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COMPOSITES

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ANALYSIS OF DISCONTINUITIES EFFECT IN RELATION TO ATTENUATION LEVEL IN GLASS FIBRE REINFORCED POLYMER COMPOSITES

In recent years, composite structures have become very popular for different applications, predominantly in the aerospace industry. Their mechanical properties provide very useful materials in aviation as a primary structure. It is well known that glass/epoxy polymer composite is characterised by its lightweight and corrosion resistance in comparison to traditional materials. The main aim is to guarantee durability and safety during the manufacturing process in the case of the possible appearance of structural defects. Porosity and delamination detection is a very important factor in solving the problem of quality. One of the basic non-destructive testing methods of detecting discontinuities in structures used in aviation is ultrasonic inspection with C-scan images. Their objective is to analyse the composite structures particularly for quality inspection in aviation. This paper presents research about the relationship between the attenuation level of an NDT technique (C-scan) and void content in glass/epoxy composites. An analysis of the microstructure and characteristics of discontinuities are presented and discussed. The observations have given the results of three distinguished types of microstructures depending on the attenuation level in ultrasonic testing. The level of void content for this specific type of composite was determined from 2% to 5% and this can be classified as a medium quality composite structure. A linear dependence was found between the attenuation level in ultrasonic inspection and the percentage of voids content in a glass fiber reinforced polymer composite. The correlation between ultrasonic inspection and the analysis of microstructure is a useful method in composite structures engineering.

Keywords: glass/epoxy composites, microstructure, voids, ultrasonic inspection

ANALIZA WPŁYWU NIECIĄGŁOŚCI W ODNIESIENIU DO POZIOMU TŁUMIENIA W KOMPOZYTACH POLIMEROWYCH WZMACNIANYCH WŁÓKNEM SZKLANYM

Struktury kompozytowe w ostatnich latach stanowią bardzo popularne rozwiązania w odniesieniu do różnych zastosowań, w szczególności w lotnictwie. Dobre właściwości mechaniczne predysponują te materiały na odpowiedzialne części struktur. Kompozyty polimerowe wzmacniane włóknem szklanym charakteryzują się niską gęstością i odpornością korozyjną w porównaniu do tradycyjnych materiałów. Głównym zadaniem podczas ich wytwarzania jest zagwarantowanie trwałości i bezpieczeństwa w procesie produkcji ze względu na możliwość wystąpienia defektów w strukturze. Wykrywanie porowatości i delaminacji stanowi istotny czynnik w rozwiązywaniu problemu jakości. Defektoskopia ultradźwiękowa jest jedną z podstawowych metod nieniszczących do wykrywania nieciągłości, szczególnie w strukturach stosowanych w lotnictwie. W artykule przedstawiono badania wpływu poziomu nieciągłości na poziom tłumienia metodą obrazowania C-scan w kompozytach polimerowych wzmacnianych włóknem szklanym. Zaprezentowano analizę mikrostruktury oraz charakterystykę nieciągłości. Wyróżnione zostały trzy typy mikrostruktury w zależności od poziomu tłumienia. Poziom porowatości dla badanej struktury kompozytowej został określony w zakresie od 2% do 5%, klasyfikując tę strukturę jako średniej jakości. Wykazano liniową zależność pomiędzy poziomem porowatości a procentowym udziałem porowatości w kompozycie polimerowym wzmacnianym włóknem szklanym. Korelacja pomiędzy badaniami ultradźwiękowymi i analizą mikrostruktury stanowi pomocną metodę w inżynierii struktur kompozytowych.

Słowa kluczowe: polimerowe materiały kompozytowe wzmacniane włóknami szklanymi, mikrostruktura, porowatość, badania ultradźwiękowe

INTRODUCTION

In recent years, composite structures have become very popular materials for different applications, predominantly in the aerospace industry. Their large spectrum of properties like strength and stiffness-to-weight ratios, fatigue characteristics and corrosion resistance comprise several extremely distinctive advantages for application in lightweight primary structures. One of the main and significant purposes is to guarantee the durability and safety of the structures [1]. Therefore the problem of quality in the composite structure designing and manufacturing process represents a very important issue.

Non-destructive testing (NDT) is the basic method used in the quality inspection of the composite structures in aircraft. Many NDT techniques such as eddy currents, magnetic, liquid penetrate inspection and ultrasonic etc. are used for detecting voids, delaminations, disbonds and other defects in composite structures during production inspection or in a laboratory [1]. Among these methods, ultrasonic inspection with C-scan images are the basic testing method to analyse composite structures particularly for quality inspection in aviation structures [2]. Decreasing or complete elimination of structural defects such as: porosity, voids and delaminations is a very important factor in solving the problem of declined quality.

