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Effect of Laser Induced Tin Oxide (SnO_2) Nano particle

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Abstract-*The study of Nano particles and its influence with laser plays a major role in material characterisation. Particularly, metal oxide semiconducting materials are low cost and have significant properties in laser materials processing. Now in this study, we report the synthesis and influence of laser treated Nano powder SnO_2. The laser treated SnO_2 samples were characterized by X-ray diffraction, UV-Visible absorption, FTIR spectra, Dielectric studies, DLS spectra and SEM. From the X-ray analysis, we observed that the laser treated $SnO_2$is with the structure of tetragonal rutile crystalline and the crystalline size is in the range of 4-6nm. The UV-Vis spectrum confirms the presence of nanoparticles in the range 191-193 nm. The optical band gap values of SnO_2 nanoparticles were calculated about 3.5-3.8eV. Fourier Transform Infrared spectroscopy reveales, the presence of tin oxide (SnO_2).The dielectric studies of laser treated samples of SnO_2 show appreciable results compared to samples without laser irradiation. Dynamic light scattering (DLS) studies revealed that the particle size distribution of Tin oxide SnO_2 nano-particles size in the range 8.8 nm - 62.5 nm. The SEM analysis of Tin oxide (SnO_2) nanoparticle also shows that the structure is tetragonal rutile in shape. The particle size varies from 20 nm – 161.2 nm.*

Key Words: **Structural, Optical and Dielectric properties of SnO_2 nanoparticles Laser irradiation.**

1. INTRODUCTION

Metal oxide semiconductor SnO_2 attracts greater attention because of high conductivity, transparency and sensitivity. In recent years, influence of laser on semiconductors finds a newer dimension in material science. Particularly, selective laser melting is a powder based additive manufacturing capable of producing industrial applications. Currently there is a greater interest in industry for generating objects with high geometrical complexity [1]SnO_2 particles, yield wide band gap and is mainly due to its chemical, electronic and optical properties[2] and also due its nano structured form[4].This material has a wide range of applications from gas sensors, liquid crystals, photovoltaic cells and conducting electrodes. In general, the band gap energy of semiconducting Nano crystals is mainly dependent on particle size. With the use lasers on metal oxides give further enhancement in band gap energy and this can be achieved with the use of suitable laser processing parameters such as laser power, scan speed .,etc. This paper investigates, the effect of with and without laser irradiation on SnO_2nano particles with different time exposures. The structural analysis of laser treated samples of SnO_2particles were studied using x-ray diffraction as well as spectroscopic investigation such as UV and FTIR were also studied. Dielectric properties of the samples for various frequencies are also investigated. DLS spectra and SEM the particle size investigated.

2. EXPERIMENTAL PROCEDURE

Commercially available SnO_2 powder (Sigama-Aldrich,99.9% purity) is used for investigation (composition is given in Table-1).Laser treatments are carried out by using 5.0mw low power He-Ne gas laser with red light of (wavelength 633nm) and diode laser with green light of 5mw power (wave length 532nm).The structural analysis of with and without laser irradiated samples are analysed using PANalytical's X-ray diffractometers(copper Kα radiation of wave length λ as 1.54060 Å and 1.54443Å).This system recorded the intensity as a function of Bragg's angle. While the average crystalline size of the particles can be estimated using full width at half maximum (FWHM) value of the x-ray diffraction peaks. The optical and spectroscopic investigation of the samples such as UV and FTIR studies are performed using Perkin Elmer UV/VIS spectrophotometer (λ 365) and Perkin Elmer FT-IR spectrometer. The Dielectric studies are performed using Digital LCRZ Meter TH2816A (50Hz - 200KHz) with various frequencies. The DLS spectra (Measuring the Particle Size Distribution) recorded by Particulate system Nano plus Zeta/nano particle analyzer instrument. SnO_2 nano particles are analysed using SEM (ZEISS SEM instrument) Micrographs. The laser processing parameters used for laser irradiation of samples are given in Table-2 and the properties of sample used is given in Table-3.

