HEATING POWER OF COATED FeCoV MAGNETIC NANOPARTICLES

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science

By

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ABSTRACT

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The effect of coatings on the heating curves of magnetic nanoparticles was studied in this Thesis. Iron cobalt vanadium (FeCoV) nanoparticles with oxide (CoO-Fe₂O₃) and graphite (C) coating to prevent oxidation, were synthesized through thermal plasma processing method. The magnetic nanoparticles had an average diameter of 30 nm. In the case of FeCoV with oxide coating, the initial diameter of the pure FeCoV nanoparticles on average was 30 nm but after oxidation 5 nm thick oxide layer was created. As a result of this oxidation, the magnetic core of FeCoV nanoparticles was shrunk from 30 nm to 20 nm in diameter. Graphite coating with a thickness of 10 nm was added uniformly to 30 nm in diameter of FeCoV nanoparticles. These magnetic nanoparticles were exposed to an ac applied magnetic field and their heating responses were measured. The measurements were done at frequency of 175 kHz and intensity of the magnetic field with different current values of 5 A, 10 A, and 15 A. The heating performance of magnetic nanoparticles described by Specific Absorption Rate (SAR) was calculated by finding the initial slope of the heating curve with respect to time. It was found that the maximum value of SAR was obtained when applied frequency and current were at 175 kHz and 15 A, respectively. Results were analyzed to find the coating effect on the heating rate. The most significant conclusion based on our research is to see that FeCoV magnetic nanoparticles with graphite coating had higher heating power than FeCoV with oxide coating.
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DEDICATION

I lovingly dedicate this Thesis to my parents Mr. Ali Alshammari and Mrs. Sara Ali Fares for their endless love, support and encouragement. I also dedicate this Thesis to King Abdullah bin Abdulaziz Al Saud with a special feeling of gratitude to him.
I. INTRODUCTION

The magnetic properties are of great importance in the advancement of the nanoparticles technology specifically in areas like engineering and biomedical sciences. One of the primary advances that have taken place in the nanomaterials recently is based on the usage of the coating in molding their specific properties during the process of magnetic nanoparticles’ fabrication. To explore these unusual properties of magnetic nanoparticles scientists developed various coating techniques. It appears that besides surface coating the other parameters such as nanoparticle size, surface to volume ratio, morphology and so on are playing important role in their potential applications [1].

In the past few years, scientists have come up with different methods that can be used to synthesize the magnetic nanoparticles. With better understanding of these syntheses more sophisticated methods of coating nanoparticles were discovered. For example, in order to ensure stability and to prevent agglomeration due to the nanoparticles’ interaction the surface coating of nanoparticles is required [2]. For a nanocrystalline structure to be able to operate at elevated temperatures, grain growth must be minimized. It was found that a highly resistive secondary stable phase at the grain boundaries is necessary to control the grain growth. However, this resistive coating should be thin enough to allow for magnetic coupling to exist between neighboring grains [3].

GOALS OF MY THESIS

The main aim of this research is to give experimental and theoretical explanation of the coating effect (oxide or graphite) on the heating rate and heating power of the soft magnetic iron cobalt vanadium (FeCoV) nanoparticles subjected to externally applied ac magnetic field. The coating layer of oxide or graphite preserves above all crystallinity and physical properties of FeCoV soft
magnetic nanoparticles. FeCoV magnetic nanoparticles used in this study were prepared by thermal plasma processing method (RF plasma torch) with a slight modification to produce two different coating layers: oxide and graphite on the surface of magnetic nanoparticles. The heating power (SAR) of nanoparticles and effects of intensity and frequency of ac magnetic field externally applied in the system including coating effect were studied. The reasoning of my Thesis was to find the relationship between coating compositions and their corresponding heating powers in addition to the theoretical understanding of mechanisms behind it.

**CHAPTERS SUMMARY**

Chapter I (Introduction) outlines on the importance of magnetic nanoparticles and goals of the Thesis research are explained.

Chapter II (Introduction to Magnetism) reviews fundamental concept of magnetism and introduces different types of magnetic order.

Chapter III (Heating Mechanisms in Ferromagnetic Nanoparticles) reviews hysteresis and relaxation loss mechanisms of heat creation by nanoparticles placed in ac applied magnetic field.

Chapter IV (Synthesis and Characterization of Ferromagnetic Nanoparticles) describes the synthesis procedures and some properties for the FeCo and FeCoV with and without coating.

Chapter V (FeCo and FeCoV System) summarizes physical properties of FeCo and FeCoV.

Chapter VI (Experimental Setup for Measurement of Heating Rate and Specific Power Loss) outlines experimental setting and presents the procedure used for the heating of magnetic nanoparticles in the presence of externally applied alternating magnetic field.

Chapter VII (Experimental Results) calculates the heating rate from the initial slope of temperature versus time and summarizes the results of the experiment.
Chapter VIII (Discussion) describes the heating performance of the magnetic nanoparticles in terms of the specific absorption rate (SAR) and analyzes the results from all the performed experiments.

Chapter IX (Conclusion and Future Goals) summarizes the important results and what was achieved during this study. Also, suggests ideas for future research.
II. INTRODUCTION TO MAGNETISM

All matter [4] responds to a static, external magnetic field $\mathbf{B}$ by producing a magnetic field of its own, $\mathbf{B}_{\text{self}}$ (Eq.(1))

$$\mathbf{B}_{\text{tot}} = \mathbf{B} + \mathbf{B}_{\text{self}}$$  \hspace{1cm} (1)

Far away from matter, $\mathbf{B}_{\text{self}}$ is dipolar in nature (it can be characterized by a macroscopic magnetic dipole moment $\mathbf{m}$).

The magnetic moment of

- a **paramagnet** points parallel to $\mathbf{B}$,
- a **diamagnet** points anti-parallel to $\mathbf{B}$.

When the external field is removed, the induced magnetic moment vanishes except in a ferromagnetic material, which is a special case of a paramagnet where $\mathbf{m}$ remains non-zero when $\mathbf{B} = 0$. A superconductor is a special case of diamagnetic material where $\mathbf{B}_{\text{tot}} = 0$ inside the superconductor (see, Fig. 1).

![Diagram](image)

**PARAMAGNET**  
**SUPERCONDUCTOR**  
**DIAMAGNET**  
**$B = 0$, FERROMAGNET**

Fig. 1. Response of paramagnet, superconductor, diamagnet, and ferromagnet to an external magnetic field $\mathbf{B}$. $\mathbf{m}$ is the induced magnetic moment.
MAGNETIZATION

Magnetization is a function $M$ used to characterize the details of rearrangement of internal currents that occurs when matter is exposed to an external magnetic field $B$. The external field which initiates the magnetization process in the matter is produced by external source referred as a free current density $j_f$. The source of $B_{self}$ is called magnetization or bound current density $j_m$ leading to the total current density $j$ (Eq.(2))

$$j = j_m + j_f$$

(2)

Experiments show that there are two distinct sources of magnetization current density on the microscopic scale of matter:

- the orbital motion of electrons around nuclei (responsible for the magnetism in diamagnets and superconductors),
- the rotational motion (spin) of electrons, protons, and neutrons (responsible for the magnetism in paramagnets and ferromagnets).

Classical electrodynamics defines a magnetization $M$ and a magnetization current density $j_m$ for both orbital and spin magnetism.

SPIN MAGNETIZATION

The magnetic fields produced by e.g., horseshoe magnets are caused by the cooperative alignment of electron spins, which behave as a point-like magnetic dipoles in ferromagnetic matter. An array of $N$ electrons with spin magnetic moments $m_k$ located at positions $r_k$ are characterized by a spin magnetic moment per unit volume, or spin magnetization, Eq.(3)

$$M_s = \sum_{k=1}^{N} m_k \delta(r - r_k)$$

(3)
The magnetic field produced by the spin magnetization (3) is the same as the magnetic field created by spin magnetization current density (Eq.(4))

\[ \mathbf{j}_s = \nabla \times \mathbf{M}_s \quad (4) \]

For magnetic sample bounded by a sharp surface, a surface magnetization current density appears in connection with the presence of the surface spin magnetization (Eq.(5))

\[ \mathbf{K}_s = \mathbf{M}_s \times \mathbf{n} \quad (5) \]

where \( \mathbf{n} \) is a local outward normal unit vector to the surface of the magnet.

