄGANIE LAMINATÓW TYTAN/KOMPOZYT WŁÓKNISTY PRZY RÓŻNYCH WARTOŚCIACH TEMPERATURY

Przedstawiono wyniki badań wytrzymałości na rozciąganie laminatów hybrydowych składających się z cienkich warstw tytanowych oraz warstw kompozytów polimerowych wzmacnianych włóknami szklanymi oraz węglowymi. Próby przeprowadzono w temperaturze –120, RT (23°C) oraz 85°C. Podczas badań wyznaczono podstawowe właściwości mechaniczne, takie jak: wytrzymałość na rozciąganie, moduł Younga oraz odkształcenie przy zniszczeniu. Przeprowadzone próby wykazały, że wraz ze spadkiem temperatury wytrzymałość laminatów wzmacnianych włóknem szklanym wzrasta od 21 do 26% w zależności od układu, natomiast wytrzymałość laminatów wzmacnianych włóknem węglowym spada od 6 do 8% wraz ze spadkiem temperatury. Wartości modułu Younga dla wszystkich badanych układów wzrastają od 3 do 7% wraz ze spadkiem temperatury badania. Odmienną tendencję odnotowano dla wartości odkształcenia przy zniszczeniu, która zmniejsza się wraz ze spadkiem temperatury od 1 do 11%. Wyniki badań wytrzymałości na rozciąganie uzyskane dla temperatury podwyższonej (85°C) nie różnią się znacząco od wyników uzyskanych w temperaturze odniesienia (RT). Makroskopowa analiza zniszczonych próbek wykazała występowanie charakterystycznych, dominujących form zniszczenia w postaci zerwania włókien, pękania osnowy, w tym delaminacji oraz trwałych deformacji blach tytanowych.

Słowa kluczowe: **laminaty metalowo-włókniste, wytrzymałość na rozciąganie, temperatura, tytan**

INTRODUCTION

Fibre metal laminates (FMLs) belong to hybrid composite materials [1]. They consist of alternating thin metal layers and layers of fibre reinforced polymers connected together. These materials are characterised by a high strength to density ratio, impact resistance and high fatigue strength [2-4]. FMLs are used mainly in the aircraft industry [5]. The area of application of FMLs shows that there is a wide spectrum of environmental aspects potentially influencing the properties of the above mentioned materials. Due to their complex structure, FMLs can have different specifications of mechanical response and of the failure process, both in the case of monolithic metals and polymer composites [6, 7]. This is why there are many studies currently examining the behaviour of FMLs in different environmental conditions and under different mechanical loads [8-10]. One might also observe how researchers' attention is now increasingly focused on numerical modelling in FMLs, considering failure and damage development processes as well [11, 12].

The issue of temperature fluctuations is connected not only with differences in dimensions but also with changes in the physical and mechanical properties of the materials used as FML components. Metals with a regular, face-centred cubic crystal system remain plastic and resistant to cold cracking even at lower temperatures [13]. Aluminium alloys, widely used in FMLs, also belong to this group. One exception is pure titanium which, although it has a hexagonal crystal system, retains its beneficial mechanical properties at lower temperatures [14]. The upper temperature limit for the use of individual alloys is determined by the phase balance scope which, in the case of titanium, is much greater than the potential of thermoset resin matrix composites [15, 16].

The properties and behaviour of titanium-composite laminate components are examined in a wide temperature range. Paper [17] and [18] present the results of studies on employing a glass-epoxy composite to produce liquefied gas containers. In work [17] fatigue tests carried out at 4, 77 K and at room temperature reveal that the threshold of energy released during the development of delamination is higher at lower temperatures, whilst its growth rate is lower. The results of the study described in [18], on interlaminar shear strength (ILSS) determined by the short-beam method prove that laminate strength increases at low temperatures. The authors point out that friction on the component interface is one of the reasons for the increased strength.

Carbon fibre reinforced polymers, examined in [19], show a tendency to accumulate microscopic cracks when exposed to multiple thermal cycles reaching cryogenic ranges. Work [20] reveals that the strength of the carbon-epoxy composite decreases, probably as a result of the increased stress intensity, and that the density of microscopic cracks increases within the composite. The reason behind the reduced strength of the carbon-epoxy composite is most likely the considerable increase in brittleness of carbon fibres in this temperature range, and consequently, a higher susceptibility to rapidly developing microscopic cracks within the laminate. Work [21] presents the issue of epoxy resin resistance to cold cracking, examined at cryogenic temperatures. The authors argue that the resistance depends on the strength of the molecular structure and the possible stress relaxation on the face of the microscopic cracks. In order to increase the total strength of the molecular structure, it is vital to achieve a higher curing density. Despite the fact that when it comes to epoxy resins the resistance to cold cracking is often higher at low temperatures due to the decreased potential energy of chemical bonds (the potential energy of the bonds appears in negative numbers), the increase in internal stresses stemming from the differences of the coefficient of thermal expansion (CTE) may lead to microscopic cracks within the matrix and to weakening of the composite [22-24].

