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Study of the Effect of Laser Energy on the Structural Properties of Lead Sulfide Nanoparticles

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Abstract:

Lead sulfide nanoparticles (PbS) are utilized in various applications across multiple fields. A set of PbS nanocomposites was prepared on glass substrates at room temperature and under a pressure of 2.5 × 10-2 mbar using pulsed laser deposition (PLD). The laser energy used ranged between 300 mJ and 600 mJ, with a wavelength of 1064 nm and 100 pulses. The structural properties of the prepared PbS nanoparticles were analyzed using X-ray diffraction (XRD) at different laser energies (300, 400, and 500 mJ). The results confirmed the well-ordered structure of the PbS nanoparticles. The analysis showed that the composition of the metal nanoparticles was polycrystalline, with lead sulfide exhibiting the best orientation. It was observed that all films had a polycrystalline, cubic structure. The surface topography of the films was studied using an atomic force microscope (AFM). As the concentration of PbS nanoparticles increased, the inhibition effect decreased, providing insights into the surface morphology. The results indicated that the average grain size was 86.49 nm at 500 mJ laser energy, with further detailed results presented in the tables of the paper.

Keywords: Nanoparticles, (PbS), Pulse laser deposition, XRD, AFM, uv.

Introdction

Pulsed Laser Deposition (PLD) is a technique that utilizes a pulsed laser to remove surface layers of materials or alter geometric shapes by directing short, high-power laser pulses at the target surface [1,2]. This process relies on the interaction between the laser and the surface material, where the laser's light energy is converted into heat, causing the surface material to evaporate and be removed [3]. Pulsed laser ablation is applied in numerous fields, such as removing surface layers from hard and soft materials, glass, biological, electronic, and other substrates. It preferentially employs high-power laser pulses exceeding 10^8 W/cm². Most preparations of semiconductor materials like PbS using this technique result in the formation of nanostructures [4]. Nanocomposites are a key area of nanoscale research that has led to the development of marketable products. The term "nanocomposite" was first introduced in 1986, and since then, the field has grown exponentially. The science and engineering of nanocomposites have impacted various fields, including metals, plastics, ceramics, biomaterials, and electronic materials. However, some skeptics believe that the "nano" phenomenon is more hype than actual productivity [5]. Nanotechnology involves a wide range of nanomaterials and reactions, including those used in water purification. Nanoparticles, transmembranes, and nanopowders are utilized to detect biological and chemical substances such as copper, zinc, cadmium, lead, nickel, and mercury. They are also applied in nutrient detection, targeting substances like nitrites, nitrates, phosphates, organic matter, ammonia, cyanide, viruses, antibiotics, and parasites [6]. Lead Sulfide (PbS) is a significant semiconductor material characterized by a direct small band gap, an exciton Bohr radius of 18 nm, and an energy gap of approximately 0.4 eV at 300 K [7]. PbS is classified as an IV–VI compound semiconductor, featuring a cubic lattice and a cubic unit cell. Due to these characteristics, PbS is ideally suited for infrared detection applications. Its distinctive optical properties also enable numerous significant applications in the production of photovoltaic devices, such as sensors and nanoscale solar cells. Pulsed laser ablation (PLD) can effectively regulate the size and shape of PbS nanoparticles (NPs) [8]. In recent years, nanotechnology has thrived, leading to the development of various applications due to the unique properties of nanomaterials. In the visible region, PbS exhibits a significant absorption coefficient of approximately $10^5 cm^{-1}$ [9]. The fabrication of nanostructures can be accomplished using a variety of processes, including thermal evaporation, spray deposition, chemical bath deposition (CBD), and plasma atomization [10,11]. This study aims to fabricate PbS nanostructures and investigate the effect of varying laser pulse energy on their structural characteristics. Solid-state laser ablation (PLD) is a rapid, simple, and versatile method for preparing nanoparticles; however, most applications require thin layers of nanomaterials [12]. Nanotechnology includes a wide range of nanomaterials and reactions that are used in water decontamination. Nanoparticles, transmembrane materials, and nanopowders are utilized for detecting biological and chemical substances, such as copper, zinc, cadmium, lead, nickel, and mercury, as well as in nutrient applications for detecting compounds like nitrite, nitrate, organic phosphates, ammonia, cyanide, viruses, antibiotics, and parasites [13,14]. Nanomaterial oxides have garnered significant attention due to their unique electrical and chemical properties. Their chemical stability, combined with the efficiency of the pulsed laser ablation process, allows for accurate and rapid results, as well as precise control over the thickness, shape, size, and internal structure of the target material [15,16]. Additionally, this method is environmentally friendly, producing minimal vibrations and noise, which enhances the quality of industrial and scientific products while reducing production time. Structural characteristics were assessed using X-ray diffraction (XRD), revealing that all films are polycrystalline, with laser energy used ranging from below 600 mJ [17,18]

