DIAGNOSIS OF PHYSICAL AND MECHANICAL PROPERTIES OF CONSTRUCTIONAL CARBON FIBER REINFORCED PLASTICS (CFRP)

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The most pressing problem of improving the reliability of the air-ship equipment is the problem of improving its quality as the main indicator for reliability assessing of high-duty assemblies. The control of the components and equipment assemblies reliability has gained considerable importance these days. It depends not only on the material properties but also on its latent structural inhomogeneities that lead to deviation of material physical-mechanical characteristics from the nominal values.

For diagnostics of polymer composites (PC) physical and mechanical properties diagnostics methods using acoustic, heat, electrical and other non-destructive testing methods are used but in other quality. Ultrasonic methods are the main of the named above (by the number of problems solved). These methods do not require bulky hardware and can be applied not only in laboratories but also in workshops and even in the field [1, 2].

Technical diagnostics methods are indirect and are based on correlation liaisons establishing between diagnostics parameters (material acoustic properties) and physical and mechanical PC characteristics by comparing results of destructive and non-destructive testing [3].

Diagnostics of physical and mechanical properties of non-metallic constructional materials (mainly fiberglass) has been studied for several decades already [4], but these methods are not widely used. The same is abroad and it is due to the low accuracy of physical and mechanical properties diagnostics by acoustic methods and instability of the results [5].

In VIAM there has been developed a new research field to diagnose physical and mechanical properties and composition of PCs to determine elastic and strength properties, porosity, density, content of matrix and filler, the degree of PC matrix polymerization in the airframe and other high-duty products by the laser - acoustic method of ultrasonic control by using the correlation equations [6, 7]. Their parameters are amplitude, temporal and spectral characteristics of acoustic signals that are defined directly in the construction without destruction. The informational content of the receivable acoustic signals that passed through material has been studied and complexation methods that provide better precision of physical and mechanical properties detection have been suggested. It is shown that for the increase of diagnostics parameters informational content it is reasonable to use the technique of full-range acoustic spectrum analysis in order to get frequency dependence of attenuation coefficient and ultra sound waves and these dependences transformation when material composition changed.

Use of laser excitation of elastic fluctuations impulses of no more than 0.05 ms allows to hold spectral analysis of sensed acoustic signals in a wide frequency band from 0.1 MHz to 10 MHz. This allows to increase accuracy and reliability of diagnostic properties of CP by a non-destructive method. It has been found out that the main criteria for selecting a single or complex parameter in the diagnostic evaluation of physical-mechanical characteristics and CP composition is the correlation coefficient (or correlation ratio), and the reliability criterion of this factor.

Both acoustic and physical and mechanical material properties are determined by material structure and composition that is why there is correlation between them. However the correlation ratio when using traditional diagnostics parameters (speed of ultrasonic oscillations impulses and their attenuation) is not always enough for practical use. That is why research and development in VIAM was aimed at finding crucially new informational CP properties diagnostics parameters that can be measured with high accuracy and veracity in a detail or in a CP construction without destruction. Also research and development in VIAM was aimed at solving the problem of diagnostics parameters complexation to establish closer correlation of diagnostics complex parameters with the CFRP definable parameters [8, 9].

To define density, porosity and CFRP composition the laser-acoustic method of ultrasound reflection method has been chosen [10, 11]. This non-destructive testing method is based on laser excitation of elastic fluctuations impulses in the object under control, receiving of impulses that passed through the object under control and were reflected from the surface of two medias separation that differ by characteristic impedance (from the borders of the objects under control or inhomogenieties), receiving the reflected impulses and received signals parameters analisys.

To receive powerful broad band sound impulses the laser thermooptical sound excitation has been used – the optico-acoustic effect [12]. The range and temporary form (and a frequency spectra accordingly) of the thermo-optically excitated ultrasound impulse is determined by the temporary correspondence of the laser impulse absorption intensity and thermo physical parameters of the absorbing medium (light absorption index, heat expansion index, heat conduction, thermal diffusivity). Herewith the range of the thermo-optically excitated ultrasound impulses can reach hundreds of MPa within the spectral range 0,1 – 20 MHz.

In figure 1 A-scan is shown. It is a temporary track of the signals that have passed through the object under control directly and indirectly. Here $2\Delta t = t_1 - t_0$, where t_1 is the time of the first bottom impulse indication; t_0 is the time of indicating an impulse reflected from the object under control surface.

