Synthesis and characterization of Structural and magnetic properties of electrodeposited Cobalt Iron thin film

A Project Report

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Master of Science

By

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I declare that this written submission represents my ideas in my own words and where others' ideas or words have been included, I have adequately cited and referenced the original sources. I also declare that I have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea/data/fact/source in my submission. I understand that any violation of the above will be a cause for disciplinary action by the Institute and can also evoke penal action from the sources that have thus not been properly cited, or from whom proper permission has not been taken when needed.

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This thesis titled "**Synthesis and characterization of structural and magnetic properties of cobalt iron thin film**" by Ravi Yadav is approved for the degree of Master of Science from IIT Hyderabad.

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Finally I wish to express my sincere gratitude to all the research scholars of Physics department at IIT Hyderabad

Thank you very much.

Dedicated to

My Professors

Abstract

In this project, I fabricated thin films of cobalt iron alloy of different thickness by electrodeposition technique and studied the domain and dynamics of the domain walls by taking measurements from magnetic force microscopy (MFM). I measured the roughness by taking measurements from Atomic force microscopy. I characterized the films with XRD which showed the crystal structure of the film. The SEM images of Cobalt iron film exhibited nano crystallized structure and the variation of granular size as a function of the potential at which the film deposited.

Contents

CHAPTER 1

INTRODUCTION TO MAGNETIC THIN FILMS

1.1 Magnetic thin films and its relevance in magnetic data storage technology

Thin films have gained lot of research attention in the last few decade and are expected to play a very important role in the development of Nano science and nanotechnology. Thin film have been explored with a wide range of potential applications in every field of physics like optics mechanics, magnetics, electricity etc. Diffusion barrier, protection against corrosion, data storage application, squids insulating / conducting coating are some applications of thin films Data storage is one of the important and potential application in the spectrum of application of thin film and the good thing is that the researches are going very strong in this field. This can be seen in the large increase in the density storage of computer hard drives. Now a days a 100s of Gigabytes of hard disk storage is very common in home PC and laptops. This increase in storage capacity is a result of human effort and great interest of people in this field. People are having interest in this field is because the magnetic hard disk are very cheap as compare to semiconductor storage devices. The large increase in magnetic storage density over the years can be summarized by the so called “Kryder’s Law”. According to this law the storage density is exponentially increasing over the years. This law is quite good from the last 30 years. Actually this kryders law is equivalent to the Moore’s law for data storage. But this kryders law may not be true in the future because the current magnetic data storage has matthe current magnetic data storage technology has been reached a fundamental limit. In order to improve the storage

density in the magnetic disk we have to go to smaller size magnetic element with large storage capacity. But when we go to very small size only a very small amount of is require to demagnetize it. If a small amount of thermal energy is sufficient to demagnetize that magnetic element. This phenomenon is known as superparamagnetism and at that much low dimension we cannot use those magnetic elements for data storage application.

The current magnetic technology is based on the longitudinal magnetic media where the magnetization lies in the plane of magnetic film and require lot of space to store one bit. Recently IBM did one research in which they magnetize 12 atom longitudinally to store one bit. But the new technology is based on the perpendicular recording or writing and bit patterned media. In this new technology we are supposed to go to very small size magnetic element by increasing the anisotropy energy of the thin film to magnetize them perpendicular to the plane. This greatly enhance the storage density. Longitudinal and perpendicular recording have their thermal stability limits at about 300GB/in^2 and 1Tb/in^2 respectively

In this project, we shall not be too ambitious. I will just fabricate the Cobalt iron and Cobalt iron boron thin films by electrodeposition technique having in plane magnetization and their surface characterization by using SEM, XRD, AFM and optical profile. Study dynamics of domains and domain walls by MFM, PPMS and effect on magnetic properties by adding Boron, thiourea.

1.2 Basic concept of magnetism in thin films

Element with different magnetic properties responds differently to an applied magnetic field. The hysteresis loop of different element is different. This hysteresis loop give the information about how the material is magnetized and demagnetize during the application of cyclic magnetic field. The shape of the hysteresis loop depends on many factors such as shape of the sample and the material itself well the direction of the applied magnetic field. Some important quantities are coercitivity Hc , the remanent magnetization Ms and the saturation field Hsat.

Remanent or retentivity is the remaining magnetization in the sample at H=0 as the applied field is going from saturation magnetic field to zero magnetic field. Coercitivity Hc is the negative applied field is required to reduce the magnetization from romance magnetization to zero magnetization. The saturation field is the required applied magnetic field to saturate the magnetization along the direction of the applied field. When we apply a magnetic field the

magnetic moments of the sample tries to orient in the direction of applied and when all the magnetic moment orient themselves in the direction of applied field that condition is known as saturation magnetization. This is solely a function of type of material and does not depend on geometry. The magnetization curves are usually measured by Physical Property Measurement System (PPMS) and Vibrating Sample magnetometer (VSM). We can calculate the hysteresis loop by theoretical calculation.

In this theory the main aim is to calculate the equilibrium magnetization configuration for the entire magnetic body in the presence of an external applied magnetic field. The general approach of the theory is to first compute the total energy of the system as a function of its magnetization configuration. Then one tries to minimize the total energy to find the stable equilibrium magnetization configuration in the presence of the applied field.

The total energy of a thin film can be written as a sum of energy from different contribution

$$E_{tot} = E_{ex} + E_H + E_{an} + E_m \qquad (1.1)$$

E_{ex} refers to the exchange energy contribution ε_H is Zeeman energy term, ε_{an} is the anisotropy energy and ε_m is the magnetostatic energy. All of these energy terms depend on the magnetization configuration in magnetic thin films.

The exchange energy originates from quantum interation there is no classical analog of this interaction. This is a very strong interaction between the spins that tries to align the neighbor spin to orient parallel. Hence when the spins are not aligned parallel in the same direction the exchange energy start to build up. The exchange energy can be written as

$$E_{ex} = -2 \sum_{<i,j>} J_{ij} S^2 \cos \varphi_{ij}$$

where J_{ij} is the exchange integral between spin i and j. this exchange integral is columbic in nature . S is the spin at each atomic site. The ϕ_{ij} is the angle between the adjacent spin. This means when ϕ_{ij} =0 the spins are aligned parallel and when ϕ_{ij} = 180 the spins are antiparallel. For ferromagnetic materials this exchange integral is positive.

Zeeman energy E_H describe the magnetic potential energy associated with the system when it is placed in the externally applied magnetic field. This energy is coming in the system because of

the particular orientation of magnetization with respect to applied magnetic field. This can be expressed as

$$E_H = -M \cdot H \quad (1.3)$$

Zeeman energy can be minimized when applied magnetic field and magnetization are parallel to each other.

The anisotropy energy E_{an} can arise from different sources. This term simply means that the magnetic properties depend on the direction in which they are measured. Anisotropy is exploited in the design of most magnetic materials of commercial importance. As a result the nature of the magnetic anisotropy is an important factor in determining the suitability of a magnetic material for a particular application

There are several kinds of anisotropy

Crystal anisotropy, formally called magneto crystalline anisotropy

Shape anisotropy

Stress anisotropy

Crystalline anisotropy

Crystalline anisotropy is intrinsic to the material; however, remaining are induced anisotropies.

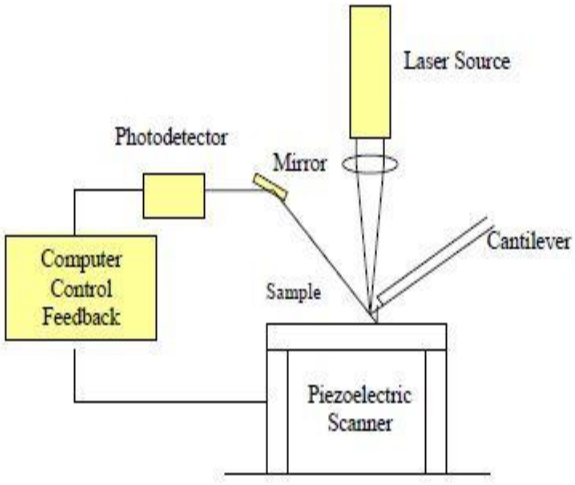
Now that we have seen some important energy terms that contribute to the total energy of the system.

