

Photoluminescence-Based Real-Time Monitoring for Dimensional Characterization of Nanostructures Fabricated by Direct Laser Writing (DLW)

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Abstract:

Real-time monitoring during nanostructure fabrication is essential to ensure fabrication accuracy and process reliability. In Direct Laser Writing (DLW), optical microscopy is commonly employed to observe the fabricate on process; however, it only provides visual images and cannot accurately determine the dimensions or geometry of the fabricated structures. This study proposes a photoluminescence (PL)-based monitoring technique for real-time dimensional characterization during DLW fabrication. The proposed method utilizes differences in PL intensity between polymerized nanostructures and the surrounding photoresist. By scanning a low-power laser probe through the fabrication region and recording the PL response, the shape and dimensions of embedded nanostructures can be reconstructed during fabrication. Experimental results demonstrate that the proposed PL-based monitoring technique can accurately reconstruct the geometry of fabricated structures. After compensation of systematic measurement errors, the average dimensional errors were reduced to 0.48 μm and 0.32 μm in the horizontal and vertical scanning directions, respectively. These results demonstrate the potential of the proposed technique as a non-destructive real-time monitoring method to improve the efficiency, accuracy, and reliability of DLW-based nanostructure fabrication.

1. Introduction

Many researches and applications of nanostructures are used in order to obtain special properties that are used according to the requirements, such as for electronic[1], mechanic[2,3], and also biocompatible components[4,5]. And many researches are being done to develop nanostructures

with unique characteristics., such as for excellent thermal conductivity[6,7], enhanced chemical reactivity[8–10], improved mechanical properties[11, 12], and tunable optical properties[13, 14].

Nanostructures will be characterized to determine the success of the fabrication process, which usually consists of properties that are greatly influenced by

their shape and dimensions [15,16]. Nanostructures for metamaterial absorbing applications, the amount of absorption power related to frequency and wavelength is greatly influenced by the shape and dimensions [17,18]. The refractive index, which is the main parameter that greatly affects the performance of photonic sensors, is highly dependent on the shape and dimension of the structure [19,20]. The dimension of the nanocoating is closely related to corrosion resistance by reducing the possibility of oxidation on the surface[19,21]. The volume, shape, and dimension of droplets in drug delivery, tissue engineering, and cell encapsulation in microfluidics are greatly influenced by the shape and dimension of the channels [22,23]. One of the main problems in nanostructure fabrication is related to monitoring that can guarantee the success of nanostructure fabrication. Currently, cameras can be used to monitor the results of nanostructure fabrication during the fabrication process, but they can only provide visual shapes, but it cannot provide the dimension of the resulting nanostructures. And there are several testing methods for nanostructures, Scanning Electron Microscope (SEM) and Atomic Force Microscope (AFM) are techniques that are widely used to characterize the shape and dimensions of nanostructures[24], but these methods can be carried out after the nanostructure fabrication process and require special treatment.

To overcome the problem of failure when fabricating nanostructures using DLW, this research was carried out which can provide a detailed monitoring method that can provide the shape and dimension of nanostructures during fabrication process. The technique used is based on the difference in photoluminescence intensity between the nanostructure and the photoresist that covers it. By using laser power below the threshold for structure formation, this technique does not cause damage to the nanostructures .

2. Methods

The experiments were conducted using a Direct Laser Writing (DLW) system, as shown in Figure 1a, a femtosecond Ti:Sapphire laser (Mai Tai, Spectra-Physics) was used for both nanostructure fabrication and real-time monitoring. The objective lens used to focus the laser beam is a lens with a numerical aperture of 1.42, and oil immersion oil is applied between the objective lens and the glass substrate to reduce the deviation of the laser beam towards the photoresist[24]. To filter the light entering the recorder which is photoluminescence light, a short-pass filter (FESH0550, Thorlabs) is used. The photoresist used is SZ2080 which is added with 1% 4,4-bis (diethylamino) benzophenone (BIS) photoinitiator, which it will emit photoluminescence with a wavelength of 530+20 nm when exposed by laser beam [25].

The principle of detailed monitoring using photoluminescence technique is based on the difference in photoluminescence intensity of the fabricated structure and photoresist. As shown in Figure 1b, The observed reduction in photoluminescence (PL) intensity when the laser probe traverses a polymerized structure is primarily attributed to the consumption of the BIS photoinitiator during the cross-linking process, which significantly reduces the density of available fluorophores compared to the surrounding liquid photoresist. Therefore, by scanning the laser focal spot along the X-, Y-, and Z-axes and monitoring variations in PL intensity, the shape and dimensions of embedded structures can be reconstructed.

