Fabrication and Characterization of high density vertically electrodeposited ferromagnetic Nanostructures in AAO Templates

International Islamic University Islamabad, Pakistan

Faculty of Basic and Applied Sciences

Department of Physics (Nanotechnology)

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Supervisor: DrNaeem Ahmad

Assistant Professor at FBAS (IIUI)

Nanostructure

Nanotechnology

Fabrication and Characterization of High Density Vertically Electrodeposited Ferromagnetic Nanostructures in AAO Templates

BY

Ahmad Saeed

(269-FBAS/MSPHY/S14)

This thesis is submitted to department of physics International Islamic University Islamabad for the award of degree of MS Physics

2.8.16

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Department of Physics

Final Approval

It is certified that the work presented in this thesis entitled "**Fabrication and Characterization of high density vertically electrodeposited ferromagnetic Nanostructures in AAO Templates**" by **Ahmad Saeed**

Registration No **269-FBAS/MSPHY/S14** is of sufficient standard in scope and quality for the award of degree of MS (Physics) from International Islamic University, Islamabad

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Dated: 01-07-2016

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MS THESIS COMPLETION CERTIFICATE

Mr **Ahmad Saeed** Registration No **269-FBAS/MSPHY/S14** student of Master of Science has submitting his research thesis titled "FABRICATION AND CHARACTERIZATION OF HIGH DENSITY VERTICALLY ELECTRODEPOSITED FERROMAGNETIC NANO STRUCTURES IN AAO TEMPLATES ."
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DEDICATED
To
All those great persons of this beauteous land "PAKISTAN", who sacrifices their lives, future and hard work for our better tomorrow

Acknowledgements

I would like to quote that this work would not have been possible without the support of some people who were always there to help and support me The very first I would like to thank my supervisor Professor **Dr. Naeem Ahmad**, for his help, support generosity and the worthy ideas for the success of this work I would also like to thank the HOD of the department of physics, IIUI, **Dr. Waqar Adil Syed** and other faculty members for their valuable discussions and suggestions on my research work I would like to thank my friends and colleagues at Spintronic lab, IIUI, for their help and inspiration I thank **Mr. Fahad Hassan**, **Mr. Usama Bashir**, Mr Muhammad Suleman and **Mr. Affan safeer** for their valuable advices and huge support on my research Furthermore, I would like to thank my family and friends for their immeasurable support Thank all of you, this MS would not have been possible without you

The Spintronics Lab acknowledges the Institute of Physics, Chinese Academy of Sciences (CAS), China, for providing characterization facilities like VSM FMR and SQUID

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Chapter 1

Introduction to nanoscience & technology

1.1 Study at nano level scale

In nano science and technology all the studies and experimentations are done at atomic level The scale of nano can be understood by many ways From a very common definition it is the one billionth part of a meter [1-2] On another way one can understand it in a good way, just by simply thinking about a single hydrogen atom whose atomic radius is experimentally understood that is 0 053 nm Now if we place five (05) hydrogen atoms together in a line then the length of this line will be referred to 1 nm almost as shown in figure 1

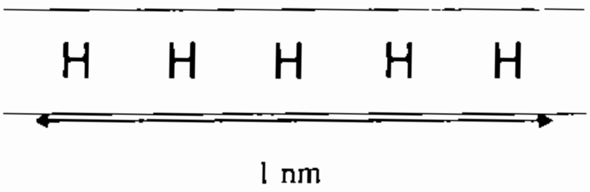

Figure 1 1 Five H atom in a row makes the length of 1 nm

In this way we can say that it is the study of matter at atomic level where the simple Newton laws does not hold The quantum mechanics rules play a vital role in the study of matter at nano level There are different nano structures of different dimensions which are fabricated and characterized by the nano scientist for the betterment of human s life and their needs Scientists and engineers presently working on many nano structures but the ferromagnetic nano structures and semiconductors are most prominent for different data storage devices and energy storage devices respectively

1.2 High surface to volume ratio

As compared to bulk material high surface to volume ratio of nanomaterials is also very important regarding their chemical reactivity This is because of the availability of greater number of atoms present at the surface of the nanomaterials Actually the atoms are unexposed at bulk level specially those atoms which are inside the material but at nano level maximum atoms have opportunity to react because of larger surface area the atoms inside the material are now lacking the covering which they have in bulk form The whole phenomenon [3] is shown in figure 2

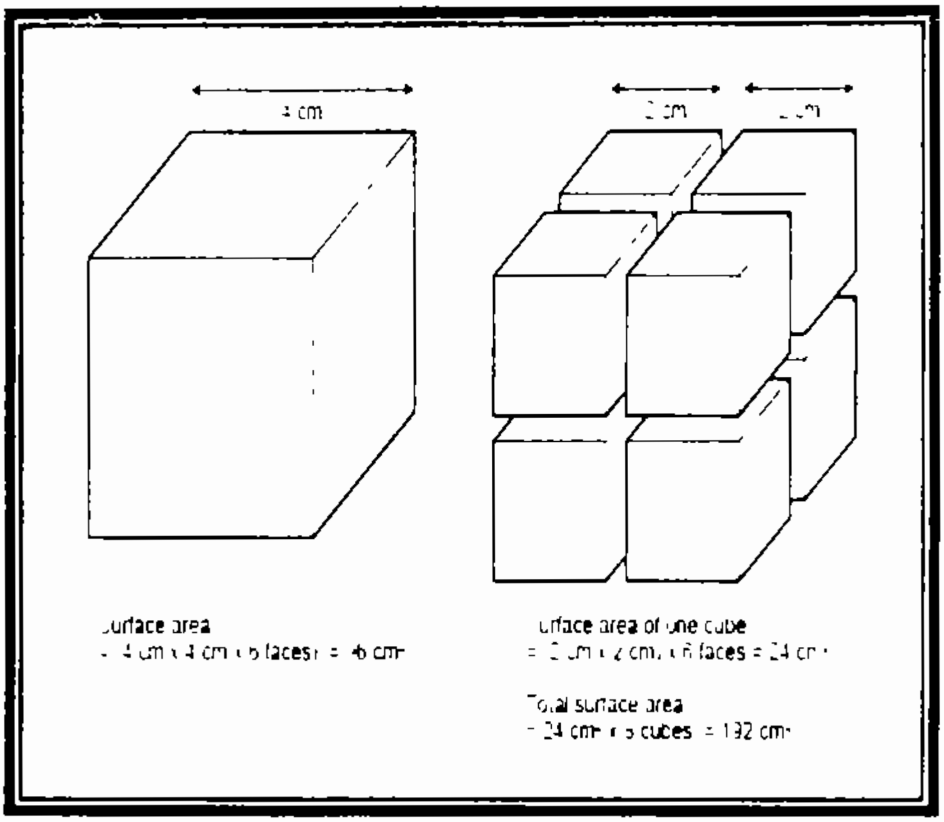

Figure 1 2 Basic comparison of surface area between bulk and nano material

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All those materials whose one dimension is at least ranges from one nanometer to hundred nanometers are called nano materials and the science and technology concerned with them is called nano science and technology Such materials are fabricated synthesis or manufactured by two different approaches usually known as top down and bottom up approach

1.3 Surface energy

Surface energy is almost directly proportional to the surface area and the materials surface area depends upon the dimensionality (at nano level) of the material The surface area becomes prominent at nano dimensions that intern increase the surface energy as well This is actually because of more number of atoms exposed on the surface of atoms because of nano dimensions This phenomena explained by the figure 1 3 given below [4]

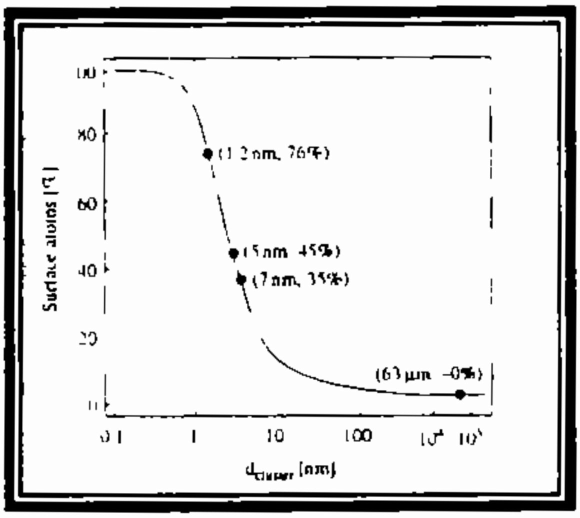

Figure 1.3 The percentage of surface atoms changes with the palladium cluster diameter

The graph shown in the figure 03 illustrates that when the size of the particle squeezes to nano range from um scale then a vast increase in percentage of atoms exposed and the surface energy become larger to the extent of seven orders of the magnitude

In case of singular surface as shown in figure 04, to which we want to subdivide into two surfaces we need the following amount of energy

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Where, N_b is the number of broken bonds, ρa the atomic density of atoms per surface and ϵ the bond strength of the material

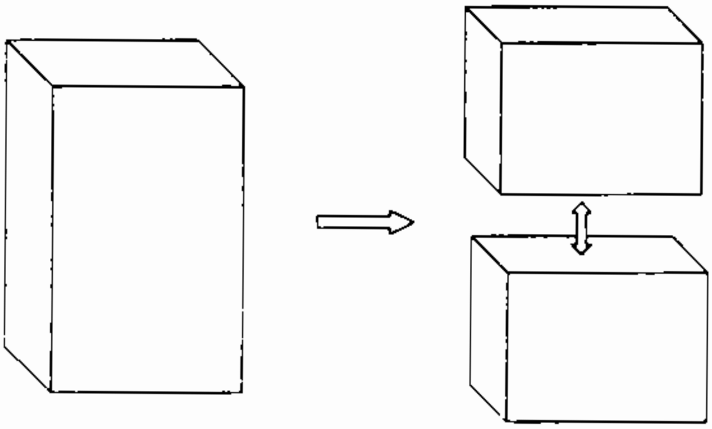
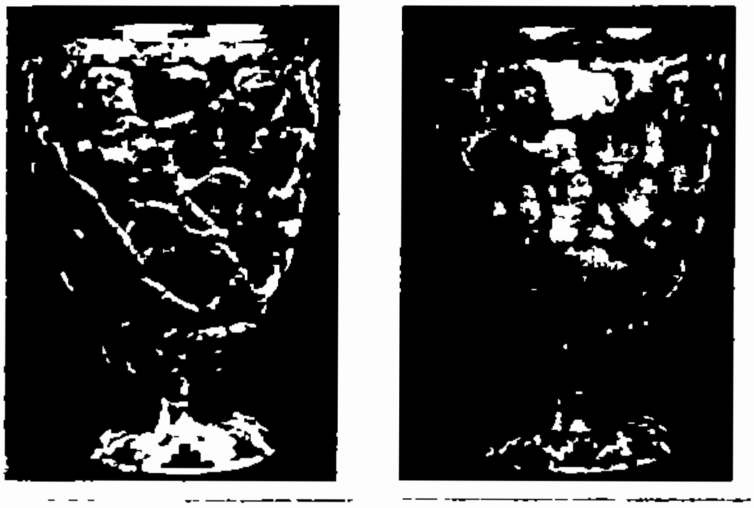


Figure 1.4 Schematic showing two new surfaces being created by breaking a rectangular solid into two pieces

1.4 Review of nano milestones

According to history point of view of nanotechnology there are many milestones having great worth Everyone have a simple question in their mind that how log nanotechnology been utilized?

If we look back to the thousands of years then we will come to know that pottery is one of the major branch of industry that unconsciously using the nano particles Such type of pottery object was Lycurgus chalice in the late fourth century AD and right now this nano based oldest chalice can be seen in the British museum This Roman chalice was famous because of its two colors green and translucent red The appearance of green color was due to reflectance of light and when the chalice was directly focused by light it appears translucent red Romans also attributed to these colors with their ancestors heroes who worked well in the eye of peoples Actually these colors are due to the 70 nm silver and gold particles [4]



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The nanotechnology now-a-days is because of the hard work of the scientists in scientific research that has been going on for over the centuries The famous scientists who attract the attention while reviewing the nanotechnology milestones because of their nice scientific contributions are enlisted below with their discoveries and inventions

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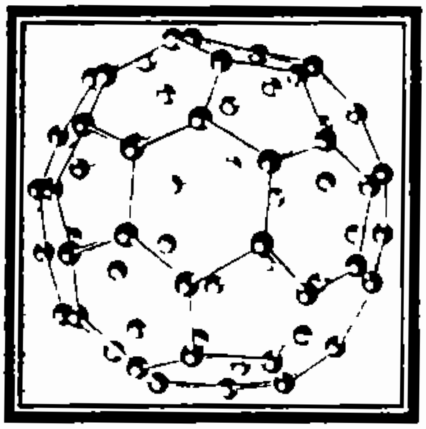
- A new type of carbon just like a spherical football made of 12 pentagons or 20 hexagons panels Smalley Robert Curl and Harold Kroto discovered the C-60 It is also known as buckminsterfullerene The aforementioned scientists were awarded with the Nobel Prize in 1996



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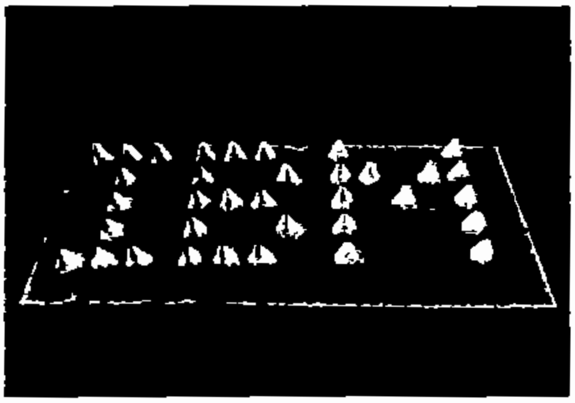

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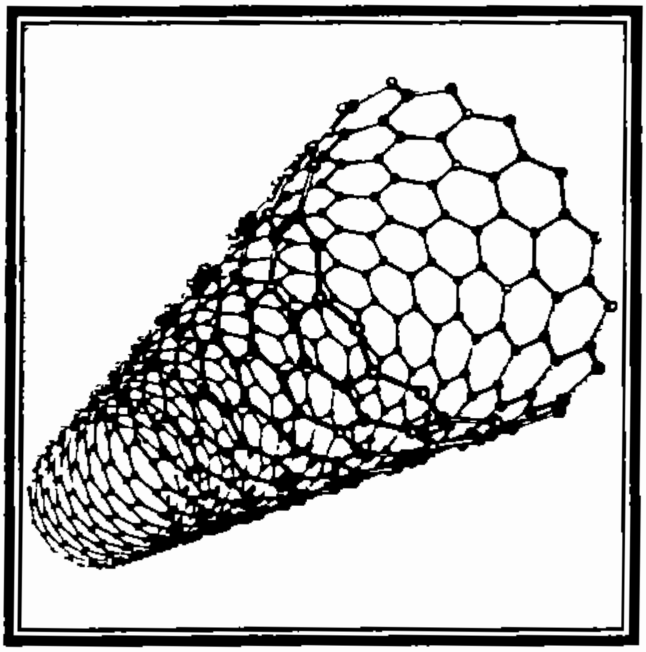
  - The great man Sumino Iijima discovered a process to fabricate the one dimensional carbon nano structures called **nanotubes.** The length of these structures was about one micron and the diameter range is 4 nm to 30 nm [6] These tubes are made of carbon atoms sheets which further rolled down to make first single walled nano tube (SWNT) in 1993 as shown in the figure below



