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SYNTHESIS AND CHARACTERIZATION OF CARBON NANO SPHERES USING NICKEL CATALYST BY THERMAL CHEMICAL VAPOR DEPOSITION

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ABSTRACT
Discovery of new nanostructures like carbon nanospheres are predicted to be a huge impact on applications of memory devices. In this paper Carbon nanospheres were grown on thin film nickel catalyst using thermal chemical vapour deposition. Different temperature was used to study the growth of nanospheres; Acetylene and Argon was used as precursors. Prior that nickel film was deposited on silicon substrate by using e-beam evaporation techniques. The prepared nickel film was annealed at different temperatures and it characterized by atomic force microscopy. It's indicated the when temperature increased film roughness is also increasing. After preparation of carbon nanospheres were studied by Scanning electron microscopy and Energy dispersive X-ray analysis. The growth carbon nanospheres were not in uniform size at 800°C. At the temperature of 850°C and 900°C shows the tower and agglomerated spheres. Energy dispersive X-ray analysis confirms the carbon and nickel elements. Growth mechanism was also discussed.

KEYWORDS
Carbon nanospheres, Nickel thin films, e-beam evaporation and Chemical vapor deposition.

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INTRODUCTION
Carbon nanostructure has different forms such as Carbon nanotubes (CNT)[1], Carbon Nanospheres (CNS), nanodiamond, and Fullerene etc. Carbon nanospheres are black and it's made up graphitic layers, based on the sizes carbon spheres can be classified into carbon beads (>1µm), carbon Nanospheres (50 nm to 1 µm), carbon onions (2 to 20 nm) and Fullerene (< 2 nm. Carbon nanospheres are attracted the explorers because of wide range of

applications in biology, clean energy technology and electronics.

The CNS are prepared by different techniques, sol gel emulsification[2], electro spinning[3] arc discharge[4], chemical vapor deposition[5,6] muffle furnace[7]. Also nanospheres formation as additional or impurity growth of carbon nanotube and graphene. Chemical vapor deposition is suitable method to produce carbon nanostructures such as carbon nanotubes[8,9] and carbon nanospheres because it's versatile technique.

The preparation of CNS required catalysts and precursor in Chemical vapor deposition techniques, so far used catalysts are metal thin films as Fe, Zn, Ni, and Co, Kaolin. Supported transition metal catalysts etc, in that decompose of Acetylene precursor (C_2H_2) high in nickel catalyst. So far to our knowledge, the growth of nanospheres using Ni as a catalyst with acetylene precursor at different temperature using chemical vapor deposition, which have not been yet reported.

In current study, we deposited Nickel (Ni) and film on silicon (Si) substrate using e- beam evaporation technique, the prepared film used as catalyst to growth of CNS at different temperatures. The prepared nickel thin film was characterized by Atomic Force microscope (AFM). Microstructure study was carried out by Scanning Electron Microscope (SEM) for CNS. The Elemental composition was found out using Energy dispersive X-ray Analysis. (EDAX).

EXPERIMENTAL PROCEDURE
Nickel (Ni) film was deposited using e-beam evaporation technique. The e-beam evaporation system (Hind high Vacuum) used for deposition is with source of Ni pellet prepared from commercially available powder. The thickness of the film was maintained at 20 nm. The film thickness was measured by a crystal thickness monitor. The target and the substrate distance were kept at 15 cm. The substrate holder able to rotate which will give the uniform thickness for all the substrate. The deposition vacuum was kept at 10^{-5} mbar. The deposition was done at using 5kv gun.

The rate of deposition is 7A$^{\circ}$/Sec. Prior to the deposition Si substrate were cleaned using Acetone and kept it in ultrasonic bath for 15 min with Millipore water.

The prepared Ni film was kept in the middle of the alumina tube in chemical vapor deposition. The alumina tube was evacuated by rotary pump at 750mm/hg level to clean the chamber. Argon (Ar) was introduced in the chamber while the system was heating up to required temperature; Acetylene (C_2H_2) was used at the growth temperature, maintained at 60 min, after the growth the C_2H_2 was shut off. The chamber allows cooling down under the Argon atmosphere.

The detailed experimental set up was given in Table No.1. All other parameter was same except the Temperature.

