The Elastic Moduli Measurement of High-Melting Nanoparticles Modified Metal Matrix Composites with the Laser Optoacoustic Method

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Abstract
In the present work we proposed and realized the laser optoacoustic method of local elastic moduli measurement of isotropic metal-matrix composite materials. The experimental setup allows to carry out the measurements of phase velocities of longitudinal and shear acoustic waves in the frequency band 0.5÷50 MHz. The elastic moduli are calculated with the elasticity theory from the measured phase velocities. The investigated composite samples were manufactured by reactive cast of aluminum with titanium microparticles (07 series) and the mixture of titanium and synthetic diamond nanoparticles added into the melt (09, 010 series). The elastic moduli of 07 series samples grow with the increase of the melt dwell time before sample forming. The elastic moduli of 09, 010 series samples are 15-20% less than that of the aluminum matrix. So the laser optoacoustic method can be helpful in choosing of the optimal conditions of materials manufacturing with enhanced strength properties.

Keywords: laser optoacoustics, ultrasound, elastic moduli, composite materials, phase velocity

1. Introduction
The problem of nondestructive testing of composite materials is very urgent, because structural changes arising during materials manufacturing and exploitation can significantly reduce their strength. For example, decrease of elastic moduli can be up to 20% [1] through the aging process. Therefore it’s very important to develop new operative nondestructive methods, that allow to control the local changes of strength properties of items in a process of manufacturing and exploitation. Particularly the determining of service life of components is very important. In the present work we proposed and realized experimentally the method of local elastic moduli measurement of isotropic metal matrix composite materials based on laser excitation and piezoelectric detection of ultrasound [2] (the laser optoacoustic method). It provides the measurements of phase velocities of longitudinal and shear acoustic waves in a sample study and calculation of the Young’s, shear moduli and Poisson’s ratio according to the elasticity theory. Its main advantage over the conventional ultrasonic methods (using piezoelectric oscillators [3]) is the effective generation of short and powerful probe acoustic pulses that is necessary for testing of heterogeneous composite materials with strong absorption of ultrasound.

2. The principle of laser thermo-optical excitation of ultrasound – optoacoustic effect
The principle of laser thermo-optical excitation of ultrasound is schematically shown in the Figure 1. The laser beam falls on the absorbing medium front surface normally. The absorbing medium is called optoacoustic (OA) source. Nonuniform non-stationary heating of near-surface region occurs during the absorption of the laser pulse. This in turn causes mechanical stresses in the subsurface region of the absorbing media, so longitudinal acoustic waves pulses spread into the absorbing medium \( p(\tau) \) and the transparent medium \( p_{tr}(\tau_{tr}) \), where \( \tau=\tau_0 \) and \( \tau_{tr}=t+z/c_{0tr} \) are the times in coordinate systems moving...
with the corresponding velocities. The velocities of longitudinal acoustic waves in the absorbing medium and the transparent medium are \( c_0 \) and \( c_{0tr} \).

![Diagram](image)

**Figure 1.** The principle of laser optoacoustic effect

The amplitude and the temporal form of laser-excited ultrasound pulse (OA signal) is governed by the time dependence of the laser pulse intensity and thermophysical parameters of the absorbing medium, such as light absorption and thermal expansion coefficients and heat capacity. By absorption of Q-switched laser pulses (\( \tau_L \) is of the order of several nanoseconds, the pulse energy is \( 10 \div 20 \) mJ) the amplitude of OA signals can reach hundreds of bars in a frequency range from hundreds of KHz to hundreds of MHz [2]. So the OA effect allows producing powerful wideband ultrasound signals and its application in the systems of ultrasonic nondestructive testing and evaluation of mechanical properties of composite materials is very promising.

### 3. Experimental setup

The experimental laser optoacoustic setup is shown in the Figure 2.

![Setup Diagram](image)

**Figure 2.** Laser optoacoustic setup

A pulse of Nd:YAG laser at the fundamental harmonic is absorbed in the OA source – the plate of optical filter blue-green glass. The ultrasonic signal excited in the OA source – the reference ultrasonic pulse – passes through an investigated sample and is registered with the
specially designed wideband piezoelectric detector being in the acoustic contact with the sample through the immersion liquid (distilled water). The OA source, the sample and the detector are mounted in the special OA cell. The typical size 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 size determines the locality of testing in the lateral plane. Electric signals from the piezoelectric detector come in the two-channel digital oscilloscope Tektronix TDS 1200 (analog bandwidth 100 MHz), the signal-to-noise ratio of the registration system is 50÷60 dB. The main characteristics of the laser optoacoustic system are presented in the Table 1.