Table -1: chemical composition of SnO_2

Element	Content (in %)
Tin	78.76
Oxygen	21.21

Table -2: Laser processing parameters of samples

Sample Number	He-Ne laser irradiation	Diode green laser irradiation
1	Without laser	Without laser
2	15min	-
3	30min	-
4	-	15min
5	-	30min

Table- 3: Properties of SnO_2

Property	Characteristics
Solubility	Soluble in alkalis, acids and insoluble in alcohol
Magnetic susceptibility	-4.1×10^{-5} cm^3/mol
Density	6.95 g/cm^3
Molar mass	150.71 g/mol

3. RESULTS AND DISCUSSION

3.1 UV-Visible studies

The absorption of UV radiations on the nano powder SnO_2 sample with and without laser irradiation shows appreciable results. From the data the band gap energy for the laser treated sample varies from 3.6eV to 3.8eV and the sample without laser irradiation is 3.5eV.The result proves that the laser influences the nano particle SnO_2 towards band gap energy, particularly for large laser irradiation time for the both He-Ne and Diode lasers.Table-4 shows the band gap energy values for different laser irradiation with two different time exposures. Fig-1(a)- Fig-5(b) shows the UV-Visible spectrum of with and without laser irradiated samples.[2]

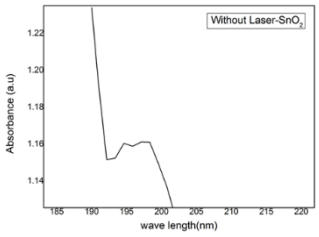

Fig-1(a) : UV-Vis Absorption spectra cutoff wavelength (sample 1)

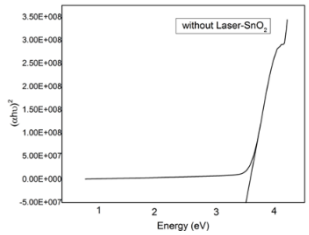

Fig-1(b) : UV-Vis Absorption spectra band gap energy (sample 1)

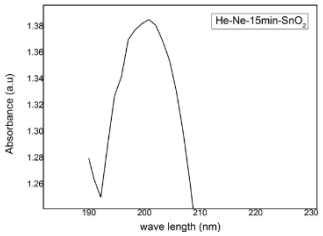

Fig-2(a) : UV-Vis Absorption spectra cutoff wavelength (sample 2)

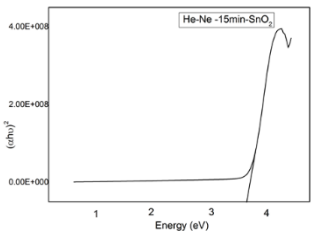

Fig-2(b): UV-Vis Absorption spectra band gap energy(sample 2)

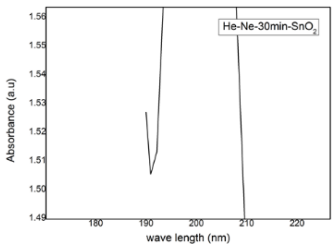

Fig-3(a) : UV-Vis Absorption spectra cutoff wavelength (sample 3)

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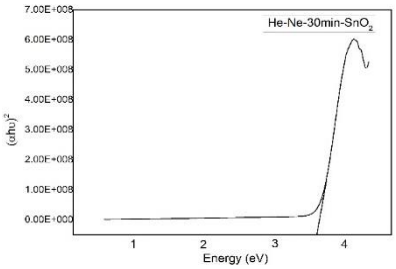

Fig-3(b) :UV-Vis Absorption Spectra band gap energy (sample 3)

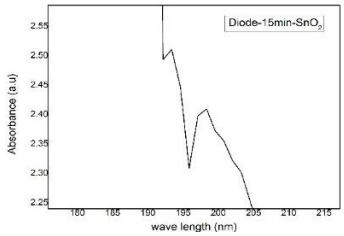

Fig-4(a) : UV-Vis Absorption spectra cutoff wavelength (sample 4)

