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**Novel Magneto-LC resonance Sensors for Industrial and Bioengineering Applications**

Ongard Thiabgoh  
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Novel Magneto-LC resonance Sensors for Industrial and Bioengineering Applications

by

Ongard Thiabgoh

A dissertation submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy
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DEDICATION

To

My Mother, Father, and brothers who have shaped my life.
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The scientific studies associated with material engineering and device miniaturization are the core concepts for future technology innovation. The exploring and tailoring of material properties of amorphous magnetic microwires, recently, have revealed remarkable high sensitive magnetic field sensitivity down to the picoTesla regime at room temperature. This superior magnetometer is highly promising for active sensing and real-time monitoring building block for modern industrial devices and healthcare applications.

The low-field, high sensitivity regime of the GMI response over a wide frequency range (1 MHz - 1 GHz) in the Co-rich melt-extracted microwires was optimized through novel Joule annealing methods (single- and multi-step current annealing techniques). Optimization of current value through multi-step current annealing (MSA) from 20 mA to 100 mA for 10 minutes is the key to improving the GMI ratio, and its field sensitivity up to 760% and 925%/Oe at $f \approx 20$ MHz. The respective GMI ratio and field sensitivity are 1.75 times and 17.92 times higher than those of the as-prepared counterpart. The employment of the MSA technique successfully enhances the surface domain structures of the Co-rich microwires. This alternative tailoring method is suitable for improving the GMI sensitivity for a small field detection. The high sensitive response of the GMI to a weak magnetic field is highly promising for biomedical sensing applications.

Real-time monitoring of position, motion, and rotation of a non-stationary object is crucial for product packaging, conveying, tracking, and safety compliance in industrial applications. The effectiveness of current sensing technology is limited by sensing distance and messy environments.
A new class of high-frequency GMI-based sensor was designed and fabricated using the optimal Co-rich microwire. The impedance spectrum from the optimal sensing element showed a high GMI ratio and high field sensitivity response at low magnetic fields. The GMI sensor based longitudinal effect was found to be more sensitive than the commercially available Gaussometers. The practical utility of the high sensitivity of the miniaturized sensor at weak magnetic fields for far-off distance monitoring of position, speed and gear rotating was demonstrated. This GMI-based sensor is highly promising for real-time position detection, oscillatory motion monitoring, and predictive failure of a rotating gear for industrial applications.

Monitoring the rate of respiration and its pattern is crucial to assessing an individual’s health or progression of an illness, creating a pressing need for fast, reliable and cost-effective monitors. A new sensor based on a magnetic coil, which is made of Co-rich melt-extracted microwire for the detection of small magnetic fields was fabricated. The 3 mm diameter coil is wound from a Co-rich magnetic microwire. Unlike some typical solenoids, the MMC is sensitive to small magnetic fields due to a significant change in impedance attributed to the high-frequency giant magneto-impedance (GMI) effect. An application of the MMC sensor for the detection of a position-varying source of a small magnetic field (~0.01 – 10 Oe) in real-time bio-mechanical movement monitoring in human was demonstrated. This newly developed MMC magneto-LC resonance technology is highly promising for active respiratory motion monitoring, eye movement detection and other biomedical field sensing applications.
CHAPTER ONE
INTRODUCTION

1.1 Research Motivation

Active sensing and real-time monitoring play an important role in modern industrial applications, modern healthcare devices, and scientific research [1-4]. The science and technology associated with sensors have grown enormously in the last few decades. In particular, magnetic field sensor technology has been extensively studied for uses in an industrial process monitoring, automatic controlling, advanced medical instrumentation, robotic controlling, antitheft systems, nondestructive testing, magnetic marking and labelling, geomagnetic measurements, space research, and many others, as illustrated in Fig. 1.1 [4-6]. A large number of magnetic sensors have been developed, such as giant magnetoresistance (GMR) sensors [7,8], fluxgate sensors [9], superconducting quantum interference device (SQUID) sensors [10], and giant magneto-impedance (GMI) sensors [11-14]. Among these magnetic sensors, those based on GMI technology have the benefit of ultrahigh sensitivity comparable to SQUID’s (detectable fields, ~pT) that operate at a room temperature, with small size, light weight, and low power consumption [6, 11]. These sensors are used in today’s mobile phones and electronic devices. The ultrahigh sensitivity of the GMI sensors also makes them very promising for detection of small biomagnetic fields.
GMI refers to a large change in the complex impedance of a ferromagnetic conductor subject to an applied dc magnetic field \([11, 15, 16]\). The origin of GMI is a skin effect in a magnetic medium resulting from an alternating current applied to the ferromagnetic conductor. It is generally known that materials with large permeability and low resistivity exhibit the GMI effect; therefore, the enhancement and trade-offs between these intrinsic material properties are crucial in optimizing the GMI for sensor applications. In addition, for high-frequency excitation, modifications of the penetration (skin) depth and magnetic permeability have strong dependence on the ac-frequency excitation and applied magnetic field. This enormous change in the impedance demonstrates the nature of time-dependent magnetic field propagating in the magnetically permeable conductor and is promising for small field detection \([11-13]\).

![Diagram of magnetic sensors and their applications](image)

**Fig. 1.1** A list of magnetic sensors and their respective applications.
It has been shown that melt-extracted Co-based microwires exhibit both high-performance GMI effect and outstanding soft magnetic properties (e.g., low magnetostriction, high saturation magnetization, high permeability, and low coercivity) [11, 15-17]. It has been reported that the GMI ratio and its field sensitivity $\eta$ of Co-based amorphous microwires, have been determined experimentally up to 300%, and 500%/Oe (at $f \approx 10$ MHz), respectively [17, 18]. Although Co-based melt-extracted microwires possess excellent mechanical and GMI properties, the internal stresses generated during the rapid cooling process, which deteriorates the GMI response in the melt-extracted microwires, must be tailored. Joule-heating is one of the most efficient techniques to improve the GMI the ratio and $\eta$ for such amorphous microwires. Under proper annealing, the release of residual stresses can promote circular magnetic domain structures, resulting in an increased GMI effect [24-26]. It has been reported that the GMI ratio of Co-based melt-extracted microwires has increased up to 368.7% and has achieved a field sensitivity as high as 623.5%/Oe, which are desirable for use in high-performance magnetic field sensors [27].

Up to now, most of the GMI studies on Co-rich melt-extracted microwires are limited to low and intermediate frequency regimes (less than 20 MHz). At higher frequencies, the magnetic field dependence of GMI in the low-field regime (below the effective anisotropy field of the wire, $H_K < \sim 5$ Oe) becomes more linear, which is desirable for sensing. However, its field sensitivity tends to decrease drastically with respect to an increase in frequency. It is, therefore, essential to tailor the circular magnetic domain structures (or the tunability of the corresponding circular magnetic anisotropy) of Co-rich melt-extracted amorphous microwires so that large linear field responses of GMI can be achieved within the low field regime. As a result, low-field detection GMI sensors with optimized field sensitivity have not been realized yet. In addition, there is an increasing need for the development of low-field GMI sensors for real-time monitoring of
industrial processes and health care. All of these have highly motivated further GMI research on Co-rich melt-extracted microwires in the high frequency regime up to gigahertz.

1.2 Objectives

The overall goal of this dissertation is to optimize the field sensitivity of GMI effect at high frequency in Co-rich melt-extracted microwires for the development of novel low-field magnetic sensors, as well as to develop a new class of magneto LC resonance sensor technology for real-time monitoring of health care and biomechanical movement. The specific objects include the following:

(i) Exploring the GMI effect over a wide frequency range (1 MHz – 1 GHz) in Co-rich melt-extracted amorphous microwires, e.g., Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{13.5}$, Co$_{69.25}$Fe$_{4.25}$Si$_{12}$B$_{13.5}$Nb$_1$, and Co$_{69.25}$Fe$_{4.25}$Si$_{12}$B$_{13.5}$Zr$_1$ (in at.%) ;

(ii) Optimizing the low-field, high sensitivity regime of the GMI response over a wide frequency range in the Co-rich melt-extracted amorphous microwires by employing novel Joule heating methods (single- and multi-step current annealing techniques);

(iii) Exploring structural and magnetic properties, e.g., amorphous, nano-crystalline, which is embedded in an amorphous matrix, surface morphology, composition, surface magnetic domain structure (SMDS) of the Co-rich microwires;

(iv) Employing microwires with an optimized GMI response as a sensing element to design and fabricate new classes of highly sensitive GMI sensors for real-time monitoring of position, vibration, and gear rotation.

(v) Integrating the high frequency GMI technology with LC-resonant circuits to develop a new class of magnetic sensor with improved sensitivity and for far-distance detection, namely
the magneto-LC resonance sensor. The sensor comprising of a magnetic coil made of Co-rich melt-extracted microwire is tested and explored.

(vi) Developing a new respiratory monitoring technology based on the optimized magneto-LC resonance sensor. Unlike typical solenoids, the magneto-LC resonance sensor is sensitive to small magnetic fields due to a significant change in impedance attributed to the high-frequency GMI effect. The magneto-LC resonance sensor is employed to detect a position-varying source of a small magnetic field for real-time respiratory monitoring of a human patient as well as real-time eye movement monitoring.

This Ph.D. dissertation also aims to advance the current fundamental understanding of correlation between the circular domain structure and the high frequency GMI behavior in Co-rich melt-extracted microwires.

1.3 Organization of the Dissertation

The dissertation is divided into seven chapters. CHAPTER ONE presents the motivation and objectives, and provides the organization of the dissertation.

In CHAPTER TWO, we present the basic concepts of magneto-impedance effect in magnetic wires and, in particular, classical electromagnetic theory of time-dependent magnetic field propagating into conducting media. Giant magneto-impedance effect (GMI), which originates from the skin effect in magnetic microwires, is demonstrated. We present fundamentals of the LC-resonance effect in ac-circuit and their recent applications. Finally, we give an overview on giant magneto-impedance (GMI) sensors and their applications.
In **CHAPTER THREE** we describe the experimental methods used in this dissertation. It begins with the description of the fabrication technique of the amorphous magnetic microwires. The sample preparation, as well as novel Joule heating treatments are explained. The structural characterization of the magnetic microwires including an x-ray diffractometry (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), and High-resolution Transmission Electron Microscopy (HRTEM) are discussed. Furthermore, measurements of the magnetic properties of the microwire samples by employing Magneto-optic Kerr Effect (MOKE) and Magnetic Force Microscopy (MFM) are described. Finally, high-frequency magneto-impedance measurement using the HP4191A RF impedance analyzer is explained.