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1.5 Classification of materials at nano scale

When we look towards the definition of nano science and technology then we understands that it is very rich in research that explains the new horizons and diverse characteristics of the materials In fact the reason is that it surrounds the very broad spectrum of research fields of different materials These materials can be any but now-a-days the following materials are on top regarding research and application point of view [10]

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In this thesis $Ni_{76}Fe_{24}$ Nanowires and $Ni_{77}Fe_{23}$ Nanotubes were deposited inside the home made anodized aluminum oxide (AAO) templates. The process of deposition was carried out by DC electrodeposition method. The composition analysis and surface morphology were explored by energy dispersive spectroscopy (EDS) and scanning electron microscopy (SEM) respectively. Scanning electron microscopy proves that the sample prepared well in desired manner. Very fine and homogeneous growth of $Ni_{76}Fe_{24}$ Nanowires and $Ni_{77}Fe_{23}$ Nanotubes was confirmed. Furthermore SEM calculation proves average diameter (D) of the$Ni_{76}Fe_{24}$ nanowires is 50 nm and length (L) of the $Ni_{76}Fe_{24}$ nanowires is 10 um. The average diameter (D) of the $Ni_{77}Fe_{23}$ nanotubes is 200 nm and length (L) of the same $Ni_{77}Fe_{23}$ nanotubes is 10μm. In order to examine the magnetic properties of the sample M-H curves were obtained by vibrating sample magnetometer (VSM) at different angles between the wire axis and external magnetic field. M-H curves prove that both nanowires and nanotubes have easy axis along the perpendicular of their axis. To some extent shape anisotropic behavior was also confirmed by M-H curves. After this we take these curves at low temperature T=5K and found that thermal energy decreases at low temperature causing an increase in saturation magnetization (Ms). Further we employed FMR to study some others magnetic parameters. With the help of FMR we obtained intensity vs H field curves and resonance vs angle curves and found out the resonance line width and easy axis as well. Results taken from these curves are consistent with the M-H curves. Finally we draw the graph of delta M curves for both nanowires and nanotubes. Magnetostatic interactions are very weak inside the $Ni_{77}Fe_{23}$ Nanotubes but on the contrary magnetostatic interactions or dipolar interactions between the $Ni_{76}Fe_{24}$ Nanowires are very strong.

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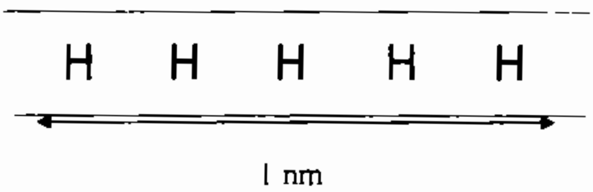

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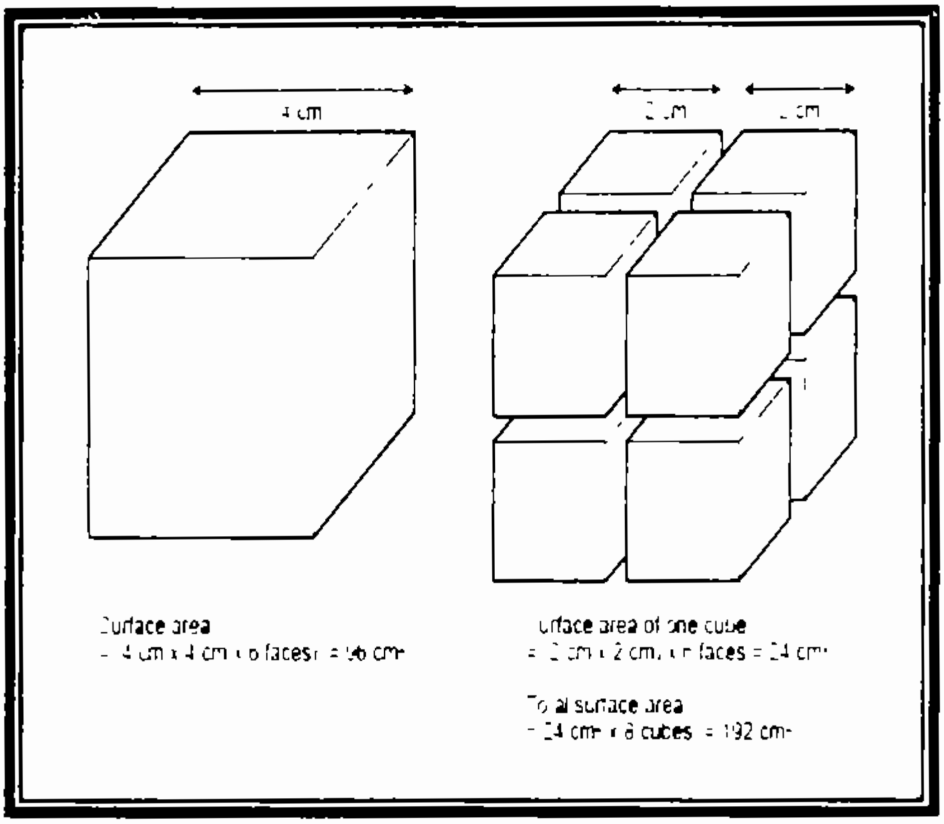

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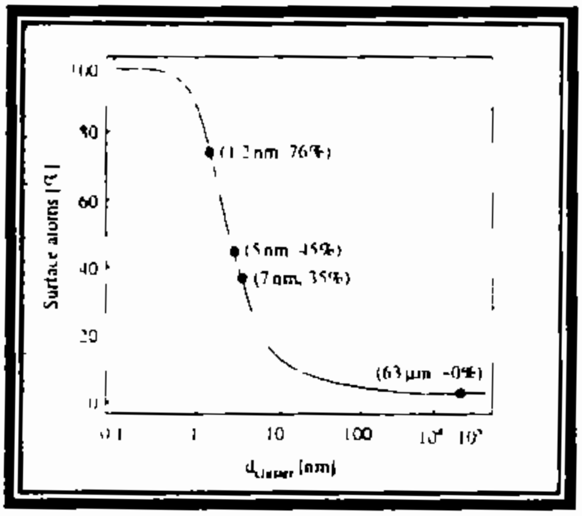

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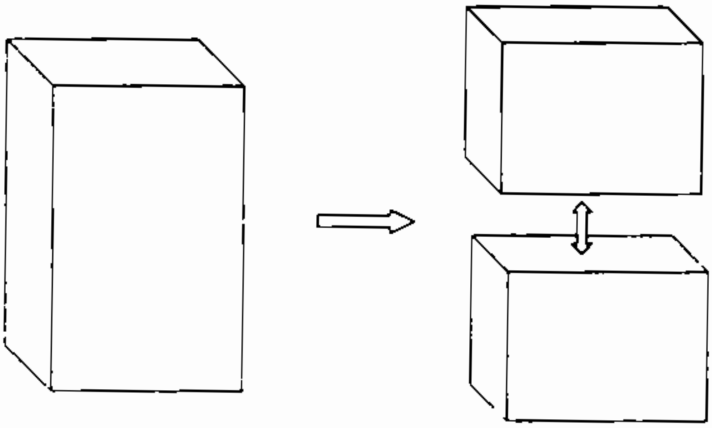

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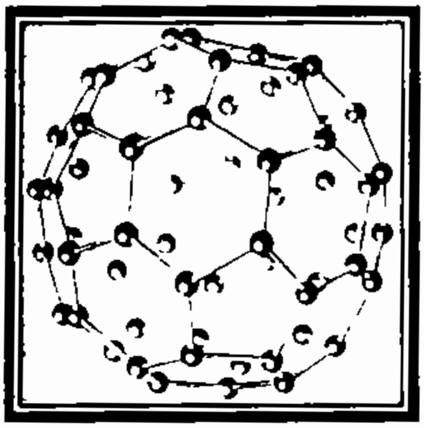

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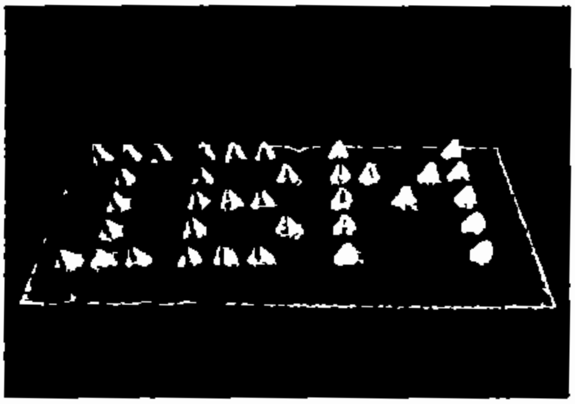

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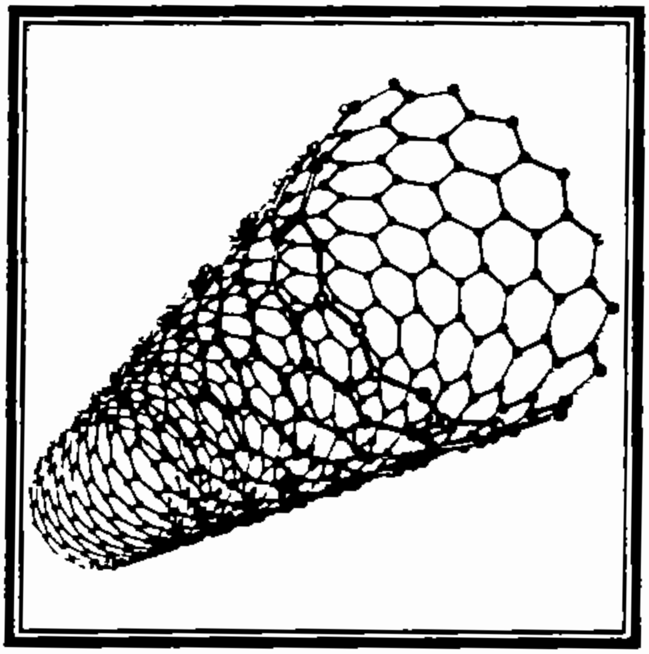

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- ✓ Semiconductor materials
- ✓ Dielectric materials
- ✓ Superconductor materials etc

When scientists and engineers try to tailor the physical or chemical characteristics of the above mentioned materials at nano level scale then they will fabricate a new sort of material called nano material. The nano materials further classified regarding their structures and dimensions weather they belongs to any of the aforementioned materials. Such nano materials fabricated through special kind of process in a clean room environment. Depending upon structure and dimensionality these nano materials are nano particles, quantum dots (QDs), nano wires (NWs), nano rods (NRs), nano tubes (NTs) and nano sheets or thin film etc. In this way we can say that nano science and technology is the science which deeply concerned with the materials structure at nano scale size due to which they exhibits improved or desired physical, chemical or biological properties [1-2]. Nano materials further grouped into the following

- ✓ Zero-Dimensional Nanostructures
- ✓ One-Dimensional Nanostructures
- ✓ Two-Dimensional Nanostructures

According to application point of view nano materials have broad scope being used in data storage devices, different electronic sensors, sports, medicine, agriculture and textile & fabrics etc. The figure 1.9 explains the above nanostructure classification of different nano materials

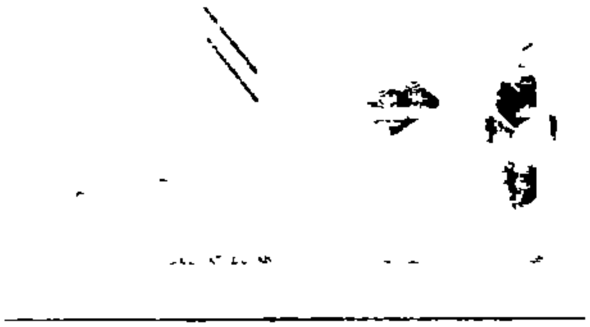


Figure 1.9 Various types of nanostructures regarding their dimensions

1.5.1 Zero-Dimensional Nanostructures

Nano particles have been developed by applying different techniques which includes top down and bottom up approaches as given below

- Milling or attrition
- Etching
- Repeated quenching
- Lithography

Nano particles with tens to several hundred diameters can be produced by attrition. The nano product i.e. nanoparticles from this particular technique have substantial shape, large volume changes as well as down-and-out thermal conductivity. In the same way lithography technique play a vital role in the fabrication of nano particles. Bottom up approach has been currently proved very effective for the synthesis of nano particles. The following bottoms up approaches are very popular now-a-days [11]

- homogeneous nucleation from vapors
- heterogeneous nucleation on substrates
- phase segregation from solid at high temperatures
- confined chemical reactions (micelles)

To synthesize 0-D structures the miniaturization of the nano products is not the only option. For device applications the synthesis and process conditions ought to be under control in such a way that the resulting products have the following distinguished properties [12]

- Uniform size distribution
- Identical (shape) morphology
- Identical crystal structure
- No agglomeration

SEM micrograph of 0-D materials given below

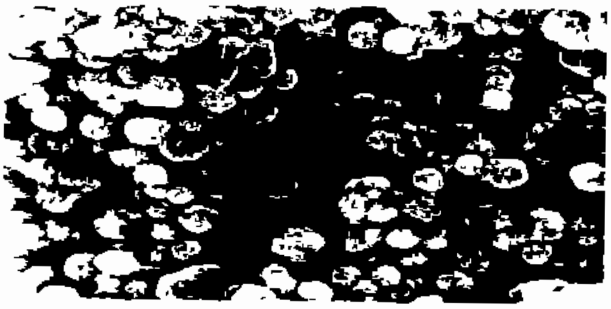


Figure 1.10 0-D materials

1.5.2 One-Dimensional Nanostructures

Nano products related to 1-D nanostructures are of different categories depending upon the different t size and diameter. The names of these categories are given as [13]

- Nano whiskers
- Nano fibers or nano fibrils
- Nano wires
- Nano rods
- Nano tubules
- Nano cables

Nano fibers and nano wires are such 1-D nano products which are considered larger than nano whiskers and nano rods The range of the diameter of nano wires and rods is less than o few hundred nano meters proved in recent research [14] While whiskers and fibers diameter range is up to several hundred nano meters to microns Generally the aspect ratio' of nano wires is greater than the nono rods Where the aspect ratio is defined as L D where L is length and D is the diameter of the concerned structure

There are some important categories of synthesis techniques for 1-D nano structures collectively These categories are given bellow [15-17]

- Spontaneous growth
- Template based synthesis
- Electrospinning
- Lithography

Spontaneous include the following steps

i Evaporation
ii Dissolution
iii Condensation
iv VLS (vapor liquid solid) growth
v SLS (solid liquid solid) growth
vi Stress induced recrystallization

The above mentioned synthesis techniques are grouped further in two ways bottom up and top down categories

Bottom up approach Spontaneous growth. Template based synthesis Electro-spinning

Top down lithography

Carbon nano tubes and ferromagnetic nano tubes are special family of 1-D structures The reason is that they are used in very advanced device fabrication equipment The ferromagnetic 1-D nano strictures are used in data storage devices [18-24]