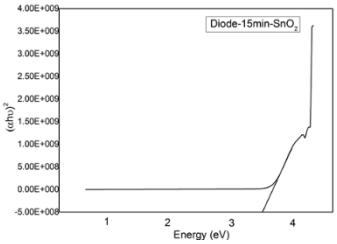

Fig-4(b) :UV-Vis Absorption spectra band gap energy (sample 4)

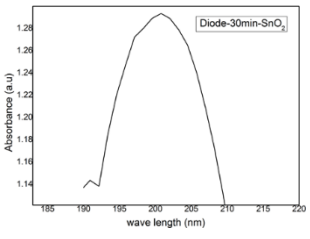

Fig-5(a) : UV-Vis Absorption spectra cutoff wavelength (sample 5)

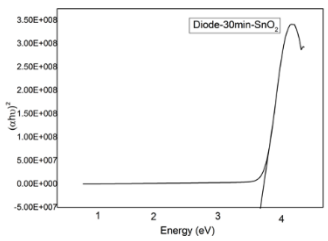

Fig-5(b): UV-Vis Absorption Spectra band gap energy (sample 5)

Table-4: Cut off wavelength and Band Gap Energy Values For SnO_2 Nanoparticles

Sample Number	Time duration of LASER Irradiation (min)	Cut Off Wavelength (nm)	Band gap energy (eV)
1	Without laser	192	3.5
2	with He-Ne laser at 15 min	191	3.6
3	with He-Ne laser at 30 min	191	3.8
4	with Green Diode laser at 15 min	192.5	3.6
5	with Green Diode laser at 30 min	193	3.8

3.2 FTIR spectral studies

The FTIR spectrum of with and without laser treated SnO_2 particles are given in Fig-6-Fig-10. [3]

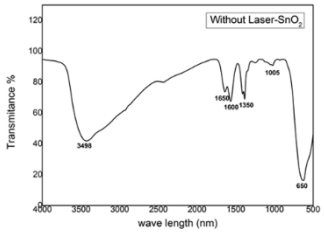

Fig-6: FTIR spectra (sample 1)

Table -5: FTIR functional analysis (Sample 1)

Wave	Group	Vibration	Intensity
3498	O-H	Stretching	Strong
1650	H-OH	Stretching	Medium
1350	Sn-OH	Stretching	Variable
1005	Sn-O	Stretching	Strong
650	Sn-O-Sn	Stretching	Strong

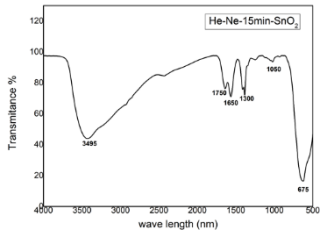

Fig-7: FTIR spectra (sample 2)

Table- 6: FTIR functional analysis (Sample 2)

Wave	Group	Vibration	Intensity
3495	O-H	Stretching	Strong
1750	C=O	stretching	Strong
1650	H-OH	stretching	Strong
1300	O-C	stretching	Medium-strong
1050	C-O	stretching	Strong
675	Sn-O-Sn	stretching	Strong

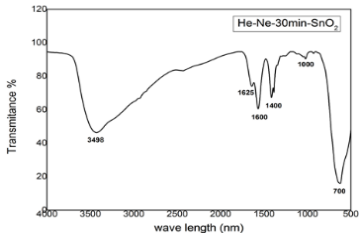

Fig-8: FTIR spectra (sample 3)

Table-7: FTIR functional analysis (Sample 3)

Wave	Group	Vibration	Intensity
3498	O-H	Stretching	Strong
1625	H-OH	stretching	Strong
1600	C=C	stretching	Medium-weak
1000	C-N	stretching	Medium
700	S-OR	stretching	Strong

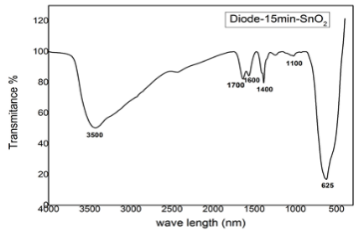

Fig-9: FTIR spectra (sample 4)

Table-8: FTIR functional analysis (Sample 3)

