

CONFIGURAÇÃO TRIDIMENSIONAL DE MATERIAIS COMPÓSITOS COMO FORMA DE ALCANÇAR ALTAS PROPRIEDADES MECÂNICAS**THREE-DIMENSIONAL SETUP OF COMPOSITE MATERIALS AS A WAY TO ACHIEVE HIGH MECHANICAL PROPERTIES****ТРЁХМЕРНАЯ ОРГАНИЗАЦИЯ КОМПОЗИТНЫХ МАТЕРИАЛОВ КАК СПОСОБ ДОСТИЖЕНИЯ ВЫСОКИХ МЕХАНИЧЕСКИХ ПОКАЗАТЕЛЕЙ**AMANKULOV, Yerdos^{1*}; PETUNINA, Irina²; LOBANOVA, Nadezhda³¹ Al-Farabi Kazakh National University, Department of Theoretical and Nuclear Physics. Kazakhstan² Federal State Budgetary Educational Institution of Higher Education "Kuban State Agrarian University named after I.T. Trubilin", Department of Higher Mathematics. Russian Federation³ MIREA - Russian Technological University, Department of Chemistry and Technology of High-Molecular Compounds named after Medvedev S. S. Russian Federation

* Corresponding author
e-mail: ayerdos@rambler.ru

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RESUMO

As resinas de polímero são lubrificantes secos populares e revestimentos protetores para produtos metálicos. No entanto, seu coeficiente de atrito é alto e isso leva a taxas de desgaste aumentadas e vida útil limitada. O reforço com cargas em nanoescala, devido à sua grande área superficial e sua distribuição uniforme dentro do volume da matriz polimérica, pode ajudar a reduzir o atrito e o desgaste. O objetivo deste trabalho foi estudar o efeito da organização tridimensional de nanopartículas de dióxido de silício com concentração de 1, 3 e 5% nas propriedades mecânicas de uma resina epóxi. A dispersão das nanopartículas foi realizada usando tecnologia de campo ultrassônico. O coeficiente de atrito foi medido usando um tribômetro de pêndulo. Resultados empíricos mostraram que a redução na concentração de nanopartículas de 5% para 1% resultou em uma redução de duas vezes nos valores do coeficiente de atrito. Além disso, verificou-se que o prolongamento do tempo de teste em experimentos com concentração de nanopartículas de 3 e 1% praticamente não afetou os valores do coeficiente de atrito. Este comportamento está associado à regulação da mobilidade da interface do polímero devido à baixa concentração de nanoinclusões e à organização tridimensional uniforme da matriz polimérica pelo campo ultrassônico, o que contribuiu para uma redistribuição uniforme das cargas aplicadas. A técnica desenvolvida pode ser aplicada a outros tipos de nanocompósitos poliméricos para estudar o efeito da organização tridimensional de cargas em nanoescala na resistência ao desgaste, resistência à flexão e resistência ao impacto.

Palavras-chave: *Nanopartículas de óxido de silício; configuração tridimensional; propriedades tribológicas, nanocompósitos.*

ABSTRACT

Polymer resins are popular dry lubricants and protective coatings for metal products. However, their friction coefficient is high, which leads to increased wear rates and limited service life. Reinforcement with nanoscale fillers, due to their large surface area and their uniform distribution within the polymer matrix volume, can help reduce friction and wear. This paper aimed to study the effect of three-dimensional organization of silicon dioxide nanoparticles with a concentration of 1, 3, and 5% on the mechanical properties of an epoxy resin. The dispersion of nanoparticles was carried out using ultrasonic field technology. The friction coefficient was measured using a pendulum tribometer. Empirical results have shown that reduction in the concentration of nanoparticles from 5% to 1% resulted in a two-fold decrease in the values of the friction coefficient. Also, it was found that prolongation of test time in experiments with a nanoparticle concentration of 3 and 1% had practically no effect on the friction coefficient values. This behavior is associated with the polymer interface mobility regulation due to the low concentration of nano inclusions and uniform three-dimensional organization in the polymer matrix by the ultrasonic field, which contributed to a uniform redistribution of applied loads. The developed technique can be

used to other types of polymer nanocomposites to study the effect of the three-dimensional organization of nanoscale fillers on wear resistance, bending strength, and impact resistance.