In **CHAPTER FOUR** the results of the enhancement of the low-field GMI response in melt-extracted Co-rich microwires by means of Joule heating are presented. The trade-off between dc current amplitudes and annealing time was systematically optimized in order to refine the domain microstructures and circular magnetic anisotropy of the amorphous microwires over a high frequency range (1 MHz – 1 GHz) for the development of highly sensitive, high frequency GMI-based sensors.

In **CHAPTER FIVE** the practical utility of the low-field detection GMI sensors with optimized field sensitivity in the melt-extracted Co-rich microwires is demonstrated. The impedance response of a new class of GMI-based sensor to a small change in an external magnetic field was measured. Reliable and accurate measurements of position and speed of a moving object by the sensor are observed. The real-time monitoring of the impedance response from a small magnet attached to a rotating gear is tested. This GMI-based sensor is highly promising for real-time position detection, oscillatory motion, and failure prediction of gear rotation.
In **CHAPTER SIX** we present a new type of sensor based on a magnetic microwire coil (MMC) for the detection of small magnetic fields, which can be applied to real-time, non-contact respiratory monitoring. The operating principle of the MMC is discussed and the effective resistance \( (R) \), reactance \( (X) \), and impedance \( (Z) \) due to a small ambient magnetic field are described. In addition, simulated respiratory patterns with various changes in amplitudes, frequencies, and waveforms are demonstrated. Through actual tests on voluntary persons, we demonstrate the high capabilities of the non-contact and non-invasive real-time measurement of the MMC sensor for respiratory rate, and eye movement monitoring.

Finally, in **CHAPTER SEVEN** we summarize the results of this dissertation work. The future work relating to this dissertation and GMI-based sensors is proposed.

### 1.4 References


CHAPTER TWO

FUNDAMENTALS OF MAGNETO-IMPEDANCE AND MAGNETO LC-RESONANCE EFFECTS IN MAGNETIC MICROWIRES

In this chapter, the basic concepts of the magneto-impedance effect in magnetic wires are presented. In particular, the classical electromagnetic theory of a propagating electromagnetic wave in conducting media and its relationship to the skin effect, which is the origin of the giant magneto-impedance effect (GMI), is demonstrated. The fundamentals of LC-resonance effect in an ac-circuit and recent applications are presented. Finally, an overview of magneto-impedance (MI) sensors and their applications is given.

2.1 Introduction

The GMI effect is a large change in the complex impedance when a high permeability conductor is subjected to an external static magnetic field [1-5]. The GMI effect in amorphous magnetic materials has been observed in several geometries, i.e., magnetic ribbons, thin films, amorphous wires, and microwires. In particular, amorphous magnetic microwires exhibit a high GMI ratio and excellent magnetic field sensitivity. Furthermore, due to their small dimension and superior mechanical properties, amorphous magnetic microwires are highly promising for micro-sensor device applications.

The GMI effect in a Co-rich amorphous wire was first independently demonstrated by Panina et al. [1], and Beach et al. [2] in 1994. This discovery reported an ~ 40% GMI ratio of an amorphous Fe$_{43}$Co$_{68.2}$Si$_{12.5}$B$_{15}$ wire, which was attractive for technological applications at that
time. Interestingly, the MI effect was first reported in a NiFe-wire, which was subjected to an external magnetic field in 1953 [6]. However, due to the very small observed impedance change, the phenomena did not receive much attention from the research community.

The GMI effect has been explored for a few decades and has received much attention from research and industrial communities. Several fabrication techniques and optimal processing conditions of the amorphous magnetic wires have been developed [7,8]. Furthermore, theoretical models based on classical electrodynamics were employed to explain and predict the GMI response [7,9].

![Fig. 2.1](image_url) *Fig. 2.1* The illustration of the wide frequency range of the GMI operations.

### 2.2 Giant Magneto-impedance (GMI) Effect in Amorphous Microwires

The GMI response in an amorphous microwire has the main contribution from the magneto-inductance effect when the ac excitation frequency is below 1 MHz. The large change in the inductance at a low frequency mainly results from domain wall movement and circumferential permeability when a microwire experiences external magnetic fields [5]. For a higher frequency range (few hundred MHz), the skin effect becomes significant and plays an essential role in the
GMI effect. While the changing penetration depth of the ac current significantly modifies the resistance of the microwire, there is a reduction in the inductive response resulting from the suppression of domain wall motion due to Eddy currents [5,7,8]. Consequently, the GMI response at a high frequency strongly depends on the skin depth and the rotational magnetization process [9]. When the excitation frequency is very high (GHz range), the GMI response shows a broader peak and exhibits a larger $H_k$. The GMI effect in this frequency range is associated with the ferromagnetic relaxation and resonance effect [8].

In this Ph.D. dissertation, focus was placed on the exploration of the GMI effect at the frequency range of 1 MHz – 1 GHz, where the skin effect and rotational magnetization are taken into account for the GMI response.

### 2.2.1 The Skin Effect

The penetration depth of a propagating electromagnetic wave into a good conductor is shown by employing Maxwell’s equations. The incorporation of Maxwell’s equations and the Landau-Lifshitz-Gilbert (LLG) equation is taken into account for the impedance response in high permeability conductors [3, 11].

![Fig. 2.2](image.png)

**Fig. 2.2** The decay of the current amplitude near the surface of the conductor due to skin effect for an ac current.
It is well known that the time-invariant (DC) current flowing in a homogenous and isotropic conductor obeys the Ohm’s law as follows:

\[ \mathbf{j} = \sigma \mathbf{E} \quad (2.1) \]

where \( \mathbf{j} \) is the current density (A/m\(^2\)), \( \sigma \) is the electrical conductivity conductor (S/m) and \( \mathbf{E} \) is the electric field (V/m).

It has been shown that the current density of a DC current flows uniformly over a homogenous and isotropic conductor. However, in the case of a time-varying (AC) current, the current tends to be concentrated near the surface of the conductor. This is due to the Faraday-Lenz law of induction [11,12]. For the quasi-static condition in a good conductor, the Maxwell’s equations state that a sinusoidal electromagnetic field in a cylindrical conductor with conductivity \( \sigma \) and permeability \( \mu \) can be described as follows:

\[ \nabla \times \mathbf{E} = -\frac{\partial \mathbf{B}}{\partial t} \quad \text{Faraday’s law} \quad (2.2) \]

\[ \nabla \times \mathbf{B} = \mu \sigma \mathbf{E} + \mu \varepsilon \frac{\partial \mathbf{E}}{\partial t} \quad \text{Ampere’s law} \quad (2.3) \]

By applying the curl of Faraday’s law and using the vector identity \( \nabla \times (\nabla \times \mathbf{E}) = \nabla (\nabla \cdot \mathbf{E}) - \nabla^2 \mathbf{E} \) and substituting the result into Ampere’s law,

\[ \nabla \times (\nabla \times \mathbf{E}) = \nabla (\nabla \cdot \mathbf{E}) - \nabla^2 \mathbf{E} = -\frac{\partial (\nabla \times \mathbf{B})}{\partial t}, \quad (2.4) \]

the result recovers a wave equation:

\[ \nabla^2 \mathbf{E} - \mu \sigma \frac{\partial \mathbf{E}}{\partial t} - \mu \varepsilon \frac{\partial^2 \mathbf{E}}{\partial t^2} = 0. \quad (2.5) \]

The general solution to the wave equation can be written as:
The dispersion relation can be achieved by substituting equation (2.5) into equation (2.4). Thus,

\[ \vec{k}^2 = i\omega\mu\sigma + \omega^2\mu\varepsilon. \] (2.7)

The wave number, \( k \), in equation (2.6) can be expressed in the real and imaginary forms

\[ \vec{k} = \vec{\alpha} + \vec{\beta}i \] (2.8a)

where,

\[ |\vec{\alpha}| = \omega \sqrt{\frac{\mu\varepsilon}{2} \left(1 + \sqrt{1 + \frac{\sigma^2}{\omega^2\varepsilon^2}}\right)^{1/2}} \quad \text{and} \quad |\vec{\beta}| = \omega \sqrt{\frac{\mu\varepsilon}{2} \left(1 + \sqrt{1 + \frac{\sigma^2}{\omega^2\varepsilon^2}} - 1\right)^{1/2}} \] (2.8b)

Thus, the propagating wave can be written as:

\[ \vec{E}(\omega, t) = \vec{E}_0 e^{i(\vec{k}\cdot\vec{r} - \omega t)}. \] (2.9)

The dispersion relation in equation (2.7) contains the electromagnetic properties of the medium.

For a good conductor, which has \( \sigma \gg \omega\varepsilon \),

\[ \alpha \approx \beta = \frac{\sqrt{\mu\sigma\omega}}{2}. \] (2.10)

It can be seen from equation (2.9) that the time-dependent electromagnetic field exponentially decays from the surface of the medium. The distance the electromagnetic field penetrates into the conductor is given by

\[ \delta(\omega) = \frac{1}{\alpha} = \frac{2}{\sqrt{\mu\sigma\omega}}. \] (2.11)
where $\delta(\omega)$ is defined as the “skin depth” and strongly depends on properties of the conducting material.

### 2.2.2 High Frequency GMI Effect in Magnetic Wire

In this dissertation, the GMI measurements were taken at a high frequency range. We employ the framework from the theory of magnetism and the dynamical response of the magnetization to explain the origin of high frequency GMI effect as follows:

$$\nabla^2 \vec{H} - \nabla (\nabla \cdot \vec{H}) = \mu_0 \sigma \frac{\partial}{\partial t} (\vec{H} + \vec{M}) \quad (2.12)$$

where $\vec{H}$ and $\vec{M}$ are the external magnetic field and spontaneous magnetization vector, respectively. The dynamical response of the magnetization from the applied external magnetic field can be described by the Landau-Lifshitz-Gilbert (LLG) equation [8,9,13-15]:

$$\frac{\partial \vec{M}}{\partial t} = -\mu_0 \gamma (\vec{M} \times \vec{H}) + \alpha (\vec{M} \times \frac{\partial \vec{M}}{\partial t}) \quad (2.13)$$

where $\gamma$ is the gyromagnetic ratio and $\alpha$ is the damping parameter. $\vec{H}$ and $\vec{M}$ are the external field and spontaneous magnetization within a domain, respectively. By applying the corresponding boundary conditions and solving the coupled equations (2.12) and (2.13) [15-17], the longitudinal impedance of the conductor for any frequency range can be expressed as

$$Z(\omega) = \frac{1}{2} k \alpha R_{dc} \frac{f_0(ka)}{f_2(ka)} \quad (2.14)$$

where

$$k = \frac{(1-j)}{\delta} = (1-j) \sqrt{\mu f \mu_0 \sigma} \quad (2.15)$$
and

\[ R_{dc} = \frac{1}{\sigma} \left( \frac{l}{\pi a^2} \right). \]  

(2.16)

In the case of a weak skin effect [11], the impedance equation can be approximated as

\[ Z(\omega) \approx R_{dc} + \frac{1}{48} \left( \frac{a}{\delta} \right)^4 \]  

(2.17)

For a strong skin effect, the impedance can be expressed by

\[ Z(\omega) \approx \frac{aR_{dc}}{2\delta} - \frac{\omega l}{4\pi a} \sqrt{\frac{2\pi \sigma \omega}{\mu_0}} j \]  

(2.18)

where \( a, l \), and \( R_{dc} \) are the diameter, length, and dc resistance of the wire, respectively.