Wave	Group	Vibration	Intensity
3498	O-H	Stretching	Strong
1625	H-OH	stretching	Strong
1600	C=C	stretching	Medium-weak
1000	C-N	stretching	Medium
700	S-OR	stretching	Strong

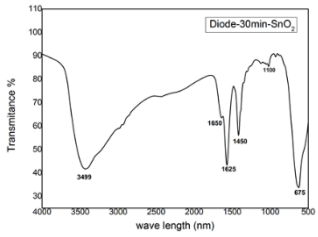

Fig-10: FTIR spectra (sample 5)

Table-9: FTIR functional analysis (Sample 5)

Wave	Group	Vibration	Intensity
3499	O-H	Stretching	Strong
1650	H-OH	Stretching	Strong
1625	H-OH	Stretching	Strong
1450	α-CH_2	Bending	Strong
1100	C-N	stretching	Medium
675	Sn-O-Sn	stretching	Strong

3.3 X-Ray Diffraction Analysis

The X-ray diffraction pattern of with and without laser treated samples are shown in Fig-11 – Fig-13.

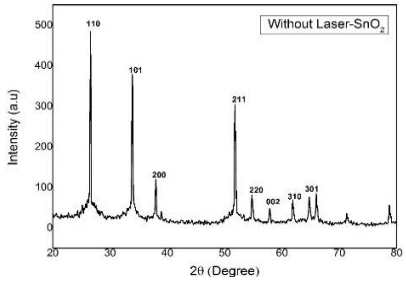

Fig- 11: XRD spectrum (sample 1)

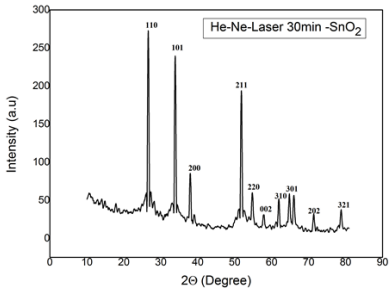

Fig- 12: XRD spectrum (sample 3)

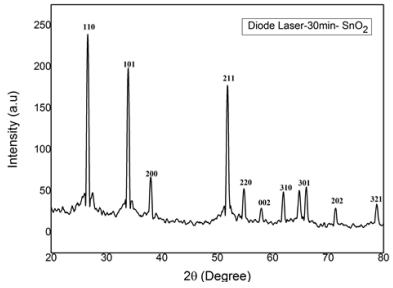

Fig- 13: XRD spectrum (sample 5)

From the X-ray diffraction studies, the structure of the SnO_2 nano particle observed is tetragonal rutile structure.The average particle size (D) was determined using the Scherer's equation [2]

$$D = K\lambda / \beta \cos\theta,$$

where, D is the crystallite size, K is the shape factor, being equal to 0.9 , λ is the X-ray wavelength, β is the full width at half maximum of the diffraction peak, and θ is the Bragg diffraction angle in degree. The average crystallite grain size calculated using Scherrer formula of SnO_2 [4] of samples is given in Table-6

Table- 10: crystallite grain size of SnO_2 samples

S. No	Sample	Grain size (nm)
1	Without laser-SnO_2	47.9
2	He-Ne laser-15min-SnO_2	48.2
3	He-Ne laser-30min-SnO_2	51.9
4	Diode laser-15min-SnO_2	42.1
5	Diode laser-30min-SnO_2	43.4

From Table-6 we observed that calculated grain size is 5.21nm in sample-3(He-Ne laser irteraction with time exposures of 30mins) which is slightly higher than the other samples.

3.4 Dielectric studies

The dielectric studies of laser treated nano particles SnO_2 are measured using LCRZ instrument for varies frequency at room temperature(starting from 50Hz to 200 KHz). the corresponding effective capacitance (Cp) and effective resistance (Rp) are measured. Finally the dielectric constants, dielectric loss and ac electrical conductivity of the samples were calculated using the expression [5,6].

$$\sigma_{ac} = (f \varepsilon \tan(\delta)) / (1.8 \times 10^{10}), \quad (1)$$

where, f is the frequency applied field in Hz, (ε) is the dielectric constant or relative permittivity and tan(δ) is the dielectric loss tangent or loss factor.

$$\text{Dielectric constant } (\varepsilon) = (Cp\ L) / (\Sigma_o\ A), \quad (2)$$

where, Cp is the measured capacitance, L is the thickness of the sample, A is the electrode area and (Σ_0) is the permittivity of free space (8.854 X 10^{-12} F/m).