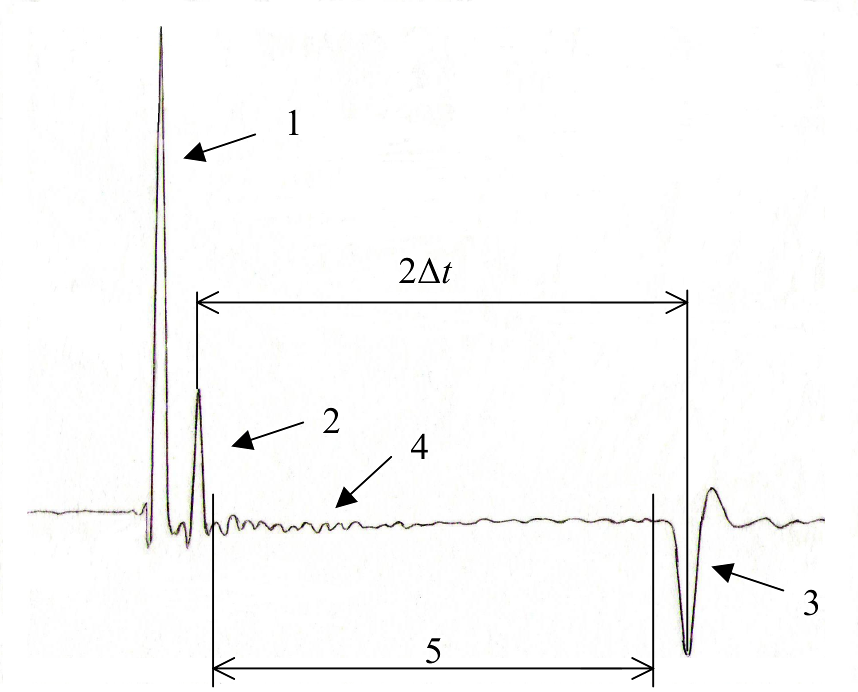

Figure 1 – Time signals (impulses) track upon their passing through the detail or construction material: 1 – OA-oscillator signal (has A amplitude); 2 – signal reflected from the object under control surface (has A_0 amplitude); 3 – bottom impulse – signal reflected from the object under control opposite side (has A_1 amplitude); $2\Delta t$ (microsecond) – time of signal passing through the material thickness directly and indirectly; 4 – structural noise appearing upon reflection from the material structure inhomogenuities mainly from pores; 5 – working range of the time track on which normed energy W_n of the structure noise is determined

The signal of the optoacoustic oscillator is partly reflected from the surface of the object under control, and partly goes through it and spresds in it as a sonic wave. The ultrasonic signal that spreads deep into the object under control, reaches its opposite side, then it is reflected and registered by receiving transducer. Besides, when spreading through the object under control material the signal disperses at the structure inhomogeneities. Part of this signal dispersed backwards reaches the receiver and is also registered with a time delay in time in regard to the reference signal coming. The time of the dispersed wave coming corresponds to the certain depth of the structure inhomogeneities bedding in the object under control (mainly pores). The frequency spectrum of the acoustic impulses (spectral range 1 – 10 MHz) dispersed backwards by all the thickness of the construction under control contains information about the structure inhomogeneity as a whole. This permits to carry out the ready-made CFRP details and constructions to diagnose their properties upon the one-side access is from one side.

The acoustic signal spectrum that is dispersed at the structure inhomogeneities consists of regular (smooth) and irregular (noise) components. The smooth part is determined by the spectrum sensitivity of the piezo-receiver in the acoustic transducer and can be detected by the smoothing of the real signal spectrum. Irregular part (as in the time form of the acoustic signal) is detected by dispersion at the CFRP structure inhomogeneities (primarily pores) and its full energy can be the feature of the medium porosity.

In VIAM there has been developed a technique to determine PC porosity by the laser-acoustic method of the ultrasound testing [13-15]. As a diagnostic parameter of the material porosity the full normed energy of the noise component is used:

$$W_n = \frac{\int_{f_{\min}}^{f_{\max}} [S(f) - S_o(f)]^2 df}{\int_{f_{\min}}^{f_{\max}} S_o^2(f) df}, \quad (1)$$

where: $S(f)$ – spectrum of impulses dispersed by the structure backwards; $S_0(f)$ – sound impulse spectrum; f – ultrasound fluctuations frequency; f_{min} and f_{max} – boundary frequencies of the spectral range.

In figure 2 the acoustic signal spectrum is shown in a sample with high pores content. Evidently the real signal spectrum has specific oscillations that are determined by structure noises due to porosity. Smooth curve is received by experimental spectrum approximation.

Normed noise component energy of the acoustic signal spread backwards is best correlated with material porosity and that is why this very characteristic has been chosen as a diagnostics parameter. CFRP porosity is defined by previously determined correlation of pores volume content V_p with the normed energy value W_n of the structure noise:

$$V_p = \varphi (W_n). \quad (2)$$

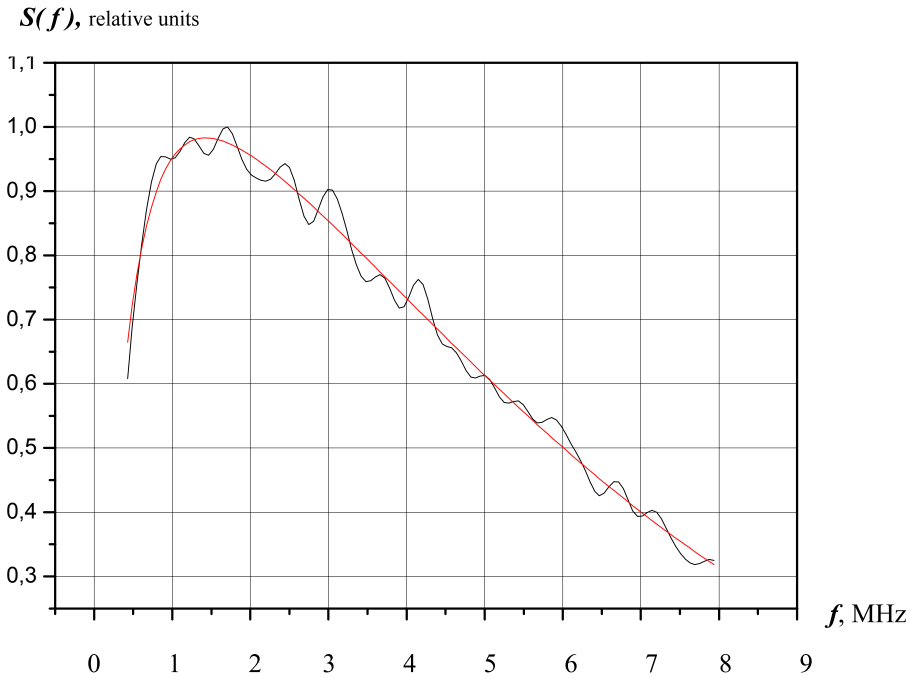

Figure 2 – Amplitude spectrum of the acoustic signal at the area 4 (see figure 1) in the porous CFRP sample:
—— - real spectrum; —— - smooth curve

In figure 3 energy W_n dependence of the acoustic signal noise spread backwards component in relative unit dependence on material volume porosity V_p in % is shown. There is rather close relationship between material porosity and noise component energy (correlation ratio R=0,95). This permits to use noise component energy as a diagnostics parameter that is determined by details and CPRF constructions non-destructive testing. The connection between volume material porosity with the structure noise energy is determined by the formula $V_p = 290{,}66\ W_n^{\ 2} + 0{,}2934\ W_n$.

The structure noise energy in the CFRP defect zone can by an order exceed energy in the flawless area. That is why it is impossible to carry out correct porosity diagnostics in the defect CFRP areas. So the CFRP diagnostics can be carried out only at those details and constructions that have successfully passed radiographic inspection and do not have any defects.

As a result the porosity connection with the structure noise energy in CFRPs with different matrix content has been received. It permits by the measured structure noise energy values to determine porosity and, what is extremely important, its low values (up to basis points).

