Production of Micro and Nano Zirconia Particle by Pulsed Laser Ablation

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ABSTRACT
In this work, zerconia nano particles have been synthesis by using laser ablation in water. AFM measurement displayed that the particle size of Zerconia was 46nm and no other phases have been noticed. The root mean square of surface roughness becomes 0.743nm after laser ablation, while before that was 2.33nm. Granularity distribution chart observed that the particle size distribution of Zerconia before laser ablation was (100-400) nm, while after that become (20-80) nm .The microstructure investigation of zerconia nano particle was carried out by using SEM.

Keywords: zerconia, nano powder, micro powder ,SEM,AFM

انتاج مسحوق نانوي بطريقة استعمال الليزر

الخلاصة
في هذا العمل تم استعمال الاشعة الليزرية لانتاج المسحوق النانوي والمايكروي لمادة الزركونيا . ان فحص الاشعة السينية اكد بان المسحوق النانوي المنتج هولمادة الزركونيا .اوضحت فحوحات AFM بان الحجم الحبيبي للزركونياهو 46 ولايوجد اي مواد اخرى. متوسط القيمة الفعالة اصبت nm 0.743 بعد استعمال الاشعة الليزرية بينما كانت 2.33 nm قبل الاستعمال. مخطط التوزيع الحبيبي للزركونيا كان nm (100 - 400) قبل استعمال الاشعة الليزرية بينما بعد الاستعمال اصبح nm (20-80) . تم فحص الجيسيمات النانوية باستعمال المجهر الالكتروني الماسح.

INTRODUCTION
Laser ablation is the process of removing material from the surface of the solid by irradiating it with a laser beam. At high laser flux, the material is typically converted to a plasma. At low laser flux, the material is heated by the absorbed laser energy and evaporates or sublimates. Usually, laser ablation refers to removing material with a pulsed laser, but it is possible to ablate material with a continuous wave laser beam if the laser intensity is high enough. The depth over which the laser energy is absorbed, and thus the amount of material removed by a single laser pulse, depends on the material's optical properties and the laser wavelength and pulse length. The total mass ablated from the target per laser pulse is usually referred to as ablation rate. Laser pulses can vary over a very wide range of duration (milliseconds to femtoseconds) and fluxes, and can be precisely controlled. This makes laser ablation very valuable for both research and industrial applications.

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In Nanomaterials which is with nanoscale dimension the surface or interface properties dominate over the bulk properties. The very large surface area of these materials may result in novel chemical and physical properties, such as increased catalytic activity, improved solubility or different optical behavior [1, 2]. Laser ablation of tetragonal zircon, a very hard Ceramic widely used in medical technology is the key to highly interesting new applications.

On the one hand, short-pulsed laser structuring is the only wear-free method to shape fully sintered substrates, as in the case of produce dental crowns. (Also) On the other hand, particles that are laser ablated from the surface of zirconia substrates can be embedded into other products to increase their surface strength. This laser-based nanotechnology approach provides the best results if ablated particles have specific characteristics such as good dispersion and small size and if a long-term fixation between such particles and the product is achieved. The investigations show how zirconia ablation by femtosecond laser pulses affects micromachining quality and efficiency on the one hand and the amount and characteristics of generated nanoparticles on the other hand, and how ablation through liquid films can combine both aims[3,4].

The investigations show a significant influence of the liquid on ablation rates, quality of micromachining, and size and amount of particulate material. Remarkably, the liquid layer does not only allow collecting nanoparticulate matter and improving the quality of micromachining, but can also enhance the productivity[5].

Depending on the focus of interest (quality of the ablated surface, amount of ablated material, size and amount of nanoparticles dispersed in liquids), it turns out to be recommendable to cover the ceramic surface with different amounts of liquids during the laser ablation process. In this study the pulsed laser ablation technique was used to produce colloidal zirconia nano particles and the main characteristics of the zirconia nano particles were investigated [6,7].