The paper presents the results of studies of the relationship between the attenuation level of the NDT technique (C-scan) and void content in glass/epoxy composites. An analysis of the composite microstructure and characteristics of discontinuities are presented and discussed.

EXPERIMENTAL PROCEDURE

The subject of examination was a glass/epoxy composite. It was fabricated from: 91111 and 92146 (Interglas), EBX 92140 (Cotech), 91240 technological woven fabrics and Epidian 6 thermosetting epoxy resin. It was made in a hand lay-up method with the following cure parameters: 60°C for 6 h, 100°C for 1 h, 120°C for 1h and 150°C for 5 h with a pressure of 0.05 MPa. The specimen size was approximately 300mm (length)×300 mm (width)×10mm (thickness).

Examinations of the attenuation levels were carried out using the through-transmission technique (TT C-scan). The sending-receiving system of the TT C-scan had the dynamics of the Y trajectory up to 90 dB and a tension amplitude up to 1000V. The whole plate was inspected using an ultrasonic failure detector that generated a quantized C-scan record with a working frequency 1 MHz automatic system. A colour for each attenuation level was generated after scanning for an actual size map of the plate. This feature was used to identify the areas of a constant porosity level [3].

The plate was divided into cube-shaped samples (cross-sections) with dimensions equal to 10/10/10 mm cut out from various areas with a different level of attenuation from 9 to 30 dB with a 1 dB degree step. Figure 1 shows a photograph of the studied plate with numerically marked samples. Then metallographic specimens were prepared.

A microstructure image analysis and voids analysis were performed by using an optical microscope (Nikon Eclipse MA200). The same samples were analyzed from three different sides. A characteristic of discontinuities (voids) was taken in a 50x scale with the aid of panoramic microscopy photographs (cross-sections). An image analysis was performed by using software for the computer analysis of microstructures (Image Pro Plus). The porosity occurrence and the area, shape and quantity of the discontinuities in the composite structure were estimated. The volume content of the voids, their location and geometrical parameters were defined in the analysis of the images.

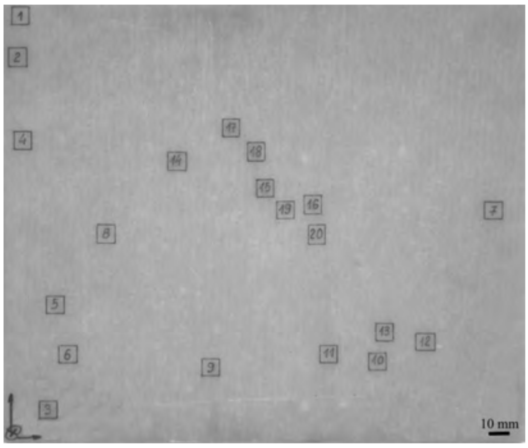

Fig.1. Photograph of plate with marked areas of sampling according to changeable attenuation level from 9 to 30 dB

Rys.1. Płyta kompozytowa z zaznaczonymi obszarami, zgodnie ze zmiennym poziomem tłumienia w zakresie od 9 do 30 dB

RESULTS AND DISCUSSION

Figure 2 shows a TT C-scan map of the examined composite plate. The distribution and location of the assumed defects can be observed. The dark area is associated with an attenuation level above 20 dB. The area of porosity is about 15%.

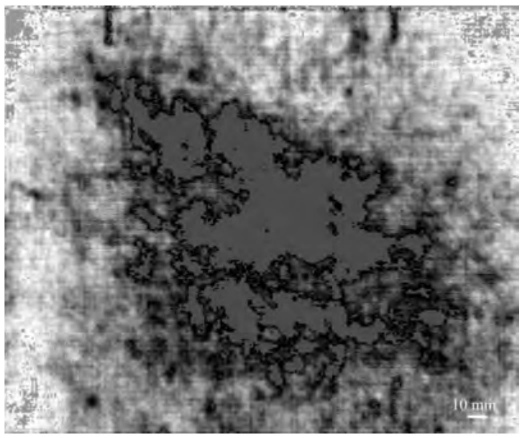

Fig. 2. C-scan images of studied glass/epoxy laminate

Rys. 2. Obrazowanie C-scan laminatu szklano-epoksydowego

Figure 3 shows typical microstructures of a glass/epoxy composite for different attenuation levels. Three types of structures depending on attenuation levels were observed: I (from 9.56 to 16.18 dB), II (from 17.2 to 23.26 dB), III (from 24.29 to 30.13 dB).

