

Laser-Ultrasonic Method for Measuring the Local Elastic Moduli of Porous Isotropic Composite Materials

Alexander A. Karabutov, Natalia B. Podymova, Elena B. Cherepetskaya, Vladimir A. Makarov, Yulia G. Sokolovskaya

Abstract—The laser-ultrasonic method is realized for quantifying the influence of porosity on the local Young's modulus of isotropic composite materials. The method is based on a laser thermo-optical method of ultrasound generation combined with measurement of the phase velocity of longitudinal and shear acoustic waves in samples. The main advantage of this method compared with traditional ultrasonic research methods is the efficient generation of short and powerful probing acoustic pulses required for reliable testing of ultrasound absorbing and scattering heterogeneous materials. Using as an example samples of a metal matrix composite with reinforcing microparticles of silicon carbide in various concentrations, it is shown that to provide an effective increase in Young's modulus with increasing concentration of microparticles, the porosity of the final sample should not exceed 2%.

Keywords—Laser ultrasonic, longitudinal and shear ultrasonic waves, porosity, composite, local elastic moduli.

I. INTRODUCTION

PRODUCTION of Composite Materials (CMs) is a promising direction for obtaining structural materials with improved mechanical characteristics. These materials have heterophase structure; they may have some porosity (volume concentration of gas pores) already in production. Fatigue-induced changes in material structure during operation of products made of CMs can lead to an increase in porosity [1]. It is known that porosity has a significant effect on the elastic and strength properties of structural materials [2]). The occurrence of porosity in the casting production of metal matrix CMs may result in that the elastic modulus of the produced material will be smaller than that of the original matrix, even with increasing concentration of high-modulus reinforcing particles [3], [4]. Thus, the development of rapid nondestructive methods for quantifying the effect of porosity on the elastic moduli of CMs is of great practical value (improvement of the manufacturing technology of CMs, estimation of the residual life of materials during operation of products, etc.).

A. A. Karabutov, E.B. Cherepetskaya, Yulia G. Sokolovskaya are with National University of Science and Technology "MISIS" and International Laser Center of M.V.Lomonosov Moscow State University, Russian Federation (e-mail: echerepetskaya@mail.ru).

N. B. Podymova is with the Department of Physics of M. V. Lomonosov Moscow State University.

V.A. Makarov is with International Laser Center of M. V. Lomonosov Moscow State University.

Traditionally, the elastic moduli of CMs are measured using mechanical static methods (tension or compression) and dynamic methods based on measuring the resonant frequencies of natural oscillations of samples. However, these methods can be used to study samples of definite geometry, such as rectangular rod samples with a fixed ratio of the longitudinal and transverse sizes. In addition, mechanical methods cannot be used to measure local elastic moduli of CMs having substantially nonuniform structure. It is known that the elastic properties of materials affect the phase velocities of acoustic waves propagating therein. Therefore, at present, wide use is made of ultrasonic methods for determining the elastic moduli of structural materials from measured phase velocities of longitudinal and transverse (shear) acoustic waves [3]–[5].

The aim of this work was to develop and implement a pulse acoustic method of quantifying the influence of porosity on the local Young's modulus of isotropic composite materials using a laser source of ultrasound. The method is based on a laser thermo-optical method of ultrasound generation [6],[7] combined with measurement of the phase velocity of longitudinal and shear acoustic waves in samples. The main advantage of this method compared with traditional ultrasonic research methods is the efficient generation of short and powerful probing acoustic pulses required for reliable testing of ultrasound absorbing and scattering heterogeneous CMs.

II. METHOD OF CALCULATING OF ELASTIC MODULUS OF ISOTROPIC COMPOSITE MATERIALS

The porosity P (the volume concentration of gas pores averaged over its volume) of a CM sample is defined by [8]

