Ultrasonic evaluation of the fibre content in glass/epoxy composites

G. Wróbel, S. Pawlak*

Division of Metal and Polymer Materials Processing,
Institute of Engineering Materials and Biomaterials,
Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland
* Corresponding author: E-mail address: sebastian.pawlak@polsl.pl

Materials

ABSTRACT

Purpose: The aim of the work was to find relationships between the selected parameter of an ultrasonic wave and the local fibre content in a glass/epoxy composite.

Design/methodology/approach: The experiments have been performed in two distinct phases. During the first phase, typical glass/epoxy composite materials with different fibre content were examined by means of through-transmission and pulse-echo ultrasonics.

In the second phase, the standard destructive method was applied to determine the actual glass content in investigated composite materials

Findings: The experimental results showed relationships between ultrasonic wave velocity and the local fibre content in investigated composite materials. This study has also assessed the ability of pulse-echo technique to carry out such a testing.

Research limitations/implications: Relationships found may be useful to local fibre content examination, but for each composite material different relationships are required to be determined, as was done in the present study.

Practical implications: The described method can be applied to the post-production quality control of a finished composite product, but in the case of composites made of different components it is necessary to determine distinct relationships for each material.

Originality/value: A new approach to the problem of fibre content examination has been demonstrated by means of pulse-echo technique. The method developed should be of interest to the industrial quality control procedures and has great importance for products with a high failure-free requirements.

Keywords: Composites; Glass/epoxy composites; Glass fibre content; Non-destructive testing; Ultrasonic wave velocity

1. Introduction

Glass fibre reinforced composites are increasingly used in aerospace, naval, and automotive vehicle due to their excellent corrosion and wear resistance, high specific strength and stiffness. In comparison with more traditional construction materials (steel and aluminium alloys), polymer matrix composites exhibit also light-weight, good fatigue performance and low through life maintenance costs [1]. However, the mechanical properties of composite materials are very sensitive to the local fibre content variations. While determining the mechanical properties, their distribution in the whole area of composite is especially important because the strength of composite highly depends on the amount of fibre in a given section [2]. Local differences in the amount of reinforcement arising during the production process decide about out-of-control variation of strength properties in a given element, which is of great importance in the case of products with high failure-free requirements. Traditionally, the fibre content is measured by three basic methods, including solvent extraction,

burn-off and acid digestion [3]. However, these methods suffer some disadvantages such as high cost of disposal of chemical wastes and the excessive time that is required to perform these tests [3]. Moreover, these methods can't be applied when components are subject to a hundred-percent inspection. Therefore, there is a need of searching for non-destructive methods of fibre content evaluation. Authors made such an attempt using ultrasonic techniques.

Ultrasonic methods are mostly developed for characterization of metals and alloys. It is an effective instrument for evaluation of elastic modules, strength, stiffness, and other essential parameters which are helpful for analysis and design of structures [4]. Unfortunately, it is impossible to compile directly achievements of ultrasonic techniques for other materials to composites and it also gives a considerable dispersion of data [5, 6]. Ultrasonic methods can be also used for analysis of destructive processes in composite materials under static and fatigue stresses and degree of degradation of composite materials, which allows an insight into the destructive processes [7-12]. Less information is available on application of ultrasonic testing for reinforcement content determination in fibre composite materials.

2. Procedure

The experiments have been performed in two distinct phases. During the first phase, glass/epoxy specimens were subjected to the ultrasonic inspection. In the second phase, the standard destructive method was applied to determine the actual glass content in investigated specimens.

2.1. Materials

The examined composite materials were made from E-glass woven fabric RT (Saint-Gobain Vetrotex Europe) with a density of 2.54 [g/cm^3] and areal weight of 800 [g/m^2], epoxy resin L 1000 (Bakelite, Germany) with a density of 1.15 [g/cm^3] and hardener VE 5195 (Bakelite, Germany) with a density of 0.93 [g/cm^3].

The composite specimens was laminated by hand lay-up, and resin matrix was cold-cured under ambient conditions (~20ºC). Specimens produced in such a way had 30÷70% weight content of glass fabric and with 10 mm average thickness. These specimens, after curing process, were thermally hardened at 50 ºC for 24 hours. The next step was to cut their rims to the final dimensions of 120x90x10 mm. In such a way 9 composite specimens with a different glass content were prepared.