Titanium/fibre reinforced polymer laminates are relatively new materials [25], therefore, in spite of intensive work in recent years [26-29], their characteristics and properties are not quite well known. Thus far, studies on the influence of temperature on titanium/fibre reinforced polymers have been conducted on a moderate scale. Paper [30] proves the high thermal stability of titanium/carbon fibre reinforced polymers with a PEEK thermoplastic matrix. The paper also presents the results of strength tests of the laminates at RT and at 220°C and durability tests in thermal cycles ranging from –65 to 135°C. It was observed that the laminate strength at the elevated temperature is lower by 52 to 60% but the test of 1000 thermal cycles did not cause any reduction in the durability. The authors of paper [31] observed that the conditioning of samples made of titanium/carbon fibre reinforced polymers at 70°C and in the environment of salt water induced a noticeable decrease in tensile strength and ILSS. Failure at the metal-composite interface was identified as the main failure mechanism after the conditioning.

The aim of the paper is to analyse the impact of temperature on the tensile strength of selected hybrid laminates. The study was conducted on titanium/fibre reinforced polymers with different types of reinforcement (glass and carbon fibres) and different configurations of fibre arrangement. The aim of the paper was also to identify the main laminate failure mechanisms at different temperatures.

METHODOLOGY

Materials and manufacturing

The study used the titanium/glass-epoxy composite (TiG) and titanium/carbon-epoxy composite (TiC). The FMLs were made of titanium alloys (Grade 2) with thicknesses of 0.3 and 0.5 mm, which were initially prepared through fluoride-phosphate treatment and the 3M EC3924B primer coating. The composite layers were made of a pre-impregnated tape containing type R glass fibres and M12 epoxy resin (Hexcel USA) and of pre-impregnated tape containing high-strength T700GC carbon fibres and M21 epoxy resin (Hexcel USA). The laminates were manufactured using the autoclave method (Scholz Maschinenbau, Germany). The laminate curing process was carried out employing the following parameters: heating rate of 2°C/min to 135°C (M12 resin system), 180°C (M21 resin system) and curing at this temperature for a period of 2 h. The pressure and vacuum used were 0.45 and 0.08 MPa, respectively. After curing, the laminates were inspected by the ultrasonic method (Olympus Ominscan MX2). The manufactured panels were cut into specimens using a water-cooled abrasive disc cutter. The configurations of the tested laminates are shown in Table 1.

The research used rectangular-shaped specimens, illustrated in Figure 1. Each series of tests used 6 specimens. The direction [0] of the test samples corresponded to the rolling direction.

TABLE 1. **Configuration of titanium/fibre reinforced polymers**
TABELA 1. **Konfiguracja laminatów tytan/kompozyt włóknisty**

Laminate	Material		Lay-up configuration
	Metal	Composite	
TiG 1	Ti 0.3 mm	R-glass/M12 epoxy	[2/1] (Ti/0/Ti)
TiG 2	Ti 0.5 mm	R-glass/M12 epoxy	[2/1] (Ti/0/Ti)
TiG 3	Ti 0.5 mm	R-glass/M12 epoxy	[2/1] (Ti/0/90/Ti)
TiC 1	Ti 0.3 mm	T700GC/ M21epoxy	[2/1] (Ti/0/0/Ti)
TiC 2	Ti 0.5 mm	T700GC/ M21epoxy	[2/1] (Ti/0/0/Ti)
TiC 3	Ti 0.5 mm	T700GC/ M21epoxy	[2/1] (Ti/0/0/90/90/Ti)

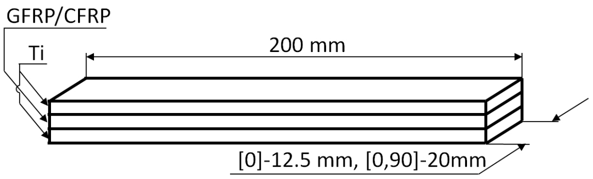

Fig. 1. Schematic diagram of construction of samples used for testing
Rys. 1. Schemat budowy próbek wykorzystanych do badań

