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# Laser Densification of Prepared $SiO_2$ Sol-Gel Thin Films

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## Abstract:

$SiO_2$ nanostructure is synthesized by the Sol-Gel method and thin films are prepared using dip coating technique. The effect of laser densification is studied. X-ray Diffraction (XRD), Fourier Transformation Infrared Spectrometer (FTIR), and Field Emission Scanning Electron Microscopy (FESEM) are used to analyze the samples. The results show that the silica nanoparticles are successfully synthesized by the sol-gel method after laser densification. XRD patterns show that cristobalite structure is observed from diode laser (410 nm) rather than diode laser (532 nm). FESEM images showed that the shape of nano silica is spherical and the particles size is in nano range (≤ 100 nm). It is concluded that the spherical nanocrystal structure of silica thin films is successfully densified by Doide laser (410 nm).

**Key words:** $SiO_2$ thin films, Sol-gel, Densification, XRD, FESEM.

## Introduction:

Nanomaterials have attracted extensive attention due to the rapid development of Nanoscience and Nanotechnology in multidisciplinary fields (1). So, nanotechnology is occupying most of the necessary applications of science and technology, namely electronics, aerospace, and medical fields. These include different design, method, characterization, and interaction processes in nanometer range (2). The silica nanostructures are synthesized using different techniques involving CVD (chemical vapor deposition), plasma synthesis, combustion method, hydrothermal techniques and sol- gel synthesis (3). Sol-Gel process could be used to fabricate various photonic materials in different forms such as coatings, optical fibers, and thin films for optical applications (4).

The sol-gel technique is used to produce silica glass via silicon alkoxide in a solution which undergoes hydrolysis and condensation polymerization at room temperature to obtain the gel. The important steps (drying and heat treatment) will rapidly densify the gel by removing water and solvents (5). Laser technology is used as a technique to densify the gel layer which represents an attractive way for processing such materials (6). Sufficient heating and coating layers for good densification result from the laser densification technique regarding no warping or melting of the substrate. Densification of Sol-Gel coating in a furnace differs from lasers in many aspects.

Heating with laser makes organic burnout take place in short time (fractions of a second) because the effective energy of the focused laser is light. On the other hand, when heating is done with an oven, the organic materials are burning out for several minutes (7). Laser densification is used to densify printed sol-gel coatings and improve its surface properties (8). In addition, it could be used in wide applications in different fields (impact on the silicalite optical characteristics, fuel cells, and glass ceramic tapes) (9-11). $CO_2$ laser and Nd:YAG laser are used for the densification process (7, 12). In this paper, our purpose is to investigate and compare the structure of prepared $SiO_2$ sol-gel thin films densified by two different lasers and study the effect of different laser wavelengths on the structure of prepared $SiO_2$ after densification process.

## Materials and Methods:

### Experimental:

### 1. Samples Preparation

The $SiO_2$ samples are synthesized by mixing (2 ml) of Tetraethoxysilicate (TEOS) as a precursor material (Sigma Aldrich, purity 99.9 %), (6 ml) of Ethanol as a solvent (purity 99.9 %), drops of HCL as a catalyst, and (4 ml) of deionized water. The mixture is stirred at the magnetic stirrer for 2 hours. The sample is left for aging about 3 days. Dip Coating Technique is used to obtain $SiO_2$ thin films. The dipping processes are repeated twice to get a thin layer of $SiO_2$ films, the films are preheated on a hot plate at 100˚C for an hour. The thickness of $SiO_2$ films is in the range (232 nm to

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260 nm) and is measured using thin film measurement system model Epp 2000.

## 2. Densification Process

The densification process is done using two different lasers. The first laser used is 532 nm DPSS laser (1.9 W), while the second laser used is 410 nm Diode laser (100 mW). The exposure time of laser irradiation is 15 minutes. The experimental setup used for the two lasers consists of: laser source, a mirror, and beam expander to extend the laser beam to whole the sample, as shown in Fig.1.