### Table 1. Characteristics of the laser optoacoustic system

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultrasonic frequency band</td>
<td>0.5÷50 MHz</td>
</tr>
<tr>
<td>Amplitude of ultrasonic pressure</td>
<td>0.01÷10 MPa</td>
</tr>
<tr>
<td>Sample thickness</td>
<td>0.1÷70 mm</td>
</tr>
<tr>
<td>Lateral locality of testing</td>
<td>1÷2 mm</td>
</tr>
</tbody>
</table>

4. **Longitudinal and shear acoustic waves phase velocities measurements and elastic moduli calculation**

The Figure 3 is the example of temporal profile of the reference ultrasonic pulse and the ultrasonic pulse once passed through a silumin matrix composite sample [4].

![Figure 3](image)

Figure 3. Temporal profile of the reference ultrasonic pulse and the ultrasonic pulse once passed through a silumin matrix composite

The absolute value of the phase velocity of longitudinal acoustic waves is determined as follows in the absence of the significant dispersion (the relative velocity change doesn’t exceed 5÷10%):

\[
    c_L = \frac{2H}{\Delta T_L},
\]

(1)
where $H$ is the sample thickness, $\Delta T_L$ is the interval between arrival times to the detector of the reference ultrasonic pulse and the ultrasonic pulse once passed through the sample (see the Figure 3). The value $\Delta T_L$ is measured between the instants of the transition through the “zero” from the compression to the rarefaction phases of the ultrasonic pulses. It is known from the theoretical model of the wideband OA signal propagation in an ultrasound absorbing medium [2], just 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.

For the measurement of shear acoustic wave phase velocity $c_s$ with OA method a laser pulse is absorbed directly in the sample with acoustically free surface [5-8]. It occurs without any damage of the sample. The example of temporal profile of an acoustic signal which is caused by absorption of a laser pulse in a silumin matrix composite sample [4] is shown in the Figure 4.

![Figure 4. Temporal profile of acoustic signal caused by absorption of a laser pulse in an aluminum matrix composite sample](image)

The exciting pulse of longitudinal acoustic waves $L$ is unipolar (compression). Rerefraction phase is caused by diffraction of ultrasound in the sample. The pulse of shear acoustic waves appears when $L$ pulse reflects from the irradiated acoustically free surface of the sample (air – sample interface). Detected waveform $S$ (Figure 4) is governed by the acoustic field of a shear wave which passes through the sample and transforms into a longitudinal one when transiting the sample-liquid interface. Not only the radiation pattern of the excited shear wave pulse influences on the profile of $S$ pulse but also the dependence of its transformation coefficient on the angle of incidence towards the sample-liquid interface and on the finite aperture of piezoelectric detector. Therefore the detected $S$ pulse is significantly longer in comparison with the $L$ pulse. The arrival of the negative peak of the $S$ pulse is determined by the propagation time of the shear acoustic wave through the sample. Pulses going after the $S$ pulse are reflections of $L$ pulse in the sample and in the immersion liquid layer. The shear wave phase velocity is determined with the interval $\Delta T_{SL}$ of arrival times to the detector of the maximum of $L$ pulse and of the minimum of $S$ pulse:

$$c_s = \frac{H}{(\Delta T_{SL} + H/c_L)}.$$  

(2)
The short duration of longitudinal and shear acoustic waves pulses provides a sufficiently high relative accuracy of phase velocities measurements: \( \delta(c_L)/c_L \approx 0.5\% \), \( \delta(c_S)/c_S \approx 1.5 \pm 2\% \). The first one is mainly determined by the measurement error of the sample thickness \( \delta H/H \approx 0.5\% \), the second one is mainly determined by the relative accuracy of \( \Delta T_{SL} \) measurement (localization of \( S \) pulse minimum, Figure 4): \( \delta(\Delta T_{SL})/\Delta T_{SL} \approx 1.5 \pm 2\% \). The Young’s, shear moduli and Poisson’s ratio are calculated with the expressions:

\[
E = \rho c_s^2 \left( \frac{3c_L^2 - 4c_s^2}{c_L^2 - c_s^2} \right),
\]

\[
G = \rho c_s^2,
\]

\[
\nu = \frac{c_L^2 - 2c_s^2}{2(c_L^2 - c_s^2)},
\]

where \( \rho \) is the density of the sample determined from the results of samples weighing in air. The maximum relative errors of elastic moduli measurements with the laser optoacoustic method are governed by the accuracy of phase velocities measurements: \( \delta E/E = 6\% \), \( \delta G/G = 4\% \), \( \delta \nu/\nu = 5\% \).

5. Investigated samples

The investigated samples are the isotropic polycrystalline composite materials based on the aluminum matrix mixed with titanium particles with the average diameter of 300 microns in the volume concentration of 3\% (07 series) or with titanium particles and synthetic diamond particles with the average diameter of 50 nm (09 series) and 150 nm (010 series) in the volume concentration of 0,25\%. The sample #070 is aluminum of 99,99\% purity. All samples were manufactured by reactive cast with adding of fillers particles into the melted matrix. The melt dwell time was changed from 2 to 90 minutes. As a result of the Al-Ti chemical reaction the intermetallic inclusions with predominance of Al3Ti-phase were formed.