The dielectric loss factor i.e. tan (δ) can be expressed by relation,

$$\tan(\delta) = \omega . Cp . Rp \ , \quad (3)$$

where, Cp is the measured capacitance, Rp is the measured resistance and ω is the angular frequency. From the Fig.14(a)-Fig.18(c) shows the graphical representation of dielectric constant, dielectric loss and a.c electrical conductivity.

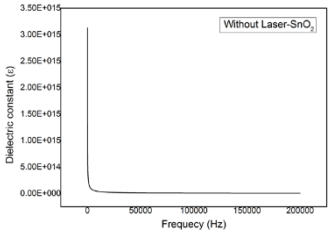

Fig -14(a): Dieletric curve of Dielectric constant (Sample 1)

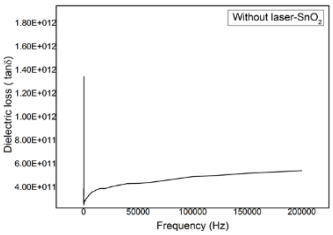

Fig -14(b): Dieletric curve of Dielectric loss (Sample - 1)

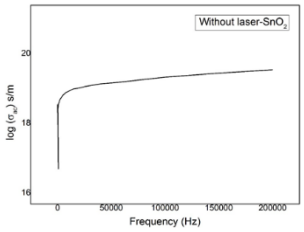

Fig -14(c): Dieletric curve of a.c electrical conductivity (Sample 1)

Table -11: Dielectric studies (Sample 1)

Frequency (KHz)	Dielectric constant (ε)	Dielectric loss (tanδ)	a.c conductivity (σ_{ac}) 10^{-5} s/m
10	3.9602×10^{10}	3.6949×10^{11}	18.9101
50	1.772×10^{10}	4.3497×10^{11}	19.153
100	7.621×10^{12}	4.928×10^{11}	19.3194
150	6.202×10^{12}	5.2046×10^{11}	19.4338
200	6.6478×10^{12}	5.4258×10^{11}	19.5321

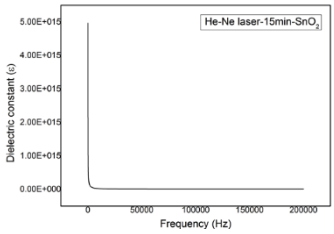

Fig-15(a): Dieletric curve of Dielectric constant (Sample 2)

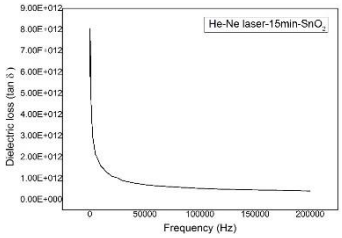

Fig-15(b): Dieletric curve of Dielectric loss (Sample 2)

Table -12: Dielectric studies (Sample 2)

Frequency (KHz)	**Dielectric constant (ε)**	**Dielectric loss (tanδ)**	**a. c conductivity (σ_{ac}) 10^{-5} s/m**
10	4.0827×10^{13}	2.5646×10^{11}	18.7647
50	1.0139×10^{13}	2.0605×10^{11}	18.7637
100	6.9678×10^{12}	2.3172×10^{11}	18.9528
150	5.9199×10^{12}	2.5429×10^{11}	19.0985
200	5.5117×10^{12}	2.7494×10^{11}	19.2263

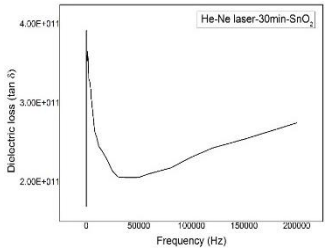

Fig -16(b): Dieletric curve of Dielectric loss (Sample 3)