Tensile test

The samples were submitted to static tension on a station equipped with a climatic test chamber, in accordance with the ASTM D 3039/D 3039M-00 standard. The study was conducted on a material strength testing station consisting of an MTS 322.31 material stress testing machine equipped with a head measuring strength up to 250 kN, model MTS 661.22D-01, calibrated in the 50 kN sub-band. An MTS 634.11F - 21 extensometer was used to measure the longitudinal strain. The experiment was controlled using a Flextest 40 controller with MTS MPT control and recording software.

The specimens were placed in hydraulic side acting grips. Those tests which were carried out at a temperature other than RT began with a few minutes' delay in order to stabilise the temperature of the specimen inside the chamber. The test was conducted through controlled movement of a servo mechanism at a constant speed of 2 mm/min until the internal composite laminate layer broke. The signals of strength measurement, servomotor movement and from the extensometer were recorded at the frequency of 10 Hz.

RESULTS

Tensile strength

Figure 2 illustrates the stress-strain curves for the TiG (Fig. 2a) and (Fig. 2b) TiC laminates. Analysis of the curves indicated that the responses of the laminates obtained during the tensile test have a complex, bi-linear character. All the examined systems are characterised by an initial linear increase both in stress and in strain, followed by a change in inclination of the curves, connected with exceeding the yield limit in the metal layers. The further increase in stress is mainly due to the elastic character of the composite material response, whose stiffness does not change until the moment of failure. The TiG laminates (Fig. 2a) are characterised by a greater decrease in stiffness after the apparent yield limit is exceeded, compared to the TiC laminates (Fig. 2b), while the elongation at failure of the TiG laminates is noticeably greater than in the case of the TiC laminates. This results from the different characteristics of materials used for the composite layers (glass and carbon fibres), because both the TiG and TiC laminates were made of identical titanium alloys. The glass-epoxy composite is characterised by a considerably smaller stiffness and strength compared to the carbon-epoxy composite, though having a greater strain range.

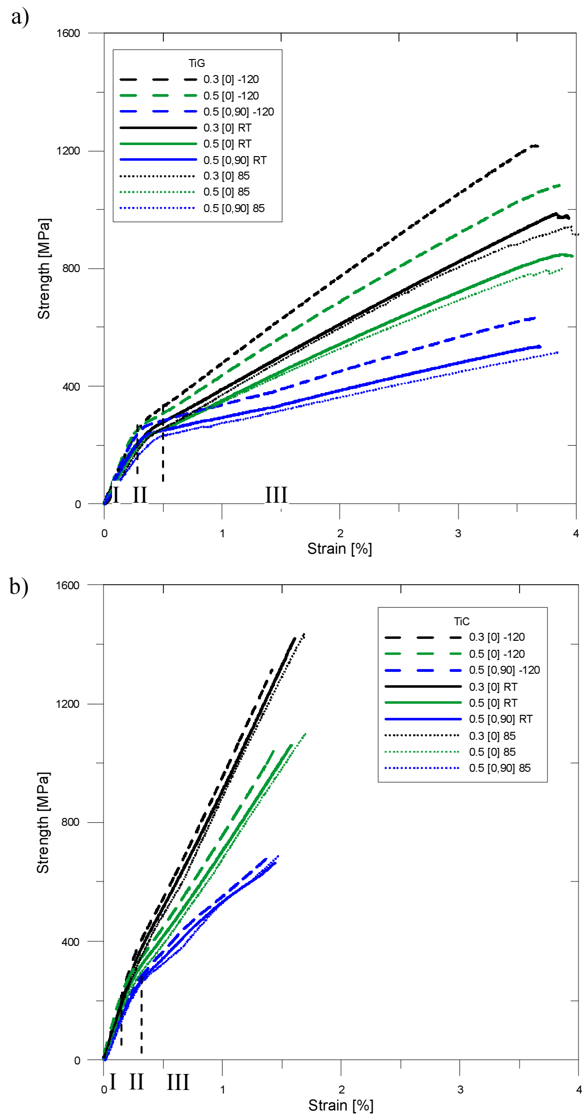

Fig. 2. Stress-strain curves for laminates: a) TiG, b) TiC at –120°C, RT and 85°C
Rys.2. Krzywe naprężenie-odkształcenia dla laminatów: a) TiG, b) TiC w temperaturze –120°C, RT i 85°C

