Formation of iron nanoparticles on quartz substrate using dense plasma focus device

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Abstract. Fabrication of nanoparticles and nanostructured materials is at the heart of modern nanoscience. In the present work, we are reporting the fabrication of Iron nanoparticles making use of high temperature, high density, and extremely non equilibrium pulsed plasma produced in 3.3 KJ Mather type Dense Plasma Focus (DPF) device. The Iron nanoparticles were deposited on quartz substrate at a distance of 5 cm above the ion source. The Iron nanoparticles are characterized structurally using X-ray diffractometer and topographically using Atomic force microscopy (AFM). The magnetic property was investigated using Magnetic Force Microscopy (MFM). It is found that these nanoparticles have a dimension in the range of 20-60 nm size and are magnetic in nature.

Keywords: Dense Plasma Focus (DPF), Magnetic material, Iron, Atomic Force Microscopy (AFM), Magnetic Force Microscopy (MFM)

1. Introduction:
The study of nanomaterials have drawn a lot of interest because of their application in many fields like electronics, magnetic, biomedicine, pharmaceutical, catalysis and material application. Properties of Ferromagnetic materials in the nanometer scale changes due to finite size effect [1]. Ferromagnetic nanoparticles are considered to be the key material from a viewpoint of the application for ultra high density magnetic storage media, medical imaging, Ferro fluids and catalysis. This leads us the way to the search of magnetic materials that could be applied to nanospintronics and ultra high-density memory storage devices [2-3]. Plasma route to nanofabrication is a bottom-up approach which is one of the interesting research field and possibly the only available laboratory technique for nanofabrication. The performance of plasma- based nanotools have proved superior to other competing nanofabrication methods and techniques [4]. Variety of plasma sources such as low density low temperature, moderate density moderate temperature, high density high temperature, equilibrium and strongly non equilibrium,
pulsed and steady state plasma had been used for deposition of thin films, nanoparticles and nanostructured materials. The results of improved adhesion on complex shaped substrates and increased reactivity with higher deposition rate for the deposition using plasma sources with moderate density and temperature which can produce ionization upto 90% are reviewed earlier [5]. It was a general misconception that high density high temperature and strongly non equilibrium fusion plasmas are not suitable for material processing and deposition of nanoparticles and nanostructured materials.

Recently, Srivastava and his research group had established in the series of the papers that high density high temperature plasma of DPF device similar to fusion plasma can be used for introducing phase changes [6-10], preparation of thin film [11-16] and nanoparticles [17-18]. It is found that this type of plasma is suitable for fabrication of nanoparticles and nanostructured materials. The deposition by DPF device is without any dc biasing, heating the substrates or annealing the deposited materials which are unavoidable in other techniques. DPF device is an efficient source of high energy ions [19-20]. In the present work, we have reported the use of high temperature (1-2 KeV) and highly dense ($10^{25}$-m$^{-3}$) pulsed argon plasma of DPF device to produce ionized Iron ions from Iron target material. These Iron ions along with argon ions move vertically upwards in a fountain like structure and get deposited on the quartz substrates which are placed at distance of 5.0 cm above the anode. These deposited Iron nanoparticles are characterized by X-ray diffractometer and Atomic Force Microscopy (AFM). XRD was characterized by using PW-370 diffractometer. AFM images have been taken using Scanning probe Microscope CP-II Digital instrument. In this work, the mode of measurement is non contact mode. The magnetic properties are also characterized qualitatively using Magnetic Force Microscopy (MFM). It images the spatial variation of magnetic force on the sample surface. It is performed with a vibrating cantilever whose tip is coated with ferromagnetic thin films such as sputtered cobalt. In this paper, the results of Iron nanoparticles deposited on quartz substrate which is obtained at a distance of 5.0 cm from the top of the anode with two focused DPF shots are reported.