The complex impedance \( Z \) consists of two components: resistance, \( R \), and reactance, \( X \). In the case of a non-magnetic conductor such as a copper wire, \( R \) increases with the operating frequency \( f_{ac} \) and exhibits a parabolic-like function due to the skin effect. In contrast, the \( X \), which is related to the inductive effect, shows a linear increase with \( f_{ac} \). For soft magnetic materials, the skin effect can be estimated [16] by

\[ \delta_m \approx a \left( 1 - \sqrt{1 - \frac{R_{dc}}{R_{ac}}} \right). \]  

(2.19)

Therefore, \( R \) and \( X \) of the magnetic microwire can be approximated as

\[ R_{ac} = \frac{\rho l}{2\pi \sqrt{(r-\delta_m)\delta_m}}, \]  

(2.20)

and

\[ X = 0.2785\mu_0 l\omega, \]  

(2.21)

respectively. The effective circular permeability (\( \mu_0 \)) can be estimated [18] as
\[ \mu_\varnothing = \frac{2\rho}{\omega \mu_0 \delta m} \]  

(2.22)

2.2.3 GMI figure of Merit

The GMI performance of the magnetic microwires is defined by the change in \( Z, R, \) and/or \( X \) due to the external dc magnetic field. The figure of merit of GMI materials is the GMI ratio [8]. The GMI ratios are defined as follows:

\[ \frac{\Delta Z}{Z} = \frac{Z(H)-Z(H_{\text{max}})}{Z(H_{\text{max}})} \times 100 \% , \]  

(2.23)

\[ \frac{\Delta R}{Z} = \frac{R(H)-R(H_{\text{max}})}{R(H_{\text{max}})} \times 100 \% , \]  

(2.24)

\[ \frac{\Delta X}{X} = \frac{X(H)-X(H_{\text{max}})}{X(H_{\text{max}})} \times 100 \% , \]  

(2.25)

where \( Z(H), R(H), X(H) \) are the impedance, resistance, and reactance of the field \( H \), respectively. \( H_{\text{max}} \) represents the maximum magnetic field by the Helmholtz coil (\( H_{\text{max}} = 115 \) Oe). In some practical cases, the GMI ratio can be defined by some other reference field \( H_{\text{ref}} \) [19]. The magnetic field sensitivity (\( \eta \)) is defined as

\[ \eta_Z = \frac{d}{dH} \left( \frac{\Delta Z}{Z} \right) \]  

(2.26)

\[ \eta_R = \frac{d}{dH} \left( \frac{\Delta R}{R} \right) \]  

(2.27)

\[ \eta_X = \frac{d}{dH} \left( \frac{\Delta X}{X} \right) \]  

(2.28)

where \( \eta_Z, \eta_R, \eta_X \) are the magnetic field sensitivity of the impedance, resistance, and reactance, respectively. The GMI ratio and \( \eta \) of a Co\(_{69.25}\)Fe\(_{4.25}\)Si\(_{12}\)B\(_{13.5}\)Nb\(_1\) microwire (\( d \sim 50 \) \( \mu \text{m} \) and 10 mm in length) are shown in Fig. 2.3 (a)-(c), and (d)-(f), respectively.
Fig. 2.3 The GMI ratio and magnetic field sensitivity of a Co69.25Fe4.25Si12B13.5Nb1 microwire (d~ 50 μm). The magneto (a)-(d) resistance, (b)-(e) reactance, (c)-(f) impedance ratio and the magnetic field sensitivity, respectively.

2.2.4 GMI Effect in Co-Rich Microwires and Magnetic Domain Structures

It is a well known fact that melt-extracted and glass-coated amorphous Co-rich microwires exhibit high GMI effect and are compatible for microelectronic circuit integration. In particular, melt-extracted Co-rich microwires, which possess high circumferential (transverse) permeability, nearly zero magnetostriction ($10^{-7}$), and low coercivity [1-3,7,20] are facile to integrate into microelectronic devices for advanced magnetic field sensors. Furthermore, the GMI-ratio and field sensitivity of the Co-rich microwires were reported to be up to 600 % and 400 %/Oe, respectively [7]. This superior GMI performance is highly promising for a small field detection. As can be seen from Fig. 2.4 (a, b), the GMI-ratio and $\eta$ in a melt-extracted Co-rich magnetic microwire ($f_{ac} = 400$ MHz) are 300% and 150 %/Oe, respectively.
The magnetic domain structure of a Co-rich microwire was hypothesized to have “bamboo-like patterns” by Mohri’s group in 1990 [21]. Basically, the core axial magnetic domains are formed in random orientations during the rapid solidification fabrication process. Then, the outer shell circular domains are formed due to the compressive stress and negative magnetostriction. Several investigations of the magnetic domain structures in Co-rich microwires using MOKE microscopy [22] and magnetic force microscopy (MFM) [23] have been recently reported (see Fig. 2.5). Turning to consider the case of RF excitation, the skin effect and the magnetization rotation of outer shell magnetic domains play a significant role in GMI effect [9]. At a high frequency, the alternating current flows only in a thin surface layer of the microwires (typically less than 10
microns). Therefore, the investigation of the surface magnetic domain structure (SMDS) employing MFM and MOKE are the key to exploring surface magnetic properties in the Co-rich microwires.

**Fig. 2.5** The magnetic domain structures of Co-rich wires. (a) Bamboo-like surface magnetic domains, (b) MOKE microscopy, and (c) MFM of Co-rich wires.

### 2.3 Effect of Annealing on the GMI Performance

The magnetic properties of amorphous materials can be improved through a post-processing treatment such as conventional heat treatment, thermal annealing with the presence of a magnetic field, torsion/tensile-stress annealing, and Joule-heating [3-5, 7].

Conventional annealing or heating amorphous microwires in time- and temperature-controlled air/vacuum furnace has shown the improvement of the magnetic properties of amorphous materials. The annealed samples exhibited magnetically softer property due to the relief of stress and strains generated during the quenching process. However, the conventional heat annealing showed a deterioration of the GMI effect in Co-rich amorphous microwires [8, 24]. Zhukov *et al.* has shown that the GMI effect in glass coated Co-rich microwires decreases after being annealed at 300ºC for 5 minutes [24]. Typically, the microstructures, electrical and magnetic properties were enhanced during the heat treatment. The decrease in the resistivity of the Co-rich microwires was also observed after the conventional annealing [8].
It is well known that applying a magnetic field to the Co-rich amorphous microwire during thermal treatment showed an enhancement of the GMI effect [25,26]. The GMI ratio and magnetic field sensitivity of a melt-extracted Co-rich amorphous microwire was enhanced through multi-angle combined magnetic-field annealing (MCMFA). The proper thermal treatment (573 K for 20 min and was increased up to 673 K for 10 min) in the presence of a 4 kOe magnetic field promoted the GMI ratio and the field sensitivity up to 310% and 30.91%/Oe, respectively. However, the increase in magnetic anisotropy and magnetic hysteresis were observed in the annealed sample due to the irreversible magnetization process [25].

Another effective technique to improve the GMI ratio in Co-rich microwire is torsion/tensile-stress annealing method [8,27]. Recently, it has been reported that applying a small tensile stress to a melt-extracted Co-rich microwire effectively improved the GMI response and their field sensitivity [28]. Further modification of stress annealing is an application of torsion-stress to a sample under conventional heat treatment. This annealing method modified the magnetic domain structures and promoted the GMI effect at a low field region in a FeSiB and CoSiB wire. As a result, the improved GMI response can be applied for small field sensing applications.

In Comparison with the aforementioned methods, the Joule heating has more advantages the others due to the thermal treatment under self-induced circular magnetic field to enhance the GMI effect. Furthermore, the application of a dc current intensity below the crystallization temperature and the Curie temperature of the materials maintains the great mechanical properties as well as improvement of the soft magnetic properties of such materials, Conventional/modified approaches of Joule heating, such as cryogenic Joule [29], twin-zone [30], combined current-
modulation [31], square-wave pulse current [32], and accumulated DC annealing [33] have been used to improve the GMI response in Co-rich wire/microwire. Basically, these post-processing treatments employ a small dc current flowing through the amorphous wire/microwire for an appropriate time. Consequently, the heat treatment from the DC-current relieved the residual stress generated during rapid quenching causing the microstructure to be relaxed. Furthermore, some local defects in the amorphous matrix are refined as a result of the thermal treatment. In the meantime, the self-induced circumferential magnetic field enhances the surface magnetic domain structures in the circumferential direction to promote the transverse magnetic anisotropy. In addition, the resistivity of the amorphous wire/microwire decreases due to the evolution of the removal of microstructure defects. Indeed, the formation of nanocrystals embedded in the amorphous matrix becomes remarkably distributed after the annealing [33,34]. Thus, the annealed microwires possess softer magnetic properties compared to the as-prepared [31,34,35]. Moreover, with proper heat treatment, the nano-crystalline size is more significant and plays a significant role in reducing the resistivity [31,35]. Application of a current intensity generating a temperature higher than the material crystallization and Curie temperature could introduce local magnetic anisotropy from partial crystallization into the microwires. As a result, the GMI properties of the annealed microwires can be deteriorated [8]. Figure 2.6 shows the enhancement of the GMI response of the Co-rich microwire after a Joule heating treatment (100 mA for 10 min).