2.2. Ultrasonic testing

Due to the need to obtain a certain amount of data sufficient for the purpose of the examination, 8 measurement points were chosen on the whole surface area of the specimen. These points were selected in such a way that it would be possible to measure exact thickness and later on ultrasonic examination in the same place (Fig. 1). Before destructive test, a group of specimens was subjected to the ultrasonic inspection.
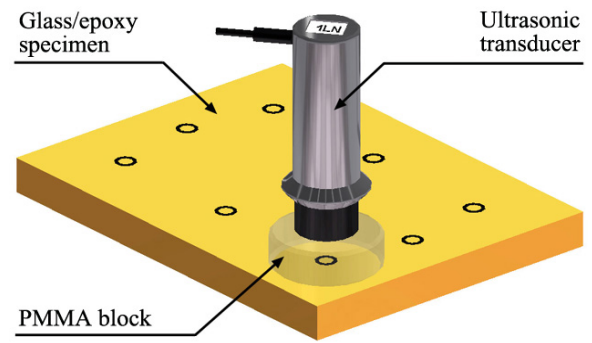

Fig. 1. Scheme of the ultrasonic testing of propagation time in selected points of the specimen

Ultrasonic longitudinal velocity in the specimens was measured using a PC UMT-12 flaw detector (Ultramet S.c., Poland) and one single transmitter-receiver 1LN 1MHz transducer with a diameter of 13 mm (Unipan, Poland). The ultrasonic instrumentation was operated in the time-of-flight mode using the pulse-echo technique. The velocity (c) of the propagating wave was determined on the basis of well-known formula 1:

$$c = 2 \cdot h / \tau, \qquad (1)$$

where:
$2 \cdot h$ – doubled thickness of the specimen in the place where the transducer was put against it [mm],
τ – time-of-flight of ultrasonic wave in [µs].

A 10 mm thick PMMA block was put between specimen and ultrasonic transducer to provide better wave matching and to provide a time delay to ensure the pulse-echo signal was not masked by the initial pulse signal. The PMMA block was chosen because of its well-known acoustic properties and it is also the material used to angle-transducers manufacturing.

As a coupling medium between the transducer and PMMA block and between PMMA block and the examined material, "Żelpol USG" (Centrum Medicum Poland) was used.

A-scans observed on the screen were characterised by a cluster of free large peaks, and these signify the reflection of ultrasound waves from the back-surface of the specimen. There were also observed many echoes reflected from glass layers of the composite specimen as well as echoes from PMMA surfaces. The reflection of ultrasound waves in a polymer composite is usually observed in an A-scan as a single peak rather than a cluster of peaks. However, the rough back surface of the specimen, with average peak-to-valley height of 1 mm, caused differences in the time-of-flight of the ultrasound waves when reflected from the peaks and valleys at the back-surface of the specimen resulting in multiple pulse-echo signals in the A-scans. Described situation has been confirmed in other publication [13].

2.3. Destructive analysis

As an appropriate method to determine actual fibre content in composite specimens made from E-glass and epoxy resin,

standard destructive burn-off method was applied. This examination was carried out in accordance with ISO 1172:2002 standard "Textile-glass-reinforced plastic - Prepregs, moulding compounds and laminates - Determination of the textile-glass and mineral-filler content - Calcination methods".

In accordance with the standard, two test specimens were cut from location on the composite to be representative of the material. It was performed with the use of a diamond circular saw blade in such a way that their weight was within the range of 2÷10 g. The next step was to dry the specimens to evaporate moisture and weighted with the use of a precision balance. Then, each specimen was put in a melting pot and baked in the temperature of 600 ºC for approximately 1 hour. That time was determined experimentally as weight of the specimens did not change after subsequent bakings. The glass content M_{glass} in each examined specimen was determined in accordance with the standard as a fraction of the initial weight and expressed by formula 2:

$$M_{glass} = (m_3 - m_1) / (m_2 - m_1) \cdot 100, \qquad (2)$$

where:
m_1 – initial weight of the melting pot in [g],
m_2 – initial weight of the melting pot with a sample in [g],
m_3 – final weight of the melting pot with calcination residue in [g].

Results of the destructive examination of the glass content and average thickness of each specimen as well as average ultrasonic wave velocity were put into table 1.

