MEASUREMENT OF LOCAL POROSITY OF PARTICLES REINFORCED COMPOSITES WITH THE LASER OPTOACOUSTIC METHOD

A.A. KARABUTOV, N.B. PODYMOVA, L.I. KOBELEVA*, T.A. CHERNYSHOVA*

INTERNATIONAL LASER CENTER, M.V. LOMONOSOV MOSCOW STATE UNIVERSITY
Leninskie Gory, Moscow 119991, Russia
e-mail: npodymova@mail.ru

*A.A. BAIKOV INSTITUTE OF METALLURGY AND MATERIALS SCIENCE
49 Leninsky pr., Moscow 117334, Russia

Abstract

In the present work we have proposed and realized experimentally the laser optoacoustic method for the measurements of local porosity of isotropic composite materials. It is based on measurements of phase velocities of thermooptically excited longitudinal acoustic waves in composite samples. The results of local porosity measurements (lateral resolution 1÷2 mm) for a number of aluminum alloy (silumin) matrix composite samples reinforced by *SiC* particles of a different mass concentration with the mean particle size of 14 μm coincide within relative inaccuracy 2-3% with the gravimetrical measurements of the average <*P*> value.

1. Introduction

The problem of nondestructive testing of the actual state of composite materials is of great importance because of essential decrease of their strength caused by defects and structural changes of a material that arise during exploitation [1]. In particular, fatigue changes of composite structure lead to the increase of the volume fraction of pores (porosity) that in turn leads to the decrease of material strength even by the absence of apparent defects. So the development of local porosity measurement methods is of great practical importance to provide valuable information for residual service life evaluation of composite products.

Typical structural fatigue changes lead to the change of attenuation and velocity of ultrasonic waves in composite materials. So one of the most widely used techniques of nondestructive testing of composites is the ultrasonic method [2]. It is based on the analysis of frequency dependencies of attenuation coefficient and phase velocity of acoustic waves in a material under study [3]. Composites are acoustically inhomogeneous materials, so to provide the quantitative evaluation of structural features one needs to carry out measurements of attenuation coefficient and phase velocity in a wide frequency band. This is due to the increase of scattering efficiency of ultrasonic waves, when its length becomes of the order of the structural inhomogeneities size. Besides the significant attenuation of ultrasound in composite materials requires the utilization of sources of powerful wideband acoustic signals. Therefore it is quite difficult to carry out the testing of samples and items of several centimeters thickness with conventional piezoelectrical transducers because of low efficiency of wideband acoustic signals excitation [4].

To overcome the difficulties of conventional ultrasonic techniques mentioned above we propose to employ the laser thermooptical excitation of ultrasound – pulse optoacoustic effect [5]. The main goal of the present work is the development of laser optoacoustic method of local porosity measurements of isotropic composite materials.

2. Investigated composite samples

In the present work a number of aluminum alloy (silumin) matrix composite samples reinforced by *SiC* particles of a different mass concentration was investigated. The samples have been manufactured by casting of molten silumin mixed with the certain amount of the fine powder of *SiC* particles with the mean size of 14 μm.

The porosity <*P*> averaged over the entire volume of the sample is expressed as:

$$< P >= (1 - r / r_0) \cdot 100\% \ , \qquad (1)$$

where the calculated density of the sample r_0 is determined with the known densities of the matrix – silumin r_{Al} = 2,735×10^3 kg/m^3, of the filler *SiC* r_{SiC} = 3,2×10^3 kg/m^3, and with the known mass concentrations n_{Al} and n_{SiC}; the actual density of the sample r is measured gravimetrically. The characteristics of the investigated samples are presented in the Table 1.

