iJRASET
International Journal For Research in Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 6 **Issue:** IV **Month of publication:** April 2018

DOI: http://doi.org/10.22214/ijraset.2018.4725

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International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 6.887
Volume 6 Issue IV, April 2018- Available at www.ijraset.com

Optimization and Statistical Analysis of Binary Composite Y_2O_3/SiO_2 Prepared by Wet Chemical Route

Navneet Kumari[1], Dr. Sunil Rohilla[2]
[1]*Department of Physics, Mewar University Gangrar, Chittorgarh- 312901 (Rajasthan), India*
[2]*Materials Science Lab, Department of Applied Physics, TIT&S Engg. College, Birla Coloney Bhiwani-127021, India*

Abstract: ***Nanopowder binary oxides consisting of Yttrium oxide (Y_2O_3) and silicon dioxide (SiO_2) was prepared by the chemical co precipitation method. The effect of process variables lattice strain of Y_2O_3:SiO_2 was studied using Cental Composite design. The results revealed that the significant factors affecting lattice strain were concentrations and the rate/speed of addition/mixing of precoursors, value and duration of annealing temperature. The optimal calculated parameters were found to be annealing temperature —300^{0}C, drop rate—20d/min, concentration of precursors1—1 (w/v), concentration of precursors2—40 mmol/l, lattice strain $\mathcal{E}\approx.000121$. The formation of Y_2O_3:SiO_2 was confirmed by Fourier transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD) studies. The strain values are calculated from W–H plot for annealed samples.***
Keywords: **Nanocomposites, Binary oxides, nanocrystallites, Central composite, nanocrystalline**

I. INTRODUCTION

Nanocomposites containing nanocrystalline rare-earth oxides (R_2O_3) and silica have been investigated widely due to their use in many fields. Among various rare-earth oxides, Y_2O_3 (yttria) has recently attracted much attention due to some interesting properties of high thermal stability, conductivity and refractive index [1]. In addition, yttria has been used as one of the potential candidate materials for some functional applications like cathode radiation tubes, anti reflection coating, protection against chemical corrosion, laser amplifier and optical communication [2,3].Y_2O_3 powder had a body-centered cubic structure with an average size of 35 nm, while the SiO_2 powder was amorphous, with narrow size distribution. Y_2O_3 is an excellent luminescent matrix[4]. Exact composition of the intergranular glassy phase is difficult to determine[5].Due to aggregation of free standing Y_2O_3 nanocrystallites limits its suitability for technological applications. In order to overcome limitations of pure nanocrystalline Y_2O_3, it has been dispersed in an optically inert and transparent host. From technological point of view, silica matrix is one of the most suitable host matrices for nanocrystallites because of its chemical inertness, ease in getting spherical particles, optically transparency, higher softening temperature and higher thermal sock resistance as well as it supports in growing nanocrystallites of embedded materials [6,7]. The development of new rare-earth oxides and silica binary systems and their characterization are important not only for technological reasons but also for obtaining a better understanding. Literature survey [8-10] reveals that formation of rare-earth oxides/silicates inside or at the surface of amorphous SiO_2 matrix depends on the synthesis method, rare-earth oxide and silica molar ratio and thermal/pressure treatment. “In the present study we report synthesis of Y_2O_3-SiO_2 nano binary oxides. Y_2O_3-SiO_2 composite find uses in a wide variety of applications, such as X-ray imaging, display monitors, laser and amplifiers for fiber–optic communication [11]. The present study, investigates the influence of variables on the nanoparticle properties. the widely useful stastical design for the development and optimization of the relationship between measured variables and a number of independent variables in the form of polynomial equations[12]. Response surface methodology (RSM, combination of mathematical and statistical techniques) has been widely used to acquire the optimal operation conditions for both laboratory and industrial processes [13]. Response surface methodology, one of the most prominent statistical modeling technique. This methodology includes various types of experimental designs such as central composite, 3 level factorial, Box Behnken , D-optimal design, user defined, one factor, miscellaneous and historical data etc., The selection of experimental design is depending on the objectives of the experiment and the number of factors to be investigated. Central Composite design is one of the most popular Response surface designs and advantage of such methodology is to provide less experimental runs and time, thus provides more efficient optimization (Chaudhary et al., 2013; Peng et al.,2013 al., 2014). it is reported by the Montgomery that , The essence of good planning is to design an eriment able

to provide exactly the type of information important for the improvement of the process for obtaining the desired material[14]. In the present study we report synthesis of Y_2O_3-SiO_2 nano binary oxides. Y_2O_3-SiO_2 composite find uses in a wide variety of applications, such as X-ray imaging, display monitors, laser and amplifiers for fiber–optic communication[15].

II. EXPERIMENTAL DESIGN

For the synthesis of Y_2O_3 via co-precipitation method, oxides of ytterium (51×10-2 mmol) were converted to their chloride salt by adding stoichiometric amount of hot diluted hydrochloric acid after which transparent solution was formed. The ammonxpeia was used as precipitation agent. Then the solution of chloride salt was added to the ammonia solution at a certain rate by means of continuous stirring. The final solution was stirred for 30 minute. The mixture was centrifuged under 2000 rpm for 8 minute. The obtained precipitate was washed with distilled water three times and dried at 80 0C for 24 hours in an electronic oven. The final powder was heated at 800 0C for 30 minutes to complete the phase transformation to Y_2O_3.