Table 1.
Thickness and determined properties of the specimens

Average thickness [mm]	Percent glass by weight [%]	Average speed of sound [m/s]
9.1	31.0	2461
9.3	37.2	2581
15.1	55.1	2744
13.8	55.9	2818
12.5	56.8	2950
13.1	57.3	2964
11.2	60.9	3014
10.7	65.2	3045
10.3	69.1	3169

3. Results amd discussion

The Results presented in this section show relationship between the propagating velocity and the content of glass in the form of woven fabrics (Fig. 2). The graph shows that the content of glass in a composite material causes an increase of the velocity with which waves propagate through the examined material, so it has been confirmed in other publications [14, 15].

For the assumed confidence interval of a random variable (–2s, +2s), the accuracy of this research method amounts to ±5,74%.
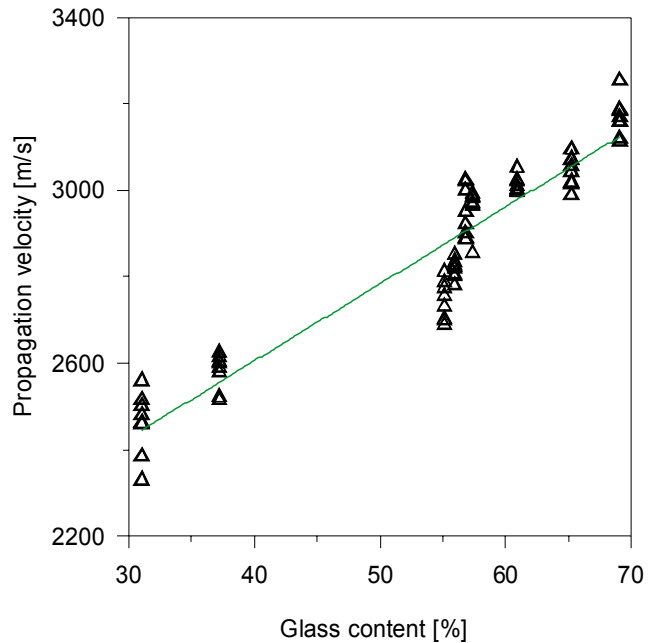

Fig. 2. Relationship between the ultrasonic wave velocity and the glass content in examined glass/epoxy specimens (obtained using pulse-echo technique)

Table 2.
Results of linear approximation of the relationship between wave velocity [m/s] and the glass content [%]

Statistical quantity	Result
Correlation coefficient, R	0.938
Standard deviation of the fibre glass content, s	2.874

For the passing velocity of an ultrasonic wave "c" and glass content "V", the result of a linear approximation (for glass in a form of woven fabrics and for the range of 30÷70%) forms the following dependence:

$$V = 0,057 \cdot c - 108,76 \qquad \pm 5,74\% \qquad (3)$$

In that relationship, V is expressed in [%], while c in [m/s].

Figure 3 shows the previous research results [15], where glass/polyester composites were investigated using through-transmission ultrasonics. The previous investigations were performed for the composites made from glass in the form of woven fabrics and polyester resin and composites made from glass in the form of mats and polyester resin, measuring the ultrasonic wave velocity (Fig. 3) [15]. Existence of different relationships between the wave velocity and the content of glass in a form of woven fabrics and in the form of mats indicates that not only the content but also geometrical form of the reinforcement as well as resin type influences the ultrasonic wave velocity. In comparison with present investigation results (Fig. 2), there is similar dispersion of data and almost the same accuracy (±6%) achieved using pulse-echo ultrasonics.
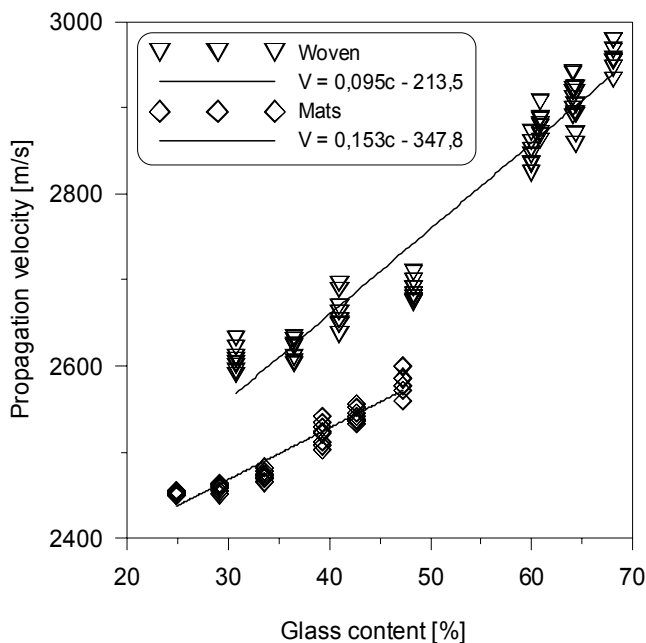
Fig. 3. Relationship between the ultrasonic wave velocity and the glass content in the form of mats and woven fabrics for glass/polyester composites (obtained using through-transmission technique) [15]