**ORBITAL AND TOTAL MAGNETIZATION**

By analogy to spin \( \mathbf{j}_s \) and \( \mathbf{M}_s \), the existence of closed internal current loops defines an orbital magnetization current density \( \mathbf{j}_0 \) or \( \mathbf{K}_0 \) (volume or surface, respectively) and an orbital magnetization \( \mathbf{M}_0 \). In Eqs.(4) and (5) if subscript \( s \) is replaced by \( 0 \), relationships between orbital current density and magnetization looks very similarly. In summary, the total magnetization is the sum of the spin and orbital magnetization, respectively (Eq.(6))

\[ \mathbf{M} = \mathbf{M}_s + \mathbf{M}_0 \quad (6) \]

And the macroscopic magnetic field of a magnetized sample is produced by macroscopic magnetization current density (Eqs.(7)-(8))

\[ \mathbf{j}_M = \nabla \times \mathbf{M} \quad (7) \]

\[ \mathbf{K}_M = \mathbf{M} \times \mathbf{n} \quad (8) \]
The integral of the total magnetization $M$ over the volume of a magnetized object defines the magnetic dipole moment of the object (Eq.(9))

$$ m = \int d^3 r \, M $$

(9)

**LINEAR MAGNETIC MATERIALS**

The magnetization of a simple magnetic materials with both linear and spatially isotropic properties obeys the following constitutive relation (Eq.(10))

$$ M = \chi_m \, H $$

(10)

where $\chi_m$ is called the magnetic susceptibility and $H$ defines the auxiliary vector field produced by free current density $j_f$ (Eq.(2)), namely, $j_f = \nabla \times H$. The total magnetic field $B$ produced by the total current density $j = j_M + j_f$ leads together with Eq.(10) to the definition of magnetic permeability $\mu$ and relative permeability $\kappa_m = \mu/\mu_0$ (Eq.(11))

$$ B = \mu_0 (H + M) = \mu \, H = \kappa_m \mu_0 \, H = \mu_0 (1 + \chi_m) \, H $$

(11)

Materials where the magnetic field $H$ induces a magnetization $M$ parallel to $B$ are called paramagnets ($\chi_m > 0$) or diamagnets for which $M$ is antiparallel to $H$ ($\chi_m < 0$). Typical values of magnetic susceptibilities are quite small for paramagnets and diamagnets: $|\chi_m| \sim 10^{-4}$ - $10^{-6}$.

**PREMANENT MAGNETIC MATERIALS**

The certain magnetic materials known as ferromagnets do not obey the linear constitutive relation Eq.(10). Soft ferromagnets in a small magnetic field behave as simple paramagnets with a value $\mu \geq 10^4$. Hard ferromagnets exhibit the non-reversible and non-single-valued behavior in
relation between $M$ and $H$ which is called magnetic hysteresis (Fig.2). For $H = 0$, the value of magnetization is zero and it follows a highly non-linear magnetization curve when the magnetic field starts to increase. The saturation value of magnetization $M_{\text{sat}}$ occurs when all the magnetic moments in the ferromagnet point in the same direction and are parallel to $H$. When $H$ decreases to zero, the ferromagnet does not follow its initial magnetization curve and a macroscopic remanent magnetization $M_R$ is established defining the fundamental property of a permanent magnet. The magnetization can be driven to zero at the value of magnetic field $H = H_c$ (coercivity) and it can saturate again by applying $H$ in the opposite direction further. Function of $M$ versus $H$ traces out the closed hysteresis loop sketched in Fig. 2. The most important factor contributing to this loop is the creation and motion of magnetic domains.

Fig. 2. Typical hysteresis loop for a ferromagnet.
MAGNETIC DOMAINS

Ferromagnetic materials in order to minimize its energy break up into an array of macroscopic regions called magnetic domains. The magnetic moments within domains are aligned along a certain crystallographic lattice due to anisotropy but the direction of \( \mathbf{M} \) is not necessarily the same in every domain. E.g., in Fig. 3, the four-domain arrangement is energetically preferable to the single-domain state of uniform magnetization with \( \mathbf{M}_{\text{sat}} \) at every point in the sample.

![Diagram](image)

a) b)

Fig. 3. Two possible magnetic domain patterns: a) multi-domain and b) single-domain ferromagnet.

The shape of hysteresis curve \( \mathbf{M}(\mathbf{H}) \) for multi-domain magnet is determined by the field-induced (an external magnetic field \( \mathbf{H} \)) movement of the walls (Bloch) between adjacent magnetic domains. The example of the magnetization process for ferromagnets where only two directions of easy magnetization are allowed is depicted in Fig. 4.
Fig. 4. Magnetic domains evolution during magnetization process.
III. HEATING MECHANISMS IN FERROMAGNETIC NANOPARTICLES

Heat in magnetic nanoparticles under externally applied ac magnetic fields can be attributed, depending on size, to one of two following mechanisms. When a material's magnetic dimensions approach the nanoscale range, its behavior can be changed and the heating mechanism is affected as well. The ferromagnetic nanoparticles placed in an alternating magnetic field convert the energy of this field into heat due to the following mechanisms [5]:

- hysteretic loss in bulk and multi-domain ferromagnetic nanoparticles,
- relaxation losses in single-domain.

HYSTERESIS LOSSES

Ordinary ferromagnetic nanoparticles exhibit regions of uniform saturation magnetization known as magnetic domains, isolated by domain walls. Domains structures exist to minimize the general magnetostatic energy of the material. When the sample dimension decreases to the nanoscale level, magnetic domains are shrinking together with domain walls but in such a manner that creation of domain walls is no longer energetically favorable. As a result of it, magnetic nanoparticle turns out to be in a shape of single domain. Hysteresis loss is present in single-domain and multi-domain nanoparticles. The magnetic nanoparticle has a tendency to rotate toward externally applied magnetic field. More often, the domain walls can move in the magnetic field in a way that some domains with the orientation closer to direction of magnetic field consolidate to one bigger domain to minimize magnetic energy of the nanoparticle. In other words, domains whose magnetic moments are positively arranged with respect to the applied magnetic field are replacing domains whose magnetic moments are oppositely oriented with regards to the magnetic field. The saturation magnetization (\(M_{\text{sat}}\)) of the magnetic nanoparticles is achieved when the domain walls are dislodged. This domain wall displacement is shown in Fig. 5.
Fig. 5. Domain wall displacement in the presence of applied magnetic field.

The relationship between magnetization and applied magnetic field (hysteresis curve) is shown in Fig. 6.

Fig. 6. Hysteresis loop.

This magnetization curve like this is known as hysteresis loop for all strengths of applied magnetic field. When all domains are parallel to one another in the same direction under
extremely large magnetic field applied to the nanoparticle, saturation magnetization is reached. We call this initial magnetization curve as a virgin curve. Because of hysteretic properties of the materials, demagnetization process will take an alternative way at which a magnetization curve will lie over the initial virgin magnetization. Magnetization curve will intersect axis at the value known as remanent magnetization (\(M_R\)) or remanence (retentivity) when magnetic field is zero. Magnetization will be zero at the coercive power or coercivity (\(H_C\)) if a magnetic field is applied in an inverse direction. The width of the enclosed area of the loop characterizes the amount of hysteresis associated with the coercivity of the material. The magnetic field inside the material does not come back to its original value but rather falls behind the externally applied field. This procedure results in loss of energy by magnetic nanoparticle in the form of heat. This loss is because of the erosion of magnetic domains while altering direction. In reality, the magnetization of the material incorporates energy which is partially restored in the process of demagnetization. The measure of energy produced during the process of magnetization and demagnetization is straightforwardly identified with the region of the hysteresis loop.

The area of hysteresis loop \(A\) is given by Eq. (12)

\[
A = \oint H dB
\]  

(12)

where \(H\) is the magnetic field and \(B\) is the magnetic induction, \(B = \mu_0 (H + M)\), \(\mu_0\) is the permeability of free space and \(M\) is magnetization. By rearranging Eq.(12), we have

\[
A = \mu_0 \oint H d(H + M) = \mu_0 \oint H dM
\]  

(13)

This area is equal to the amount of energy dissipated. If the process of magnetization and demagnetization is repeated \(f\) times per second, we will get hysteresis power loss \(P\) (\(f\) – frequency)

\[
P = \mu_0 f \oint H dM
\]  

(14)

where \(f\) is the frequency of the magnetic field.
NEEL AND BROWN RELAXATION LOSSES

There are two sorts of relaxation losses which happen in superparamagnetic nanoparticles and lead to heat generation. These losses fall into two types: Brown and Neel losses (see Fig.7). The Brown relaxation describes the rotational frictional contribution in a given medium when the nanoparticle tries to rearrange to the externally applied ac magnetic field. As the entire molecule tries to move towards the field, the suspending medium resists this rotational movement which brings expected heat. The time required by the magnetic nanoparticle to react to the externally applied magnetic field is called the Brown relaxation time \( \tau_B \) given by Eq.(15)

\[
\tau_B = \frac{3\eta V_H}{k_B T}
\]

where \( \eta \) is the fluid viscosity and \( V_H \) is the hydrodynamic volume of the nanoparticle (including coatings).