Table 1. *Characteristics of investigated samples*

sample #	Thickness *H*, mm	Mass concentration of components *n*		Calculated density r_0 ,×10^3 kg/m^3	Actual density r ,×10^3 kg/m^3	Averaged porosity <*P*>, %
		silumin	*SiC*			
1	10,70	1,0	0,0	2,735	2,714	0,77
2	10,18	0,962	0,038	2,750	2,710	1,45
3	10,98	0,923	0,077	2,766	2,665	3,65
4	4,72	0,845	0,155	2,798	2,660	4,93

The local porosity in different sites of a sample could be different because of nonuniform reinforcing fabrication method. To determine the local porosity *P* we use the theoretical model of ultrasound propagation in a porous metal [6], where the phase velocity of longitudinal acoustic waves in a material c_l depends on *P* as follows (at the values of *P* < 20%):

$$c_l^2 = c_{l_0}^2 \left(1 - P^{2/3}\right) \ , \qquad (2)$$

where c_{l_0} is the calculated with the two-phase medium model [7] phase velocity in the investigated site of a sample:

$$c_{l_0}^2 = \frac{1}{r_0^2}\left[\frac{n_{SiC}}{(r_{SiC}\, c_{l_{SiC}})^2} + \frac{n_{Al}}{(r_{Al}\, c_{l_{Al}})^2}\right]^{-1} . \qquad (3)$$

In the expression (3) $c_{l_{SiC}}$ = 11,82×10^3 m/s, $c_{l_{Al}}$ = 6,86×10^3 m/s are the known phase velocities in the filler and in the matrix [8, 9]. So to determine the local porosity *P* from the expression (2) we have to measure the phase velocity of longitudinal acoustic waves c_l in the corresponding site of the sample under study.

3. Experimental technique of phase velocity measurement of longitudinal acoustic waves

Experimental laser optoacoustic setup is shown in the Figure 1. A pulse of Q-switched Nd:YAG laser at the fundamental harmonic is absorbed in the specially designed optoacoustic (OA) source – the plate of optical filter blue-green glass, that causes the nonuniform non-stationary heating of subsurface layer of OA source and corresponding rise of the nonuniform mechanical stress. This in turn leads to excitation of the pulse of longitudinal acoustic waves (OA signal). The employment of Q-switched lasers and appropriate OA sources allows one to achieve the amplitude of OA signals up to hundreds of MPa in the frequency range up-to hundreds of MHz.

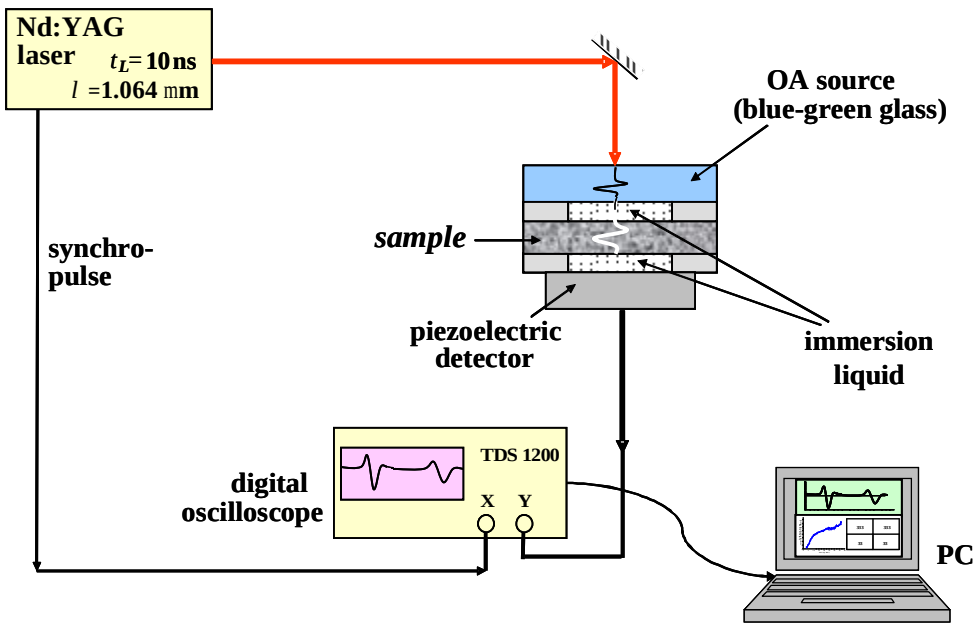

Figure 1. Laser optoacoustic setup

The OA signal excited in the OA source – the reference ultrasonic pulse – passes through an investigated sample and is detected with the specially designed wideband piezoelectric receiver

being in the acoustic contact with the sample through the immersion liquid (distilled water) (see Figure 1). The OA source, the sample and the receiver are mounted in the special OA cell.