Experimental Work

High purity Zirconia powder with particle size of 195.90 nm is pressed with 25 Mpa by using hydraulic press to form pellet with diameter of 2-inch. The zirconia pellet were ablated by pulsed laser ablation using Q-switching Nd-YAG laser Delixi DZ47-63 (wavelengths1064 nm, pulse durations 7 ns, number of pulses 300). The laser fluence was maintained at 0.14J/cm ^{2} and focused by using 10Cm positive lens with an incident angle of 45^0 on the of the target and the distance between sample and laser source 10cm with time exposure equal 10 min . The ablation process was done at room temperature. And the target was fixed by Teflon piece inside the flask and immersed at 20 mm depth in the solution (distilled water).

The Zirconia nano particles were characterized by atomic force microscopy (AFM) is carry out on AA 300 Scanning probe microscope Angstrom Advanced Inc, to examine their crystalline and to observe the surface structure. The size and the shape of Zirconia particle were estimated by mean of scanning electron microscopy type VEGA// Easy probe . The X-Ray diffraction measurement were carried out on automatic X-R diffractometer (ADX 2500) operating with a Cu-Ka radiation at voltage of 30 KV and a current of 20 mA with a Ni-filter and a ascan rate of 1.28 degree/min.

Results and Discussion

The laser ablation of the Zirconia was performed in water at low laser fluence of ($0.14J/cm^2$) the color of the solution was changed after laser solution. The results of AFM test (CPSM) image Surface Roughness Analysis is listed in table(1).These results indicated that the particles of Zirconia within the range of nano particle.

Fig. (1) Shows the SEM images of Zirconia NPs, both nano particles and submicron particles present with both spherical and irregular shapes.

Scanning electron microscopy images of fracture surfaces of Zirconia particles are presented in Figs(1-4). All the samples have dense, reasonably uniform, fine-grained microstructure with an average grain size of around 80 nm. The appearance of the surface suggests intergranular fracture. The laser fluence may be played an important role in ablated particle's shapes, size and structure.

Figures (1-4) show SEM images of fracture surfaces of Zirconia with different magnifications. The particle size that estimated from AFM investigation agrees well with that obtained from SEM.

Figure(5) represent the 2D- AFM image of zirconia particles before laser ablation, while 3D -AFM image of zerconia particles before laser ablation display in figure (6).

Figure (7) show a granularity distribution chart of Zirconia before ablation by laser, while figure (8) shows a granularity distribution chart of Zirconia after ablation by laser. It was normalized to represent. The figures indicated that the the particle size of ablated Zirconia ranges before laser ablation from (100 – 400) nm, But The average particles size ranging become (20-80) nm after ablation depending on the laser fluence as shown in figures (7)and (8).The ablated Zirconia particles are aligned vertically ,on the other hand ,the observed particles have non spherical shape. Agglomeration of some particles is observed. The granularity distribution chart of Zirconia particles was nearly Gaussian. Figure (9) represent the 2D- AFM image of zirconia particles after laser ablation, while **3D** -AFM image of zirconia particles after laser ablation display in figure (10).Figure (11) shows the particle size test for zirconia after laser ablation, while Figure (12) displays the particle size test for zirconia before laser ablation. The X-Ray diffraction pattern of the powder sample is shown in figure (13). All reflections show the presence of two polymorphic forms ZrO_2 ICSD 18190(01-072-1669) & ICDS 15983(01-072-0597) which are (monoclinic & tetragonal).The major reflection of the patterns for tetragonal phase which is the metastable phase . The highest intensity of the reflection with 2 ɵ = 28.30 was for monoclinic Zirconia phase, while 2 ɵ = 30.57 is the diffraction angle of tetragonal Zirconia.

Table (1) AFM test (CPSM) image Surface Roughness Analysis.

