A study of physical and mechanical characteristics of polymer composite materials by ultrasonic technique

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Abstract. This work is concerned with the determining of physical and mechanical characteristics (dynamic elastic modulus along the direction of reinforcement, porosity, density, percentage of binder, strength) of fiberglass plastics by ultrasonic technique. The techniques, which are currently used for evaluation of the above-mentioned parameters of polymeric composite materials, are presented. The ultrasonic technique of physical and mechanical characteristics determining of glass fiber reinforced plastics for radio engineering purpose on the base of quartzitic, glass and siliceous fillers is proposed.

1. Introduction
Polymer composite materials (PCM) are of great commercial and technological interest due to their special properties: high specific strength, stiffness and elastic modulus; good damping ability and corrosion resistance; high fatigue life and low coefficient of thermal expansion [1-4]. Of the entire variety of PCMs, fiberglass plastics — PCMs based on inorganic oriented fabric quartz, glass, and silica fillers — should be distinguished. Fiberglass is one of the most common PCMs, combining high strength, low density, good dielectric properties and reasonable price [5-7]. To ensure the high quality of fiberglass products, it is necessary to use new solutions in the development and creation of methods and means of non-destructive testing (NDT). This will eliminate the flaw at the early stages of manufacturing products, control the correctness of the technology parameters, and evaluate the reliability, manufacturability [8, 9]. An important area of NDT quality of materials, parts, products and structures made of PCM is the determination of physical and mechanical characteristics, which allows us to switch from spot defining these properties on specially manufactured samples for their control on final products without their destruction or damage. This increases the reliability of product quality assessment and reduces material costs.

2. Techniques of non-destructive testing of physical and mechanical characteristics of PCM
The determination of the physical and mechanical characteristics of PCMs using NDT techniques is a difficult task, which is due to the wide variety of PCMs, specific design features of them and manufacturing technology, a wide range of physical and mechanical characteristics, and a variety of types of defects that arise during the manufacturing process [10]. The main techniques of non-destructive testing physical and mechanical characteristics of PCM [11]: dielectric constant; on thermal effects; electrical resistance; acoustic methods.
Acoustic techniques have been widely used to determine the following physical and mechanical properties of PCMs: elastic properties (elastic modulus, shear modulus, Poisson's ratio), strength (tensile, compression, bending, torsion, shear, etc.), technological (density, humidity, the content of individual components, etc.), structural (anisotropy of the material), the content of inclusions and other properties [12].

In standard [13], non-destructive testing of PCMs was carried out using ultrasonic waves by acousto-ultrasonic (AU) technique according to the correlation dependence of the SWF criterion (stress wave factor) on the diagnostic parameter. The following main variants of the SWF criterion can be selected: amplitude, when the criterion is defined as the maximum of the amplitude signal; countable criterion based on the number of the signal exceeds the threshold value; energy criterion determined by the energy of the signal (the amplitude integral over time or the integral of the signal spectrum in the frequency range of interest). The disadvantage of this method is that it does not allow us to determine the strength characteristics, but only makes it possible to identify structural heterogeneities.

In [12, 14], to determine the strength characteristics of PCM, it was proposed to use the integral of the optimal signal spectrum as the SWF criterion:

$$\sigma = \varphi \left[ \int_{f_{\text{min}}}^{f_{\text{max}}} S(f) \frac{\Sigma_{i=1}^{n} [S_{1i}(f) - S_{2i}(f)]}{f} \right],$$

where \(\sigma\), \(\varphi\) are strength and the sign of correlation consequently; \(S(f), S_{1i}(f)\) and \(S_{2i}(f)\) are spectra of pulses of ultrasonic vibrations transmitted in the PCM control object, witnesses specimens after manufacture and subjected to damaging influences; \(n\) – number of witness specimens; \(i\) is serial number of measurements; \(f_{\text{min}}\) and \(f_{\text{max}}\) are the range of spectral boundary frequency. The technique allows determining only the strength characteristics of carbon plastics.

In [15–18], techniques for determining the physical and mechanical characteristics of PCMs from the correlation dependence of the velocity, amplitude, or attenuation of ultrasonic waves on the diagnostic parameter with a through or echo-pulse monitoring scheme are presented. The disadvantage of this technique is the low accuracy of determining PCMs characteristics in the direction of reinforcement. Determination of physical and mechanical characteristics with the aid of acoustic resonance and the method of acoustic emission described in [19-22]. The disadvantages of these techniques are the need of loading the structure, the complexity of the equipment and the low reliability of the control.