A. Characterization of sample

Complementary methods were used to characterize annealed samples. In order to determine the crystallite size and lattice constant, XRD patterns of samples were recorded by using a Philips X-ray powder diffractometer PW/1710 having GIXRD geometry; with Ni filter, using monochromatic CuK radiation of wavelength 1.5418Å at 50KV and 40mA. The divergence of scanning beam on the source slide was controlled with the help of 0.15mm slit. The specimen were scanned in the range 200 to 800 . Fourier transform infrared spectrometer has been used to study the IR properties of prepared sample in the Mid-IR range, 4000-400 cm-1 using Perkin- Elmer instrument. Absorption spectra of the samples were studied with the help of Lambda 750 (Perkin Elmer) spectrophotometer in the wavelength range of 200–800 nm.

B. Statistical method and data analysis

The study explored the four main processing parameters in the method of Y_2O_3-SiO_2 was optimized using 3-level, 4-factor central composite experimental design. Concentrations of Y_2O_3. (x1), concentration of SiO_2(X2),Annealing temperature(X3) and rotation rate (X4) were used as independent variables on the basis of preliminary trials. Strain was selected as dependent variable and the effect of independent variables on Size and Strain was studied at 3 levels i.e., low (−1), middle (0), high (+1).The experimental design and statistical analysis of data were done using the Design Expert software (version 9.0.6.2). For this study. Experimental conditions for the tests conducted are summarized in Table 1,The Strain of nano particle based on the results of a preliminary set of experiments,

Table 1:-Experimental Design for Strain.

Factors	High level	Medium level	Low level
Con Y_2O_3(X1)	1	0	-1
Con SiO_2(X2)	1	0	-1
Temperature(X3)	1	0	-1
Drop rate(X4)	1	0	-1

Statistical analysis was performed using DesignExpert1 software. In this study, the optimum operation conditions strain was obtained by analyzing the relationships between the variables (con Y_2O_3,Con SiO_2,Temperature and Drp) and the response (Strain). The behaviour of the RSM in this study was expressed by the following polynomial equation[16-18]

$$Y = +\sum_{i=1}^{n} AiXi + e \qquad (1)$$

where Y is the response variable Ai, represents the linear effect regression terms; n is the number of independent variable; and e is the random error. Coefficient of determination (R^2) was used to describe the accuracy of the model; F value (Fisher variation ratio) and probability value (Prob > F) were applied to evaluate the significance of the model terms [16,19]
Design Expert to detect any outlier and unreliable result and collected data was in the acceptable range to be used to develop the

C. Statistical analysis and modeling

In the Central composite design performing 30runs the experimental results for particle size and strain, data was evaluated by model. Regression analysis was applied to develop the best-fit model using the collected data. In Table2, Design summary by RSM

for central composite experiments design has been given. While in Table 3, data obtained by RSM for strain(Response summary) has been given .

Table2: Design summary:-

		Factor 1	Factor 2	Factor 3	Factor 4	Response 1
Std	Run	A(con y_2o_3)	B(con sio_2):	C:temperature	D:drp	Strain
		G	g	^oc	r/m	---
27	1	-1	0	1	-1	0.000243
1	2	1	-1	0	-1	0.000158
22	3	-1	-1	$^{-1}$	-1	0.000119
16	4	1	1	1	1	0.000121
17	5	-1	0	0	0	0.000243
29	6	0	0	0	0	0.000443
8	7	1	1	1	-1	0.000589
28	8	0	0	0	0	0.000456
24	9	0	0	0	1	0.000444
21	10	0	0	-1	0	0.000589
20	11	0	1	0	0	0.000456
2	12	1	-1	-1	-1	0.000467
13	13	-1	-1	1	1	0.000378
10	14	1	-1	-1	1	0.000478
23	15	0	0	0	-1	0.000213
11	16	-1	1	-1	1	0.000401
26	17	0	0	0	0	0.000335
9	18	-1	-1	-1	1	0.000571
18	19	1	0	0	0	0.000374
5	20	-1	-1	1	-1	0.000368
6	21	1	-1	1	-1	0.000178
14	22	1	-1	1	1	0.000375
7	23	-1	1	1	-1	0.000215
4	24	1	1	-1	-1	0.000349
3	25	-1	1	-1	-1	0.000568
12	26	1	1	-1	1	0.000459
25	27	0	0	0	0	0.000375
15	28	-1	1	1	1	0.000167
30	29	0	0	0	0	0.000389
19	30	0	-1	0	0	0.000465

Table 3: Response summary.

Response	Name	Units	Obs	Analysis	Minimum	Maximum	Mean	Std. Dev.	Ratio	Trans	Model
R1	Strain	---	30	Polynomial	0.00012	0.000589	0.00038283	0.00012526	4.867	Power	Linear

The response Strain (Y1) was predicted by a linear equation shown as Eq. (2) below.