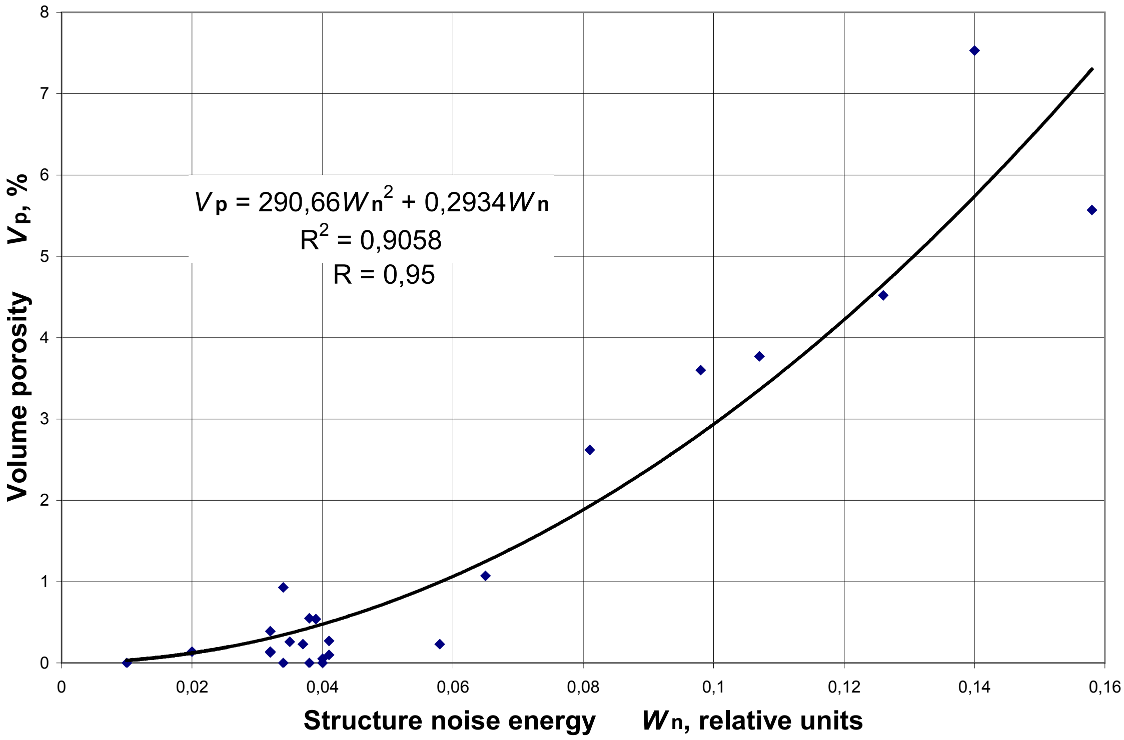

Figure 3 – Connection of the pores volume content V_p in CFRP VKU-17KE0.1 with the energy W_n of the structure noise

Structure noise energy W_n is determined for the preliminary porosity estimation. If the porosity is between 0 – 1 %, one can start determining reinforcing fiber content in the CFRP and CFRP porosity in accordance with the determined correlation relationship. If the material porosity is more than 1%, the error of the reinforced fabric volume content determination in CFRP and CFRP porosity as the increasing pores number in the material more and more affects the results of the diagnostics parameter determination. If the material porosity is more than 2% the fiber content determination and CFRP density by the suggested technique becomes incorrect.

Fiber content in CFRP is determined by the defined correlation relationship of the mass fiber content with the main diagnostics parameters:

$$M_F = \varphi_1 (c), \tag{3}$$

CFRP density ρ is determined when the material porosity in not more than 1 % by the determined correlation relationship of material density with the same diagnostics parameter:

$$\rho = \varphi_2 (c). \tag{4}$$

The main diagnostics parameter that is speed *c* of ultrasonic waves in CFRP is detected by the program method by the time Δt of the signal passing and the object under control wall thickness *h*:

$$c = 2h / \Delta t. \quad (5)$$

Thickness *h* of the CFRP construction wall in the control zone is determined by measuring instruments, ultrasonic gauge or by the construction drawing.

Let us view the example of the main diagnostics parameter changing – ultrasonic impulses speed *c* at filler content change in CFRP at constant material porosity. At filler content increase the impulses speed is growing as the ultrasound signal is passing the biggest part of the way through the material in which the speed of the ultra sound spreading is higher. At low material porosity (up to 1% by the volume) increase in the filler content will lead to material density increase and to the increase in ultrasound fluctuations impulses speed *c*.

In figure 4 the correlation of ultrasound fluctuations speed *c* with weight content M_F of carbon fiber in CFRP built for low-porosity samples is shown. In figure 5 correlation of ultrasound fluctuations speed *c* with CFRP density ρ is shown.

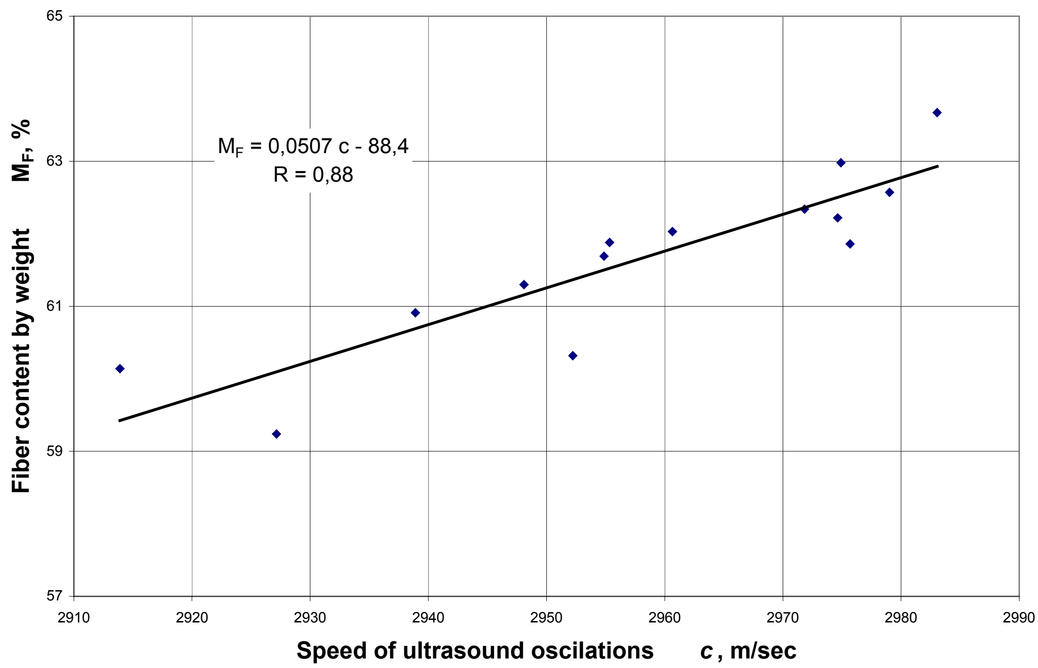

Figure 4 – Correlation of ultrasound fluctuations speed *c* with weight content M_F of carbon fiber in CFRP VKU-17KE0,1