Careful analysis of the curves in particular groups suggests that their location and shape depend on the configuration of the laminate layers and the test temperature. Depending on the configuration of the layers, the laminates examined at RT are characterised by a different initial stiffness (Stage I) and after the conventional yield limit (Stage III). The stiffness for individual systems is determined by the stiffness of the components in particular ranges and depends on their volumetric proportion in the laminate structure. The curves obtained at −120°C for all the TiG systems in the stress-elongation system considerably differ from the reference curves (RT) and from the curves obtained at 85°C. As regards the TiC laminates, the individual curves obtained at different temperatures are rather similar to each other, compared to the TiG laminates. What is more, the laminates examined at −120°C have the steepest characteristics in reference to the horizontal axis in the whole spectrum of the study (Stages I-III).

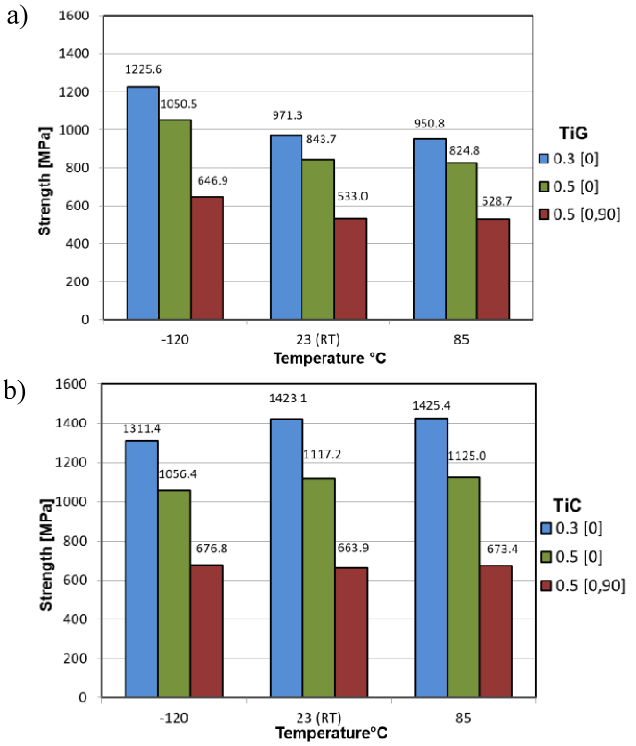

Fig. 3. Tensile strength of laminates: a) TiG, b) TiC as a function of temperature

Rys. 3. Wytrzymałość na rozciąganie laminatów: a) TiG, b) TiC w funkcji temperatury

Figure 3 illustrates a comparison of the average values obtained in the tensile strength test of titanium-fibre reinforced polymers. The test allowed the authors to determine: the tensile strength, Young's modulus E and strain at failure ε at −120°C, RT and 85°C. Figure 3a shows the average tensile strength of the TiG laminates, while Figure 3b shows the tensile strength of the TiC laminates.

The obtained values indicate that the tensile strength of all the examined laminates depends on the investigation temperature. It was noted that the strength of the TiG laminates increases by 21 to 26% on average for individual systems as the temperature drops, while the strength of the TiC laminates decreases by 6 to 8% as the temperature drops, except for TiC 05 [0, 90], whose strength at −120°C is slightly greater than at RT (0.1%). The increase in tensile strength of the TiG laminates along with a decrease in temperature corresponds to the increase in tensile strength of the laminate components - the titanium and GFRP composite at a decreased temperature, described in [14, 18], which may be the most plausible reason for the observed tendency. The key contributor to the decreasing strength of TiC laminates at decreased temperatures is most likely the increase in internal stress and the simultaneous decrease in the carbon fibre reinforced composite layer strength, whose failure ended the tests. The average tensile strength at 85°C of all the tested laminates differs only slightly compared to the reference test and does not exceed 3%.