The nanostructure that is still inside the photoresist is subjected to development process for removing the photoresist and obtain nanostructures. The development process is carried out by placing the photoresist in 1-propanol:isopropanol (1:1), and cleaning nanostructures by placing the

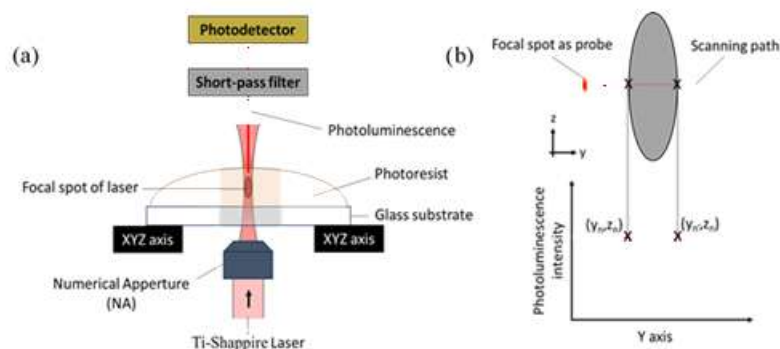


Figure 1.(a) The setup of Direct Laser Writing (DLW) for nanostructures fabrication and monitoring using photoluminescence technique, (b) Principle of photoluminescence technique for detailed monitoring in DLW.

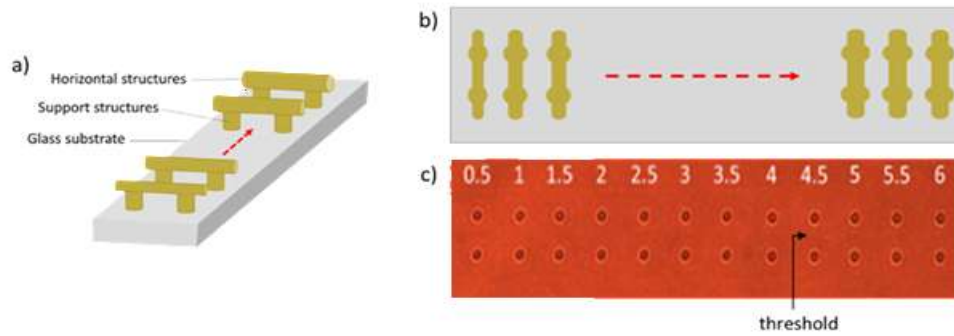


Figure 2. (a) Nanostructures for test resolution and detailed monitoring in DLW, (b) Topview of nanostructures for test resolution and detailed monitoring in DLW (c) Image of resolution test for determining the threshold of laser power for fabrication

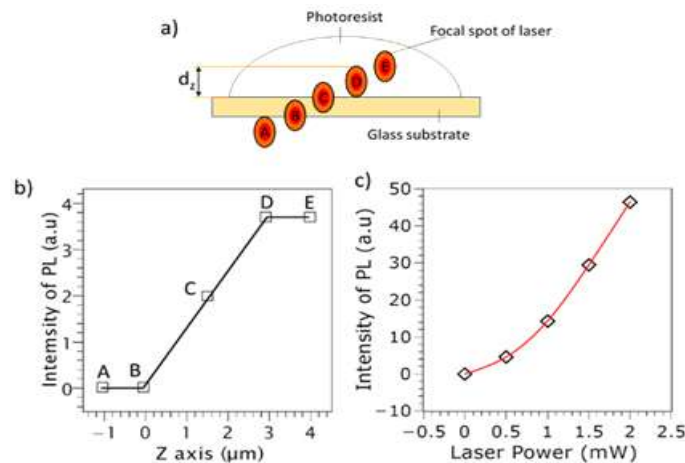


Figure 3. (a) Positioning the laser focal spot to determine the intensity of PL. (b) The intensity of PL corresponds to each position of the laser focal spot. (c) The intensity of PL corresponds to the laser power at the position of the laser focal spot within the photoresist.