Fig. 7. Brown and Neel relaxation processes.

The Neel relaxation describes the rotation of the individual magnetic moments toward the external magnetic field. The magnetic moment rotates away from the crystal easy axis towards the field to minimize potential energy when an external magnetic field is applied. The remaining
energy is then discharged as heat into the system. It infers that Neel relaxation is due to the internal rotation of the nanoparticle’s magnetic moment. The typical time for orientation changes is known as Neel relaxation time $\tau_N$ given by Eq.(16). Neel relaxation time $\tau_N$ can happen when nanoparticle movement is blocked

$$\tau_N = \frac{\sqrt{\pi}}{2} \tau_0 \frac{K V}{e^{K V / k_B T}}$$

where $\tau_0$ is the attempt time (generally $10^{-9}$ sec), $V$ is the volume of ferromagnetic nanoparticle, $k_B$ is Boltzmann’s constant, $K$ is the nanoparticle anisotropy constant, and $T$ is the absolute temperature. As two relaxation processes are occurring simultaneously, there is an effective relaxation time given by $\tau$ (see, Eq. (17)).

$$\tau = \tau_N \tau_B / (\tau_N + \tau_B)$$

From above equations, it is clear that Brown relaxation time varies with the radius of the nanoparticle in third power and fluid viscosity coefficient in the first power while Neel relaxation time depends on the volume of the nanoparticle ($V$) and on the magnetic anisotropy energy ($K$). Relative relaxation mechanisms for magnetite ($K = 43$ kJ/m$^3$) nanoparticles in water (low viscosity $\eta = 0.0009$ kg/m•sec) are represented in Fig.8. The effective relaxation time is represented by a blue line while time for Neel relaxation and Brownian relaxation are represented by red and dashed line, respectively. The crossover between Neel and Brown regime corresponds to maximum value of heat (known as specific absorption rate (SAR)) which occurs roughly at a critical radius of the order 5.5 nm and relaxation time is of the order of $10^{-6}$ sec.
Fig. 8. Effective relaxation time for iron as a function of the radius of the nanoparticles in water with a 2 nm magnetic coating at $T = 300 \, \text{K}$ [6].

We can see from Eq. (16) that Neel relaxation time depends on the anisotropy constant. In order to maximize the heating of the nanoparticles, we need to change core size of nanoparticle only. In general, because heating response has a linear dependence on viscosity coefficient of surrounding medium, an increase in its viscosity produces longer Brown time constant. As a result, Brownian contributions to heating process can be completely eliminated. The total volumetric power generation $P$ is given by Eq. (18)

$$P = \mu_0 \chi'' \pi H_0^2 f$$  \hspace{1cm} (18)

where $H_0$ is the intensity of ac magnetic field, $f$ is the frequency of the applied field and $\mu_0$ is the magnetic susceptibility of vacuum. The frequency dependence of nanoparticle relaxation time can be given through the complex susceptibility. The magnetic susceptibility is $\chi = \chi' - i \chi''$.
with $\chi'$ is the in-phase (real) component and $\chi''$ is the out-of-phase (imaginary) component. This susceptibility is described by the following Eq. (19)

$$\chi'' = \chi_0 f \tau/[1 + (f\tau)^2]$$

where $\chi_0 = M_{sat}^2 V/k_B T$ is static susceptibility. At low frequencies ($f\tau<<1$), i.e., in the superparamagnetic regime, the losses increase with the square of frequency as given by Eq. (20)

$$P = \mu_0 \chi_0 \pi H_0^2 f^2 \tau$$

while for ($f\tau >> 1$) losses saturate and become independent on frequency Eq. (21)

$$P = \mu_0 \chi_0 \pi H_0^2 / \tau$$

The specific absorption rate (SAR) for monodisperse nanoparticles (no variation in size) is equal to volumetric power generation $P$ given by Eq. (22)

$$P = \mu_0 \pi H_0^2 \{ \chi_0 f \tau/[1 + (f\tau)^2] \}$$

This is the heat delivered to the medium where the ferromagnetic nanoparticles are submerged. The size dependence of the relaxation time leads to a maximum of the SAR. It is important to stress that large heating power output occurs only for magnetic nanoparticles with well-defined size distribution (and anisotropy) and with the mean diameter properly adjusted to the applied frequency [7]. Therefore, the highest SAR can be achieved only for careful adjustment of magnetic field parameters (frequency $f$ and amplitude $H_0$) with appropriate properties (size and anisotropy) of nanoparticles used in experiment.
IV. SYNTHESIS AND CHARACTERIZATION OF FERROMAGNETIC NANOPARTICLES

Magnetic nanoparticles are offering great potential application in engineering and biomedicine. The magnetic properties of nanoparticles can be used as data storage application in electronics. Iron oxide nanoparticles are commonly used for biomedical applications due to their biocompatibility and biodegradability and also as magnetic carriers for drug delivery and cell separation [1][8].

Because the iron oxide nanoparticles have a relatively low saturation magnetization ($M_S$) in comparison to transition metal magnetic nanoparticles, they have to be larger in diameter [9]. Unfortunately, the use of larger nanoparticles can cause a problem such as an embolism and damage to blood vessels especially if they aggregate [10]. So, it is preferable to use smaller nanoparticles that have enhanced magnetic properties [8]. However, Co or Fe nanoparticles are hard to acquire in view of their sensitivity to oxidation in the environment, which diminish their magnetic properties [11]-[12]. As a result, magnetic FeCo alloy nanoparticles become particularly interesting because of their high saturation magnetization and resistance against oxidation [8]. Moreover, FeCo alloy is even more desirable due to the exceptional properties, which includes a high saturation magnetization, high permeability, and high Curie temperature. These particular properties of FeCo nanoparticles are being used in highly sensitive magnetic sensors [13] and magnetic carriers [14], and they have excellent catalytic properties [1][15].

There are various developed methods for synthesis of magnetic nanoparticles such as microemulsions, sol–gel synthesis, hydrothermal reaction, and thermolysis of precursors, and others [16]. Synthesis of magnetic nanoparticles is a complex process, so every method has a specific procedure and conditions [17].
One of the best definition of microemulsions has been presented by Danielsson and Lindman [18]. According to them microemulsion is a system of water, oil and an amphiphile which is a single optically isotropic and thermodynamically stable liquid solution. The aqueous phase may contain salt(s) and/or other ingredients, and the "oil" may actually be a complex mixture of different hydrocarbons and olefins. There are three basic types of microemulsions which are direct (oil dispersed in water), reversed (water dispersed in oil) and bicontinuous (Fig. 9).

![Fig. 9. Possible microemulsion structures, (a) spherical w/o, (b) spherical o/w, and (c) bicontinuous [19].](image)

The sol-gel process is a wet-chemical technique that uses either a chemical solution (short for solution) or colloidal particles (nanoscale particle) to produce an integrated network (gel). The sol-gel process may be described as follows: it is a formation of an oxide network through polycondensation reactions of the molecular precursors (metal alkoxides and chlorides) in a liquid. They undergo hydrolysis and polycondensation reactions to create a colloid which is a system composed of nanoparticles dispersed in a solvent. The sol changes then towards the formation of an inorganic network incorporating a liquid phase (gel). Formation of a metal oxide connects the metal centers with oxo (M-O-M) or hydroxo (M-OH-M) bridges resulting in metal-oxo or metal-hydroxo polymers in solution. After a drying, the liquid phase is removed from the
gel. Then, the calcination favors further polycondensation and increases mechanical properties (Fig. 10).

Fig. 10. Schematics of sol-gel process [20].

