



# A method for ultrasonic quality evaluation of glass/polyester composites

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## ABSTRACT

**Purpose:** The aim of the present study was to find relationship between the ultrasonic wave velocity or attenuation coefficient and the local fiber content in glass/polyester composites.

**Design/methodology/approach:** Experimental data have been obtained using ultrasonic wave velocity and attenuation measurements. To determine the actual fiber content in the composites, needed to build proper relationships between considered factors, the standard destructive analysis was applied. For ultrasonic non-destructive testing, through-transmission technique was used.

**Findings:** Experimental results have shown that the longitudinal wave velocity increases almost linearly with an increase of the fiber content in the investigated specimens. The second considered parameter of an ultrasonic wave, namely attenuation coefficient was not correlated with the glass content.

**Research limitations/implications:** The propagation velocity of the ultrasonic waves can be affected, apart from fiber content, by interfacial stresses and changes of elastic modulus in polymer matrix during long-lasting service time. These factors overlap and which of the two can affect the ultrasonic wave to a higher degree is yet to be established. Further work is needed in this area.

**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 constituent materials it is necessary to determine distinct relationships for each composite.

**Originality/value:** The results obtained would be of considerable importance in the industrial applications to achieve a first estimate of fiber content variations in polymer composite materials.

**Keywords:** Non-destructive testing; Ultrasonic wave velocity; Glass/polyester composites; Fiber content; Quality control

## PROPERTIES

### 1. Introduction

Fibre reinforced polymer 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 traditional construction materials such as steel and aluminium alloys, polymer matrix composites exhibit also significant weight savings, good fatigue performance and improved

service-time performance [1]. It is known that the mechanical properties of fiber reinforced composites, among others, are local fiber content dependent. Local variations in the reinforcement content 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 subjected to a hundred-percent inspection. A wide range of application for these materials raises a necessity for non-destructive investigation methods, mainly at the stage of post-production quality assurance and during

component service-time. That demand is particularly important for the quality assessment of products with high failure-free requirements being mass-produced.

The content of reinforcement can be also treated as a one of the quality criteria of finished composite products. There are many research works directed towards understanding how reinforcement content can affect chosen properties of composite materials [2÷5]. To improve manufacturing quality it is necessary to develop methods, which are suitable for the purpose of reinforcement content determination. Traditionally, the fibre content is measured by three basic methods, including solvent extraction, burn-off and acid digestion [6]. These methods, due to their destructive character cannot be applied when components are subjected to a hundred-percent inspection. The observations above suggest that there is a need to develop the non-destructive methods to be applicable for presented purpose. From a wide range of non-destructive techniques, ultrasounds seem to be the most promising for this purpose, because the propagation of mechanical waves is elastic properties and density of the medium dependent [7, 8]. It is also known that propagation of ultrasonic waves is sensitive to the variations in the microstructure and mechanical properties. Therefore, establishing a relationship between the microstructural (fiber content) and ultrasonic evaluation results could be very useful for improving the process parameters and controlling the quality of the products [8]. So far, ultrasounds have found many applications in non-destructive testing of composite materials [9-18].

Ultrasonic testing of materials, including the polymer composites, consists of introducing waves into objects, scanning of these objects with a transducer(s) moved over their surface and detection of signals caused by the waves propagating through these objects [19,20].

Ultrasonic waves are most frequently used to determine properties of materials [22] and to detect possible inclusions in composites. Ultrasounds are also used in material defect examination and geometric distribution of these defects in new, in-service and damaged products, as well as for measurement of their thickness [11,13,26]. Ultrasonic methods can be also used for analysis of destructive processes in composite materials under static and fatigue stresses [27], which allows an insight into the destructive processes, assessment of the degree of degradation of composite materials [14].

Parameters that characterise an ultrasonic wave used in the diagnostic of materials are [13,20]:

- $c$ : propagation velocity;
- $\alpha$ : amplitude-attenuation coefficient

$$\frac{dA}{A} = -\alpha dx, \quad (1)$$

where:

$A$  - wave amplitude,  $dx$  – path of  $dA$  amplitude drop;

- $\gamma$ : energetic attenuation coefficient

$$\frac{dI}{I} = -\gamma dx, \quad (2)$$

where:

$I$  - wave intensity,  $dx$  – path of  $dI$  wave intensity drop.