Keywords: *Silicon oxide nanoparticles; three-dimensional setup; tribological properties, nanocomposites.*

Аннотация

Полимерные смолы - популярные сухие смазочные материалы и защитные покрытия для металлических изделий. Однако их высокий коэффициент трения приводит к повышенному износу и ограниченному сроку службы. Упрочнение наноразмерными наполнителями из-за их большой площади поверхности и их равномерного распределения в объеме полимерной матрицы может помочь снизить трение и износ. Целью данной работы было изучение влияния трехмерной организации наночастиц диоксида кремния с концентрацией 1, 3 и 5% на механические свойства эпоксидной смолы. Диспергирование наночастиц осуществлялось с помощью ультразвуковой обработки. Коэффициент трения измеряли с помощью маятникового трибометра. Результаты эксперимента показали, что снижение концентрации наночастиц с 5% до 1% привело к двукратному снижению значений коэффициента трения. Кроме того, было обнаружено, что продление времени испытаний в экспериментах с концентрацией наночастиц 3 и 1% практически не повлияло на значения коэффициента трения. Такое поведение связано с регулированием подвижности границы раздела полимеров из-за низкой концентрации нановключений и однородной трехмерной организации в полимерной матрице ультразвуковым полем, что способствовало равномерному перераспределению приложенных нагрузок. Разработанная методика может быть использована для других типов полимерных нанокompозитов с целью изучения влияния трехмерной организации наноразмерных наполнителей на износостойкость, прочность на изгиб и ударопрочность.

Ключевые слова: *наночастицы оксида кремния; трехмерная организация; трибологические свойства, нанокompозит*

1. INTRODUCTION:

In widespread use of various mechanical systems such as parts of friction units (e.g., bearings, compressor cylinders) (Lampaert *et al.*, 2018; Xiang and Gao, 2017), metal forming and cutting tools (Flegler *et al.*, 2020; Idusuyi and Olayinka, 2019), transport mechanisms, and technological machines for various industries (Cho and Bhushan, 2016; Holmberg *et al.*, 2017), constant monitoring of friction and wear of structural parts is required. As the main binder component that enhances tribological properties, various liquid, dry lubricants are used (Mushtaq and Wani, 2017; Zhu *et al.*, 2019) or polymer coatings applied to parts.

Due to the limited use conditions for an extended period under challenging conditions, the various physical and chemical properties of different types of lubricants significantly reduce their effectiveness and durability (Busch *et al.*, 2016; Singh *et al.*, 2020). If due to restrictions on the use, liquid lubricants cannot be used, then polymer coatings are considered the best option for controlling friction and wear. However, polymeric materials must meet certain requirements for practical application in various mechanisms, such as high mechanical strength and durability, the required stiffness, the corresponding coefficient of thermal expansion (Zalaznik *et al.*, 2016). For example, for some

polymer composite coatings, low values of both the coefficient of friction (<0.2) and the wear rate ($<10^{-6}$ mm³/Nm) were achieved; additional lubrication was not required in the contact zone (Rodriguez *et al.*, 2016).

However, under conditions of high loads and mechanism speeds, polymer composites should have both high wear-resistant properties and self-lubrication characteristics. To solve the first condition, various types of polymer matrix fillers are usually introduced, which can vary in shape and size, such as particles and fibers (Gad *et al.*, 2017). As for the second condition, based on numerous previous studies, it was found that the peeling of the upper layers of the coatings followed by the transfer of thin lubricating films with wear residues significantly improved the tribological characteristics of polymer composites (Cui *et al.*, 2020; Pham *et al.*, 2019; Saravanan *et al.*, 2017; Zhang *et al.*, 2017).

Despite the significant advantages, polymer composites have several disadvantages, such as the loss of inherent mechanical properties when exposed to a liquid medium. For example, in contact with seawater, which includes a set of different salts, the surface layer enters into a chemical reaction with salt, which leads to a decrease in the efficiency and durability of the polymer coating.