No.	Results	Before laser ablation(nm)	After laser ablation (nm)
1	Sa(Roughness average)	1.21	0.431
2	Sq(Root mean square)	2.33	0.743
3	S sk(Surface skew ness)	2.97	2.03
4	Sku(surface Kurtosis)	21.7	12.3
5	Sy(Peak-Peak)	30.7	8.09
6	Sz(Ten point Height)	30.7	7.87
7	Particle size	100-400	20-80
8		36.92	14.22

CONCLUSIONS

1- Colloidal Zirconia NPs of various sizes can be synthesized by using a simple method of nanosecond pulsed laser ablation with wavelength of 1064 nm.
2- The control on the size of particles and size by using low laser fluence $0.14J/cm^2$which offered the smallest dominant particle size.
3- The root mean square of surface roughness becomes 0,743nm after laser ablation, while after that was 2.33 nm.
4- the particle size distribution of Zirconia before laser ablation was (100-400) nm, while after that become (20-80) nm
5- The ablated Zirconia particles are aligned vertically and the observed particles have non spherical shape.

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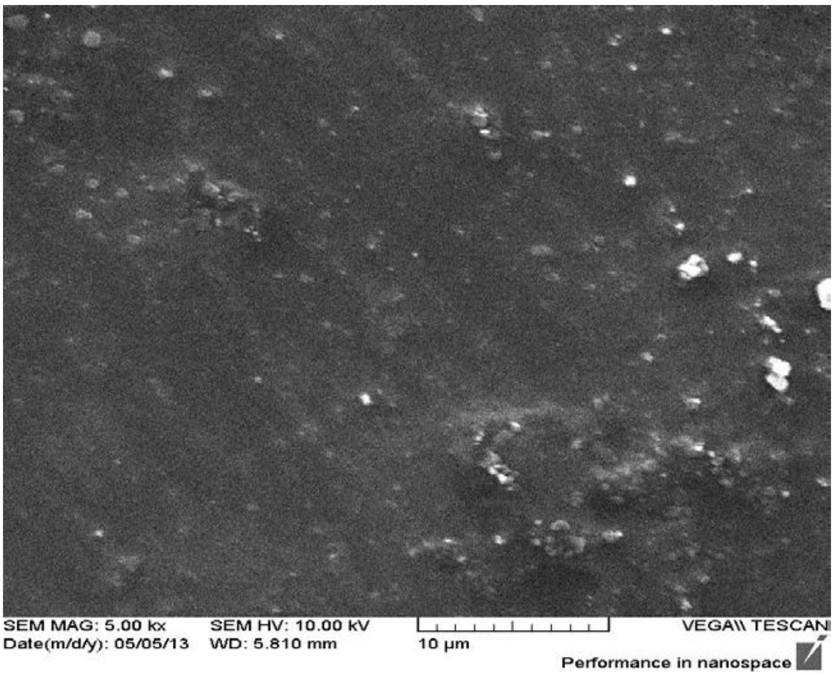

