

Synthesis and Characterization of Silica-Nickel Nanocomposite through sol-gel route

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Abstract : The optimum combination of experimental variables- temperature, time of heat treatment under nitrogen atmosphere and amount of Ni-salt was delineated to find the maximum yield of nanophase Ni in the silica gel matrix. The size of Ni in the silica gel was found in the range 35 and 61nm. In the last two decades synthesis, characterization and understanding a material with lower dimensions have become the most interesting area of research due to their novel properties and potential applications in different fields.

Keywords: Sol-gel; Ni, Nano phase, Nanocomposites

I. Introduction

The synthesis and characterization of nanostructured materials have recently been studied world over because of their novel properties and potential applications in many fields and effort has been given for basic understanding(1-3) . Many methods have been used of which sol-gel technique is more versatile(4-6) .This technique has been exploited to synthesize metal insulator nanocomposites which have possible applications in optical switches and single electron transistor (7), fine soft magnetic materials(8) , microwave absorbing and shielding materials(9) , and super paramagnet(10)etc. The kinetics of the reduction of the metal salts in silica gel matrix has been widely studied. Detailed kinetic analysis have been carried out ,for in situ reduction of transition metal salts in silica(11) and silica- alumina gel matrix(12) to understand the mechanism of reduction and to compute the activation energy. This would help to control and tailor make the nanocomposites. The amount of metal along with other morphological characterization would influence the property (13).

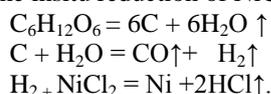
II. Experimental Procedure

The silica-Nickel gels were prepared through the hydrolysis followed by poly-condensation of tetraethyl-orthosilicate (TEOS) in water/ethanol solution under acidic condition. In this typical process, each one with TEOS:H₂O:C₂H₅OH ratio of 1:1:2 by volume respectively is prepared as follows. First a homogeneous solution is prepared with 1 ml of distilled water, containing required amount of NiCl₂, 100% excess glucose over stoichiometric requirement of complete reduction of NiCl₂, 5 ml of ethyl alcohol; and 4ml of distilled water under stirring. A second homogeneous solution of 5 ml C₂H₅OH and 5 ml tetraethyl orthosilicate(TEOS) is also prepared in a beaker under constant magnetic stirring. The first solution is added drop wise to the second one under continuous stirring with a magnetic stirrer followed by the addition of a few drops of 8 (N) HCl as a catalyst to the solution. The resulting solution is left for gelling at room temperature. We prepare the samples for 5 Wt%, 10 Wt%, 15 Wt%, 20 Wt%, of nickel with 100% excess glucose over that required for the complete reduction of NiCl₂ .

The heat treated samples were ground and passed through 400 mesh and XRD patterns of these were taken using Cu K α monochromatic radiation in Regaku make Ultima III, XRD instrument. The TEM images have been recorded with a JEOL JEM 1010 microscope. The sizes of nanoparticles from each TEM image were measured and the corresponding size histogram have been realised using specific software.

III. Results and Discussion:

The insitu reduction of NiCl₂ in silica gel occurs as follows as reported elsewhere in details [5]



The nanoporous silica gel matrix plays a very important role in this insitu reduction. The first one is that the nanopores restrict the reduced metal to be in the nanometric dimension. Secondly it is important to note that the reactions occur in nanopores simultaneously throughout the bulk of silica gel matrix which make kinetics of

reduction much faster. Thirdly since the nano sized metal is entrapped in the silica matrix subsequently there is hardly any oxidation.

Fig1-4, represent the XRD patterns of the silica gel samples containing 5wt%,10wt%, 15Wt% and 20wt% Ni heat treated at 550°C for 12 minutes. In each figure, peaks corresponding to Ni are marked.

Scherrer formula, given below has been used to compute the crystallite size of nickel,

$$t = 0.9\lambda/B\cos\theta$$

Where,

t is the average crystallite size in \AA

B is the width of the peak at half maximum in radian,

and λ is the wave length of X-ray in \AA .