1.3 Why Cobalt – Iron thin film?

Cobalt is the only alloying element that substantially increases the Curie temperature and the saturation magnetization of iron. The alloys from about 30 to 50% Co all have room temperature saturation magnetization about 10% higher than iron, and Curie temperatures limited by a bcc to fcc phase transformation just under 10008C. The 50–50 alloy, sold under various names, has low anisotropy and relatively high permeability, but quickly develops long-range order which makes it brittle. The addition of 2% V slows the ordering and allows the resulting alloy to be rolled into sheet form after rapid cooling from above the ordering temperature. Lower cobalt content alloys have less desirable soft magnetic properties, but cost less. The Fe–Co alloys are used where the highest saturation magnetization and/or a high Curie point is important: in the pole pieces of electromagnets, in beam-focusing lenses for electron microscopes, and in aircraft motors, generators, and transformers operating usually at 400 Hz

CHAPTER TWO
ABOUT INSTRUMENTS

2.1 Atomic Force Microscopy



AFM is a scanning probe technique which is used for the Nano scale measurement. The atomic force microscope (AFM was invented in 1986 by Binnig, Quate, and Gerber. Atomic force microscopy (AFM) provides a 3D profile of the surface at Nano scale, by measuring forces between a sharp tip(which is made up of antimony doped silicon) and surface which is at very short distance(0.2-10 nm tip-sample separation). The tip is mounted at the end of a flexible cantilever which is made up of silicon, which bends in respond to the force exerted on the tip by the sample The AFM tip “gently” touches the surface and records the small force between the probe and the surface.

Atomic force microscopy consist of two main modules and every module has its own work. The first module consist of the piezoelectric scanner that moves the sample in the X, Y, and Z directions. The second module is called the AFM detection system. This system includes a laser

source, a cantilever, a mirror, a photodiode, a photo detector, and computer control. The laser is focused at the back of the reflective cantilever. As the tip scans the surface of the sample, the laser beam is bounced off the cantilever into the photodiode. The difference in light intensities between the upper and lower photodiodes is sent to the photo detector, and the signal is then sent off to the computer control feedback loop. The feedback loop attempts to keep the cantilever deflection constant by maintaining a constant distance between the cantilever and the sample. This can be done by moving the scanner at each (X, Y) position in the Z direction, hence, adjusting the voltage applied to the scanner. The voltage, then, is converted to a cantilever deflection. The standard cantilever tips are typically Si_3N_4 or silicon.

The tip is placed at the end of the flexible cantilever. When the tip approaches toward the sample it experience the amount of force which is dependent on the spring constant (stiffness) of the cantilever and the distance between the tip and the sample. This force is expressed in term of hooks law

$F=-k.x$

F= Force

K= spring constant

X= cantilever deflection

If the spring constant of cantilever is less than surface stiffness the cantilever bends and then we can measure the deflection from cantilever.

This typically results in forces ranging from nN (10) to µN (10-6) in the open air.

What are tips and cantilever made of?

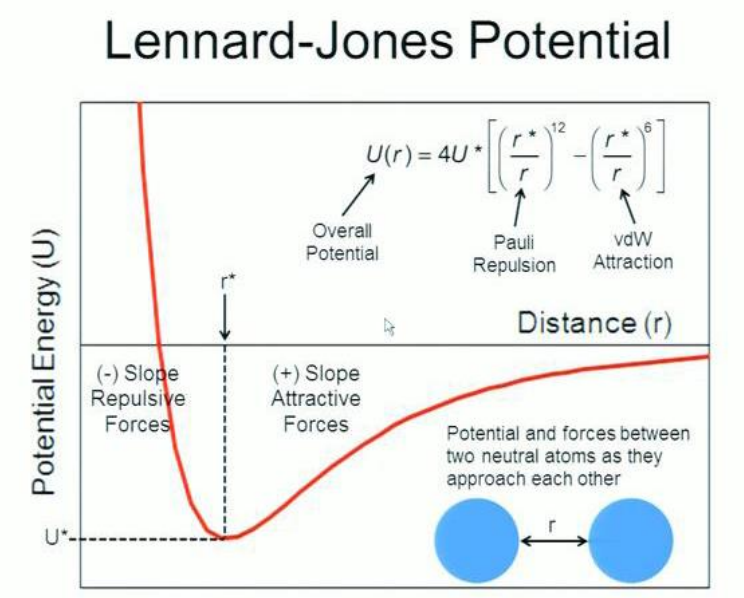
The probes that we are using (i.e tip and cantilever) are generally made from Si or Si3N4.The Tip is generally pyramidal and tetrahedral in shape Depending on the length, materials and shapes we can moderate the spring constants and the resonant frequency (the frequency at which the resonance condition is achieved). The geometry of the tip greatly affect the lateral resolution of the atomic force microscopy. The silicon can be doped and make conductive and allowing tip sample bias to be applied for electrical measurements. Silicon nitride tips are not conducting These tips can be coated with different materials for some other additional measurements like magnetic force microscopy (MFM) and chemical force microscopy (CFM).

The cantilever is a long beam with a tip is located at the end. IN AFM s, the motion of the tip is deflected by the reflecting laser.

For contact mode AFM the cantilever needs to deflect easily without damaging the sample surface or tip.
Therefore it should have a low spring constant, this is achieved by making it thin (0.3–2 μm).
It also needs a high resonant frequency to avoid vibrational instability, so is typically short (100–200 μm).
V-shaped cantilevers are often used for contact mode as these can provide low resistance to vertical deflection, whilst resisting lateral torsion.

FORCES MEASURED BY AFM

Tip and sample may experience many force depends upon their separation. Different forces will dominate over different distance. Some forces are attractive and some are repulsive like long range electrostatic, short range electrostatic, short range polarization Pauli repulsion, Vander Waals forces etc. When the distance between the tip and sample is very small in the AFM the interaction that plays a dominant role is Vander Waals force or interaction. This interaction measurement is heavily dependent on the geometry of the tip. There are many theoretical approaches or models that give the interaction picture. The model for the Vander Waals force (attractive force) and Pauli repulsion (repulsion at very short distance due to overlap of electrons clouds) is Lennard- Jones potential. The Vander Waals interaction can be felt by the tip and sample when the separation is 10nm. These force arise because of the temporary fluctuation of dipoles.



However long-range interactions (i.e. capillary, electrostatic, and magnetic) are significant further away from the surface. During contact with the sample, the probe predominately experiences *repulsive Van der Waals forces* (contact mode). This leads to the tip deflection described previously. As the tip moves further away from the surface *attractive Van der Waals forces* are dominant (non-contact mode).

OPERATING MODES OF AFM.

AFM can be operate in 3 primary modes

- Contact mode AFM
- Intermittent contact (tapping mode AFM)
- Non- Contact mode AFM

Contact mode AFM

In contact mode the tip of the AFM touches the sample surface through the adsorbed fluid layer. The detector monitors the changing cantilever deflection. In this mode the scanning speed is high. But the problem with this mode it can distort the soft samples and the effect of lateral and strong normal force may reduce the resolution of the image. In this mode the tip can also damage.

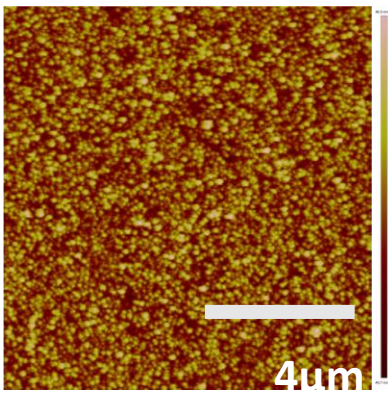
Intermittent contact (Tapping mode)

In tapping mode the cantilever oscillates at or slightly below resonant frequency. The amplitude of oscillation in tapping mode is vary from 20nm to 100 nm. In this mode tips lightly “taps” on the sample during scanning, contacting at the bottom and swing. Because the forces on the tip change as the tip-surface separation changes, the resonant frequency of the cantilever is dependent on this separation. In tapping mode lateral forces almost eliminated and the force is low so there is very less damage to soft samples. But the problem is low scan rate as compare to contact mode.