Figure (1) SEM test for zerconia with magnification of 5.00 kx

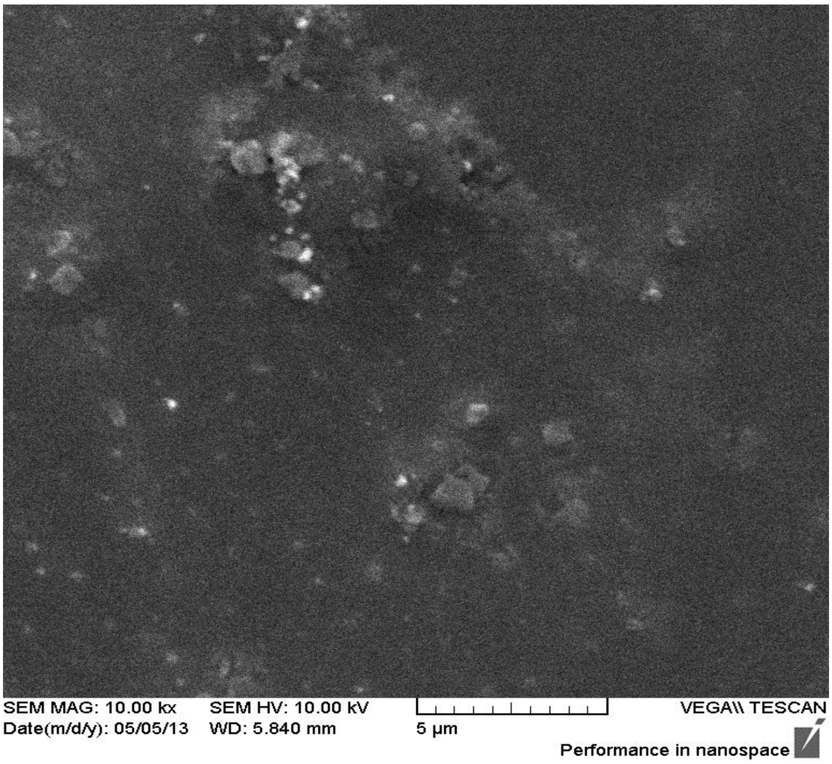

Figure (2) SEM test for zerconia with magnification of 10.00 kx

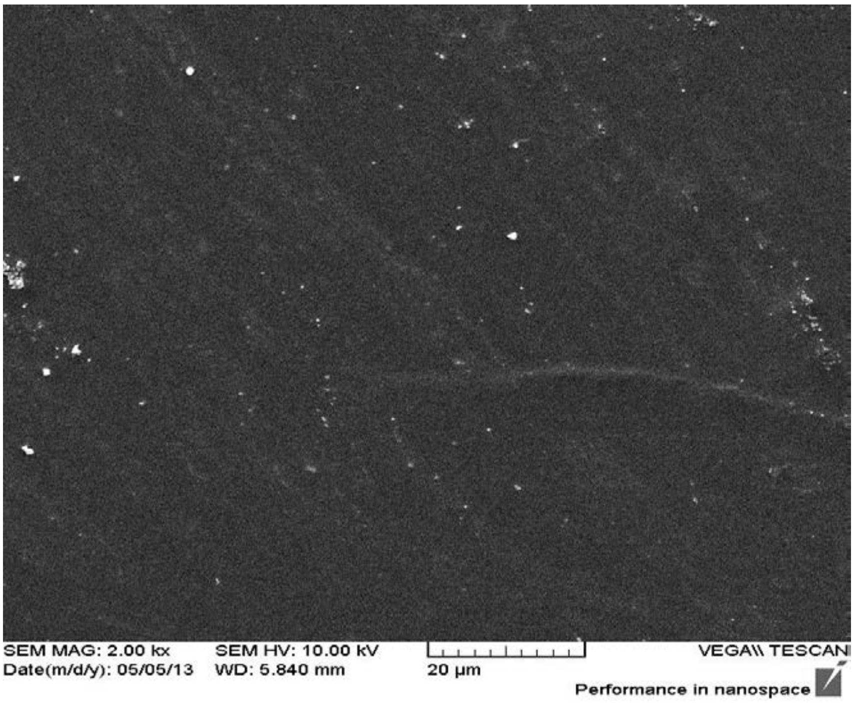

Figure (3) SEM test for zerconia with magnification of 2.00 kx.