Figure 2 shows the temporal profile and the amplitude spectrum of the reference ultrasonic pulse of blue-green glass OA source. The small duration and the large amplitude of the reference pulse allow to test the composite samples of a thickness from hundreds of microns to several centimeters, and also for strong scattering and absorptive materials (with the ultrasound attenuation coefficient value up to 10÷20 cm^{-1}).

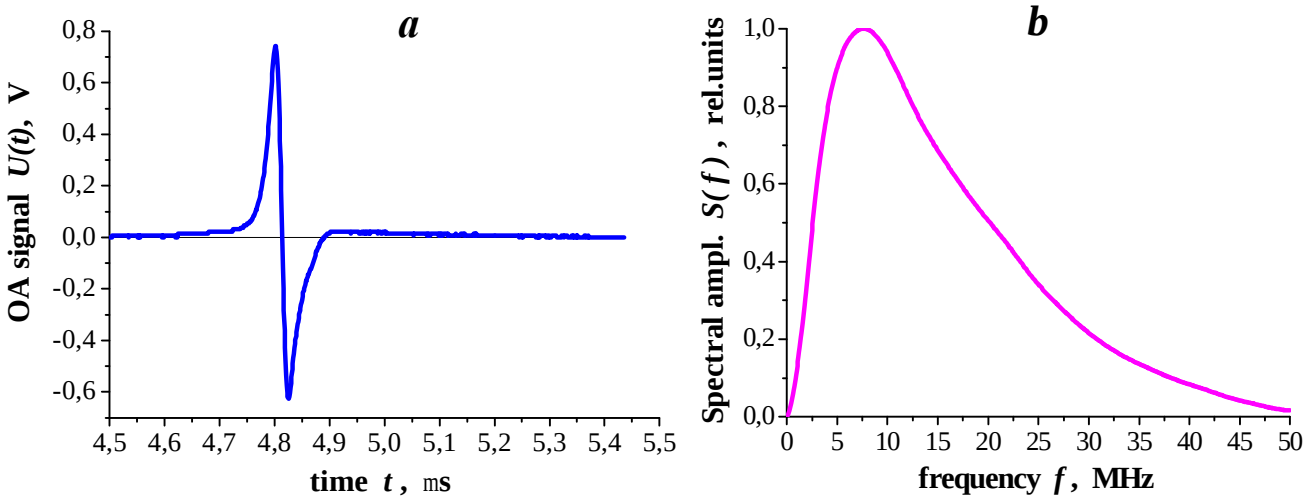

Figure 2. Temporal profile *(a)* and amplitude spectrum *(b)* of the reference ultrasonic pulse

The cross-section of the reference ultrasonic beam irradiated into the sample coincides with the mean diameter of the laser beam and is approximately 1,5 mm. This dimension determines the locality of testing in the lateral plane.

Electrical signals from the piezoelectric receiver come in the two-channel digital oscilloscope Tektronix TDS 1200 (analog bandwidth 100 MHz), the signal-to-noise ratio for the registration system is 50÷60 dB. The amplitude and phase spectra of acoustic signals are calculated with the standard code of the fast Fourier transform (FFT) taken into account the reflection coefficients of acoustic waves at the sample - immersed liquid interfaces in the optoacoustic cell and also the diffraction effect on the spectra of pulses. The frequency dependencies of the attenuation coefficient and the phase velocity of longitudinal acoustic waves in the sample are calculated in real time due to high signal-to-noise ratio and high stability of temporal profile of the reference ultrasonic pulses. The main characteristics of the laser optoacoustic system are presented in the Table 2.