RESULTS AND DISCUSSION
Scanning Electron Microscope (SEM)
From the Figure No.3 (a) shows a sphere shaped carbon is appears at temperature of 800°C. Theses spheres are not in mono size. The diameter of the sphere is nanometers to micrometers. The image indicates the growth temperature is not enough all the catalyst becomes spheres, it shows the Ni grains all over the film with the spheres, Figure No.3 (b) shows the higher magnification image of carbon nano spheres, it has perfect Sphere shape At the Temperature 850 °C growth of CNS shown in Figure No.3 (c) The surface of the substrate Appears to be totally covered by the carbon deposition, for the nickel grains and grain boundaries can no longer be detected in this sample. Carbon spheres are less uniform in size ranges between 750 to 850 nm, no trace of Ni catalyst due to higher temperature. All the catalyst become spheres, the carbon sphere are formed one on another because of excess flow of acetylene. From the Figure No.3 (d) shows the tower of spheres and the spheres are so dense in this temperature the sphere sizes are not uniform it was few hundred nanometer to micrometer. Due to high temperature more carbon deposition on the catalyst particle and

it leads the fast deposition of carbon from precursor and it become a tower of spheres.

Growth Mechanisms

Figure No.4 (a) shows nickel catalysts particle formation and carbon deposition from Acetylene precursor. Optimum temperature leads to balanced carbon supply and catalyst particle formation allows formation of nanotubes, but here low temperature leads to island formation and less carbon soluble in catalyst particle and low catalyst and carbon supply made the sphere formation. Figure No.4 (b) the excess supply of carbon leads to new growth of spheres on the top of grown spheres, this is because on the tip of the carbon sphere may contain the catalyst particle, this is the growth site of other spheres but less growth time allowed formation of small sphere. Figure No.4 (c) Continues supply of carbon made the tower of spheres Figure No.4 (d) excess carbon supply and low catalyst support leads to agglomerated spheres.

Energy Dispersive X-ray analysis (EDAX)

Figure No.5 (a) shows the EDAX spectrum of carbon spheres prepared using Acetylene precursor, indicate that the maximum amount (96.42 %) of carbon is present and slight amount (3.58 %) nickel also were there. The carbon quantity indicates the prepared material were the carbon spheres, Ni is derived from the catalyst. From the spectrum of Figure No.5 (b) we observed that 95.81 % weight of carbon and 4.19% of Gold. This graph also indicates maximum carbon spheres were presented. The gold peak arising because of gold coating done, prior to imaging.

Atomic force microscopy (AFM)

Figure No.6 shows 3 Dimensional Images of atomic force microscopy with different annealing temperature. The scanning was done on dynamic mode on the area size 10μm. All the images were acquired in ambient air. The images indicate that the granules are in various sizes. Its observed in all the films and are distributed evenly. There are no regular shapes and separation in granules structure. The Figure No.6 (a) shows as deposited film nickel on silicon shows the relatively smooth films than the rest of the films, there are not much big granules. As a function of Annealing temperature Figure No.6 (b) 800°C (c) 850°C and (d) 900°C, the island size, valley and island shape become bigger when annealing temperature is increased. The rms values are 40.20nm, 70.22, 120nm, 175.18 nm respectively for as deposited film annealing temperature of 800°C, 850°C and 900°C. The sample roughness is increased when annealing temperature increased. These island help as catalyst for growth of carbon nanospheres.

Table No.1: Experimental details

S.No	Sample	Temperature (°C)	Acetylene (C_2H_2) (ml / min)	Argon (Ar) (ml / min)
1	Ni on Si Thickness: 20 nm, Growth time 60 min	800	100	30
		850	100	30
		900	100	30