Figure 5. The increase of Al3Ti volume concentration in the investigated composite samples
The structure, chemical and phase composition of the samples were investigated by optical and scanning electron microscopy [9]. The increase of the volume concentration of Al$_3$Ti intermetallic phase depending on the melt dwell time of the material is shown in the Figure 5. These intermetallic inclusions affect the elastic moduli of the obtained composite material because of difference in mechanical properties of the matrix and intermetallic phases. Also during the casting process air pores may appear, that causes decrease of elastic moduli and strength of manufactured composite materials. The volume concentration of pores (porosity $P$) averaged over the sample volume is calculated with the expression:

$$P = (1 - \rho / \rho_0) \cdot 100\%,$$

where $\rho_0$ is the predictable density of the sample, calculated from the known densities $\rho_i$ and volume concentrations $n_i$ of the composite material components:

$$\rho_0 = \sum_i (\rho n)_i,$$

where the index «$i$» indicates corresponding component: Al$_3$Ti, unreacted titanium Ti, diamond particles D and aluminum Al.

The parameters of investigated composite materials are shown in the Table 2. All samples were prepared as the discs of the diameter $d = 2.5$ cm and of the thickness $H = 0.5 \div 1.2$ cm. It is significant to notice, that there is no possibility for such geometry to produce uniaxial tension or compression stress in samples for the conventional mechanical measurement of Young’s modulus.

**Table 2. Parameters of investigated samples**

<table>
<thead>
<tr>
<th>Melt well time, min</th>
<th>07 series Modifier Ti</th>
<th>09 series Modifiers Ti, D (50 nm)</th>
<th>010 series Modifiers Ti, D (150 nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$#_0$</td>
<td>$\rho$, g/cm$^3$</td>
<td>$P$, %</td>
</tr>
<tr>
<td>2</td>
<td>071</td>
<td>2,702</td>
<td>1,8</td>
</tr>
<tr>
<td>20</td>
<td>072</td>
<td>2,657</td>
<td>3,5</td>
</tr>
<tr>
<td>60</td>
<td>073</td>
<td>2,694</td>
<td>2,4</td>
</tr>
<tr>
<td>90</td>
<td>074</td>
<td>2,754</td>
<td>0,3</td>
</tr>
</tbody>
</table>

6. Experimental results

As the reference sample the pure aluminum (#070) was used. Measured values of longitudinal and shear acoustic wave phase velocities and elastic moduli coincide within the margins of errors with the reference data for aluminum [10]. This fact confirms the reliability of the laser optoacoustic method for the measurement of the phase velocity and elastic moduli.
Table 3. Comparison of OA measurements results and reference data for the sample #070

<table>
<thead>
<tr>
<th></th>
<th>$c_L$, ( \times 10^3 ) m/c</th>
<th>$c_s$, ( \times 10^3 ) m/c</th>
<th>$E$, GPa</th>
<th>$G$, GPa</th>
<th>$\nu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference data for Al</td>
<td>6.26</td>
<td>3.08</td>
<td>69±72</td>
<td>25±26.5</td>
<td>0.31±0.33</td>
</tr>
<tr>
<td>The results of OA</td>
<td>6.28±0.03</td>
<td>3.13±0.05</td>
<td>71±3</td>
<td>26±1</td>
<td>0.34±0.01</td>
</tr>
</tbody>
</table>

The results of OA measurements of elastic moduli of all investigated composite samples are shown in the Figure 6. The values of elastic moduli of 07 series samples increase with the growing of melt dwell time. This is due to increase of volume concentration of Al₃Ti with higher elastic moduli values and decrease of material porosity (Table 2). So the intermetallic hardening of material takes place. The values of elastic moduli of 09 and 010 series samples don’t exceed the values of aluminum for all dwell times within the margins of errors. The probably reason is the porosity growth in the samples (Table 2). Relatively high porosity (5-9%) can appear because of the addition of diamond particles, which act as local centers of pores formation.

Figure 6. The results of OA measurements of composite samples elastic properties
6. Conclusions

Local elastic moduli of high-melting particles reinforced isotropic composite samples were measured with the laser optoacoustic method with the high accuracy. The thickness and the diameter of samples can be in all proportions. Elastic moduli of aluminum-matrix composite material with intermetallic phase Al₃Ti particles increase with the rise of melt dwell time, that is the strengthening of the material. However there is no strengthening by adding the diamond nanoparticles in melt, because they act as the local centers of pores formation, that in turn leads to decrease of elastic moduli values. The developed laser optoacoustic method enables one to analyze experimentally the influence of the chemical composition, sizes and the concentration of reinforced particles on elastic properties of isotropic composite materials.

References