K. Byrappa in 2001 [21] defined hydrothermal reaction as “any heterogeneous chemical reaction in the presence of a solvent (whether aqueous or non-aqueous) above room temperature and at pressure greater than 1 atm in a closed system.” Hydrothermal synthesis is a method to produce materials by using physical and chemical processes taking place in aqueous solutions at temperatures above 100°C and pressures above 1 atm. The method is based on the ability of aqueous solutions including water to dilute at temperature of 500°C and pressures (up to 300 MPa) substances insoluble under normal conditions: some of them are oxides, silicates, and sulfides. The synthesis is carried out in autoclaves which are sealed steel cylinders that can withstand high temperature and pressure for a long time [22]-[23].
One of the most important processes to make magnetic nanoparticles is thermal decomposition, or thermolysis, which is a chemical decomposition caused by heat. The reaction is usually endothermic as heat is required to break chemical bonds in the compound undergoing decomposition. As an example to fabricate bimetallic FeCo nanoparticles reductive decomposition of Fe (III) acetylacetonate (Fe (acac)₃) and Co(II) acetylacetonate (Co(acac)₂) in a mixture of surfactants and 1,2-hexadecanediol (HDD) under a gas mixture of 93% Ar + 7% H₂ at 300 °C is used [24].

Fig. 11. Thermal decomposition setup for producing magnetic nanoparticles.
SYNTHESIS OF C-COATED AND OXIDE-COATED FeCo/FeCoV ALLOY NANOPARTICLES

The FeCo alloy has been selected because of their higher magnetic induction value and high transformation temperature from magnetic to non-magnetic phase. To maintain stability of FeCo nanoparticles to different coatings were used. First, carbon coated (C-coated) FeCo [C] nanoparticles, starting with a mixture of Fe-Co metal precursors, were produced by using a radio frequency (rf) plasma torch where carbon (C) source was acetylene used as a carrier gas [25]. A rf plasma system (Fig. 12) consists of a TEKNA PL 50 type plasma torch head and a gas expansion-reaction vessel connected to a filtering unit.

![Diagram](image.png)

**Fig. 12.** (a) A detailed schematic representation of plasma torch head. (b) The rf plasma torch reaction with reaction chamber and porous metal [3].

A 50 kW, 3 MHz power supply (lepel) was used. Argon gas flowing at 40 standard liters per minute (slpm) was used as plasma gas and the sheath gas was consisted of 80 slpm Ar mixed with 9 slpm of hydrogen. A 6–10 mm Fe powder and 1.6 mm Co powder mixture was used as a starting feedstock and injected axially into the plasma stream through an injection probe with the
aid of a screw driven powder feeder and 3 slpm Ar as a carrier gas. Acetylene was introduced into the carrier gas and powder mixture between powder feeder and injection probe as a carbon source for coating purposes [26].

Secondly, to guarantee stability also the oxide coating of FeCo nanoparticles were produced as an alternative by using the same technique with carbon coating which is the rf plasma torch method. During the process of making these nanoparticles, starting with a mixture of Fe-Co metal precursors, the controlled amount of air was introduced into the plasma gas mixture as an oxidation agent in order to synthesize it and to control a thickness of the protective oxide layer. The plasma power was kept at 50 kW and argon flowing at 40 slpm was used as the plasma gas while the sheath gas consisted of 80 slpm of Ar mixed with 9 slpm of hydrogen. The pressure in the reactor was maintained at 250 Torr during the synthesis.

Due to further improvement of magnetic properties as far as coercivity is concerned of FeCo nanoparticles on initial stage of their fabrication (plasma torch synthesis), Fe and Co metallic precursors were mixed together with vanadium powder [3].
V. FeCo AND FeCoV SYSTEM

Relatively low ductility iron-cobalt based alloys exhibit interesting magnetic properties which characterized by high Curie temperature, high saturation magnetization and permeability, and also low hysteretic losses. These alloys discovered by Elmen in 1929 [27] are expensive, so their applications were restricted to the cases where a small volume and high performances were critical. The discovery of the FeCo-2V alloy in 1932 by White and Wahl [28] was an important step in achieving an alloy of industrial acceptance. Few new compositions with solute additions tended to reduce the saturation magnetization resulting in general increase of its coercivity leading to degradation of its magnetic performances. To date the most remarkable role of vanadium (V) in the FeCo-X system is its improvement of the ductility of the equiatomic alloy. The microstructural aspects of FeCo alloys in an attempt of better understanding of magnetic or mechanical properties are key factors to study V role in this system.

A near-equiaatomic FeCo alloys are bcc at low temperature and fcc at temperature above \( \sim 983^\circ C \). The bcc phase orders to a B2 structure at temperatures below \( \sim 730^\circ C \). Phase equilibria in the FeCo system are represented by the phase diagram in Fig. 13. Equiatomic FeCo alloys have a bcc structure (\( \alpha \) in Fig. 13) below \( \sim 983^\circ C \) and B2 structure (\( \alpha_2 \) in Fig. 13 below \( \sim 730^\circ C \)), with a lattice parameter given by [29]

\[
\begin{align*}
\alpha_a &= 0.28236 \text{ nm} \\
\alpha_{a2} &= 0.28250 \text{ nm}
\end{align*}
\]

and a fcc one (\( \gamma \) in Fig. 13 above \( \sim 983^\circ C \)), with a lattice parameter given by:

\[
\gamma_t = 0.35438 \text{ nm}
\]
Fig. 13. Fe-Co binary phase diagram [30].

The FeCoV system in regions of interest for compositions close to equiatomic FeCo with vanadium additions reduce both the $\alpha/\alpha + \gamma$ and $\alpha + \gamma/\gamma$ transition temperatures (see Fig. 14).

Fig. 14. The $\alpha/\alpha + \gamma$ and $\alpha + \gamma/\gamma$ boundaries as a function of vanadium content in an equiatomic FeCo alloy, as measured by Martin and Geisler [31] by thermal analysis (lines are average of values measured during cooling and heating) [31,25,32].
It is commonly accepted that FeCo undergoes an ordering transition around 730°C where the bcc structure takes the CsCl (B2) ordered structure (Fig. 15).

![Fig. 15. bcc and ordered B2 structure.](image)

Iron-cobalt alloys have the highest magnetization saturation of all known magnetic alloys (Fig. 16).

![Fig. 16. Slater-Pauling curves showing the mean atomic moment for a variety of binary alloys as a function of their composition [33].](image)
Although the maximum saturation is obtained around 35% cobalt, equiatomic compositions offer a considerably larger permeability for a similar saturation, as illustrated in Fig. 17. As for soft magnetic systems in general, the coercivity of FeCo alloys depends strongly on the microstructure.

Fig. 17. Initial and maximum permeability for FeCo alloys, the annealing temperature influences strongly the maximum permeability, in this case most likely because 1000\(^{\circ}\)C lies in the two-phase region [33].

The saturation magnetization is generally regarded as independent of the microstructure. As indicated earlier (Fig. 18), an increase of saturation has been observed following ordering, which in turns has been used as an indicator of the order parameter. Although the moments of Co atoms
does not depend on their environment, that of Fe does, as it increases from 2.2 $\mu_B$ in pure Fe to about 3 $\mu_B$ (Fig. 19) in ordered equiatomic FeCo [33]-[35].

![Graph showing variation of saturation magnetization as a function of temperature](image)

**Fig. 18.** Variation of the saturation magnetization as a function of temperature [36].

Typical saturation values for ordered binary FeCo [37] or FeCo-2V [38]-[39] are about 2.35 T. Chen [33] investigated the influence of ternary additions on the saturation of FeCo alloys, and found that most (Ti, V, Cr, Ni, Cu) had a detrimental effect, with the exception of Mn. This author also reported that, while Ti, V and Cr order antiferromagnetically, Ni and Mn order ferromagnetically. In addition, Mn displays an atomic moment larger than that the average one for FeCo (~3 $\mu_B$). This has been further investigated theoretically by Reddy et al. [41] using the cluster variation method. Their results also indicate that the impurities Al, V, Mn and Ru preferentially occupy the Fe site, with the substitution of Fe by V being the only one energetically favorable. Chen reports a solubility limit of 7 at% for Mn in FeCo [34] (Koster [42]
Fig. 19. Magnetic moments of Fe and Co on binary alloys of different compositions [35]. Saturation magnetization of Fe$_{1-x}$Co$_x$ alloys as a function of composition. The straight line is that for mixture of pure elements. The formation of a solution is accompanied by an increase in the saturation magnetization [40].

...reporting about 5.5 at \%, and the possibility to achieve an average atomic moment of 2.43 \( \mu_B \) for a 5 at\% addition. Saturation is usually regarded as being independent, to a large extent, of the microstructure. Small variations can however be detected; for example, Fingers and Kozlowski [43] report different values of saturation for the same alloy depending on the exact heat-treatment conditions, as illustrated in Table I.

Table I. Saturation magnetization for a FeCo-2V-0.3Nb alloy after different heat treatments.