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

where ρ is the density of the sample calculated from the known volume of the sample and the results of its hydrostatic weighing or weighing in air, ρ_0 is the calculated density of the solid phase of the sample determined from the known densities of the matrix ρ_m and the filler ρ_f and their volume concentrations n_m and n_f in this sample

$$\rho_0 = n_m \rho_m + n_f \rho_f \quad (2)$$

According to the theory of elasticity, the Young's modulus E_0 of an isotropic composite without pores (P is zero) is calculated by

$$E_0 = \rho_0 C_S^2 \frac{3C_{L0}^2 - 4C_S^2}{C_{L0}^2 - C_S^2} \quad (3)$$

where ρ_0 is calculated by (2); C_{L0} is the theoretically calculated phase velocity of longitudinal acoustic waves in the CM without pores, and C_S is the measured phase velocity of shear acoustic waves in the sample. The possibility of using the experimentally obtained value of C_S to calculate Young's modulus is due to the fact that porosity does not affect the shear stiffness of the sample, and a reduction of C_S due to the scattering of shear waves by pores for sufficiently small porosity (less than 5%) can be neglected. To calculate C_{L0} , it is proposed to use the following model of propagation of longitudinal acoustic waves in a two-phase (two-component) medium

$$C_{L0}^2 = \frac{1}{\rho_0} \left(\frac{n_m}{\rho_m C_{Lm}^2} + \frac{n_f}{\rho_f C_{Lf}^2} \right)^{-1} \quad (4)$$

Here the phase velocities of longitudinal acoustic waves in the matrix C_{Lm} and filler C_{Lf} of the composite are assumed to be known. Young's modulus of the samples are calculated by (3) in which ρ_0 and C_{L0} are replaced by the measured densities ρ and the phase velocity of longitudinal acoustic waves C_L for each CM sample.

The Poisson's ratio ν_0 is defined by

$$\nu_0 = \frac{C_{L0}^2 - 2C_S^2}{2(C_{L0}^2 - C_S^2)} \quad (5)$$

III. METHOD FOR MEASURING PHASE VELOCITIES OF LONGITUDINAL AND SHEAR ULTRASONIC WAVES IN AN ISOTROPIC COMPOSITE

Thermo-optical sources of ultrasound produce acoustic signals of short duration about 100 ns, which provides high accuracy in measuring the travel time of an acoustic wave even in samples of small thickness of 100 μm and more [9]. An ultrasonic beam with a typical diameter of a few millimeters can be used to produce and measure the phase velocity of transverse (shear) acoustic waves. The ultrasonic pulse generated in an OA source (the reference signal) propagates in the sample and is detected using a broadband piezoelectric transducer having acoustic contact with the sample. To ensure this contact, the OA source, sample, and piezotransducer are separated by layers of an immersion fluid (in this case, distilled water) and are mounted into a cell,

which is an optoacoustic measuring cell. The transverse size of the test region is determined by the transverse size of the generated ultrasonic beam equal to the characteristic diameter of the laser beam. Electrical signals from the piezotransducer are sent to a Tektronix type dual-channel digital storage oscilloscope; the time of start of the oscilloscope is synchronized with the moment of laser pulse generation. The signal-to-noise ratio of the recorded electrical signals is 50-60 dB. The laser OA system has the following characteristics: operating frequency range 0.2-50.0 MHz, pressure amplitude of ultrasonic pulses 0.01-10.00 MPa, sample thickness 0.1-70.0 mm, and transverse size of the testing region 1-2 mm.

The samples of composites based on a matrix of AK12M2MgN alloy (silumin) reinforced with particles of silicon carbide, SiC, with an average diameter of 14 μm and various volume concentrations were studied. The density of the solid phase of each sample ρ_0 was calculated by (2) using the known density of the filler $\rho_{SiC} = 3200 \text{ kg/m}^3$ [10], the density of the silumin matrix $\rho_A = 2740 \text{ kg/m}^3$, and the specified volume concentrations of the matrix n_A and the filler n_{SiC} . The density of silumin was calculated from the known densities and mass concentrations of the alloy components. The density of each sample ρ was calculated from the mass measured by weighing in air and the calculated volume. Accordingly, the porosity of each sample P averaged over its volume was determined by (1). Parameters of the investigated samples of the CM are given Table I. From Table I, it follows that the averaged porosity of the sample increases with increasing concentration of the SiC filler.

