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Laser Diffraction on Particles of a Damaged Surface Layer of Piezoceramics

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The phenomenon of diffraction of laser radiation by particles of a damaged surface layer of a piezoceramic deposited on a plane-parallel glass plate was studied. A diffraction pattern is clearly observed in reflected and transmitted light, which is an alternation of light and dark concentric rings. These rings correspond to the conditions of diffraction maxima and minima. A bright spot is observed in the center of the diffraction pattern. The dependence of the radius of diffraction rings on the thickness of the glass plate was established. The diffraction angles at a given value of the order of maximum for different thicknesses of the glass plate were the same. The particles of the damaged surface layer of the piezoceramic, which scatter light, have different characteristic sizes. The study of particles using optical microscopy showed that their sizes are in the range of ~ (1 ÷ 3) μm. The shape of the grains is predominantly close to spherical. The Fraunhofer approximation was used to analyze the diffraction pattern. The distribution of the light intensity of the diffraction pattern depending on the diffraction angle has been analyzed. The simulation of the total scattered intensity as a function of the angle for particles with a diameter of 1.7 μm and 3.0 μm has been performed. The diffraction angles become smaller as the particle size increases. The distribution of the light flux in the diffraction pattern is constructed. This graph shows that most of the scattering intensity is concentrated near the center of the diffraction pattern, where the vast majority of the laser radiation falls. As the diffraction angle increases, the relative intensity of the maxima obeys the regularity 1 : 0.71 : 0.25 : 0.13. The size of the particles of the damaged surface layer of the piezoceramic was estimated using the laser diffraction method. The average particle diameter of the surface damaged layer of the piezoceramic is 1.7 μm. It can be argued that particles of such sizes make the main contribution to the creation of the diffraction pattern obtained by us under these experimental conditions.

Keywords: Laser diffraction, Particles, Damaged surface layer, Piezoceramics.

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1. INTRODUCTION

Piezoceramic materials based on solid solutions of lead zirconate-titanate PZT are currently the most promising and widely used in electronic engineering for the creation of piezoelectric transducers [1, 2]. In the process of technological operations for the manufacturing of piezoceramic materials, a surface damaged layer is formed on their surface. Its formation is due to the operations of mechanical processing of piezoceramic samples, deviations from the manufacturing technology and changes in the chemical composition of the surface upon contact with various cleaning solutions. The presence of such a layer leads to a deterioration in the quality of piezoceramic materials, worsens the adhesion of metal electrodes, and therefore requires its study and consideration in order to minimize its effect on the electrical characteristics of piezoelectric elements manufactured on their basis.

The study of the properties of the surface layer of the PZT piezoceramics depending on the surface treatment technology was studied in a number of works. A technique for controlling the integrity of the surface during ultra-precise grinding of polycrystalline ceramics PZT has been developed. Defects including porosity and fractural damage induced in the subsurface area were investigated. The proposed technique provides for optimal surface treatment to obtain excellent surface roughness and high surface flatness with minimal texture damage [3].

The surface microstructure of PZTNB-1 piezoceramics has been studied in comparison with the tetragonal PZT-19. The size distribution of grains and porous, their shape peculiarities were revealed with using computer treatment of visualized structural elements [4].

The effect of abrasive polishing on the surface topography and domain structure of PZT ferroelectric ceramics, using atomic force microscopy was studied. It has been established that polishing treatment removes non-ferroelectric passive layers formed during material preparation, and also leads to the formation of a new surface layer with properties different from those in the bulk [5].

The features of the surface layer formed during mechanical processing of the PZT ceramic surface were studied. A correlation has been established between microhardness and domain sizes. It is shown that the properties of the surface layer depend on the particle size of the diamond paste used for polishing [6].

Determining the size of particles with a diameter of several microns is a rather difficult task due to the high measurement error using various instruments. This problem can be more successfully solved using the diffraction method, which allows obtaining results with higher accuracy.

One of the modern methods for studying small particles is laser diffraction [7]. The phenomenon of laser diffraction has practical applications for determining the size of small

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particles and their statistical size distribution.

Laser diffraction method is based on the inversely proportional dependence of the light diffraction angle on the particle size. This method uses a laser as a light source. The particle size can vary over a wide range. In practice, laser diffraction is usually applied over a size range of about 30 nm – 1,000 μm [7].