In the case of the first type of microstructure (attenuation level from 9.56 to 16.18 dB), the small voids are essential in the dominant category that is shown in Figure 3, where the cross-section corresponds to a 11.26 dB level of attenuation. The voids have irregular near-circular shapes. Perpendicular and longitu-

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dinal yarns are well visible. Fiber orientation and fiber clustering for the planar and through-the-thickness views are the same in all microstructures. The second type of voids are distinguished as ellipsoidal voids between different fiber rows and in this case the attenuation level is from 17.2 to 23.26. The microstructure in Figure 3b corresponds to 17.2 dB. In comparison to Figure 3 a and b, the third microstructure corresponds to 27.2 dB and has got cigar-shaped and ellipsoidal voids but first of all it has a few delaminations. It is a representative of the type in the attenuation range from 24.29 to 30.13 dB. These voids are concentrated in the centre part of the cross-section in the samples, only between plies. Delaminations encounter resistance which has an influence on their shapes. Regardless of the samples cross-section, the cigar-shaped voids were present on the whole area of the cutting samples.

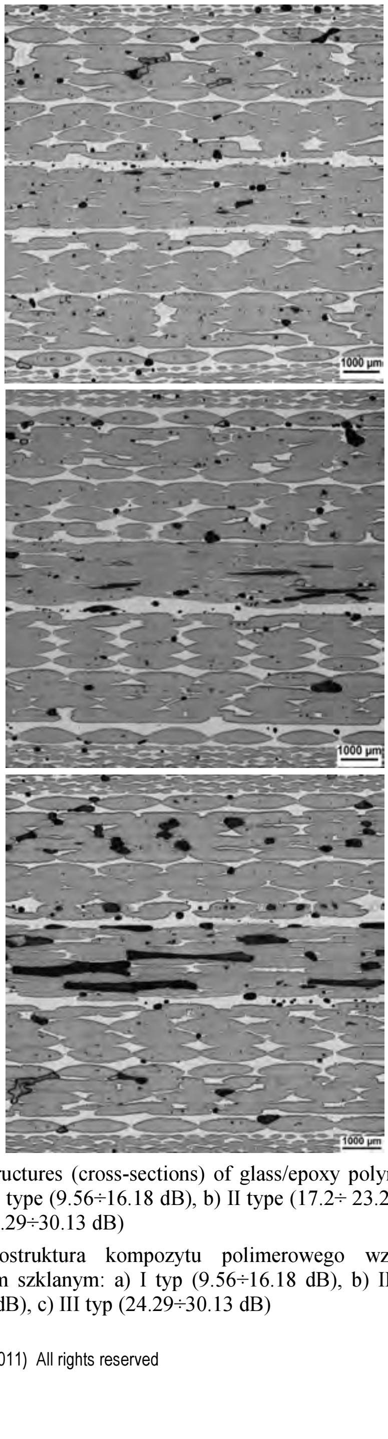
a)
b)
c)


Fig. 3. Microstructures (cross-sections) of glass/epoxy polymer composite: a) I type (9.56÷16.18 dB), b) II type (17.2÷ 23.26 dB), c) III type (24.29÷30.13 dB)

Rys. 3. Mikrostruktura kompozytu polimerowego wzmacnianego włóknem szklanym: a) I typ (9.56÷16.18 dB), b) II typ (17.2÷ ÷23.26 dB), c) III typ (24.29÷30.13 dB)

Table 1 presents three types of microstructures which were distinguished during the microstructure observations. Their values depended on the geometric shape and aspect ratio, which ranged from 0÷1.5 and 1.5÷3 and above 3. The aspect ratio specifies the reports between the major axis and the minor axis of the ellipse equivalent to the object (the elongation characteristic of the object). The analysis shows an increase in the mean area of the voids content. The first type of microstructure has the highest percentage of the void area where the aspect ratio is from 1.5 to 3, which is similar to the second type of distinguished microstructures. The void width in the second group is the largest, having an aspect ratio above 3. The third type of microstructure demonstrates the largest sizes of perimeter which confirms the highest values of attenuation.