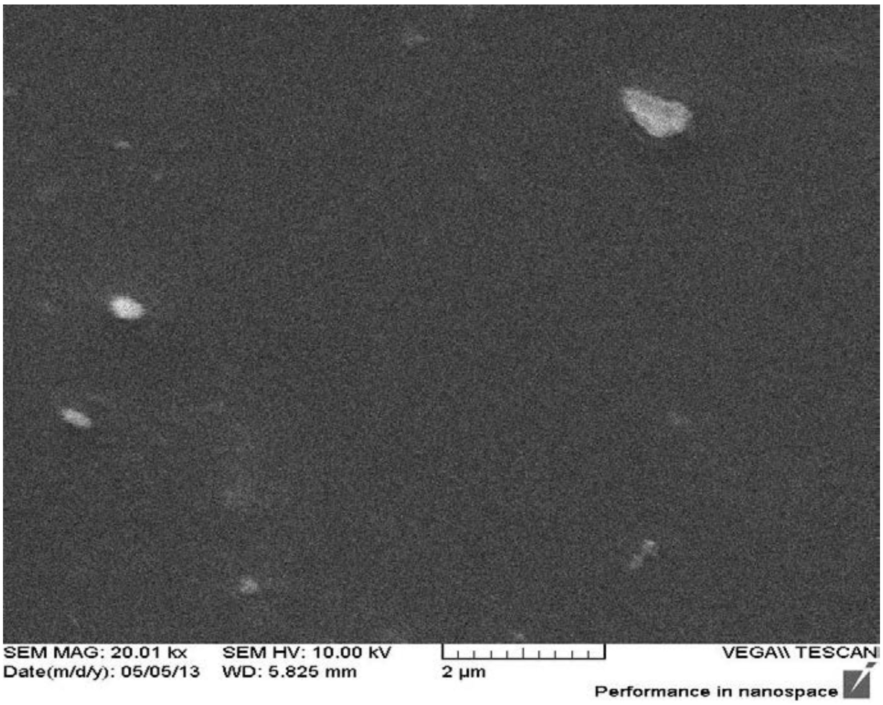

Figure (4)SEM test for zerconia with magnification of 20.00 kx

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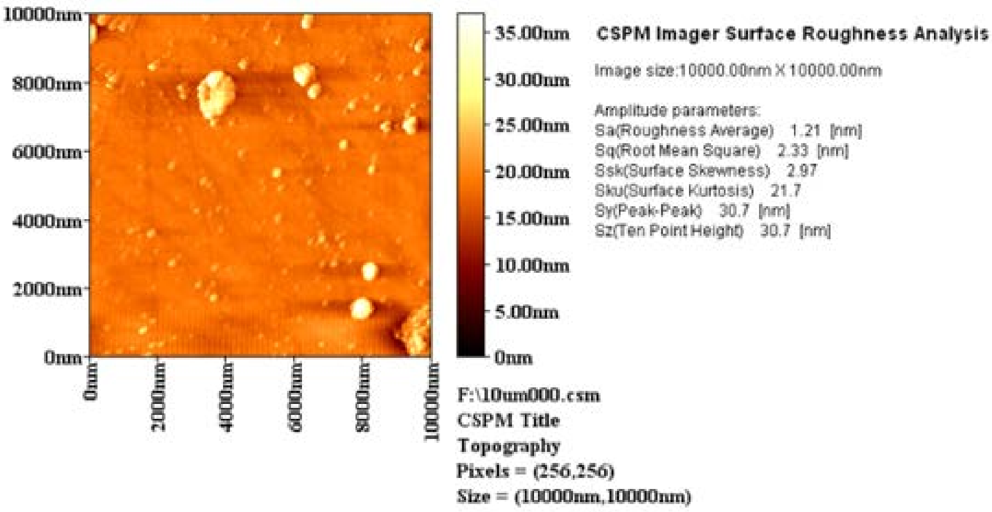

Figure (5) 2D- AFM image of zerconia particles before laser ablation.

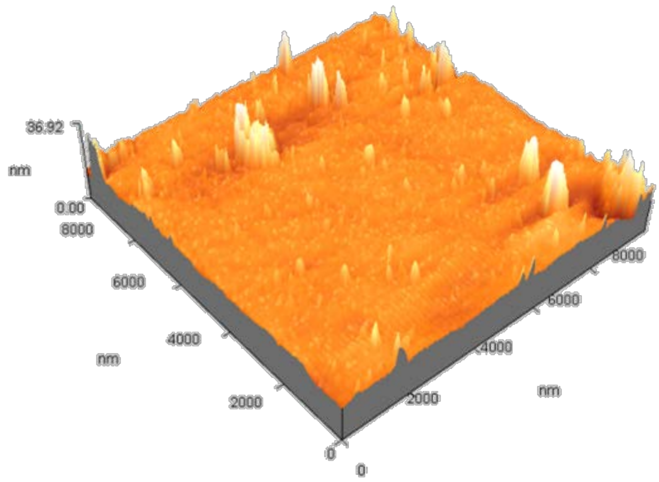

Figure (6) 3D -AFM image of zerconia particles before laser ablation.