Each of the above listed techniques has its own limitations, making impossible to use it for NDT determination of physical and mechanical characteristics of different-thickness parts of ogival form made from fiberglass with woven angle-ply lay-up and high porosity.

To solve this problem, it was proposed to use a comprehensive control technique, which consists in applying two techniques for measuring the parameters of ultrasonic waves: along the direction of reinforcement and perpendicular to the PCM layers. Measurement of the parameters of ultrasonic wave’s perpendicular to the layers makes it possible to increase the control sensitivity due to the possibility of using a higher signal frequency and allows local control (region with a diameter of 5 mm) of the material structural characteristics, which seems impossible to control by the standard method [13]. It is also possible to increase the accuracy of determining the parameters in the direction of reinforcement, by means of increasing the information content of the SWF criterion, which can be used to calculate not only the parameters of the ultrasonic signal obtained by the AU method, but also the parameters of ultrasonic waves propagating perpendicular to the PCM layers at higher frequencies. Figure 1 shows the schemes of the control techniques proposed for use.
Figure 1. Measurement schemes of acoustic signal parameters; a – echo method, b – AU method, c — passage method; 1, 2 - source and receiver of ultrasonic waves.

As the receiver and source of ultrasonic vibrations, direct contact piezoelectric transducers are used. Using the echo technique and the transmission technique, the speed of ultrasonic wave’s propagation (C) and the signal amplitude (A) are determined perpendicular to the PCM layers. Moreover, control is carried out in the frequency range from 2 to 10 MHz. At higher frequencies, the sensitivity of the technique increases, but at the same time, the attenuation of the ultrasonic signal in the PCM increases. Therefore, the exact value of the working frequency is determined experimentally depending on the wall thickness, product shape, ultrasonic wave attenuation, acoustic contact and other factors.

There is a correlation between the physical and mechanical characteristics of PCM and the parameters of ultrasonic vibrations propagating in it, in particular, ultrasonic waves interact with pores: they scatter, bend around them, etc. With an increase in the number of pores, these interactions occur more and more, which leads to more attenuation and decrease of the speed of ultrasonic waves. Thanks to such relationships, the physical and mechanical characteristics of PCM can be determined from the previously constructed correlation dependencies of the diagnostic parameters on the parameters of the ultrasonic waves.

When controlled with the help of acoustic-ultrasonic technique, the velocity and amplitude of the ultrasonic waves are determined along the direction of reinforcement. The control is determined at frequencies from 0.2 to 1.5 MHz, the operating frequency is obtained experimentally for a specific fiberglass. Using the AU method, it is possible to determine the direction of layer reinforcement, the elastic and strength properties of PCM from the previously constructed correlation dependencies of the diagnostic parameter on the calculated SWF criterion. To obtain a multi-parameter correlation, one can calculate the SWF criterion, which contains information about the parameters of acoustic signals measured by various techniques. A promising direction is the construction of correlation dependencies of the PCM characteristics on the SWF criterion calculated from the spectral components of acoustic signals.

The proposed comprehensive ultrasonic technique for determining the physical and mechanical characteristics of the fiberglass can be outlined by the following steps:
1. Control of discontinuity (non-glued, delamination, cracks, etc.) by NDT techniques (impedance, thermovision, ultrasonic shadowing, computer radiation tomography, etc.);
2. Measurement of the parameters of ultrasonic waves in the direction perpendicular to the layers by the passage technique or echo one using direct probes at a frequency of 5-10 MHz;
3. Determination of the structural characteristics of PCMs according to the correlation dependencies on the SWF criterion;
4. Measurement of the ultrasonic waves parameters along the direction of reinforcement by the acousto-ultrasonics technique (if necessary, on a different sounding base) using a probe at a frequency of 0.2-1.5 MHz;
5. Determination of physical and mechanical characteristics of PCMs in the direction of reinforcement from preliminary constructed correlation dependencies of the control parameter of the SWF criterion. For each specific type of PCM, it is necessary to determine the most informative SWF criterion, taking into account the structural features of the material. The proposed techniques are implemented using commercially available equipment and can be used for evaluation and determination of the elastic, strength and structural parameters of PCM parts.

