Nanoparticles and Nanostructured Cobalt Deposition using Dense Plasma Focus Device and their Characterization

W.P. SINGH and M.P. SRIVASTAVA
Department of Physics and Astrophysics, University of Delhi, Delhi-110007, INDIA
Savita ROY
Department of Physics, Kalindi College, University of Delhi New Delhi-110008, INDIA

(Received: 6 October 2008 / Accepted: 15 January 2009)

Nanofabrication through plasma route has been used for fabrication of Nanoparticles and Nanostructured materials as it is indicated that plasma route is superior to other physical and chemical techniques. Srivastava has established, the deposition of nanoparticles through plasma route on different substrates using Dense Plasma Focus device (DPF). DPF is a source of hot (1-2 keV) and dense (10^{26} m^{-3}) plasma. The hot and dense argon plasma formed during focus phase causes the ablation of the cobalt disc placed at the top of the anode. Brass holder is inserted from the top of the plasma chamber and is placed above the anode. The cobalt ions along with the argon ions move upwards in a fountain like structure and get deposited with three focused DPF shots on the silicon substrates placed at a distance of 5.0 cm above the anode. The structural properties have been studied using X-ray diffractometer. The surface morphology of the deposited cobalt has been investigated using Atomic Force Microscope (AFM). The nanoparticles have dimensions in the range of 40-80 nm. Magnetic Force Microscope (MFM) studies show that the nanoparticles are magnetic in nature.

Keywords:Cobalt Nanoparticles, Dense Plasma Focus Device, Atomic Force Microscope, Magnetic Force Microscope.

1. Introduction

Nanomaterials exhibit physical and chemical properties which can be tailored and are extremely different from the properties of bulk materials. Magnetic properties also change in the nanoparticles of magnetic materials. This paves the way to the search of magnetic materials that could be applied to nanospintronics and ultra high-density memory storage devices. Ferromagnetic nanoparticles are considered to be the key material from a viewpoint of the application for ultra high density magnetic storage media [1].

Nanofabrication of materials in laboratory through plasma route is established [2-4] to be one of the best routes. In chemical route, one follows the top down approach whereas the biological processes in nature follow the bottom up approach. In laboratory it is possible to adopt the bottom up approach for nanofabrication through plasma route. Plasma sources fall in different categories such as low density low temperature, moderate density moderate temperature, high density high temperature, equilibrium and strongly non-equilibrium, pulsed and steady state plasmas. Ion sources produced through plasmas are widely used for material processing. Many methods such as glow discharge, rf sputtering, magnetron sputtering, ionized physical vapor deposition (IPVD) etc. to produce ions having either low density low temperature or moderate density moderate temperature plasmas. The ions under such plasma conditions produce films and nanoparticles which in many cases require substrate biasing, substrate heating or annealing of the films. Recently, it has been established that ions produced in fusion plasmas under extreme conditions of density, temperature and in strongly non-equilibrium state are very much suited for the preparation of nanoparticles [2-4] as they don't require substrate heating or biasing or annealing of nanoparticles deposited. One such device used is Dense Plasma Focus (DPF) device.

Dense plasma focus device is a source of ions and electrons [5, 6]. High energy and high fluence argon ions produced in DPF device have been used for the deposition of nanostructures and nanoparticles. Srivastava [2,3] has discussed extensively the deposition technique and characterization of Nanoparticles. Earlier DPF device have also been successfully used for initiating phase changes [7-11] as well as the deposition of thin films [12-17] , nanoparticles [2-4] and more recently for

Author's e-mail: sav1roy@yahoo.co.in

FePt [18].

In the present work, we have reported the use of high temperature (1-2 KeV) and highly dense argon plasma (10^{26} m^{-3}) in Dense Plasma Focus device to produce fully ionized cobalt ions using cobalt on the top of the detachable anode. These fully ionized cobalt ions are deposited on the silicon substrates which are placed at a distance of 5.0 cm above the anode. These deposited cobalt nanoparticles are characterized structurally with X-ray diffractometer (XRD) and surface morphologically by atomic force microscope (AFM). The magnetic properties of nanoparticles have been investigated using Magnetic Force Microscope (MFM).

2. EXPERIMENTAL SET-UP

The Dense Plasma Focus (DPF) Device produces high temperature (1-2 KeV) high-density plasma (10^{26} m^{-3}) for duration of about hundred nanoseconds [2, 3, and 6]. The DPF is a coaxial gun accelerator consisting of two coaxial electrodes separated by an insulator sleeve. We have used in the present work a DPF device of Mather type [5, 6]. The schematic of DPF Device showing various subsystems is shown in Fig.1.