Both, wave propagation velocity and element thickness measurement methods can utilise the same measurement device. Most flaw detectors allow measuring wave-propagating time and, on the basis of wave propagation velocity, also thickness of the wall.

Measurements are usually performed using pulse-echo and through-transmission method and with the use of single or double transducers [9].

Previous research results [23] have shown that the local fiber content in glass/epoxy composites is detectable by ultrasonic measurements using pulse-echo technique. It was also presented that inhomogeneous distribution of the reinforcement, which significantly degrades the mechanical properties, and ultrasonic wave velocity are closely related [23].

The primary objective of present research is directed towards understanding the effect of fiber content on the ultrasonic wave velocity or wave attenuation in glass/polyester composites. For this purpose, two groups of specimens were ultrasonic tested and also subjected to the damage investigation.

## 2. Experimental

### 2.1. Procedure

The ultrasonic testing and destructive analysis was conducted to investigate the effect of fiber content and attenuation coefficient on the ultrasonic wave velocity in glass/polyester specimens. For damage analysis and ultrasonic measurements, the specimens were prepared with the variation of glass content by conventional hand lay-up. The specimens were investigated using through-transmission ultrasonic wave velocity and attenuation coefficient measurements. The damage analysis was applied to determine the exact fiber content in considered materials.

### 2.2. Materials

Investigated composite specimens were made of cross - ply plain weave [0/90] E-glass fabric and mats (Saint Gobain Vetrotex Europe), polyester resin „Aropol M 105 TB” (Ashland Finland) and initiator „Butanox M-50” (Akzo Nobel Polymer Chemicals). The details about constituent materials are summarized in Table 1.

Glass fiber reinforced polyester composites were fabricated by hand lay-up, with variation of glass content. The polyester resin was cold-cured under ambient conditions (~20 °C). The composites prepared in such a way had a glass content at the range of 30÷70% (wt.) for woven fabrics and 25÷50% (wt.) for mats. The average thickness of composites was about 10 mm. The variation of glass content was achieved using different number of glass layers with the same total thickness of the specimens. Polyester matrix, after curing process was thermally hardened at 50 °C for 24 hours.

Table 1.  
The properties of constituent materials

Parameter	Unit	E-glass	Polyester resin
Density	g/cm <sup>3</sup>	2.58	1.10
Tensile strength	MPa	3500	55
Elastic modulus	GPa	75	3.6

### 2.3. Wave velocity measurements

Ultrasonic velocity in the specimens was measured using a MG 2000S ultrasonic thickness meter (AZ Industry Supplier, Warsaw, Poland). The schematic view of the ultrasonic investigation is shown in Fig. 1. The apparatus had build-in function to measure the propagating time and was operated in the time-of-flight mode using through-transmission technique. For longitudinal velocity measurements, two standard (longitudinal wave) transducers of 2 MHz frequency were used. The accuracy of velocity measurements was within  $\pm 1 \text{ ms}^{-1}$ . Eight measurement points were chosen on the surface area in such a way that there would be no side-wall effect on ultrasonic wave.

For longitudinal velocity, eight measurements were made and that values have been reported (Fig. 3. and 4.). As a coupling medium at the transducers-specimen surface, the USG-gel (Centrum Medicum Poland) was applied.

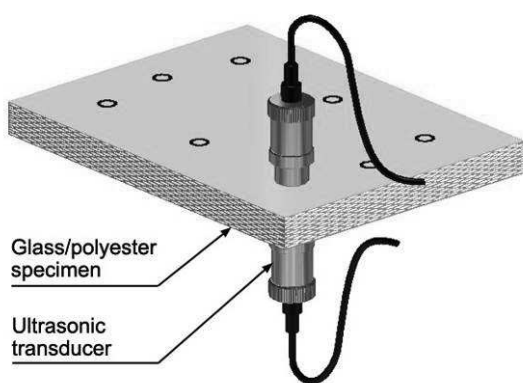


Fig. 1. Scheme of the ultrasonic testing of wave velocity

The ultrasonic wave velocity ( $c$ ) was determined on the basis of well-known formula 3:

$$c = h / \tau, \quad (3)$$

where:

- $h$  - thickness of the specimen in the place where transducers were put against it,
- $\tau$  - time-of-flight of the ultrasonic wave.