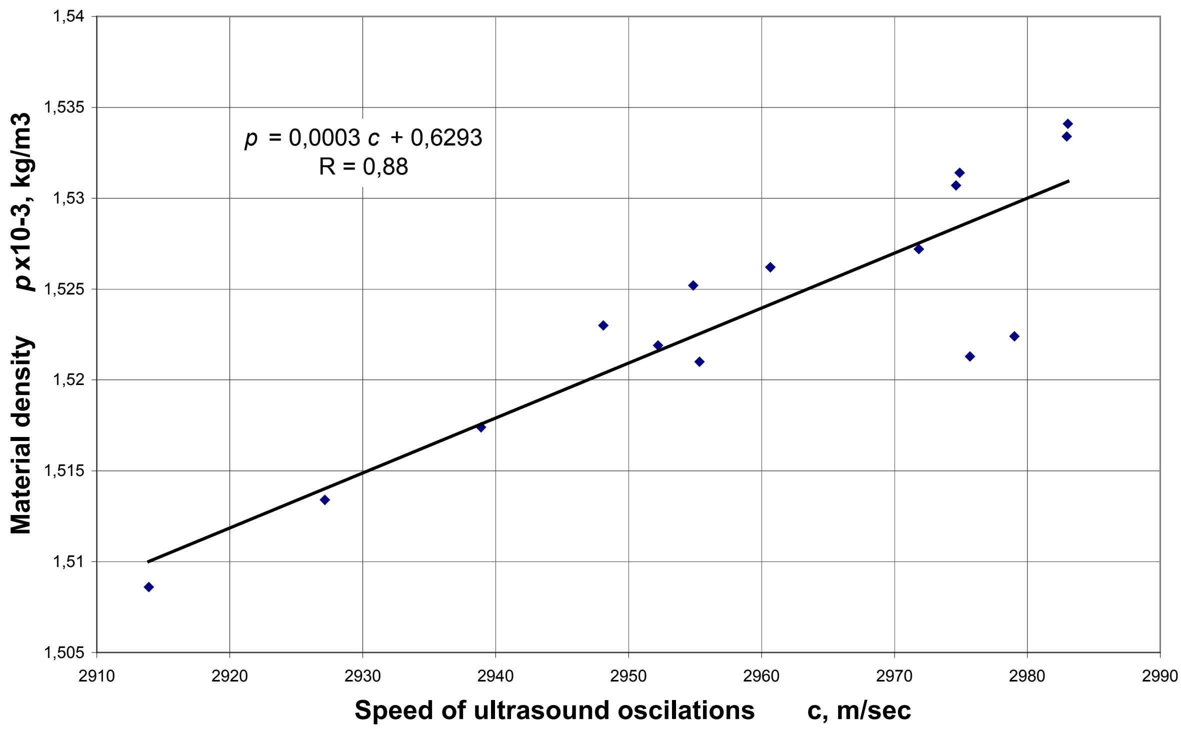

Figure 5 – Correlation of ultrasound fluctuations speed *c* with material density ρ CFRP VKU-17KE0,1

For low porosity samples the following regression equations and correlation indexes have been received V_p = 0 – 1 %:

– for correlation of ultrasound fluctuations speed *c* with the fiber weight content M_F:

$$M_F = 0{,}0507\, c - 88{,}4, \qquad (6)$$

correlation ratio R = 0,88;

– for correlation of ultrasound fluctuations speed *c* with material density ρ:

$$\rho = 0{,}0003\, c + 0{,}6293, \qquad (7)$$

correlation ratio R = 0,88.

Relative error of determining by the non-destructive method:

– CFRP porosity is not more than ±10 % (i.e. error in absolute values for porosity of 1% does not exceed ±0,1 %, so the porosity determination result is between 0,9 – 1,1 %);

– CFRP density is not more than ±1 % (i.e. error in absolute values for density 1500 kg/m^3 does not exceed ±15 kg/m^3, so the density determination result is 1485 – 1515 kg/m^3);

– fiber content in CFRP by weight is not more than ±4 % (i.e. error in absolute values at fiber weight content 60 % is not more than ±2,4 %, so the result of fiber volume content determination is 57,6 – 62,4 %).

When implementing CFRPs into large-dimensioned integral constructions the necessity to prepress details preliminarily using material heating occurred. In this case some adhesive polymerization takes place. The exceedence of prepressed half-finished products hardening degree leads to the lowering of elements connection strength in the integral construction.