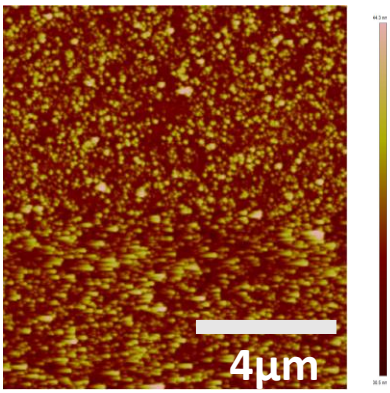
Non- Contact mode

. In a non –contact mode the cantilever oscillates near the surface of the sample but does not touches the surface of the sample. The oscillation of cantilever in this mode is slightly above the resonant frequency. In non-contact mode both normal and lateral forces are minimized so this mode is good for soft samples. Using this mode we can measure atomic resolution in ultra-high vacuum environment. But the problem with this modes is slow scan speed as compare to contact modes.

Co-Fe with solution Ph 3.0 (-1.1V, 250 s)



Co-Fe with solution Ph 3.0 (-1.2V, 100 s)



Here are the AFM images of cobalt iron thin film sample. The color contrast is changing depending on the distance between the sample and tip.

2.1 Magnetic Force microscopy (MFM)

The magnetic force microscopy is one of the most elegant method to study surface magnetic properties with very good resolution. The basic MFM principle is same as that of the AFM technique. We can say that MFM is AFM with the flexible cantilever is equipped with a magnetic tip or probe on its end. In MFM the measurement is coming because of the interaction of the magnetic tip and magnetic sample. When the tip approaches toward the magnetic sample and when the separation is nearly in the range of few Nano meter the cantilever start deflect because we know the magnetic forces are long range order and at a distance of few 100 nm it dominates over the short range forces like Vander Walls forces, Pauli repulsion etc. MFM can operated in non-contact mode and tapping mode.

MFM operational modes

The MFM is operating in two basic modes

1. Static (DC) mode
2. Dynamic (AC) mode

In a static mode the cantilever carries over the magnetic force incidence as a product of tip sample interaction

Where the deflection of cantilever is measured in terms of hooks law

$$\boldsymbol{F} = -k\,\Delta z$$

Where k is the spring constant of the cantilever

And Δz is the deflection in the cantilever because of the magnetic interaction of tip and sample.

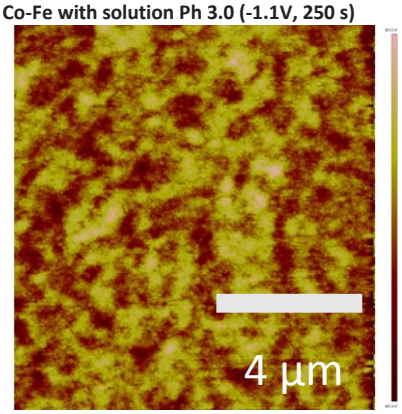
In Dynamic mode regime the cantilever can oscillate at or very close to its resonance frequency as we keep up. It is possible to represent a cantilever as a harmonic oscillator with a resonant frequency f

f=root (keff/m)*1/2pi

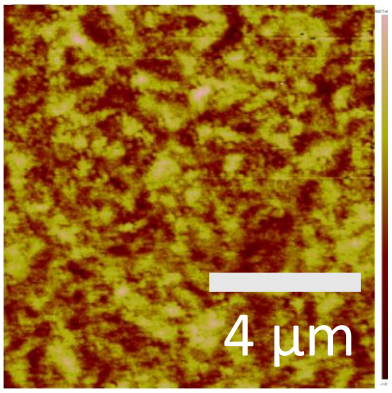
Where keff is the effective spring constant and m is the effective mass for the whole system i.e tip+ cantilever

Now the effective spring constant can be written in two components one is the spring constant of the cantilever and other is the force gradient with respect to the tip sample separation and from some we can find the equation of frequency of oscillation of cantilever.

Co-Fe with solution Ph 3.0 (-1.1V, 250 s)



Co-Fe with solution Ph 3.0 (-1.2V, 100 s)



2.2 SCANNING ELECTRON MICROSCOPE

A scanning electron microscope is a type of electron microscope that is used for observation of specimen surface. When the specimen is irradiated by the fine electron beam from the electron gun. These electron interacts with the atom of the sample and because of this interaction secondary electron are emitted from the specimen surface. These secondary electrons can be detected in the detector and topography image of the surface can be observed by two dimensional scanning of the electron probe over the surface.

Why images are visible?

When electron ejected from the electron gun and reaches toward the specimen or sample, the coming electrons enter in the specimen and scattered within the specimen and slowly lose their energy and adsorbed in the specimen this can be shown in figure. Depending on the energy of coming electrons inside the specimen decide the scattering range of electrons. The scattering range is also depends on the atomic number of the elements making up the specimen and the density of constituent atoms. If the energy is higher, the scattering range is large and if the atomic number is large the scattering range is smaller. This phenomenon can be understood by Monte Carlo simulation.

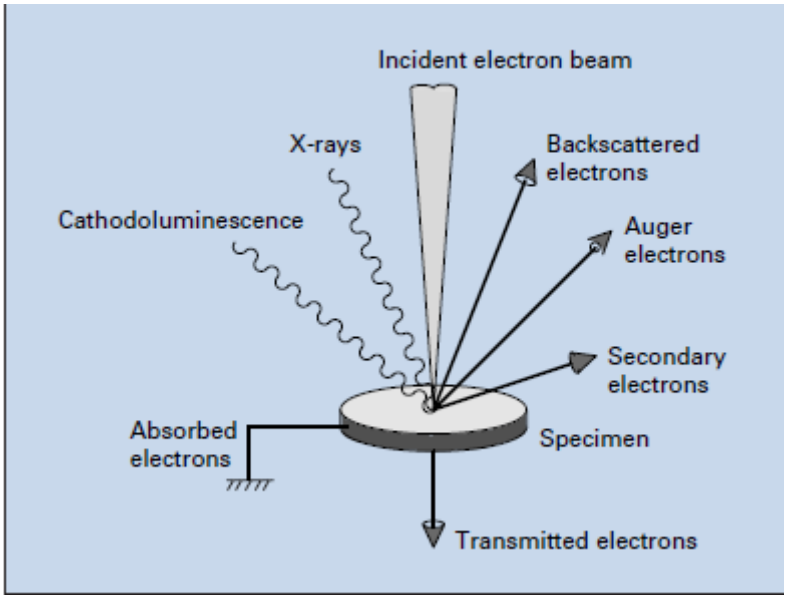


Figure : emission of various electrons and electromagnetic waves

Secondary Electrons

When the incident electron beam enters the specimen, the secondary electron are produced from the emission of the valence form the constituent atom in the specimen. Since the energy of the secondary electron is very small, those region are quickly adsorbed by the specimen. Only those generated at the top surface of the specimen are emitted out of the surface. This means these secondary electrons are very sensitive to the surface. When the incident electron beam is enters perpendicular to the specimen, the amount of emission of secondary electrons are larger when the incident at some obliquely. The difference in the brightness of the crystal structure is due to the difference of the incidence angle of the electron beam**.** Since the secondary electrons are very less energy it is influenced by the potential near the specimen surface. When the specimen is electrically charged and the secondary electron is often used to measure the operating voltage of a circuit in a semiconductor device.