Polymer nanocomposites are a class of

composite materials. The polymeric material matrix is reinforced with one or more types of nanoobjects smaller than 100 nm (Li *et al.*, 2016) to improve performance. The most common materials used as a matrix for polymer nanocomposites are various epoxies, nylon, polyepoxide, polyetherimide, and others. Unlike traditional polymer composites with a high filler density (about 60%) of different kinds of micrometer size particles, a lower density (less than 5%) of fillers is used in polymer nanocomposites. Recently, many new polymer nanocomposites have appeared, which have some unique properties and provide an opportunity for further implementation in the industry. For example, transparent conductive nanocomposites reinforced with nanotubes have been developed for use as electrodes of solar cells. Amorphous polymers with nanoparticles have been introduced into production due to the high scratch resistance of various transparent coatings in cell phones and CD technology (Wang and Alexandridis, 2016). In general, the use of nanoparticles to reinforce the matrix of traditional polymer composites increases mechanical, optical, or conductive properties.

The advantage of nanosized fillers is that, due to their size, they prevent cracks on the surface, which characterizes the increased ductility and strength of the polymer coating. As shown in the work of Roy *et al.* (2007), the introduction of nanoparticles into the polymer matrix contributes to an increase in the electric tensile strength and endurance. Also, it provides insignificant optical scattering on existing defects (Hanemann *et al.*, 2009). Because of the large surface area of the fillers, nanocomposites have a large volume of interphase boundaries, which differ significantly from the properties of a pure polymer. Depending on the volume of the interphase boundaries and its uniform distribution in the polymer matrix, the composite properties can differ significantly from each other. For example, to improve the tribological properties of composite coatings, it is necessary to observe the following rules, called the mixture rule. Namely, the strength modules of polymer nanocomposites should be directly proportional to the volume ratio of nanosized fillers when both phases are aligned to bear the same amount of load (Rivière *et al.*, 2016). Still, this optimal mechanical state is usually challenging to achieve due to aggregation and uneven distribution of the nanofiller. Surface optimization often helps to improve the dispersion of nano inclusions. Even with a concentration of up to 5%, it does not provide uniform load distribution, and friction coefficients remain high enough

(Domun *et al.*, 2015). The layer-by-layer application of polymer and nanofiller layers provides a structure with a more uniform dispersion of fillers (Li *et al.*, 2017). However, a layered structure containing a continuous interface between the phase components, due to the anisotropy in the layers, also limits the uniform distribution of the applied load, which leads to rapid wear of the coating.

The development of the design of continuous composite materials consisting of a network of physically separated two phases is a promising technique for overcoming the dispersion limitation of nanofillers in a matrix. The basis of this method is a phased preparation, consisting of the preparation of a porous matrix (template) and infiltration of the nanofiller into the pores. Three-dimensional matrix formation using PnP technology, which includes single-stage exposure through an appropriate phase mask, is the most effective method for constructing a matrix with a nanoscale step (less than 100 nm) (Park *et al.*, 2010). This method is effective, but very energy-consuming and expensive, and such low availability leads to a decrease in its application possibilities.

Among the most used reinforcing fillers, carbon fibers, short glass fibers, carbon nanotubes, or carbon nanofibers are used to improve the tribological properties of nanocomposite coatings (Yan *et al.*, 2013). However, various types of fillers have several disadvantages. For example, increasing the volume of carbon nanotubes to 1% usually greatly simplifies manufacturing a composite due to its porous structure. Still, often nanotubes scratch the surface, which damages the coating. The most effective way is a combination of reinforcing elements (carbon fibers with nanoparticles), which can significantly improve the tribological characteristics of polymers (Visco *et al.*, 2016). However, the selected objects are more difficult to distribute evenly due to their intricate design, unlike spherical nanoparticles. Besides, there is not enough research on the influence of nanoparticles' organization on improving the tribological properties of polymer composites.

The purpose of this research was to study the silicon dioxide nanoparticles' ultrasonic dispersion effect on the uniform distribution of the composite in the volume of the polymer matrix to improve tribological properties. Abrasion resistance tests under cyclic loading were carried out on three series of samples of polymer nanocomposites with different volume contents of nanoparticles.

2. MATERIALS AND METHODS:

$$\Delta p = R_a U \quad (2)$$

2.1. Preparation of materials and samples

Three series of samples were obtained using standard technology. Colloidal quartz nanoparticles (Aerosil R8200) with average particle sizes of 15 nm with 1, 3, and 5 %, respectively, were dispersed in epoxy resin (DER331, DOW). Samples of series 1 contained 1% of nanoparticles, series 2, and 3 had 3 and 5% of nanoparticles, respectively. A cycloaliphatic amine hardener was used (HY 2954; Huntsman). Volume fractions of fillers were calculated, taking into account their weight and density. Densities of pure epoxide and nanoparticles SiO₂ were 1.14 and 2.00 g/cm³, respectively.