TABLE I
PARAMETERS OF CM SAMPLES

| Sample number | H, mm | n | | $\rho_0 \text{ kg/m}^3$ | $\rho \text{ kg/m}^3$ | P, % |
|---------------|-------|-----------|------|-------------------------|-----------------------|------|
| | | AK12M2MgN | SiC | | | |
| 1 | 11,20 | 1,00 | 0 | 2,74 | 2,72 | 0,7 |
| 2 | 10,20 | 0,97 | 0,03 | 2,75 | 2,71 | 1,5 |
| 3 | 7,85 | 0,93 | 0,07 | 2,72 | 2,67 | 3,6 |
| 4 | 4,75 | 0,86 | 0,14 | 2,80 | 2,67 | 4,6 |

Presumably, this is due to features of the manufacturing technology of the material (nonuniform reinforcement). For the same reason, the local porosity in different parts of the same sample may be different, which affects the local Young modulus. Since the porosity of the samples does not exceed 5%, in calculating the Young's modulus of the composite without pores by (3), we can use the measured phase velocity of shear acoustic waves in the sample. All tested samples were plane-parallel disks with a diameter of 40 mm, whose surfaces were polished by a corundum abrasive powder having an average particle diameter of 20 μm . It should be noted that in samples with this ratio of the thickness and diameter, it is impossible to create strictly uniaxial tension required for measuring Young's modulus using the mechanical method of tension or compression. The mechanical and acoustic properties of the obtained CM samples allowed them to be considered isotropic since the shape of the filler particles is

quasi-spherical and the particles are almost uniformly distributed over the volume of each sample.

IV. RESULTS AND DISCUSSION

The absolute value of phase velocity of longitudinal acoustic waves is given by

$$C_L = 2H / \Delta T_L \quad (6)$$

where H is the thickness of the sample, ΔT_L is the difference in time of arrival at the piezotransducer between the ultrasonic pulse passed one times through the sample and the pulse passed thrice through the sample and reflected from the interfaces between the sample and the immersion fluid layers. This method of measuring the phase velocity is called the time-of-flight method. The typical temporal shape of the longitudinal acoustic wave pulse passed through samples (Table I) and the pulse passed three times through the sample are recorded. The recorded shape of the wave S is determined by the acoustic field of the shear wave transformed into a longitudinal wave in passing from the sample to the immersion fluid. In this case, the shape of the wave S is influenced by the direction of the shear wave, determined by the transverse size of the longitudinal wave beam L , the dependence of the transformation coefficient on the angle of incidence of the shear wave on the interface between the sample and the immersion fluid, and the finiteness of the piezotransducer aperture. Therefore, the recorded pulse S , determined by the shear wave, is highly extended compared with the longitudinal wave pulse L , and the arrival time of the negative peak of the signal S is determined by the time of shear wave propagation in the sample. The pulse following this signal is the pulse of longitudinal waves L reflected in the immersion liquid layer between the sample and the piezotransducer. The measured difference ΔT_{LS} in time of arrival at the piezotransducer between the maximum of the pulse L and the minimum of the pulse S and the measured phase velocity of longitudinal acoustic waves C_L are used to determine the phase velocity of shear waves C_S in the sample

$$C_S = H(\Delta T_{LS} + H / C_L)^{-1} \quad (7)$$

The error of time-of-flight measurements of phase velocities is fairly small: about 0.5% for longitudinal waves and about 2% for shear waves. For longitudinal ultrasonic waves, the measurement accuracy is determined primarily by the maximum relative error of measurement of the sample thickness. Table II shows the results of optoacoustic measurements of the local Young's modulus and Poisson's ratio at the center (E_1, ν_1) and on the periphery (E_2, ν_2) of each CM sample. Fig. 1 shows the results of calculations of the average values of Young's modulus E for different volume concentrations of SiC particles n_{SiC} based on (3) and (4).

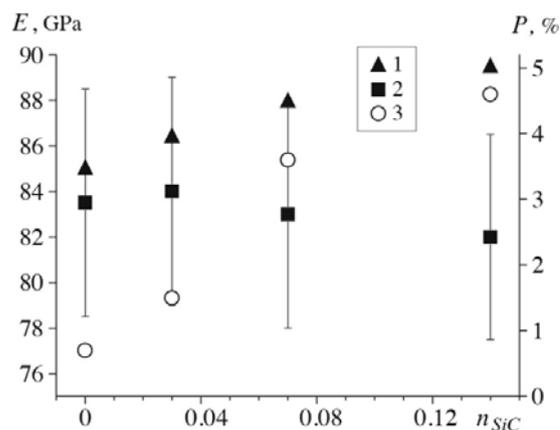


Fig. 1 Young's modulus (1 and 2) and porosity (3) of CM samples based on silumin versus volume concentration of the filler SiC: (1) results of calculations by formula (1.3); (2) results of measurements