SEM micrographs of 1-D structures

Figure 1 11 1-D material

1 5 3 Two-Dimensional Nanostructures

The well-known two dimensional nano structure is the thin film as show in the fig 1 12 If e look back in history, we come to know that more or less a century ago scientist had a sound knowledge about the two dimensional nano structures in the firm of thin film many methods for the synthesis of this two dimensional structure developed in that time and with the passage of time scientist and engineers brought development and betterment to the synthesis process All of such synthesis techniques which were developed previously, now a days they are widely used in different kinds of industries, for different scientific applications and devices like solar cells and biosensors etc the deposition technique of thin film grouped in two main streams given below

- Vapor based deposition

The examples of VBD of thin film are

i Sputtering
ii atomic layer deposition (ALD)
iii evaporation
iv molecular beam epitaxy (MBE)
v chemical vapor deposition (CVD)

- Liquid based growth

The examples of LBG are very well known which are given below

i Electrochemical deposition (ECD)
ii Chemical solution deposition(CSD)
iii Langmuir deposition

iv Blodgett films

v Self-assembled mono layers (SAMs)

Electro chemical deposition is very cost effective because rest of the techniques utilizes ultra high vacuum that's why researchers give preference to electrochemical deposition on the vacuum based synthesis techniques

Figure 1.12 Material with 2-dimension

1.6 Brief history of magnetism

Magnetic materials history grows up with the science's history Regardless the fact that the previous history of magnetism is indistinct but its characteristics of attracting pieces of iron bewitched the peoples of thousands of years ago Fe_3O_4 (lodestone magnetite) was the first magnetic material that was found in Magnesia (right now known as Turkey) Initially the magnetic device was fabricated by the lodestone magnetite The Chinese are the pioneer of making first useful device by which they measure the directions of earth's magnetic field as shown below in figure 1 13

In 1064 first professional navigational compass fabricated This invention plays a vital role in the discovery of America and Africa in 1492 and 1433 respectively There were many wrong concepts regarding the origin of magnetism but in but a first scientific and logical text "On the Magnet" by William Gilbert in 1600 overthrew them This effort put the scientists in the right path and later on in 1820 Hans Christian Oersted discovered that a current carrying conductor have the magnetic field around it

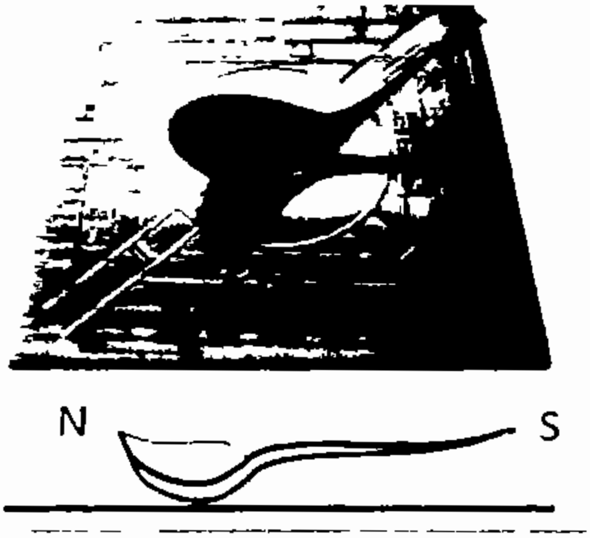


Figure 1 13 The earliest magnetic device from China South pointer

After this the success goes on in this field and electromagnetic induction was discovered by the Michael Faraday in 1821 The breakthrough of magneto optic and the Maxwell's equation about unified theory of electricity, magnetism and light in 1845 and 1864 respectively was also a nice achievement This brings the many improvements in the human's life in the form of electric motors, street lights and telegrams [25]

1.7 Phenomena of magnetism

Materials show the response of attraction or repulsion in the presence of external magnetic field Mostly materials show such type of phenomena, some reflects strong and some give weak response to the applied magnetic field Depending upon this magnetic response or behavior of the material they are grouped in more or less three or four categories [26]

Further there are mainly two types of magnetism

- electromagnetism
- permanent-magnetism

The former term is related with the magnetic field produced by the unidirectional motion of electrons inside the current carrying conductor, which can be found by the right hand twist rule The later term is related with the magnetic field produced by the spin and orbital motion of electron inside the atom around the nucleus [27]

As discussed above that the microscopically the magnetic effect generated inside the atom is because of the intrinsic spin and orbiting motion of electron that further become the property of the whole material This is shown schematically in the following figure 1 13

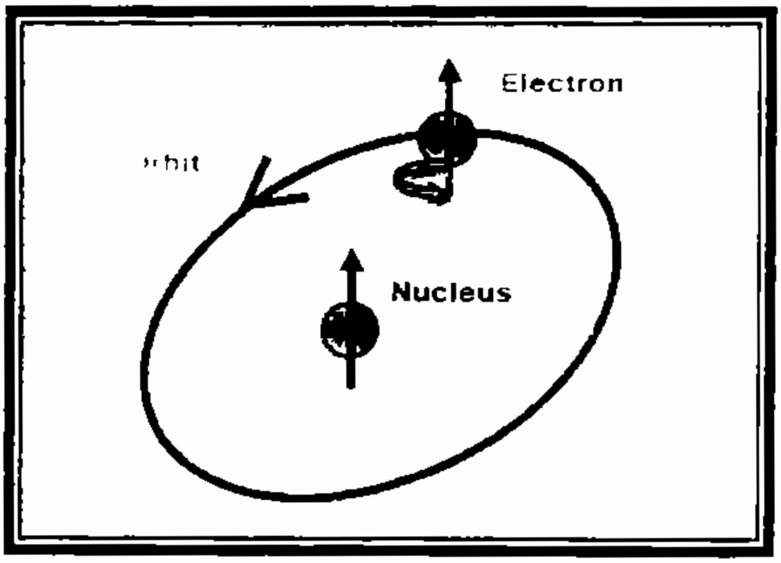


Figure 1 14 Two intrinsic motion of electron about nucleus spinning and orbiting

Actually the spin moment and orbital moment is because of this intrinsic motion of electron around the nucleus as shown in figure 1 14 These two moments of electron will further combine to produce the total magnetic moment

It is observed that the magnetic moments cancel each other which results almost zero or weak magnetic field but sometimes it is also observed that they cooperate with each other to generate strong magnetic field this amazing property of the material depends upon the filling and infilling the atomic orbits The strong magnetic field is because of the unpaired electrons while the filled orbits shows weaker magnetic field because the opposite spins cancels out each other effect [28]

Following different behavior of magnetism is expected from the magnetic material

- o diamagnetism

- paramagnetism
- ferromagnetism
- antiferromagnetism

When material is subjected to the external magnetic field then one must observed the aforementioned magnetic behavior because of magnetic moments of the atoms [29]. Figure 1.14 give the broad view of magnetic materials inside the periodic table of elements.

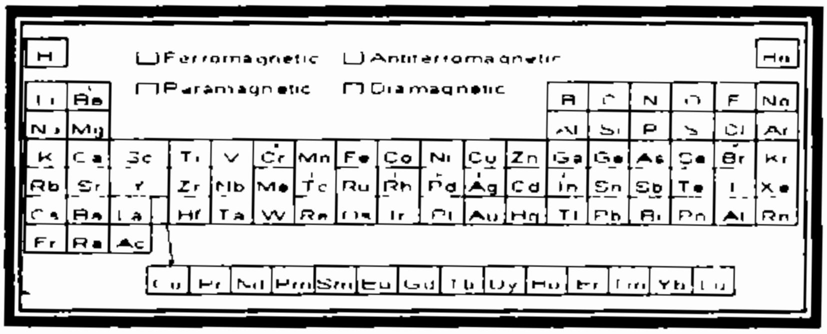


Figure 1.15 Periodic table of magnetic materials

1.7.1 Diamagnetic material

As all the orbitals filled and there are no unpaired electrons due to which there will be no magnetic moments inside the diamagnetic materials. The magnetic susceptibility χ_m of diamagnetic materials is less than zero almost in the order of -10^{-5} [30-31]. This is due the fact that such materials shows negative magnetization there magnetic moments orient themselves contrary to the applied external magnetic field H. When the external magnetic field is removed they still show some negative magnetization as show in figure 1.16.

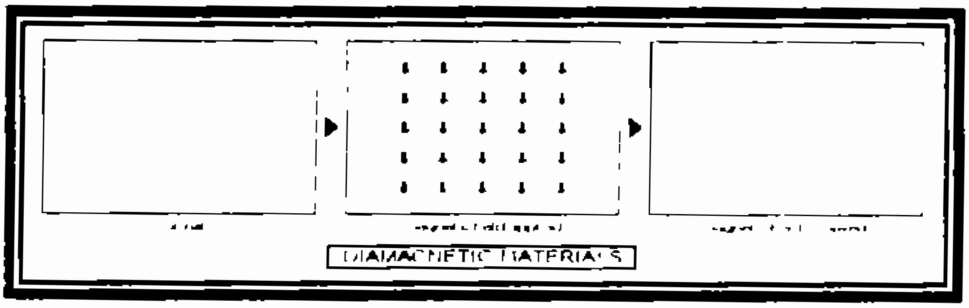


Figure 1.16 Diamagnetic materials when (a-left) no field is applied (b-centre) field is applied (c-right) field is removed

1.7 2 Paramagnetic materials

Paramagnetic substances are those substances in which partially filled atomic shells or obits are present That s why unpaired s electrons become the cause of magnetic moment These magnetic moments are oriented irregularly inside the material as shown in the figure 1 16

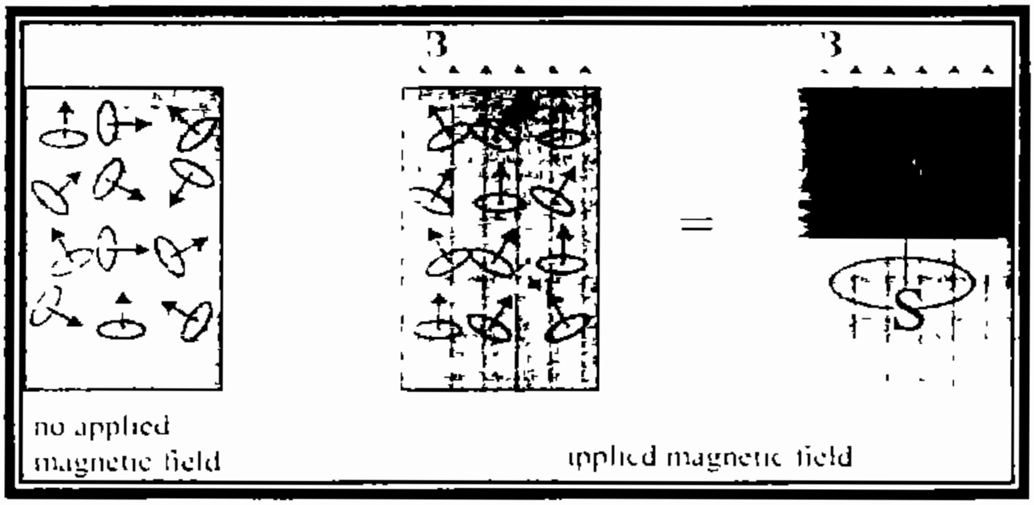


Figure 1 17 Paramagnetic materials when (a-left) no magnetic field (b-right) applied magnetic field

We concluded that in the presence of external magnetic field the magnetic moments aligned themselves in the direction of external magnetic field and a total magnetization $M=M_o$ observed in the material which results the positive susceptibility

1 7.3 Ferromagnetic material

In ferromagnetic materials the magnetic moment of each adjacent atom is aligned parallel When such materials subjected to the external magnetic field then their atomic magnetic moments arrange themselves in the direction of external field and they will remain in the same position even after the external field becomes equal to zero Co Fe, Ni etc are the best examples of ferromagnetic materials The magnetic susceptibility of these materials is very high up to the order of 10^6 [32]

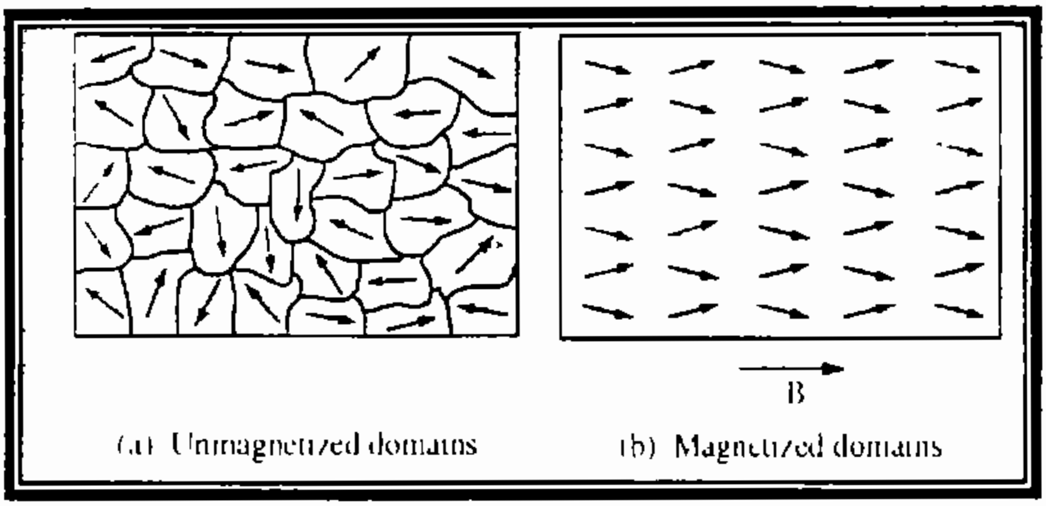


Figure 1 18 Ferromagnetic material when (a) no magnetic field (b) magnetic field

1 7.4 Anti ferromagnetic material

In anti ferromagnetic substances the magnetic moments of atoms aligned oppositely as illustrated in the figure 1 18 given below Below the Neel temperature let's suppose if one group is upward then the other surly downward which results in zero spontaneous magnetic field On certain temperature when they are subjected to external magnetic field they will respond like a paramagnetic material with positive but smaller values of magnetic susceptibility

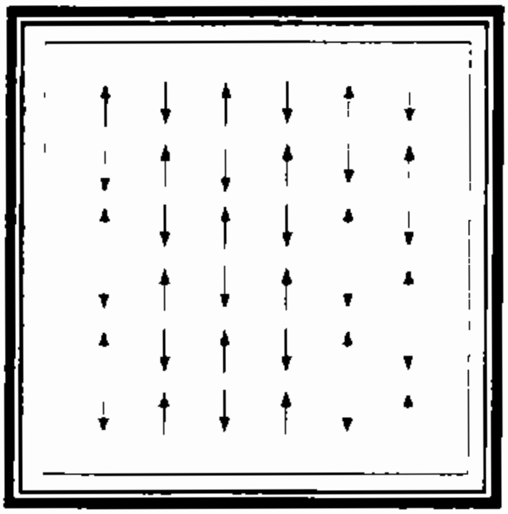


Figure 1 19 Anti-ferromagnetic material

1.8 Central principles of magnetism

The net magnetic induction out of magnetic material can be found out with the help of following equation

$$B = \mu_o H + \mu_o M \quad (1)$$

OR

$$B = \mu_o (H+M) \quad (2)$$

When we apply external stimulus **H** (external magnetic field) on the magnetic material due to which internal magnetization **M** produced inside the material the aggregate effect of these two fields is denoted by **B** as given in equation 1 and equation 2 Where **B** is also known as magnetic moment per unit volume of the material