2.2. Ultrasonic dispersion technique for three-dimensional structure of nanoparticles

The use of an ultrasonic field for dispersion in a liquid medium has many significant advantages compared to other methods, such as low energy consumption, high productivity, and the ability to obtain ultrafine particles with a repeatable result (Ma *et al.*, 2018).

The main characteristics describing the propagation of a sound field in a liquid medium are the pressure and sound speed in the medium. Propagating in a liquid, sound creates compression and rarefaction areas in which pressure changes occur for the average external statistical pressure (Herzfeld and Litovitz, 2013). Sound pressure is the main quantitative characteristic of sound and the main parameter of acoustic measurements.

The amount of energy of the wave motion, which is transferred in one second through the unit area, placed perpendicularly to the direction of wave propagation, is called the sound intensity and is expressed through the following formula:

$$I = \frac{1}{2} \frac{p_0^2}{R_a} \quad (1)$$

where, p_0 - sound pressure amplitude, R_a - acoustic impedance.

Acoustic impedance depends only on the properties of the medium, and sound pressure depends on the characteristics of the medium, such as the density and speed of sound propagation in the medium and on the amplitude and cyclic frequency of the ultrasonic wave.

The dependence of the acoustic velocity U in a liquid medium on sound pressure is expressed through the following formula:

In a liquid medium, the main mechanism of ultrasonic dispersion of powder materials is the occurrence of the cavitation effect (Gaynutdinova *et al.*, 2016), the essence of which is the formation of cavities or bubbles with a local decrease in pressure in the liquid to the pressure of saturated vapor. To stimulate cavitation in a liquid, it is necessary to stimulate a sound wave of a certain intensity, associated with other parameters from the formulas (1-2). Knowing one of them, one can find other characteristics when calculating an acoustic reactor to stimulate cavitation.

It is known that cavitation nuclei in a liquid can be randomly distributed. As a result of the oscillations, the bubbles reach a larger size, and the accumulations of bubbles under the action of the arising sound flows move at a sufficiently high speed in the volume of the liquid. The scattering and reflection of ultrasonic waves depending on changes in the time and space of the cavitation region lead to a significant averaging of the field, and the interference pattern is smoothed out, and the field acquires a fairly clear small-scale diffuse character. This diffuse nature, which is necessary for research, depends on the intensity of sound, which is governed by the clarity of the interference pattern. The basis of this technique includes the following - the application of an ultrasonic field leads to the acceleration of nanoparticles in volume of the polymer. The acceleration intensity can be adjusted to provide the most necessary conditions for a uniform distribution of particles by adjusting the sound intensity.

A series of samples was tested on an ultrasonic unit UP200St with a wide frequency range (up to 20 kHz). In a flat glass bath, two emitters were installed opposite each other to smooth the interference pattern. The intensity of the ultrasonic wave was adjusted by leveling the interference pattern on the surface of the liquid, an example of which is shown in Figure 1.

The dispersion of nanoparticles in the resin of each series of samples was carried out for 30 minutes. Then, the parts were coated with mixtures for tribological studies, and for curing were placed in an oven at 70 °C for 8 hours, then for 8 hours at 120 °C.

2.3. The methodology of tribological studies

Tribological tests were carried out on a pendulum tribometer with microprocessor processing of experimental data. In tests on a pendulum tribometer, the components of a friction

pair from a titanium sample with a surface hardness of 12.63 GPa were used, coated with previously obtained polymers of series 1, 2, and 3, respectively. The installation parameters were as follows: the length of the pendulum - 0.51 m, the weight of the load - 35.8 N, and the initial deflection angle - 20°.

The formula calculated the coefficient of friction:

$$f = \frac{\Delta A}{4(n-1)r} \quad (3)$$

where f – coefficient of friction; ΔA – decrease in the amplitude of the pendulum over the period (m); r – radius of the roller of the friction support unit (m); n – number of oscillation cycles.

3. RESULTS AND DISCUSSION:

The results of the experimental measurements of tribological properties are presented in Figure 2.

