Laser optoacoustic method of local porosity measurement of particles reinforced composites

N B Podymova¹, A A Karabutov², L I Kobeleva³ and T A Chernyshova³

¹Faculty of Physics, M.V. Lomonosov Moscow State University, Leninskie Gory, 119991, Moscow, Russia
²International Laser Center, M.V. Lomonosov Moscow State University, Leninskie Gory, 119991, Moscow, Russia
³A.A. Baikov Institute of Metallurgy and Materials Science, 49 Leninsky Prospect, 117334, Moscow, Russia

E-mail: npodymova@mail.ru

Abstract. In the present work we have realized experimentally the laser optoacoustic method for the measurements of local volume fraction of air pores (porosity $P$) of isotropic composite materials. It is based on measurements of phase velocities of thermooptically excited longitudinal ultrasonic waves in composite samples in the frequency range $0.5 \div 50$ MHz. The local porosity (lateral resolution $1 \div 2$ mm) is determined using the dependence of phase velocity of longitudinal acoustic wave on porosity for porous metals and theoretical calculation of phase velocity in a composite with the two-phase medium model. 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 $\mu$m was investigated. The samples were disks with the diameter $d = 40$ mm and the porosity in the center and in the periphery area of each sample was determined. The increase of mass concentration of the filler SiC leads to the growth of $P$. The results coincide within relative inaccuracy of 2-3% with the gravimetrical measurements of average $<P>$ value, the porosity in the center of each sample was slightly higher than in the periphery.

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.

The structural fatigue alteration leads to the change of attenuation and velocity of ultrasonic waves in composite materials. So the most widely used technique of its nondestructive testing is the ultrasonic one [2-3]. Composites are acoustically inhomogeneous, 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. Besides the significant attenuation of ultrasound in composite materials requires the utilization of sources of powerful wideband acoustic signals.
Therefore it is difficult to carry out the testing of samples and items of several centimeters thickness with conventional piezoelectrical transducers because of low efficiency of wideband signals excitation and of technical problems to provide the uniform frequency response of an ultrasonic source and detector in a wide spectral band [4]. The latter feature could reduce the dynamic range by testing of materials with the strong frequency dependence of ultrasound attenuation.

To overcome these difficulties 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 and brief theoretical analysis

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:

\[
< P > = (1 - \rho / \rho_0) \cdot 100\%
\]

(1)

where the calculated density of the sample \( \rho_0 \) is determined with the known densities of the matrix – silumin and the filler SiC, and with its known mass concentrations; the actual density of the sample \( \rho \) is measured gravimetrically.

The local porosity in different areas 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 \( P < 20\% \)):

\[
c_l^2 = c_0^2 \left(1 - P^{2/3}\right)
\]

(2)

where \( c_0 \) is the calculated with the two-phase medium model [7] phase velocity in a sample:

\[
c_0^2 = \frac{1}{\rho_0^2} \left[ \frac{n_{SiC}}{(\rho_{SiC} c_{SiC})^2} + \frac{n_m}{(\rho_m c_m)^2} \right]^{-1}
\]

(3)

In the equation 3 \( n_m \) and \( n_{SiC} \) are the mass concentrations of silumin and SiC in the sample under study, \( \rho_m = 2.735 \times 10^3 \text{ kg/m}^3 \), \( \rho_{SiC} = 3.2 \times 10^3 \text{ kg/m}^3 \), \( c_m = 6.86 \times 10^3 \text{ m/s} \), \( c_{SiC} = 11.82 \times 10^3 \text{ m/s} \) are the known densities and phase velocities of longitudinal acoustic waves for the matrix and the filler [8, 9]. So to determine the local porosity \( P \) from the equation 2 we have to measure the phase velocity of longitudinal acoustic waves \( c_l \) in the corresponding area of the sample under study.

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

We used the experimental setup (figure 1) for optoacoustic investigations in the transmission mode [7, 9]. A pulse of Q-switched Nd:YAG laser at the fundamental harmonic (\( \lambda = 1.064 \mu\text{m} \)) is absorbed in the specially designed optoacoustic (OA) source – the plate of optical filter blue-green glass, that leads to excitation of the pulse of longitudinal acoustic waves (OA signal). The acoustic signal excited in the OA source – the reference ultrasonic pulse – passes through an investigated sample and is detected with the specially designed wideband piezoelectric detector being in the acoustic contact with the sample through distilled water. The OA source, the sample and the detector are mounted in the special cell. The small duration (\( \approx 200 \text{ ns} \)) and the large amplitude (up to several of MPa) of reference ultrasonic pulses allow to test both thin composite samples (the thickness from hundreds of microns) and samples of the thickness up to several centimeters of strong ultrasound scattering and absorptive
materials. 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 detector come into the two-channel digital oscilloscope (analog bandwidth 100 MHz), the signal-to-noise ratio for the registration system is 50–60 dB. The dispersion of the phase velocity of longitudinal acoustic waves is calculated in real time due to high signal-to-noise ratio and high stability of temporal profile of reference ultrasonic pulses.

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 5–7\% in the investigated frequency band 0.5–50 MHz):

\[ c_l = \frac{2H}{\Delta T_l}. \]

where \( H \) is the known thickness of the sample, \( \Delta T_l \) 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” – passed through the sample and reflected at the sample - immersed liquid interfaces. So the uncontrolled influence of 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.

As an example the typical temporal profile of these two pulses in a silumin matrix composite sample is shown in figure 2 [9]. The time interval \( \Delta T_l \) is measured between the instants of transition through the “zero” from the compression to the rarefaction phases of the ultrasonic 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. The small duration of the longitudinal acoustic wave pulses provides sufficiently low relative inaccuracy of the phase velocity measurement: \( \delta c_l = 0.5\% \). This value is governed basically by the relative inaccuracy of the sample thickness measurement: \( \delta H = 0.5\% \), because of the value of \( \Delta T_f \) (see figure 2) is determined accurate within 2±3 ns, that is of the order of \( 10^3 \Delta T_f \) for the sample thickness \( H \sim 10 \text{ mm} \).

4. Experimental results and discussion
As the test samples a number of aluminum and copper samples have been studied. The experimental results: 

\[
\begin{align*}
    c_{l, Al} &= (6.28 \pm 0.03) \times 10^3 \text{ m/s}, \\
    c_{l, Cu} &= (4.72 \pm 0.02) \times 10^3 \text{ m/s}
\end{align*}
\]

coincide within the inaccuracy limits with the reference data [8]. This fact confirms the reliability of the laser optoacoustic method for the measurement of the phase velocity of longitudinal acoustic waves in isotropic solids.

All composite samples were the discs of the diameter \( d = 40 \text{ mm} \). We determined the porosity in the center (\( P_1 \)) and in the periphery (\( P_2 \)) areas of each sample using the equation 2 with the measured values of the phase velocities in these areas \( c_{l,1} \) and \( c_{l,2} \) and the calculated value of \( c_{l,0} \) (the equation 3) for the given sample (see table 1). 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, 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.

| sample # | \( n_{SiC} \) | \( c_{l,0} \), m/s | \( c_{l,1} \), m/s | \( c_{l,2} \), m/s | \( P_1 \), % | \( P_2 \), % | \( <P> \), % |
|---------|----------------|-----------------|-----------------|-----------------|-------------|-------------|--------------|
| 1       | 0.00           | 6680            | 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         |

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

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