Materials and Methods

In this study, the effect of laser energy on the structural properties was investigated in the films laboratory at the University of Wasit College of Science, specifically within the Physics Department. The pulsed laser deposition (PLD) method was employed for this purpose. The experiment was conducted in a vacuum chamber at a pressure of 2.5×10^{-2} mbar. A hydraulic piston (SPECAC) was used to manufacture the lead sulfide target under a pressure of 6 tons for 10 minutes, resulting in a target disc with a diameter of 1.5 cm and a thickness of 0.3 cm. Glass slides were utilized in this research to examine the morphological properties of the lead sulfide (PbS) nanostructures. The Nd:YAG laser was operated with parameters including a wavelength (λ) of 1064 nm, energy (E) levels of 500, 400, and 300 mJ, frequency (f), and a shot count (n) of 100 pulses. The incident Nd:YAG SHG Q-switching laser beam strikes the target surface at an angle of approximately 45 degrees when entering through the window. The system is equipped with two vacuum systems, with the first being a two-stage rotating vacuum system. Lead sulfide (PbS) was prepared on a glass substrate at room temperature and pressure. The system includes a vacuum chamber, along with pressure and temperature gauges. Atomic Force Microscopy (AFM) and X-ray Diffraction (XRD) were employed to evaluate the nanostructure and analyze the crystal structure and morphology. In this study, these measurements were taken, and as will be demonstrated later, they are among the most significant structural analyses in the research. Topographical emission measurements revealed that laser energy has a critical and significant effect on the intensity of the spectral lines, with the intensity increasing as the laser energy rises. This study adhered to the Helsinki Declaration and received approval from the Ethics Committee and the Wasit Education Directorate (Date: 2024). All participants provided informed consent.

Results and discussion

XRD test

This section discusses the characterization results of the nanoparticles (NPS), focusing on crystal composition, phase analysis, morphology, particle size, and the measurement of various functional groups in the nanoparticles using specific techniques such as X-ray Diffraction (XRD). The XRD spectra of lead sulfide (PbS) produced by pulsed laser deposition (PLD) are illustrated in the figures to investigate crystal size, interplanar spacing, and the various combinations in the films. Using PLD technology, it was possible to calculate X-ray neutralization under preparation conditions including room temperature, and laser energies of 300, 400, or 500 mJ, within a vacuum of 2.5×10^{-2} mbar. X-ray diffraction analysis with a YAG laser operating at a wavelength of 1064 nm was conducted to understand the crystalline structure, crystalline size, interplanar distances, and the positions of the peaks for the films produced at varying energies. The Debye-Scherrer equation was employed to determine the crystal size of the samples. The distance between crystal planes was determined using Bragg's law. The full width at half-maximum peak (FWHM) in radial angles, Bragg's angle, and the X-ray wavelength (0.15406 nm) were utilized in the analysis of the XRD pattern of the composite nanostructure. The presence of both materials is confirmed by the appearance of PbS peaks in the diffraction pattern. This paper provides a detailed account of the growth process of the nanostructures and their composite forms. The sample displays a set of peaks that represent the structure of the deposited membrane at specific angles, indicating the development of crystalline planes when 300 mJ of laser energy was applied, as illustrated in Figure 1, which confirms its multi-crystalline structure. Figure 2 presents the X-ray diffraction (XRD) spectra at 400 mJ of laser energy, revealing that the sample also possesses a multi-crystalline structure. This sample includes a collection of peaks that represent the structure of the deposited membrane at various angles, along with the emergence of crystalline planes at those peaks. Figure 3 displays the X-ray diffraction (XRD) spectra at 500 mJ of laser energy, confirming that the PbS films exhibit a multi-crystalline structure. However, a slight shift in the position of the peaks is observed, attributed to the interaction process that combines two phases within the same particle **[**19,20**]**.