The highest intensity peak in each case has been used to compute the crystallite size of the Ni for Silica-5 wt% Ni, 10 wt% Ni, 15 wt% Ni and 20 wt% Ni nanocomposite respectively.

Fig. 5-8 represent the TEM image of the silica - 5 wt% Ni, 10 wt% Ni, 15 wt% Ni, 20 wt% Ni nanocomposite respectively, heat treated at 550°C for 12 minutes in N_2 atmosphere. The Nickel particles of nanometric dimension are clearly visible in TEM image (Figs). It is to be noted from the TEM images that agglomeration is more for when the Nickel content is more, e.g. 10, 15 & 20 wt%. This is probably the reason why the image sizes estimated from TEM for these samples are significantly larger than those which are measured by scherrer formula. However, in TEM images for all the samples 5wt% to 20wt%Ni there is the distribution of size. This is because the nickel nuclei which are formed at the longer stage through reduction can grow for longer time.

The particle size of nickel was calculated (ref. Scherrer formula) and directly measured from the TEM images tabulated below in the table 1. From the table it is observed that the particle sizes of Nickel changes from 35 to 61 nm as the Nickel content in the Silica –Nickel nanocomposites increases from 5wt% to 20wt% of Ni..

The value of $\text{Sin}\theta/\lambda$ the hump in each of samples (ref. Fig 1- 4) is 0.116, very close to 0.12 which is attributed to amorphous silica[13]. This is plausibly due to slow kinetics of crystallization of amorphous silica arising out of lower reduction temperature for a shorter period of time. It is observed that with the increase in the content of Nickel, the relative amount of silica decreases, as a result of which the height of silica hump reduces & becomes negligible small for 20wt% Ni sample (ref. Fig.4).

Fig.9-12 represents the SAED (Selected Area Electron Diffraction) patterns for the above samples. From this fig we compute compute the interplaner spacing and compare with the standard dhkl of Nickel. Table 2 shows that the agreement between the standard and experimental dhkl are good. This suggest that d_{hkl} of Nickel and this is agree mental with the findings of XRD(Ref. JCPDS)

Fig. 13-16 represents the lattice image. The interplanar spacing (.21nm) matches reasonably well with the standard value of metallic nickel(.202nm) (JCPDS Card no.—1051) This further confirms the presence of metallic Nickel.

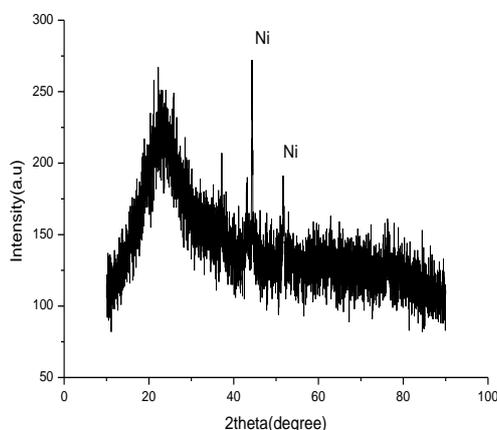


Fig 1 XRD pattern of sample containing Silica -5wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

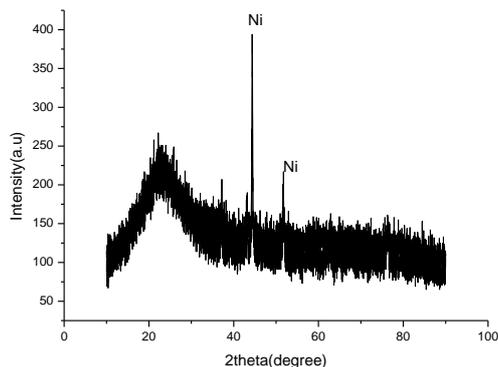


Fig 2 XRD pattern of sample containing Silica-10wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere.