The pulse-echo technique seems to be more convenient, but there are also some disadvantages like that the extreme care must be taken for instrumentation use and calibration, and high skills of the operator are required in order to obtain correct results. A great importance for good resolution and accuracy of the experimental results is also in the choice of the transducer frequency. These observations demonstrate the difficulties that can be expected during the NDT of such composites using pulse-echo technique.

4. Conclusions

The ultrasonic wave velocity as a function of glass fibre content was determined from a specially prepared specimens. The results obtained by pulse-echo technique shows differences of wave velocity for the specimens with different glass content. The study has also assessed the ability of pulse-echo technique to carry out such a testing. The described method can be applied to the post-production quality control of a finished composite product, but in the case of composites made of different components it is necessary to determine distinct relationships for each material.

The evaluation of the reinforcement content may be improved by using ultrasonics in combination with other NDT techniques such as thermography, which is out of the scope of this paper.

References

[1] L.A. Dobrzański : Fundamentals of Materials Science and Physical Metallurgy. WNT, Warszawa 2002 (in Polish)
[2] S.B. Heru, J. Komotori, M. Shimizu, Y. Miyano: Effects of the fiber content on the longitudinal tensile fracture behavior of uni-directional carbon/epoxy composites, Journal of Materials Processing Technology, Vol. 67, 1997, 89-93
[3] Y.D. Huang, L. Liu, Z.Q. Zhang, Y. Wan: On-line monitoring of resin content for film impregnation process. Composites Science and Technology 58, 1998, 1531-1534
[4] G. Marsh: Finding flaws in composites. Reinforced Plastics, 2002, 22-28
[5] G. Nesvijski Edouard.: Some aspects of ultrasonic testing of composites. Composite Structures 48, 2000, 151-155
[6] C. Potel, T. Chotard, J.F. Belleval, M. Benzeggagh: Characterization of composite material by ultrasonic methods. Composites Part B, 1998, 159-169
[7] [7] Vaccaro C., Akers D.: Damage assesment in a SMC composite by means of ultrasonic techniques. Review of Progress in Quantitative Nondestructive Evaluation. Vol 13, Plenum Press, New York, 1996
[8] M. Rojek, J. Stabik, G. Wróbel: Ultrasonic methods in diagnostics of epoxy-glass composites. Journal of Materials Processing Technology, 2005, 162-163
[9] K. Maslov, Y.R. Kim, K.V. Kinra, J.N. Pagano: A new technique for the ultrasonic detection of internal transverse cracks in carbon-fibre/bismaleimide composite laminates. Composite Science and Technology 60, 2000, 2185-2190
[10] C. Scarponi, G. Briotti: Ultrasonic technique for the evaluation of delamination on CFRP, GFRP, KFRP composite materials. Composites: Part B 31, 2000, 237-243
[11] G. Wróbel, Ł. Wierzbicki: Ultrasounds in diagnosis of strength changes in laminates put in ageing conditions, 3rd Scientific Conference on Materials, Mechanical and Manufacturing Engineering, 2005, 575-580
[12] K. Imielińska, M. Castaings., R. Wojtyra, J. Haras, E. Le Clezio, B. Hosten: Air-coupled ultrasonic C-scan technique in impact response testing of carbon fibre and hybrid: glass, carbon and Kevlar/epoxy composites, Journal of Materials Processing Technology, Vol. 157-158, 2004, 513-522
[13] A.P. Mouritz, C. Townsend, M.Z.Shah Khan: Non-destructive detection of fatigue damage in thick composites by pulse-echo ultrasonics. Composites Science and Technology 60, 2000, 23-32
[14] S. Ochelski: Experimental methods in construction coposites mechanics. WNT, Warszawa 2004 (in Polish)
[15] G. Wróbel, Ł. Wierzbicki, S. Pawlak: Ultrasonic quality evaluation method for polyester glass laminated materials. 11th International Scientific Conference on Contemporary Achievements in Mechanics, Manufacturing and Materials Science. Gliwice – Zakopane, 2005, 1040-1044