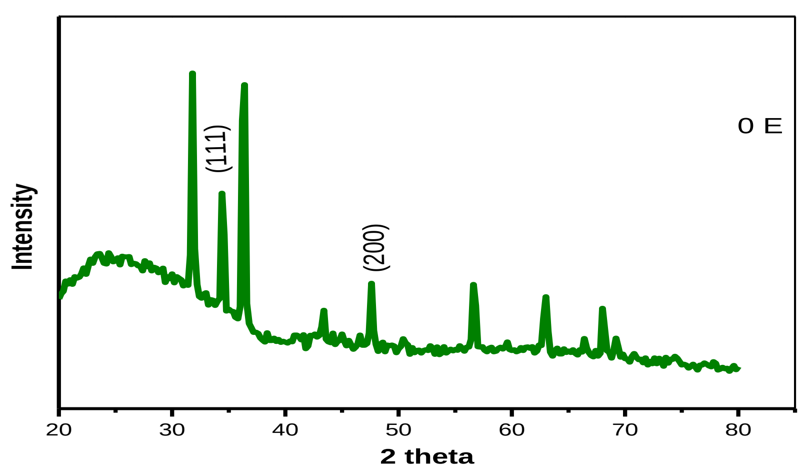

Figure 1: The XRD spectra of the PbS nanostructure generated by PLD at a laser energy of 300 mJ.

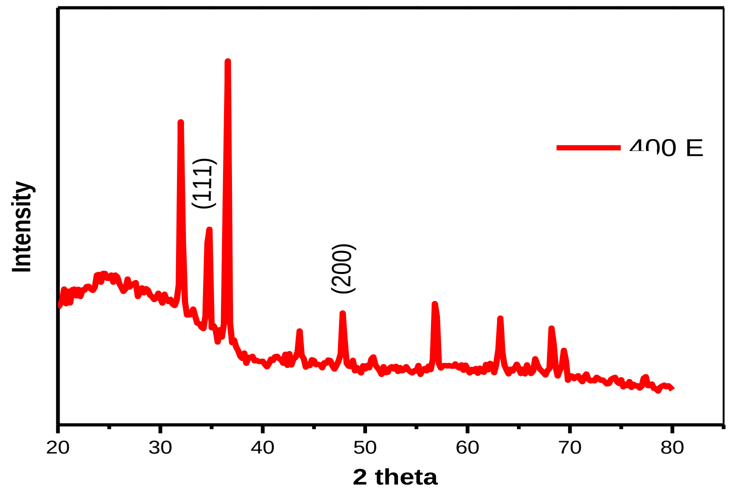

Figure 2: The XRD spectra of the PbS nanostructure generated by PLD at a laser energy of 400 mJ.

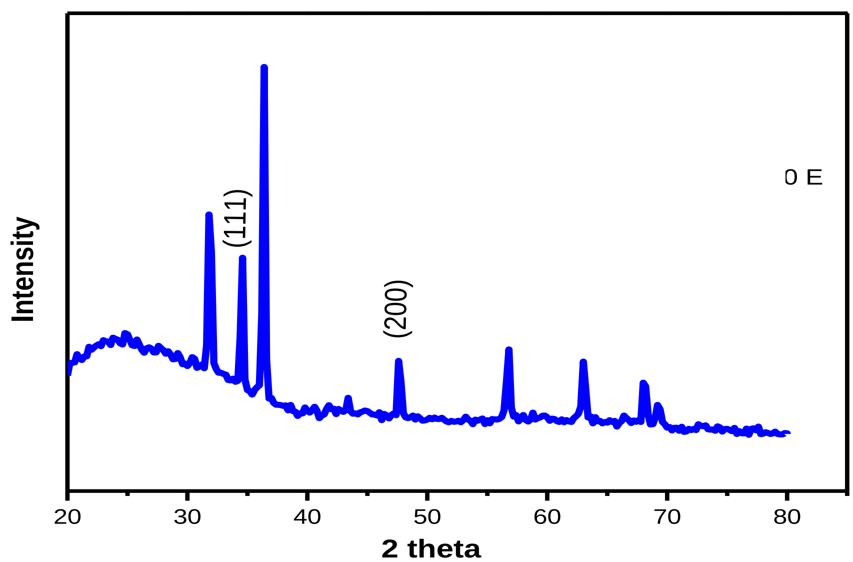

Figure 3: The XRD spectra of the PbS nanostructure generated by PLD at a laser energy of 500 mJ.