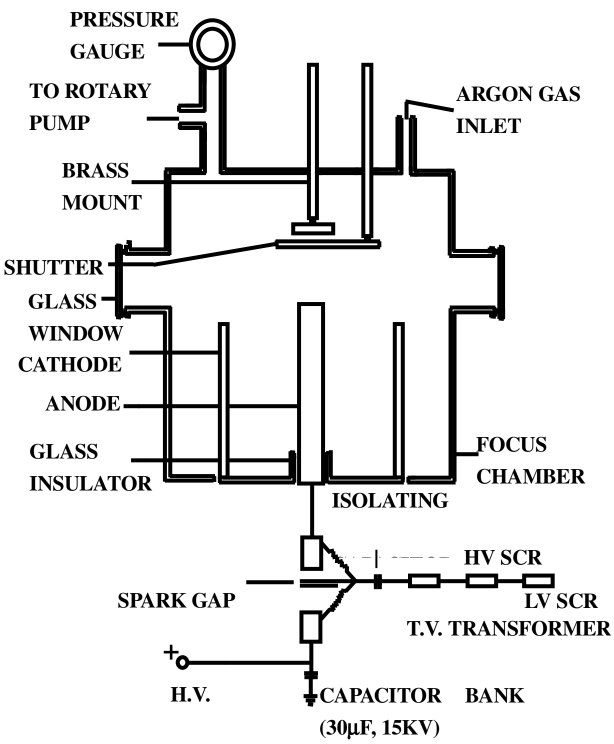

Fig.1. Dense Plasma Focus device with its modifications

The various subsystems of DPF are (i) focus chamber made of chromed mild steel. It is a cylindrical chamber consisting of coaxial assembly of electrodes with one anode surrounded by six symmetrically placed cathodes around it, (ii) the anode is made up of copper with a cobalt disc inserted in it, (iii) High voltage power supply to charge the capacitors, (iv) the spark gap as fast switching device to transmit the high voltage from capacitor to the electrodes inside the focus chamber, (v) the triggering electronics to activate the spark gap switches and (vi) focus diagnostic data acquisition system. The DPF Device is having energy of 3.3 kJ that is obtained by discharging a 30 μF capacitor charged to15 kV. This device is used for obtaining nanoparticles of magnetic material Viz. Cobalt.

N-type Silicon substrate with orientation (111) is cleaned and mounted on the brass substrate holder and is placed inside the vacuum chamber. The axial movement of substrate holder and its distance from anode is controlled by a brass rod from the outside. The focus chamber is evacuated and filled with argon gas. The argon pressure inside the chamber is maintained in the range of 80-100 Pa for getting good focusing. The capacitor bank is charged to 15 KV by high voltage charger and then discharged through the electrode assembly by triggering the spark gap arrangement. The plasma is formed on the top of the anode during radial pinch phase. After a few unfocused shots, a good focus is obtained which is evident from the sharp peak in the Voltage probe signal that is displayed on the oscilloscope (Tektronix TDS 784). A shutter, placed in between the top of the anode and the substrate, prevents ions from reaching the substrates when a good focusing signature on the storage oscilloscope is not obtained. The shutter is removed after the device gives good focusing. Each focused shot in this device is of ~ 1μs duration and the Cobalt ions are produced subsequently. The argon ions along with cobalt ions move upward in a fountain shape and cobalt ions get deposited on the silicon substrate. It was observed that silicon substrates get damaged when the distance between anode and substrates is less than 5.0 cm. Thus, the results of nanoparticles deposited at a distance of 5.0 cm from the top of the anode with three focused DPF shots are reported in this paper.

3. RESULTS AND DISCUSSION

X-ray diffraction was carried out using CuKα radiation. The X-ray spectrum of deposited cobalt nanoparticles on silicon substrates for three DPF shots is shown in Fig.2. It shows a peak at 2θ value of 61.87° corresponding to [hkl] plane of [102] with d-spacing of 1.498 Å [19]. Since the nanoparticles have short range order their d spacing may be at variance with the d spacing of bulk crystal lattice having long range order. The 2θ value from X-ray diffraction pattern when compared with JCPDS file database matches with the α-Co having (hcp) structure [19].