Laser diffraction operates on the principle of Fraunhofer diffraction. This is true only if the particles are large compared to the wavelength of light, or if the ratio of the refractive indices of the dispersed and continuous phases is clearly different from unity [8].

Laser diffraction method is typically used in line-of-sight mode, which collects and analyzes near scattering from an ensemble of particles larger than the wavelength. Based on the theory of Fraunhofer diffraction, the problem of particle size distribution is solved [9].

The fundamentals of the theory of absorption and scattering of light by small particles were considered by C.F. Bohren, D.R. Huffman [10].

Laser diffraction method based on the Fraunhofer diffraction is the most widely used for particle size analysis due to ease of use, speed, and reproducibility. The pattern of a diffraction image depends on the shape of the particle, and the size of the pattern depends on the size of the particle. The directions of research on determining the shape of non-spherical particles are considered [11].

The theory of laser diffraction is a special branch of the theory of electromagnetic scattering. It is based on Maxwell's equations and their solutions. Depending on the size of the particles in relation to the wavelength of the laser radiation, different theories can be used. The Fraunhofer approximation, which is closer to geometrical optics than other approximations, is commonly used in laser diffraction techniques to determine particle sizes [12].

It is proposed to use the laser diffraction method not only to determine the particle size distribution of mixtures, but also to determine the number of particles [13].

The modern laser diffraction method is used to measure the grain size of powder in building materials and the particle size distribution in relation to their diameter [14].

To measure particle sizes, a method is proposed for analyzing the pattern of optical radiation scattering by microparticles obtained using a digital camera [15].

The laser diffraction analysis method is based on measuring the scattering angle and intensity of light after it passes through the particles. It is an instant, convenient and non-destructive method for evaluating inorganic powders [16].

Smart laser diffraction analysis provides information on the size, shape and concentration ratio of two-component heterogeneous model mixtures of particles with the accuracy better than 92 %. In contrast to commonly-used laser diffraction schemes, in which a large number of detectors are needed, the machine-learning-assisted protocol makes use of a single far-field diffraction pattern contained within a small angle (~ 0.26°) around the light propagation axis [17].

2. METHODOLOGY OF THE EXPERIMENT

PZT oxide piezoceramics based on lead zirconate-titanate $Pb(Zr,Ti)O_3$ solid solutions with various modifying additives was chosen as the object of research. The studies were carried out on piezoceramic blanks of industrial composition PZT-19. The removal of the surface damaged layer from the surface of piezoceramic blanks was carried out mechanically using a grinding belt. Particles of the damaged surface layer were deposited on plane-parallel glass plates of various thicknesses. An LGN-207A helium-neon laser with a radiation wavelength $\lambda = 0.6328$ μm was used as a coherent light source.

In our experiment, the thickness of the glass plates was 1.5 mm, 4 mm, and 5 mm. The distance from the laser radiation source to the glass plate was 0.15 m. The laser beam was directed onto a flat parallel glass plate with particles of the damaged surface layer of the PZT piezoceramic at a right angle. The experiment and calculations were carried out for reflected light. The diffraction pattern was clearly observed regardless of the viewing angle, both in reflected and transmitted light. The diffraction pattern was photographed with a Canon EOS 600D digital SLR camera. The radii of the diffraction rings were measured at the center of the light rings corresponding to the diffraction maxima.

The scheme of the experimental setup is shown in Fig. 1.

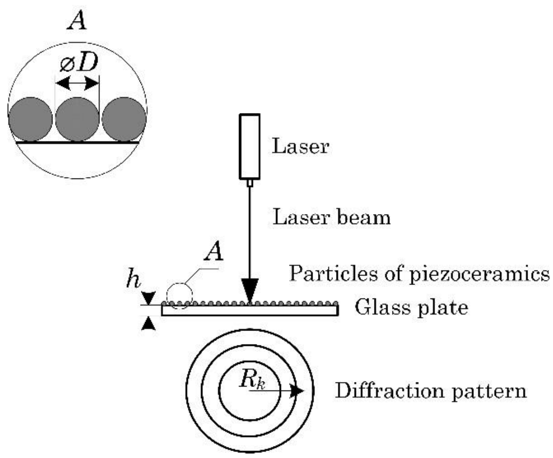

Fig. 1 – Scheme of the experimental setup

The microstructure of the surface damaged layer was also studied using optical microscopy on a MMU-5C metallographic microscope at a magnification from × 640 to × 1280.