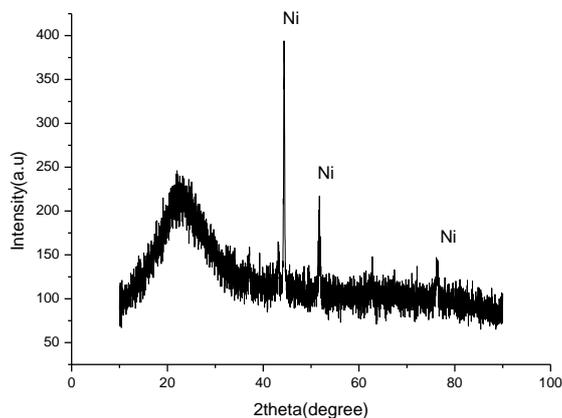


Fig 3 XRD pattern of sample containing Silica -15wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

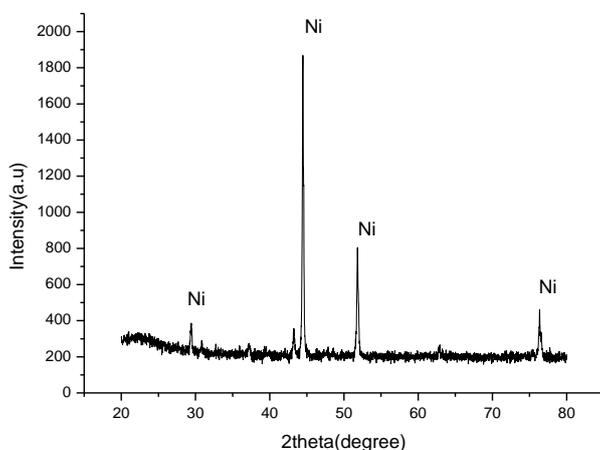


Fig. 4 XRD pattern of sample containing Silica -20wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

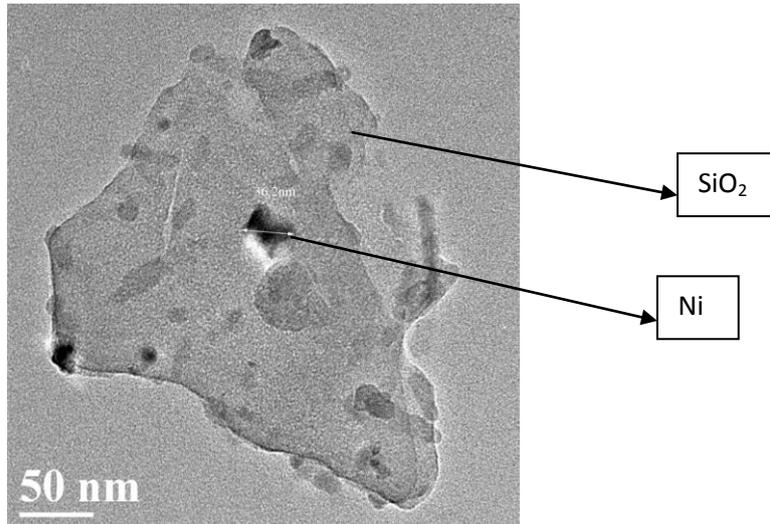


Fig. 5 TEM image of sample containing Silica - 5 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

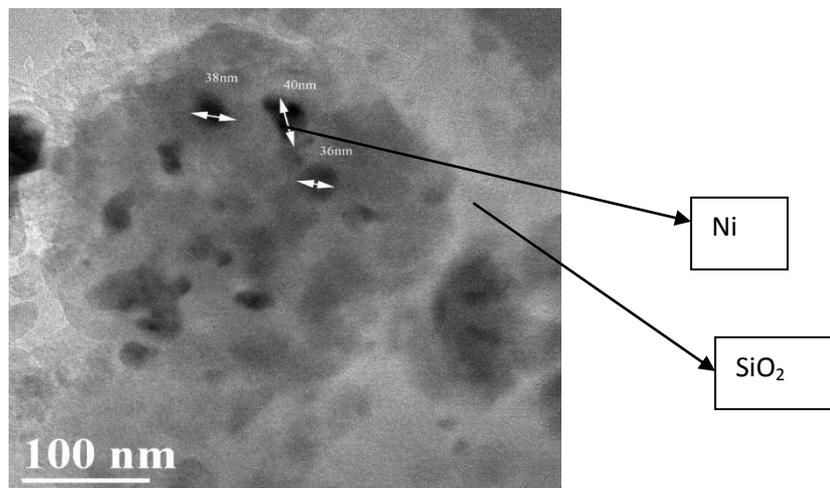


Fig. 6 TEM image of sample containing Silica - 10 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