μo = constant of permeability = $4\pi \times 10^{-7}$ Wb A-m

Tesla (T) or (Wb/m^2) is the system international unit of **B**, while Am^{-1} is the system international unit of **H & M**

1.9 Magnetic Permeability

If we talk about free space then there will be a direct relation possible in between **B & H** we can write for free space that **B = H** But in case of magnetic material the magnetic induction **B** become larger during the presence of external magnetic field **H** to what extent the magnetic induction become larger this will decide the term μ known as "magnetic permeability" and that can be easily understand from the following relation

$$B = \mu H \quad (3)$$

From equation 3 we can write the relation for "magnetic permeability" as

$$\mu = B/H \quad (4)$$

Hence we can say that it is the quotient of magnetic induction over the exposed external magnetic field as given in equation 4, while the SI unit of magnetic permeability is Wb/A-m

Relative permeability (μ_r) is another parameter by which we can understand the further properties of the material, which is the quotient of μ/μ_0

The way we increase the intensity of external field in the same way the volume of internal magnetization of the magnetic material increases this phenomena is linked with the magnetic susceptibility The magnetic susceptibility of the material can be understood by the following expression

$$M = \chi_m H$$

Or

$$\chi_m = M/H \tag{5}$$

It is cleared from the above formula of magnetic susceptibility that it is unit less because it is the ratio of such quantities (M & H) those posses the same SI units

There is also a mathematical relation between susceptibility and magnetic permeability that can be developed by the manipulation of the above equations as,

$$\chi_m = \mu_r - 1 \tag{6}$$

1.10 Magnetization processes and hysteresis

The magnetization carry through the ferromagnetic materials is very remarkable regarding different research and device fabrication point of view The well known materials regarding this point of view are Ni, Co, Fe and some of their alloys

The rudiment property of ferromagnetic substances is their response to the external magnetic field H The magnetic property explained or reveled by the VSM in M-H loop describes the non-linear response of material's internal magnetization M to the H (external applied field) as shown in the figure 16 given below The M-H loop is also known as magnetization hysteresis curve

In order to obtain M-H curve for the understanding of magnetic behavior of FM material it is place in the VSM characterization tool This tool has ability to generate the curve between M and H Initially the sample gets saturated in one direction because of the application of external field H and in the direction of H Actually all the randomly oriented magnetic moment of all the domains inside the material arranged themselves with the external magnetic field H produced by the super-magnets or electromagnetic coils This point is marked with M_s in the figure 1 17 When the external field reverses from its maximum value towards the minimum value the magnetized domain also reverses but not moves back linearly with the field H even though the H becomes zero but there is still some magnetization inside the material This phenomenon is of remanent and symbolized by the M_r inside the curve on y-axis At this stage few magnetic moments of the domains get reversed with the field H that s why we observe M_r inside the material which is because of previously saturated magnetic domains After this the external field becomes negative so that to obtain zero magnetization inside the material The magnitude of H where the M is almost zero is known as the coercive field H_c as shown in the M-H curve At this stage the domains once again is so arranged that they cancel out each other effects which results in null or zero magnetization We can also say that at H_c fifty percent of the domains reversed against the saturated direction of M_s When we further go increasing the H in opposite direction the rest of the domains get saturated in totally opposite direction until to reach the opposite magnetization saturation For demagnetization of ferromagnetic substance thermal heating applied

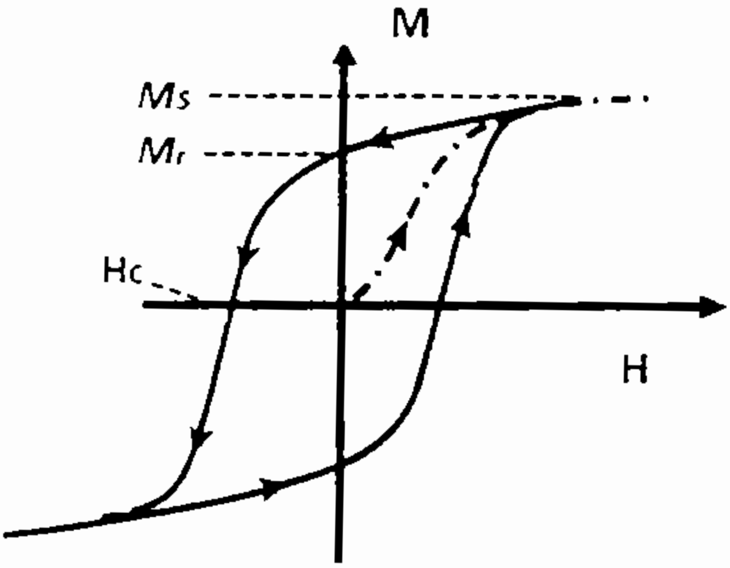


Figure 1 20 M-H curve of a ferromagnetic material

Some of the important ferromagnetic materials have the following saturation magnetization (M_s) values given in the table 01

Table 01 Values of saturation magnetization of FM materials

Material	M_s (emu/cm^3)
Ni	485
$Co_{55}Fe_{45}$	1950
$Ni_{80}Fe_{20}$	800

1.11 Shape anisotropy

The shape of the sample and aspect ratio as well, has the great impact on demagnetization energy or the shape anisotropy of the sample or material. It's uniquely a pure property of the material that depends upon the structure dimensionality. The magnetization process inside the prolate spheroid and sphere is totally different because difference in structure (semi major and minor axis). In sphere the magnetization occurs uniformly in all directions rather in prolate spheroid. Their magnetization process occurs easily along the cylindrical axis rather than diametrically. At nano level especially when we talk about the one dimensional structure like nano wires and nano tubes this effect have its own great effects and worth because it defines or alter some other magnetic properties of the material i.e. it will limits the magnitude of coercivity in FM materials. In a way to comprehend the concept of shape anisotropy let us take the example of prolate spheroid which is very close to the samples structure fabricated in this presented research work i.e. NW's. The prolate spheroid is given in the figure 1.20

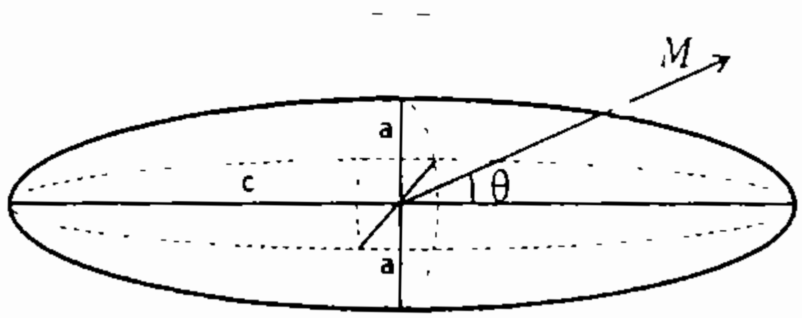


Figure 1 21 Prolate spheroid's schematics for shape anisotropy

When external magnetic field of some suitable magnitude magnetizes the prolate spheroid to some extent M by making angle θ with the " c" (prolate major axis) then the E_{MS} "magnetostatic energy can be mathematically given below,

$$E_{MS} = \frac{1}{2} M^2 \sin\theta^2\,(Na - Nc) \qquad (7)$$

Where the demagnetization factors towards a-axis and c-axis are illustrated by the N_a & N_c respectively [33]

The shape anisotropy constant can be written as in the present case of prolate spheroid

$$K_s = \frac{1}{2} M^2 (Na - Nc) \qquad (8)$$

While the "shape anisotropy constant" K_s specify the aspect ratio (which is in general, length over the diameter) in order to find the shape anisotropy For instance taking $N_a = 2\pi$ and $N_c = 0$ then we have left with this relation [38-39]

$$E_{MS} = \frac{1}{2} M^2 (2\pi - 0)$$

$$E_{MS} = M^2 (\pi - 0)$$

$$E_{MS} = M^2 \pi \qquad (9)$$

That s why it can be thought-out that NW's and NT's have their easy axis along the wire and tube length respectively

1.13 1-D magnetic nano structures & their applications (NW's)

Here is the bird's eye view of the nano wires of various materials and especially magnetic materials A nano wire is such 1-D structure in which the transport of charge carrier in two dimensions is prohibited Because of this type of nano structure nano wires and nano tubes have diverse area of applications regarding technological device point of view Normally the diameter range of the NT's and NW's is in the order of manometer while the length is in the range of some nm to few microns Due to these unique size ranges of these nano structures become the cause of tremendous quantum mechanical effects As compared to bulk the nano range structures have extraordinary physical properties and characteristics i e "electrical, optical and magnetic characteristics' Presently the nano scientists are researching and investigating their great properties for many potential applications [34]

In this regard many kinds of nano wires and nano tubes are fabricated of different suitable materials given to the table 02

Table 02: Nano wires of different materials [35]

Metallic	**Semiconducting**	**Insulating**
Nickle	Silicon	silicon dioxide
Platinum	Indium Phosphide	TiO_2 etc
Gold etc	GaN etc	

1.13.1 Magnetic Nanowires

Now-a-days magnetic nano materials and their structures are at the top priority basis of the researchers The world's cutting edge research on the magnetic nano wires and tubes is the big explanation of their importance being utilized in the different patterned magnetic data storage devices like MRAM, RACE TRACK memory and substance for microwave applications etc [36-37] The mystery of magnetic one dimensional structures is still under discussion and researchers tries to explored their essential scientific issues such as, "shape anisotropy as opposed to magneto-crystalline anisotropy, establishing the easy axes for typical preparing conditions and description of magneto-static interaction between wires" There are numerous methods to fabricate the 1-D magnetic structures, but electrochemical fabrication process is one of the best and cost effective techniques (AC & DC fabrication process) Possin was the scientist who published for the first time the deposition of NW's by the electrochemical deposition process in 1970 At that time he made use of track mica films for the deposition of NW's [38-39]

CoMn alloy Ni, Ni Sn and NiFe NW's has been fabricated for different technological applications Experimentally it is proved that the retantivity, dipolar interaction between the wires and coercivity can be tailored by changing the diameter and lateral length of the wire By changing these magnetic properties we can use these NW's in different data storage devises Cobalt manganese alloy with large aspect ratio is considered good for random access memory [40-41] High coercivity is the big demand of the data storage devices that is fulfilled by the high aspect ratio and with packing density at least $10^{11}\frac{wires}{cm^2}$ [42] In this research work Ni_xFe_{1-x} nano wires and nano tubes are fabricated and characterized for the purpose of data storage devises We try our level best to produce the nano wires of high density and homogeneous in nature as further explained in the upcoming chapters

Chapter 02

Synthesis of 1-D nanostructures

2.1 Introduction

The growth of nano structures for different multifunctional applications is carried out with different routes and methods Technically or scientifically there are mainly two approaches for the fabrication of different nano structures and these two approaches are (i) "Top down Approach" & (ii) "Bottom up Approach"

On the behalf of above mentioned approaches there are further techniques to produce nano structures These techniques have their own merits and demerits regarding their production cost and yield quality The following schematic diagram illustrates these nano fabrication techniques with respect to "Top down & Bottom up Approaches"

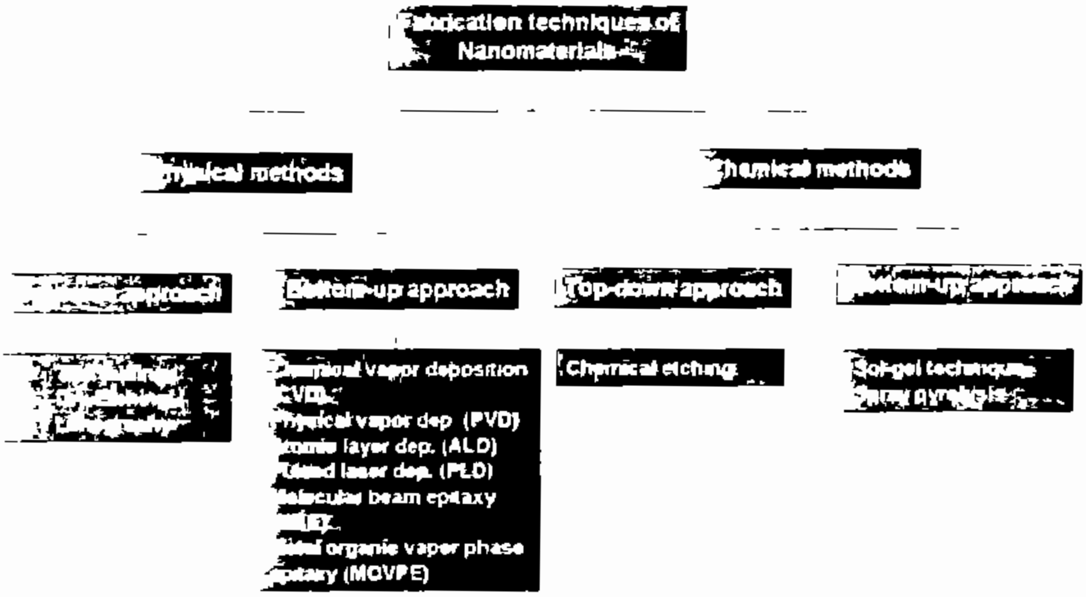


Figure 2 1 Systematic diagram of fabrication technique of nanomaterials synthesis [43]

2.2.1 Top-down technique

It is the process of splitting up of the material from bulk level to a very minute level of nano meter range Overall we can say that the top down approach is basically miniaturization of bulk material up to the nano or micro level extent The main problem with this technique is that it creates imperfections within its nano products But on the other hand it the nano production from this method is very cost effective and very easy as compared to the rest of the techniques Top down approach includes the "electron beam lithography high-energy wet ball milling gas-phase condensation aerosol spray and atomic force manipulation" as its main techniques to synthesize the nano products

2 2.2 Bottom-up Technique

Bottom up technique is the reciprocal bottom up technique regarding concept and as well as experimental lab working In this context it will be quite right to say that the bottom up technique is the name of sewing or knitting of atom to make the desired nano products The well known methods which comes under the heading of this technique are "hydrothermal synthesis Organo metallic chemical route solgel synthesis, colloidal precipitation, revere-micelle route, template assisted sol-gel" etc Figure 2 2 illustrate the all science about top down and bottom up techniques

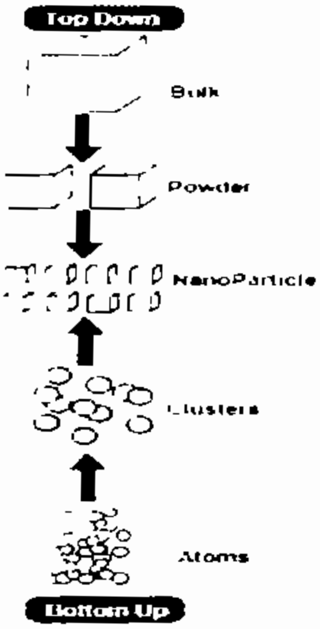


Figure 2 2 Top Down and bottom up Approach of synthesis processes [44]

Electro-chemical deposition is an elegant, cost effective and versatile in the deposition of nano structures (NW^{a}, NT^{b} & NR^{c}) among all the above mentioned techniques The fabrication of any kind of nano structure electro-chemical deposition employs the concerned structure's metal pattern which is known as template The templates are electrically insulators because of oxidative layer and their formula is Al_2O_3 Bio medical sensors, non-linear optics, magneto optics, semiconductors micro electro mechanical system and all other technologies required suitable nano structures for present and future technological applications or device fabrications [55-65]