Y1=3.828E-004+5.694E-005*X1+5.839E-005*X2+7.050E-005*X3+3.489E-005X4 ---- (2)

where Y1 is the strain, X1 is the concentration of A, X2 is the concentration of B, X3 is the annealing temperature,X4 is the drop rate per minute .To evaluate the statistical significance of the quadratic model. F-test was conducted for the analysis of variance (ANOVA). The ANOVA tests results for the out put Strain is as shown in Table 4.

III. RESULT AND DISCUSSION

It is here very difficult to define ANOVA in a simple word precisely but yet we try, We can say that when several ideas are synthesized and they can be used for multiple purposes-ANOVA comes in frame. The collection of statistical models which are used to analyze the differences among group means and the procedures associated with them, For example, "variation" between groups, Ronald Fisher, the father of “ANOVA” was an evolutionary statistician and biologist. In the ANOVA, the observed variance in a particular variable, partitioned into components attributable to different sources of variation. ANOVA provides a statistical test which is used to check whether or not the means of several groups are equal and then generalizes the t-test to more than two groups. It is also used to compare three or more groups or variables for statistical significance. suited to a wide range of practical problems. ANOVA, a particular form of statistical hypothesis testing strongly used in the analysis of experimental data. A when a probability (p-value) comes out to be less than a threshold (significance level) now this type of result become statistically significant, justifies the rejection of the null hypothesis(all groups are simply random samples of the same population). In short, we can say that ANOVA is a statistical tool, which can be used in several ways to develop and confirm an explanation for the observed data. we can also do the given things with the help of ANOVA. It is computationally elegant, relatively robust against violations of its assumptions. With the help of ANOVA, multiple samples can be compared (statistical analysis). In the same way, it can also be used to analysis of a variety of experimental designs. ANOVA "is probably the most useful technique in the field of statistical inference. To obtain the difference between the variance of observations and the variance of means the result multiplied by the number of observations in each treatment,

Table 4: for ANOVA for Response Surface linear model..

ANOVA for Response Surface Linear model						
Analysis of variance table [Partial sum of squares - Type III]						
	Sum of		Mean	F	p-value	
Source	Squares	Df	Square	Value	Prob > F	
Model	2.311E-007	4	5.778E-008	6.45	0.0010	Significant
X1-con A	5.837E-008	1	5.837E-008	6.52	0.0172	
X2-con B	6.137E-008	1	6.137E-008	6.85	0.0148	
X3-temperature	8.946E-008	1	8.946E-008	9.99	0.0041	
X4-drp	2.191E-008	1	2.191E-008	2.45	0.1304	
Residual	2.239E-007	25	8.957E-009			
Lack of Fit	1.918E-007	20	9.589E-009	1.49	0.3495	not significant
Pure Error	3.214E-008	5	6.428E-009			
Cor Total	4.550E-007	29				

The fundamental technique deviding the total sum of squares (SS) into components related to the effects used in the model. For example, the model for a simplified ANOVA with one type of treatment at different levels. The number of degrees of freedom (DF) , partitioned in a similar way: one of these components (that for error) specifies a chi-squared distribution describings the associated sum of squares, while the same is true for "treatments" if there is no treatment effect. F-test can be used in ways like F-test can be used in both therefore one-way or single-factor ANOVA. By comparing the F-test statistic we can test statistical significance. The critical value of F is a function of the degrees of freedom of the numerator and the denominator and the

significance level depends upon the (α). Null hypothesis rejected only in case where F ≥ FCritical. The null hypothesis can also be rejected if probability is less than or equal to the significance level (α). F-test can be considered to be nearly optimal , to minimize false negative errors for a fixed rate of false positive errors (i.e. maximizing power for a fixed significance level). The F-test's p-values nearly approximate the permutation test's p-values. The approximation is particularly close when the design is balanced. Probability Values (p-Values)Probability values (P-values) It does not necessarily measure the importance of a regressor. An important regressor may have a large (nonsignificant) P-value if the sample is small, if the regressor calculated over a narrow range, For large measurement errors, or another closely related regressor can included in the equation. An unimportant regressor always have a very small P-value in a large sample. Computing a confidence interval for a parameter estimate gives you more useful information than just looking at the P-value, but confidence intervals can not solve problems of measurement errors in the regressors or highly correlated regressors.

Table 5 : Coefficient of determination (R^2),(ANOVA test result).

Std. dev	9.464E-005	R-Squared	0.5079
Mean	3.828E-004	Adj. R- Squared	0.4292
C.V. %	24.72	Pred. R-Squared	0.3347
PRESS	3.027E-007	Adeq. Precision	11.4267