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>Time (h)</th>
<th>Grain size (( \mu m ))</th>
<th>Saturation magnetization (T)</th>
</tr>
</thead>
<tbody>
<tr>
<td>704</td>
<td>1</td>
<td>1.13</td>
<td>2.37</td>
</tr>
<tr>
<td>720</td>
<td>1</td>
<td>1.68</td>
<td>2.40</td>
</tr>
<tr>
<td>720</td>
<td>2</td>
<td>2.80</td>
<td>2.43</td>
</tr>
<tr>
<td>732</td>
<td>1</td>
<td>2.33</td>
<td>2.40</td>
</tr>
</tbody>
</table>

Increasing further the amount of ternary addition leads to significant reduction in saturation,
resulting from both the dilution effect due to the addition of V or Nb, and from the precipitation of non-magnetic particles [44] as illustrated in Table II.

Table II. Saturation magnetization for different FeCo based alloys after furnace cooling from 760 °C, data from [44].

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Saturation magnetization (T)</th>
<th>Volume fraction of second phase (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FeCo-2V</td>
<td>2.32</td>
<td>-</td>
</tr>
<tr>
<td>FeCo-3.6V</td>
<td>2.29</td>
<td>not measured</td>
</tr>
<tr>
<td>FeCo-1Nb</td>
<td>2.34</td>
<td>7</td>
</tr>
<tr>
<td>FeCo-2Nb</td>
<td>2.29</td>
<td>11</td>
</tr>
<tr>
<td>FeCo-3Nb</td>
<td>2.20</td>
<td>14</td>
</tr>
</tbody>
</table>

The coercivity is often seen as an important parameter if low losses are to be achieved. The coercivity is, in general, affected by most types of defects. This includes dislocations, grain boundaries, and precipitates. The coercivity depends on the grain size as follows [45] (Eq.(18)):

\[
H_C \sim \left( \frac{3}{D} \right) \left( kT_C K_1/aM_{sat} \right)^{1/2}
\]  

(18)

where \( H_C \) is the coercivity, \( D \) the grain size, \( M_{sat} \) the magnetization saturation, \( K_1 \) the magnetocrystalline anisotropy, \( T_C \) the Curie temperature and \( a \) the lattice constant. The most often quoted relationship between coercivity and non-magnetic particle distribution is due to Kersten [45] (Eq.(19)):

\[
H_C \sim \left( \delta_w K_1/M_{sat}\mu_0 r \right)^{2/3} V_f^{(2/3)}
\]  

(19)

where \( \delta_w \) is the wall thickness, \( \mu_0 \) the permeability of vacuum, \( r \) the average radius of the particles and \( V_f \) their volume fraction. This does not apply if the wall thickness is greater than the particle size.

The different grain sizes should have been obtained by varying the annealing time at the same temperature, as the concentration of quenched-in vacancies may otherwise vary and affect the result. Clearly, the samples should also be well annealed if comparisons are to mean anything.
Fig. 20 shows a number of measurements made on a variety of FeCo-X alloys. In general, the dependence of coercivity on the grain size is in good agreement from one study to another, as long as the alloy is well-annealed, single-phase FeCo-X alloy. A satisfying fit is provided by Eq. (20):

\[ H_C = 34.2 + 758 D^{-1} \]  

(20)

where D is a diameter expressed in μm and \( H_C \) in A m\(^{-1} \).

![Graph showing coercivity as a function of grain size](image)

Fig. 20. Coercivity of a variety of FeCo-2V based alloys (all are annealed and single-phase) as a function of their grain size.

As mentioned earlier, the use of equiatomic FeCo alloys as opposed to lower Co content is mainly driven by their very high permeability. There are surprisingly little studies reporting directly either the initial or maximum permeabilities of FeCo based alloys. Although there is evidence for a decrease in permeability in FeCo-2V during long-term exposure to intermediate temperatures (450-550˚C) [46], one can only speculate, for lack of direct evidence, that this is a consequence of \( \gamma_2 \) precipitation as observed by Ashby et al. [32].
The impact of Cu and W additions in FeCo-2V alloys on their coercivity has been discussed above. Orrock also reports saturation for different applied fields, showing a significant decrease in permeability, even for additions which had no impact on coercivity, as shown in Table III.

Table III. The magnetization at 4000 and 40000 A m\(^{-1}\), showing that, while saturation is little affected by the addition of Cu or W to FeCo-2V, the magnetization at intermediate applied fields is significantly reduced.

<table>
<thead>
<tr>
<th></th>
<th>FeCo-2V</th>
<th>FeCo-V-Cu</th>
<th>FeCo-V-W</th>
</tr>
</thead>
<tbody>
<tr>
<td>(M_{4000\text{A/m}})</td>
<td>2.23</td>
<td>1.99  1.90 1.65</td>
<td>1.79  1.73 1.69</td>
</tr>
<tr>
<td>(M_{40000\text{A/m}})</td>
<td>2.34</td>
<td>2.28 2.23 2.18</td>
<td>2.20 2.19 2.19</td>
</tr>
</tbody>
</table>

Works on FeCo-V-Ni [47,48] indicate a detrimental effect on the permeability: the maximum permeability of this alloy, after optimum heat-treatment, is found to be about 2.5 times lower than that of FeCo-2V. A similar observation was made on FeCo-Nb alloys, with FeCo-1Nb wt% showing a maximum permeability about 3 times lower than FeCo-2V.

The low ductility of the binary alloy are attributable to a grain boundary embrittlement, and it is most often observed to fracture inter-granularly in both ordered and disordered conditions. The role of vanadium in promoting ductility in samples quenched from high temperatures seems to be related to its suppressing the grain boundary embrittlement. The ordered state remains brittle due to the limited numbers of slip systems, however this time failing mainly by intragranular fracture, and with detectable deformation (\(~ 4 – 5\%\)). Other elements have been found that have the same influence on ductility, such as Nb or Ta. The coercivity is strongly dependent on the microstructural features, which in most commercial alloys is limited to grain boundaries. The problem is more complex when precipitates are present, as it is clear that their influence on the coercivity cannot be rationalized without more
detailed information than usually published (size distribution, location). In contrast to the coercivity, the saturation magnetization is slightly dependent on the microstructure.
VI. EXPERIMENTAL SETUP FOR MEASUREMENT OF HEATING RATE AND SPECIFIC POWER LOSS

In this project, the magnetic nanoparticles are subjected to applied ac magnetic field. As a result, the nanoparticles release thermal energy and heat the environment. So, the released heat by particular amount of nanoparticles is calculated based on measurement of the sample temperature as a function of time. The amount of heat produced during this experiment depends on the heating rate which is characterized by a quantity called Specific Absorption Rate (SAR). The accurate measurement of the mass of nanoparticles used during the measurement is important. For the purpose of relative comparison of the heating power for different nanoparticles, approximately the same mass of magnetic nanoparticles was used. To meet the above requirements and proper measurements of the heating rate of nanoparticles, the system must consist with the following parts: a temperature probe, a function generator, a coil, a power supply, a current supply, a chiller, a vacuum pump, sample container, and an accurate mass balance.

TEMPERATURE PROBE

The primary parameter for the determination of SAR values is the temperature measurement. The fiber-optics temperature probe used here is very precise without affecting the medium whose temperature is being measured. The temperature probe used in this project is FISO Inc. FOT-L Temperature Sensor. This temperature sensor has following characteristics [49].

1. It is fiber-optics temperature sensor which enables accurate, stable and repeatable measurements of temperature. It has an accuracy of .0001 K.

2. It has a Fabry-Parot cavity constituted by two optical fibers precisely assembled into a glass capillary. The length of the cavity changes with temperature variations due to the difference in the coefficient of thermal expansion between optical fiber and the glass capillary.
3. The temperature measurements are based on variations of the reflected light when compared to the emitted light due to thermal expansion of the glass used within the sensor.

4. It is immune to EMI and RFI since the sensors are not electronically active. Hence they do not emit nor are they affected by any EM radiations and weather.

5. It has miniature size and microscopic contact area so it can be inserted even if the apparatus does not support temperature sensing inherently. Also, due to small size of the sensor, the thermal inertia is virtually reduced, which allows ultra-fast temperature monitoring.

6. It is sheathed with polytetrafluoroethylene (PTFE) and has a temperature range from as low as 
   −40°C up to 300°C (−40°F to 572°F).

7. It has a very low heat capacity so it does not give in or take heat to the material and there will be no effect on the temperature of the material being measured.

8. The structure of the sensor has an influence on minimum amount of the sample needed to assure that the sensitive part of the sensor was imbedded in the sample.