The demand of such future technological application is fulfilled by anodic aluminum oxide (AAO) templates which solves all the key challenges of the present nano-fabrication technology The main key challenges are given below

- ✓ Low cost
- ✓ Large production of nano structures
- ✓ Homogeneous growth
- ✓ Versatile in yielding nano structures
- ✓ Have control over growth

In this way we can say that templates based nano fabrication is having more worth and value as compared to the other synthesis techniques i e e-beam & ion beam nano-lithography, Vapor liquid-solid growth etc [56-58]

We have great control over the aspect ratio (length over the diameter) and diameter of nano-structures by using AAO templates [59-61]

AAO templates have great power of attraction for scientist and researchers because of their high density and regular distributions of pores [62-63]

In this way for the sake of metallic nano-structures this technique is playing a vital role in research and technology

2.2 AAO templates

Whatman is the company which commercially synthesizes the templates There are two main types of templates which are formed by annodization process The names of the templates are (1) anodized aluminum oxide templates, 2) polycarbonate membrane templates

Anodic aluminum oxide consists of uniform cell structure having hexagonal self organized nano pores The growth of nano pres is vertical to the membrane surface, while each pore is parallel to each other as shown in figure 2 3, given below

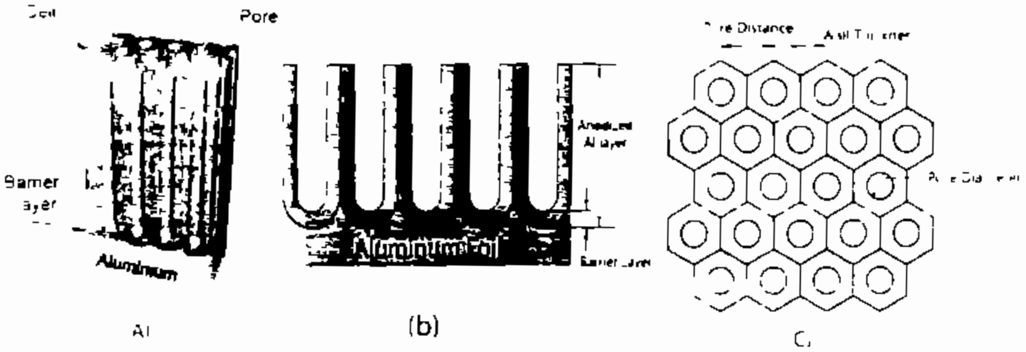


Figure 2 3 (a) Schematic diagram of anodic aluminum oxide (AAO) template and (b) side view of AAO templates (c) top view [70]

The following experimental parameters or physical conditions have great impact on the structural characteristics of the AAO cylindrical pores in such a way that we can tune up the, inter pore distance (D_{int}) lateral length pore diameter (D_p) and regularity of the AAO pores

- ✓ Applied voltage (V_{app})
- ✓ Temperature
- ✓ Time
- ✓ Nature of acidic solution like H_2SO_4 or oxalic etc
- ✓ Purity of acids
- ✓ Molarity of acids [65]

Initially AAO templates were formed as a protective layer because it has the property of anti corrosion Such type of AAO templates coatings were also used in mechanical industry where the surfaces of different kinds of tools were made protective and well finished or smooth [66-67] There is a specific process known as two step annodization by which homogeneously self organized pore membrane fabricated [58] According to this method the following D_p and the corresponding to D_{int} were published in the scientific literature with the following size,

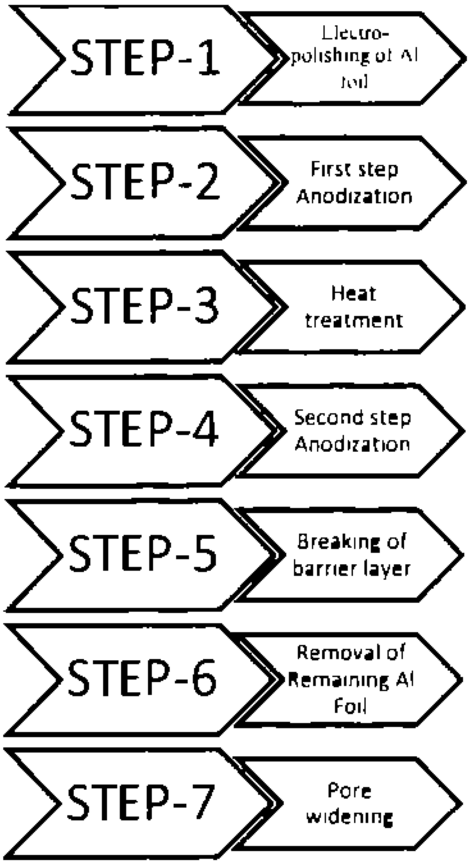
D_p = 6 nm to 200 nm

D_{int} = 65 nm to 500 nm [68-70]

Different kind of impurities imperfections and the grain size of Al foil may have strong impact on the final structural parameters of AAO templates The research in the field of every area of science and technology is reinforced with the versatility of AAO templates [71]. The pore density of AAO templates is in the range of 10^{11} p/cm^2 by which researcher may able to synthesize a comprehensive numbers of nano wires simultaneously. The other amazing feature of AAO templates is that when they react with phosphoric acid their pore diameter becomes wider [72]. From figure 2.3 it is cleared that AAO templates have 2-D array of uniform size cylindrical and hexagonal packing of pores. Furthermore we can also say that AAO templates are best substrate to deposit different metals like Ni, Co, Fe and Cu.

2.3 Experimental steps for the fabrication of AAO templates

We can prepare a very fine quality of AAO templates inside the graduate physics lab easily by performing the following scientific experimental steps.



2.3.1 Electro-Polishing of Al Foil

This electrochemical process of electro-polishing was performed at room temperature. During this process the irregular surface of Al foil were made smooth for the growth the AAO templates. During this chemical process the dirt from the surface of Al foil was seen removed clearly just after the 60 to 70 seconds. The whole chemical process almost takes at least 3 minutes. Following acids with certain proportion were used to prepare the electrochemical solution.

$$HClO_4\text{-}25\% \text{ and ethanol-}75\% \ (1:4 \text{ by volume})$$

About 9 volts constant DC voltage was applied for three minutes for electro-polishing. At the end of chemical reaction the Al strip was removed from the apparatus and rinsed with distilled water instantly. A very fine and smooth reflecting shiny surface appeared when it gets free from moisture and completely dried as shown in the figure 2.4.

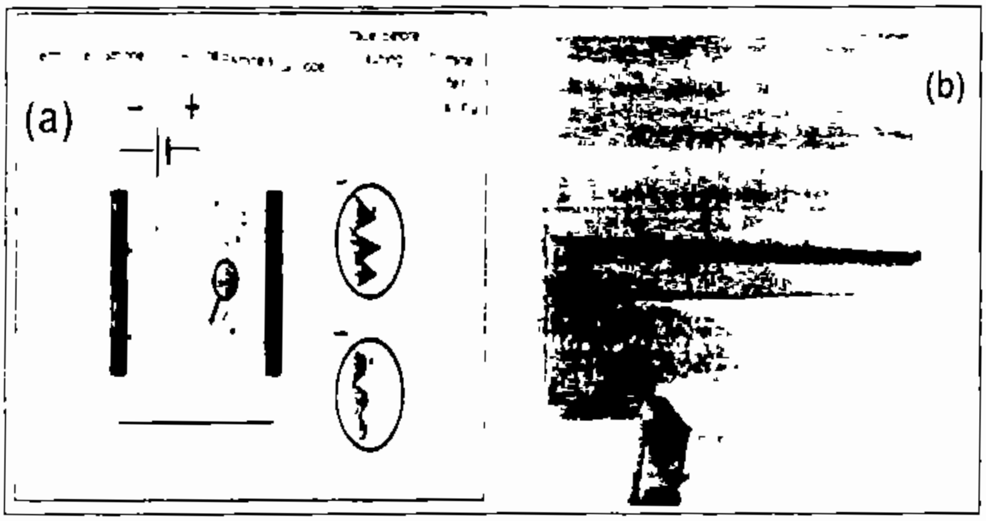


Figure 2.4 (a) Schematic view of electro-polishing (b) Electro-polished Al foil

2.3.2 First Anodization of Al foil

After getting the electro-polished Al foil the next process of anodization were performed inside the specific acidic electrolyte, phosphoric acid of Molarity 0.3 M. During this process a stratum of Al_2O_3 deposited which made the Al foil insulator. The first anodization was performed at 5 ^{0}C for four hours under constant value of DC voltage 60 V. The templates formed in first step of anodization little bit irregular but they were made smooth and regular in second step of anodization. The chemical reaction which taken place on both cathode and Al strips which was connected with anode can be written in the form of in the following chemical equations

- The Overall anodization of Al is
  - $2Al + 3H_2O \rightarrow Al_2O_3 + 3H_2$
- The Anodic reaction taking place at metal-oxide boundary is
  - $2Al + 3O^{2-} \rightarrow Al_2O_3 + 6e$
- The Anodic reaction proceed at oxide-electrolyte interface
  - $2Al^{3+} + 3H_2O \rightarrow Al_2O_3 + 6H^+$
- When porous oxide layer is formed
  - $2Al \rightarrow 2Al^{3+} + 6e$
- The reaction at cathodes in which hydrogen gas is librated
  - $6H^+ + 6e \rightarrow 3H_2$

The experimental setup for the first anodization is shown in the following figure

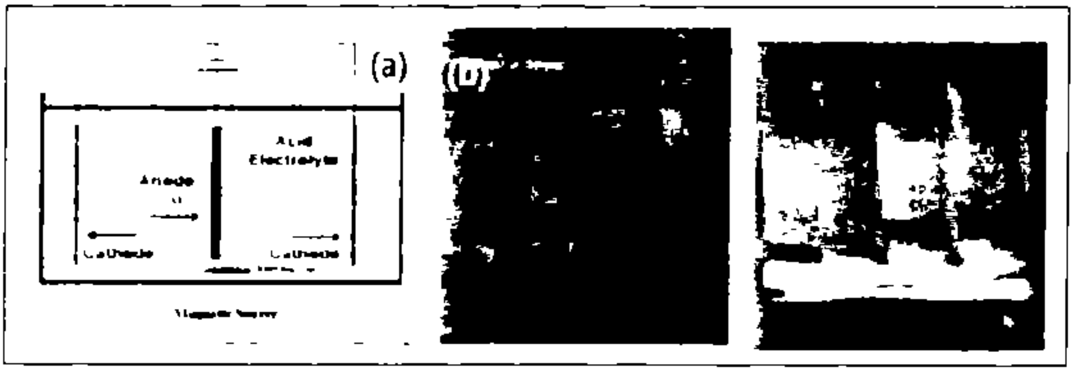


Figure 2 5 (a) experimental set up for Anodization and (b) Al foil after first Anodization

2.3.4 Second anodization of Al foil

The growth of AAO templates is actually the growth of oxide layer i e Al_2O_3 The direction of deposition or the evolution of this oxide layer is entirely electric field dependent which is setup on the Al strip, dipped in acidic electrolyte, as an anode This is also observed that during first time anodization process the gradual evolution of oxide layer was not so smooth almost irregular in nature schematically shown inside the figure 2 6 given below

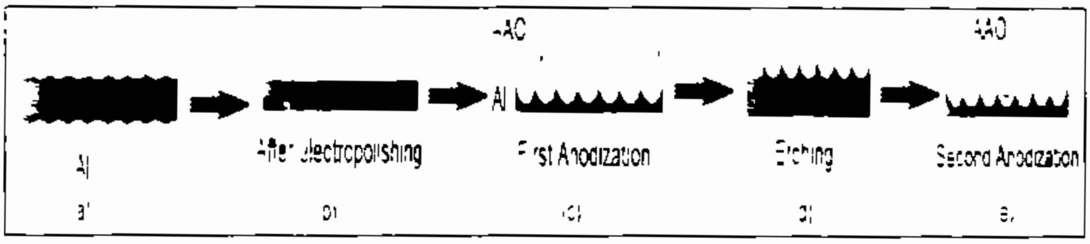


Figure 2.6 (a) Al foil before electro-polishing (b) After Electro-Polishing (c) Fist Anodization (d) etching process (d) Second Anodization

As it is high demand of the better research to grow regular, homogenous and straight pores of AAO that's why we cannot rely on the first annodization. Now in a way to solving this problem of irregularity of AAO pores the strip was dipped inside the yellow solution and put into the oven at 60 °C for one hour and thirty minute. This process is also called heat treatment. The yellow solution ingredients are H_3PO_4 6 wt % and CrO_3 1.8 wt % acid. During this process all the irregularities of the AAO oxide layer were etched and the Al strip was only left with the footmarks of the earlier evolution or growth of oxide layer. After getting this Al strip it was further rinsed with distilled water and fastens with a support inside the lab to dry it at room temperature. Now this Al strip is ready for the second annodization the annodization parameters will remain same but reaction time is increased up to 12 to 14 hours for the sake of better and latterly long and well ordered homogenous structure of AAO templates. The structure or the geometry of AAO templates right now entirely on the same track or foot prints of the previous but the only good thing is that it is not like before i.e. irregular it is now fully ordered form with hexagonal structure of AAO as shown in the figure 2.7 given below

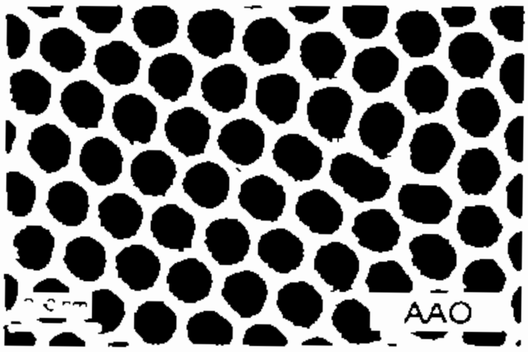


Figure 2.7 SEM, hexagonal structure of AAO templates

The value of current does not remain constant even the value of voltage remain constant throughout the reaction. This is because the fact that the growth of oxide layer continuously increasing and it is insulator in nature due to which current doesn't remained same and drops to some extent with the passage of time.

2.3.5 Detaching of barrier layer

The barrier of AAO templates were removed at the end of second anodization. The Al strip was not removed from the acidic electrolyte. It was kept in dipped position and the process of barrier removing started. The process is so simple, it is just the stepping down the value of voltage after some specific time. About one volt is dropped after three minutes. During this process of barrier breaking the value of current dropped gradually until reached the minimum value i.e. zero m A, at that time the barrier layer completely removed from the bottom of templates. The Al strip was then removed and from the apparatus and rinsed with the distilled water.

2.4 Factors affecting the evolution of AAO template

Following factor can affect the growth of AAO templates in different ways

- ✓ Anodization voltage
- ✓ pH of electrolyte
- ✓ Anodization time

The researchers finally finds answer of mystery that the shape and size of AAO templates especially their diameter, entirely depends upon the anodization voltage. The barrier layer can also be tune up with the anodization voltage. The size of nano pore diameter is directly proportional with the anodization voltage and it ranges from 1 nm to 1.2 nm per volt [73].