TABLE II
RESULTS OF OPTOACOUSTIC MEASUREMENTS OF YOUNG'S MODULUS AND POISSON'S RATIO OF CM SAMPLES

| Sample | E_1 , GPa | E_2 , GPa | ν_1 | ν_2 |
|--------|-------------|-------------|-------------|-------------|
| 1 | 83±5 | 84±5 | 0,326±0,016 | 0,328±0,016 |
| 2 | 84±5 | 84±5 | 0,330±0,016 | 0,320±0,016 |
| 3 | 83±5 | 83±5 | 0,319±0,016 | 0,318±0,016 |
| 4 | 80±5 | 80±5 | 0,323±0,016 | 0,321±0,016 |

The dependence of the porosity P of the sample on the value of n_{SiC} is also shown in Fig. 1. Calculation results show that the addition of silicon carbide particles with a volume concentration of 14% should increase the Young's modulus by almost 6% compared to the initial Young's modulus of the matrix, but this does not occur due to an increase in the porosity of the material. It can be seen that for a porosity of the sample $P \approx 5\%$, the decrease in the Young's modulus relative to the calculated value is 8%. Thus, to provide an increase in the Young's modulus of the CM based on AK12M2MgN silumin with increasing volume concentration of the filler SiC during its mechanical mixing into the matrix melt, the porosity the final sample should not exceed 2%.

V. CONCLUSIONS

It was proposed and experimentally implemented an acoustic pulsed method using a laser source of ultrasound for quantitative evaluation of the combined effect of porosity and concentration of dispersed filler on the local Young's modulus of isotropic metal matrix CMs. This method is suitable for nondestructive measurements of Young's modulus in samples 0.1–70.0 mm thick with a transverse size of the test area 1–2 mm. For the investigated CMs based on silumin with the addition of reinforcing microparticles of silicon carbide, SiC, we determined the maximum porosity of the prepared sample above which the effect of porosity on the reduction in Young's modulus is more significant than the effect of the filler on the increase of this modulus. The developed laser ultrasonic method can be used for nondestructive testing of the local acoustic and mechanical properties of CM.

This method can be used in experimental studies of the effect of the composition, size, and concentration of reinforcing fillers on the elastic properties of composite materials.

ACKNOWLEDGMENT

The work was carried out with financial support from the Ministry of Education and Science of the Russian Federation *in the framework of Increase Competitiveness Program of NUST «MISiS»* (№ K1-2015-025)

REFERENCES

- [1] Springer handbook of nanotechnology / Ed. B. Bhushan. Springer-Verlag, Berlin (2007), 1916 p.
- [2] A.D. Romig, jr, M.T. Dugger, P.J. McWhorter. *Acta Mater.* 51, 5837 (2003).
- [3] Yu Golovin. *Introduction to nanotech. Machinery, M.* (2007). 496 pp.
- [4] P.D. Tall, S. Ndiaye, A.C. Beye, Z. Zong, W.O. Soboyejo, H.-J. Lee, A.G. Ramirez, K. Rajan, *Mater. Manuf. Processes* 22 (2007) 175-179.
- [5] E. Wibowo, C.Y. Kwok, *J. Micromech. Microeng.* 16 (2006) 101-108.
- [6] A.K. Nanda Kumar, C.K. Sasidharan Nair, M.D. Kannan, S. Jayakumar, *Mater. Chem. Phys.* 97 (2006), 308-314.
- [7] M. Callisti, F.D. Tichelaar, B.G. Mellor, T. Polcar, *Surf. Coat. Technol.* 237 (2013) 261-268.
- [8] V. V. Polyakov and A. V. Golovin, "Elastic Characteristics of Porous Materials," *Prikl. Mekh. Tekh. Fiz.* 34 (5), 32-35 (1993) (*J. Appl. Mech. Tech. Phys.* 34 (5), 625-628 (1993)).
- [9] V. E. Gusev and A. A. Karabutov, *Laser Optoacoustics* (Nauka, Moscow, 1991) (in Russian)
- [10] S. Yu. Davydov, "Effect of Pressure on the Elastic Properties of Silicon Carbide," *Fiz. Tverd. Tela* 46 (7), 1169-1173 (2004).