Coefficient of determination (R^2), adjusted R^2 and predicted R^2 values were used to evaluate the fitness of the model. Adjusted R^2, which adjusts for the number of explanatory terms in a model relative to the number of data points. It is the is a modification of R^2[16-20]. How well a regression model predicts responses for new observations is represnted by, The predicted R^2 [21].The residual error to the pure error from triplicated experimental design points are compared by the lack of fit . In model, the p-value for lack-of-fit is 0.3495, which is greater than 0.0010, indicating that the lack-of-fit is not significant relative to the pure error. However, a model with reasonable R^2 value is acceptable with significant lack-of-fit [22-24]. Table 5 gives information regarding data for Coefficient of determination (R2),(ANOVA test result). The Model F-value of 6.45 implies the model is significant. There is onlya 0.10% chance that an F-value this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case X1,X2,X3 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The "Pred R-Squared" of 0.3347 is in reasonable agreement with the "Adj R-Squared" of 0.4292;i.e. the difference is less than 0.2."Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable, ratio of 11.426 indicates an adequate signal. This model can be used to navigate the design spaceThe red line was produced by the software based on the externally studentized to define outliers, . as shown in the diagnostics plots outlier exists in the plot indicating that the model is consistent with all the data. All the externally studentized residual were randomly scattered across the graph and Furthermore, there is no significant distribution pattern for all the diagnostics plots graphs. The residuals are normally distributed if the points on the plot follow a straight line [25] Normal probability plot of the studentized residuals to check for normality of residuals. Studentized residuals versus predicted values to check for constant error. Externally Studentized Residuals to look for outliers, i.e., influential values. Box-Cox plot for power transformations. A three-dimensional surface plot Figs. respectively, to provide a better visualization of the statistically significant factors derived from the statistical analysis.

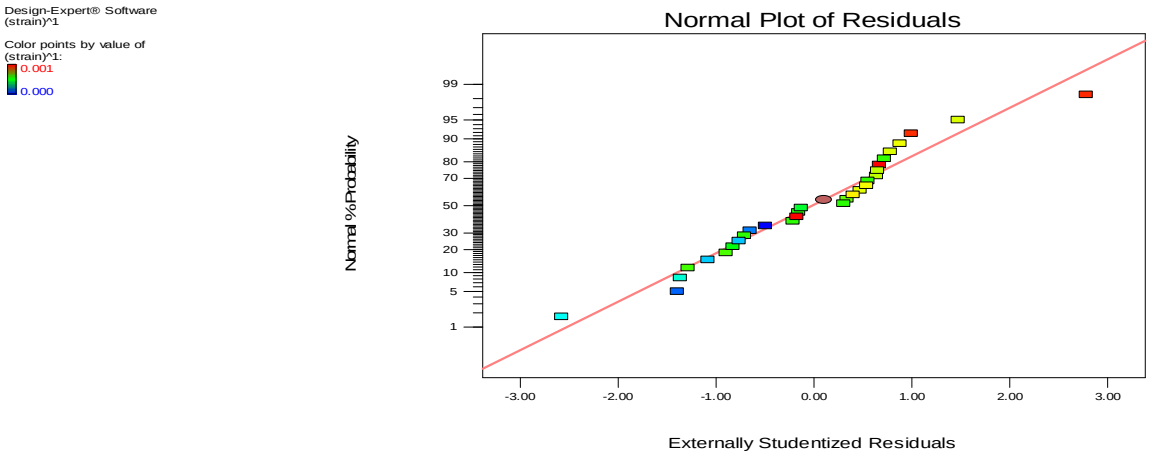

Fig1:Normal plot of Residuals(Externally Studentized).

The red line was produced by the software based on the externally studentized to define outliers as shown in the diagnostics plots Fig.(1-5). The outlier exists in the plot indicating that the model is consistent with all the data. All the externally studentized residual were randomly scattered across the graph and furthermore, there is no significant distribution pattern for all the diagnostics plots/graphs. The residuals are normally distributed if the points on the plot follow a straight line. Normal probability plot of the studentized residuals are to check for normality of residuals. Studentized residuals versus predicted values are to check for constant error. Externally Studentized residuals are to look for outliers, i.e., influential values.

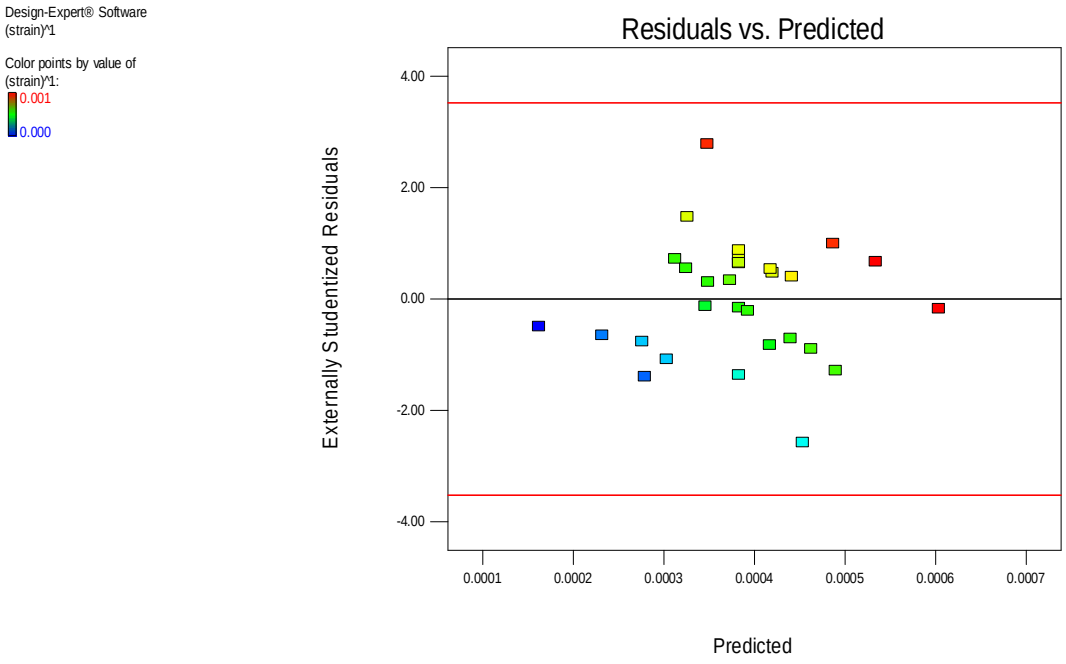

Fig2:Residuals vs. Predicted(Externally studentized)