To measure CFRP matrix polymerization degree [16, 17] the main diagnostics parameter is determined that is the joint parameter b_1 equal to the product of the signal A_0/A_1 weakening and time Δt of its passing through the material under control in the direction of its thickness:

$$b_1 = (A_0/A_1) \cdot \Delta t, \quad (8)$$

where A_0 – impulse amplitude reflected from the upper surface of the object under control (mV); A_1 – amplitude of the first bottom impulse i.e. signal amplitude reflected from the opposite wall surface of the object under control (mV); Δt – time of signal passing through the object under control gauge (microsecond):

$$\Delta t = (t_1 - t_0) / 2, \quad (9)$$

where t_1 – time of the first bottom impulse indication (microsecond); t_0 – time of impulse indication reflected from the upper surface of the object under control (microsecond).

Diagnostics parameter b_1 is calculated by the program method by the results of the primary diagnostics parameters definition results (A_0/A_1, Δt) during non-destructive testing.

The experiments prove that the parameter has been chosen correctly. Thus when increasing the material hardening degree both multipliers decrease and when decreasing the material hardening degree they increase. They change in one direction and their product amplifies these changes.

The matrix polymerization degree β in the object under control made of CFRP VKU-17KE0,1 is determined according to the graph built by the detected correlation of the main parameter b_1 with the matrix polymerization degree β at material porosity $V_p \leq 1$ % or by the equation of this connection:

$$\beta = \varphi (b_1). \quad (10)$$

The factor impeding the matrix polymerization degree β definition by the parameter mentioned above is the porosity that influences the dependency $b_1 = \varphi (\beta)$. To take into account the material porosity it is necessary to define the value of the normed energy W_n of the structure noise.

Correlation of the reaction groups conversion degree in PC matrix can be written as:

$$\beta = \varphi\,(b_1, W_n). \qquad (11)$$

In figure 6 the correlation of diagnostics parameter b_1 with the VKU-17KE0,1 matrix polymerization degree built for the samples with the low material porosity is shown. The following regression equation and correlation coefficient has been received:

$$\beta = 191{,}08 \cdot b_1^{-0{,}6063},\ R = 0{,}96. \qquad (12)$$

Mechanical as well as physical CFRP characteristics are mainly determined by physical and chemical components characteristics, their ratio, material porosity. Main material acoustic characteristics are also determined by these factors – ultrasound fluctuations speed and attenuation that are called diagnostics parameters as these parameters can be determined during non-destructive constructions testing. CFRP mechanical characteristics are determined by the defined correlation (statistic) connection with the diagnostics parameters.

The CPRF strength properties in the construction are determined by the defined correlation of the joint diagnostics parameter b_2 with the material strength τ_{sh} at interlaminar shear

$$\boldsymbol{\tau_{sh}} = \varphi_1(b_2) \qquad (13)$$

and material strength σ_{com} at pressure

$$\boldsymbol{\sigma_{com}} = \varphi_2\,(b_2), \qquad (14)$$

where b_2 – the joint diagnostics parameter equal the index k ratio to the time Δt of its passing in the direction of the construction wall thickness.

Coefficient k is defined by the program method by the signals amplitude characteristics and it is the opposite value to the signal attenuation in the material when passing through the object under control thickness. The program defines the k coefficient as:

$$k = A_1 / A, \qquad (15)$$

where A_1 is the bottom signal amplitude, i.e. signal amplitude reflected from the opposite wall surface of the object under control;

A is the signal amplitude that has entered the object under control.

The joint diagnostics parameter b_2 is calculated by the program method by the primary diagnostics parameters:

$$b_2 = k / \Delta t = A_1 / (A \cdot \Delta t), \qquad (16)$$

where Δt is the time of signal passing through the construction wall thickness.