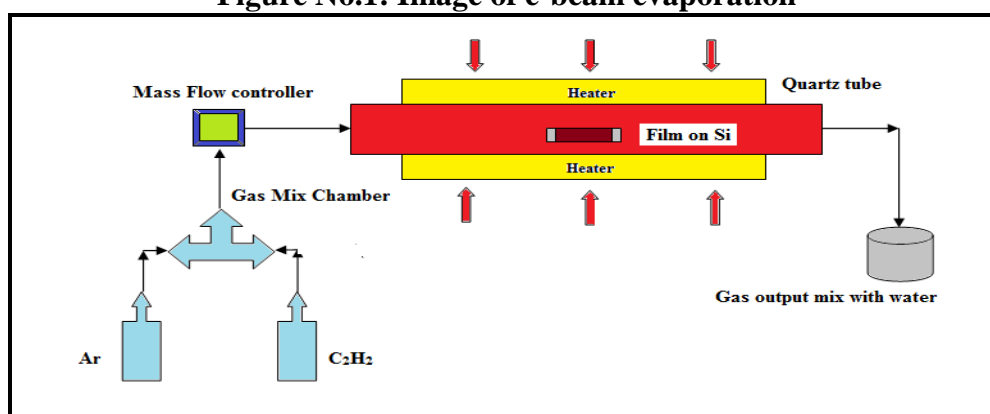**Figure No.1: Image of e-beam evaporation**
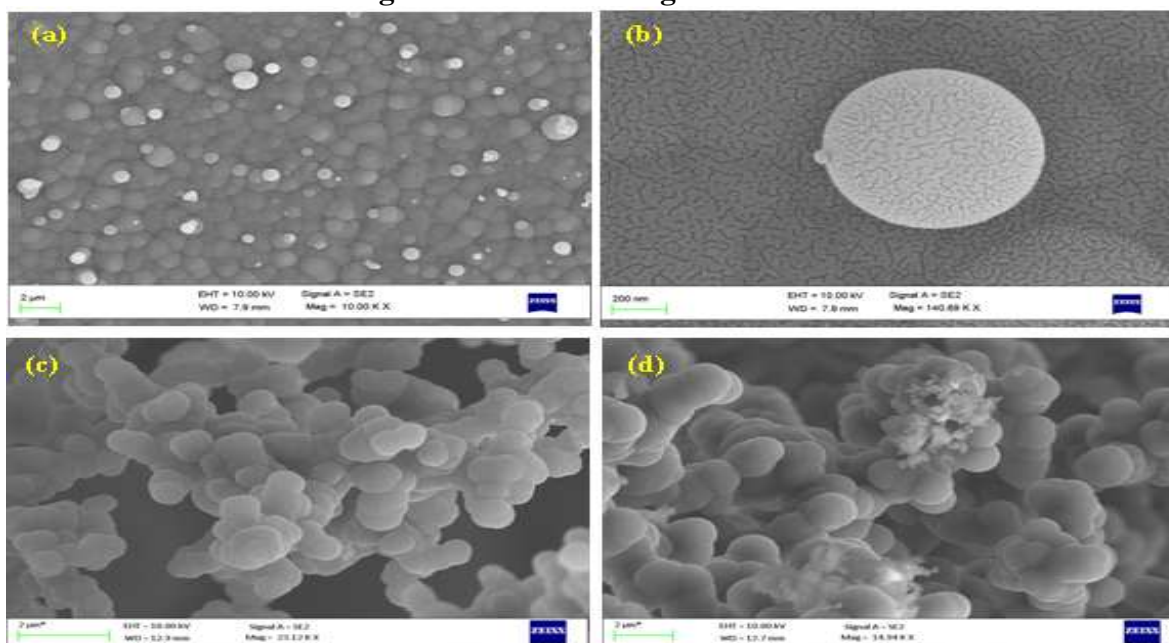

Figure No.2: Block diagram of CVD

Figure No.3: Shows the annealing temperature of (a) 800 °C lower magnification, (b) higher magnification (c) 850 °C (d) 900°C
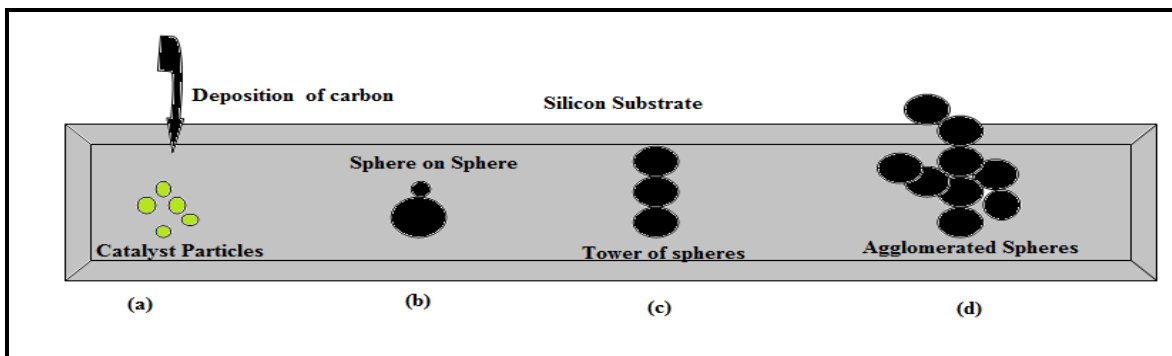
Figure No.4: Shows the Growth mechanism of CNS
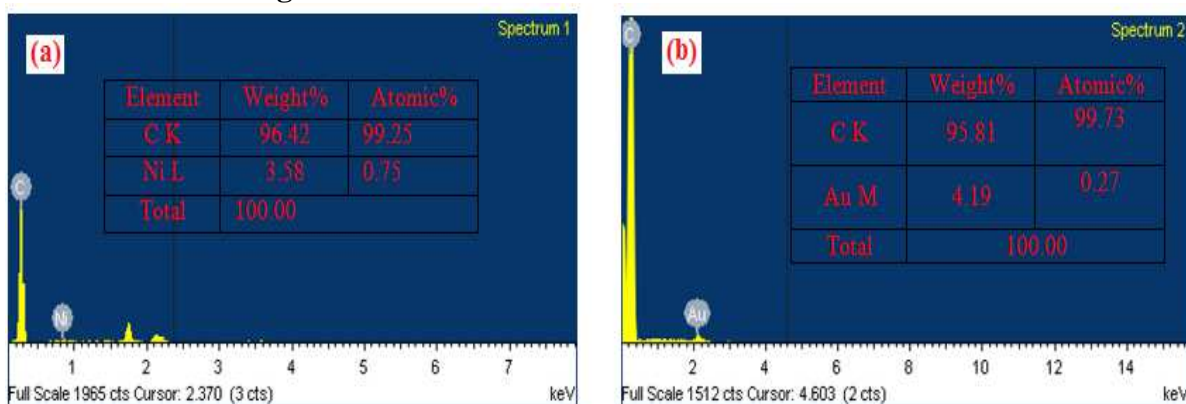