3. EXPERIMENTAL RESULTS AND THEIR DISCUSSION

We have discovered the phenomenon of diffraction of light scattered by particles of a damaged surface layer of PZT piezoceramics deposited on a plane-parallel glass plate. A diffraction pattern is clearly observed in reflected and transmitted light, which is an alternation of light and dark concentric rings. These rings correspond to the conditions of diffraction maxima and minima (Fig. 1). In the center of the diffraction pattern, there is a bright spot – Airy disc.

The diffraction pattern arises as a result of diffraction scattering of light by particles of a damaged surface layer of the PZT piezoceramic. The occurrence of diffraction maxima and minima of the light intensity is due to the

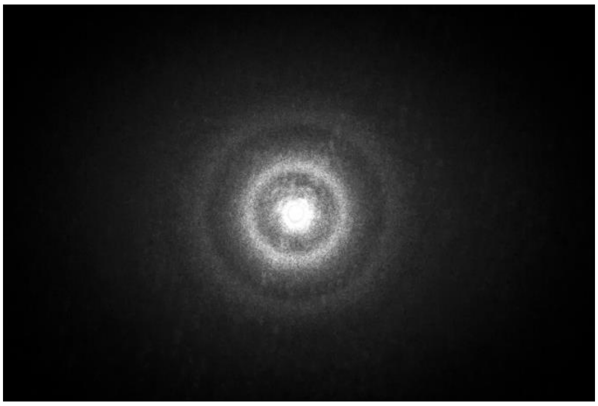

Fig. 2 – Photograph of the diffraction pattern of laser radiation on particles of a damaged surface layer of PZT-19 piezoceramic on a glass plate 1.5 mm thickness

scattering of light by particles and the interference between the rays, the scattered particles of the damaged layer and their reflection from the rear surface of the glass plate. The lengths of the optical paths of these rays do not differ significantly, as a result of which the necessary optical path difference arises, despite the fact that the thickness of the glass plate significantly exceeds the wavelength of light [18].

A coherent beam of light, diffracted by a single particle in different directions, expands as it passes through the glass. Since there are many particles in the plane of the cross section of the laser beam, and they are located randomly, then, due to the equal probability of all values of the phases of light waves diffracted in each direction, the intensities of light waves diffracted by different particles will add up.

The diffraction angle φ was calculated on the basis of experimental data on measuring the radius of diffraction rings and the thickness of the glass plate, based on the following condition:

$$\varphi_k = \arcsin\frac{R_k}{\sqrt{4h^2 + R_k^2}}, \tag{3.1}$$

where R_k are the radii of light diffraction rings; $k = 2, 4, 6$; h is the glass plate thickness.

The geometric substantiation of formula (3.1) is the construction shown in Fig. 3.

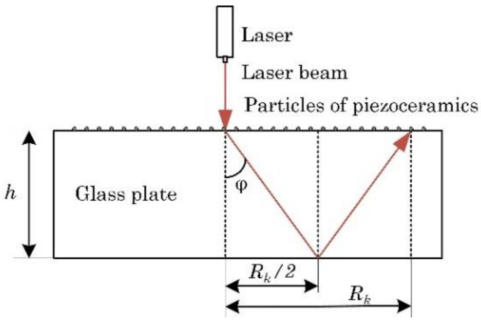

Fig. 3 – Scheme of the passage of an optical beam through a glass plate

Experimental data on the values of the radii of light diffraction rings for glass plates of different thicknesses are shown in Table 1.

Table 1 – The values of the radii of the diffraction rings for different thicknesses of the glass plate

Thickness of glass plate, mm	Radii of diffraction rings		
	R_2, mm	R_4, mm	R_6, mm
$h_1 = 1.5$	3	6	9
$h_2 = 4.0$	7	14	21
$h_3 = 5.0$	10	20	30

The numbering of rings in the formulas starts from the first dark ring from the center. The values of the diffraction angles corresponding to the light intensity maxima for different thicknesses of the glass plate are summarized in Table. 2. The diffraction angles at a given value of k for different thicknesses of the glass plate have similar values (can be considered the same within the measurement error).