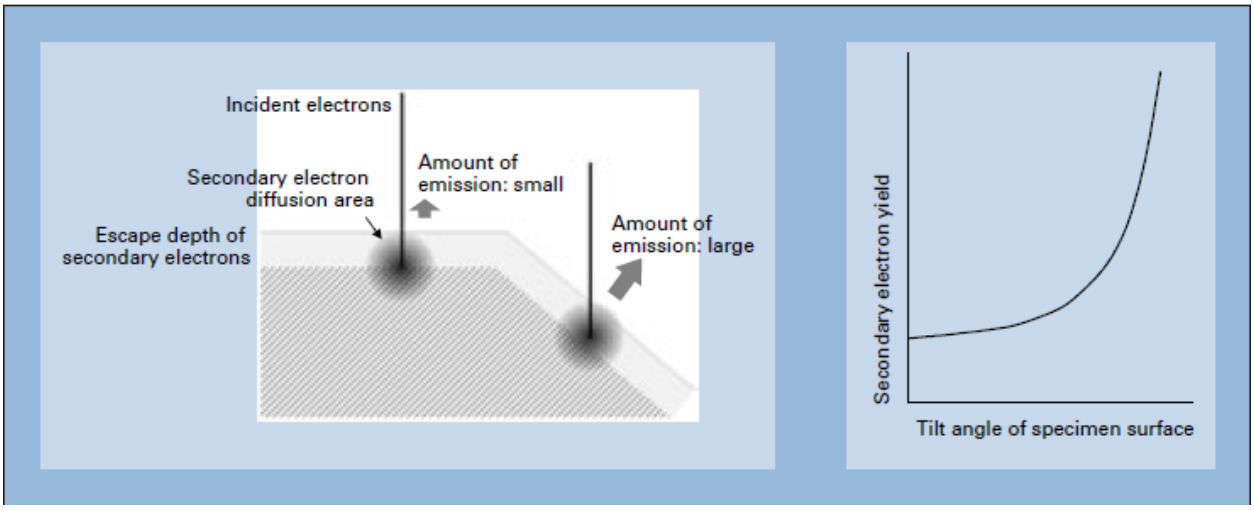
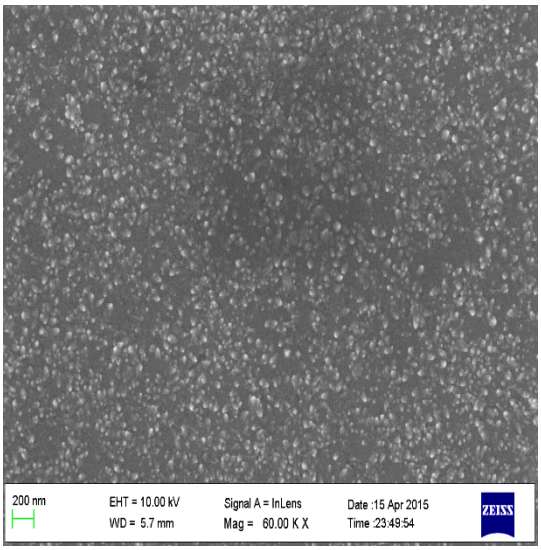
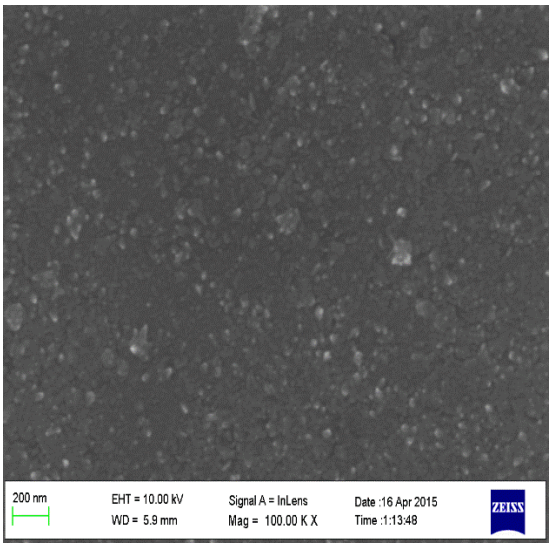


Figure : Relation between the incidence angle of the electron probe and the secondary electrons.

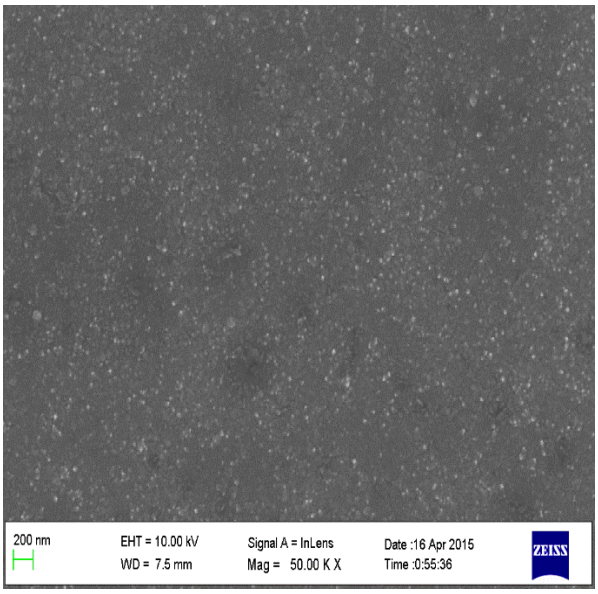
SEM Images of Cobalt Iron film



CoFe (-1.1V) 250 sec



CoFe(-1.15V) 100 sec

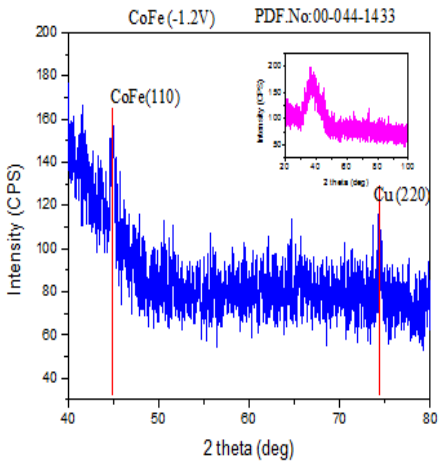


CoFe (-1.2V) 100S

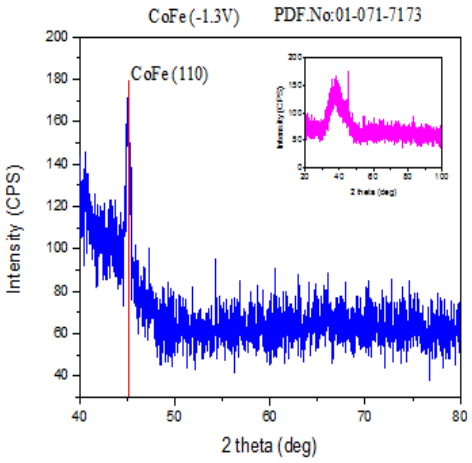
Here are the SEM images of Cobalt iron thin film sample which were deposited for different time and different potential. These images showing the size of nano granular are changing as the diposition time and diposition potential changes

2.3 Grazing Incidence X-ray Diffraction (GIXRD)

X- Rays technique have been used for decades to study the structure of bulk crystalline materials. They interact weakly with matter as compare to the electrons and thus are not normally thought as surface sensitive. As X-rays are weakly interacting with matter it can penetrate up to significant distances (typically of the order of .1 -10mm,) depend on the X- ray energy and the material on which they incident. Now a days a very intense source of X-rays are available and it is possible to obtain surface information by looking for the deviation from the bulk scattering. **In** other words, one scatters X-rays 'by brute force from the entire sample, and looks for the small part of the scattering that is due to the surface. This is done primarily when the bulk is a good single crystal, because then the scattering from it is largely limited to Bragg peaks. The excess scattering between Bragg peaks can then be attributed to a reconstructed surface or an adsorbed monolayer. This is the basic idea of 'truncation rod' analysis, so called because the scattering due to a surface is in the form of rods normal to the surface, rather than Bragg points, in reciprocal space (see later in the article). This method has been used extensively in the study of surfaces of inorganic single crystals, and of monolayers and thin films deposited on these crystals. Its disadvantages are that the substrate must be a good single crystal with little scattering between Bragg peaks, and the truncation rod profiles must generally be fitted using assumed models in order to extract information, as in the case of reflectivity.



