



Investigation of Densified SiO₂ Sol-Gel Thin Films Using Conventional and DPSS Laser Techniques

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Abstract: The prepared nanostructure SiO₂ thin films were densified by two techniques (conventional and Diode Pumped Solid State Laser (DPSS) (532 nm). X-ray diffraction (XRD), Field Emission Scanning electron microscopy (FESEM), and Atomic Force Microscope (AFM) technique were used to analyze the samples. XRD results showed that the structure of SiO₂ thin films was amorphous for both Oven and Laser densification. FESEM and AFM images revealed that the shape of nano silica is spherical and the particle size is in nano range. The small particle size of SiO₂ thin film densified by DPSS Laser was (26 nm), while the smallest particle size of SiO₂ thin film densified by Oven was (111 nm).

Keywords: SiO₂ thin films, sol-gel, Densification, FESEM.

Introduction

Nanotechnology has rapidly occupied all necessary fields of science and technology such as electronic, aerospace, defense, medical, and dental. This involves the design, synthesis, characterization, and application of material and devices on the nanometer scale [1]. One of the most common minerals on earth and a basic component of soil, sand, and rocks is Silica nanoparticles (SiO₂ NPs) which exists in both amorphous and crystalline forms. SiO₂ NPs preparation is simple and of low cost [2]. It has specific physical, optical, and chemical properties that have led to its broad use in

different fields. They are used as semiconductive material, catalyst, rubbers paints, plastics, biomedicine, and effective materials [3].

The conventional methods cannot produce a high-quality SiO₂ nanoparticles but Sol-Gel method is one of the most substantial methods for preparing high-quality SiO₂ nanostructure [4]. It is a versatile method with its high process rapidity, suitability for continuous production and collection of chemical precursors. The most important advantage of sol-gel processing is the possibility of precise control on the microstructure of the deposited film, such as the pore volume, pore size and surface area, as

compared to conventional coating methods [5]. Sol-gel method has been widely used as a method for the preparation of nanoparticles. It has several advantages such as synthesis may be carried out at low temperature, desired pH to yield high purity and, also, the reaction kinetics of the process may be controlled by varying the composition of the reaction mixture [6]. Laser technology was used as a technique to densify the gel layer which represents an attractive way for processing such materials [7]. Sufficient heating and coating layers for good densification resulted from laser densification technique regarding no warping or melting 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 light. On the other hand, when heating is done with an oven the organic materials are burning out for several minutes [8]. In this paper, our aim is to investigate and compare the prepared SiO₂ nanostructure thin films conventionally densified and by Diode Pumped Solid State (DPSS) Laser.

Experimental Part

1. Densification Process

The prepared SiO₂ thin films were densified by two techniques conventional and DPSS Laser (532 nm). Some of the thin films were densified by oven at 200 °C for 2 hours, and others by CW DPSS Laser (532 nm, 1.9 W) with an exposure time of 15 minutes. The experimental setup consisted of: laser source, a mirror, and

beam expander to extend the laser beam to whole the sample, as shown in Fig.(1).

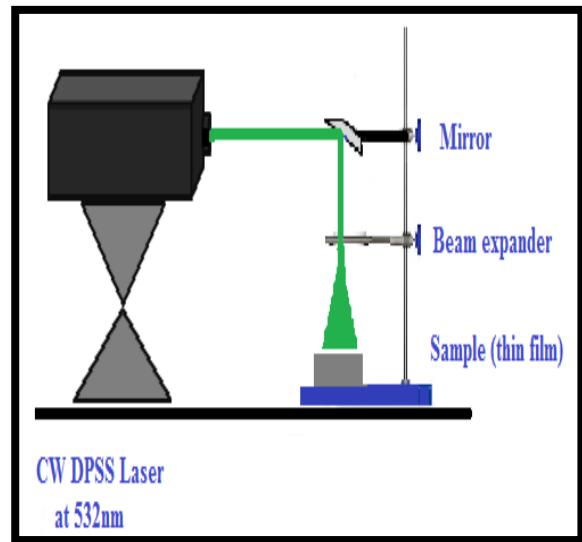


Fig. (1): The Experimental Setup.