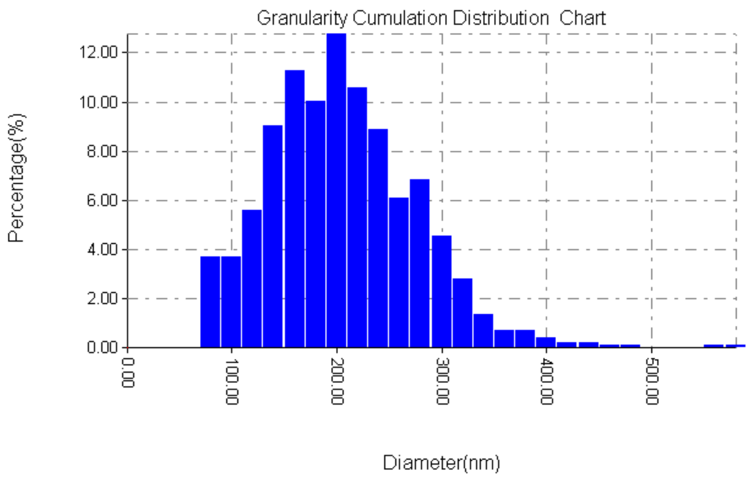

Figure (7) particle size test for zerconia before laser ablation

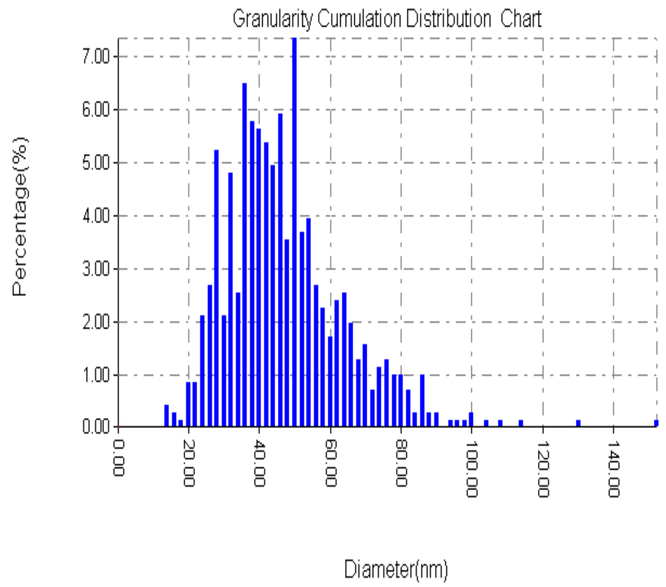

Figure (8) particle size test for zerconia after laser ablation

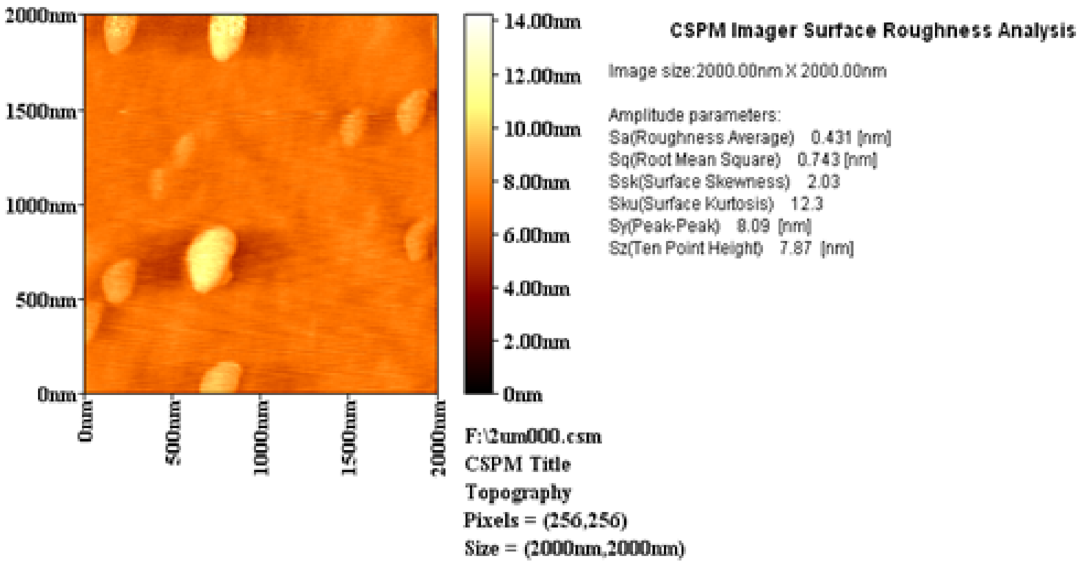