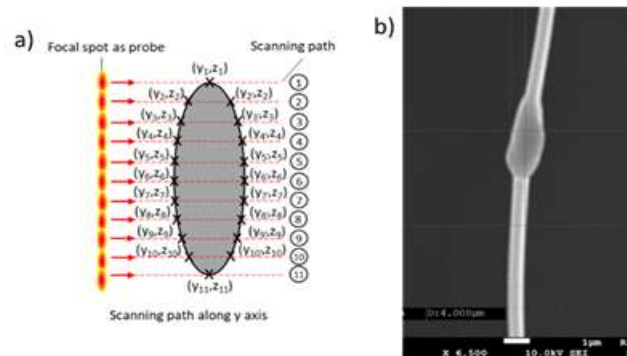


Figure 4. (a) Horizontal scanning to carry out measurements for detailed monitoring of fabricated horizontal structures, (b) additional structures formed due to repeated subthreshold laser excitation

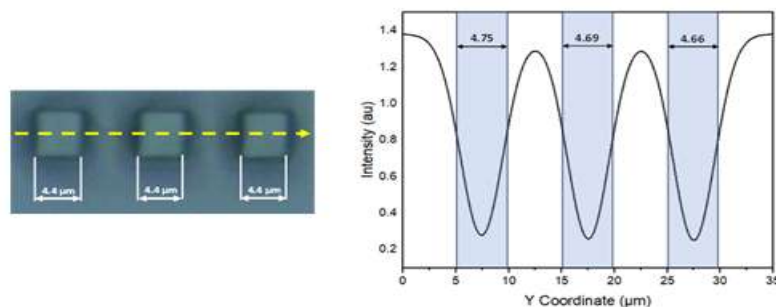


Figure 5.. (a) Image of monitoring the creation of a rectangle in the DLW process, the yellow arrow line is the direction of the scanning process using the PL technique. (b) Results of measuring the cross-section of the rectangle in the horizontal direction using the PL technique

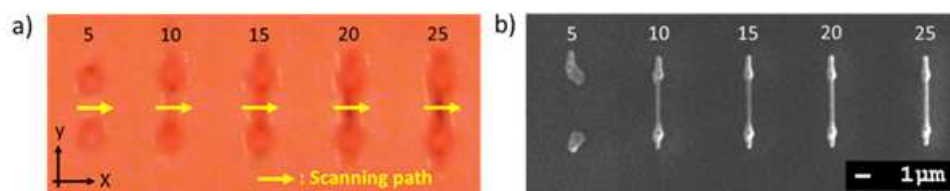


Figure 6..(a) Image from a camera used for monitoring during nanostructure fabrication with laser power 5 mW – 25 mW, **(b)** SEM image of nanostructures resulting from the fabrication process with laser power 5 mW – 25 mW that has been carried out in the development process

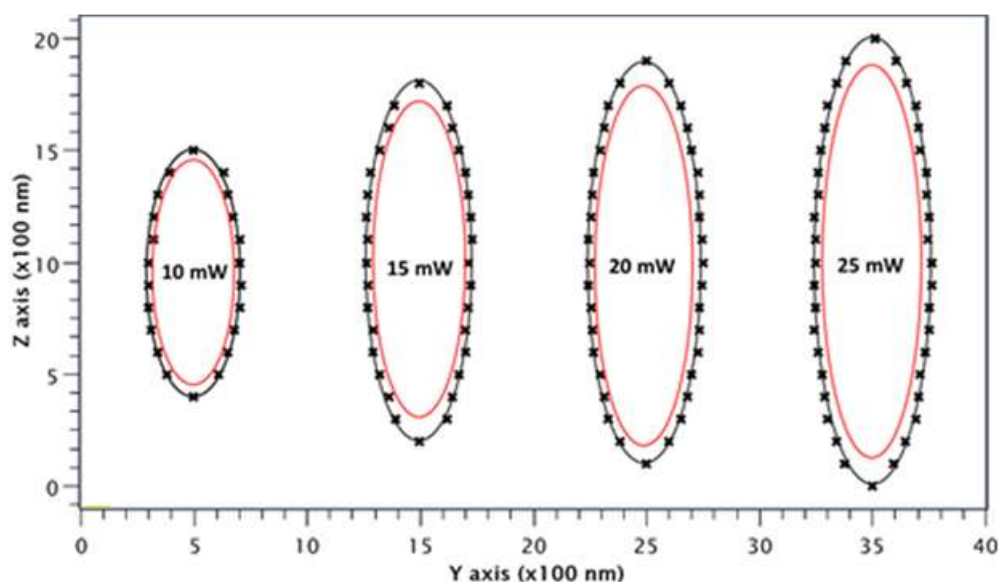


Figure 7. The results of nanostructure measurements with the shape as in Figure 5a using the PL technique in the horizontal direction.

nanostructures in ethanol, then the nanostructures were dried with a dryer (JCPD-5). The resulting nanostructure will be characterized using Scanning Electron Microscopy (SEM). The results of measurements using SEM will be used as a basis for comparison with the results of measurements using the photoluminescence technique to determine the accuracy of the photoluminescence technique.