### 2.4. Wave attenuation measurements

Attenuation measurements have been performed using the 512-type ultrasonic flaw detector produced by Scientific Equipment Industry "Unipan", Warsaw Poland. The device allows reading amplification in decibels [dB] with a set level of amplitude. The measuring stand also comprised additional equipment for perpendicular (relative to the transducers) hold of the specimen immersed in the coupling medium. The equipment together with an examined specimen are shown in Figure 2.

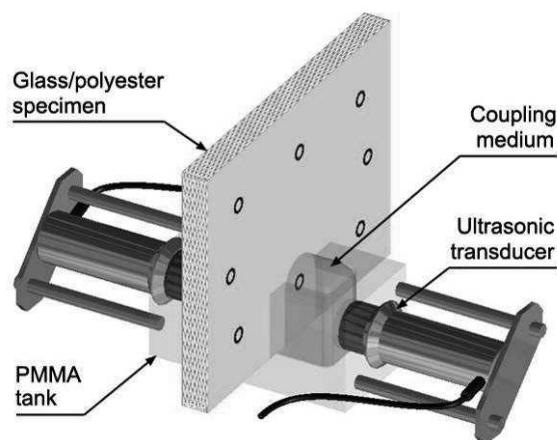


Fig. 2. Scheme of the ultrasonic testing of wave attenuation

The transducers used in examination had a frequency of 1 MHz. The specimens were fastened in a PMMA tank in such a way that the chosen measurement point were in axis with the transducers. The tank was filled with the coupling gel, which level was monitored and kept constant. For a given position of the specimen, amplification was adjusted according to proper level of the signal amplitude and amplification ( $A$ ) was read in decibels. Wave attenuation coefficient  $\alpha$  [dB/m] was determined on the basis of the formula 4 [25]:

$$\alpha = \alpha_0 + 1/l \cdot 23 \log(A_0/A_1) + 1/l \cdot 23 \log(1 - R^2), \quad (4)$$

where:

- $\alpha_0$  - attenuation coefficient in the liquid,
- $A_0$  - impulse amplitude after propagating through the liquid, between the transducers, without the measured material,
- $A_1$  - amplitude in the liquid after a single propagating through the material,
- $R$  - reflection coefficient equals to:  $\left| (\rho_2 \cdot c_2 - \rho_1 \cdot c_1) / (\rho_2 \cdot c_2 + \rho_1 \cdot c_1) \right|$ ,
- $c_1$  - propagating velocity in the coupling medium,
- $c_2$  - propagating velocity in the specimen,
- $\rho_1$  - density of the coupling medium,
- $\rho_2$  - specimen density,
- $l$  - specimen thickness.

### 2.5. Damage investigation

The damage analysis was performed according to the method described in ISO 1172:2002 [24]. The whole population of specimens were tested in order to achieve the information about actual glass content from each specimen. 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  $2 \pm 10 \text{ g}$ . 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 about  $600 \text{ }^\circ\text{C}$  for

Table 2.  
Results of damage investigation for specimens made of woven fabrics and mats

Specimen no.	Actual glass content [%]	Specimen no.	Actual glass content [%]
1W	30.7	1M	24.8
2W	36.4	2M	29.1
3W	41.0	3M	33.6
4W	48.3	4M	39.2
5W	59.9	5M	42.6
6W	60.8	6M	47.2
7W	64.0	7M	52.8
8W	64.3	8M	57.3
9W	68.1	-	-

W - woven fabrics, M - mats

approximately 1 hour. That time was determined experimentally as weight of the specimens did not change after subsequent bakings. The glass content  $M_{\text{glass}}$  in each examined specimen was determined in accordance with the standard as a fraction of the initial weight and expressed by formula 5:

$$M_{\text{glass}} = (m_3 - m_1) / (m_2 - m_1) \cdot 100, \quad (5)$$

where:

- $m_1$  – initial weight of the melting pot in [g],
- $m_2$  – initial weight of the melting pot with a specimen in [g],
- $m_3$  – final weight of the melting pot with calcination residue in [g].

Results in [% (wt.)] achieved from damage investigation for specimens made of woven fabrics and mats are given in Table 2.

### 3. Results and discussion

Comparing the values of fiber content and ultrasonic wave velocity in investigated materials it became clear that these two properties are closely related. The results of ultrasonic testing of the wave velocity for specimens made of glass in the form of woven fabrics are presented in Figure 3. The graph shows that the glass content in a composite material causes an increase of the velocity with which waves propagate through the examined material. The results show that in general the ultrasonic velocity increases almost linear with an increase in glass content. This is in agreement with findings of other researchers [8, 21]. The results of linear approximation for considered specimens are shown in Table 3 and 4. Existing dispersion of data in the wave velocity (Fig. 3. and 4.) can be attributed to the glass content in examined specimens and possible local absence of glass on the path of ultrasonic wave.