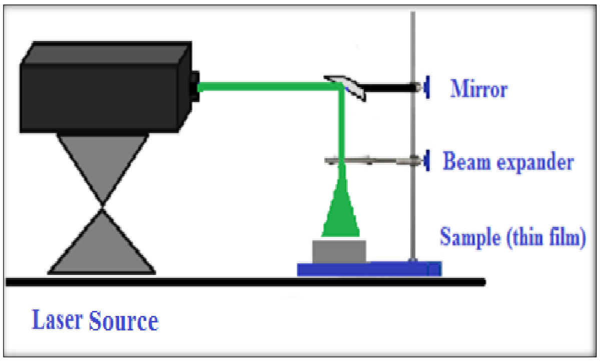



**Figure 1. The Experimental Setup Used for Densification Process.**

## Results and Discussion:

### 1.X-Ray Diffraction

X-Ray diffraction patterns of $SiO_2$ nanostructure thin films prepared by Sol-Gel synthesis method is characterized by X-Ray Diffractometer (XRD). Fig.2 shows that the crystobalite structure appears clearly for the diode laser (410 nm) at the position (2θ= 21.7°) which is in good agreement with J. R. Martinez et al. (13), whereas other references observe the amorphous structure at different diffraction angles (23°, 16° to 30°) (14-17) which is approximately obtained from diode laser (532 nm) at the position (2θ=24.2°).

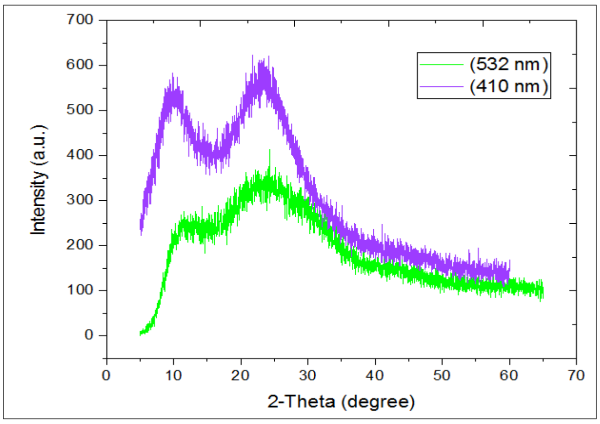



**Figure 2. XRD Patterns of $SiO_2$ Samples Densified by : DPSS Laser (532 nm), and Diode Laser (410 nm).**

### 2. Fourier Transformation Infrared Spectrometer (FTIR)

The FTIR spectra for $SiO_2$ samples after densification process with two different Diode lasers are shown in Fig.3 which show three bands of Si–O–Si bond vibration. The bands of H–O–H bending vibration of $H_2O$ and O–H vibrations are from different species. Si–OH band vibration appears clearly. The appearance of Si-Si bond is due to oxygen vacancies. Symmetric and asymmetric fundamental stretching vibrations of $CH_2$ and $CH_3$ groups belonging to alkoxide and solvent residues appear for diode laser (532 nm) only due to its amorphous structure. The wavenumbers and FTIR bonds for $SiO_2$ samples are summarized in Table 1. These results are in good agreement with (18-22). FTIR results indicate that Silicon Dioxide has been successfully synthesized.

**Table 1. FTIR Bonds and Wavenumbers for $SiO_2$ Samples.**

| Bond Type | Wavenumber (1/cm) for DPSS Laser (532 nm) | Wavenumber (1/cm) for Diode Laser (410 nm) |
|---|---|---|
| Si–O–Si | **487** | **487** |
| Si–O–Si | **833** | **815** |
| Si–O–Si | **1024** | **1024** |
| Si-Si | **605** | **605** |
| Si–OH | **942** | **942** |
| H–O–H | **1334** | **1334** |
| $CH_2$ and $CH_3$ groups | **2860** | **—** |

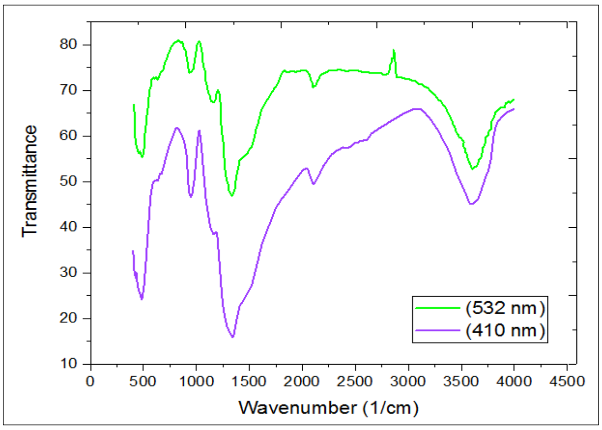
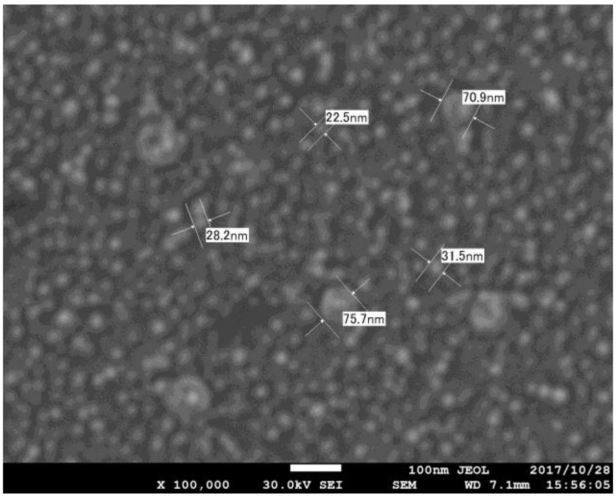