3. Results and Discussions

The research began by determining the threshold parameters for the laser excitation process on SZ 2080 photoresist with 1% BIS photoinitiator. This procedure was performed to determine the minimum laser power required to initiate structure formation in the photoresist. The laser power threshold value that has been obtained will be used as laser power in the scanning process for measurement. The laser parameters used in the laser excitation process in the photoresist range from 0.5 mW to 6 mW with laser focal spot velocity of 100 $\mu\text{m/s}$. The threshold power was subsequently used as a reference for selecting the probe laser power employed during the measurement process as in Figure 2a. and Figure 2b., the structures are horizontal structures supported by two support pillars on a glass substrate.

The horizontal structures are the part that will be used to determine the laser power threshold, that the laser power used is 0.5 mW to 6 mW. Figure 2c shows that the laser power threshold to form a horizontal structure is 4.5 mW, which is shown by a thin and slightly bent horizontal line. The photoluminescence intensity generated by the probe laser during scanning was recorded and analyzed. When the laser excites the photoresist, it will emit photoluminescence which is different from when the laser excites the photoresist material which has undergone photopolymerization. When a photoresist is excited by a laser, a reaction will occur in the photoresist and produce light scattering in the form of photoluminescence with a certain wavelength, which usually has a longer wavelength than the laser light used for excitation.

To determine the dimension of the focal spot used as a probe in this research, a scanning was carried out in the vertical direction (Figure 3a), by moving the laser focal spot in the z-axis direction from under the glass substrate (A) and through the glass substrate (B). until inside the photoresist (C-D-E). That when the laser focal spot is under the glass substrate and not yet in the photoresist (A, B), the photopolymerization process does not occur so it does not release energy in the form of

photoluminescence, which means the photoluminescence intensity is 0. After part of the laser focal spot is in the photoresist (C), that at position C, half the focal spot of the laser beam is inside the photoresist, then excitation of the laser beam on the photoresist produces photoluminescence of 1.8 au. The larger the laser focal spot that is inside the photoresist (between C-D), the greater the intensity of the photoluminescence produced (Figure 3b) and the greatest photoluminescence intensity is obtained when the entire focal spot is inside the photoresist (D, E), that positions D and E have the same photoluminescence intensity of 3.7 au. From the change in intensity value from positions B to D, it can be used to determine the height of the laser beam focal spot (dz) of $3\text{ }\mu\text{m}$. Based on the focal spot height (dz), a calculation was then carried out taking into account the laser wavelength used (800 nm), and a focal diameter of $0.76\text{ }\mu\text{m}$ was obtained. Detailed determine the reference point of nanostructure. And in Figure 3c, it can be seen that the photoluminescence intensity is directly proportional to the laser power used for excitation. Photoresist that has been excited and the photopolymerization process has occurred will not produce photoluminescence because when the laser is excited, no more photopolymerization reactions occur which release energy of photoluminescence. Monitoring using direct laser writing use focal spot as a probe to measure the workpiece. The probe that will be used is a laser with laser power that does not cause photopolymerization which causes structures to form in the photoresist measurement data. Based on the data obtained as in Figure 2c, in this study a probe was used in the form of a laser with a laser power of 0.5 mW , that this laser power produces a photoluminescence intensity of around 0.9 au , and is far below the laser power that causes photopolymerization, around 10% of the photopolymerization threshold (4.5 mW).

To obtain shape and dimensions data from the horizontal structure resulting from DLW fabrication, scanings were carried out in the y-axis direction with several steps in the z-axis direction (Figure 4a). To obtain the accuracy of the monitoring using the photoluminescence technique, a horizontal structure was also fabricated with one pole in the z-axis direction and a horizontal structure was also made and installed on the pole structure, and the position increase in the z-axis direction was $0.1\text{ }\mu\text{m}$. The focalspot is moved from under the glass substrate to the inside of the photoresist, to obtain the height of the horizontal structure. Measurements were carried out on a structural sample in the form of a horizontal structure supported by 2 supports on the right and