Figure (9) 2D-AFM image of zerconia particles after ablation

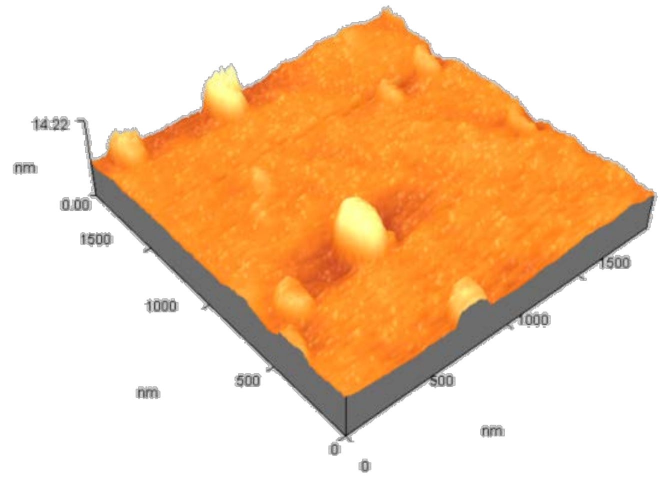

Figure (10) 3D-AFM image of zerconia particles after laser ablation

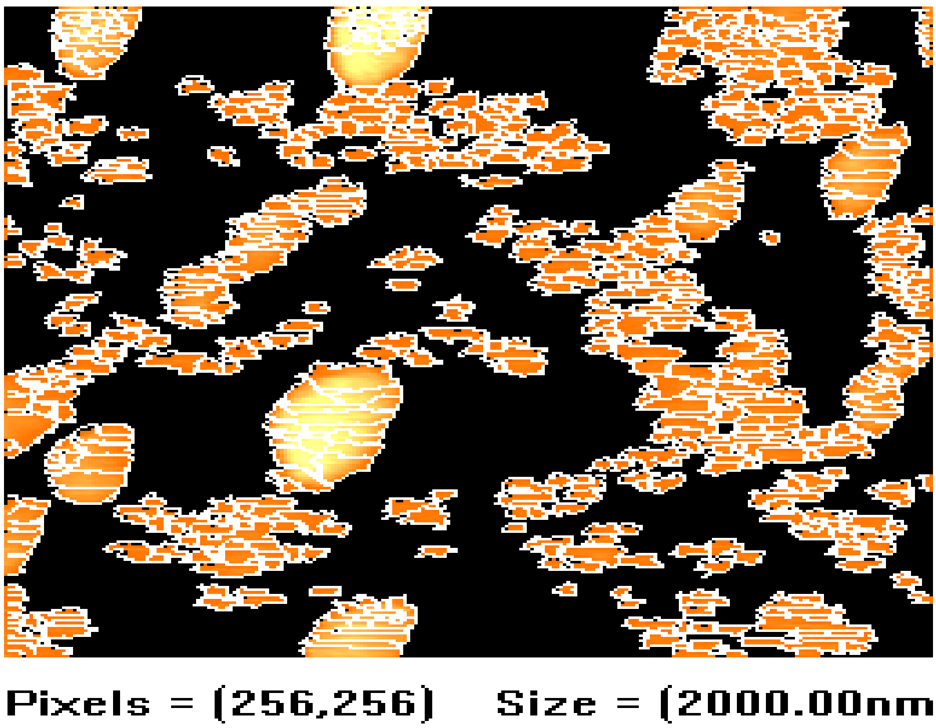

Figure (11) particle size test for zerconia after laser ablation.