CoFe(-1.2V,100sec)ph 3.0



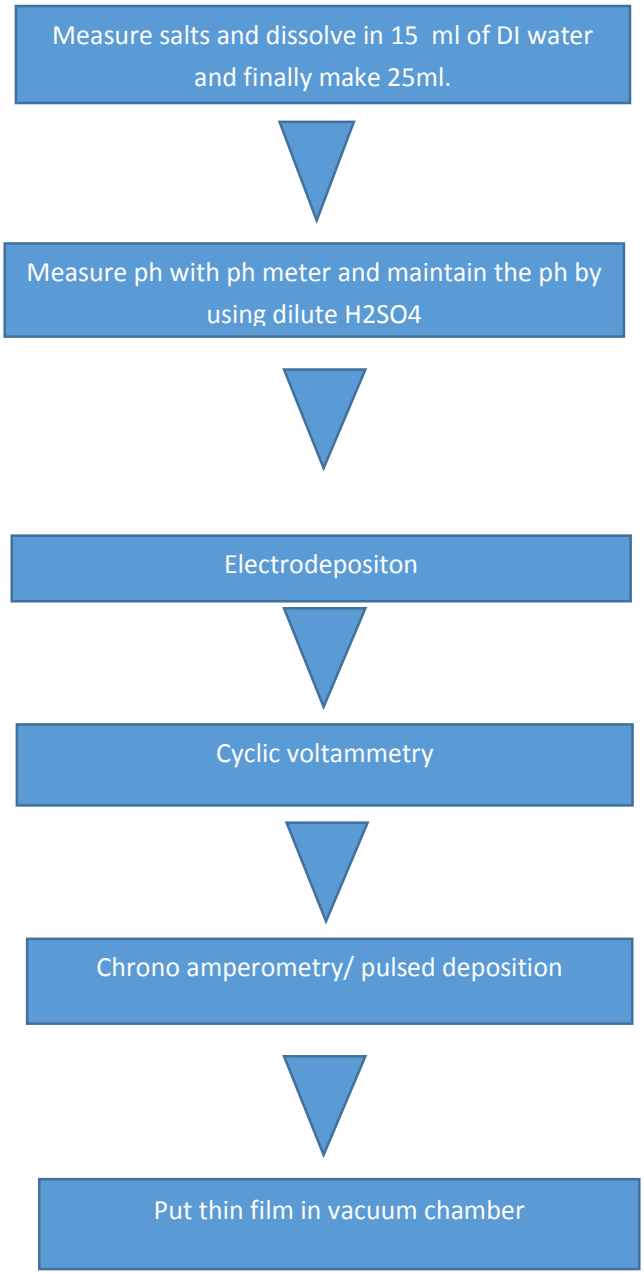
CoFe(-1.3V,50sec)Ph 2.4

CHAPTER 3
EXPERIMENT

3.1 Experiment

In this project I fabricate Co-Fe thin film by electrodeposition technique. The electrodeposition of Fe group metals and alloys are of great interest because of their very good magnetic properties. It has been already shown that the electrodeposited Fe – group alloy films show excellent soft magnetic properties. Co-Fe alloy system is one of them. Previous work shows that this Co-Fe alloys shows highest saturation magnetization of 2.45T and a very low coercitivity of about 2 Oe. And to meet the strong demand for high performance magnetic write- heads to increase the storage density this type of property is required. The previously developed CoNiFe alloys gives saturation magnetization of 2.0- 2.1 T and now a days this is in practical use. Here I am using this electrodiposition technique because this technique is very cheap as compare to other technique s like sputtering , pulsed laser deposition, chemical vapor deposition etc. And on the other hand this technique is useful as an industrial process and now a days the writing heads are prepared using electrodeposition technique.

Preparation of thin film



Chemical used

Here I prepared two solution of cobalt iron one with have ph 2.4 and other with ph 3.0 by adding these salts

The first solution is prepared by these salt and maintain ph 2.4 by the use of dilute sulphuric acid.

COMPOUND	CONCENTRATION
NH_4CL	.3M
H_3BO_3	.4M
$COS0_4.7H2O$	.05M
$FESO_4.7H20$	.04M
SLS	.0003M
SACCARINE	.004M

The second solution I prepared by using these chemicals and maintain the ph 2.4

compound	concentration
Sodium cirtate	0.3M
H_3BO_3	0.4M
$COS0_4.7H2O$	0.03M
$FESO_4.7H20$	0.04M
SLS	.0003M
Sodium sulphate	0.1M

3.2 Electrodeposition

Electro deposition process for producing a dense, uniform, and adherent coating of metal and alloys, upon a surface by the act of electric current. Electroplated products are widely used in industries, such as automobile, ship, air space, machinery, electronics, jewelry, defense, and toy industries. Here I am using electro-deposition technique to grow Thin Films of CoFe alloys on Copper sputtered Si(100) with adhesive of Ti(20nm) using this technique we can control the thickness of deposition by controlling the time of deposition.

Working principle:

Electrolyte is the electrical conductor which carried current between the two electrodes. Depending on the application of current in the system the positive and negative ions in the electrolyte move toward cathode and anode. This migration of ions through the electrolyte constitutes the electric current in that part of the circuit.

The metallic ions of the salt in the electrolyte carry a positive charge and are thus attracted to the cathode. When they reach the negatively charged work piece, it provides electrons to reduce those positively charged ions to metallic form, and then the metal atoms will be deposited onto the surface of the negatively charged work piece.

Oxidation/Reduction

All electron transfer reaction can be understood by electron transfer reactions known as oxidation and reduction. In oxidation/reduction reaction the substance which take electrons known as oxidant (or oxidizing agent) and the substance which loosing electrons known as reductant (or reducing agent). In electrolysis cathode is attached to the negative pole of the electric source, it supplies electrons to the electrolyte. On the contrary, an anode is connected to the positive pole of the electric source; therefore, it accepts electrons from the electrolyte. Various reactions take place at the electrodes during electrolysis. In general, reduction takes place at the cathode, and oxidation takes place at the anode.

Anode and Cathode Reactions

Electrodeposition or electrochemical deposition (of metals or alloys) involves the reduction of metal ions from electrolytes. At the cathode, electrons are supplied to cations, which migrate to the anode. In its simplest form, the reaction in aqueous medium at the cathode follows the equation:

$Co^{+2} + 2e^{-} = Co$

$Fe^{+2} + 2e^{-} = Fe$

with a corresponding anode reaction. At the anode, electrons are supplied to the anions, which migrate to the anode. The anode material can be either a sacrificial anode or an inert anode. I am using inert electrode.

SURFACE PREPARATION

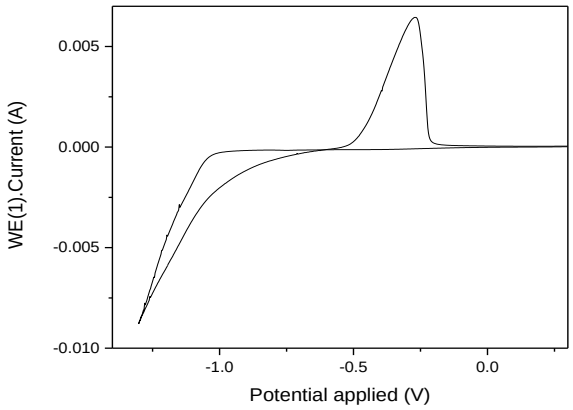
Before deposition of film on the substrate we have to go through following procedure surface cleaning of substrate. Surface cleaning is one of the most important part of deposition of thin films. The purpose of surface cleaning is to remove the contaminants such as dust and the films from the substrate. The surface contamination can be extrinsic, composed of organic debris and mineral dust from the environment or preceding processes. It can also be intrinsic, such as a native oxide layer. Contaminants and films interfere with bonding, which can cause poor adhesion and even prevent deposition

Surface Cleaning

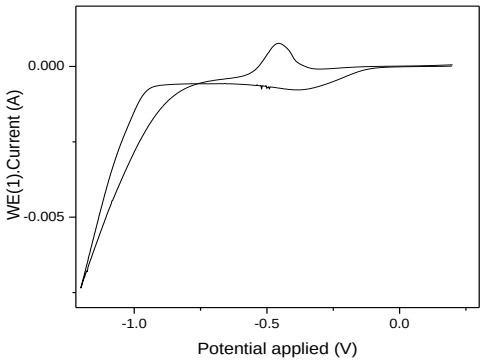
I used chemical approach, in this method I put my substrate into the bath of very dilute H2SO4 for 5 mins and then I washed with DI , IPA , Acetone sequently and then finally used for growing film of CoFe.