After the model has been fitted, predicted and residual values are calculated, graphed, and output. The predicted values, calculated from the estimated regression equation; the raw residuals were calculated as the observed minus the predicted value. Often other forms of residuals were used for model diagnostics, such as studentized or cumulative residuals. Some procedures may calculate predicted mean values, standard errors of residuals and individual predicted values.

Let one may consider the observation of which is the row of regressors, is the vector of parameter estimate and for the residual variance (the mean squared error) it is S2. The leverage value of the observation is defined as

hi = wixi'(x'wx)-1xi

where x is the design matrix for the observed data, xi' is an arbitrary regressor vector (possibly but not necessarily a row of x), w is a diagonal matrix with the observed weights on the diagonal, and wi is the weight corresponding to xi'.

Then the predicted mean and the standard error of the predicted mean are

$\hat{Y}i = xi'\beta$

$STDERR(\hat{Y}i) = \sqrt{(s2\, hi/wi)}$

The standard error of the individual (future) predicted value yi can be calculated as

$STDERR(yi) = \sqrt{s2(1 + hi)/wi)}$

If the predictor vector $\mathbf{x}_i$ corresponds to an observation in the analysis data, then the raw residual for that observation and the standard error of the raw residual are defined as

RESIDi = Yi = xi'β

$STDERR(RESIDi) = \sqrt{s2(1 - hi)/wi}$

In the case where model assumptions may be reasonable in that situation Residuals, will appear to be normally distributed, close to statistically independent and have a constant variance. It will not differ for different treatment groups

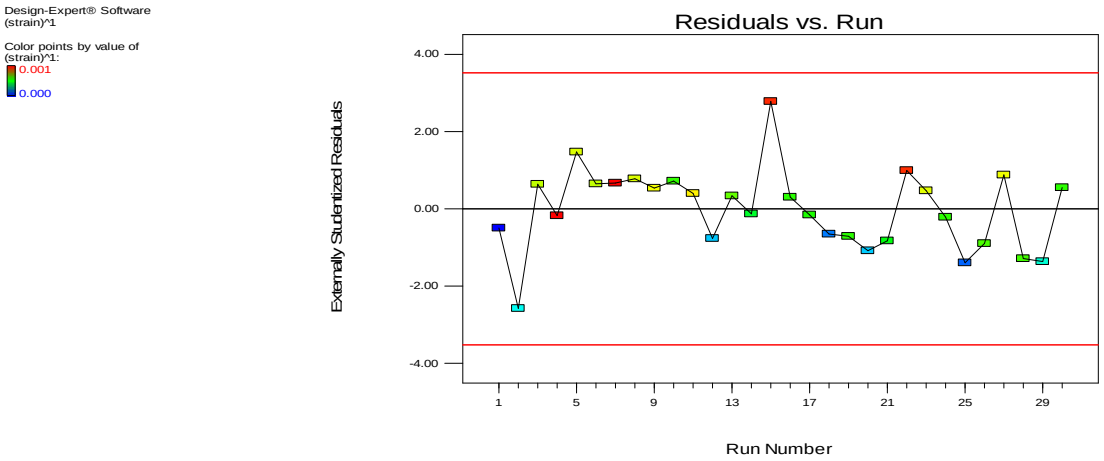

Fig3:Residual vs. Run Number(Externally studentized).

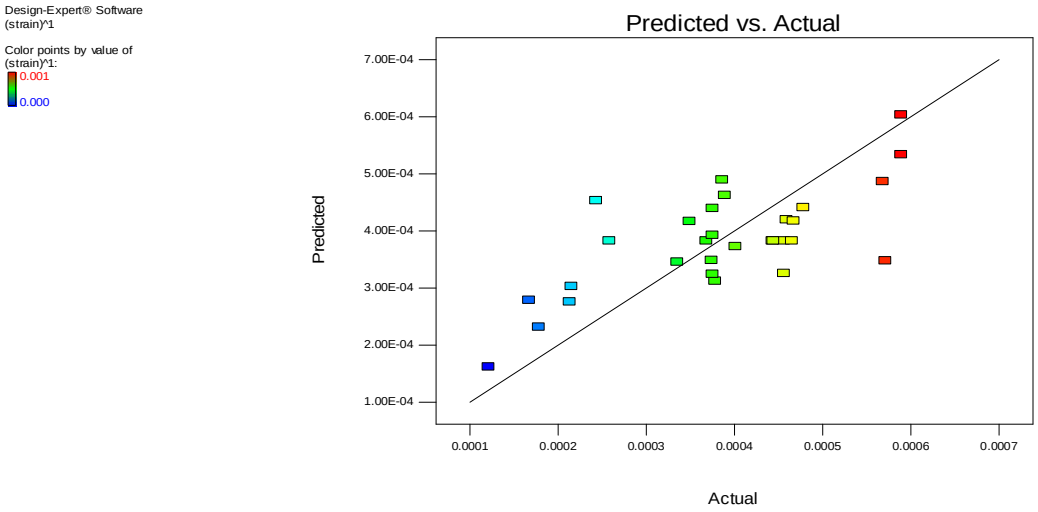

Fig4: predicted versus Actual.