9. It offers accuracy and reliability in extreme temperature and hostile environmental conditions.

10. The fiber optic lead cable can be up to several meters long without affecting the quality or the accuracy of the results.

Specifications of FOT-L temperature sensor are summarized in Table IV and Fig.21 shows FOT-L temperature sensor.

Table IV. Specification of FOT-L temperature sensor.

<table>
<thead>
<tr>
<th>Specification</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature range</td>
<td>40°C to 250°C</td>
</tr>
<tr>
<td>Resolution</td>
<td>0.001°C</td>
</tr>
<tr>
<td>Accuracy</td>
<td>0.01°C</td>
</tr>
<tr>
<td>Response time</td>
<td>≤ 0.5s</td>
</tr>
</tbody>
</table>
**FREQUENCY OF OPERATION**

The amount of thermal energy released by magnetic nanoparticles strongly depends on frequency. These time varying magnetic fields directly affect human tissues and can cause nerve simulations and impact the neurological functioning of the human body [50]-[51]. Setting a frequency range for the heating measurements is an essential part of the experiment. This frequency should be safe for human tissues because the higher frequency will produce a higher temperature raise in the medium. Also, at low frequency (f < 100 kHz) the heating mechanism is different from that of high frequency (f > 100 kHz). In the low frequency regime, induced current and dielectric absorption causes stimulation of muscle cells. In the high frequency, a tissue heating is the main problem which can damage the human body. Human tissue is not able to resist the extreme heat generated by the thermal impact. As a result, one particular frequency of 175 kHz was used as the operating frequency in the experiment.

**MAGNETIC FIELD AND CUSTOM COIL**

A custom made coil in the form of a solenoid was used in order to produce a uniform magnetic field around the FeCoV magnetic nanoparticles. The strength of the magnetic field generated by a single current carrying wire is inversely proportional to the distance from the center of the wire.
while the magnetic field of the solenoid strength does not depend on the distance from the axis or on the cross-sectional area [52]. Fig. 22 shows pattern of magnetic field lines in a solenoid of the finite length, in general. The magnetic field of an infinitely long solenoid or copper sheet coil is given by Eq.(21), respectively

\[
B = \mu_0 \frac{I}{2\pi r}
\]  

(21)

where \( \mu_0 \) is a vacuum permeability and \( I \) is an intensity of current [53]. Our custom-made coil with diameter of \( 2r = 3 \) cm and length of \( 4 \) cm and consists of insulated copper sheets wrapped around each other 20 times in the form of a spiral solenoid was used.

Fig. 22. Magnetic field of custom-made coil with insulated copper sheet.

POWER SUPPLY AND FREQUENCY GENERATOR: ALTERNATING MAGNETIC FIELD (AMF) SYSTEM

The custom-made power supply (a black box in Fig. 23) is used for the production of an alternating current at the range of kHz. This alternating current is delivered to the custom coil.
The variable frequencies produced by a frequency generator with custom made power supply are shown in Fig. 23.

![Image of black box of custom made power supply and frequency generator](image)

Fig. 23. Black box of custom-made power supply and frequency generator.

Using an external frequency generator to set the resonant frequency of the system. The frequency generator was manufactured by BK Precision and named as BK Precision 4011A function generator. It is able to sweep the frequency from 0.5 Hz to 5 MHz. It has four digits LED display. Fig. 24 shows BK Precision Function generator [54].

![Image of BK Precision Frequency generator](image)

Fig. 24. BK Precision Frequency generator.

**CHILLER AND VACCUM PUMP**

In this project, there are two factors: high currents and high frequencies (above 100 kHz) which generate heat and heat up the copper coil. As the frequency increases the heating of the coil will
increase further. The coil has to be cooled externally in order to dissipate the generated heat. So, due to the set up high heat capacity (4.187 J/g°C), water was injected into it by the water chiller in order to keep the water that cools the coil externally at constant temperature. All the experiments were done at room temperature. The copper sheets of the custom coil were cooled by distilled water [50]. The water pump maintains a constant flow-rate in the tubes. However, there will be some heat transfer to the sample by convection because the water being pumped into the coil was not able to take all amount of the heat released by the coil. In order to recover this issue, vacuum pump [55] has been connected to the coil enclosure. The vacuum pump eliminates heat transfer from the coil to the sample. In summary, water is needed to cool the coils and a vacuum section inside the coils is needed to insulate the sample from the hot coils.

![Vacuum pump](image)

Fig. 25. Vacuum pump.
Fig. 26. Complete AMF system including custom-made power supply, frequency generator, current supply, and custom-made coil.

SAMPLE CONTAINER

The sample was placed in nuclear magnetic resonance (NMR) tubes with outer diameter of 5 mm and small inner diameter of 4.57 mm with length of 7 inches. Fig. 29 shows a sample in NMR tube.

Fig. 27. NMR tube with a sample.
MASS BALANCE

A very precise mass balance with accuracy of ± 0.0001 g was used. To avoid any effects from surrounding conditions, the mass balance was placed inside a lab hood (Fig. 28). First, mass of each tube was measured alone. Then, we used a weighing paper to transfer sample into the tube, then the total mass of tube and a sample were measured together. We used the same amount of sample for measurements. The mass of nanoparticles was calculated by subtracting mass of tube from total mass of nanoparticle sample and tube together (see Table V). Measurements were done with a dry sample.

Table V. Mass of sample and total mass of the tube and sample.

<table>
<thead>
<tr>
<th>Mass of sample (C)</th>
<th>0.0107g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total mass of the tube and sample (C)</td>
<td>2.5729g</td>
</tr>
<tr>
<td>Mass of sample (Oxide)</td>
<td>0.0112g</td>
</tr>
<tr>
<td>Total mass of the tube and sample (Oxide)</td>
<td>2.5852g</td>
</tr>
</tbody>
</table>

Fig. 28. Mass balance.

EXPERIMENTAL PROCEDURE

Iron-cobalt vanadium (FeCoV) nanoparticles with average diameter of 30 nm were coated with graphite or oxide with the thickness of 10 and 5 nm, respectively. A fiber optics temperature
probe was inserted inside. The minimum amount of the sample is needed to ensure that the sensitive part of the sensor submerges into the sample. To set the tube in the middle part of the coil all the time, tube containing sample along with temperature probe was secured by a rubber holder. Firstly, water chiller and then vacuum pump was turned on. The above mentioned arrangement of tube containing sample was put inside the custom-made coil and kept for around 10 minutes in order to ensure stability of the vacuum inside the coil and also that the heat is no longer dissipated into the surroundings keeping our system under steady state condition. Power supply and function generator was turned on and a frequency of 175 kHz was set in function generator. FISO Evolution software was opened and it was ensured that software measures temperature, else device was refreshed from the software. Recording time of the frequency of data collection was set to 30 minutes. Sample was then subjected to magnetic heating by turning on current supply to 15 A, and sample was allowed to heat up until it reached $80^\circ$C on the outside temperature probe, then current supply was turned off. Sample was allowed to cool down for 13 minutes. Then whole system was turned off. After this tube containing sample and probe was taken out from the coil. The same experimental procedure was followed for 5 and 10 A. Experiments with each sample was repeated for one frequency (175 kHz) and three different current values. All the data for temperature variation of nanoparticles was recorded by the FISO software. A typical sample plot is shown in Fig. 29 (FeCoV) without coating at 15 A and 175 kHz).
Fig. 29. Temperature variation of heating curve for FeCoV nanoparticles at 15 A and 175 kHz.
VII. EXPERIMENTAL RESULTS

In this Thesis, we are interested in the heating rate of the FeCoV magnetic nanoparticles subjected to ac externally applied magnetic field. The temperature changes with time, during the heating process, were measured by custom-made AMF system and recorded by using FISO evolution software. The typical graph of temperature change (degree Celsius) versus time (seconds) is plotted in Fig. 30.

Fig. 30. Temperature variation of heating curve for FeCoV (oxide-coated) nanoparticles at 15 A and 175 kHz.