3.2 Cyclic voltammetry

Cyclic voltammetry became a popular tool to studying electrochemical reaction from last fifteen-twenty year. Cyclic voltammetry is a type of potentiometric electrodeposition. In cyclic voltammetry experiment the working electrode potential linearly changed with time. In cyclic voltammetry the working electrode potential will go to opposite potential and again return back to initial potential and make a cyclic loop between working electrode current and potential. This cyclic loop is called cyclic voltammogram trace.



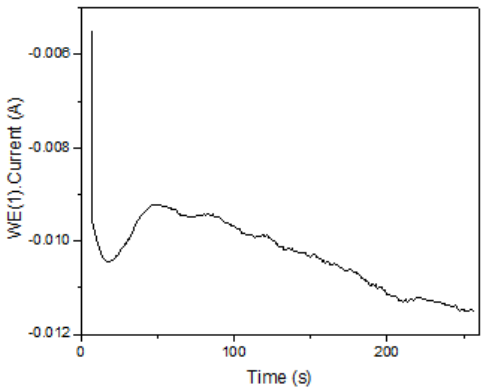
Cyclic voltammetry of Co-Fe solution having ph2.4



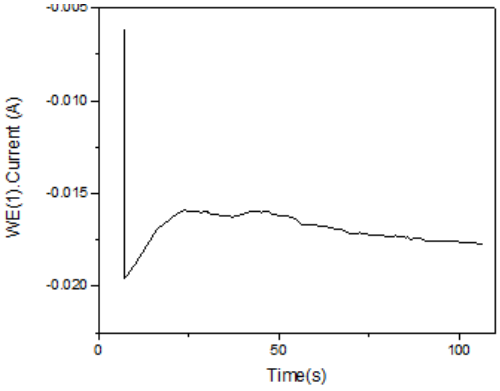
Cyclic voltammetry of Co-Fe solution having Ph 3.0

3.3 Chronoamperometry

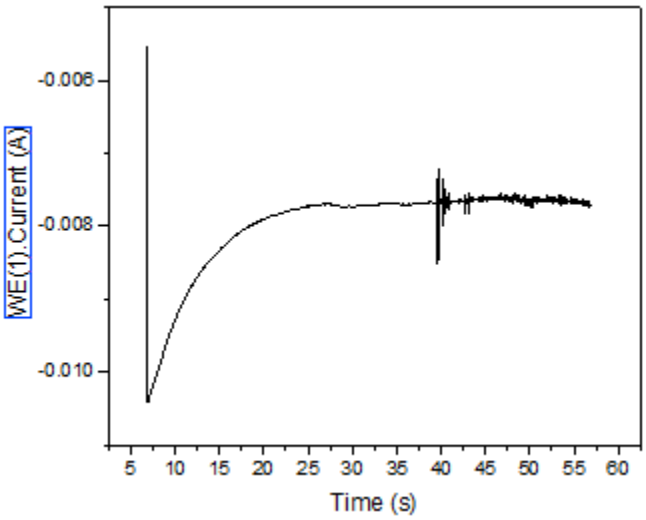
Chronoamperometry is an electrochemical technique in which the potential of the working electrode is stepped and the resulting current from faradaic processes occurring at the electrode (caused by the potential step) is monitored as a function of time.



Cobalt iron (-1.1V, 100sec) ph3.0



cobalt iron (-1.2V, 100 sec) ph3.0

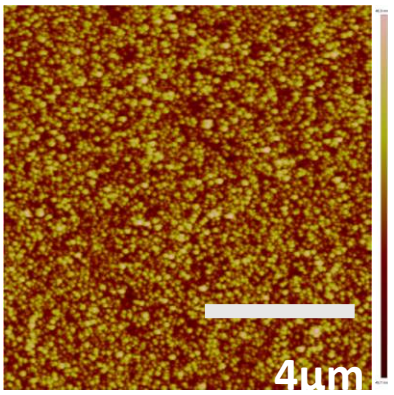


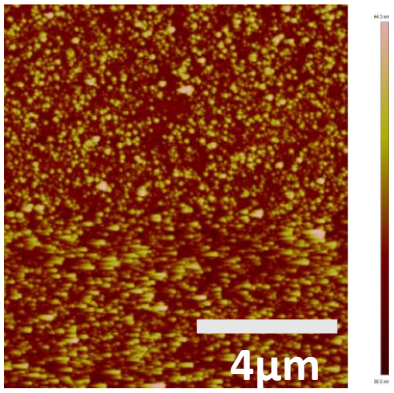
Cobalt iron thin film (-1.3,50 sec) ph 2.4

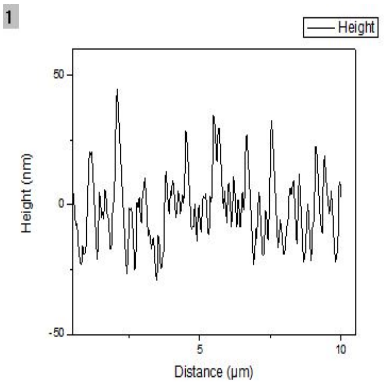
CHAPTER 4
RESULT AND DISCUSSION

4.1 Atomic force microscopy and magnetic force microscopy images

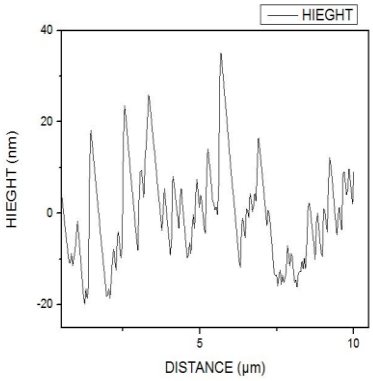
1.1.1 Atomic force microscopy



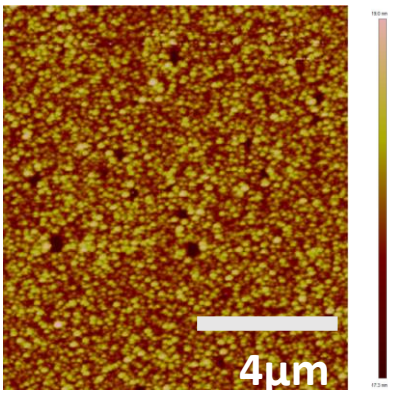


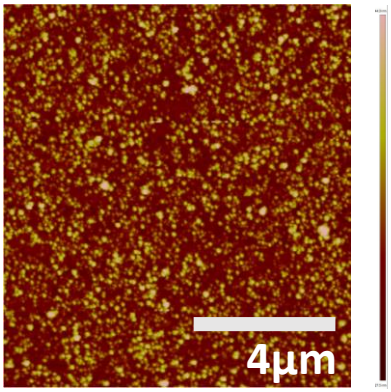


Co-Fe with solution Ph 3.0 (-1.1V, 250 s)



Co-Fe with solution Ph 3.0 (-1.2V, 100 s)





Co-Fe with solution Ph 2.4 (-1.3V, 50s) Co-Fe with solution Ph 3.0 (-1.3V, 50s)

Here we are showing the AFM image of cobalt iron film deposited at different potential , time duration and the Ph. this is a scan over the range of 10 micrometer at a lift hieght of 20nm. Picture 1 is AFM image of lthe Cobalt iron film sample which was deposited for 250 second at voltage -1.1 V. In picture 1 the showing the rougness of 12.3nm and the graph is showing how the particles are distributed over the area under 10 micrometer. Depending on the distribution of particles on the surface of the film they interact with interact with the AFM. Because of this interaction the cantilever bend and this bending is dectected on the detector.