The next parameter that affects the evolution and growth of the AAO templates is pH value of the acidic electrolyte. There are three main types of acidic electrolytes used for that purpose

- ○ Sulfuric acid (H_2SO_4)
- Oxalic acid ($H_2C_2O_4$)
- Phosphoric acid (H_3PO_4)

The molarity and pH of each acid have its own impact on the formation of templates geometry. It is experimentally noticed that the pore diameter is relatively small by sulfuric acid as compared to the rest of the acids But the anodization time is predominantly reduced by using sulfuric acid as an electrolyte The above mentioned electrolytes have their own fix value of working voltages and molarities upon which they work soundly The pore size and regularity of the pore cylinders is experimentally observed very good of larger size by phosphoric acid [74- 75]

The formation of AAO templates means the growth of oxide layer on the Al foil The growth of oxide layer can be increased by increasing the anodization time simply In this way we can say that the lateral length of the nano pores can be affected by the anodization time If the reaction time is larger than as a result of this the lateral length of the templates also becomes larger [76] The following SEM images give proof of this fact in increase of lateral length of templates that is fabricated into the oxalic acid of 30 gram/l at room temperature with 40 V of anodization voltage

Figure 2 7 The thickness of AAO template with the anodization time (a) 3 hours (40 um) (b) 6 hours (58 um) and (c) 15 hours (123 um) [83]

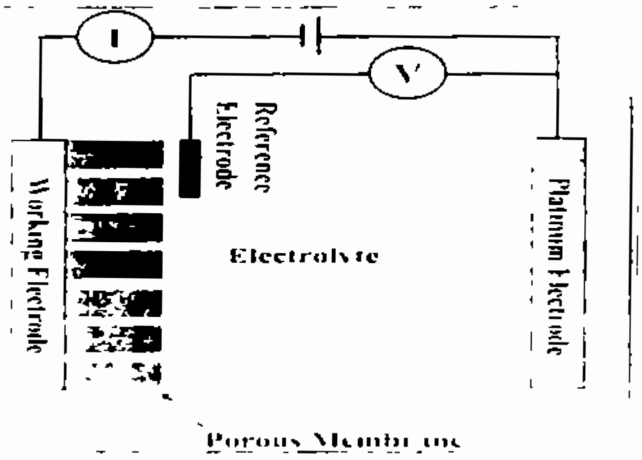
2.5 Electrodeposition Technique

Electrodeposition technique is just like electrolysis process In this technique the two electrodes dipped inside the acidic electrolyte and then current passed through them During this process the solution decomposes into positive and negative ions that further get deposited to the opposite polarity electrodes

Electrodeposition technique is very useful as compared to the rest of physical techniques This technique is very cost effective and very versatile to fabricate different nano structured specially nano wires and nano tubes There are three main types of electrodeposition techniques In this technique researcher can use the lab environment for the fabrication of nano structures in other words there is no need of any expensive apparatus or equipments like ultra high vacuum technology etc The deposition rate of this technique is relatively high in comparison with the other physical deposition techniques All those elements and compounds that feel or experience the electrostatic force can be deposited by using electrodeposition techniques Following chemical parameters play a vital role in depositing the metal elements

- local current density
- electrolyte concentrations
- buffer capacity
- pH
- leveling agents
- brighteners
- surfactants
- contaminants
- temperature
- agitation
- substrate properties
- cleaning procedure

The schematic view of electrodeposition apparatus is shown in the figure given below In this research work the deposition of 1-d nano structure carried out with electrodeposition technique in laboratory made AAO templates



Figurer 2.8 Electrochemical deposition

2.6 Template assisted electrochemical deposition

There are two types of templates the first one is anodized aluminum oxide (AAO) and polycarbonate membranes These templates are available in market made by the Whatman Company" but both of them can also be prepared inside the lab of the same quality One can also prepared the choice of its own templates regarding pore size and nano cylinders length or on the other hand can also buy from the market for research work

2.7 Types of Electrodeposition

Basically there are three well established and well practiced electrodeposition methods which are given below

i DC (Direct Current) electrodeposition

ii Pulse current electrodeposition

iii Alternating Current (AC) Electrodeposition [78-79]

In DC (Direct Current) electrodeposition we need such type of AAO which is completely detached from the substrate (Al) In this regard one should deposit the at least 20 μm thick oxide layers so may able to remove easily After this detaching process the detached layer undergoes the process of puttering of conductive layer from any one of its end The sputtered conducting layer has thickness range from the few nm to 300 nm depending upon the nature of the deposited structure The electrodeposition techniques other than DC electrodeposition technique do not required the detachment process of AAO templates The mostly used method of deposition is AC deposition method because of its simplicity and easy handling and good results for the nano wires [80]

2.8 Evolution process of Nanowires

The evolution process of nano wires starts from the bottom of templates that is sputtered with the Cu conducting layer and placed at the negative electrode known as working electrode When the sputtered templates dipped inside the electrolyte along with the working electrode then the current passed through the electrolyte during this process the solution splits up into its positive and negative ions The desired ions moves towards the templates and get deposited inside the pores of the templates Specific and suitable amount of negative potential applied to the working electrode for the deposition of respective positive ions [81-82]

2.9 Synthesis of ferromagnetic $Ni_{80}Fe_{20}$ nanowires and nanotubes

In order to fabricate excellent permolly of $Ni_{80}Fe_{20}$ DC electrodeposition technique employed The selection of this technique is because of its own worth of excellent nano products like nano wires and nano tubes We used self made AAO templates for the fabrication of permolly mention above

2.9.1 Growth of $Ni_{80}Fe_{20}$ NWs

The recipe of chemicals used for the purpose of $Ni_{80}Fe_{20}$ NW s is given in the table 2 1

Table 2 1 Chemicals used for $Ni_{80}Fe_{20}$ NWs

Chemicals	**Suitable Ratio**
$NiSO_4$ $6H_2O$	12 44 g
$FeSO_4$ $7H_2O$	0 54 g
H_3BO_3	4 g
SDS	0 02 g
Sodium Citrate	0 06 g
Thiorca	0 03 g

By using the above recipe 250 ml electrolyte were prepared The self made AAO templates were sputtered with Cu layer of 300 nm by ULVAC to make them conducting Furthermore the AAO templates were pasted above the working electrode and then finally exposed to the electrolyte Then a -1V applied to the working electrode of this three electrode cell The electrolysis process takes place during which positive ions produced and they get deposited to the inside of the AAO templates The process of reduction took placed here because of gain of electron from the working potential The process starts for different durations of time in order to make more than one samples

2.9.2 Growth of $Ni_{80}Fe_{20}$ NTs

For the growth of $Ni_{80}Fe_{20}$ NT's following recipe of the chemicals were made use as shown in the table 2.2

Table 2.2 Chemicals used for $Ni_{80}Fe_{20}$ NTs

Chemicals	Suitable Ratio
$NiSO_4 \cdot 6H_2O$	0.2579 – 0.5 g
$FeSO_4 \cdot 7H_2O$	0.05 – 0.058 g
Triton-100-x	0.06 – 0.05 g
SDS	0.0072 – 0.01 g
Sodium Citrate	0.025 – 0.04 g
Thiora	0.0068 – 0.009 g

In away to making nano tubes a 30 nm thin layer of Au was sputtered on one side of the AAO templates. Then we follow the same procedure inside the three electrode cell as followed for the nano wires. The same value of -1V is used for the reduction of positive ions in the AAO templates. But at this time the positive ions get reduced by getting electrons just with the walls of AAO templates and become solidify to finally form the NT's of the afford mentioned permolly. The whole experimental setup is shown inside the figure 2.9 given below.

Chapter 03

Characterization techniques

3.1 Introduction

Just after the deposition of ferromagnetic nano structures i.e. NW's & NT's the next step of the research is to investigate the hidden science or physics at the nano level. For this purpose the deposited material sample was sliced into different appropriate unit size portions for the sake of different characterization techniques. Following are the major techniques by which the sample undergoes to collect the raw data for the analysis of different phenomenal feature of NiFe permolly.

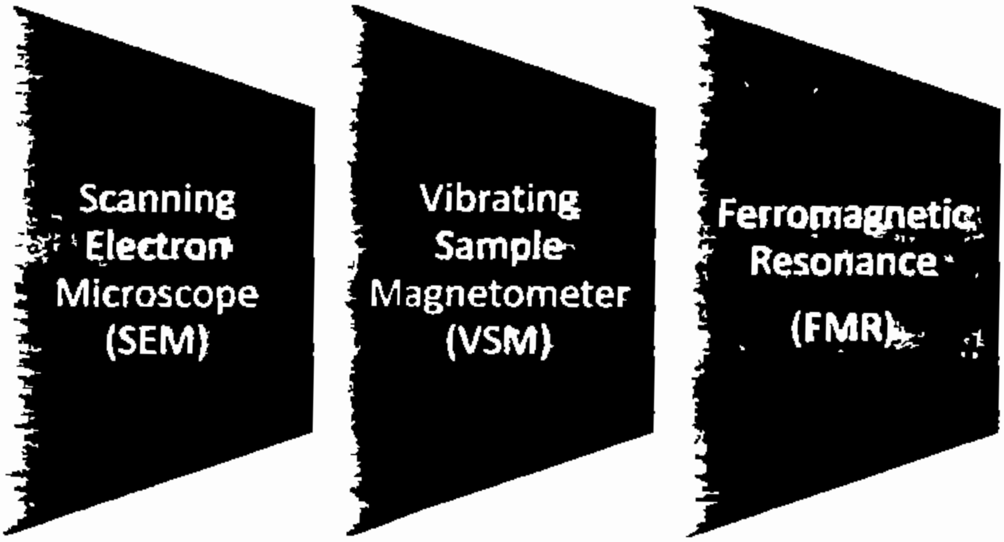


Figure 3.1 Important techniques of this research project

The scanning electron microscopy of the sample provides the clear image of the sample at different optical zooming size in general. The surface topography will be explained through this technique. One can easily understand that weather the desired structure formed or not like in our research we will see that weather the NW's or NT's formed in a good way or not. The regularity and some other measurements regarding the geometry of the structure can also be taken from the SEM. Well the Vibrating Sample Magnetometer (VSM) provides the information regarding magnetism of the material. It provides the M-H curve of the sample from which many properties like coercivity and remenance of the material can be obtained FMR is also an advanced technique by which we can find out the some ferromagnetic response of the sample by applying external magnetic field The further explanation of each technique is given below one by one

3.2 Scanning Electron Microscope (SEM)

High resolution images of a sample surface can be created using scanning electron microscope SEM It could also give information about the surface morphology of the specimen It comprises of high energy electron beams The electrons emitted from the electron gun due to the heating of filament using a high voltage The electrons are accelerated by potential difference and then focused onto the sample surface by magnetic lenses SEM has condenser and objective lenses which are used to focus the beam of electrons coming from electron gun 0 4 nm to 5 nm is the range of the focus beam by condenser lens While 0 2 to 40 keV is the range of beam energy A number of signals are obtained by the interaction of beam with the sample These signals are by auger electrons secondary electrons, back scattered electrons and characteristic X-rays There are different types of detectors present in SEM for different signal detection Back scattered electrons are helpful for viewing the image Secondary electrons are used for the topography and surface morphology SEM is a nondestructive technique as no volume loss takes place when the production of X rays with the interaction of electrons takes place But sometimes sample almost become useless after SEM characterization when sample is insulator Actually a conducting layer of gold (Au) sputtered on the surface of sample to make it interacting for the beam coming from electron gun and further focused by the magnetic lenses A Back scattered electrons are used for the elemental distribution in the sample Characteristic X rays are emitted when the beam of electrons removes some inner shell electrons and high energy electrons are caused to fill the inner shell These emitted X-rays are used for the composition of element For the production of picture detected signal and beam position are important parameter The resolution of SEM does not depend on the spot size of electron but rather on the wavelength associated with this fundamental particle SEM has a magnification power from 20 to 50000X and has a resolution of 50 nm to 100 nm Small signals are amplified and displayed on the computer screen

The SEM comprises of mainly following parts

a) Electron gun that emits electrons due to thermionic emission or field emission process

b) Magnetic lenses for controlling and focusing of the electron beam onto the sample

c) Sample chamber in which a sample is placed This chamber is very sensitive to vibrations

d) Detectors for detection of various signals resulting due to the interaction of electron beam and surface electrons of the sample

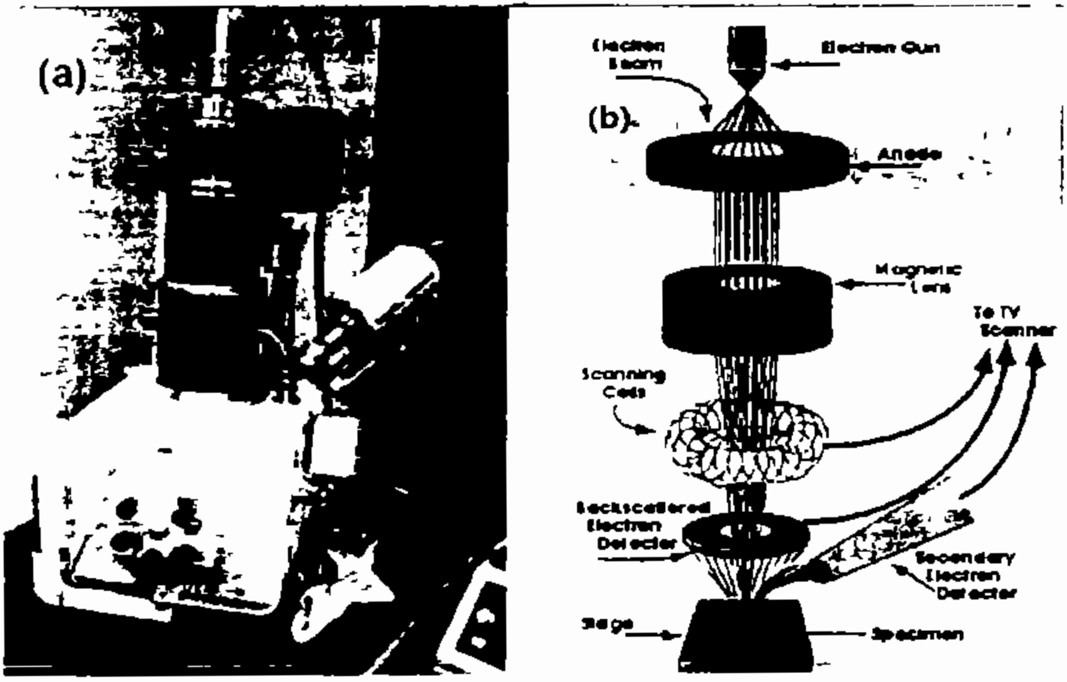


Figure 3.2 (a) Scanning electron microscope (b) Block diagram of Scanning electron microscope