As was mentioned previously, the high frequency GMI effect in a Co-rich microwire strongly depends on the skin effect and rotational magnetization process of the transverse magnetic domains. During Joule heating, the self-induced magnetic field, one key parameter of enhancing the surface domain structure, promotes the transverse magnetic anisotropy in the Co-rich microwire [8,36]. Consequently, the GMI response increases due to the higher transverse
permeability promoting ease of magnetization rotation [5,16,36]. The well-defined SMDs of an annealed Co-rich microwire using MFM have been explored [31]. The SMDs of the as-prepared microwire are not as well defined [31,34,35]. After the Joule annealing treatment, the circular domain structure is present, which is consistent with the other reports through MOKE measurement [37]. The acquired transverse anisotropy of SMDs in the annealed Co-rich microwire is shown in Ref. 37. The hysteresis loop from a longitudinal MOKE signal shows an induced perpendicular anisotropy in the annealed microwire [37-39], which promotes the GMI effect in the Co-rich microwire. Therefore, the Joule heating method is a versatile technique to optimize the GMI effect and mechanical properties of Co-rich amorphous microwires.

![Fig. 2.6](image)

**Fig. 2.6** The enhancement of GMI response of a Co-rich microwire using Joule annealing.

### 2.4 High Frequency Magneto LC-resonance Effect

Generally, an electrical circuit is a connection of a voltage source ($\varepsilon$) and circuit elements such as a resistor ($R$), inductor ($L$), and/or capacitor ($C$) together in parallel and/or series fashion (see Fig. 2.7). The supplied voltage can be either in the form of dc or ac sources, which depends on the purpose of the circuit. Basically, a resistor is considered as a passive device and dissipates
energy in a circuit. The capacitor and inductor are circuit components that can store energy in electrostatic and magnetic forms, respectively. For example, a capacitor stores energy through the buildup of electrostatic energy from its charging process. An inductor stores energy through a created electromagnetic field when the current flows through inductor. These circuit elements are well known and show an ideal response when they are subjected to a dc current source or a low frequency ac-source (below 1 MHz) [40-42].

![Fig. 2.7 Schematics of (a) series and (b) parallel connections of ac currents.](image)

2.4.1 The RLC Resonance Circuits

Recently, LC resonant sensors and their integrated circuits have been widely used for real-time environmental sensing [43], food quality monitoring [44], biomolecule detection [45] and health care device applications [46]. To appreciate the nature of the resonance effect in an LC circuit, consider a simple case of an RLC circuit containing $R$, $X$, and $C$ in series and with a voltage source $E(t)$. The circuit is shown in Fig. 2.7 (a). By applying the Kirchhoff’s rule for a loop of current, the sum of the potential difference around the single loop can be written as

$$L \frac{dI}{dt} + RI + \frac{q}{C} = E(t).$$  \hspace{1cm} (2.29)

By taking $I = I_0 e^{i\omega t}$ and $q = I/i\omega$, then the equation can be written as
\[ i\omega LI_0 + \frac{l_0}{\omega C} + RL_0 = \varepsilon_0 e^{i\omega t}. \] (2.30)

Then, the current as a function of the time is
\[ I(t) = \frac{\varepsilon_0 e^{i\omega t}}{R + i(\omega L - \frac{1}{\omega C})}. \] (2.31)

We employ \( Z = \frac{\varepsilon(t)}{I(t)} \), the complex impedance of the current loop becomes
\[ Z = R + i \left( \omega L - \frac{1}{\omega C} \right) = R + Xi \] (2.32)

where \( |Z| = \sqrt{R^2 + \left( \omega L - \frac{1}{\omega C} \right)^2} \) and \( \phi = \tan^{-1}\left( \frac{X}{R} \right) \).

Series or parallel ac circuits are interesting when the phase angle \( \phi \) becomes zero because they exhibit resonance phenomena. The resonance frequency of the circuit can be written as
\[ \omega = \frac{1}{\sqrt{LC}} \] (2.33)

For the series circuit, the current \( I(t) \) attains its highest value at this point. Similar analysis can be used for the parallel case, however, the complex impedance reaches a maximum for the parallel circuit when the excitation frequency attains the resonance frequency. The impedance spectrum of series and parallel resonance circuits are shown in Fig. 2.8. As can be seen the sharpness of the resonance curve depends on its resistance and can be determined using the dimensionless quantity “Q”. This Q value is known as “quality factor at resonance” and can be defined as
\[ Q = \frac{\omega L}{R} = \frac{1}{R} \sqrt{\frac{L}{C}} \] (2.34)
The Q factor is used to determine the bandwidth of the resonator and the degree of energy loss. It is interesting to remark that the characteristic frequency of the parallel resonance has been applied for advanced sensing applications.

![Fig. 2.8 The current and impedance responses of the RLC resonance circuits.](image)

### 2.4.2 The Non-ideal Behavior of an Inductive Coil

In the absence of a resistance component, the circuit is called “LC resonator” or “tank circuit”. Recently, the impedance spectrum of LC resonators has been proposed for modern sensing applications. For example, a resonance circuit has been used to remotely monitor the bacterial respiration and bacteria detection on a tooth [46]. In this case, an inductively-coupled LC tank circuit has been integrated into a microfluidic resonance detection (MRD) device [45], a parallel 3D-printed LC-resonance circuit was employed to monitor the quality of liquid food [44], and the real-time monitoring of the growth of bacteria in the environments using the coupled resonance circuit and wireless communication has been demonstrated [43].

As was mentioned previously, the elements of a resonance circuit are well behaved for a low-excitation frequency. The non-ideal behavior of the components, however, is observed when
the operating frequency is up to the RF-frequency range [40-43]. For instance, a carbon-composition resistor, made of a carbon thin film or granular carbon, exhibits decreasing resistance with increasing frequency. A wire-wound resistor behaves like an inductance for a high frequency operation. These non-ideal behaviors of the components are due to the manufacturer construction of the elements [40].

Since an inductor is fabricated by a wound conductor or coiled wire, the inductor’s response exhibits a stray capacitive effect at a high frequency operation. In particular, the close proximity of the coils becomes significant for RF excitation due to a strong skin effect. A voltage between each of the coils is analogous to small capacitors connected in series. The theoretical model for the complex impedance of the inductor has been proposed in Ref. [43] (see Fig. 2.9) and can be expressed as

\[
Z(\omega) = R_s + jX_s, \quad (2.35)
\]

where

\[
R_s = \frac{R}{(1-\omega^2 L C)^2 + (\omega R C)^2} \quad (2.36)
\]

and

\[
X_s = \frac{\omega (L - R^2 C) - \omega^2 L^2 C}{(1-\omega^2 L C)^2 + (\omega R C)^2} \quad (2.37)
\]

where \(C, R,\) and \(L\) are the stray capacitance, dc resistance, and inductance of a solenoid.
2.4.3 Magneto LC Resonance Effect

The utility of a magnetic microwire integrated with an inductive circuit has been demonstrated by Le and Phan [47], Devkota et al. [48], He and Shiwa [49], and Zhukov et al. [50]. The Co-rich magnetic microwire was introduced as a core sensing element into an inductive coil. Thus, the coil inductance increases when it experiences an external magnetic field. The inductive coil sensor showed great sensitivity when it was employed to scan Japanese thousand-yen bill, which was printed with magnetic ink. Moreover, the field sensitivity of multiwire-based coil can reach up to 1957 %/Oe ($f_{ac} \sim 0.1$ MHz), which paves a pathway for a highly sensitive magnetic sensor [51].

The concept of integrating a magnetic microwire and LC-resonance circuit was demonstrated by Kim et al. [52] and Le et al. [53]. In this configuration, a micro-inductor with a microwire core was fabricated. The microinductor was integrated with an external capacitor to create an LC resonance effect. Interestingly, the GMI ratio of the integrated LC-resonance based sensor can reach up to 400,000% measured at a resonance frequency of 518.51 MHz. However, the LC-resonant GMI curves exhibit a broader peak response located at large applied field ($H_{ext} \sim$...
25 Oe). Therefore, the employment of the GMI sensing element coupled with LC resonant circuits may be useful for high-field sensing applications. In addition, the LC-type magneto-impedance (LCMI) sensor device has been fabricated and developed for magnetic field detection. The resonance frequency of the LCMI sensor is given as follows [53]:

\[
\omega = \frac{1}{\sqrt{LC - \left(\frac{R}{L}\right)^2}}
\]  

(2.38)

where \(\omega\) is the angular frequency of the integrated LC tank circuit. \(R\), \(L\), and \(C\) are the resistance, inductance, and capacitance of the LCMI circuit.

### 2.5 GMI-based Sensor Applications

Since the demonstration of the GMI effect in the Co-rich wire in 1994, much interest has been focused on the utility of the GMI sensing element for magnetic sensor applications. One year later, a micro-magnetic sensor based on the GMI effect in the Co-rich wire was introduced for highly sensitive magnetic sensors [54]. Then, the first sensitive linear micro-magnetic sensor based on the GMI effect incorporated with a pick-up coil was illustrated [55]. A few years later, Aichi Steel Company developed an electronic compass from an MI-sensor chip for commercial smart phones and wrist watches [56].

The concept of using a GMI sensor for position detection was demonstrated in 1996 by Valenzuela et al. [57]. The Co-rich wire fabricated by the in-rotating-water technique was used to construct a GMI-based sensor for industrial applications. The detection of a moving object with a permanent magnet attached has been proposed for object tracking. Moreover, the transportation traffic monitoring using a CMOS MI sensor has been developed as a prototype for traffic control [58]. Then, the utility of a GMI sensor for automobile guidance was demonstrated by Honkura
For this system, the MI sensor with a linear response and temperature stability was placed in front of the demonstration car and it detected magnetic markers embedded into the road. Thus, the GMI sensor is highly promising for transportation census and monitoring a vehicle location.

Recently, the GMI-based sensors have been used for biomolecule/functionalized magnetic particle detection. The CoFeSiB amorphous glass coated microwire integrated with a functionalized polymer has been used for detecting amorphous micro-particles and superparamagnetic beads [60,61]. Amorphous particles of diameter ~ 40-60 μm were detected by the functionalized MI sensor [61]. Therefore, the Co-rich microwire has the potential to become a GMI-based biosensor. However, to obtain the higher response and be able to detect a smaller particles/lower concentration, a more sensitive sensing element should be developed. Moreover, the contactless measurement using a Co-rich wire/microwire would be a new generation of magnetic biosensors.

![Fig. 2.10](image)

**Fig. 2.10** (a) GMI response of the MI sensing element and (b) the nT (nanotesla) sensor, which was invented by Aichi Micro Intelligent Corporation. Reproduced from ref. [62], with the permission of Aichi Steel Corporation.