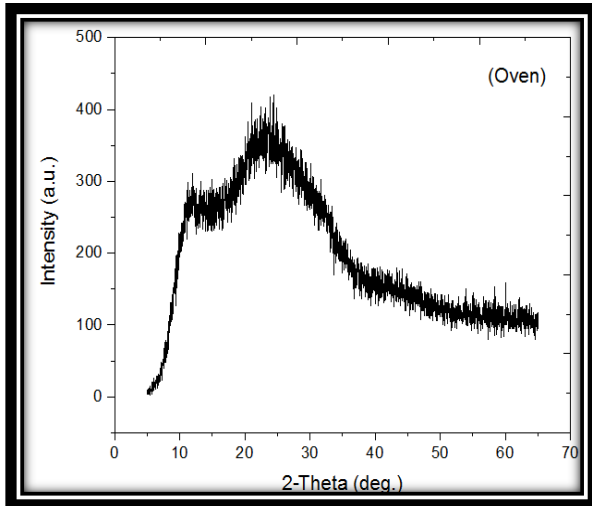
2. Characterization Measurements

Structure and morphological properties of SiO₂ nanostructure thin films were investigated by X-Ray Diffraction (XRD), Atomic Force Microscope (AFM) and Field Emission Scanning Electron Microscopy (FESEM).

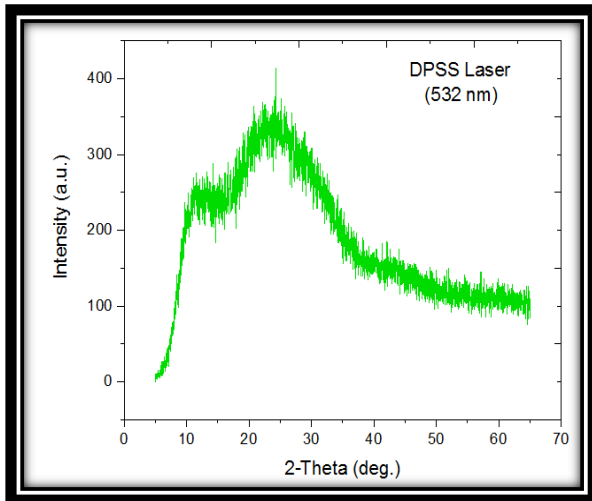
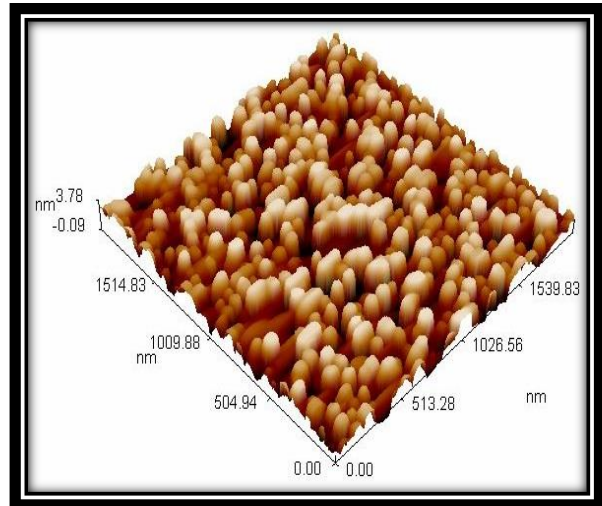
Results and Discussion

1. X-Ray Diffraction

The X-ray diffraction patterns of densified SiO₂ thin films showed that the pattern of SiO₂ sample densified by oven gave a broad band at the position $2\theta=24.5^\circ$ at the left side of the pattern. When the DPSS laser was used for the densification process, the band had a little shift to the lower value of the diffraction angle ($2\theta=24.2^\circ$) and the intensity became higher. These results indicate that the structure of SiO₂ sample is amorphous in nature and the particles formed are tiny nanocrystal as shown in Fig.2, which are in good agreement with [9-11].