2. Experimental set-up:
The Dense Plasma Focus (DPF) used in this work is a 3.3 kJ Mather type device which is powered by 30 µF, 15 kV fast discharging energy storage capacitor. It produces high temperature (1-2 KeV) high-density plasma ($10^{25}$-m$^{-3}$) for duration of about hundred nanoseconds [6-7, 19-20]. The DPF is a coaxial gun accelerator consisting of two coaxial electrodes separated by an insulator sleeve. The schematic of DPF Device showing various subsystems is shown in figure 1. The various subsystems of DPF are (i) focus chamber is a cylindrical chamber made of chromed mild steel which consist 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 Iron disc inserted on the top of 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. It is used for deposition of Iron nanoparticles on quartz substrates. Quartz substrates are cleaned and mounted on the brass substrate holder and are placed above the anode inside the focus chamber. The axial movement of substrate holder and its distance from the 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 at 80Pa 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. High density and high temperature plasma is formed on the top of the anode during radial pinch phase. After a few unfocussed shots, a good focus is obtained which is evident from the sharp peak in the Voltage probe signal that is displayed on the storage oscilloscope (Tektronix TDS 784). A shutter, placed in between the top of the anode and the substrate, prevents ions from reaching the substrates till a good focusing is obtained as is evident from the voltage probe signal.
obtained on the storage oscilloscope. The shutter is removed thereafter. The Iron in fully ionized state is produced by the hot and dense argon plasma on the top of the anode. The argon ions along with Iron ions move upward in a fountain like structure in post focus phase.

These energetic ions lose their normal component of the momentum to the quartz substrate and get deposited on it. Subsequently, Iron nanoparticles are deposited on quartz substrates which are placed at a distance of 5 cm above the anode for two focused DPF shots.

3. Results and discussion:
X-ray diffraction (XRD) was carried out using CuK$_\alpha$ radiation to identify the crystal structure of Iron nanoparticles deposited on quartz substrate for two focused DPF shots. The X-ray diffraction pattern shows no peak indicating amorphous nature of the particles.

Figure 2. AFM image of Iron nanoparticles on quartz.
The AFM image of deposited Iron on quartz substrate placed at a distance of 5.0 cm above the anode is shown in figure 2. The area scanned is 1 µm × 1 µm. It shows nanoparticles of Iron which are nearly spherical in shape. The roughness measurement and particle size were determined directly using software of CPII digital instrument.

From a typical line analysis roughness average, maximum height of the profile above the mean line, mean of maximum height above mean line, maximum peak-to-valley height and mean of peak-to-valley height were estimated to be 3.19 nm, 6.65 nm, 4.23 nm, 16.36 nm and 8.87 nm respectively. The size of the particles was determined by critical dimension analysis and was found to be in the range of 20-80 nm with a typical particle size shown by arrowhead to be 40 nm.

The topography, magnetic phase and lift mode phase images from MFM of Iron nanoparticles deposited on quartz substrate at a distance of 5.0 cm from the top of anode with two focused shots are shown in (a), (b) and (c) of figure 3 respectively. The arrowhead in figure 3(a) shows a magnetic domain of typical dimension 40 nm. The image shows dark areas along with bright structures of approximately 100 nm which are randomly distributed all over the image. The brightness of the black region reflects the depth of the ferromagnetic phase embedded in the sample as shown in figure 3(b).

**Figure 3.** MFM image of Iron nanoparticles on quartz substrate (a) Topography (b) Phase image (c) lift mode phase image.
The darker the area in the image more magnetic is the region [21] as magnetic tip of cantilever magnetizes these grains producing attractive force and give rise to dark magnetic domains. The lift mode scanning image gives better information about the magnetic nature of samples. This is because the distance between the cantilever and the tip of the sample is larger. So, the dominating force of interaction between them is magnetic in nature.

4. CONCLUSION
It has been demonstrated that high-density high temperature and under extreme non-equilibrium plasma conditions such as found in DPF device can be used for deposition of magnetic nanoparticles of iron. The deposition by DPF device is without any de biasing or heating the substrates or annealing which is unavoidable in other techniques.

5. ACKNOWLEDGEMENT
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6. References:
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