Table 1: The XRD analyse parameters of pbs nanocoposite.

Sample	Av. C.S PbS	Phase	card No
300 mJ Energy	14.67	Cub.PbS	96-901-3404
400 mJ Energy	20.14	Cub.PbS	96-901-3404
500 mJ Energy	28.66	Cub.PbS	96-901-3404

AFM test

The AFM images presented in Figures were captured at pulsed laser energies of 300, 400, and 500 mJ. These images reveal the formation of semi-circular clusters with granular sizes, along with the presence of a smooth surface and excellent adhesion to the glass substrate. This measurement was conducted in the laboratory. As the laser intensity increases, the amount of material removed leads to an expansion of the granule size, resulting in an increased surface area of the nanostructure. Consequently, there are fewer particles in this region. The layers of the nanostructure formed during the deposition process ascend to the surface through the interaction of the ablation with byproducts from the nanostructure, which occur within the plasma, ions, and uncharged ablation fragments, each exhibiting different diffusion rates. A key finding from this measurement is presented in the table below: The ions correspond to high-energy (rapid) fragments, and their velocity is influenced by the frequency of collisions between the uncharged fragments and charged particles. The following table presents the key parameters observed. The occurrence of larger granules, resulting from multiple impacts, is amplified due to high-intensity laser ablation or cutting, which enhances the roughness of the nanostructure's surface, as indicated by the atomic force microscopy (AFM) results. Both the AFM and X-ray diffraction (XRD) measurements are explained in this context. This study involved the preparation of lead sulfide (PbS) powder using a laser method, which is characterized by its low economic cost compared to other preparation techniques. Three sample systems were examined. Energy dispersive X-ray spectroscopy (EDX) analysis on the PLD system samples provided insights, with the AFM diffraction spectrum being investigated as well. The lead sulfide nanoparticles utilized in this research exhibited an average crystal size in a cubic phase.

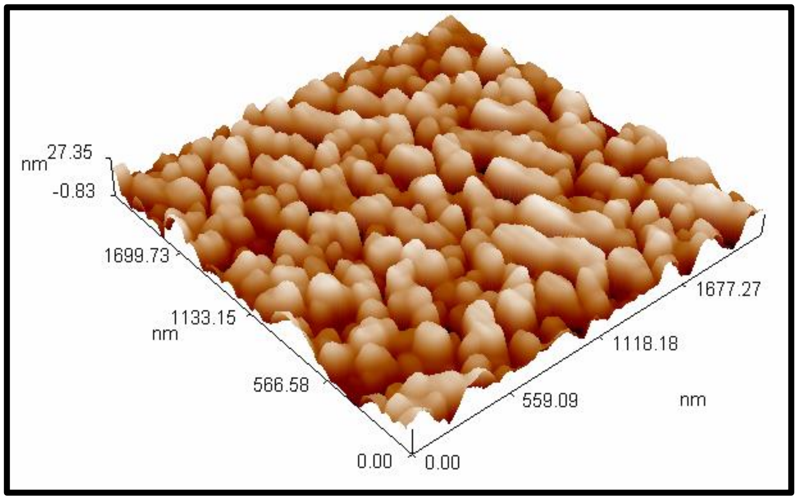

Figure 4: Topographical images of PbS nanocomposite at a laser energy of 300 mJ.