To date, Aichi Steel Company distributes a new commercial MI-sensor technology for nT field detection [62]. The GMI response of the amorphous wire of the MI sensing element and the
commercial magnetic sensor (nT sensor) are shown in Fig. 2.10. Due to its ultra-high sensitivity, high speed response, and low power consumption, the MI sensor is highly promising for biomagnetic field sensing, chemical measurements, and industrial detection. The proposed major applications are electronic compasses, vehicle sensors, rotary detection in automobile, non-destructive testing, safety registration, and food quality control, and bio-magnetic detection (see Fig. 2.11).

![Diagram of magnetic fields and potential applications](image)

**Fig. 2.11** The potential applications of the MI-sensor proposed by Aichi Micro Intelligent Corporation. Reproduced from ref. [62], with the permission of Aichi Steel Corporation.

### 2.6 References


CHAPTER THREE

EXPERIMENTAL METHODS

This chapter presents the experimental methods used in this dissertation. It begins with the description of the fabrication technique of the amorphous magnetic microwires. The sample preparation, as well as novel Joule heating treatments are explained. The structural and morphological characterizations of the magnetic microwires including an X-ray diffractometry (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), and High-resolution Transmission Electron Microscopy (HRTEM), are discussed. Furthermore, measurements of the magnetic properties of the microwire samples by employing Magneto-optic Kerr Effect (MOKE) magnetometry and Magnetic Force Microscopy (MFM) are described. Finally, high-frequency magneto-impedance measurements using the HP4191A RF impedance analyzer are explained.

3.1 Fabrication of Melt-Extracted Magnetic Microwires

It is established that rapid quenching of certain melt alloys forms an amorphous metallic alloy [1, 2]. In general, amorphous metallic alloys do not have crystalline structures, which is known as “non-crystalline solid” or “bulk metallic glass”. Their atoms are randomly distributed and are lacking of long-range atomic order. Consequently, they exhibit high electrical resistivity due to carriers scattering from the atomic disorder [3]. Owing to non-crystalline structure of atoms, the amorphous metallic materials possess low magnetic anisotropy and ultra-soft ferromagnetic
properties. Recently, there have been some interests in the mechanical and soft magnetic properties of such materials in terms of the fundamental studies and industrial applications [3-5].

As was mentioned earlier, amorphous magnetic wires are highly promising for technological applications, e.g., magnetic sensors, bio-detection, and microwave absorption. Several well-known fabrication techniques [6,7], such as Taylor-wire process, in-rotating-water quenching, and melt-extraction methods have been employed for the amorphous microwire fabrication. However, the melt-extraction method has some advantages over other methods; it processes high rapid solidification rate (up to $10^6$ K/s), provides faultless surface and cylindrical geometry, and maintains superior magnetic properties of the fabricated wires [8]. Therefore, the melt-extraction method has been employed in order to produce the high quality Co-rich microwires in our experiments.

Firstly, the mother alloys of Co (99.999%), Fe (99.99%), Si (99.999%), B (99.99%), and Nb (99.99%), each with an arbitrary nominal composition were gathered in a Boron nitride (Bn) crucible under a metal wheel (quench wheel). Then, the composite alloys were melted at high temperature using an induction coil. Following that, the amorphous microwires with diameter ~20-60 μm were extracted by the copper wheel (3.2 cm in diameter and 60 degrees of the knife edge angle) under pure Argon. The feeding rate of the molten alloy was maintained at ~30 μm/s and the linear speed of the quenching wheel was kept constant at 30 m/s. The schematic diagram of the melt-extraction method and the picture of an experimental set-up for the melt-extraction are shown in Fig. 3.1. In addition, the microwire diameter was controlled by the speed of the quenching wheel and the dip-level of the rotating wheel. Consequently, high quality Co-rich microwires of uniformly circular shape were attained. The typical length of the quenched microwires is few
meters. All high quality microwire samples that were used in our research were fabricated and provided by our collaborator, Prof. Sun’s group of Harbin Institute of Technology (HIT), Harbin, China.

Fig. 3.1 (a) A schematic diagram of the melt-extraction method, and (b) a picture of the experimental setup for the melt-extraction machine (courtesy of H. Shen from HIT).

3.2 Sample Preparation

Amorphous magnetic microwires with nominal compositions of Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{13.5}$, Co$_{69.25}$Fe$_{4.25}$Si$_{12}$B$_{13.5}$Nb$_1$, and Co$_{69.25}$Fe$_{4.25}$Si$_{12}$B$_{13.5}$Zr$_1$ (in at.%) were fabricated by means of the melt-extraction method, which was mentioned previously. The fabricated samples have a uniform circular shape and a typical size distribution ~30-60 µm in diameter. In our experiments, we chose high quality Co-rich microwires from a long strand and then measured their diameters using a micrometer. The selected microwire was cut and soldered to SMA ports employing a heated solder gun and a solder wire. Fig. 3.2 (a)-(b) shows both the optical and SEM images of as-cast Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{12.5}$Nb$_1$ microwires. A single microwire and mounted microwire on copper ground plane and micro-strip holders are shown in Fig. 3.2 (c)-(d), respectively.
3.2.1 Joule Heating with Single-step Current Annealing

Firstly, three sets of as-cast Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{12.5}$Nb$_{1}$ microwires with a diameter of about 40 μm and 10 mm in length were prepared for the low current annealing. The microwires were annealed with dc current with magnitudes of 5 mA, 10 mA, 20 mA, and 30 mA for 10 minutes, in order to understand the effects of dc current annealing on the GMI response. Then, the second and third sets were annealed with the same current intensities for 20 and 30 minutes (see Fig. 3.3), respectively. The experimental setup for the single-step Joule heating is shown in Fig. 3.4.
Fig. 3.3 Schematic diagrams showing the single-step and multi-step Joule heating treatments (from left to right).

3.2.2 Joule Heating with Multi-step Current Annealing

Further study is aimed at enhancing the GMI response by employing the multi-step dc current annealing method. Three sets of as-cast Co$_{69.25}$Fe$_{4.25}$Si$_{12}$B$_{13.5}$Nb$_{1}$ microwires with a diameter of ~50 μm and 10 mm in length were prepared. The first sample, the as-cast microwires, was mounted on a sample holder using a soldering material. Then, the GMI-response for each as-cast sample was investigated. The as-cast microwires were subjected to a dc current of 100 mA for 10 minutes as a single-step current annealing (SSA). For multi-step current annealing (MSA), the as-cast microwires were firstly subjected to a 20-mA dc current for 10 minutes (see Fig. 3.3), and then the magnitude was increased by 20 mA for every 10 minutes until the annealed current intensity reached 300 mA. These parameters were tuned in order to optimize the GMI response following our previous systematic study [9]. The experimental setup for the Joule heating treatment is illustrated in Fig. 3.4.
3.3 Characterization of Melt-extracted Magnetic Microwires

3.3.1 Structural Characterization

We characterized the Co-rich melt-extracted microwires by an x-ray diffractometry (XRD) using a Bruker AXS D8 Focus Diffractometer. The atomic weight percentage of the elements present in the amorphous microwires was investigated by means of energy dispersive spectroscopy (EDS) using a JEOL JSM-6390LV SEM. The composed elements of the sample were confirmed using the EDS technique. The surface nano-crystallites of Co-rich melt-extracted microwires were characterized using a high-resolution transmission electron microscopy (HRTEM) using Tecnai G2F30 TEM.

3.3.1.1 X-ray diffractometry. An x-ray diffraction is a common method that is used to investigate crystal structures and atomic spacing of crystalline materials. An x-ray was discovered by Roentgen in 1895 [10]. Later on, the interatomic spacing in the crystalline (~ 1 Å), which acts
as three dimensional diffraction grating for the newly discovered x-ray was demonstrated by Laue [11]. Owing to the nature of electromagnetic radiation, an x-ray exhibits wave properties, i.e. reflection, interference, diffraction, and polarization. Therefore, the interaction of the incident rays with the crystal planes of the atoms produces successive constructive interference patterns when the conditions satisfy Bragg’s law:

\[ 2d_{hkl} \sin \theta = n\lambda, \]  

(3.1)

where \( n \) is an integer and \( d_{hkl} \) is the inter-planar lattice spacing. \( \theta \) is the angle of diffraction and \( \lambda \) is the wavelength of the x-ray that is generated by the diffractometer.

Basically, the x-ray diffractometer consists of three components; an x-ray tube to generate an x-ray, a sample holder and an x-ray detector as shown in Fig. 3.5 (a). An x-ray is produced when the beam of electrons strikes a metal target, i.e. Mo, Cr, Fe. Then, the x-ray beam is collimated to imping on the parallel planes of atoms. The constructive interference of the diffracted beam occurs when the condition satisfies the Bragg’s Law and is detected by a scintillation detector. For different configurations, the design can either rotate the sample holder or the x-ray source and detector to perform the diffractometer operation. Typically, the x-ray diffraction is used for non-destructive characterization of materials properties such as lattice constant determination, phase identification, crystal structures, orientation, thin-film thickness and quantity [9-11]. The characterized materials can be in the forms of powder, thin-film, single crystal, or bulk.
In our experiment, we employed a Bruker-AXS D8 Focus powder diffractometer with Cu anode ($\lambda \sim 1.5406 \text{ Å}$) for the structural characterization of the microwire samples. The as-prepared and annealed microwires were cut into very small pieces and were mounted on a sample holder made of white nylon, respectively. Then, the sample holder was mounted on a sample stage. The scanning angle was started at 2$\theta$ from 20 to 80 degrees using 0.05 degree increment. The scan speed was set-up for 5 degrees per minute. The measurement process was controlled by the XRD Commander program created as a user-friendly interface on a computer.

3.3.1.2 Scanning electron microscopy. The capability to obtain topographic images from nanometer (nm) to micrometer ($\mu$m) scale has made Scanning Electron Microscopy (SEM) a powerful tool for microstructural characterization and surface morphology analysis [13]. The high-resolution micrograph of the SEM that is obtained from the interaction between the electron beam
and a micro-volume sample, i.e., back scattering electron, secondary electron, and characteristic x-ray allows the observation of surface morphology of solid materials. Moreover, the measurement of the characteristic x-ray from the stimulated high-energy electron beam allows the composition analysis. This material characterization method is known as “Energy Dispersive x-ray Spectroscopy (EDS)” [14].