left of the horizontal structure which was attached to the glass substrate, as shown in Figure 2a, and shown in Figure 4a, an image of the results of observations with an optical microscope. Scanning for detail monitoring was done by scanning. In the scanning process, in addition to excitation with laser power and speed below the threshold, the distance between scanning processes must be determined so that no new structures are formed. Figure 4b shows the formation of a new structure as a result of repeated excitation at a power below the threshold. This occurs as a result of the accumulation of sufficient power for polymerization to occur. Laser power above the threshold will cause surface modifications to the structure, similar to those used in laser ablation and coating[26, 27]. Scanning to determine the shape of the horizontal structure in the z-axis direction is shown in Figure 4a. Figure 6b shows the scanning process with a power of 1 mW , a focal spot speed of $100\text{ }\mu\text{m/s}$, and a distance between scanning processes of $1\text{ }\mu\text{m}$, which a new structure is formed around the horizontal structure. Therefore, in this study, the scanning process parameters used are a laser power of 0.5 mW , a speed of $100\text{ }\mu\text{m/s}$, and a distance between scans of 1 m . And Figure 6b shows that no new structures are formed around the monitored horizontal structure. The monitoring process experiment was conducted on a square structures located within the photoresist as shown in Figure 5a. The monitoring process was carried out by scanning in the horizontal direction through the inside of the beam indicated by the yellow dashed line. The results of this scanning process are shown in Figure 5b, which there are differences in PL intensity when passing through the photoresist and the square structures within the photoresist. And it was determined that the dimension of the cubes in the X-axis direction is $4.4\text{ }\mu\text{m}$. when compared to the measurement results with the microscope, there is a difference of $0.03\text{ }\mu\text{m}$. It can be concluded that measurements using this PL technique produce high accuracy. The structures are created through photopolymerization of photoresist using direct laser writing techniques. on the glass substrate. After the structure has been formed in the photoresist, the dimensions of the horizontal structure formed are then measured. The measurement technique is carried out by carrying out a scanning process in the horizontal (y-axis) and vertical (z-axis) directions, so that the cross-sectional shape and dimensions of the horizontal structure is obtained, which can be seen in Figures 6a and 6b. The horizontal structure that will be used as an experimental sample was fabricated using variable laser power parameters of 5 mW to 25 mW and a fixed focal spot speed of $100\text{ }\mu\text{m/s}$. The scanning process is carried out in the vertical direction or in the z-axis direction (Figure 4a).

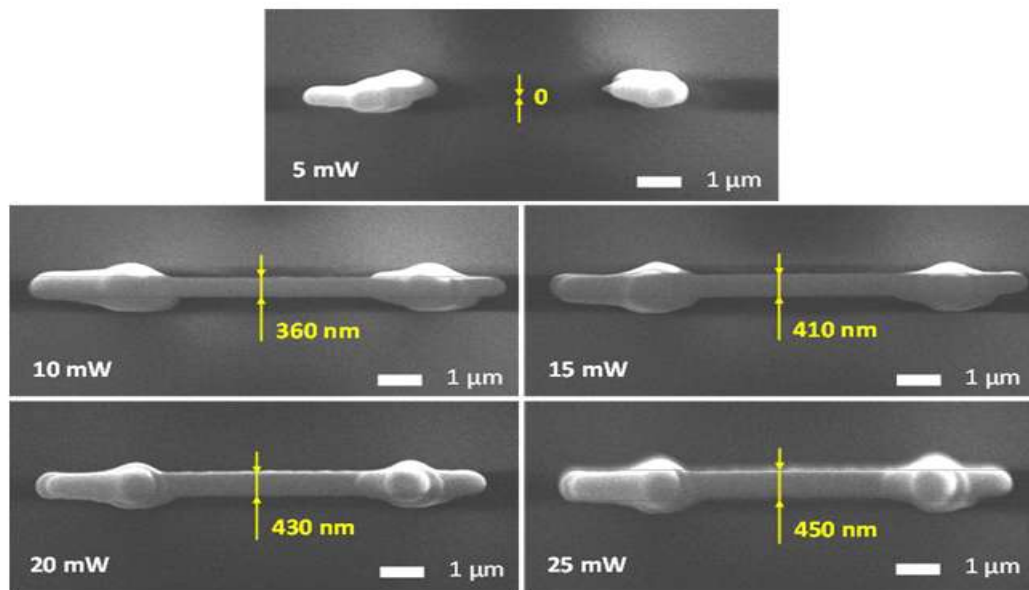


Figure 8. SEM images of several horizontal structures supported by two poles at their ends, in which horizontal structures were made with laser powers of 5 mW, 10 mW, 15 mW, 20 mW, and 25 mW