Author's e-mail: sav1roy@yahoo.co.in

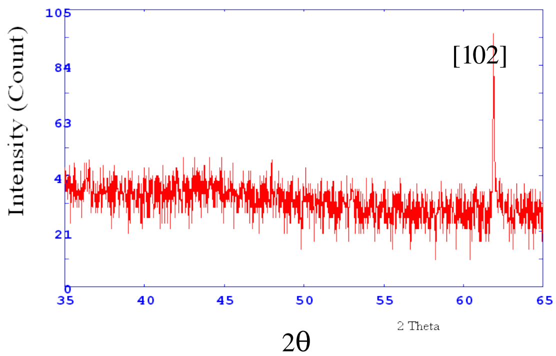

Fig.2. X-ray Diffraction pattern of deposited nanoparticles of Cobalt

Atomic force microscopy images have been taken using Scanning probe Microscope CP-II digital instrument. In this work the mode of measurement is non contact mode. The AFM images of deposited Cobalt on n-type silicon (111) substrate placed at a distance of 5 cm above the anode is shown in Fig. 3. The area scanned is 5 µm × 5 µm and 1 µm × 1 µm shown respectively in Fig. 3(a) and Fig. 3(b). Fig.3(a). show the tubular structures which on observing at higher resolution of atomic force microscope, scanning an area of 1µm × 1µm show the presence of nanoparticles. (b) of Fig. 3 shows cobalt nanoparticles of different sizes arranged inside the tubular structures. The diameter of the particles in (b) of Fig.3 is found to be in the range of 40nm – 80 nm. The AFM pictures were analyzed by SPM lab analysis software. The arrow 1 on (b) of Fig.3 indicates a typical nanoparticles whose radius is found to be 20 nm. A standard roughness measurement was done for Z-line (indicated by arrow 2). The parameter Ra gives the roughness average along Z-line to be 4.40 nm. The maximum height of deposit above the mean line drawn on the surface is given by parameter Rp

$$Rp = Z\min - \overline{Z}$$

Where Z is scaling line on the image. The value of Rp is 22.47 nm as evident from data points. The mean of maximum height above mean line is given by

$$Rpm = \frac{1}{Y}\sum_{i=1}^{Y}\langle Rp \rangle i$$

Author's e-mail: sav1roy@yahoo.co.in

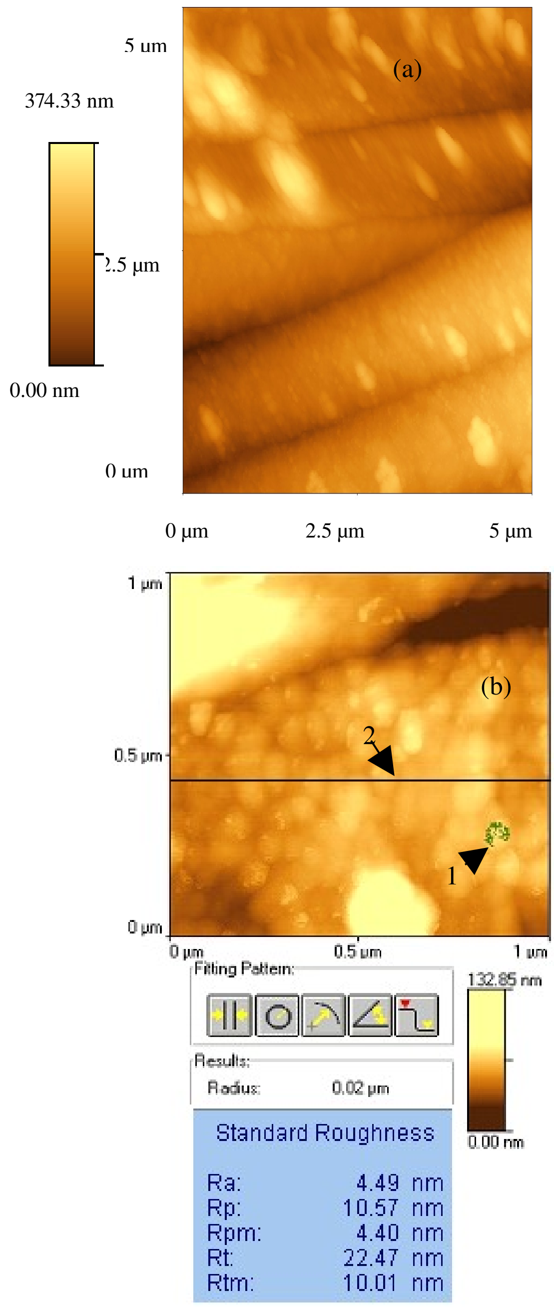

Fig. 3: AFM images of cobalt nanoparticles with scanned area of (a)5 µm ×5 µm and (b) 1 µm ×1µm.

Where Y=20. In Veeco algorithm for line profile measurement of this type, 20 highest features in profile are considered. The value of R_{pm} is estimated to be 10nm. Let Zmax be the point where the height of nanoparticles is maximum and Zmin be the point where the height of nanoparticles is the minimum *i.e.* valley, then maximum peak to valley height is given by

$$Rt = Z\max - Z\min$$

The value of R_t is estimated to be 4.49 nm which indicates fairly uniform deposition. The mean of peak to valley for the entire profile is given by

$$Rtm = \frac{1}{Y} \sum_{i=1}^{Y} \langle Rt \rangle i$$

and *Rtm* was estimated to be 10.57nm.