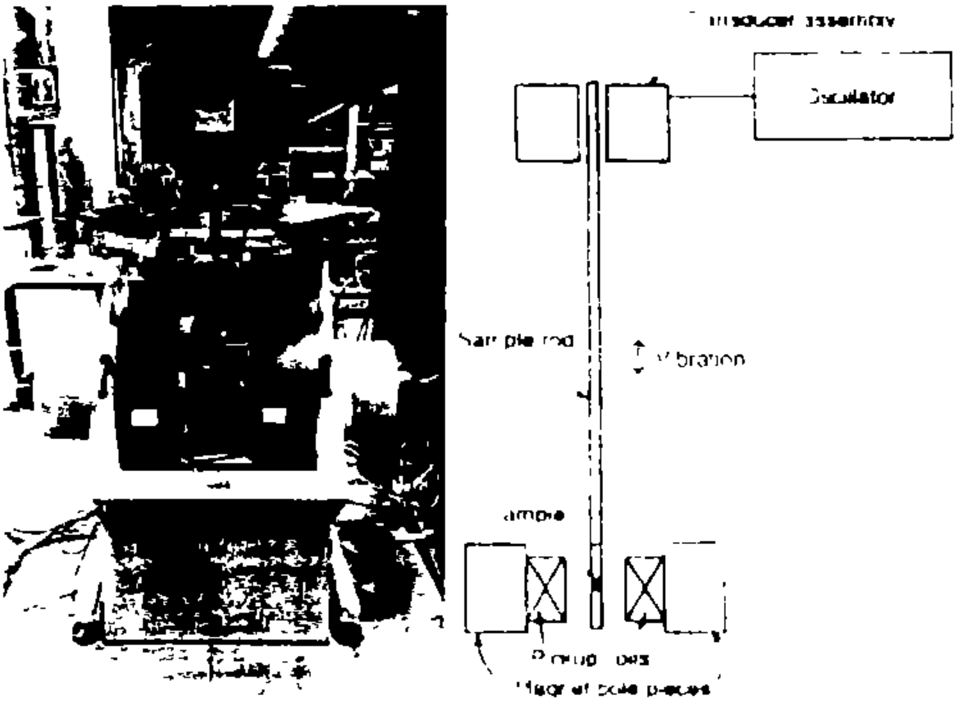
3.2.1 Important precautions regarding SEM

The sample should be moisture free High vacuum is required for the operation of SEM Sample must be conducting for the proper operation of SEM for non-conducting materials the sample surface is made conducting by coating of some conducting material like gold or copper

3.3 Vibrating Sample Magnetometer (VSM)

For the measurements of magnetic materials and magnetic behavior vibrating sample magnetometer is a widely used device The principal of VSM is based on Faradays law of electromagnetic induction which is change in the magnetic field produces an induced electric filed The information about the changing magnetic field is given by this electric field For the characterization of magnetic behavior of e materials VSM is considered to the conventional technique of characterization we record the magnetic moments as a function of applied magnetic field and temperature

VSM is the most common instrument used for the magnetic measurements like hysteresis loops It was developed by S Foner and Van Osterhart



K H J Buschow and F R de Boer *Physics of Magnetism and Magnetic Materials*
Kluwer Academic/Plenum Publishers 2003

Figurer 3 3 Lab view and the schematic diagram of the VSM

A uniform magnetic field is fall on the sample during the measurements Magnetic field is generated by an electromagnet or by a superconducting magnet The samples mounted in the center of uniform magnetic field are oscillated by the vibrator The vibrator is a type of velocity transducer which is like mechanically coupled loud speaker A set of suitable pick up coils detects the magnetic flux changes with the time as the sample is oscillated The electric field is caused in the pick-up coils by the changing magnetic field as stated by Faradays law Magnetization of sample is proportional to the induced current produced in sample Induced current will have a higher value if the sample possesses higher magnetization VSM has a built in amplifier which amplifies the induced current VSM is interfaced with the computer which controls and monitors software that gives information on how the magnetization depends on the constant magnetic fields strength and magnetization value

3.4 Fundamental theory of ferromagnetic resonance

For the study of ferromagnetic materials ferromagnetic resonance FMR is a very useful characterization technique Ferromagnetic resonance is known as the motion of ferromagnetic material magnetization M about the applied external magnetic field H The rf transverse magnetic field hrf absorbs energy in the process of resonance physically which occurs with the match of frequency with precessional frequency

The strength of magnetic field and the orientation of material are responsible for the precession frequency The most important parameters are measured by it that are total magnetic moment, Curie temperature, relaxation mechanism and elementary excitation etc

For the phenomenon of FM resonance the resonance condition is considered to be a single domain magnetic particle having ellipsoid shape The sample is set into microwave cavity with the static applied magnetic field H along z-axis

A resonance is observed at a frequency given by

$$\hbar\,\omega = g\mu B\sqrt{[H + (N_x - N_z)M][H + (N_y - N_z)M]} \qquad (1)$$

Here the demagnetization factors in the directions x, y and z are Nx, Ny, Nz respectively g is the spectroscopic factor known as Lande factor Bohr magneton is represented by

$$\mu B = e\hbar/2\mathrm{me}$$

The geometry of the magnetic material which could be ellipsoid thin film and sphere etc is affected by the demagnetization factor

The classical micro spin vector M magnetization is replaces the spins which is a classical approach to ferromagnetism We can directly obtain the time dependence of magnetization by calculating the torque on M by an **effective** field H_{eff},

$$\frac{dM}{dt} = -\gamma\, M \times H_{eff} \qquad (2)$$

Here γ is the gyromagnetic ratio that is equal to

$$\gamma = g\mu_b h$$

The undamped magnetization precession is represented by equation (2) In finite time the actual changes of magnetization are known to decay from experiments Within several nanoseconds the reversal of magnetization is gained towards the direction of H by the occurrence of a damping mechanism That can be explained mathematically just by the following changes

$$\frac{dM}{dt} = -\gamma M \times \left(Heff - \frac{\alpha}{\gamma Ms}\frac{dM}{dt}\right) = -\gamma M \times H_{eff} + \frac{\alpha}{Ms} M\frac{dM}{dt} \qquad (3)$$

Here α is the dimensionless Gilbert damping constant which is of order 10^{-2} in ferromagnetic thin films After Gilbert introduces the damping term in the equation (3) is known as Landau-Lifshitz-Gilbert equation The sum of applied static field H, the dynamic field (h_{rf}) and internal field (H_{in}) is the total effective magnetic field H_{eff} Magneto crystalline, shape and magneto elastic anisotropies are constituted by internal field Therefore torque acting on M can be described by this equation The rotation of magnetization is lead towards the external magnetic field direction by this torque By applying a dynamic field the decay in precessional motion caused by damping becomes continuous As shown in figure 3 4 to next page

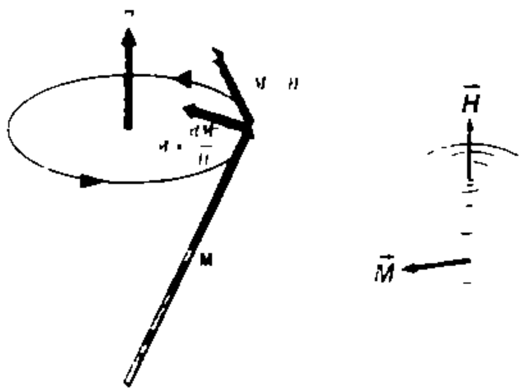


Figure 3 4 (a) Torque components exerted on the magnetization M by rotational field H (b) Motion of M for constant H

3.5 Conventional Ferromagnetic Resonance Flip-chip based technique

The magnetic excitation in the frequency domain measurement is sinusoidal magnetic field h_{rf} and the vector network analyzer detects the response of sample We can apply the magnetic field to the nanowires which satisfy the FMR condition the field can be applied along perpendicular and parallel directions

The fig shows the main components of the setup The coplanar waveguide CPW which have characteristic impedance of 50 ohm is connected to VNA using coaxial cables and microwave connectors Teflon insulated coaxial cables and SMA connectors are often used for such radio frequency connections They have a band width of 18 GHz and are low priced as well The used cables should not have a metallic reinforcement

Material parameters are obtained by flip-chip based measurements, these are important for data storage devices technologically The schematic of flip chip measurement setup is shown in fig magnetic field perpendicular to RF magnetic field and parallel to nano wires is applied, so that resonance condition must be satisfied

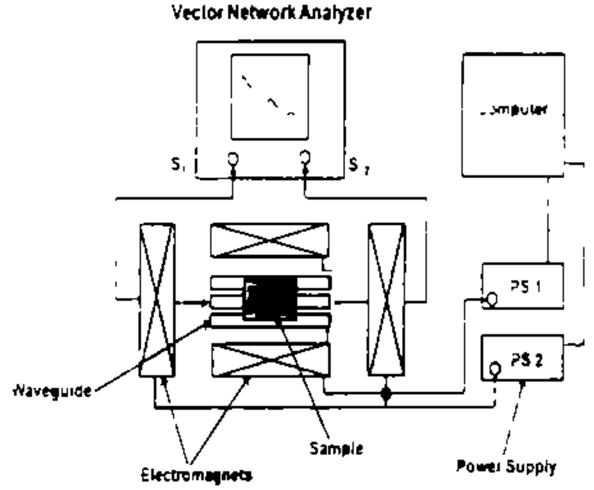


Figure 3 5 Schematic representation of VNA-FMR system perpendicular to the 1-d sample geometry (NWs & NTs) Behind the screen reason is that those angles where the resonance is minimum will be referred as the easy axis The graph 4 5a) and 4 5b) both explains that the easy axis lies at almost at 90 degree By these results we can also predict the shape anisotropy of the sample which is also perpendicular to the both nanowires and nanotubes

$$\frac{dM}{dt} = -\gamma M\times\left(Heff - \frac{\alpha}{\gamma Ms}\frac{dM}{dt}\right) = -\gamma M\times H_{eff} + \frac{\alpha}{Ms}M\frac{dM}{dt} \qquad 4\ 1$$

The above equation is known as "Landau-Lifshitz-Gilbert" α is the Gilbert damping constant" its magnitude or value depends upon the nature of the material Here γ is gyromagnetic ratio" and its value is given below·

$\gamma = ge/2mC = 2\ 21\times10^5$ m A^{-1} s^{-1}

4.3.3 ΔM Curve

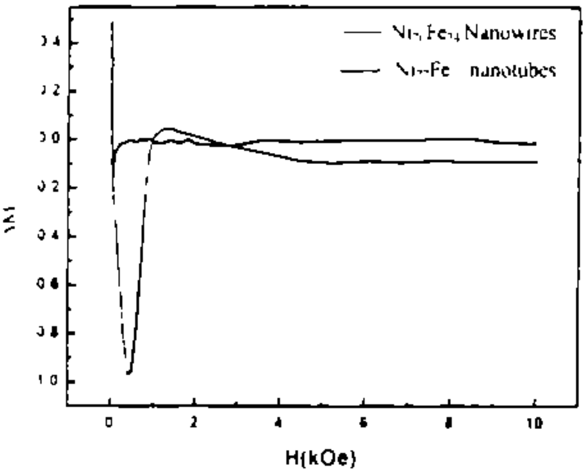


Figure 4 6 ΔM Curve for both NiFe Nanotubes and Nanowires

It is very important to know that what type of magnetic interaction taking place inside the magnetic material, which is important for the future applications like data storage devices In this regard we cannot neglect the worth of ΔM curves There are many other methods to explain the magnetic interactions but relatively the Henkel analysis [83] ΔM curves derived from VSM parameters is relatively more advanced and unsophisticated for the said purpose In order to know about the magnetic interaction of $Ni_{76}Fe_{24}$ Nanowires and $Ni_{77}Fe_{23}$ Nanotubes we also draw a graph as shown in figure 4 6 As we have one-dimensional nano structure that is why depending upon the pore size and their density magnetic interactions are possible

In the Fig 4 6 we observe a deviation from the linear line It can be noted that delta M curves have peak negative values which are typically associated with magnetostatic interactions that tend to destabilize the magnetized state or easy magnetization reversal of the nanowires This is the big evidence about presence of magnetic interactions inside the $Ni_{76}Fe_{24}$ Nanowires and $Ni_{77}Fe_{23}$ Nanotubes The black line shows the ΔM curve of $Ni_{77}Fe_{23}$ Nanotubes while the red line is the ΔM curve of the $Ni_{76}Fe_{24}$ Nanowires Actually ΔM plot can be justified or defined as the deviation from the idealized linear line If the delta M curve goes straight parallel to the ideal line or zero line then it clearly means that there are no particular magnetic interactions inside the material

As we see that the black ΔM curve starts from positive and just reached near to the idealized linear line and then gets parallel with it This indicates that the magnetostatic interactions are very weak inside the $Ni_{77}Fe_{23}$ Nanotubes While on the other hand the red ΔM curve makes a prominent negative peak that indicates the magnetostatic interactions or dipolar interactions between the $Ni_{76}Fe_{24}$ Nanowires These strong dipolar interactions are because of magnetostatic energy of the domains as a function of applied external magnetic field These ΔM result are consistent with that of M-H loops and FMR results in which easy axis is found to lie perpendicular to the nanocylinder axis due to dominance of magnetostatic interactions and weakness of shape anisotropy

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3.5 Conventional Ferromagnetic Resonance Flip-chip based technique

The magnetic excitation in the frequency domain measurement is sinusoidal magnetic field h_{rf} and the vector network analyzer detects the response of sample We can apply the magnetic field to the nanowires which satisfy the FMR condition the field can be applied along perpendicular and parallel directions

The fig shows the main components of the setup The coplanar waveguide CPW which have characteristic impedance of 50 ohm is connected to VNA using coaxial cables and microwave connectors Teflon insulated coaxial cables and SMA connectors are often used for such radio frequency connections They have a band width of 18 GHz and are low priced as well The used cables should not have a metallic reinforcement

Material parameters are obtained by flip-chip based measurements, these are important for data storage devices technologically The schematic of flip chip measurement setup is shown in fig magnetic field perpendicular to RF magnetic field and parallel to nano wires is applied, so that resonance condition must be satisfied

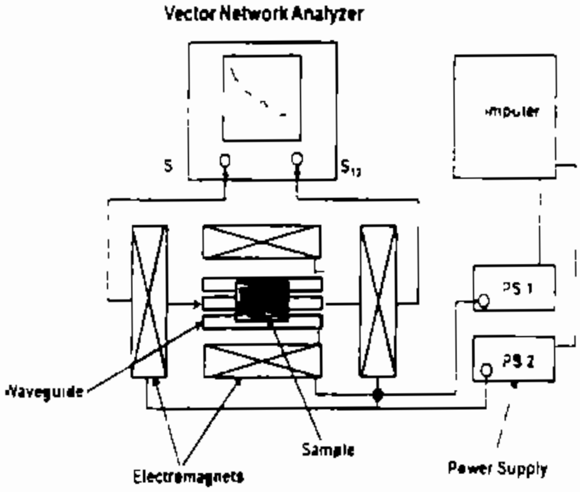

Figure 3 5 Schematic representation of VNA-FMR system

Chapter 04

Results and discussion

4.1 Ni$_{76}$Fe$_{24}$ Nanowires and Ni$_{77}$Fe$_{23}$ Nanotubes

DC electrochemical deposition method was employed to synthesize Ni$_{76}$Fe$_{24}$ Nanowires and Ni$_{77}$Fe$_{23}$ Nanotubes inside the anodized alumina oxide (AAO) templates. Then these nanowires and nanotubes were further characterized by SEM, VSM and FMR to investigate their different properties especially ferromagnetic properties and surface morphology etc. A comprehensive discussion on results of all characterization techniques are given below.