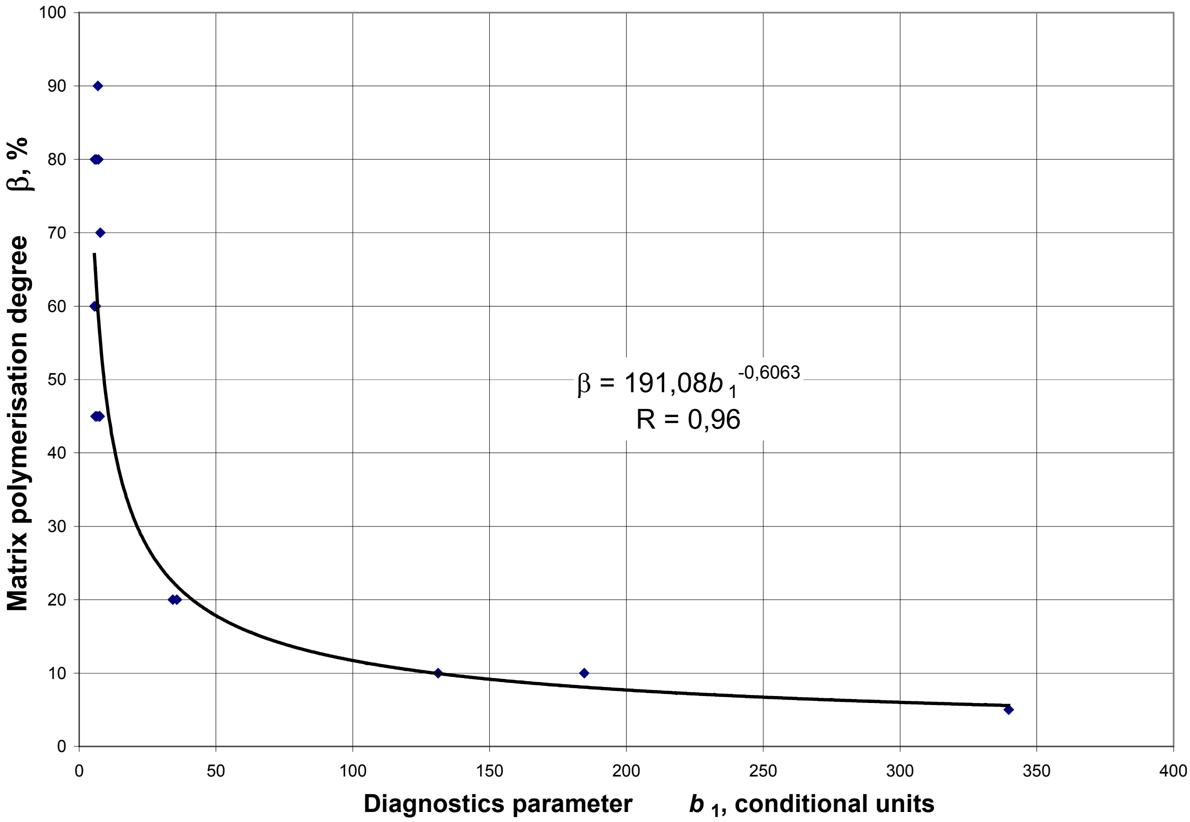

Figure 6 – Correlation of the diagnostics parameter b_1 with the CFRP matrix polymerization degree β

In the present work the laser excitation of elastic vibrations in the object under control is used. This allows to receive a very short impulse (impulse length is not more than 0,05 microsecond) providing significant advantages at spectral analysis as in the object under control the acoustic signal with a very wide spectrum is formed (within the range 0,1 – 10,0 MHz).

Let us study an example of the joint diagnostics parameter b changing when CFRP strength properties changing at constant and minimum material porosity. At material strength increase A_1 / A value will also grow and the time Δt of the signal passing through the wall thickness will be decreasing. As a result the joint diagnostics parameter will be increasing. At the material strength decrease A_1 / A value will be decreasing and the time Δt of the signal passing through the wall thickness will be increasing. As a result a result the joint diagnostics parameter will be decreasing.

During work performance the correlation of the joint diagnostics parameter with the strength at CFRP shift and compression has been determined. The experimental data has been electronically processed to determine the correlation ratio of the diagnostics parameter with the strength upon CFRP shift and compression and diagrams of the joint parameter b_2 (determined by the apparatus UDL-2M) connection with the strength at CFRP shift and compression (determined by the destructive method – material porosity is less than 1%) plotted.

In figure 7 the correlation of the diagnostics parameter b_2 with the strength upon the CFRP shift (built for the samples with the low material porosity – less than 1%) is shown and in figure 8 the correlation of the diagnostics parameter b_2 with the strength upon CFRP shift (also built for the samples with the low material porosity) is shown.

The following regression equations and correlation coefficients for the samples with the low porosity (V_p from 0 % to 1 %) have been received:

- for the correlation of the diagnostic parameter b_2 with the strength τ_{sh} upon CFRP shift:

$$\tau_{sh} = 306{,}6\, b_2 + 58{,}825, \qquad (17)$$

correlation ratio $R = 0{,}90$;

- for the correlation of the diagnostics parameter b_2 with the strength σ_{com} upon CFRP compression:

$$\sigma_{com} = 18300\, b_2 + 490{,}48, \qquad (18)$$

correlation ratio $R = 0{,}88$.

The error when using the method to determine the CFRP strength upon shift and compression does not exceed ±8 %.