TABLE 1. **Characteristic of voids in glass/epoxy composite**
TABELA 1. **Charakterystyka porowatości w kompozycie szklano/epoksydowym**

Type of microstructure	Aspect ratio	% Voids area	Mean area μm^2	Size length µm	Size width µm	Perimeter µm
I	0÷1.5	27	2531	4÷275	4÷238	11÷934
	1.5÷3	49	3487	7÷679	3÷264	13÷1775
	3<…	24	10045	12÷1377	2÷448	12÷3904
II	0÷1.5	20	4497	4÷439	4÷336	12÷1587
	1.5÷3	46	10133	4÷1074	2÷510	10÷3576
	3<…	34	30923	7÷2328	2÷545	16÷6027
III	0÷1.5	15	4279	5÷470	3÷364	12÷1447
	1.5÷3	40	8259	2÷912	2÷460	2÷2959
	3<…	45	100110	6÷5511	2÷577	12÷17524

Table 2 presents the measurements of voids content for the selected area of the reference plate with definite attenuation. The voids content increased approximately from 2% to 5% along with the rising attenuation level.

The results of the correlation between the attenuation level and the void content are shown in Figure 4 where the linear increase fitting curve describes the dispersion in some attenuation level. This suggests that an increase in the attenuation level can significantly influence an increase in the voids content.

Porosity is one of the most common manufacturing induced defects in composite laminates [4]. The defect size, location and distribution are significant factors characterizing the voids which must be taken into account when estimating the effects of voids on flexural fatigue life and mechanism. It was found that voids have a major effect on the propagation of cracks between plies [5] and it was also proved that the interlayer-shearing strength and compression strength depend on porosity [6]. According to Staffan [7] who identified two types of voids: cylindrical voids within fibre bundles and spherical voids between bundles [5], this experiment confirmed his thesis, which was also confirmed by Bowles and Frimpong [8] who found cylindrical voids between the plies in low void content

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laminates and larger, more spherical voids in high void content laminates. They observed that the distribution of voids in low void material was less homogenous [5].

TABLE 2. **Attenuation levels and voids content for glass/epoxy composite**

TABELA 2. **Poziom tłumienia i zawartość porowatości w kompozycie szklano/epoksydowym**

Samples	Attenuation, dB	Void content, %
1	9.56	2.14
2	10.25	2.35
3	11.26	1,91
4	12.23	2.50
5	13.20	2.05
6	14.20	2.20
7	15.20	2.37
8	16.18	3.42
9	17.20	3.63
10	18.24	2.91
11	19.56	3.50
12	20.22	3.26
13	21.22	3.73
14	22.36	3.37
15	23.26	4.84
16	24.29	4.06
17	25.34	3.96
18	26.26	3.86
19	27.20	5.46
20	30.13	5.36

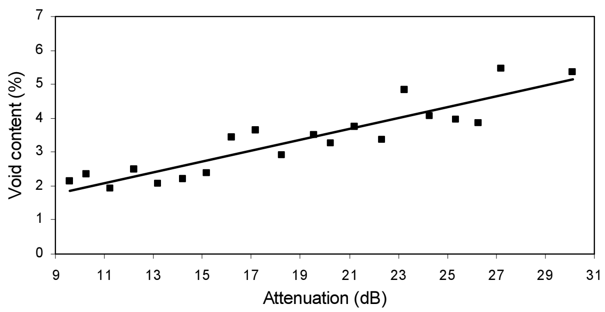

Fig. 4. Correlation between void contents and attenuation levels for glass/epoxy laminate

Rys. 4. Zależność pomiędzy udziałem porowatości a poziomem tłumienia w laminacie szklano/epoksydowym

Laminates that contain more than 5% of voids are normally unacceptable in aerospace applications. A statistical average of void percentages from the cross-sections is taken as an equal to the void volume fraction. That is why Purslow [9] suggested a quality classification of polymer composites as based on the porosity level (V_p - %). Purslow assumed: $V_p < 0.2\%$ - excellent quality; 0.2÷0,5% - very good quality; 0,5÷1% - good; 1÷2% - medium quality; 2÷5% - average quality; $5\% \leq V_p$ - low quality. Although Purslow's classification is only for unidirectional composites, many engineers and constructors use his criterion for their base in the evaluation of very many compound structures made from polymer composites [10]. In this experiment all of the analyzed samples were classified as being medium, average or even low qualitative in statistical measurements of dimensions such as width, length, area and perimeter.

The ultrasonic inspection gives quantitative grading evaluation criterion that will be greatly useful in engineering applications. The resin viscosity affects the resin flow and also the transport of voids and, to a limited degree, their formation and growth [11]. The experimental results obtained in this work will help to develop the model of wave propagation in porous composite materials for voids characterization [6].