4.2 Surface morphology

The scanning electron microscopy has been used to delve the surface morphology of the NiFe permolly sample but before this to launch the sample inside the vacuum chamber of the SEM it was made sure that the sample is moisture free and conducting. Especially in case of AAO template assisted sample it was dissolved inside the 0.1 M NaOH aqueous solution at temperature T=60°C and Time = 2 hours prior to put into the SEM sample holder. A beam of 20 keV was used to investigate the sample surface. SEM uses the wave nature of the particle to explore the topography of the sample. The SEM images results are shown in the figure 4.1

Figure 4.1 SEM results of nano wires (left image) and nanotubes (right image)

SEM micrographs confirm the uniform and regular structure of nanowires and nanotubes as shown above The length and diameter of nanotubes and nanowires were also measured from the scanning electron microscope The average diameter (D) of the $Ni_{76}Fe_{24}$ nanowires is 50 nm and length (L) of the $Ni_{76}Fe_{24}$ nanowires is 10 um The average diameter (D) of the $Ni_{77}Fe_{23}$ nanotubes is 200 nm and length (L) of the same $Ni_{77}Fe_{23}$ nanotubes is 8um Figure 4 1 also shows a homogeneous and highly ordered growth The length and diameter of each 1-D nano structure can be increased by increasing the deposition time and voltage in electrodeposition method

4.3 Magnetic Characterization

4.3.1 MH loops of nanowires and nanotubes

The magnetic properties of $Ni_{76}Fe_{24}$ Nanowires and $Ni_{77}Fe_{23}$ Nanotubes were investigated by using vibrating sample magnetometer which proves to be highly sensitive to characterize the sample's magnetic properties DMS Model 4HF Vibrating Sample Magnetometer (VSM) of ADE Technology was employed and its sensitivity range lies almost in the range of 10^{-3} emu

The $Ni_{76}Fe_{24}$ Nanowires and $Ni_{77}Fe_{23}$ Nanotubes have prominent magnetic properties Their magnetic properties were explored by hysteresis (M-H) curves as shown in figure 4 2 These (M-H) curves were obtained by applying the external magnetic field at different angles, ranging from 0^0 to 90^0 Easy axis magnetization direction' is one of the most important magnetic parameter that can be found by calculating the remenent squareness (Mr Ms) with the help of graph plotted above in figure 4 2 a) and 4 2 b) for different angles As we have 1-d structures, that is why we have calculated the squareness along the both axes parallel (H at 0^0) and perpendicular (H at 90^0) of the samples As it is clear from the above cited graph of nanotubes, the squareness of M-H loop is much larger along the perpendicular direction On the other hand the sheerness is larger along the axis of the nanotubes Hence consequently we can say that the easy axis of the nanotubes lies perpendicular to their axis

In case of nanowires majestic trend is observed in the saturation magnetization At every angle there will be a fix saturation magnetization (Ms) and corresponding remenent magnetization Mr The Ms and Mr decreases side by side with the decrease of angle At first glance it seems that the easy axis lies along the axis of the nanowires but calculation proves that the squareness is larger along the perpendicular direction of nanowires axis Hence the easy axis of nanowires is along perpendicular to the wire axis

From both M-H graphs (nanowires and nanotubes) a clear cut difference observed regarding easy axis which conforms the shape anisotropic behavior of the nanowires and nanotubes

The direction of magnetic moments reversed as the external magnetic field reverses its direction, is called magnetization reversal There are many magnetization reversal mechanisms inside the magnetic materials and depending upon these mechanisms the magnetic moments reverses its direction with respect to the external field As we observe in case of nanowires that the Hc is inversely proportional to the angle of the external field that s why the magnetic moments reverse because of the curling mode

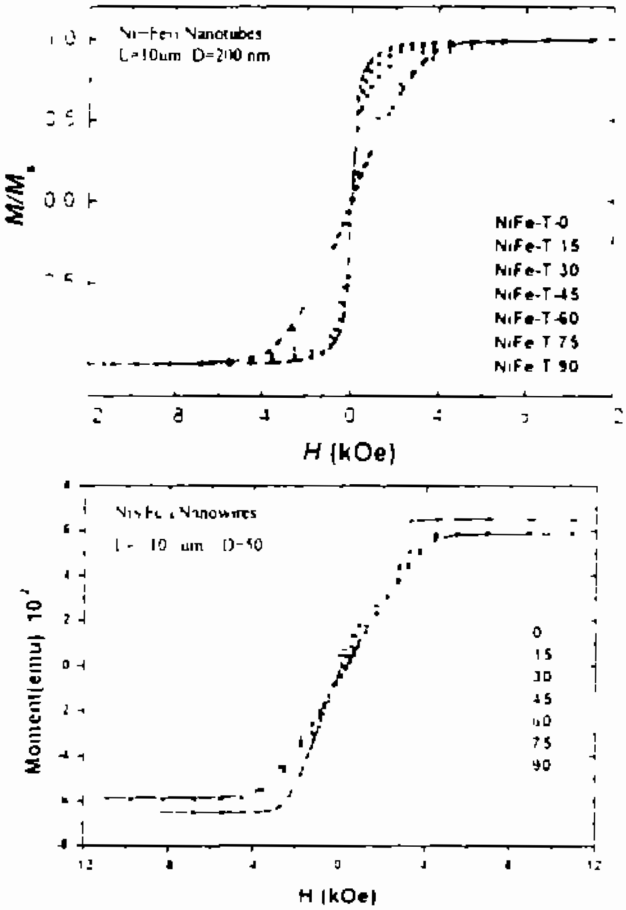

Figure 4 2 a) nanotubes hysteresis (M-H) curve and b) nanowires hysteresis (M-H) curve

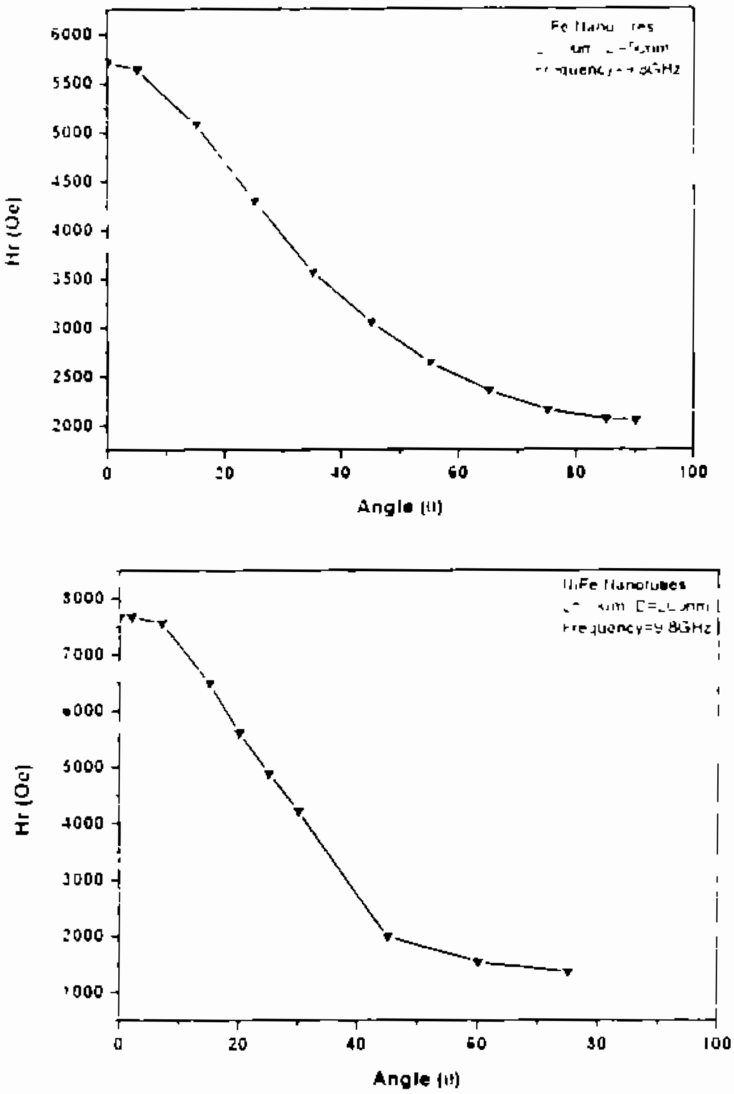

Fig 4 5 a) resonance graph of $Ni_{76}Fe_{24}$ nanowires 4 5b) resonance graph of $Ni_{77}Fe_{23}$ nanotubes

The both samples of $Ni_{76}Fe_{24}$ Nanowires and $Ni_{77}Fe_{23}$ Nanotubes have same lateral length of 10 um but with different diameters as illustrated in the above graph

The magnetic field was applied at various angles from 0 degree to 90 degree as shown in the Fig 4 5 a) resonance graph of $Ni_{76}Fe_{24}$ nanowires 4 5 b) resonance graph of $Ni_{77}Fe_{23}$ nanotubes

The figure shows the angular dependence of resonance field obtained when intensity line cuts x-axis There is a great analogy and consistency of these FMR results with the M-H loops These graphs conforms that the easy axis of nanowires and nanotubes lies in the perpendicular to the 1-d sample geometry (NWs & NTs) Behind the screen, reason is that those angles where the resonance is minimum will be referred as the easy axis The graph 4 5a) and 4 5b) both explains that the easy axis lies at almost at 90 degree By these results we can also predict the shape anisotropy of the sample which is also perpendicular to the both nanowires and nanotubes

$$\frac{dM}{dt} = -\gamma M\times\left(Heff - \frac{\alpha}{\gamma Ms}\frac{dM}{dt}\right) = -\gamma M\times H_{eff} + \frac{\alpha}{Ms}M\frac{dM}{dt} \tag{4 1}$$

The above equation is known as "Landau-Lifshitz-Gilbert" α is the "Gilbert damping constant" its magnitude or value depends upon the nature of the material Here γ is "gyromagnetic ratio" and its value is given below

$\gamma = ge/2mC = 2\ 21\times10^{5}\ m\ A^{-1}\ s^{-1}$

4.3.3 ΔM Curve

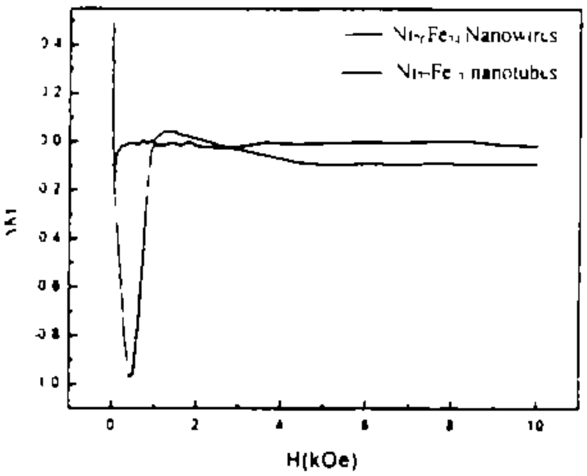

Figure 4 6 ΔM Curve for both NiFe Nanotubes and Nanowires

It is very important to know that what type of magnetic interaction taking place inside the magnetic material, which is important for the future applications like data storage devices In this regard we cannot neglect the worth of ΔM curves There are many other methods to explain the magnetic interactions but relatively the Henkel analysis [83] ΔM curves derived from VSM parameters is relatively more advanced and unsophisticated for the said purpose In order to know about the magnetic interaction of $Ni_{76}Fe_{24}$ Nanowires and $Ni_{77}Fe_{23}$ Nanotubes we also draw a graph as shown in figure 4 6 As we have one-dimensional nano structure that is why depending upon the pore size and their density magnetic interactions are possible

In the Fig 4 6 we observe a deviation from the linear line It can be noted that delta M curves have peak negative values which are typically associated with magnetostatic interactions that tend to destabilize the magnetized state or easy magnetization reversal of the nanowires This is the big evidence about presence of magnetic interactions inside the $Ni_{76}Fe_{24}$ Nanowires and $Ni_{77}Fe_{23}$ Nanotubes The black line shows the ΔM curve of $Ni_{77}Fe_{23}$ Nanotubes while the red line is the ΔM curve of the $Ni_{76}Fe_{24}$ Nanowires Actually ΔM plot can be justified or defined as the deviation from the idealized linear line If the delta M curve goes straight parallel to the ideal line or zero line then it clearly means that there are no particular magnetic interactions inside the material

As we see that the black ΔM curve starts from positive and just reached near to the idealized linear line and then gets parallel with it This indicates that the magnetostatic interactions are very weak inside the $Ni_{77}Fe_{23}$ Nanotubes While on the other hand the red ΔM curve makes a prominent negative peak that indicates the magnetostatic interactions or dipolar interactions between the $Ni_{76}Fe_{24}$ Nanowires These strong dipolar interactions are because of magnetostatic energy of the domains as a function of applied external magnetic field These ΔM result are consistent with that of M-H loops and FMR results in which easy axis is found to lie perpendicular to the nanocylinder axis due to dominance of magnetostatic interactions and weakness of shape anisotropy

4.4 M-H loop at different temperatures

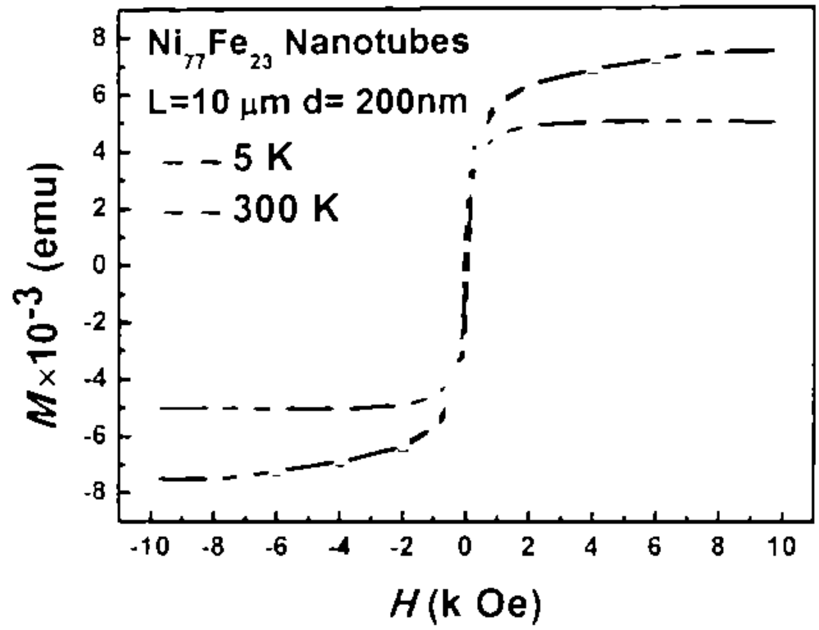

Figure 4 7 M–H curves of NiFe NTs at temperatures T = 5 K and 300 K

In order to see the behavior of such nanotubes at low temperature the M-H curve at T=5K were taken while the lateral length of nanotubes is approximately 10 um with diameter of 200 nm and compared with that of T=30K It is very clear from the above cited graph that a drastic change of pattern observed with the change of temperature from 300 K to 5 K Actually this may be the settlement of the magnetic moments towards the such crystallographic axis which are attributed to the easy axis of the said material of the NTs i e NiFe ($Ni_{77}Fe_{23}$ Nanotubes) At low temperature the magnetic moments of the different domains are arranged in singular direction because of low thermal energy consequently less external magnetic field required for its magnetization The another aspect from the same phenomena is that the resultant response of the material regarding saturation magnetization is much higher at low temperature as compared to the higher one, on the same magnetizing external field This thing also leads towards the minimization of magneto-crystalline energy due to which high saturation of magnetization produced at low temperature on the same external magnetizing field [85] as shown in figure 4 7

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