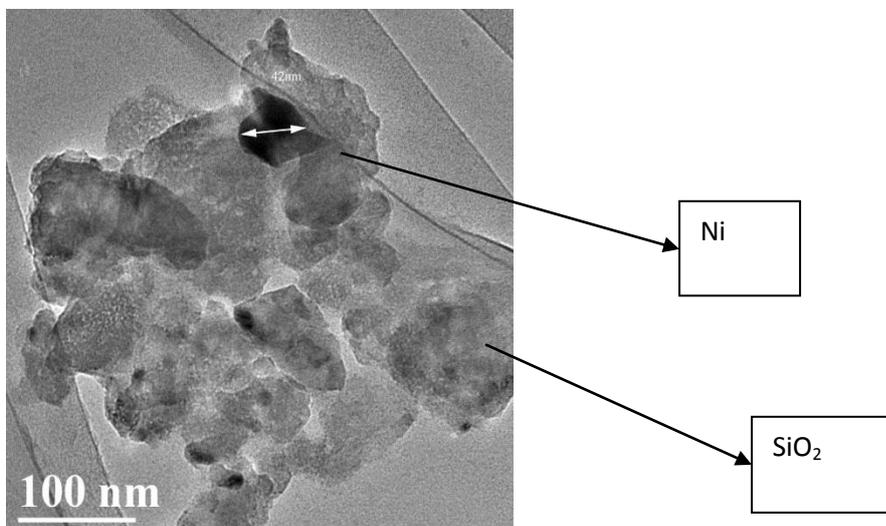


Fig. 7. TEM image of sample containing Silica - 15 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

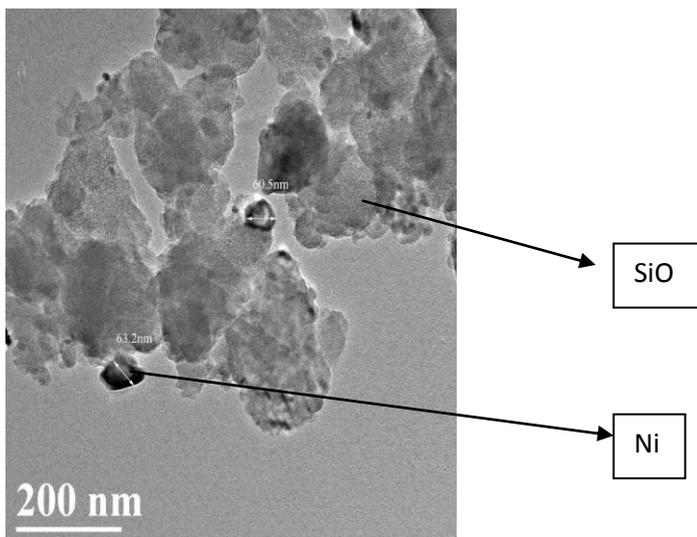


Fig. 8 TEM image of sample containing Silica - 20 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

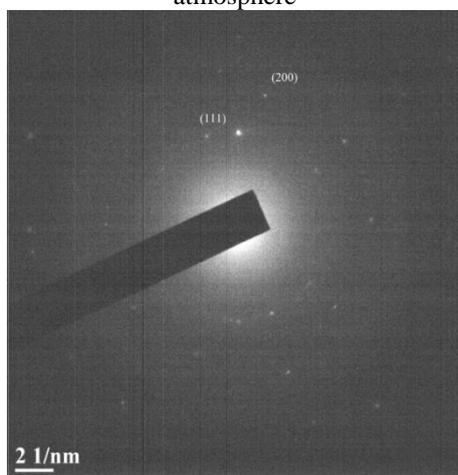


Fig. 9 SAED pattern of sample containing Silica - 5 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