MFM images the spatial variation of magnetic force on the sample surface. Magnetic Force microscopy is performed with a vibrating cantilever whose tip is coated with ferromagnetic thin films. For magnetic measurement, the tip of the cantilever is magnetized and held at 15 nm to 20 nm above the sample so that the force of interaction is dominated by the magnetic force. Magnetic domains exert either attractive or repulsive force on the tip of the cantilever which causes the change in phase and amplitude.

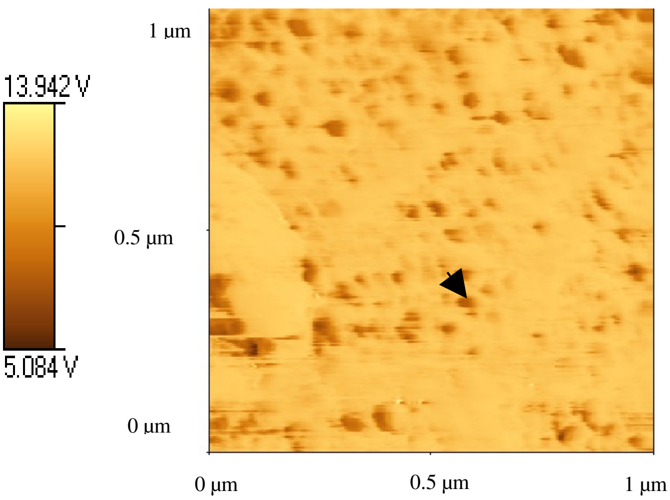

Fig.4 The magnetic phase Image of Cobalt nanoparticles.

Fig. 4 gives the magnetic phase image of nanoparticles deposited on Silicon substrate with three focused shots. The darker region show the region of attractive force and lighter region represent repulsive force of attraction between the tip and the sample surface. The typical size of magnetic domains (shown by arrowhead) is about 10 nm.

4. CONCLUSION

Nanoparticles of dimensions in the range 40 nm – 80 nm of Cobalt have been obtained on silicon (111) substrate by plasma-assisted nanofabrication method using Dense Plasma Focus Device. XRD establishes that α--Co nanoparticles, having hcp structure are deposited. MFM studies further establish that they are magnetic in nature with a typical dimension of a magnetic domain of 10 nm.

Author's e-mail: sav1roy@yahoo.co.in

5. ACKNOWLEDGEMENT

W.P. Singh is grateful to CSIR, New Delhi for the award of Senior Research Fellowship (SRF).

[1] Li.Yang,*et.al.,*Journal of Industrial and Engineering Chemistry **14,** 22 (2008).

[2] M.P. Srivastava, Scheriften Des Forschungszentrum Julich Gmbh Ed. H.J.Kunze, *et.al.*, **34,** 40 (2003).

[3] M.P. Srivastava, Advanced in Applied Plasma Science **6**, 227 (2007).

[4] K. Ostrikov, Reviws of modern physics. **77**, 489(2005).

[5] J.W .Mather, Physics Fluids**, 75** 1964

[6] S.Lee *et.al*.,Am J. Physics **56,** 62 (1993).

[7] R S.Rawat, *et.al*., Phys. Rev. **B 47,** 4858 (1993).

[8] R.Sagar and M.P.Srivastava: Phys.Lett. A.**183,** 209 (1996).

[9] M.P.Srivastava, *et.al.* Phys.Lett.A**. 215,** 63 (1996).

[10] P. Agarwal, *et.al*, Phys. Lett. A **231** 434 (1997).

[11] P. Agarwal, *et.al*., Physica C **313,** 87 (1999).

[12] C.R. Kant, *et.al*., Phys.Lett .A **226,** 212 (1997).

[13] C.R. Kant, *et.al.,*Phys. Lett. A **239,** 109 (1998).

[14] M.P. Srivastava, *et.al*., PAT/3.16.19/98060/93

[15] R. Gupta, *et.al.,* J. Phys. D: Appl. Phy. **37,** 1091 (2004).

[16] M.P. Srivastava and R. Gupta: Plasma Sour. Sci. technol **13,** 37 (2004).

[17] M.P. Srivastava and R.Gupta, Advanced in Applied Plasma Sci. **2,** 161 (1999).

[18] J.J.Lin, *et.al*., J.Phys.D: Appl. Phys. **41,**135213 (2008).

[19] F. Seitz, *The theory of solids* (McGraw-Hill Book Company, Inc) Chap.1, p.7