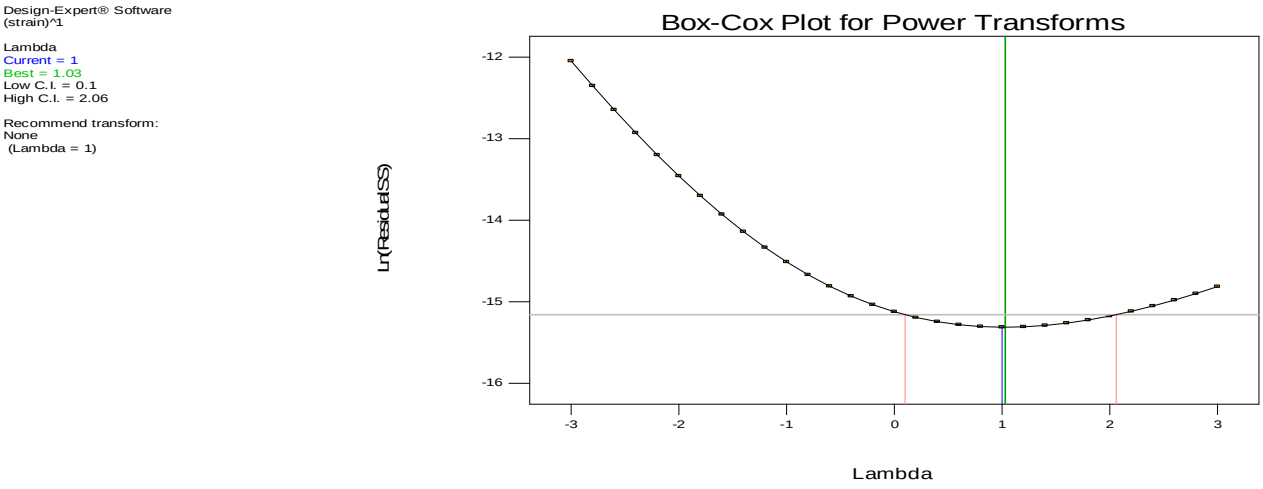

Fig5:Box-Cox Plot for Power transforms(Ln of Residual/Lambda).

The assumption of normality are followed by many statistical tests and intervals . The assumption of normality often leads to those tests which are simple, mathematically tractable, and powerful compared to tests that do not make the normality assumption. But some time, many real data sets are in fact not normal. In Table 6 dta for Box-cox Power Transformation has been given. However, an appropriate transformation of a data set may often yield a data set that follows a normal distribution. This increases the applicability and usefulness of statistical techniques based on the normality assumption. The correlation computed between the vertical and horizontal axis variables of the probability plot and is a convenient measure of the linearity of the probability plot and we know that the more linear the probability plot, the better a normal distribution fits the data

Table 6:Box-cox Power Transformation

Box-Cox Power Transformation Constant	95% CI	95% CI	Best	Rec.
k	Low	High	Lambda	Transform
0.000	0.100	2.06	1.03	None

A. XRD

X-ray crystallography is a tool used for identifying the atomic and molecular structure of crystal, in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. XRD results of the samples annealed at different annealing conditions have been shown in Fig(16). The fig.6, shows the diffraction peaks at $2\theta \sim 20.62^0$ (211), 29.30^0 (222), 33.94^0 (400), 36.06^0 (411), 38.06^0 (420), 40^0 (332), 43.64^0 (134), 47.06^0 (521), 48.68^0 (440), 50.28^0 (433), 53.36^0 (611), 56.32^0 (145), 57.76^0 (622), 59.18^0 (136), 60.58^0 (444), 62^0 (543), 63.38^0 (046), 64.66^0 (721). These peaks shows the cubic structure of Y_2O_3 [JCPDS file no. 741828].