Table 2 – Values of diffraction angles corresponding to maxima of light intensity

Thickness of glass plate, mm	φ_2, rad	φ_4, rad	φ_6, rad
$h_1 = 1.5$	0.45	0.79	0.98
$h_2 = 4.0$	0.41	0.72	0.92
$h_3 = 5.0$	0.46	0.79	0.98

We found that the following relation between the radius of the diffraction maximum and its order is satisfied for a given thickness of the glass plate:

$$\frac{R_k}{k} = const. \tag{3.2}$$

It should be noted, that the particles of the damaged surface layer of the PZT piezoceramic, which scatter light, have different characteristic sizes. The study of particles using optical microscopy made it possible to determine the size of the particles and the nature of their distribution on the surface of the glass plate. In Figure 4 shows a photograph of the particles of the damaged surface layer of the PZT-19 piezoceramic, obtained using an MMU-5C microscope at ×640 magnification. The photograph shows that the particles are randomly distributed over the surface of the glass plate. The distance between adjacent particles varies within ~ 1 – 10 μm. Particle sizing was performed using a photograph taken at ×1280 magnification. It is shown in Fig. 5. Analysis of this photograph showed, that the particle sizes are in the range of ~ (1 ÷ 3) μm. The shape of the grains is predominantly close to spherical. There are also conglomerates of particles ~ (5 ÷ 10) μm in size.

The results obtained are consistent with the data on the study of the PZT piezoceramic surface. According to these studies, the surface structure of the PZT-19 piezoceramic is a conglomerate of grains with pores. The average grain size is 4.6 μm and the average pore size is 10.2 μm. According to AFM microscopy data, the depth of the relief of the piezoceramic surface is 1.7 μm, the grain roughness is about 0.2 μm, and the depth of grain boundaries is up to 1 μm [4]. The presented data make it possible to assert that the particles under study correspond to the damaged surface layer.

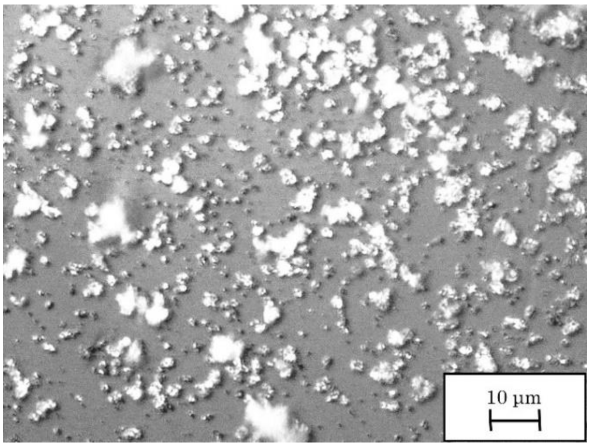

Fig. 4 – Photograph of the particles of the damaged surface layer of the PZT-19 piezoceramic at × 640 magnification

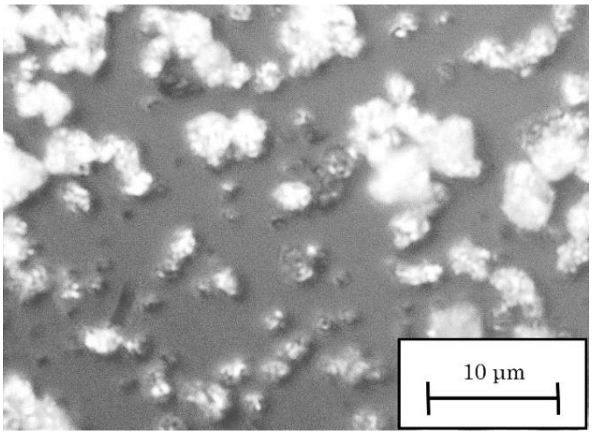

Fig. 5 – Photograph of the particles of the damaged surface layer of the PZT-19 piezoceramic at × 1280 magnification

The diffraction pattern shown in Fig. 2 is a superposition of interference patterns of diffraction scattering of many individual particles. In this case, the concentration of particles should be low enough to avoid particle overlap and multiple scattering. On the other hand, the concentration should be high enough to achieve an acceptable signal-to-noise ratio [12]. The standard method for evaluating laser diffraction data is based on the Fraunhofer approximation. Fraunhofer diffraction is also known as direct scattering or low-angle laser light scattering [12]. The Fraunhofer diffraction pattern is the Fourier transform of the particle projection. Fraunhofer's theory assumes that the investigated particles have a spherical shape.