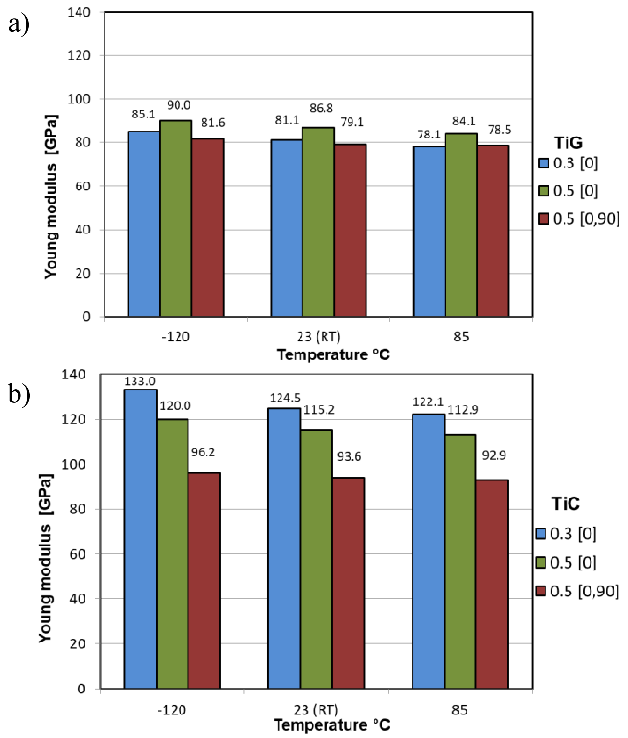

Fig. 4. Average Young's modulus for laminates: a) TiG, b) TiC as a function of temperature

Rys. 4. Wartość średnia modułu Younga dla laminatów: a) TiG, b) TiC w funkcji temperatury

The average Young's modulus illustrated in Figure 4 obtained during the tests indicate a link between temperature and the laminate structure. All the tested configurations showed an increase in stiffness at the lowered temperature (−120°C) and a slight decrease in stiffness at the raised temperature (85°C). The maximum increase in the average stiffness obtained at the lowered temperature was 5% for the TiG laminates and 7% for the TiC laminates respectively. In both cases the maximum was obtained for the systems with the greatest volumetric proportion of composite, i.e. 0.3 [0]. Similar to the tensile strength, the stiffness obtained at the increased temperature (85°C) differs only slightly from that obtained in the reference conditions. This is probably caused by the smaller difference in temperature between the reference conditions and the raised temperature than between the lowered temperature and reference conditions. Moreover, in the above mentioned ranges no changes occur in the structure of the laminate components to significantly impact the obtained results.

The average strain at failure, defined as the moment of loss of consistency and failure of the composite layer, illustrated in Figure 5, is much higher for the TiG than TiC laminates for all the systems and temperatures. In the case of this parameter, the properties and configuration of the composite layers play a key role, which confirms the previous observations.

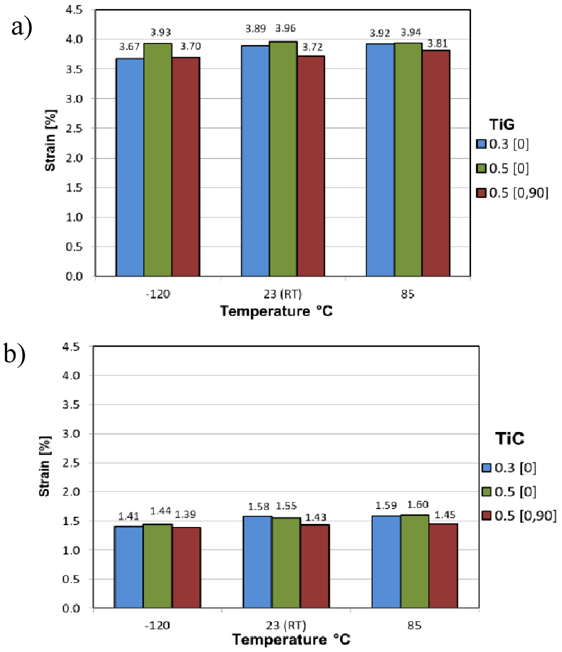

Fig. 5. Average strain at failure for laminates: a) TiG, b) TiC as a function of temperature

Rys. 5. Wartość średnia odkształcenia przy zniszczeniu dla laminatów: a) TiG, b) TiC w funkcji temperatury

A thorough analysis of strain at failure indicates that the examined materials show a decrease in strain at failure as the temperature drops both in the TiG and TiC group, reaching 8% for TiG and 11% for TiC in the 0.3 [0] system in both cases. As in the case of tensile strength and stiffness, no significant change in strain at failure was observed at the raised temperature compared to the reference temperature.

Failure analysis

Figure 6 depicts the comparison of representative specimens revealing the main forms of failure achieved during the tensile strength test.