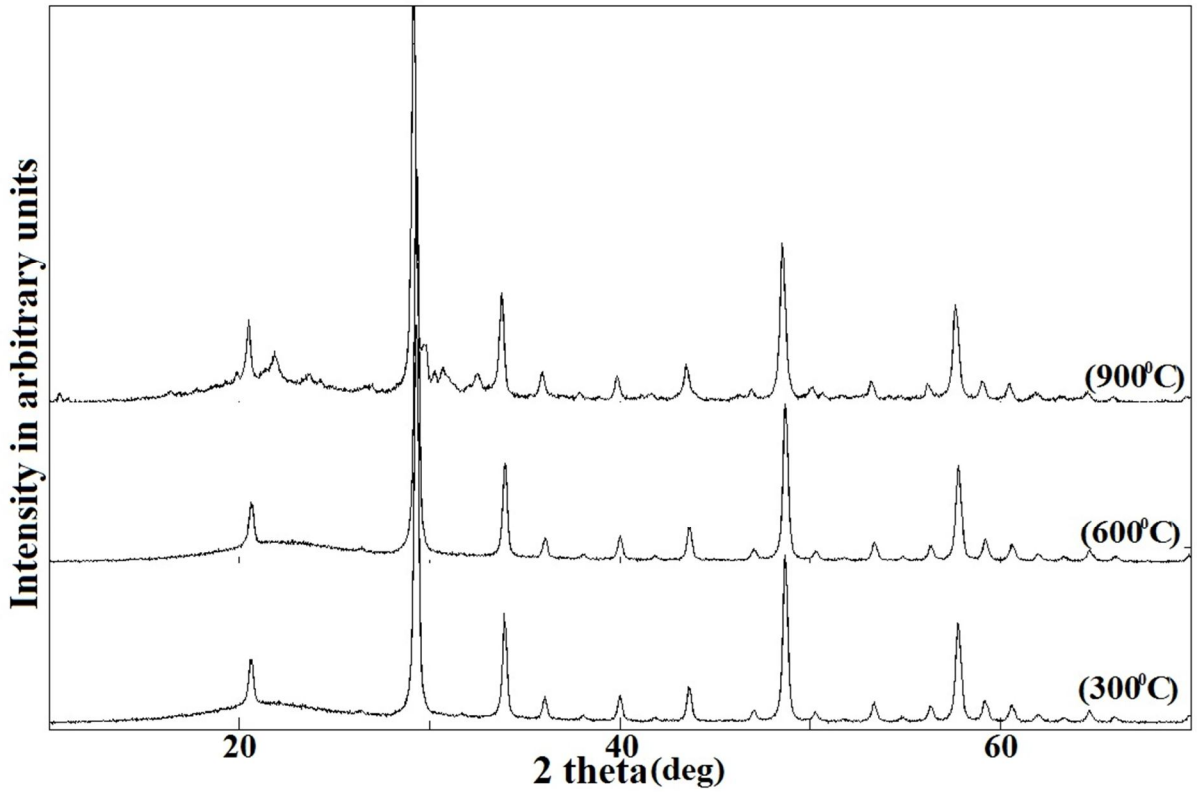

Fig.6 : XRD pattren of sample annealed at temperature 300ºC, 600ºC, 900ºC. showing sharpning of peak at higher temperature.

B. FTIR

Fourier transform infrared spectroscopy is a technique which enables us to identify the organic, inorganic materials and specially the presence of impurity phase in natural or synthesized materials. Fig (7) shows the FTIR spectrum of as prepared Y_2O_3/SiO_2. The peaks at 3447.07 cm^{-1} assigned to the stretching vibration of H–O–H. The absorption bands around 1636.16 cm^{-1} due to bending of H–O–H absorbed at silica surface. The two medium absorption bands around 799.14 cm^{-1} and 467.18 cm^{-1} are attribute to

symmetric stretching and bending vibrations of Si-O-Si bond. Band appeared around 563.01 cm^{-1} attribute to stretching vibrations of Y-O bond [10,11].The FTIR spectrum of Y_2O_3/SiO_2 annealed at 900 C shows in the fig (7). In this fig. peak at 3422.71 cm^{-1} shows several broad absorption band of the O-H. Peak at 1108.16 cm^{-1} and 801.49 cm^{-1} due to the symmetric and asymmetric stretching vibrations of Si-O-Si bond.