In practice, the Fraunhofer approximation is applied to particles larger than a few microns, or to highly absorbing particles (with an absorption coefficient greater than 0.5), or to particles with a significantly different contrast of refractive index relative to the medium ($n > 1.2$). Since the PZT solid solution is isotropic crystals with a refractive index $n = 2.572 \pm 0.025$ [19], we use the Fraunhofer approximation in further calculations. The use of the Fraunhofer approximation also corresponds to the fulfillment of the criterion $\alpha >> 1$ [14]:

$$\alpha = \frac{\pi D}{\lambda}, \tag{3.3}$$

where α is a dimensionless parameter, D is the particle size (its diameter), λ is the wavelength of the light emitted by the laser.

Scattering of unpolarized light with intensity I_0 incident on one spherical particle is described by the relation:

$$I(\theta) = \frac{I_0}{2k^2a^2}\left[\left(S_1[\theta]\right)^2 + \left(S_2[\theta]\right)^2\right], \tag{3.4}$$

where $I(\theta)$ is the total scattered intensity as a function of the angle θ, θ is the scattering angles used for mathematical modeling, I_0 is the intensity of the incident light (modulus of the Poynting vector), $k = 2\pi/\lambda$ (wavenumber), λ is the wavelength of the light emitted by the laser that it is in the air), a is the distance from the light-scattering particle to the detector, and are the dimensionless complex functions describing the change in the amplitude in the perpendicular and parallel polarized light components, respectively, depending on the angle θ with respect to the forward direction [20].

In the Fraunhofer approximation, which describes the diffraction of light on the contour of a particle, scattering is considered in the near forward direction (i.e., the angle θ is small). In this case:

$$\left(S_1\right)^2 = \left(S_2\right)^2 = \alpha^4\left[\frac{j_1(\alpha\sin\theta)}{\alpha\sin\theta}\right]^2. \tag{3.5}$$

Then, equation (3.4) is simplified to (3.6) and represents an analytical solution for spherical particles as if they were opaque disks [21]:

$$I(\theta) = \frac{I_0}{k^2a^2}\alpha^4\left[\frac{j_1(\alpha\sin\theta)}{\alpha\sin\theta}\right]^2, \tag{3.6}$$

where $j_1(\alpha\sin\theta)$ is the first-order spherical Bessel function of the first kind.

The spherical Bessel functions can be calculated using the Rayleigh formula (3.7):

$$j_n(x) = (-x)^n\left(\frac{1}{x}\frac{d}{dx}\right)^n\frac{\sin(x)}{x}. \tag{3.7}$$

The angular dimensions of the diffraction rings (the positions of the minima and maxima of the intensity) are determined by the zeros of the Bessel function. We used the built-in function in MathCad $js(n, x)$, where n is the order (integer), x is a real dimensionless scalar, $x = \alpha\sin\theta$.

The result of modeling the total scattered intensity as a function of the angle in the MathCad system for particle sizes D = 1.7 μm and D = 3.0 μm is shown in Fig. 6. We see that the diffraction angles become smaller with increasing particle size. The larger the particle, the more light it scatters and the more it scatters in the forward direction. For very small particles, scattered light is weaker and almost isotropic.

An analysis of the results of optical microscopy shows that the investigated particles of the surface damaged layer of the PZT piezoceramic have different sizes and shapes. In laser diffraction, each particle of many particles of different sizes creates a certain diffraction pattern; they all overlap each other, which lead to an increase in the width of individual diffraction rings.

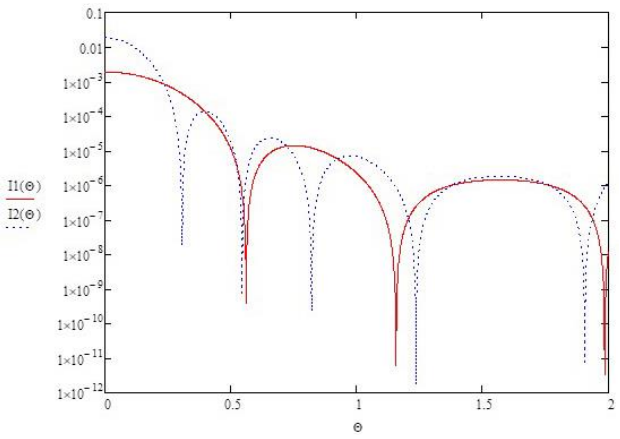

Fig. 6 – The result of modeling the total scattered intensity as a function of the angle in the MathCad system from 0 to 2 rad for particles with sizes of 1.7 μm (——) and 3.0 μm (• • •)

The “line profile” tool in the Tracker software tool allowed us to build on the basis of the photograph shown in Fig. 2, the distribution of the light flux in the diffraction pattern (Fig. 7).