HEATING RATE

Since our interest is the heating power, we are looking for the maximum rate of change in the temperature as a function of time from the graph at the inflection point of the heating curve. First step is to find a fit of third degree polynomial to the heating curve in order to calculate analytically the inflection point. Secondly, a second derivative of the polynomial equal to zero locates this inflection point. Finally, the maximum heating rate (dT/dt, rate of change of sample
temperature with respect to time) is calculated at the inflection point. A best fit (cubic approximation) is represented by following Eq. (18) where temperature $T$ is a function of time $t$ and $a$, $b$, $c$, $d$ are the coefficients of the third degree polynomial.

$$T(t) = at^3 + bt^2 + ct + d$$  \hspace{1cm} (22)

The second derivative of Eq. (22) was set to zero to get inflection point $t_{\text{INF}}$. Finally, slope or heating rate was calculated at inflection point (see, Eq.(23))

$$\text{Slope} = \frac{dT}{dt} \text{ at } t = t_{\text{INF}}$$  \hspace{1cm} (23)

A typical plot with cubic approximation is shown in Fig. 31. The blue curve in the insert to Fig. 31 corresponds to best fit to the curve during the only heating process. The original experimental data have the error bars. These maximum heating rates at inflection points were used to find the specific absorption rate (SAR) or heating power of FeCoV nanoparticles.

![Graph showing temperature variation and cubic approximation](image)

**Fig. 31.** Temperature variation of heating curve with error bars for FeCoV (graphite-coated) nanoparticles at 10 A and 175 kHz with a fitting curve by 3rd order polynomial in the insert.

The rest of the experimental data for heating rate of FeCoV magnetic nanoparticles coated with oxide and graphite (to prevent oxidation) are depicted in Figs.32-39 for three different values of I.
= 5 A, 10 A, and 15 A at frequency of 175 kHz only. Each graph has an insert which contains analytical approximation of this part of the heating curve which is approximated by third order polynomial in terms of time dependence (Eq. (22)) needed for calculation of inflection points.

Fig. 32. Temperature variation of heating curve for FeCoV (oxide-coated) nanoparticles at 15 A and 175 kHz.

Fig. 33. Temperature variation of heating curve for FeCoV (oxide-coated) nanoparticles at 10 A and 175 kHz.
Fig. 34. Temperature variation of heating curve for FeCoV (oxide-coated) nanoparticles at 5 A and 175 kHz.

Fig. 35. Temperature variation of heating curve for FeCoV (graphite-coated) nanoparticles at 15 A and 175 kHz.
FeCoV (graphite)
f = 175 kHz
I = 10 A

Fig. 36. Temperature variation of heating curve for FeCoV (graphite-coated) nanoparticles at 10 A and 175 kHz.

FeCoV (graphite)
f = 175 kHz
I = 5 A

Fig. 37. Temperature variation of heating curve for FeCoV (graphite-coated) nanoparticles at 5 A and 175 kHz.
Fig. 38. Time variation of heating curve for FeCoV (oxide) nanoparticles sample heated up at different intensity (5, 10, and 15 A) and at 175 kHz.

Fig. 39. Time variation of heating curves for FeCoV (graphite) nanoparticles’ sample heated up at different intensity (5, 10, and 15 A) and at 175 kHz.
A RF plasma torch method was used to produce a disordered bcc $\alpha$-FeCo phase magnetic nanoparticles with a thin protective oxide layer fabricated from metal precursors [56]. Due to a continuous precursor powder feed rate, nanoparticles (after additional annealing at elevated temperature to create ordered nanocrystals) size observed was 30 nm in an average and this average size was confirmed by statistical particle size analysis carried on the TEM micrographs (nanoparticles with an oxide coating, Fig. 40).

The magnetic characterization of these nanoparticles by using SQUID and VSM magnetometer showed relatively high coercive force with a value of $H_C = 250$ Oe and a low saturation magnetization $M_{sat} = 190$ emu/g. Both values were measured at room temperature. The temperature dependence of coercivity and saturation magnetization is depicted in Fig. 41 and 42, respectively. The presence of ferrite $\text{CoFe}_2\text{O}_4$ with antiferromagnetic order was detected in this coating and confirmed by thermomagnetic data. An antiferromagnetic coupling between FeCo
core nanoparticle and ferrite (oxide) coating was indicated by hysteretic measurement from a shifted hysteresis loop.

Fig. 41. Temperature dependence of coercivity for FeCo-oxide.

Fig. 42. Temperature dependence of saturation magnetization for FeCo-oxide.
Since antiferromagnetic Neel temperature $T_N \approx 520 \, ^0\text{C}$ of CoFe$_2$O$_4$ is much lower than the ferromagnetic Curie temperature $T_C \approx 950^0\text{C}$ of FeCo, diminishing antiferromagnetic exchange coupling between coating and nanoparticle’s core with increasing temperature results in lower coercivity (see Figs. 41 – 42). However, the explanation of the higher coercivity and lower magnetization saturation, in general, in nanocrystalline FeCo-oxide systems like this is connected with surface and interface anisotropy. Neel proposed a model [57] which concludes that atoms in a vicinity of the reduced symmetry (at a surface or at an interface) including lattice misfit between core of the nanoparticle and coating gives rise to anisotropy that is different from the bulk anisotropy. It leads to the creation of the canting phase of the surface moments and the higher corecivities for FeCo-oxide magnetic nanoparticles.

A RF plasma torch method was also used to produce a disordered bcc $\alpha$-FeCo phase magnetic nanoparticles with a thin protective graphitic carbon layer [26] to avoid oxidation. Acetylene was introduced into the carrier gas and powder mixture between powder feeder and injection probe as a carbon source for coating purposes. An average nanoparticle diameter of 50 nm (including carbon coating) was in reasonable agreement with TEM observations of nanoparticle size with a Gaussian type size distribution. Saturation magnetization and coercivity in FeCo with carbon coating subjected to annealing at 800 $^0\text{C}$ (crystallization temperature) are equal respectively to $M_{\text{sat}} = 168$ emu/g and $H_C = 260$ Oe.

The alloy investigated in this Thesis includes 1.8 % of vanadium as an additional constituent to oxide or carbon-coated FeCo. In addition to improvement of its mechanical properties, a role of vanadium added to FeCo alloy significantly reduces the presence of CoFe$_2$O$_4$ ferrite in the system by reducing ferrite to form vanadium oxide [3]. Synthesized magnetic nanoparticles yielded magnetic saturation magnetization $M_{\text{sat}}$ between 185-215 emu/g and coercivities $H_C$ at
around 290 Oe at room temperature. Higher than expected coercivity values are due to the nonhomogeneous microstructure resulting from interactions with vanadium. In summary, Tab.VI lists \(M_{\text{sat}}\) and \(H_C\) for FeCo magnetic nanoparticles with oxide and carbon coating without and with vanadium addition at room temperature.

Table VI. Magnetization saturation and coercivity for FeCo and FeCoV magnetic nanoparticles at room temperature.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>(M_{\text{sat}}) (emu/g)</th>
<th>(H_C) (Oe)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FeCo-Oxide</td>
<td>190</td>
<td>130</td>
</tr>
<tr>
<td>FeCo-C</td>
<td>168</td>
<td>260</td>
</tr>
<tr>
<td>FeCoV-Oxide and C</td>
<td>185-215</td>
<td>290</td>
</tr>
</tbody>
</table>

**SPECIFIC ABSORPTION RATE (SAR)**

SAR is a measure of the heating ability of FeCoV magnetic nanoparticles in a dry form ([58]-[61]) and usually defined as heat produced by magnetic nanoparticles subjected to ac externally applied magnetic field over time as shown below (Eq.(24))

\[
\text{SAR} = \frac{Q}{\Delta t}
\]  

(24)

Heat \(Q\) produced by Neel mechanism only and defined as heat capacity \(C\) multiplied by temperature change \((Q = C \Delta T)\) results in Eq. (25))

\[
\text{SAR} = C(\Delta T/\Delta t)
\]  

(25)

where \((\Delta T/\Delta t)\) is the initial slope of time-dependent temperature curve or heating curve in units of \(^{0}\text{C}/\text{min}\). On the other hand, heat capacity is defined as follows (Eq. (26))

\[
C = m_{\text{np}}c_{\text{np}} + m_5c_5 + m_{\text{T}}c_{\text{T}}
\]  

(26)

where \(m_{\text{np}}, c_{\text{np}}, m_5, c_5,\) and \(m_{\text{T}}, c_{\text{T}}\) are masses of FeCoV, graphite coating, oxide coating, and NMR glass tube and respectively their specific heat capacities. We define the SAR\(_{\text{FeCoV-C}}\) as SAR
(Eq. (25)) divided by $m_{np}$ of FeCoV nanoparticles and a new expression of the heat efficiency of FeCoV nanoparticles ($\text{SAR}_{\text{FeCoV-C}}$) in units of Watt per gram is given by Eq. (27)

$$\text{SAR}_{\text{FeCoV-C}} = \left[ (m_{np}c_{np} + m_{s}c_{s} + m_{t}c_{t})/m_{np} \right] (\Delta T/\Delta t) \quad (27)$$

while $\text{SAR}_{\text{FeCoV-oxide}}$ is defined by dividing SAR (Eq. (25)) by $m_{np}$ plus $m_{\text{oxide}}$ of FeCoV nanoparticles because of the magnetic property of the oxidation layer (CoO–Fe$_2$O$_3$). It leads to (Eq. (28))

$$\text{SAR}_{\text{FeCoV-oxide}} = \left[ (m_{np}c_{np} + m_{\text{oxide}}c_{\text{oxide}} + m_{t}c_{t})/(m_{np} + m_{\text{oxide}}) \right] (\Delta T/\Delta t) \quad (28)$$