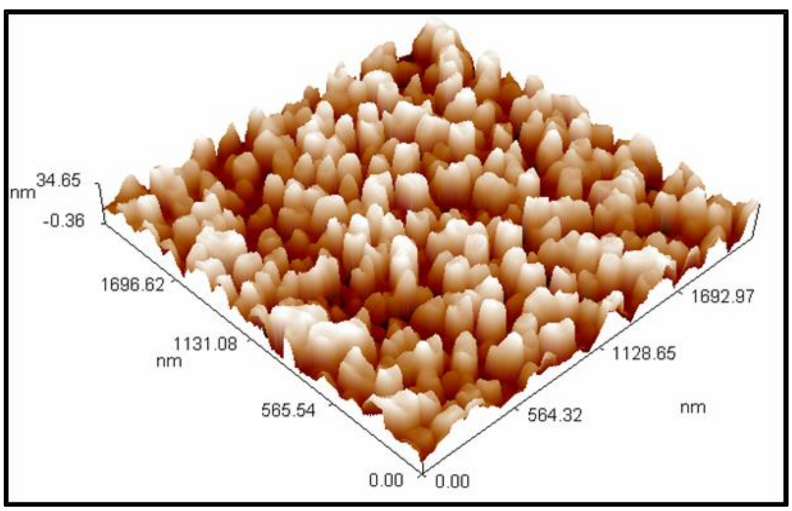

Figure 5: Topographical images of PbS nanocomposite at a laser energy of 400 mJ.

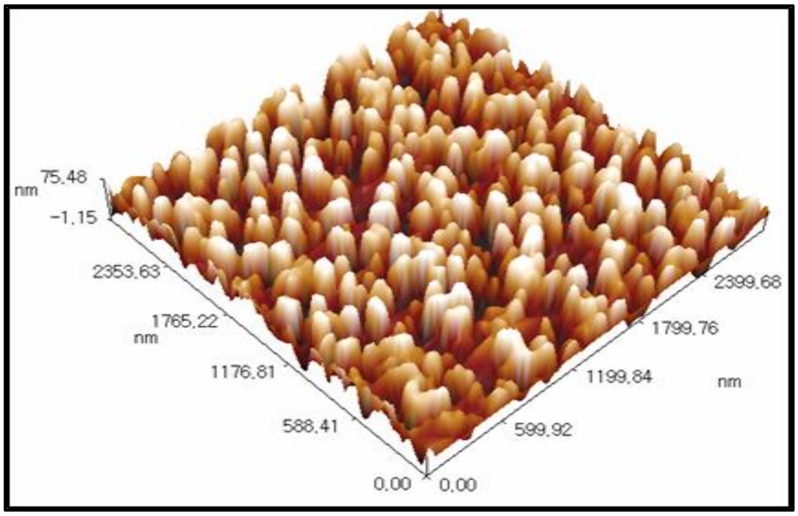

Figure 6: Topographical images of PbS nanocomposite at a laser energy of 500 mJ.

Table 2: AFM Analysis Parameters of PBS Nanocomposites

Sample	Grain size (nm)	Ave. Roughness (nm)
300 mJ Energy	61.65	4.31
400 mJ Energy	76.78	5.51
500 mJ Energy	86.49	8.34

UV test

Using UV–Vis spectroscopy to determine the optical properties of PbS, Fig. 7 shows the absorption characteristics of the nanoparticles at various temperatures. The band gap energy of the nanoparticles was measured at 300, 400, and 500 mJ of laser energy. This measurement, expressed in nanometers, indicates that the nanomaterial exhibits high crystallinity. The calcination of the samples reveals absorption at specific wavelengths. The PbS nanoparticles exhibit a transition in absorption from the UV to the visible light spectrum. The UV-visible absorption spectra of the nanoparticles are presented in Fig. 7. As illustrated in the figure, there is a strong excitonic absorption peak in all samples. This peak is attributed to the significant binding energy of excitons and the excellent optical quality of the nanoparticles [20]. With increasing content, the redshift in the band edge absorption peak is minimal. The calculated band gap of the PbS nanoparticles shows a decline, consistent with previous studies. Two primary factors may contribute to variations in band gap energies: the quantum size effect and modifications to the electronic structure. The decrease in the band gap observed in Fig. 8 is unlikely to be attributed to the quantum size effect in the as-synthesized samples. It is reasonable to expect a slight decrease in the band gap energy (Eg) with an increase in laser energy concentration, possibly due to doping-induced band edge bowing.