(a)



(b)

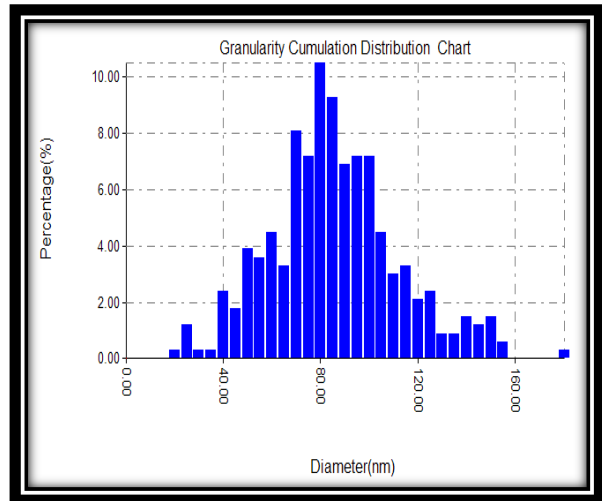
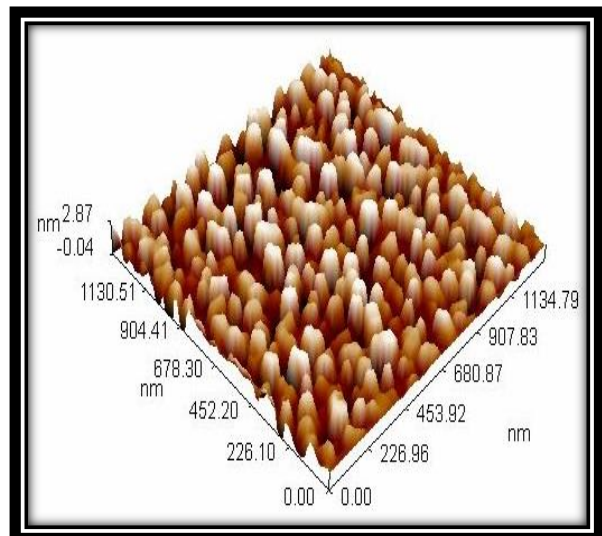


Fig. (2) : XRD Patterns of SiO₂ Thin Films Densified by : a- Oven b- DPSS Laser.

Fig.(3): AFM images for SiO₂ nanostructure thin film densified by Oven, a three-dimensional image, and size distribution histogram. The particle size 50±100 nm is dominated.

2. Atomic Force Microscope (AFM)

The surface morphology of the prepared SiO₂ nanostructure thin films densified by oven and DPSS laser samples was investigated using Atomic Force Microscopy (AFM) as shown in Fig.(3, 4). A granular microstructure and a flat texture, with the lowest surface roughness for SiO₂ sample densified by Laser, while at oven densification, the grains get larger and combine to make denser coatings, but the basic structure remains unchanged.



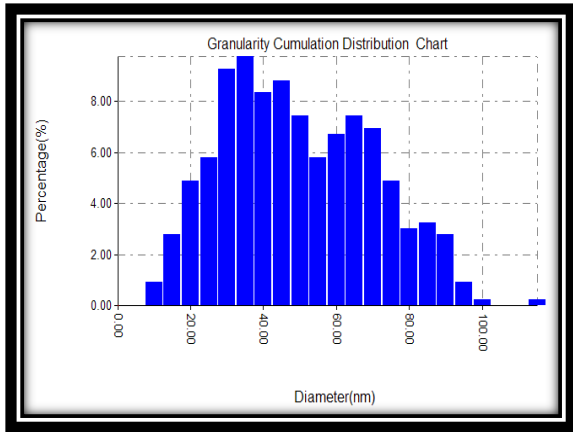
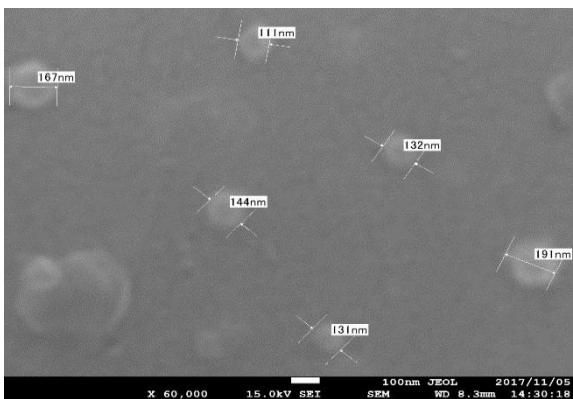