General physical properties of NMR glass tube (Tab.VII) and mass of FeCoV nanoparticles used in experiment with graphite (C) coating ($m_c$), oxide (CoO–Fe$_2$O$_3$) coating ($m_{\text{oxide}}$) and NMR glass tube used for SAR measurements and their respective heat capacities are summarized in Tabs.VIII-X. Finally, SAR (W/g) values for FeCoV nanoparticle samples are presented in Tab. XI (an accuracy of experimental measurements was done within approximately 5 percent) with their heating rates (Tab.XII) defined as $\Delta T/\Delta t$ at the inflection point of heating curve = slope at this point. Finally, graphs representing $\Delta T/\Delta t$ and SAR as a function of magnetic field intensity for FeCoV with graphite and oxide coating are plotted in Figs. 43-44.

Table VII. Borosilicate glass (NMR glass tube) properties.

<table>
<thead>
<tr>
<th>Borosilicate glass (NMR glass tube)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal conductivity [W/(m K)]</td>
<td>1.13</td>
</tr>
<tr>
<td>Specific heat [ J/(g°C)]</td>
<td>0.753 - 0.837</td>
</tr>
<tr>
<td>Coefficient of thermal expansion [1/°C (x10$^{-6}$)]</td>
<td>3.30</td>
</tr>
</tbody>
</table>
Table VIII. Calculated masses of FeCoV, graphite coating and oxide coating and their specific heat capacities.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Calculated mass (g)</th>
<th>Heat capacity at 300K (J/g °C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FeCoV-C (no coating)</td>
<td>0.0053</td>
<td>0.1000</td>
</tr>
<tr>
<td>FeCoV-Oxide (no coating)</td>
<td>0.0042</td>
<td>0.1000</td>
</tr>
<tr>
<td>C</td>
<td>0.0054</td>
<td>0.7100</td>
</tr>
<tr>
<td>CoFe₄Ο₄/CoΟ·Fe₂Ο₃</td>
<td>0.0070</td>
<td>0.6503</td>
</tr>
</tbody>
</table>

Table IX. Specific heat capacities and mass for FeCoV nanoparticles with graphite and oxide coating and for NMR glass tube.

<table>
<thead>
<tr>
<th>Name</th>
<th>Heat capacity at 300K (J/g °C)</th>
<th>Mass (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NMR glass tube</td>
<td>0.7950</td>
<td>2.5622</td>
</tr>
<tr>
<td>FeCoV-C</td>
<td>0.4079</td>
<td>0.0107</td>
</tr>
<tr>
<td>FeCoV-Oxide</td>
<td>0.4441</td>
<td>0.0112</td>
</tr>
</tbody>
</table>

Table X. Samples densities, molar heat capacities and molar masses.

<table>
<thead>
<tr>
<th>Name</th>
<th>Density (g/mc³)</th>
<th>Molar heat capacity (J/K mol)</th>
<th>Molar mass (g/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FeCoV</td>
<td>8.12</td>
<td>16.57</td>
<td>165.72</td>
</tr>
<tr>
<td>Fe₂Ο₃</td>
<td>5.24</td>
<td>104.77</td>
<td>159.69</td>
</tr>
<tr>
<td>CoO</td>
<td>6.44</td>
<td>55.22</td>
<td>74.93</td>
</tr>
<tr>
<td>CoFe₄Ο₄/CoΟ·Fe₂Ο₃</td>
<td>5.63</td>
<td>152.58</td>
<td>234.63</td>
</tr>
<tr>
<td>C</td>
<td>2.25</td>
<td>8.53</td>
<td>12.01</td>
</tr>
</tbody>
</table>
Table XI. SAR values of FeCoV nanoparticles at 175 kHz for three current values of 5 A, 10 A, and 15 A.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>SAR at 175 kHz (W/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>15 A</td>
</tr>
<tr>
<td>FeCoV-C</td>
<td>24.25</td>
</tr>
<tr>
<td>FeCoV-Oxide</td>
<td>11.90</td>
</tr>
</tbody>
</table>

Table XII. Heating rate of FeCoV nanoparticles at 175 kHz for three values of current 5 A, 10 A, and 15 A.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Heating rate at 175 kHz $\Delta T/\Delta t$ ($^0$C/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>15 A</td>
</tr>
<tr>
<td>FeCoV (oxide)</td>
<td>4.1151</td>
</tr>
<tr>
<td>FeCoV (graphite)</td>
<td>3.9886</td>
</tr>
</tbody>
</table>

Fig. 43. Rate of change of heating curve for coated FeCoV magnetic nanoparticles with current.
We can see from Fig. 43 that the iron cobalt vanadium nanoparticles with graphite or oxide coated surfaces are behaving in the same manner as far as heating curve is concerned. It means that with increasing value of the current, which is related to increasing intensity of ac magnetic field, heating rate of these nanoparticles slightly increases. The slight difference between these two curves is due to the coated material. The FeCoV nanoparticles coated with oxide have a slightly higher heating rate than FeCoV nanoparticles with graphite coating due to the presence of magnetic oxide layer Fe₂O₃·CoO on the surface. The main role of this layer is to prevent oxidation of FeCoV nanoparticles but because of their magnetic properties, there is slight contribution of this layer to heating mechanism. The heating rate slightly favors FeCoV with oxide coating which is opposite to heating power or SAR (Fig. 44). It appears that due to combination of heat capacities of different constituents and their respective masses (see, Eqs. (27) and (28)) leads to stronger heating power of FeCoV magnetic nanoparticles with graphite.

Fig. 44. Effect of current on SAR of coated FeCoV magnetic nanoparticles’ samples.

![Graph showing SAR vs. Current for FeCoV/CoO·Fe₂O₃ and FeCoV/C nanoparticles](image-url)
coating (Fig. 44). As a result, we can conclude that the graphite coating of FeCoV magnetic nanoparticles has a notable effect on their heating power by preserving magnetic core of the sample in contrast to oxide coating of FeCoV nanoparticles which results in the depletion of magnetic core hampering its power. In addition, heating power and rate (Figs. 43 and 44) roughly around 10 A approaches saturation suggesting that further increase in magnetic field intensity has no significant effect on heat production. It suggests that at 10 A the amplitude of magnetic field reaches coercive force $H_C$ (see Tab.VI) and further increase above this value of 10 A has no effect on heating power or rate.
IX. CONCLUSION AND FUTURE GOALS

The iron-cobalt-vanadium with oxide or graphite coating is recognized as extremely soft magnetic materials with a high saturation magnetization $M_{\text{sat}}$, a high Curie temperature $T_C$, and a relatively low coercivity $H_C$. The major application of FeCoV as a soft magnet for high temperature applications in electric engines can be considered as a core of electromagnets to improve their losses. Unfortunately, oxidized FeCoV magnetic nanoparticles (with an increase in brittleness) have a higher coercivity in comparison to the bulk material. This could be one of possible mechanisms to cause increase in $H_C$. To avoid the oxidation, two different types of coating of FeCoV nanoparticles were studied: oxide or graphite coating. Heating rate was measured and heating power has been calculated to characterize heating capabilities of these nanoparticles to sort it out their potential applications. It appears that FeCoV magnetic nanoparticles coated with oxide have much lower heating power than coated with graphite so they are more suitable for application in electromagnetic machines. However, FeCoV magnetic nanoparticles with graphite coating and high heating power can be used in hyperthermia to destroy cancerous cells when they are properly administered in a human body including their additional functionalization. Also heating measurements could lead to a very straightforward technique of coercivity measurements for soft magnetic nanoparticles by simple establishing a value of magnetic field intensity when heating power or heating rate is saturated.

One of the most interesting aspects of further studies is to see how thicknesses of coatings (oxide or graphite) are affecting heating power in relation to potential applications in electromagnetic machines or in hyperthermia treatment.
X. REFERENCES


[53] http://hyperphysics.phy-astr.gsu.edu/hbase/magnetic/solenoid.html (online link).