The conducted macroscopic observations indicated the existence of composite layer failure in the form of broken fibres and matrix cracks, including delamination (see Fig. 5). No fractures were observed within the titanium layers, however, permanent deformation occurred, confirming that the yield limit of the metal was exceeded before the composite layer broke, which was identified in the stress-strain curves (Fig. 2a,b). However, considerable strain of the metal layers perpendicular to the plane of the layers (Fig. 6e, j) may indicate that its underlying cause was the process of a rapid release of energy during the failure of the composite layers.

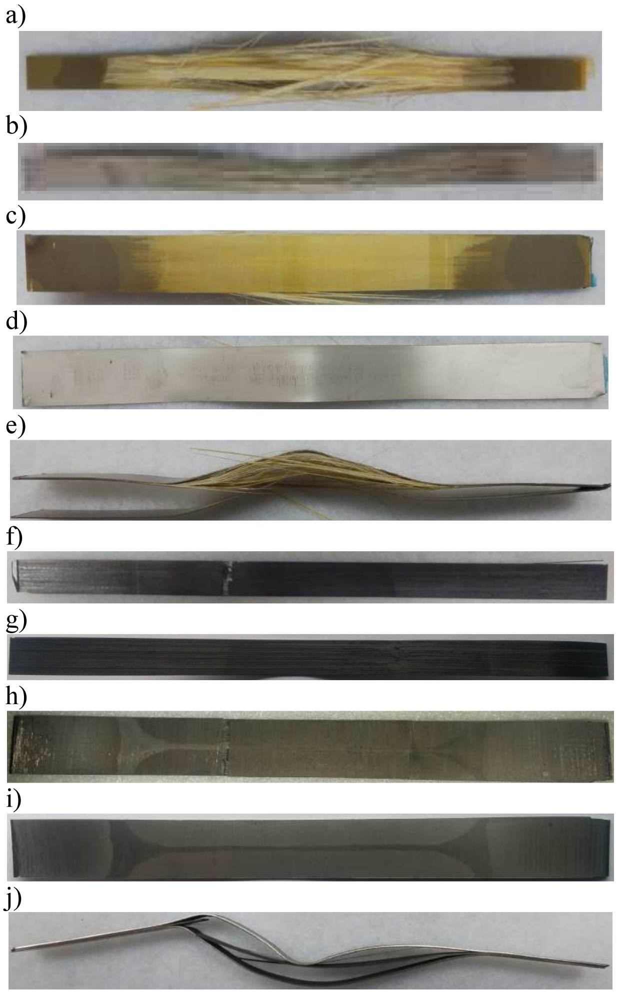

Fig. 6. Specimens after tensile strength test: a-e) TiG, f-j) TiC

Rys. 6. Próbki po badaniu wytrzymałości na rozciąganie: a-e) TiG, f-j) TiC

The failure observed within the composite layers appears in two predominant forms: broken fibres (Fig. 6a,f) and cracks in the matrix located between individual fibres (Fig. 6c,h). When it comes to the composite layers placed parallel to the breaking force, the breaking of fibres is the predominant failure mechanism. In the case of such layers, matrix cracking is the only accompanying secondary phenomenon which can be observed clearly especially for glass fibres, most likely having smaller adhesion to the matrix compared to carbon fibres. The layers perpendicular to the breaking force reveal only the existence of cracks within the matrix, due to their low strength regarding the fibres and the fact that in such a configuration the fibres cannot transfer loads effectively.

Another observed form of failure is delaminations both at the metal-composite interface and between composite layers placed perpendicular towards each other (Fig. 6 a-j). Delaminations at the metal-composite interface are vast and encircle the whole tested area of the specimen, except for the grips. Their extent may be connected with an insufficient strength of the bond at the metal-composite interface, despite the anodising process and the use of a primer.

CONCLUSIONS

The study presents the results of tensile strength tests conducted on hybrid laminates consisting of thin titanium layers and glass and carbon fibre reinforced polymer composite layers. The tests were carried out at the temperatures of −120, RT, and 85°C.

Particular parameters determined in the tensile strength test depend on the temperature of the test. A decrease in temperature causes an increase in stiffness and a subsequent decrease in strain at failure for all the tested laminates. Titanium/glass-epoxy reinforced laminates are characterised by a higher tensile strength at lower temperatures, while titanium/carbon-epoxy reinforced laminates obtained lower values than at the reference temperature.

The analysis of the failed specimens revealed the existence of characteristic, prevailing forms of failure for all the specimens, namely breaking fibres, cracking matrix, including delamination and permanent strain of the titanium sheets.

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