Table 2. *Characteristics of the laser optoacoustic system*

Ultrasonic frequency band	0,5÷50 MHz
Amplitude of ultrasonic pressure	0,01÷10 MPa
Sample thickness	0,1÷70 mm
Lateral locality of testing	1÷2 mm

The absolute value and the dispersion of the phase velocity c_l of longitudinal acoustic waves in the sample of the known thickness H are determined from the measured phase spectra of the reference ultrasonic pulse, $j_0(f)$, and of the ultrasonic pulse passed through the sample, $j(f)$:

$$c_l(f) = \frac{2p\, fH}{j(f) - j_0(f)} \quad . \tag{4}$$

To enhance the accuracy of the phase velocity measurements it is useful to determine the phase difference of the ultrasonic pulse once passed through the sample, and of the pulse after the "tripple pass" in the sample – passed through the sample and reflected at the sample-immersed liquid interfaces. So the uncontrolled influence of possible difference in the liquid layers thickness could be eliminated by the mounting of OA cell for detection of the reference signal without the sample and the OA cell with the sample.

The absolute value of the phase velocity of longitudinal acoustic waves is determined as follows in the absence of the significant dispersion (the relative change of velocity doesn't exceed 5÷10% in the investigated frequency band 0,5÷50 MHz):

$$c_l = \frac{2H}{\Delta T_l} \quad , \tag{5}$$

where ΔT_l is the difference of arrival times to the receiver of the ultrasonic pulse once passed through the sample and of the pulse after the "tripple pass" in the sample. As an example the typical temporal profile of these two pulses in a silumin-matrix composite sample is shown in the Figure 3 [9]. The time interval ΔT_l is measured between the instants of transition through the "zero" from the compression to the rarefaction phases of the ultrasonic pulses. The attenuation of ultrasound affects on the duration of both the compression and the rarefaction phase and the "single pass" and the "triple pass" pulses cover a different distance in the sample. Therefore the duration of both the compression and the rarefaction phases will be different for these two pulses. As it follows from the theoretical model of the wideband OA signal propagation in an absorbing medium [5], just the propagation velocity of the "zero" point of the bipolar OA pulse is the most close to the acoustic wave phase velocity in the absence of the significant dispersion.

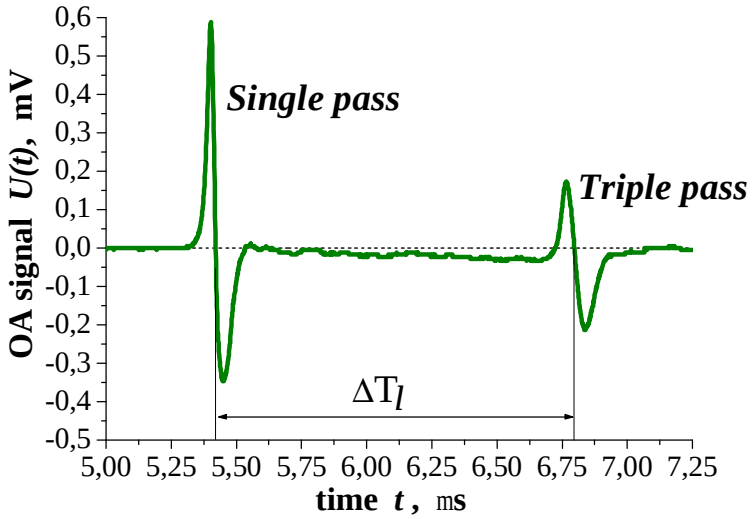

Figure 3. Typical temporal profile of the OA pulse of longitudinal acoustic waves once passed through the silumin-matrix composite sample and after the “tripple pass” in the sample [9].

The small duration of the longitudinal acoustic wave pulses provides sufficiently low relative inaccuracy of the phase velocity measurement: $d(c_l) \approx 0{,}5\%$. This value is governed basically by the relative inaccuracy of the sample thickness measurement: $\delta H \approx 0{,}5\%$, because of the value of ΔT_l (see Figure 3) is determined accurate within 2÷3 ns, that is of the order of $10^{-3}\,\Delta T_l$ for the sample thickness $H \sim 10$ mm.