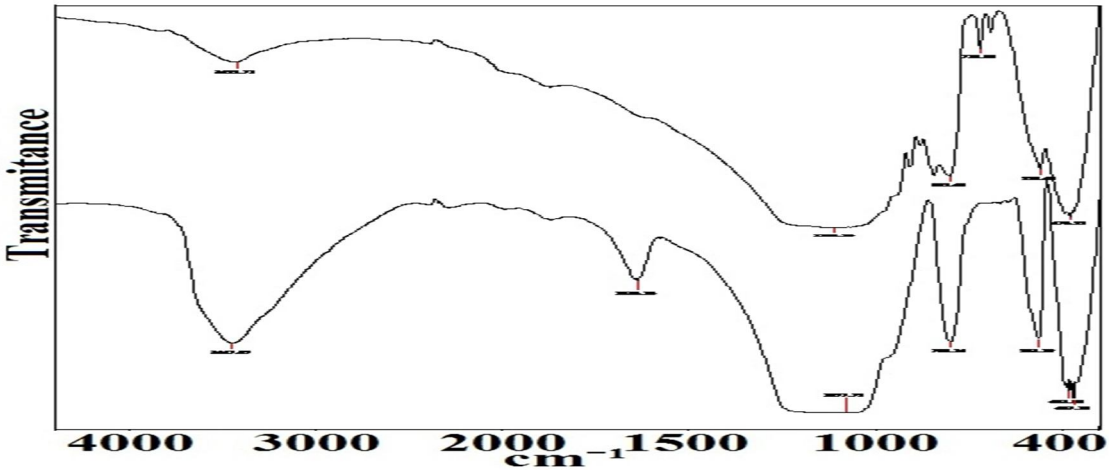

Fig. 7: FTIR

IV. CONCLUSION

Nanocrystalline Y_2O_3/SiO_2 powder with average size of 28 nm has been effectively prepared by chemical coprecipitation method. The cubic nature of yttria nanoparticles with crystalline silica is confirmed by XRD. The strain effect in peak broadening is calculated by W-H Plot. FTIR spectrum of samples analyzed the functional group and characteristic bond of the used precursors. Data were analyzed using Design Expert_ version 9.0.6.2 software. The significant effect of independent factors were analyzed using ANOVA. The Model F-value of 6.45(for strain) implies the model is significant. The correlation coefficient of determination R^2 was 0.5079 for strain indicating that the observed results fitted well with the model prediction, and the effect was also reported in the form of 3D perturbation plots.from 3D plot we concluded that size of particle is strongly affected by concentration and temperature and strain is affected by drop rate and concentation of sio_2 .

V. ACKNOWLEDGMENT

We are deeply grateful to Director of the T.I.T. Institution. He gives insightful comments and suggestions. I am particularly grateful for the assistance given by the Puran sir.

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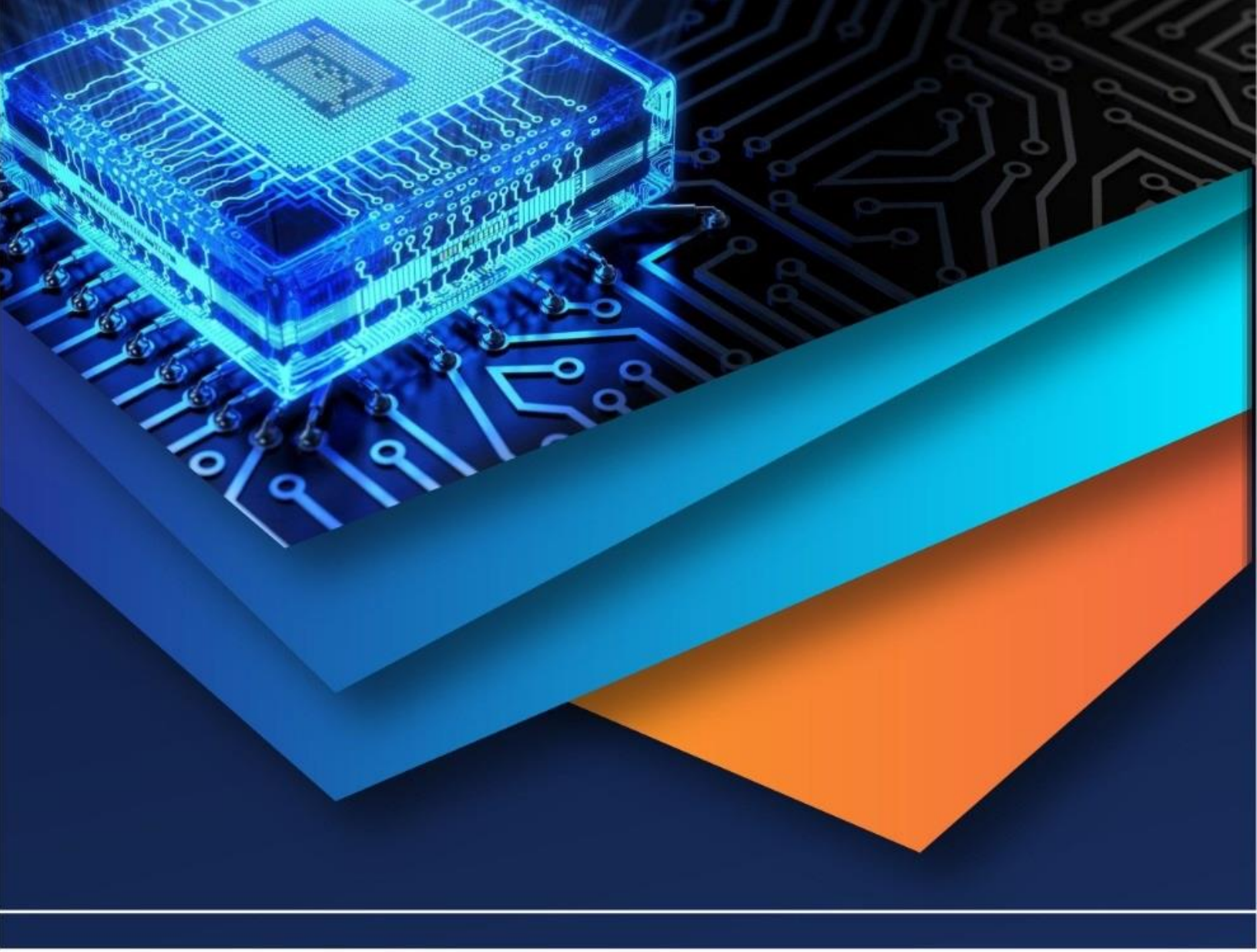

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