According to Liu et al, a greater void content causes an increase of the levels and linear correlation between porosity and the absorption coefficient, which can be observed for porous composite structures [11]. In order to correlate the measured void contents with the attenuation, the slope of the attenuation curve was calculated by a linear fit to the attenuation data and it was plotted as a function of voids content [6].

The Table 1 presents the measured void content for a selected area of the reference plate with definite attenuation. As mentioned before, the acceptance criterion according to Purslow [9] is the range of the attenuation level from 9 to 13 dB. The samples from 6 to 18 show an average quality and the ones from 19 and 20 represent a low quality.

Attenuation was found to be linearly dependent on the frequency, and its magnitude and slope were larger for laminates with more voids. It is therefore important to use prior knowledge about laminate structure and pore morphology in estimating void content from an attenuation slope [6]. The smallest absorption coefficient corresponds to low porosity laminates. The samples with the flat elliptical voids and delaminations gave a higher fractional velocity decrease than the samples with more spherical voids at all tested frequencies [6].

Costa et al defined the regions by a straight line as a function of the size and shape of voids in carbon/epoxy laminates. A transition region, representative of intermediate behaviour due to the simultaneous presence of small and large pores, can also be observed [3].

Examinations of the correlation between the voids content and attenuation levels are required for other composite materials for future investigations in this field.

CONCLUSIONS

The following conclusions can be drawn from this study:

1. The analysis and microstructure observations have shown three types of composite structures depending on the attenuation level in ultrasonic testing. Circular voids (aspect ratio 0÷1.5), circular/ellipsoidal (aspect ratio 1.5÷3) and ellipsoidal/delaminations (aspect ratio above 3) were observed. It may be concluded

that the porosity occurrence with ellipsoidal/delamination shapes (Type III of the structure) has a fundamental influence on the increase in the attenuation level determined by the ultrasonic testing.
2. The level of voids content for this specific type of composite was determined as 2÷5% and it can be classified as a medium structure of composite. The porosity occurrence is probably connected with the technology of producing the composite by the Hand Lay-Up method.
3. The linear dependence between the attenuation level determined in ultrasonic inspection and voids percentage in glass/epoxy composite, within the range of voids content 2÷5%, was found.
4. Ultrasonic inspection and the analysis of microstructure are useful methods in analysing composite structures.

Acknowledgements

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REFERENCES

[1] Ciliberto A., Cavaccini G., Salvetti O., Chimenti M., Azzarelli L., Bison P.G., Marinetti S., Freda A., Grinzato E., Porosity detection in composite aeronautical structures, Infrared Physics and Technology 2002, 43, 139-143.

[2] Yashiro S., Takatsubo J., Toyama N., An NDT technique for composite structures using visualized Lamb-wave propagation, Composites Science and Technology 2007, 67, 3202-3208.

[3] Costa M.L., Franscino M. de Almeida S., Rezende M.C., The influence of porosity on the interlaminar shear strength of carbon/epoxy and carbon bismaleimide fabric laminates, Composites Science and Technology 2001, 61, 2105.

[4] Lopes C.S., Remmers J.J.C., Gürdal Z., Influence of porosity on the interlaminar shear strength of fibre-metal laminates, Key Engineering Materials 2008, 383, 33-52.

[5] Chambers A.R., Earl J.S., Squires C.A., Suhot M.A., The effect of voids on the flexural fatigue performance of unidirectional carbon fibre composites developed for wind turbine applications, International Journal of Fatigue 2006, 28 1390-1395.

[6] Joeng H., Hsu D.K., Experimental analysis of porosity-induced ultrasonic attenuation and velocity change in carbon composites, Ultrasonics 1995, 33, 3, 200-202.

[7] Staffan T., Void formation and transport in manufacturing of polymer composites, Maskinteknik/Stomningslara, Available from: www.epubl.luth.se/avslutade/0348-8373/184

[8] Bowles K.J., Frimpong S., Void effects on the interlaminar shear of unidirectional graphite reinforced composites, Journal of Composite Materials 1992, 26.

[9] Purslow D., On the optical assessment of the void content in composite materials, Composites 1984, 15(3), 207--210.

[10] Bełzowski A., Stasieńko J., Ziółkowski B., Kamińska A., Niektóre kryteria akceptacji defektów w kompozytach na przykładzie laminatu ciętego strumieniem wody, Kompozyty (Composites) 2004, 4, 12, 390.

[11] Liu L., Zhang B.M., Wang D.F., Wu Z.J., Effects of cure cycles on void content and mechanical properties of composite laminates, Composite Structures 2006, 73, 303-309.

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