Fig. 7 – The graph of the distribution of the light flux in the diametrical section of the diffraction pattern, obtained on the basis of the analysis of the photograph shown in Fig. 2 in Tracker. Along the *0Y* axis – the luminous flux in Lm, along the *0X* axis – the distance in cm

This graph is similar to the radial light intensity distribution graph. It shows that most of the scattering intensity is concentrated near the center of the diffraction pattern, where the overwhelming majority of the laser radiation falls. The relative luminous flux in the center equal to 1. With an increase in the diffraction angle, the relative intensity of the second, third, and fourth maxima obeys the pattern 1 : 0.71 : 0.25 : 0.13 (see Table 3). With this approach, we did not take into account the amplitude characteristic of the camera, which shows how the intensity of the incident light is related to the brightness of the image.

It is known [22], that in Fraunhofer diffraction on a single spherical particle, the relative intensity at the maxima is described by the relation $I_{max}/I_0 : I_{2max}/I_0 : I_{4max}/I_0 : I_{6max}/I_0 = 1 : 0.0175 : 0.0042 : 0.0016$. With an increase in the number of particles, we obtain several diffraction patterns that overlap each other; along with this we obtain an increase in the contrast of the diffraction pattern.

Table 3 – The value of the relative luminous flux in the diffraction maxima

k	Ring of Intensity	Relative luminous flux, Φ_{max}/Φ_0
0	The central maximum	1.00
2	The second maximum	0.71
4	The third maximum	0.25
6	The fourth maximum	0.13

It is known [12, 22], that during Fraunhofer diffraction on a spherical particle, the minima and maxima are related to the size of the particles by the ratios given in the Table 4.

Table 4 – Relation of diffraction minima and maxima to particle size and wavelength

k	Ring of Intensity	φ_k
0	The central maximum	0
1	The first minimum	$\arcsin(1.22\lambda/D)$
2	The second maximum	$\arcsin(1.64\lambda/D)$
3	The second minimum	$\arcsin(2.23\lambda/D)$
4	The third maximum	$\arcsin(2.68\lambda/D)$
5	The third minimum	$\arcsin(3.24\lambda/D)$
6	The fourth maximum	$\arcsin(3.70\lambda/D)$
7	The fourth minimum	$\arcsin(4.24\lambda/D)$

Let us determine the particle size of the damaged surface layer of the PZT piezoceramic based on the condition of the maximum diffraction for light rings:

$$D_2 = 1.64\frac{\lambda}{\sin\varphi_2}; D_4 = 2.68\frac{\lambda}{\sin\varphi_4}; D_6 = 3.70\frac{\lambda}{\sin\varphi_6}. \quad (3.8)$$

The calculation results are given in the Table.5.

Table 5 – Particle size of the damaged surface layer of PZT piezoceramics

Particle diameter	Thickness of glass plate		
	h_1 = 1.5 mm	h_2 = 4.0 mm	h_3 = 5.0 mm
D_2, μm	2.3	2.6	2.3
D_4, μm	1.5	1.6	1.5
D_6, μm	1.2	1.3	1.2
D_{cp}, μm	1.7	1.8	1.7

An analysis of the results of calculating the particle size showed that their diameter is in the range of (1.2-2.6) μm. The average particle diameter of the surface damaged layer of the PZT piezoceramic is D_{av} = 1.7 μm. The relative measurement error, based on formula (3.8), taking into account (3.1), was determined as follows:

$$E = \frac{\Delta D}{D} = \sqrt{\left(\frac{\Delta\lambda}{\lambda}\right)^2 + \left(\frac{\Delta h}{h}\right)^2 + \left(\frac{\Delta R_k}{R_k}\right)^2}.$$

The values of the first two components of the error are negligible; they are equal to 0.007 % and 0.05 %, respectively. The main contribution comes from the error in measuring the radius of diffraction rings, which amounted to 10 %. The value of the relative error at a confidence level of 0.9 was $E \approx 10$ %. Consequently, the average particle diameter is D_{av} = (1.70 ± 0.17) μm. It can be argued that particles of such sizes make the main contribution to the creation of the diffraction pattern obtained by us under these experimental conditions.