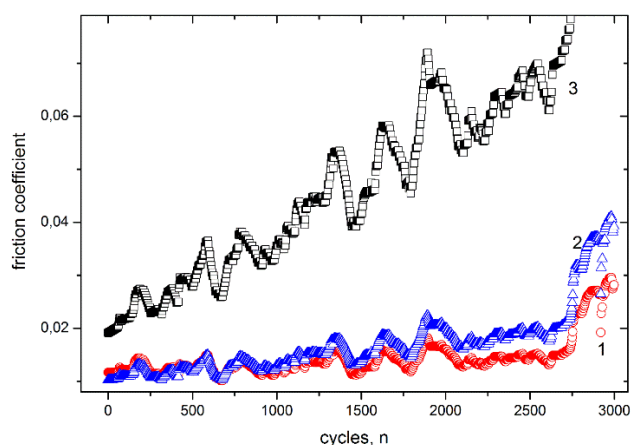


Figure 2. Time dependences of a friction coefficient f for a series of samples with different content of nanoparticles SiO_2 in the polymer matrix: 1 – series 1, 2 – series 2, 3 – series 3, respectively

As can be seen from Figure 2, a sample of series 3 with a maximum amount of introduced filler (in a volume of 5%) shows high values of the coefficient of friction f . Also, the sawtooth shape of the curve indicates the occurrence of defects in the test, such as cracks or tearing of the films. Large jumps in values and a sharp increase in f values over time indicate (1) a large volume fraction of nanofiller can adversely affect the structure and properties of the polymer coating or (2) there are agglomerates of nanoparticles, for breaking which ultrasonic frequency is not enough.

In recent works (Bittmann *et al.*, 2009; Kumar *et al.*, 2016; Ma *et al.*, 2018) similar results were obtained, according to which the optimal

dispersion process of nanoparticles in epoxy resin was achieved due to a systematic change in the parameters of the ultrasonic dispersion process. The effectiveness of applying the ultrasonic dispersion technique consists of using the maximum allowable amplitude of the ultrasonic device and the dispersion time in proportion to the volume of the material processed by ultrasound. However, some shortcomings of the chosen method were also found, such as the presence of residual agglomerates of nanoparticles. This explains the result obtained in sample 3.

However, samples with a smaller amount of reinforcing filler (series 1 and 2) demonstrate a significant improvement in the strength properties of nanocomposite coatings. As can be seen from Figure 2, the values of friction coefficient f for the samples of series 1 and 2 are almost two times less than for samples of series 3. Besides, there are smaller deviations in f values frequency and a slower growth rate over time (up to 2700 oscillation cycles of the pendulum). The results obtained in this work are in good agreement with the work of Zhang *et al.* (2011), where it has been shown that the increase in the volume fraction of the filler to 5 % leads to a decrease in tensile strength, and, consequently, the deterioration of tribological properties. In this work, the authors used a different approach to the uniform distribution of particles in the polymer matrix. Zare (2016) proposed a methodology for studying the effect of agglomeration of spherical nanoparticles in polymer nanocomposites. This methodology showed reliable results for determining the volume fraction of agglomerated nanoparticles in nanocomposite samples. According to the results of calculations by this technique for various samples, it was shown that the degree of agglomeration increased with an increase in the content of nanofiller and a decrease in particle size. In addition, it was found that agglomeration reduced the efficiency of nanoparticles in the polymer matrix, which led to a decrease in the mechanical properties of composite samples. The results of theoretical modeling are in excellent agreement with the experimental results obtained in this paper.

There were other studies of the effect of SiO_2 nanoparticles on the formation of a transfer film and the tribological properties of epoxy composites reinforced with different types of fillers (Zhang *et al.*, 2016). These studies showed that the presence of silicon dioxide nanoparticles at the interface gave rise to a transfer film with different structures and functional properties. The inclusion of different nanofillers of the same or different type

in the epoxide matrix improves the tribological properties. However, as shown above, it is not entirely effective. There was also a study of the dynamics of silicon dioxide nanoparticles at the interface between the polymer and the nanoparticle (Holt *et al.*, 2014) using various research methods.