Table 3.  
Results of linear approximation of the relationship between the propagating velocity [m/s] and the glass content [%] for woven fabrics

Statistical quantity	Result
Correlation coefficient, R	0.97
Standard deviation of the fibre glass content, s	2.93

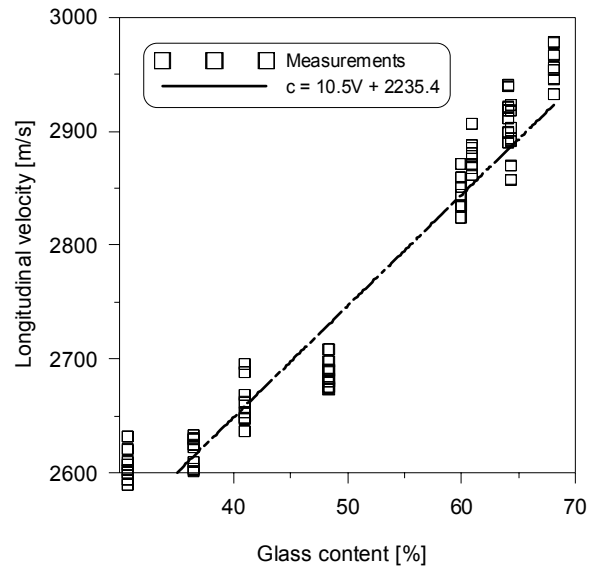


Fig. 3. Relationship between the ultrasonic wave velocity and the glass content (for glass in the form of woven fabrics)

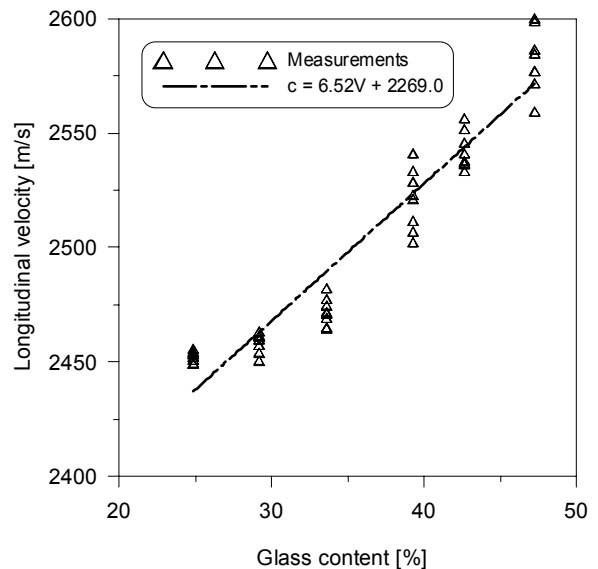


Fig. 4. Relationship between the ultrasonic wave velocity and the glass content (for glass in the form of mats)

For the assumed confidence interval of a random variable  $(-2s, +2s)$ , the accuracy of this research method amounts to  $\pm 5.86\%$ .

For the propagating velocity of an ultrasonic wave ( $c$ ) and glass content ( $V$ ), the result of a linear approximation (for glass in the form of woven fabrics and for the range of  $30 \div 70\%$ ) forms the following linear relationship:

$$V = 0.0955 \cdot c - 213.5 \quad \pm 5.86\%, \quad (6)$$

where,  $V$  is expressed in [%], and  $c$  in [m/s].

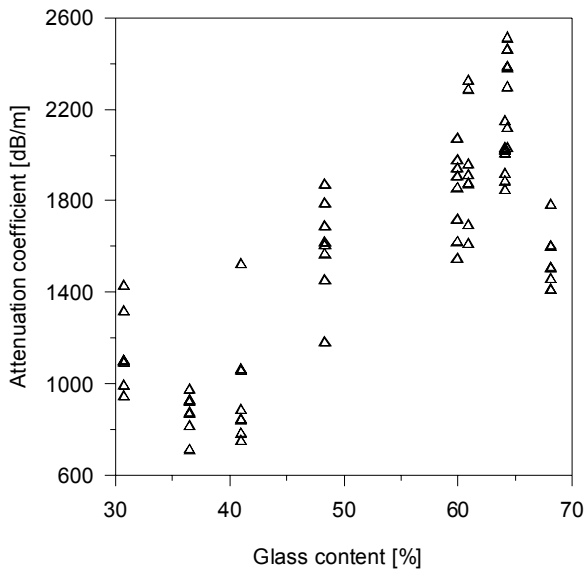


Fig. 5. Relationship between the ultrasonic wave attenuation and the glass content (for glass in the form of woven fabrics)