The image formation of the SEM is quite sophisticated; however, the back scattering electrons and the offspring from the interaction between the beam and sample are the key information to produce the SEM images. **Fig. 3.6 (a)** illustrates the interaction between the beam electrons interacting with a specimen. As can be seen, the back scattering electron, secondary electron, and characteristic x-ray, which are the offspring from the interaction, are employed to form the SEM images. **Fig. 3.7 (b)** shows the characteristic x-ray that is generated from the transition electron from the K and L shells. These emitting spectra are used for the EDS element composition analysis.

**Fig. 3.6 (a)** An interaction between the SEM beam and a sample, and **(b)** the characteristic x-ray that is generated from the transition electron from the K and L shells.
In our measurement, the magnetic microwires were cut and mounted on the SEM sample holder using high conductive carbon tape. The JEOL JSM-6390LV Scanning Electron Microscope was used to explore the surface morphology of the samples. Energy Dispersive Spectroscopy (EDS) system integrated with the JEOL JSM-6390LV SEM was employed to investigate the nominal composition of the present elements in the Co-rich microwire samples. Fig. 3.7 (a)-(c) shows the JEOL JSM-6390LV SEM, mounted samples, and SEM micrograph, respectively.

Typically, the JEOL JSM-6390LV SEM is operated in an automatic vacuum system ~7.5×10⁻⁷ Torr for a high-vacuum mode. A collector, light guide, and photomultiplier tube are used to produce secondary electron imaging (SEI). The compositional contrast, topological, and shadow imaging are produced from the back scattered electron imaging (BEI) detected by a semiconductor device. The magnification range is from 30x to 3000,000x. Moreover, the EDS system (INCAx-sight) operated under liquid nitrogen cooled environment is used for qualitative and quantitative element analyses of the microwire samples.

Fig. 3.7 (a) JEOL JSM-6390LV Scanning Electron Microscope, (b) Mounted microwire samples on the SEM holder, (c) SEM micrograph of a magnetic microwire.
3.3.1.3 High-resolution transmission electron microscopy. Attaining ultimate spatial resolution down to atomic level plays an essential role in modern microscopy. At present, the observation of atomic configuration of solid state materials through high-resolution transmission electron microscopy (HRTEM) provides useful information for chemical occupancy, lattice defect, and interface analysis [14,15]. Recently, the discovery of new materials, e.g., topological insulators, two dimensional Wander-Wall materials, amorphous magnetic materials has employed HRTEM to detect their atomic spacing and structure levels. Therefore, the attainment of high-resolution imaging at the atomic level is crucial for modern material analysis [16].

It is established that the obstacles for optical microscopy at the atomic level resolution is the diffraction limit from the light source. The SEM microscopy has spatial resolution ~10 nm due to the interaction volume of electron beam and specimen. The HRTEM has advantage over other techniques because it utilizes an interference pattern from the interacting electron, which is diffracted from the specimen. The analysis of the interference pattern using the phase shift allows the formation of the resolution of TEM images ~0.2 nm [14]. In general, the TEM can be operated in three different modes based on the image types (bright, dark, and high-resolution images), as shown in Fig. 3.8. When the transmitted electron beam is selected through the objective aperture, the bright-field image is formed. On the otherhand, if the diffracted electron beam is allowed to pass the aperture to produce the dark-field image, the high-resolution image can be achieved when both the transmitted and diffracted electrons are allowed to pass the aperture. Then, the phase information from the interference is analyzed to form high-resolution images. A more detailed analysis of image processing and HRTEM simulation are presented in Ref. 10.
Fig. 3.8 The modes of the TEM operation. (a) The bright-field and (b) dark-field.

In our experiment, the microstructure of the as-prepared and annealed microwires was characterized by HRTEM method using a Tecnai G2F30 TEM microscope. The magnetic microwires were prepared by using a Gatan 691 ion beam thinner. In order to reduce the sample thickness down to a nanometer scale, the ion beam thinners were focused on the samples for 3-5 hours using an applied voltage of 3.5 kV. The details of the HRTEM sample preparation can be found in Ref. 17. Then, the thin-layer of the magnetic microwire was used for the HRTEM microscopy. The HRTEM microscopy of the samples was performed by our collaborator, Prof. Sun’s group of Harbin Institute of Technology, China.
3.3.2 Measurements of Magnetic Properties

In this sub-section, the surface magnetic domain structure (SMDS) of the magnetic microwires is discussed. We employed a Magnetic Force Microscopy (MFM) and magneto-optics Kerr effect to investigate the SMDS of Co-rich microwires, which is explained in the following sub-section.

3.3.2.1 Magnetic force microscopy. Scanning probe microscopy has been widely used in modern microscopies, i.e., scanning tunneling microscopy (STM), atomic force microscopy (AFM), and scanning near-field optical microscopy (SNOM). Basically, these microscopies employed the tunneling current between a conducting probe and the surface atoms of a specimen, the van der Walls force acting on a tip and a sample, and scattering light through a sub wavelength and optical fiber guidance to produce images, respectively. However, these methods do not allow the quantitative investigation of magnetic property of materials that are being examined [18,19].

The spatial resolution of magnetic imaging, which is equal to 100 nm was first demonstrated in 1987 [18]. The magnetic force between a magnetized tip and a thin-film recording head was observed. Later in the same year, the surface domain distribution of Ni sample was examined through the development of single domain tip of AFM. These findings were highly promising for the submicron quantitative analysis of magnetic media. Nowadays, the high-resolution of magnetic force microscopy (MFM) has been widely used for quantitative analysis of domain image in magnetic materials. In general, MFM measures the intensity of the stray magnetic field generated by the sample, which is acting on a magnetized tip. The schematic illustration of the magnetic tip and the sample is shown in Fig. 3.9. As can be seen, the magnetized tip detects the magnetic force owing to the interaction between the MFM tip and the stray field of the sample. The magnetic force acting on the tip can be expressed by [19]:

\[\text{force} = \text{MFM} \times \text{sample} \times \text{tip} \]
$$F_z(r) = \int \mu_0 m(\hat{r}) \cdot \frac{\delta^2 H}{\delta z^2}(r + \hat{r}) \, d^3r,$$

(3.1)

where $m(\hat{r})$ is the magnetization of a volume element of the tip and $H$ is the stray magnetic field acting on the magnetic tip.

![Fig. 3.9 (a)](image)

(a) The basic principle of MFM microscopy, and (b) a schematic of the magnetized tip and magnetic sample.

Recently, new materials such as magnetic topological insulators, two dimensional van der Waals materials, which are promising for technological application, have employed the MFM to characterize the magnetic response of such materials. Moreover, the MFM is capable of investigating the magnetic domain structures of nano-patterning reconfigurable magnetic landscapes, domain wall motion in sub-micron magnetic wires as well as the magnetic domains of Fe-based magnetic microwires. Therefore, MFM is a versatile technique and is suitable for characterizing the SMDS of magnetic microwires.

In this experiment, we employed MFM technique to characterize the SMDS of the as-prepared and annealed microwires. The MFM images were conducted using a Bruker Dimension Icon in a tapping-lift mode where the cantilever is driven slightly below its natural resonance.
frequency to maximize the change in the oscillation amplitude. The lift height was kept constant at 100 nm for all samples [20]. The MFM microscopy was performed by our collaborators from Harbin Institute of Technology, China.

3.3.2.2 Magneto-optic Kerr effect (MOKE) magnetometry. The interaction between the electromagnetic wave and magnetic materials provides a power tool for the studies of magnetic samples. Typically, the magneto-optic effect refers to the change in the optical properties of media when the materials interact with an external magnetic field [21]. This effect can be observed through the transmitted/reflected electromagnetic radiation. There are two important magneto-optic phenomena: Faraday and Kerr effects.

![Fig. 3.10](image)

Fig. 3.10 The schematic diagrams showing (a) polar, (b) longitudinal, and (c) transverse Kerr effect.

The phenomenon is known as “Faraday effect” when light propagates through a transparent medium and such propagation strongly depends on the rotation direction of sample’s magnetization. On the other hand, the “Kerr effect” occurs when the light is reflected from a magnetized material in the presence of magnetic fields. For the latter case, the reflected light from the ferromagnetic materials can be used to investigate the magnetic properties of materials. There are three types of the basic study of Kerr’s effect: polar, longitudinal, and transverse Kerr effect.
They are based on the orientation of the sample magnetization with respect to the plane of incidence of the light beam, as shown in Fig. 3.10.

In fact, the Faraday and Kerr effects originate from the spin-orbit interaction and Zeeman effect [20]. In the macroscopic level, however, the Kerr effect is described by the dielectric tensor of the material when the light interacts with matter. The dielectric tensor ($\varepsilon$) of an isotropic media can be expressed by

$$
\varepsilon = \begin{pmatrix}
\varepsilon & \hat{\varepsilon} & 0 \\
-\hat{\varepsilon} & \varepsilon & 0 \\
0 & 0 & \varepsilon
\end{pmatrix}
$$

(3.1)

where $\varepsilon$ is a real number, and $\hat{\varepsilon}$ is an imaginary number. This imaginary part of the dielectric tensor strongly depends on the magnetization of the ferromagnetic materials.

In our experiment, the homemade MOKE magnetometer was employed to investigate the surface magnetic properties of as-prepared and annealed microwires. The homemade MOKE setup consists of a monochromatic light source ($\lambda \sim 650$ nm), linear polarizers, a converging lens, a semiconductor photodetector, a lock-in amplifier, a bipolar power supply, and a Helmholtz coil. The schematic diagram and the experimental setup for the MOKE measurement are shown in Fig. 3.11, respectively.

Firstly, the sample was mounted on a non-magnetic holder in the longitudinal direction, which is parallel to an axial magnetic field ($H_{dc}$) generated by the Helmholtz coil ($H_{max} = 115$ Oe). This sample holder was attached to a three dimensional translation. The red laser and photodetector were mounted to an optical table where the angle of incidence and reflection is $\sim 45$ degrees.
Secondly, the arbitrary directions of the polarizers were selected to be perpendicular to each other in order to observe the polarization rotation. The converging lens was introduced between the sample and the polarizer to focus the reflected light from the sample onto the photodetector.

![Diagram](image)

**Fig. 3.11** (a) A schematic diagram showing the setup for MOKE magnetometer, (b) An experimental setup for the MOKE measurement.