4. Experimental results

Phase velocities of longitudinal acoustic waves in the composite samples under study were measured by the procedure described in the Section 3 and calculated with the expression (5). As the test samples with the known values of phase velocities of longitudinal acoustic waves a number of aluminum and copper samples have been studied. The experimental results of the optoacoustic measurement and the referenced data [8] for these samples are presented in the Table 3. The experimental results coincide within the inaccuracy limits with the referenced data. This fact confirms the reliability of the laser optoacoustic method for the measurement of the phase velocity of longitudinal acoustic waves in isotropic solid samples.

Table 3. *Comparison of optoacoustic measurement results and referenced data*

material	c_l, m/s	
	OA measurements	Referenced data [8]
Al	6280 ± 30	6260
Cu	4720 ± 20	4700

All composite samples were the discs of the diameter d = 40 mm. We determined the porosity in the center (P_1) and in the periphery (P_2) sites of each sample using the expression (2) with the measured values of the phase velocities in these sites c_{l1} and c_{l2} and the calculated value of c_{l_0} (3) for the given sample (see the Table 4). The relative inaccuracy of 0,5% of the phase velocity measurements leads to the relative inaccuracy of 2÷3% of porosity determination. The experimental results show (Table 4), that the increase of the mass concentration of the filler n_{SiC} leads to the growth of the sample porosity *P*. All investigated samples have quite uniform distribution of pores, the local porosity values coincide within the inaccuracy limits with the gravimetrical measurements of the average <*P*> value. The porosity in the center of each sample is slightly higher than in the periphery.

Table 4. *Results of optoacoustic measurements*

sample #	n_{SiC}	c_{l_0}, m/s	c_{l1}, m/s	c_{l2}, m/s	P_1, %	P_2, %	<*P*>, %
1	0,00	6860	6670	6730	1,30±0,03	0,77±0,02	0,77
2	0,038	6920	6720	6740	1,43±0,03	1,18±0,02	1,45
3	0,077	6990	6550	6590	4,24±0,09	3,69±0,07	3,65
4	0,155	7140	6660	6680	4,69±0,09	4,41±0,09	4,93

5. Conclusions

In the present work we have proposed and realized the laser optoacoustic method for local porosity measurement of isotropic composite materials. It can be applied also for any type of constructional materials (metals, alloys, plastics), when the size of pores is in the micron range. This method allows one to investigate samples of the thickness of 0,1÷70 mm with the lateral resolution of 1÷2 mm. The maximum relative inaccuracy of porosity measurement is 2÷3 %.

The proposed laser optoacoustic method may be useful by technological development and improvement of the materials fabrication process and to reveal the sites in a material with the less strength before manufacturing of items and products.

References

1. *Handbook of Composites* (ed. Herakovich C.T. and Tarnopol'skii Yu.M.). Elsevier Science Pbl., 1988, 165 ps.
2. Vary A. Ultrasonic measurements of material properties. // Research Techniques in Nondestructive Testing, 1980, v. 4, p. 160-204.

3. Fitting D.W., Adler L. *Ultrasonic spectral analysis for nondestructive evaluation.* Plenum Press, 1981, 354 ps.
4. Truell R., Elbaum C., Chick B. *Ultrasonic methods in solid state physics*. Academic Press, 1969, 308 ps.
5. Gusev V.E., Karabutov A.A. *Laser Optoacoustics.* AIP, 1993, 292 ps.
6. Polyakov V.V., Golovin V.A. Influence of porosity on ultrasonic wave velocity in metals. // Technical Physics Lett., 1994, v. 20, № 11, p. 54-57.
7. Zharkii S.M., Karabutov A.A., Pelivanov I.M., Podymova N.B., Timoshenko V.Yu. Laser ultrasonic study of porous silicon layers. // Semiconductors, 2003, v. 37, № 4, p. 468-472.
8. *Handbook of Physical Quantities* (ed. Grigoriev I.S., Meilikhov E.Z. and Radzig A.A.). CRC-Press, 1997, 1395 ps.
9. Karabutov A.A., Kobeleva L.I., Podymova N.B., Chernyshova T.A. Measurement of elastic moduli of particle reinforced composites with the laser optoacoustic method. // Zavodskaya Laboratoriya - Diagnostika Materialov, 2009, v. 75, № 3, p. 27-33 (in Russian).