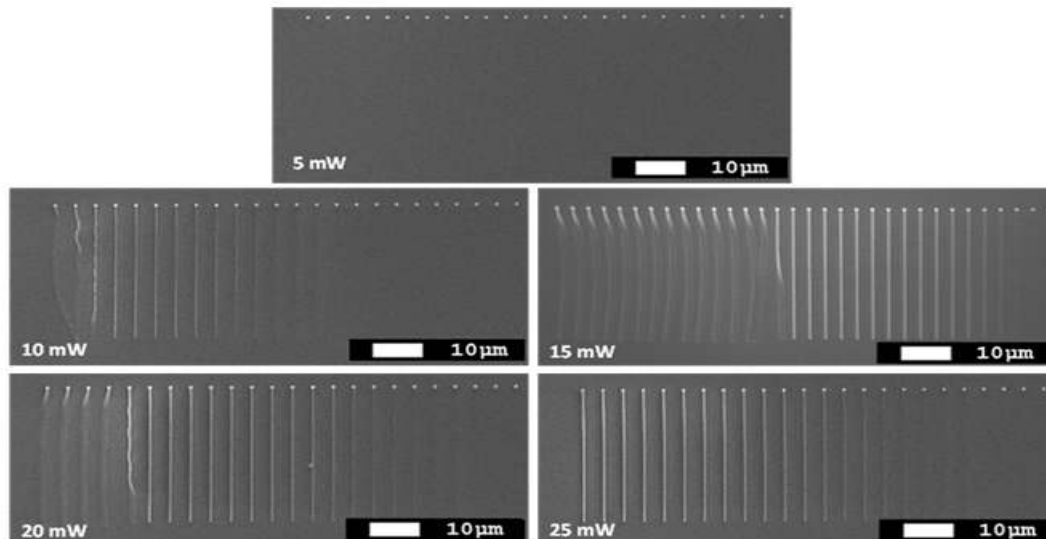


Figure 9. SEM images for measuring the vertical dimensions of microstructures fabricated by DLW with powers of 5 mW, 10 mW, 15 mW, 20 mW, and 25 mW

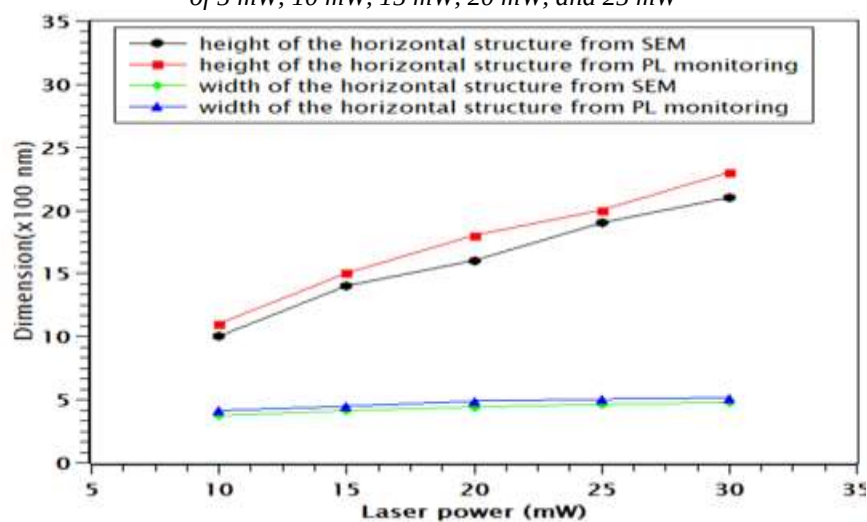


Figure 10.. Results of structural measurements using PL techniques and measurements using SEM in the horizontal direction