**Figure 3. FTIR Spectra of $SiO_2$ Samples Densified by : DPSS Laser (532 nm), and Diode Laser (410 nm).**

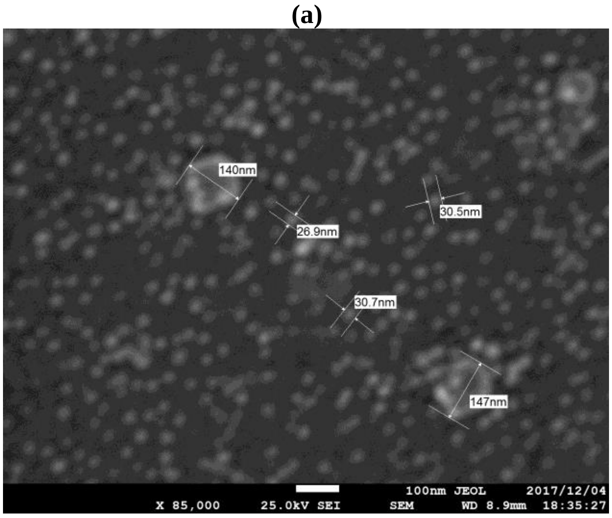
### 3. Field Emission Scanning Electron Microscopy (FESEM)

Field Emission Scanning Electron Microscopy is used to study the morphology of the prepared silica samples. FESEM can characterize the shape and size of $SiO_2$ nanostructure thin films.

FESEM images of $SiO_2$ thin films densified by the two Diode Lasers show that $SiO_2$ nanoparticles have roughly a spherical shape with small size nanoparticles around the range of less than 100 nm, Fig. 4.




**(a)**




**(b)**

**Figure 4. FESEM Images of $SiO_2$ Samples Densified by : a- DPSS Laser (532 nm), b- Diode Laser (410 nm)**

## Conclusions:

Spherical silica nanocrystals are successfully densified by diode laser (410 nm) rather than diode laser (532 nm). Cristobalite structure is clearly observed. FTIR results prove a successful preparation of $SiO_2$ nanoparticles. FESEM images show that the $SiO_2$ particles formed are in the nano range ($\leq$ 100 nm). It is concluded that the laser wavelength plays an important role in the laser densification process. $SiO_2$ could be used in chemical sensing.

## Conflicts of Interest: None.

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# التكثيف بالليزر لأغشية ثنائي اوكسيد السيليكون الرقيقة المحضرة بطريقة (Sol-Gel)

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**الخلاصة:**

تم تحضير مركب ثنائي اوكسيد السيليكون النانوي بطريقة (sol-gel) وتم تحضير اغشية رقيقة منه بتقانة الطلاء الإنغماسي. دُرس تأثير التكثيف بالليزر. تم فحص العينات المُحضّرة وتحليلها بالطرق الاتية:- الحيود بأشعة X ، مطياف تحويلات فورير بالمنطقة تحت الحمراء، والميكروسكوب الالكتروني الماسح. اظهرت النتائج بإن مركبات السيليكا النانوية كانت مُحضّرة بنجاح بطريقة (sol-gel) بعد عملية التكثيف بالليزر. بينت فحوصات XRD بإن تركيب الكرستوبالايت ظهر عند استخدام ليزر الدايود (410 nm) فضلاً عن ليزر الدايود (532 nm). الصور المأخوذة بالمايكروسكوب الالكتروني الماسح اظهرت بإن شكل المركب النانوي كان كرويا وحجومه ($\le$ 100 نانومتر). تم الاستنتاج بإن التركيب البلوري النانوي الكروي لاغشية السيليكا الرقيقة كُثفت بنجاح بواسطة ليزر الدايود (410nm).

**الكلمات المفتاحية :** أغشية ثنائي اوكسيد السيليكون الرقيقة، سول – جل، التكثيف، حيود الأشعة السينية، المجهر الألكتروني الماسح.