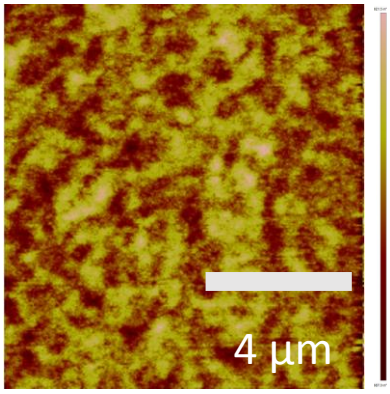
In the second image the scan is over the range of 10 micrometer of the sample and it is showing somewhat non uniformly distribution of particle on the surface on thin film. this film is deposited for 100 second at potential -1.2V. The Ph for this solution is 3.0. the measured roughness is 11.1 nm. Depending on the distribution of particles on the surface of the film they interact with interact with the AFM. Because of this interaction the cantilever bend and this bending is dectected on the detector.

Third image is a sample of Cobalt iron which was prepared with a solution of ph 2.4 at potential -1.3V, 50 sec. This is showing good uniform distribution as compare to other films. And the average roughness in this film was measured 5.30 nm.

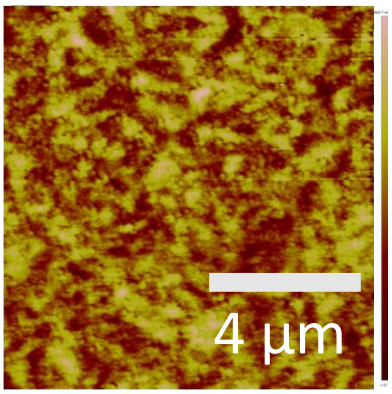
Forth image is a sample of Cobalt iron which was prepared with a solution of ph 3.0 at potential -1.3V, 50 sec. this is not showing that much good diposition. Some of the part is not deposited.

	ph	Voltage	Time	Scan range	Rougness
CoFe	3.0	-1.1	250	10µm	12.3nm
CoFe	3.0	-1.2	100	10 µm	11.1nm
CoFe	3.0	-1.3	50	10 µm	
CoFe	2.4	-1.3	50	10 µm	5.30nm

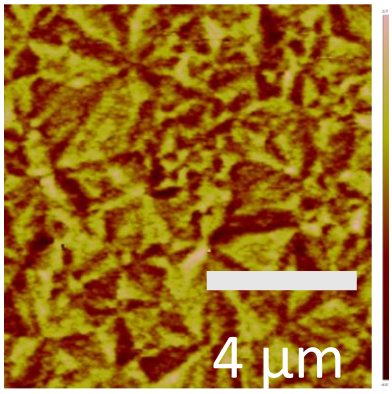
Magnetic phase



Co-Fe with solution Ph 3.0 (-1.1V, 250 s)



Co-Fe with solution Ph 3.0 (-1.2V, 100 s)

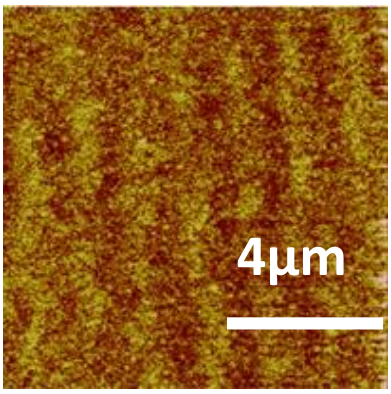


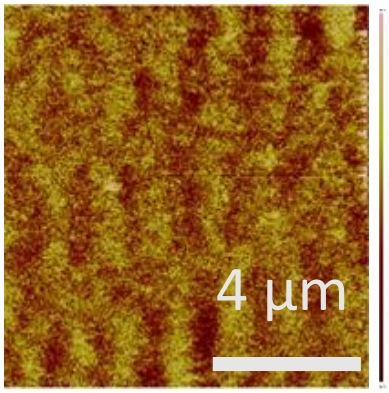
Co-Fe with solution Ph 2.4 (-1.3V, 50s)

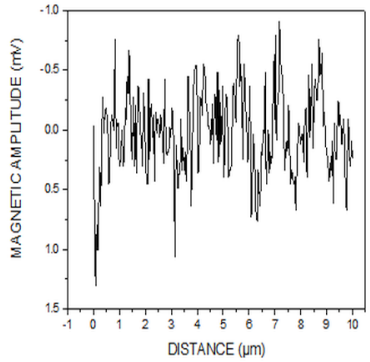
These images are the magnetic phase of different Cobalt iron thin film samples over the range of scan of 10 micron at lift height of 20 and 30nm. As we see the roughness of the cobalt iron thin film which was deposited from solution having ph 2.4. is very low as compare to other sample

which were deposited from solution having ph 3.0. the roughness sample which are deposited from solution ph 3.0 are in the range from 10 nm to 12.1 nm at a lift height of 20nm. Because of this high roughness in these thin film the magnetic contrast is not very good and the domain structure is not that much clear. But the CoFe thin film which is deposited at potential -.1.3, 50sec is showing very good domain as compare to the other film . Third image is showing very good magnetic domain.

Magnetic Amplitude





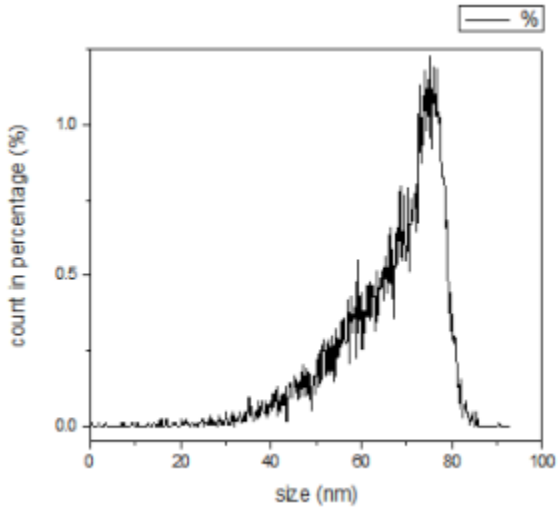


Co-Fe with solution Ph 3.0 (-1.1V, 250s)

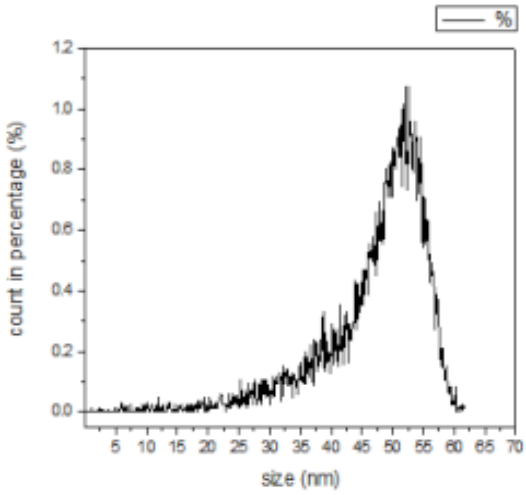
Magnitude amplitude images gives the information about the magnetic interation at a particular region during the scan of 10 micrometer. Here we can see in graph that the magntitude amplitude is fluctuating from positive value to negative values. Because the surface is having very high roughness this magnetic amplitude curve is not matches exactly with the afm curve.

Depth histogram

Co-Fe with solution Ph 3.0 (-1.2V, 100s)



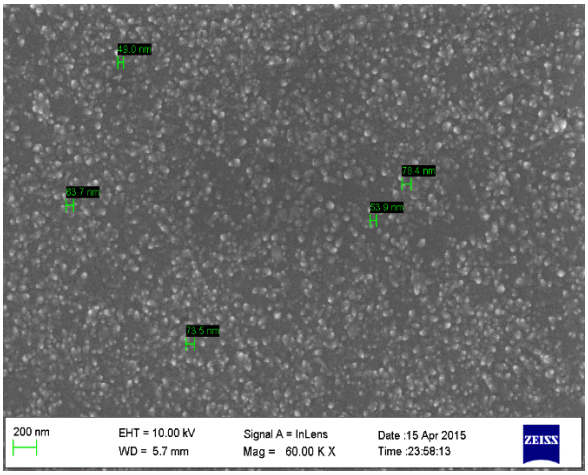
Co-Fe with solution Ph 3.0 (-1.1V,250s)



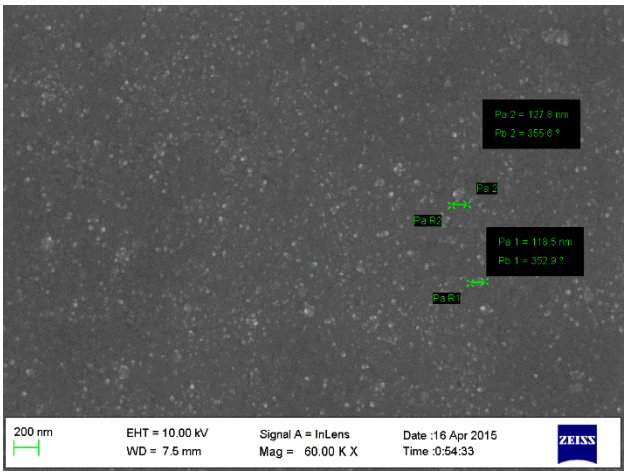
This depth histrogram give the information about the size of the nana granuluar on the thin films. From the graph we can see that the film which is deposited at potential -1.2V, 100seconds having sizes of particle or nano granuluar within the scan area is lying in the range from 70 to 75 nm and the film which was deposited at potential -1.1V, 250 seconds having mostly particles size lies in the range of 50 to 55 nm.