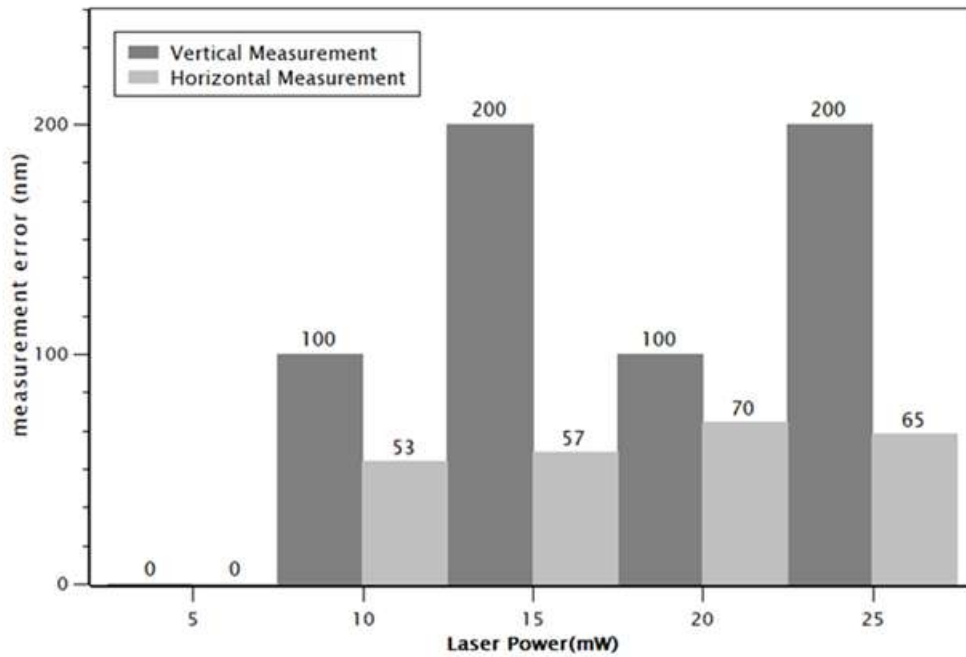


Figure 11.. Errors in measurements using the PL technique compared with SEM measurements in the horizontal and vertical directions

Table 1. Comparative dimensional analysis of fabricated structures before and after systematic error compensation by laser power 10 mW - 30 mW

Laser power for fabrication (mW)	Error Dimension (μm)			
	Horizontal Scanning		Vertical Scanning	
	Before Compensation	After Compensation	Before Compensation	After Compensation
10	1.00	0.40	1.40	0.80
15	1.00	0.40	0.35	0.25
20	2.00	0.60	0.45	0.15
25	1.00	0.40	0.40	0.20
30	2.00	0.60	0.38	0.22

Figure 6a shows the image of the structure seen in an optical microscope when the fabricated structure is still inside the photoresist, with the yellow arrow line representing the horizontal scanning direction that the scanning passes through the horizontal structure. The fabricated structure that has been removed from the photoresist is then measured using SEM as shown in Figure 6b. Horizontal scanning is carried out in the horizontal direction (y axis) by passing through the horizontal structure from the top to the bottom of the horizontal structure. Scanning was carried out several times with a step of 1 μm in the z-axis direction according to the height of the fabricated horizontal structure. Measurements are carried out by observing the laser focal spot when it touches the structure which can be seen by a decrease in photoluminescence intensity. The start and end points of the surface of the structure being measured can be seen by changes in photoluminescence intensity. One of the scanning results graphs for measuring horizontal structures can be seen in Figure 7. On the horizontal structure fabricated with a laser power of 10 mW, scanning

was carried out in the y-axis direction with a step of 1 μm in the z-axis direction, and 11 scans were obtained which changed in photoluminescence intensity when the laser focal spot hit the horizontal structure, which can be seen in Figure 5a. So it was determined that the height of the horizontal structure is 1.1 μm. Using the same scanning method, on horizontal structures resulting from laser fabrication with several different powers, a decrease in PL intensity was obtained when crossing the horizontal structure with a number of scanning steps in the z-axis direction sequentially from 15 mW, 20 mW, and 5 mW are 17 times, 19 times, and 22 times. From the number of scanning steps in the z-axis direction, it was determined that the shape of the structure is horizontal with a height of 1.6 μm, 1.8 μm, and 2.1 μm and a width of 468 nm, 500 nm, and 515 nm. For the coordinate points that the PL intensity decreases, it is indicated by a cross as can be seen in the Figure 7. And from the Figure 7, the cross-sectional shape of the horizontal structure is oval image. Figure 8 is an image of horizontal structure characterization using SEM, and from this SEM characterization it