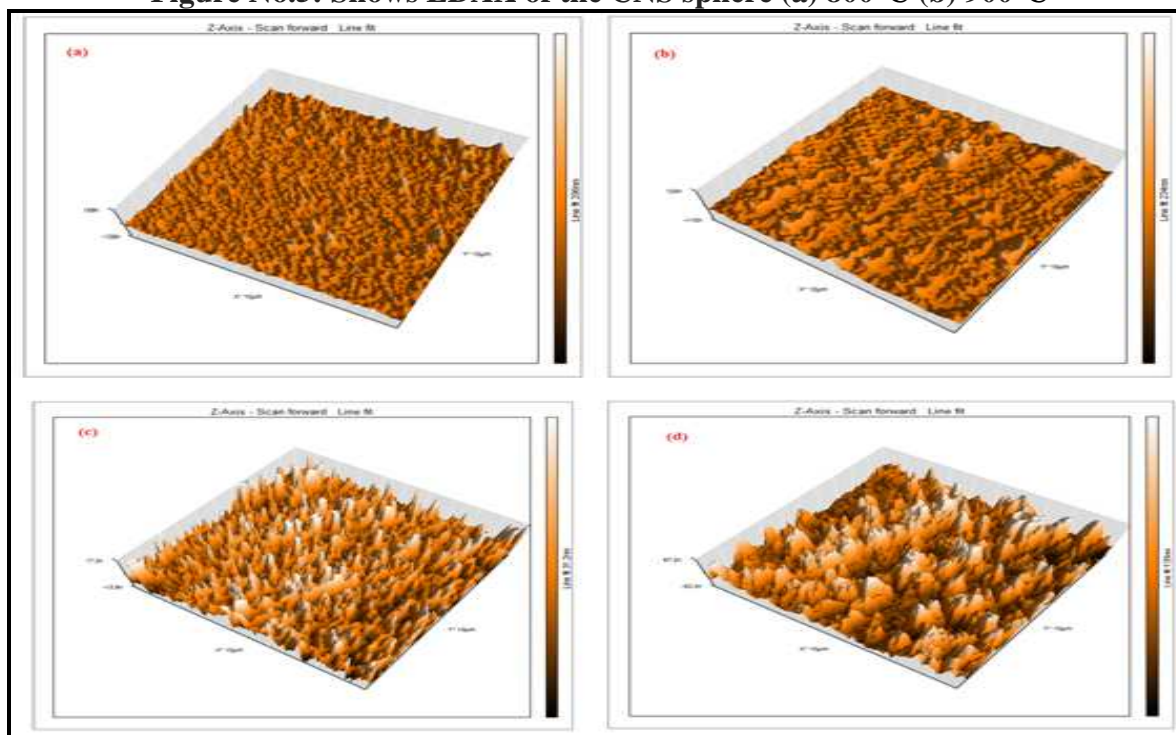**Figure No.5: Shows EDAX of the CNS sphere (a) 800°C (b) 900°C**

Figure No.6: shows the AFM images of annealing temperature at (a) As deposited film (b) 800°C (c) 850 °C (d) 900°C

CONCLUSION

In this work Nickel deposited film on silicon have showing the high roughness values with increasing annealing temperature. The growth of distinct CNS was achieved at 800°C temperature; the tower growth of CNS was at 850°C temperatures. At the temperature 900°C revealed agglomerated spheres. The EDAX confirms the elements were presented after the growth. Briefly growth mechanisms were discussed.

ACKNOWLEDGEMENT

CONFLICT OF INTEREST

We declare that we have no conflict of interest.

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