4. CONCLUSIONS

1. It has been established that on the particles of the damaged surface layer of the PZT-19 piezoceramic deposited on a glass plane-parallel plate, a diffraction pattern in the form of concentric light and dark rings is clearly observed in the reflected and transmitted laser radiation.

2. The dependence of the radius of diffraction rings on the thickness of the glass plate is obtained.

3. It was found that the diffraction angles at a given value of the maximum order do not depend on the thickness of the glass plate.

4. The particles of the damaged surface layer of the piezoceramic, which scatter light, have different characteristic sizes. The study of particles using optical microscopy showed that their sizes are in the range of ~ (1 ÷ 3) μm. the particles are randomly distributed over the surface of the glass plate. The distance between adjacent particles varies within ~ 1 – 10 μm. The shape of the grains is predominantly close to spherical. There are also conglomerates of particles ~ (5 ÷ 10) μm in size.

5. Based on the Fraunhofer approximation, an analysis was made of the distribution of the light intensity of the diffraction pattern depending on the diffraction angle. The simulation of the total scattered intensity as a function of the angle in the MathCad system was performed for particles of different diameters D = 1.7 μm and D = 3 μm. The diffraction angles become smaller as the particle size increases.

6. Using the “line profile” tool in the Tracker software tool, a graph of the distribution of the light flux in the diffraction pattern was obtained. It shows that most of the scattering intensity is concentrated near the center of the diffraction pattern, where the overwhelming majority of the laser radiation falls. As the diffraction angle increases, the relative intensity of the maxima obeys the pattern 1 : 0.71 : 0.25 : 0.13.

7. The size of the particles of the damaged surface layer of the piezoceramic was estimated using the laser diffraction method. The particle diameter is within the range (1.2-2.6) μm. The average particle diameter of the surface damaged layer of the PZT piezoceramic is D_{av} = 1.7 μm. It can be argued that particles of such sizes make the main contribution to the creation of the diffraction pattern obtained by us under these experimental conditions.

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Лазерна дифракція на частках порушеного поверхневого шару п'єзокераміки

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Досліджено явище дифракції лазерного випромінювання на частках порушеного поверхневого шару п'єзокераміки, що нанесені на плоско паралельну скляну пластину. У прямому і відбитому світлі чітко спостерігається дифракційна картина, що представляє собою чергування світлих і темних концентричних кілець, що відповідає умовам максимумів і мінімумів дифракції. У центрі дифракційної картини є яскрава пляма. Було встановлено залежність радіусу дифракційних кілець від товщини скляної пластини. Кути дифракції при заданому значенні порядку максимуму для скляних пластини різної товщини були однаковими. Частки порушеного поверхневого шару п'єзокераміки, що розсіюють світло, мають різні характерні розміри. Дослідження часток за допомогою оптичної мікроскопії показало, що їх розміри знаходяться в межах ~ (1 ÷ 3) мкм. Форма зерен переважно близька до сферичної. Для аналізу дифракційної картини застосовувалося наближення Фраунгофера. Проведено аналіз розподілу інтенсивності світла дифракційної картини залежно від кута дифракції. Змодельована повна розсіяна інтенсивність як функція кута для часток діаметром 1.7 мкм та 3.0 мкм. Кути дифракції стають меншими зі збільшенням розміру часток. Побудовано розподіл світлового потоку у дифракційній картині. Цей розподіл показує, що більшість інтенсивності розсіювання зосереджена поблизу центру дифракційної картини, куди потрапляє переважна частина лазерного випромінювання. Зі збільшенням кута дифракції відносна інтенсивність максимумів описується закономірністю 1 : 0.71 : 0.25 : 0.13. Здійснено оцінку розмірів часток порушеного поверхневого шару п'єзокераміки за допомогою методу лазерної дифракції. Середній діаметр часток поверхневого порушеного шару п'єзокераміки становить величину 1.7 мкм. Можна стверджувати, що частки таких розмірів дають основний внесок у створення дифракційної картини, отриманої нами за даних експериментальних умов.

Ключові слова: Лазерна дифракція, Частки, Порушений поверхневий шар, П'єзокераміка.