was found that in fabrication with 5 mW laser power no horizontal structure was formed. And the results of fabricating horizontal structures with laser powers of 10 mW, 15 mW, 20 mW, and 25 mW respectively are as follows: 300 nm, 410 nm, 430 nm, and 450 nm. Figure 9 is SEM image for determining the height of structures. To characterize the height of the horizontal structure via SEM, several horizontal line structures were fabricated in the y-axis direction attached to the pole structure with each step the horizontal structure was raised in the z- axis direction by 0.1 μm . The horizontal structure created uses the same parameters as when fabricating a horizontal structure supported by 2 poles, a focal spot speed of 100 $\mu\text{m/s}$ and a laser power of 5 mW – 25 mW with steps of 5 mW. The height of the horizontal structure is determined by observing the horizontal structure attached to the glass substrate, and it was determined that fabricating the horizontal structure with a laser power of 5 mW does not form a horizontal structure, and sequentially for laser powers of 10 mW, 15 mW, 20 mW, and 25 mW obtained horizontal structure heights of 1,100 nm, 1,400 nm, 1,600 nm, and 1,900 nm. Figure 10 shows the differences in the width and height of the horizontal structure according to the laser power used for fabrication. The characterization results from SEM are used as a reference for calculating the accuracy of photoluminescence measurements, and from the calculations the average tolerance of the photoluminescence technique is 84.5 nm, as shown in Figure 11. The measurement error rate of the photoluminescence method is caused by the dimension of the laser focal spot used. Therefore, to overcome this problem, a compensation technique is needed to produce accurate measurements[28, 29]. Compensation techniques are carried out by reducing or increasing the focal spot dimension in the measurement results. In addition to the influence of the laser focal spot dimension, there is interference from the signal received by the photodetector, which causes errors in determining the coordinate points which photoluminescence intensity decreases or increases.

To mitigate dimensional discrepancies caused by the finite size of the laser focal spot, a systematic compensation was applied by introducing an offset of 0.60 μm in the horizontal direction and 1.40 μm in the vertical direction, based on the calibrated Point Spread Function (PSF) of the system. Table 1 shows the monitoring detail error before and after compensation, which the average error number before compensation is 1.40 μm and 0.60 μm , and after compensation is 0.48 μm and 0.32 μm in the horizontal and vertical scanning directions, respectively. From this study, it can be concluded

that the PL technique is very suitable for use as a principle in monitoring that can produce shapes with accurate dimensions.

4. Conclusions

The photoluminescence technique proposed and applied in this study results in accurate real time monitoring of the shape and size of nanostructures. This study provides a non-destructive measurement method that can be used during nanostructure fabrication using the DLW technique. With this monitoring technique, it will reduce errors, increase the efficiency and effectiveness in microstructure fabrication using DLW. From this research, it was shown that the PL monitoring technique used in DLW can produce shapes with accurate dimensions with error levels of 0.48 μm and 0.32 μm in the horizontal and vertical scanning directions.

To improve the accuracy of monitoring using the photoluminescence method, precise determination of the laser focal spot size is required to facilitate and minimizing the difference between the photoluminescence signal and the interference signal captured by the photodetector is also crucial, which allows precise determination of the coordinates of the decrease or increase in photoluminescence intensity.

Research that can be conducted to improve the accuracy of this photoluminescence technique involves developing a monitoring method to determine the focal spot dimensions of the laser used. In addition, the use of photoluminescence methods for additional measurement methods in other fabrication techniques is a challenge so that this method becomes widely used..

Author Statements:

- **Ethical Approval:** This research did not involve human participants, animals, or any biological materials requiring ethical approval. Therefore, ethical approval was not required for this study.
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- **Author Contributions:**

- **Budi Suhendro:** Collecting experimental data, analyzing results, and writing articles.
- **Edy Yulianto:** Conceptualization, methodology, supervision, project administration, writing – original draft, and overall responsibility for the research activities.
- **Marsetiayu Ningsih:** Investigation, sample preparation, and fabrication nanostructures, characterization, data acquisition, and preliminary data analysis.
- **Suroso:** Equipment preparation, fabrication process implementation, and technical support during experimentation.
- **Diandono Kuntjoro :** Data analysis, interpretation of measurement results, and validation of experimental findings.
- **Agus Purbhadi:** Electrical system preparation, instrumentation support, photodetector data acquisition, and technical assistance.
- **Vygantas Mizeikis:** Research supervision, scientific guidance, resource provision, review, and critical revision of the manuscript.
- All authors contributed substantially to the conception and design of the study, participated in the interpretation of data, reviewed the manuscript, and approved the final version for publication.
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- **Data Availability Statement**
- The data supporting the findings of this study are available from the corresponding author upon reasonable request. All relevant data generated or analyzed during this study are included in the manuscript or are available from the authors.

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