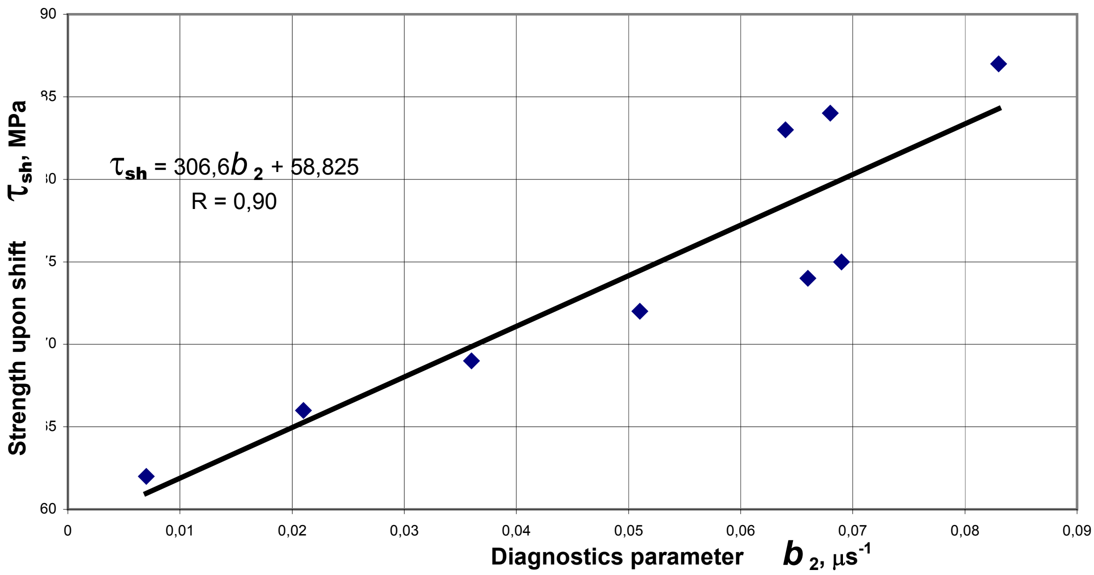

Figure – 7 Correlation of the joint diagnostics parameter b_2
with the strength τ_{sh} upon CFRP VKU-17KE0,1 shift

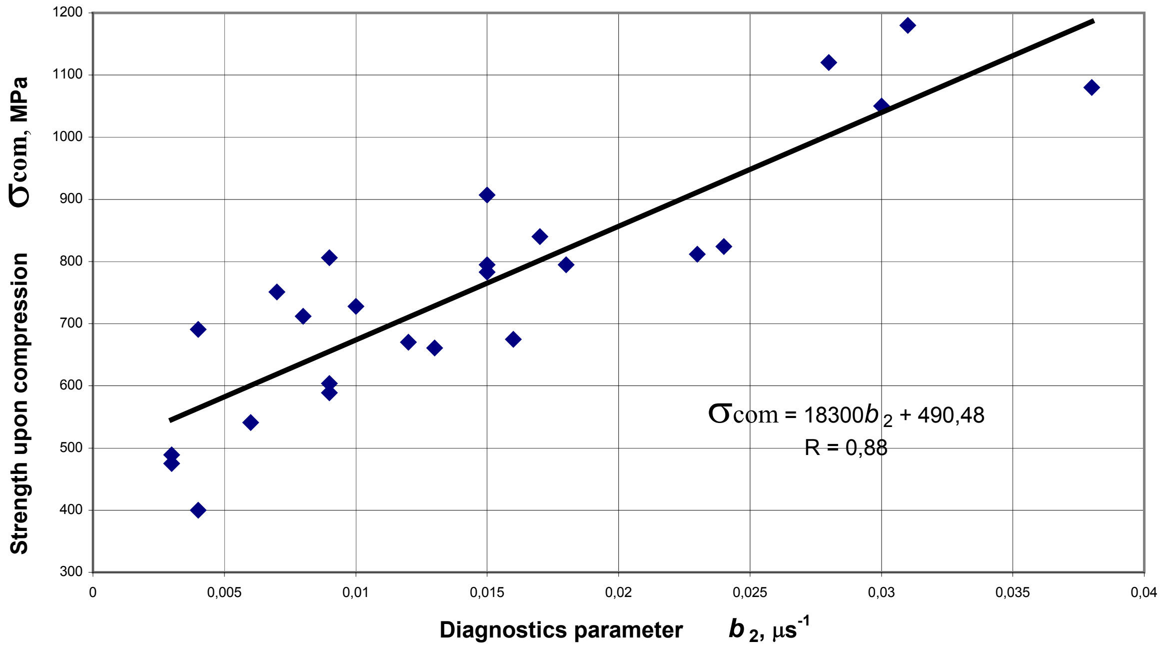

Figure 8 – Correlation of the joint diagnostics parameter b_2
with the strength σ_{com} upon CFRP VKU-17KE0.1 compression

I.e. a new approach to problems solving has been laid down in the PC diagnostics. It is based on laser oscillations excitation in the construction under control and on spectral analysis of the ultrasound oscillations impulses that have passed through the object under control and contain information about the structure and physical and mechanical properties of the CFRP. Use of the laser excitation of the elastic oscillations enables to receive very short impulses with the wide spectrum and to carry out the ultrasonic attenuation analysis in a rather wide range of frequencies. This permits to raise the accuracy and reliability of the structure diagnostics and CPRF characteristics by the non-destructive method.

As a result of works carried out in VIAM the control technologies have been developed launched methodological materials have been launched, technological recommendations and manufacturing instructions on porosity determination, density, fibre and matrix content in CFRP, have been developed as well as strength upon interlaminar shear and compression of unidirectional CFRPs, matrix polymerization degree at different stages of integral constructions production have been worked out.

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