Finally, we employed the lock-in amplifier to modulate the light source frequency \((f \sim 1.5 \text{ kHz})\) and analyze the collected light intensity from the photodetector. The numerical values of the
light intensity and the applied magnetic field were plotted in the form of xy-graph using a LabVIEW data acquisition program and are recorded as a text file for further analysis. All measurements were controlled through a general purpose interface bus (GPIB) using the LabVIEW program.

3.4 High-frequency Magneto-impedance Measurements

3.4.1 Working Principle of HP 4191A RF Impedance Analyzer

A network analyzer has played an important role in measuring circuit performances and the device under test (DUT). Instead of using voltage-current-ratio, the network analysis provides more accurate and precise measurements by measuring the impedance circuit response (low frequency method). Moreover, the network analysis is one of the versatile tools that is used to study material properties.

For a radio frequency (RF) measurement, the sinusoidal signal is generated from the impedance source ($Z_s$) and is transmitted to the impedance load ($Z_L$) in order to consider a transmission and reflection wave from the DUT. The reflected wave becomes zero if the load impedance is equal to the characteristic impedance ($Z_0$). On the other hand, the sinusoidal wave will be reflected back toward the source if the $Z_L$ is not equal to $Z_0$. This reflected wave is used to obtain the desired characteristics of the sample [22]. The ratio of the reflected wave to the incident wave is defined as the reflection coefficient $\Gamma$ and can be expressed as [22, 23]:

$$\Gamma = \frac{V_{\text{ref}}}{V_{\text{inc}}} = \Gamma_x + j \Gamma_y,$$  \hspace{1cm} (3.1)

In practice, the measured length of the propagation line is automatically compensated through the standard calibration procedures. The impedance value of the load can be expressed as [24]:

58
where \( j \) is an imaginary unit, \( Z_0 \) is the internal impedance of the analyzer (50 \( \Omega \)), \( R \) and \( X \) are the resistance and reactance of the test sample, respectively. The relationship between \( R \), \( X \), \( \Gamma \) and \( Z_0 \) can be written as:

\[
R = Z_0 \frac{1 - \frac{\Gamma^2 x^2}{1 - \frac{\Gamma x}{(1 - \frac{\Gamma x}{2})^2 + \frac{\Gamma y}{2}}}}{1 - \frac{\Gamma x}{1 - \frac{\Gamma x}{2}}}
\]

\[
X = Z_0 \frac{2 \Gamma y}{1 - \frac{\Gamma x}{2}}
\]

**Fig. 3.12** A schematic diagram showing a high-frequency magneto-impedance measurement through a transmission line.

### 3.4.2 Standard Calibration

Firstly, after turning-on the HP4191A RF impedance analyzer, a standard calibration is mandatory for all measurements. We selected the feature “Local” from the machine front-panel in order to control the HP4191A manually from its front panel. Then, we chose the target frequency range between 1 MHz – 1000 MHz using the front panel of the HP4191A. As 50 increments are allowed per frequency calibration, we attained 50 target-frequencies for the impedance
measurement. In fact, the smaller frequency span can be selected, however, only 50 target-frequencies is calibrated.

Secondly, the transmission line was mounted on the HP4191A RF. Another end-port of the coaxial line was directed to the center of an unbiased current Helmholtz coil for a standard calibration. The standard procedure employing short-, open-, and 50 \( \Omega \) connectors were carefully carried out as follow:

(1) the short-circuit connector was first gently mounted on the end-port of a fixed coaxial line (~50 cm in length). Then, the “Start” button was selected to perform the frequency-sweeping calibration.

(2) we repeated the same procedure for the open-, and 50 \( \Omega \) connectors, respectively. After finishing the general standard calibration, the “Local” button was unselected in order to allow a personal computer to control the HP4191A RF through the general purpose interface bus (GPIB).

(3) it is worth mentioning that the different frequency ranges for the impedance measurement can be performed. However, the calibration procedure must be repeated for each new selected target-frequency before operating the machine.

3.4.3 Standard Material Testing

The Cu wire (AWG39) was cut and soldered to SMA ports using a heated soldering gun (heating temperature ~750 °F). The SMA ports of the sample were connected to the coaxial line and 50-ohms terminator, respectively. Then, a wide frequency sweeping for the impedance measurement was operated through a LabVIEW program. In order to validate our experimental data with the classical skin-depth theory [25], we constructed a MATLAB program to calculate
the impedance response of the measured $Z$ of the Cu wire ($d \sim 86 \, \mu m$ and 7 mm in length). The experimental data and calculations of $R$, $X$, and $Z$ are shown in Fig. 3.13. As can be seen, the experiment and theoretical prediction show an excellent agreement. In addition, the experimental data for $R$, $X$ and $Z$ as well as the effective circumferential permeability extracted from the Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{12.5}$Nb$_1$ microwire ($d \sim 50 \, \mu m$ and 7 mm in length) [26] show a good agreement with the reported result in Ref. 27 (see Fig. 3.14). Therefore, the prepared sample was ready for high-frequency magneto-impedance measurement.

![Fig. 3.13](image1.png)

**Fig. 3.13** (a) Experimental data and (b) calculated values of $R$, $X$, and $Z$ for a Cu wire.

![Fig. 3.14](image2.png)

**Fig. 3.14** (a) Experimental data of $R$, $X$, and $Z$ and (b) effective circumferential permeability extracted from the Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{12.5}$Nb$_1$ microwire.
In this Ph.D. dissertation, the complex impedance of the test samples was measured in the frequency range of 1 MHz to 1 GHz using the HP4191A RF impedance analyzer via a transmission line technique [28]. An axial magnetic field ($H_{dc}$) was applied to the samples in the ±115 Oe range using a Helmholtz coil. The dc current that was used to generate $H_{dc}$ was controlled by the lock-in amplifier and a bipolar power supply. The complex reflection coefficient at the beginning of the microwire airline was measured and converted to complex impedance ($Z$) as before. The experimental setup for the high-frequency impedance is shown in Fig. 3.15.

![Fig. 3.15 An experimental setup for a high-frequency magneto-impedance measurement.](image)

In order to interface all instrumentation units together, the test devices and magnetic field generator are controlled through a general purpose interface bus (GPIB), using LabVIEW data acquisition program. The resistance ($R$), reactance ($X$), and complex impedance ($Z$) were recorded using the LabVIEW data acquisition program. Furthermore, we define magneto-impedance (MI) magneto-resistance (MR), magneto-reactance (MX) as follows:
\[ MI = \frac{Z(H) - Z(H_{\text{max}})}{Z(H_{\text{max}})} \times 100 \%, \quad (3.7) \]
\[ MR = \frac{R(H) - R(H_{\text{max}})}{R(H_{\text{max}})} \times 100 \%, \quad (3.8) \]
\[ MX = \frac{X(H) - X(H_{\text{max}})}{X(H_{\text{max}})} \times 100 \%, \quad (3.9) \]

where \( Z(H) \) is a complex impedance at the field \( H \), and \( H_{\text{max}} \) represents the maximum induced magnetic field by the Helmholtz coil (\( H_{\text{max}} = 115 \text{ Oe} \)), respectively. The magnetic field sensitivity of GMI (\( \eta \)) is defined as

\[ \eta_z = \frac{d}{dH}(MI) \quad (3.10) \]
\[ \eta_R = \frac{d}{dH}(MR) \quad (3.11) \]
\[ \eta_X = \frac{d}{dH}(MX) \quad (3.12) \]

where \( \eta_z, \eta_R, \eta_X \) are the magnetic field sensitivity of the impedance, resistance, and reactance, respectively.

### 3.4 Summary

The experimental methods used in this dissertation have been presented. Melt-extraction technique, which has been employed for the Co-rich magnetic microwire fabrication, is demonstrated. The sample preparation, single- and multi-step current annealing treatments are explained. The structural and magnetic properties of the Co-rich microwires are characterized using XRD, SEM, EDS, HRTEM, MOKE, and MFM. The measurement of high-frequency magneto-impedance as well as standard calibration methods have been elaborated in this chapter.
3.5 References


CHAPTER FOUR

TAILORING HIGH-FREQUENCY GMI RESPONSE IN CO-RICH MELT-EXTRACTED MICROWIRES

Note to the reader.

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In this chapter, we present a systematic study of the high frequency GMI response in melt-extracted amorphous microwires using the Joule annealing technique. We have employed single- and muti-step current annealing to enhance low-field, high-frequency magneto-impedance (MI) response and surface magnetic domain structures (SMDS) in the melt-extracted amorphous microwires. We have explored structural and magnetic properties of the as-prepared and annealed magnetic microwires. The improvement of the high-frequency MI response in Co-rich amorphous microwires is promising in low field detection of sensitive magnetic microsensors.

4.1 Single Step Joule Annealing Effect

4.1.1. Introduction
Amorphous magnetic materials (“metallic glasses”) have been the subject of great attention in recent years due to their outstanding soft magnetic properties, i.e., low magnetostriction, high saturation magnetization, high permeability, and low coercivity, which are promising for sensing technology applications [1-3]. The discovery of a giant magneto-impedance (GMI) effect, which refers to a large change in the complex impedance of a ferromagnetic conductor in response to a dc magnetic field, in this type of material [4,5] has made it ideal for applications in weak field sensing and biodetection [2,6,7]. GMI relates to a skin depth effect in a magnetic medium resulting from an alternating current applied to a soft ferromagnetic conductor. The major contributors to the skin depth (δ) are ac frequency (f), electrical resistivity (ρ), and circumferential permeability (μθ), and they are related by $\delta = \sqrt{\rho / \pi \mu_0 f}$ [2,4,5]. The most tunable parameter is μθ, which is associated with domain-wall displacement and rotational magnetization.

In recent years, progress in fabrication and characterization of the amorphous magnetic materials such as wires [8], microwires [9-12], ribbons [13,14], and thin films [15] has been extensively studied. Various technologies of microwire fabrication, such as in-rotating-water quenching [10], glass-coating [11], and melt-extraction [12] have been employed to improve mechanical properties and GMI effect [16]. Among these techniques, melt-extraction provides great mechanical properties, sufficient length scales (30-60 μm in diameter, and typically several meters in length), tailoring ability, and pronounced GMI properties [17,18]. However, internal stresses are introduced during the rapid cooling process, which can deteriorate GMI response in amorphous microwires. Joule-heating is one of the most efficient techniques for improved GMI ratio and magnetic field sensitivity (η) for amorphous microwires. Under proper annealing conditions the evolution of amorphous microstructures, such as the release of residual stresses, can enhance circular magnetic domain structures resulting in an increased GMI effect [19-21].
It has been reported that the GMI ratio and η of Co-based amorphous microwires, parameters which determine the ability of a sensing application, has been experimentally achieved up to 300%, and 500%/Oe (at \( f \approx 10 \text{ MHz} \)), respectively [22,23]. The improvement of the GMI effect at low frequency (250 kHz) was also reported in Joule-heated Co-based amorphous wires using low dc current annealing in vacuum [24]. Zhukov et al. [25] tailored the GMI effect in amorphous Co-rich microwires by controlling the internal stress to improve GMI effect. As a result, the GMI ratio has been achieved up to 500% and pronounced its high field sensitivity into the gigahertz frequency range. Tiberto et al. [26] performed both Joule- heating and conventional furnace annealing studies on microwires and measured GMI at high frequency (45 MHz – 6 GHz). It was found that the Joule-heating technique improved the GMI ratio but the conventional technique did not. However, the authors note that crystallization in the amorphous microwires can deteriorate the GMI effect.