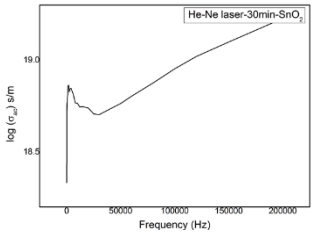

Fig -16(c): Dieletric curve of a.c electrical conductivity (Sample 3)

Table-13: Dielectric studies (Sample 3)

Frequency (KHz)	Dielectric constant (ε)	Dielectric loss (tanδ)	a. c conductivity (σ_{ac}) 10^{-5} s/m
10	2.4496×10^{13}	1.5746×10^{12}	19.3311
50	8.1654×10^{12}	7.0149×10^{11}	19.2017
100	6.2806×10^{12}	5.2796×10^{11}	19.2653
150	5.7022×10^{12}	4.5827×10^{11}	19.338
200	5.3756×10^{12}	4.0719×10^{11}	19.386

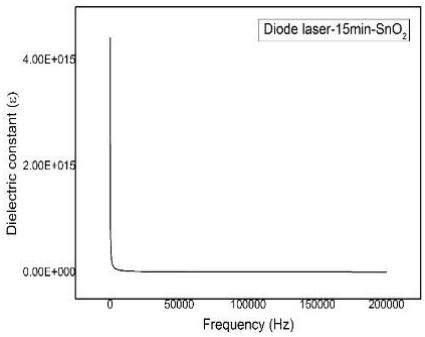

Fig -17(a): Dieletric curve of Dielectic constant (Sample 4)

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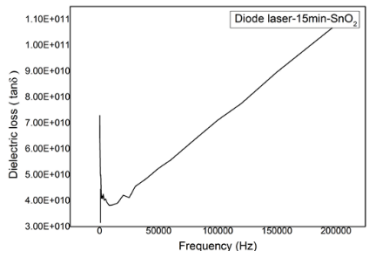

Fig -17(b): Dieletric curve of Dielectric loss(Sample 4)

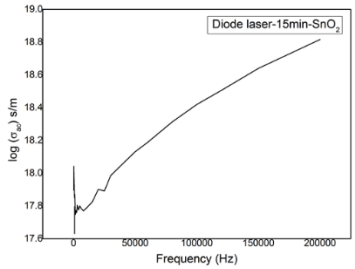

Fig -17(c): Dieletric curve of a.c electrical conductivity (Sample 4)

Table-14: Dielectric studies (Sample 4)

Frequency (KHz)	Dielectric constant (Σ)	Dielectric loss (tanδ)	a. c conductivity(σ_{ac}) 10^{-5} s/m
10	6.6480×10^{13}	2.6038×10^{11}	18.983
50	1.5446×10^{13}	2.3186×10^{11}	18.9978
100	9.1181×10^{12}	2.4512×10^{11}	19.094
150	7.0087×10^{12}	2.6512×10^{11}	19.1899
200	6.0560×10^{12}	2.8531×10^{11}	19.2833

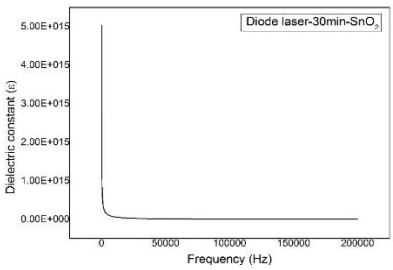

Fig -18(a): Dieletric curve of Dielectric constant (Sample 5)

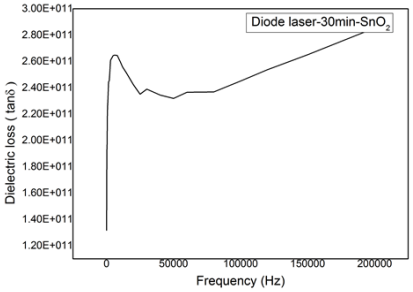

Fig -18(b): Dieletric curve of Dielectric loss (Sample 5)