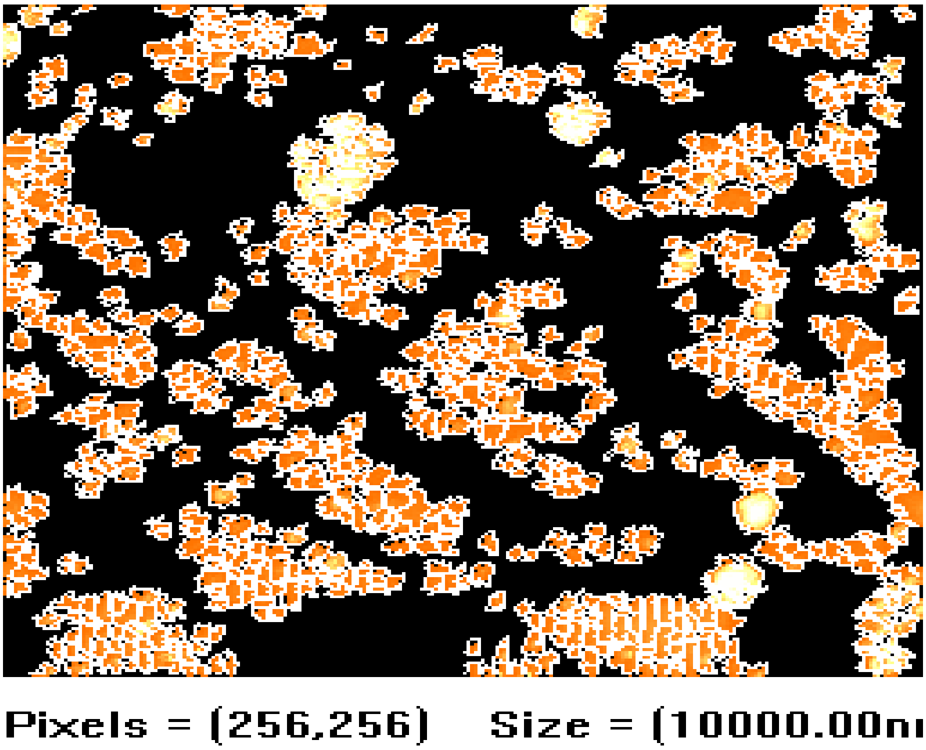

Figure (12) particle size test for zerconia before laser ablation

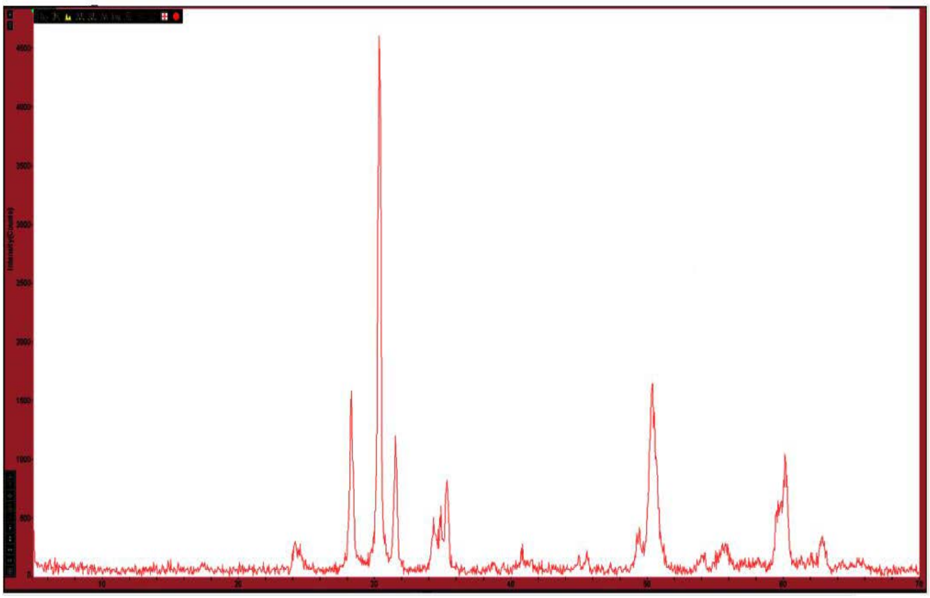

Figure (13) The X-ray Diffraction pattern of the sample