Fig.(4): AFM images for SiO₂ nanostructure thin film densified by DPSS Laser, a three-dimensional image, and size distribution histogram. The particle size 20±85 nm is dominated.

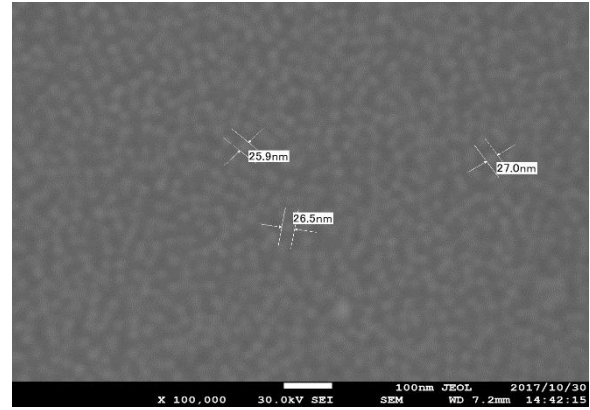
3. Field Emission Scanning Electron

Microscopy (FESEM)

The densified SiO₂ thin films were characterized by Field Emission Scanning Electron Microscopy (FESEM). FESEM images of SiO₂ thin films densified by oven and laser showed that SiO₂ nanoparticles had roughly spherical shape with small size nanoparticles around the range of less than 100 nm, Fig.5 (a, b). FESEM images showed that the smallest particle size of SiO₂ thin film densified by Laser was (25.9 nm), while the smallest particle size of SiO₂ thin film densified by oven was (111 nm). This gives an indication that the laser densification produces nanoparticles smaller than oven densification.



(a)



(b)

Fig. (5) : SEM images of SiO₂ samples densified by : a- an oven, b- DPSS Laser.

Conclusions

Spherical and amorphous densified silica nanoparticles thin films could be observed for both conventional and DPSS laser densification. The amorphous form of SiO₂ nanoparticles appears from XRD and shows that the silica particles are formed by small nanocrystals. The small particle size of SiO₂ thin film obtained by DPSS Laser is (26 nm) , while the smallest particle size of SiO₂ thin film densified by oven is (111 nm) which gives an indication that the laser densification yields nanoparticles smaller than oven densification.

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التحقق من تصليب غشاء رقيق لمادة SiO₂ sol-gel باستخدام التقنيات الاعتيادية وتقنية ليزر الحالة الصلبة المضخ بليزر الثنائي DPSS Laser

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الخلاصة : الاغشية الرقيقة المحضرة من ثنائي اوكسيد السيليكون ذو البنية النانوية كُنُفَت بتقنية التكتيف الاعتيادي والتكتيف بليزر الدايدود (532 nm). فحصت العينات المُحضرة بالطرق الاتية:- الحيود بأشعة X , ومجهر القوة الذرية , والميكروسكوب الالكتروني الماسح. اظهرت نتائج الحيود بأشعة X بان تركيب ثنائي اوكسيد السيليكون كان غير متبلور. الصور المأخوذة بمجهر القوة الذرية و بالمايكروسكوب الالكتروني الماسح اظهرت بان شكل المركب النانوي كان كرويا وحجمه (≥ 100 نانومتر). اصغر جسيمة لثنائي اوكسيد السيليكون المكثف بواسطة الليزر هو (26 nm) بينما اصغر جسيمة لثنائي اوكسيد السيليكون المكثف بواسطة الفرن هو (111 nm).