3. The results of experimental work

Using the AU examination technique, the direction of reinforcement can be determined. For experimental investigation of the proposed technique, two batches of 6 samples of 120×120×1.5 mm in size were made, which are the PCMs based on a phenol-formaldehyde binder reinforced with six layers of TS 8/3 K-TO quartz fabric. The first batch was unidirectional, the second one [0; +33; -33; +63; -63; 0] lay-up.

Using an ultrasonic flaw detector and broadband ultrasonic probes in the specimens, the velocities (C) and amplitudes (A) of ultrasonic waves were measured at a frequency of 500 kHz on the base of 100 mm at different angles (αo) to the direction of the reinforcement. The results obtained are presented in Figure 2. From the presented data, it is seen, that for all specimens the maximum of ultrasonic waves velocity are observed when αo corresponds to the direction of reinforcement (Figure 2, b, d). This can be explained that maximum of modulus of elasticity in the warp direction.

On the Figure 2 (d) it is seen that despite the change in the average velocity caused by different density and porosity of the specimens, the dependence of the velocity of ultrasonic waves in the direction of reinforcement is clearly visible. According to the data in Figure 2 (a, c), one can conclude that using the signal amplitude as an informative parameter can be applied only for unidirectional layering.

![Figure 2](image_url)

**Figure 2.** Velocity and amplitude of the ultrasonic signals; a, b - in specimens with unidirectional calculation; c, d - [0; +33; -33; +63; -63; 0] lay-up.

The density (ρ), porosity (ε), and binder content (γ) were determined on specimens of 20×10×1.5 mm size consisting of six layers of TS 8/3 K-TO fabric with [0; +33; -33; +63; -63; 0] lay-up with varying density at the manufacturing stage. The velocity of the ultrasonic waves were measured by the passage technique using a probe at a frequency of 5 MHz. The obtained dependences of the density, porosity
and content of the binder samples on the measured velocities of ultrasonic waves are presented in figure 3.

![Figure 3. Correlation dependence of the structural characteristics of PCM samples on the speed of ultrasonic waves;](image)

The resulting correlation equations:

\[
\rho = 0.00000005C^2 - 0.00009250C + 1.54227852
\]

\[R^2 = 0.95\]

\[\nu = -0.000004C^2 + 0.007974C + 17.229608\]

\[R^2 = 0.96\]

\[\gamma = 0.000003C^2 - 0.004693C + 24.828420\]

\[R^2 = 0.96\]

(2)

The determination of the elastic modulus in the direction of reinforcement was carried out on specimens 250×25×1.5 mm size having six layers of fabric TS 8/3 K-TO with a unidirectional lay-up. The ultrasonic wave velocity were measured with the aid of acoustic-ultrasonic technique on 120 mm base and frequency 1 MHz in the direction of the warp direction. The calculation of the dynamic modulus of elasticity was defined according to the relation [20]:

\[
E_D = \frac{\rho C^2 (1 + \nu) (1 - 2\nu)}{(1 - \nu)},
\]

(3)

where \(\nu\) - Poisson's ratio equal to 0.11 in the warp direction.

After measuring the velocities of ultrasonic waves in the specimens, the tensile modulus ET was determined according to GOST 11262-2017 (ISO 527-2: 2012). The results is presented in Figure 4.
Figure 4. Average values of elastic moduli in lots (lot size - 10 pcs).

The Figure 4 shows that the difference between the elastic moduli in the direction of reinforcement determined in tensile testing and the non-destructive ultrasonic technique does not exceed 15%, which indicates the possibility of using the proposed technique for assessing the elastic characteristics of PCMs structures.

4. Conclusions

To find the direction of PCMs reinforcement acoustic-ultrasonic technique was used. It has been experimentally confirmed the possibility to determine the direction of fiberglass reinforcement with varying degree of impregnation.

A technique for determining the structural characteristics of PCM (density, porosity, binder content) by an ultrasonic passage technique with determination coefficient from 0.94 to 0.96 was proposed and tested.

A technique for determining the elastic modulus of unidirectional PCMs in the direction of reinforcement based on the theory of acoustoelasticity was experimentally confirmed. The error in the elastic modulus determining did not exceed 15%.

To increase the accuracy of determining the elastic and strength characteristics in the direction of reinforcement in angle-ply lay-up fiberglass plastics, it is recommended to use the correlation dependencies of PCMs characteristics on the SWF criterion, which are calculated from the parameters of the acoustic signals that passed through the control object in different directions.

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