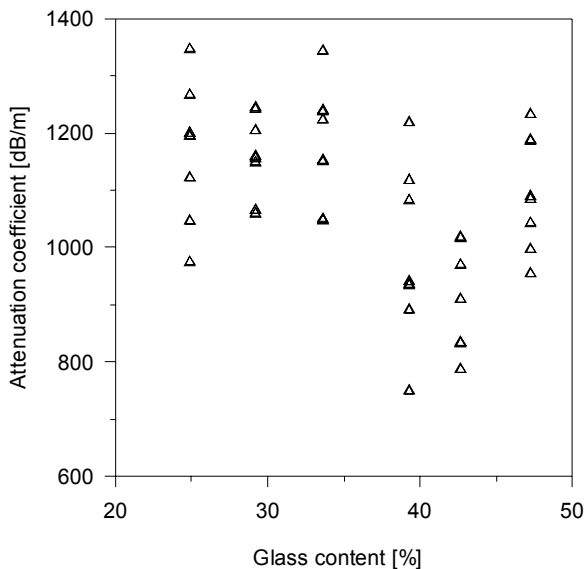


Fig. 6. Relationship between the ultrasonic wave attenuation and the glass content (for glass in the form of mats)

The similar analysis has been performed for specimens containing glass in the form of mats. Figure 4 shows the results of ultrasonic wave velocity measurements for this type of reinforcement. Obtained relationship can be present as a linear function between the propagating wave velocity and the glass content (Fig. 4).

Analogous to the previous case, increased glass content in the composite causes an increase in the propagating wave velocity.

For the assumed confidence interval of a random variable (-2s, +2s), the accuracy of this research method amounts to ±4.42%.

Table 4.

Results of linear approximation of the relationship between the propagating velocity [m/s] and the glass content [%] for mats

Statistical quantity	Result
Correlation coefficient, R	0.95
Standard deviation of the fibre glass content, s	2.21

The result of a linear approximation (for glass in the form of mats and for the range of 25÷50%) forms the following relationship:

$$V = 0.1532 \cdot c - 347.8 \pm 4.42\% \quad (7)$$

where,  $V$  is expressed in [%], and  $c$  in [m/s].

Existence of different relationship between the propagating velocity and the content of glass in the form of woven (6) and in the form of mats (7) indicates that not only the content but also geometrical form of the reinforcement influences the propagating velocity of an ultrasonic wave.

Figures 5 and 6 show the results of ultrasonic examination the wave attenuation in examined materials. The graphs reveal considerable dispersion of results, which could be caused by too small sensitivity of the measurement device or using too high frequency (1 MHz) of the transducers for such inhomogeneous material.

Due to the high dispersion of data, low correlation coefficient and relatively high standard deviations, no relationship has been found between the attenuation and the glass content in the tested materials. That can be caused, among other things, by not sufficient accuracy of the measuring device, which probably caused considerable dispersion of the results. Therefore, that results are not considered any further.

#### 4. Conclusions

In the present study, ultrasonic testing and standard damage analysis were carried out on the two groups of glass/polyester specimens, which were fabricated by conventional hand lay-up. The specimens were prepared with variation of reinforcement content. Results obtained from non-destructive analysis have proven a possibility that the propagating velocity of an ultrasonic wave can be the primary parameter in evaluation of the glass content in a glass/polyester composite material. The obtained results showed that increasing fiber content leads to an increase in ultrasonic wave velocity. The determined relationships are linear functions, different for each type of reinforcement.

The method described can be applied to the post-production quality control of finished polymer composite products, but in the case where composites are made of different constituent materials, distinct relationships should be determined.

The results have also shown that the second considered parameter of an ultrasonic wave, namely attenuation coefficient was not correlated with the glass content in the investigated composite materials.

The ultrasonic wave velocity, except from glass content, can be also affected by interfacial stresses and time-changes of elastic modulus in polymer matrix during service time indicating that further investigations are needed in this area.

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