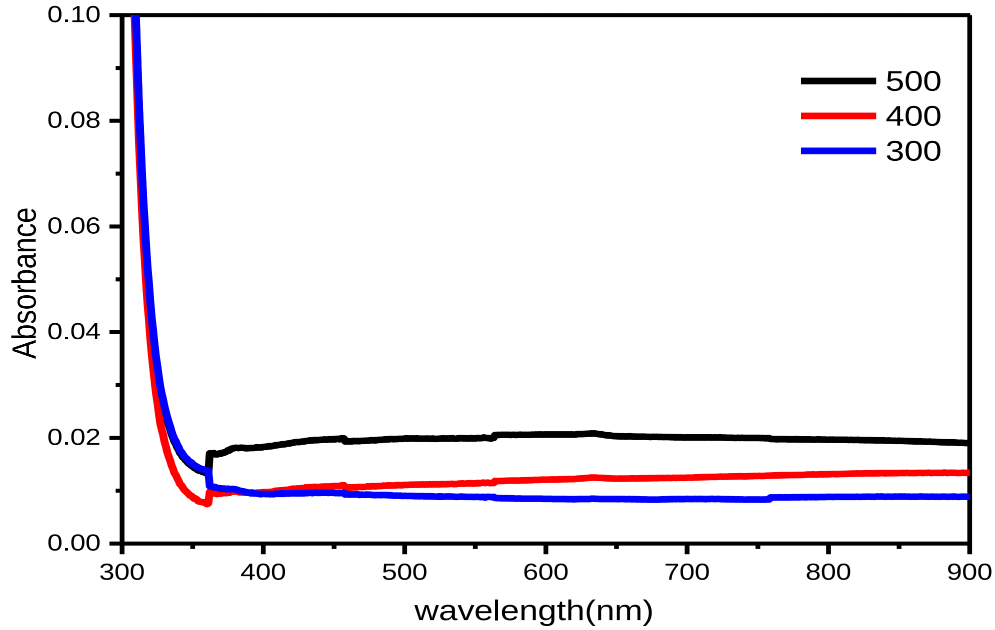

Figure 7: UV–Vis Absorption Spectra of PbS Nanoparticles at 300, 400, and 500 mJ Laser Energy.

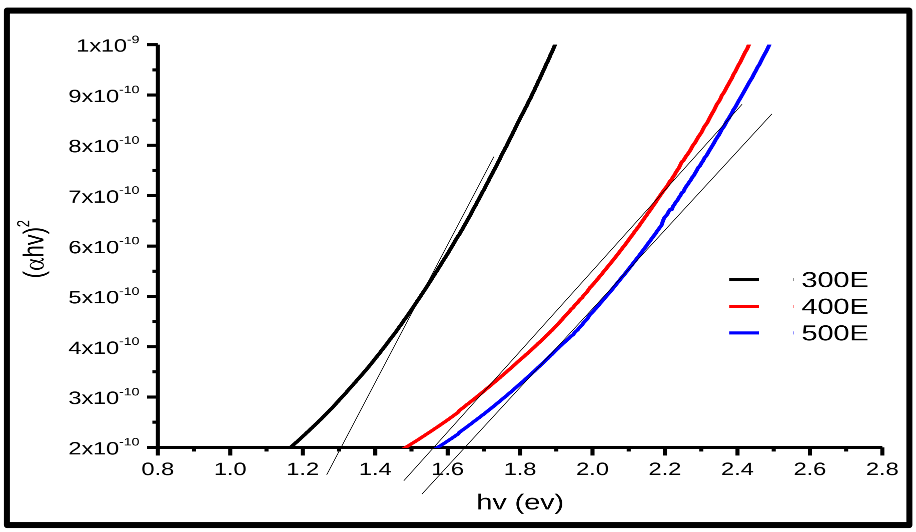

Figure 8: $(\alpha h\nu)^2$ vs. Energy of Laser for the Prepared PbS Nanoparticles at 300, 400, and 500 mJ.

Conclusion

In this study, measurements were conducted for the three produced samples, and an X-ray diffraction (XRD) test was performed. The analysis led to the following conclusions: the results revealed that the samples possess a nanostructured, polycrystalline nature. According to the findings from atomic force microscopy (AFM), the grain size increases with a higher percentage of laser energy. The bactericidal effect of the nanoparticles (NPs) agents increased with temperature. XRD results indicated that spherical lead sulfide (PbS) nanoparticles were successfully obtained. AFM results confirmed that the spherical PbS particles were embedded in the lattice and adhered to the substrate surface. Laser irradiation appeared to enhance the photocatalytic activity of PbS against Staphylococcus aureus. It was observed that the diameter of the inhibition zone decreases with an increase in the particle size of the NPs. The use of the polycrystalline structure powder prepared in this study as feedback components in a random laser system will be compared with results obtained using other methods.

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