The findings showed that the mobility of the interfacial polymer layer on the surface of nanoparticles with a lower concentration of nanoparticles is two orders lower than that of a bulk polymer. Analysis of small-angle X-ray scattering data showed that the interfacial layer has a thickness of 4–6 nm regardless of the concentration of nanoparticles. From these works, the authors conclude that due to the mobility of the interfacial polymer layer, depending on the degree of nanoparticle content, the necessary mechanical properties of the composite can be adjusted. Besides, uniform distribution can also contribute to a uniform redistribution of the applied loads throughout the polymer volume. Since the goal of this work has been to study the influence of the organization of nanoparticles in a polymer matrix in a three-dimensional setup, the results of the chosen technique demonstrate entirely new results. Thus, the following assumptions describing the results obtained can be made.

Due to the action of an ultrasonic field with a stable cavitation region, the nanoparticles are accelerated at a certain speed throughout the polymer volume, which is controlled by the sound intensity and interference pattern. The distributed nanoparticles in the polymer matrix of the composite are not static, or rather, the interface boundary is mobile. When a load is applied, this layer can compensate for large stress gradients due to its mobility (tension or compression), which leads to a uniform distribution of stresses arising in the volume, which significantly reduces the occurrence of cracks and breaks in the coatings. This explains the low values of the coefficient of friction, and its small changes over time at constant load. To confirm the above assumptions, it is necessary to carry out many additional tests, which expands the possibilities of this work for further studies.

Various approaches to the three-dimensional setup of nanofillers to improve the mechanical properties of nanocomposites have also been used. For example, Ahn *et al.* (2018, 2019), in their works, used the technique of constructing a three-dimensional template through a phase mask and filling the pores by electrolysis of particles. As the results show, the chosen method is very effective; however, it requires the

accuracy of the phased procedures and the cost of materials, which significantly complicates the process of obtaining the composite. A different approach was applied in the work of Zhao *et al.* (2018), where the authors pursued the goal of constructing a sponge structure from carbon nanotubes, which, depending on the morphology of the structure, significantly improved mechanical stiffness and allowed controlling viscoelastic characteristics. This review shows the importance of choosing a methodology for organizing nano inclusions. According to the results of this work, ultrasonic technology and a small volume of nanoscale inclusions (up to 3%) provide a significant improvement in mechanical properties and require further research in this direction. For example, this technology can be applied to other types of polymers and nanofillers to study the optical, electrical, and photoelectric properties, which significantly expands the field of experimental research.

4. CONCLUSIONS:

This paper reports on the empirical results obtained through investigating the effect of the three-dimensional organization of composite polymers, reinforced by silicon dioxide nanoparticles, on their mechanical properties. The silicon dioxide content was 1, 3, and 5%. It was found that the use of ultrasound and a smaller concentration of nanoparticles (1-3%) promote a uniform distribution of particles within the volume of the polymer matrix. The tribological analysis results revealed that a reduction in the concentration of nanoparticles from 5% to 1% resulted in a two-fold decrease in the values of the friction coefficient. The friction parameter for samples with 1 and 3% of silicon dioxide nanoparticles slightly increased over time under cyclic loading. The literature explains this behavior by referring to the regulation of the polymer interface mobility, which contributes to a uniform redistribution of applied loads across the composite volume. This slows down the coating wear. The proposed technique for organizing nano inclusions in the polymer matrix can be applied to other types of polymer nanocomposites with various properties such as wear resistance, bending strength, and impact resistance. It is expected that this proposal will help improve the mechanical properties of polymer nanocomposites.

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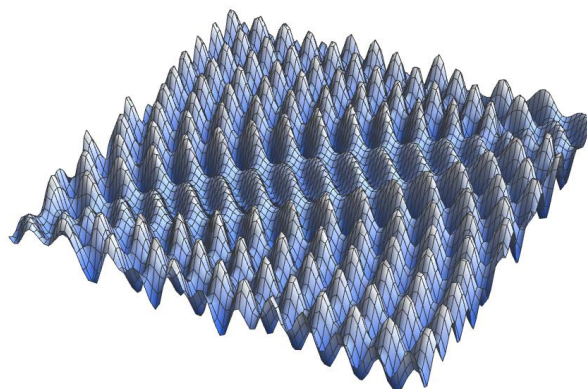


Figure 1. 3D model of the interference pattern on the surface of a liquid while equalizing the ultrasound intensity of two emitters.