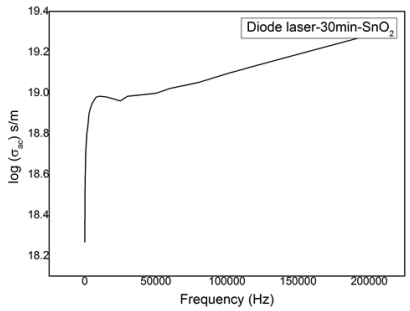

Fig -18(c): Dieletric curve of a.c electrical conductivity (Sample 5)

Table-15: Dielectric studies (Sample 5)

Frequency (KHz)	Dielectric constant (ε)	Dielectric loss (tanδ)	a.c conductivity (σ_{ac}) 10^{-5} s/m
10	2.8579×10^{13}	3.828×10^{10}	17.7838
50	9.2542×10^{12}	5.2574×10^{10}	18.1308
100	6.6412×10^{12}	7.1164×10^{10}	18.4192
150	5.8315×10^{12}	8.9691×10^{10}	18.6394
200	5.4572×10^{12}	1.0788×10^{11}	18.5157

The result shows liner decrease in dielectric constant values for increasing frequency and linear increase in dielectric loss for laser treated SnO_2 nano particles(reverse action for without laser irradiated samples) and for a.c electrical conductivity linear increase for with and without laser irradiated samples when the frequency increases.

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3.5 DLS Spectra (Measuring the Particle Size Distribution)

The DLS spectra of with and without laser treated samples are shown in Fig-19 – Fig-21.[7]

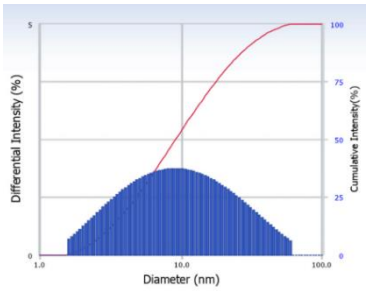

Fig-19: DLS spectra (sample 1)

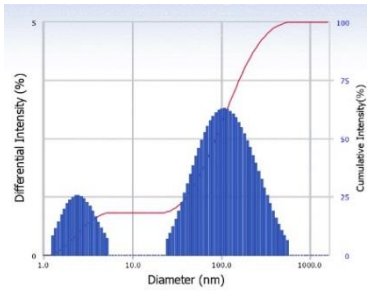

Fig-20: DLS spectra (sample 3)

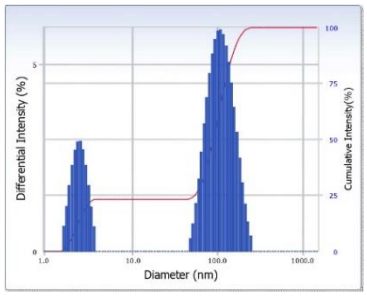

Fig-21: DLS spectra (sample 5)

Dynamic light scattering (DLS) studies revealed that the particle size distribution of Tin oxide SnO_2 nano-particles are 8.8 nm(sample-1), 62.5nm(sample-3), and 59.1 nm (sample- 5).

3.6 SEM Analysis

The SEM Analysis ofwith and without laser treated samples are shown in Fig-22 – Fig-24.[4,7]

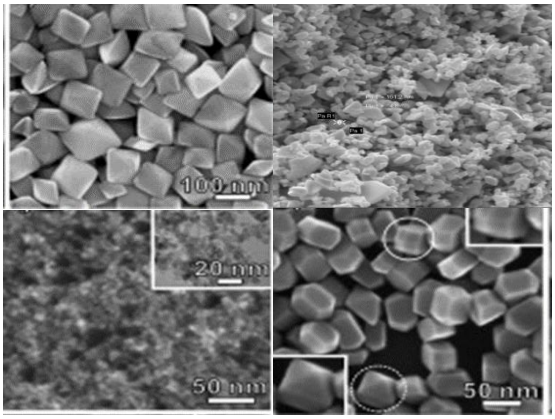

Fig-22: SEM image (sample-1)