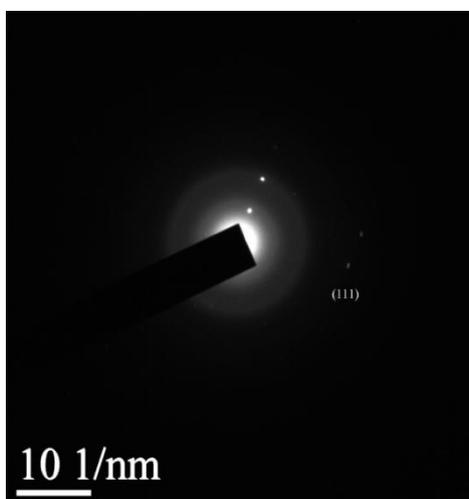


Fig. 10 SAED pattern of sample containing Silica - 10 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

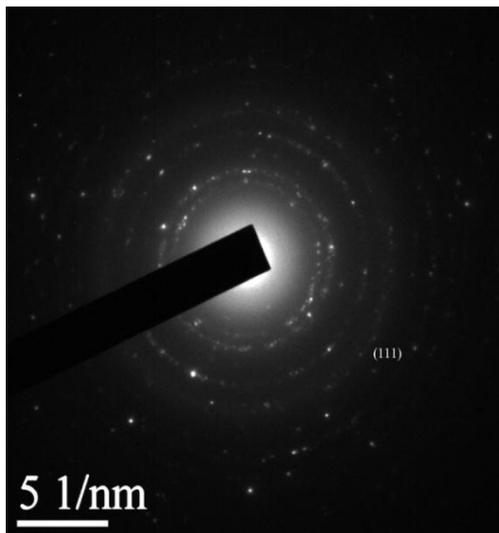


Fig. 11 SAED pattern of sample containing Silica- 15 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

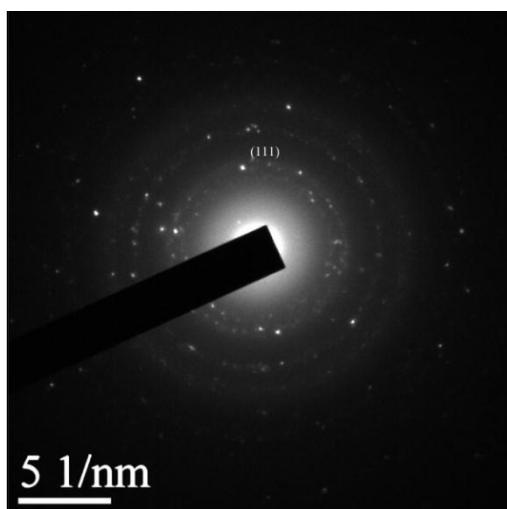


Fig. 12 SAED pattern Lattice image of sample containing Silica -20 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

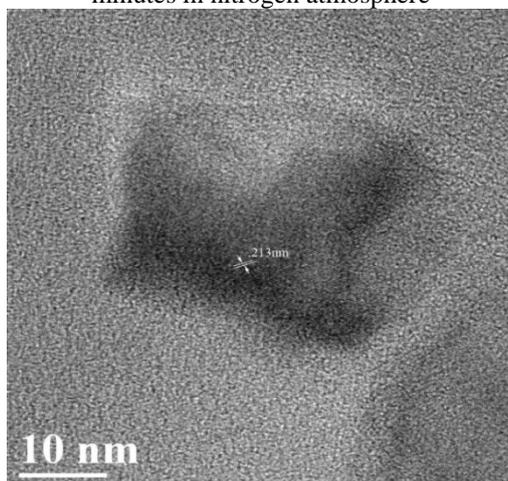


Fig. 13 Lattice image of sample containing Silica -5 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