1.2 Scanning electron microscope images

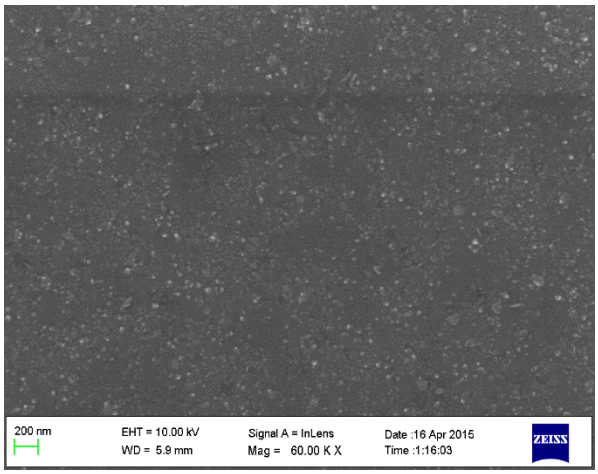
Here are the SEM images of cobalt iron thin film.



Co-Fe with solution Ph 3.0 (-1.1V, 250 sec)



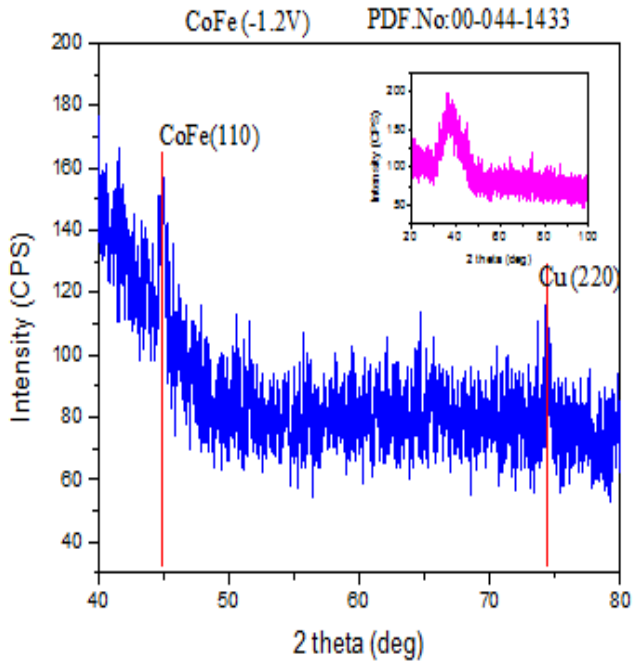
Co-Fe with solution Ph 3.0 (-1.2V, 100s)



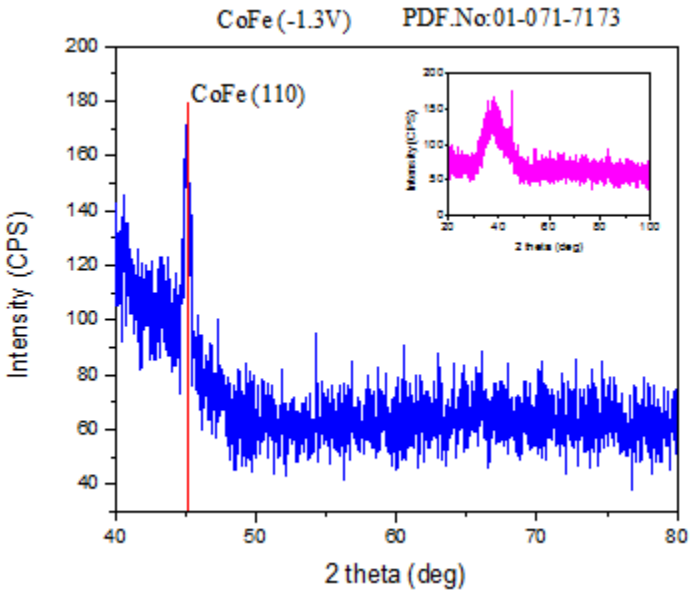
Co-Fe with solution Ph 3.0 (-1.15V, 100sec)

As we know that SEM is used for the analysis of topography image of samples. Here what we are seeing is that Cobalt iron thin film clearly exhibiting nano crystalline nature and Nano granular size is varying as we vary the deposition voltage. The cobalt iron film which is deposited at potential -1.1V have on an average granular size is vary from 50 nm to 75 nm and the Cobalt iron film which is deposited at potential -1.2V, 100 seconds some of the granular size is above 120nm.

4.3 GI-XRD Analysis



GI- XRD OF Co-Fe(-1.2V, 100sec)



GI- XRD OF Co-Fe (-1.3V, 50sec)

x- ray diffraction technique is used to determine the structure of the sample. Here we are showing thin film diffraction showing that 2theta for the first film is 44.89 degree and for the

second is 45.16 degree. As we match these result from the already known result of ICDD pdf number 0-0-049-1568 and for the second film 00-048-1818. These result shows that our thin film is having BCC structure. The first film which is deposited from a solution having ph 3.0 at potential -1.2V, 100 seconds having cobalt concentration 51.34 and iron concentration 48.66. But the second film which is deposited from the solution having ph 2.4 showing cobalt concentration 70 and iron concentration is 30.

REFERANCES

- Yang Junhua, COBALT NANOWIRE ARRAY: SYNTHESIS AND MAGNETIZATION BEHAVIOR, March 2006
- The magnetic force microscopy and its capability for nanomagnetic studies - The short compendium, A. Hendrych*,1, R. Kubínek1 and A. V. Zhukov2, 2007
- Introduction to Scanning Probe Microscopy (SPM), Robert A. Wilson and Heather A. Bullen,* Department of Chemistry, Northern Kentucky University, Highland Heights.
- A High Moment CoFe Soft Magnetic Thin Film Prepared by Electrodeposition, Tetsuya Osaka,*,z Tokihiko Yokoshima, Daizo Shiga, Kenta Imai, and Kyoko Takashima, 2003.
- Electrocrystallisation of CoFe alloys under the influence of external homogeneous magnetic fields—Properties of deposited thin film, Jakub Adam Koza∗, Franziska Karnbach, Margitta Uhlemann, Jeffrey McCord, Christine Mickel, Annett Gebert, Stefan Baunack, Ludwig Schultz, 2010.
- Electroplating, Helen H. Lou Department of Chemical Engineering, Lamar University, Yinlun Huang Department of Chemical Engineering and Materials Science, Wayne State University,
- Electrodeposited Iron Group Thin-Film Alloys Structure-Property Relationships, N. V. Myung*,z and K. Nobe*, 2001.
- GRAZING INCIDENCE X-RAY DIFFRACTION CHARACTERIZATIONOF CORROSION DEPOSITS INDUCED BY CARBON DIOXIDE ON MILD STEEL, S Sembiring, B O'Connor, D Li, A van Riessen, C Buckley and I Low, Materials Research, 2000.
- Electrodeposited Iron Group Thin-Film Alloys Structure-Property Relationship,N. V.Myung*,z and K. Nobe*2001.