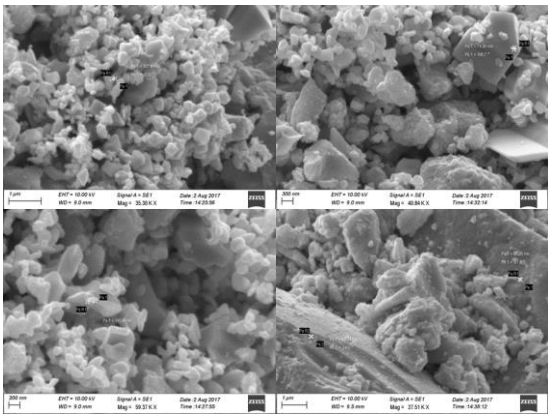

Fig-23: SEM image (sample-3)

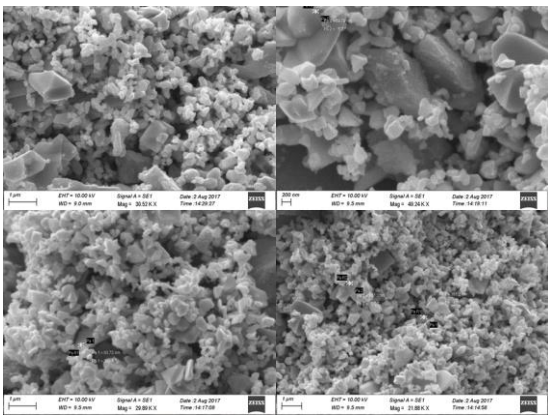

Fig-24: SEM image (sample-5)

SEM analysis of Tin oxide (SnO_2) Nano Particle, it is observed that the particles are in the tetragonal rutile shape within the particle size are in the range about 20 - 161.2nm (sample-1), 43.91-143.9nm (sample-3), and 59.79-161.2 nm (sample-5). The average particle size

observed in both SEM and XRD measurements are nearly equal values.

Table-16: comparative study of particle size for Tin oxide (SnO2) Nanoparticle (sample 1, 3 and 5).

SAMPLE	XRD Crystallite (nm)	DLS Particle (nm)	SEM Particle (nm)
1	47.9	8.8	20 – 161.2
3	51.97	62.5	43.91– 143.9
5	43.4	59.1	59.79– 161.2

4. CONCLUSIONS

In this, we investigated and reported the effect of laser heat treatment (He-Ne laser and Diode laser) on nano particles SnO_2. The results observed are given below ;

i. From the UV-visible spectrum studies, we observed thatthe band gap energy (for various laser exposures) from 3.6 eV - 3.8 eV (3.5 for sample-1 without laser irradiation).

ii. The functional groups for different peaks in FTIR are analyzed and the type of vibration as well as intensity are studied.

iii. From the X-ray diffraction studies, the structure of SnO_2nano particle (with and without laser irradiation) is tetragonal rutile structure.

iv. The crystallite grain size of the samples calculated are 47.9(sample-1) ,48.5(sample-2),51.9(sample-3), 42.1(sample-4) and 43.4 nm(sample-5) were observed from the XRD studies.

v. The dielectric properties of laser treated SnO_2 nano particles and their relation with frequency,dielectric constant,dielectric loss and a.c electrical conductivity are investigated.

vi. In general, when the laser power with increased time exposure gives improved result in band gap energy and grain size structure.

vii. Dynamic light scattering (DLS) studies revealed that the particle size distribution of Tin oxide SnO_2 nano-particles are 8.8 nm(without laser), 62.5nm(He-Ne laser treated with 30min), and 59.1 nm (Diode laser treated with30min).

viii. From SEM analysis of Tin oxide (SnO_2) Nano Particle, it is observed that the particles are in the tetragonal rutile shape within the particle size are in the range about 20 - 161.2nm (without laser),43.91-143.9nm (He-Ne laser treated with30min), and 59.79-161.2 nm (Diode laser treatea with30min).

ix. The average particle size observed in both SEM and XRD measurements are nearly equal values.

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