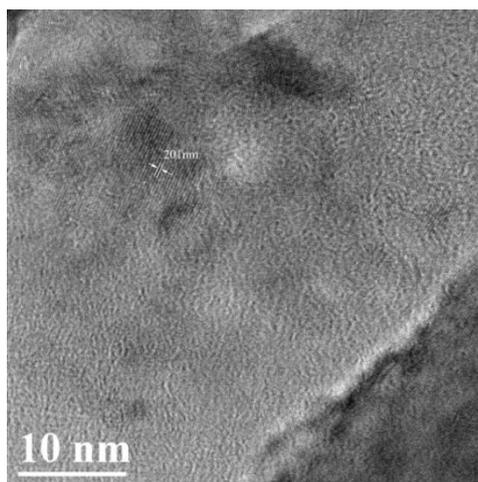


Fig. 14 Lattice image of sample containing Silica - 10 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

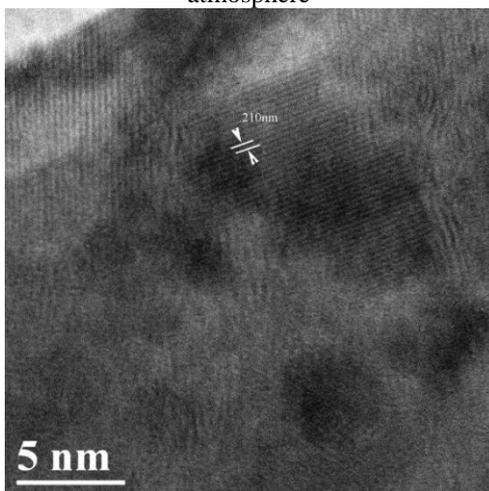


Fig. 15 Lattice image of sample containing Silica-15 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

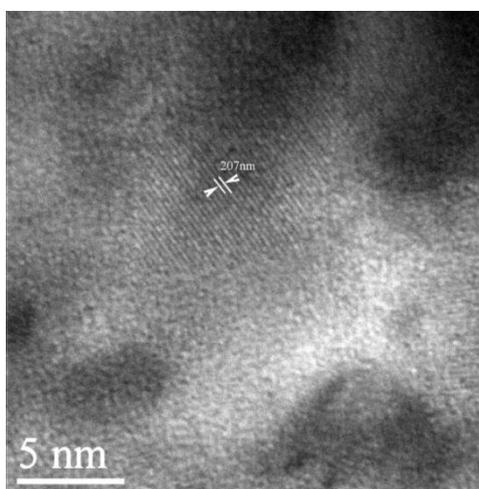


Fig. 16 Lattice image of sample containing Silica-20 wt% Ni heat treated at 550°C for 12 minutes in nitrogen atmosphere

Table 1 contains the particle sizes of Nickel in Silica-Nickel Nanocomposites

Samples	Nickel crystallite size from XRD	Nickel particle size from TEM image
Silica -5wt% Ni	35nm	36nm
Silica -10wt% Ni	40nm	39nm
Silica -15wt% Ni	52nm	53nm
Silica -20wt% Ni	60nm	61nm

Table 2 contains interplaner spacing and compare with the standard d_{hkl} of Nickel

Samples	$d_{(111)}$ value from XRD	d value from TEM lattice image	$d_{(111)}$ value from SAED pattern	$d_{(111)}$ value from JCPDS Card No. 03-1051
Silica -5wt%Ni	0.204 nm	0.213 nm	0.218 nm	0.202 nm
Silica -10wt% Ni	0.203 nm	0.212 nm	0.211 nm	0.202 nm
Silica -15wt% Ni	0.204 nm	0.206 nm	0.207 nm	0.202 nm
Silica- 20wt% Ni	0.204 nm	0.207 nm	0.209 nm	0.202 nm

IV. Conclusions

1. Silica-Nickel Nanocomposites have been successfully synthesized via sol-gel route through insitu reduction at 550°C for different percentage of Nickel.
2. The particle size of Nickel computed by scherrer is in the nanometric levels as observed from the X-ray diffraction patterns. It is observed that the Nickel particle size ranging from 35 to 61 for different Nickel content in the silica matrix from 5wt% to 20wt% is in good agreement with obtained from TEM analysis.

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