Alternative approaches such as cryogenic Joule [19], twin-zone [21], combined current-modulation [27], square-wave pulse current [28], and accumulated dc annealing [29] have been developed to successfully improve GMI response and field sensitivity of Co-based amorphous microwires. It has been shown that the GMI ratio of melt-extracted microwires increases up to 368.7% and achieved a field sensitivity up to 623.5%/Oe, which are desirable for high performance field-sensor applications [27]. However, these studies were limited in low and intermediate frequency regimes (less than 20 MHz) where maximal values of GMI ratio have not been observed.

As mentioned previously, tailoring the magnetic microwires under proper conditions shows the significant improvement of the GMI ratio. Although obtaining the higher ratio is an important parameter, the corresponding magnetic field sensitivity (η), which determines a sensor’s
qualities, must be scrutinized. The high frequency GMI response possesses two peaks due to the existence of the circular magnetic anisotropy in the microwires [30,31]. This two-peak feature, which noticeably has a maximum at low field region (on the order of a few Oersted). The field location of the peak is related to the anisotropy field ($H_k$). In order to retain both small $H_k$ and high magnetic permeability of the microwires, the peak-location and peak-depth of the GMI-response must be optimized. Of particular interest is the large depth of the GMI response near zero field, which has been extensively exploited for making highly sensitive GMI-based sensors for a weak field detection [32-36].

In this study, we enhanced the low-field GMI response in melt-extracted amorphous Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{13.5}$ microwires by means of Joule-heating. The trade-off between dc current amplitudes and annealing time was systematically optimized in order to refine the domain microstructures and circular magnetic anisotropy of the amorphous microwires over a high frequency range (20 MHz – 1 GHz) for the development of highly sensitive, high frequency GMI-based sensors.

4.1.2. Experimental

The amorphous magnetic microwires with a nominal composition of Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{13.5}$ (in wt%) were fabricated by a melt-extraction method described elsewhere [12,16]. The atomic weight percent of the elements present in the amorphous microwires was investigated by energy dispersive spectroscopy (EDS) using a JEOL JSM-6390LV SEM. The EDS spectra show 69.25 wt% of Co (used as a normalized element), 4.56 wt% of Fe (Sd. = 0.10), and 14.44 wt% of Si (Sd. = 1.10). Three sets of the as-cast microwires with diameter about 40 μm and 10 mm in length were cut and prepared from strands. The first set of the microwires was annealed with dc current
amplitudes of 5 mA, 10 mA, and 20 mA for 10 min to compare the effect of current amplitude on GMI response. Then, the second and third sets were subjected by the same current intensities for 20 and 30 min, respectively. The complex impedance was measured in the frequency range of 20 MHz to 1 GHz using the HP4191A RF impedance analyzer via a transmission line technique [37]. An axial magnetic field \((H_{dc})\) was applied to the samples in the range ±115 Oe by employing a Helmholtz coil. The resistance \((R)\), reactance \((X)\), and complex impedance \((Z)\) were recorded using a general purpose interface bus (GPIB) and LabVIEW data acquisition program through a computer. The figure of merit of the Co-rich microwire is determined by the GMI-ratio and magnetic field sensitivity \((\eta)\), which are previously defined in the chapter 3.

4.1.3. Results and Discussion

4.1.3.1 Material characterization. The amorphous nature of the microwires was characterized by an x-ray diffractometer (XRD) using a Bruker AXS D8 Focus Diffractometer. As seen from Fig.4.1 (a), the XRD pattern presents a broad hump feature at the diffracted angle of ~45 degrees, which represents short-range order of \(\alpha\)-CoFe-phase embedded in an amorphous matrix. The insets show high-resolution transmission electron microscopy (HRTEM) of the amorphous microwires (as-cast) and the corresponding electron diffraction pattern. The scanning electron microscopy (SEM) micrographs of the as-cast and annealed microwires (5 mA, 20 min) are shown in Fig.4.1 (b)-(c), respectively. The composed elements, as mentioned earlier, were confirmed using EDS as shown in Fig.4.1 (d).
Fig. 4.1 (a) The XRD pattern of amorphous Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{13.5}$ microwires for as-cast and annealed samples (5 mA, 20 min). Insets show the HRTEM image of the as-cast microwire and the corresponding electron diffraction pattern. (b) The SEM image of the as-cast microwire, its inset shows an enlarged image. (c) The SEM image of the annealed microwire (5 mA, 20 min). (d) EDS spectra of the as-cast microwire.

4.1.3.2 High-frequency GMI response. Figure 4.2 shows the field dependence of resistance ($R$), reactance ($X$), and impedance ($Z$) of the as-cast (black square) and Joule-annealed microwires (red circle) at 5 mA for 20 min for selected frequencies (20, 100, and 300 MHz). It can be observed in Fig. 4.2 (a)-(i) that $R$, $X$, and $Z$ apparently increase with increasing frequency and exhibit a two-peak feature. The two-peak behavior of a high frequency GMI effect ensures the circular magnetic anisotropy ($H_k$) in the amorphous microwires and can be described as
follows:[2,38] (i) at \( H_{dc} < H_k \), the ac magnetization mechanism of the outer shell circular domains depends on the superimposed field of \( H_{ac} \) (the induced ac field was induced by ac current), \( H_k \), and \( H_{dc} \). Owing to the inhomogeneous magnetic anisotropy in glassy microwires, the outer shell domains are not fully aligned in the circular direction and the domain configurations are tilted toward an arbitrary direction. With increasing \( H_{dc} \) in the axial direction, domain-wall movement and the rotational magnetization processes re-arrange domains due to a response from the superimposed field. Once the circular domains are oriented in their preferable positions, the ac magnetization process is easier, which makes the relative circumferential permeability increase. In fact, the relative circumferential permeability reaches its highest value at this point and pronounces the maximum \( Z \). This field point is defined as \( H_k \). (ii) at \( H_{dc} > H_k \), the GMI effect constantly decreases with increasing \( H_{dc} \), since the dominant field is in the axial direction. This reduces the ac circumferential permeability of the amorphous microwires. \( Z \) gradually decreases following a quasi-logarithmic behavior with \( H_{dc} \) increasing.

It is worth mentioning from Fig. 4.2 (a)-(c) that the as-cast microwire displays \( R \), \( X \) and \( Z \)-broadened peaks in comparison with the annealed sample (5 mA, 20 min). These sharper peaks obtained from the annealed sample at a small \( H_{dc} \) regime (~1-2 Oe) obviously promote the GMI ratios and \( \eta \), which is attractive for sensing technology applications. Nonetheless, they are much less pronounced at higher frequencies, as shown in Fig. 4.2 (d)-(i), considering the deterioration of the GMI effect in the high frequency regime. In addition, \( R \) apparently contributes more to \( Z \) than \( X \) does at 20 MHz, but both \( R \) and \( X \) significantly contribute to \( Z \) at 100 and 300 MHz.
Fig. 4.2 The field dependence of the resistance ($R$), reactance ($X$), and impedance ($Z$) of as-cast and annealed samples (5 mA, 20 min) at 20 MHz (a)-(c), 100 MHz (d)-(f) and 300 MHz (g)-(i).

In order to appreciate the enhanced GMI effect in the amorphous microwires annealed by Joule-heating, the field-dependent transport properties (MR, MX, and MI) have been studied in detail. Fig. 4.3 (a)-(i) shows the field dependence of the MR, MX, and MI of the as-cast (black square) and annealed samples (red circle) at 5 mA for 20 min. The greatest improvement in the GMI ratios was observed at 20 MHz. At this frequency in particular, MR has a larger contribution to MI than MX. It is well known that the low temperature annealing has the effect of the removal of local defects and stresses in the amorphous microwires. Considering that the MR is related to the rotational magnetization process, the remarkable change implies that reducing local defects (accompanied by a self-induced magnetic field) provided an enhanced rotational permeability in the circumferential direction. However, at 100 and 300 MHz the MI ratio (Fig. 4.3 (f), (i)) only slightly increases from the as-cast value. Unlike the enhancement in MX at 20 MHz, the ratio at higher frequencies does not improve. When the excitation frequency increases, the current gets distributed near the surface of the sample, where the circular magnetic anisotropy is prominent.
As a consequence, the rotational motion of magnetization plays an essential role in GMI response to the static magnetic field. Therefore, at rather high frequency regime, the low-current annealing has less pronounced GMI improvement because of the suppression of the domain-wall displacement and the skin effect [20].

**Fig. 4.3** The field dependence of the magneto-resistance (MR), magneto-reactance (MX), and magneto-impedance (MI) of as-cast and annealed samples (5 mA, 20 min) at 20 MHz (a)-(c), 100 MHz (d)-(f) and 300 MHz (g)-(i).

The frequency dependence of the maximum GMI ratio is shown in **Fig. 4.4 (a)** for the as-cast and Joule-annealed microwires (the microwires were annealed at different currents of 5 mA, 10 mA, and 20 mA for 20 min). **Fig. 4.4 (b)** shows various annealing times for 5 mA current. As seen from Fig. 4 (a) and (b), Joule-heating by the current amplitude of 5 mA for 20 min (red circle) markedly promotes GMI ratios up to 610% which is 1.6 times of 380 % for the as-cast one at \( f \approx 20 \text{ MHz} \). In addition, the sample annealed for 10 min at 20 mA (not shown) had a significantly improved GMI ratio about 460% at \( f \approx 20 \text{ MHz} \). Therefore, the low amplitude and self-induced field of the annealing current enhances the GMI ratio of the amorphous microwires.
Fig. 4.4 The frequency dependence of the maximum GMI ratio for the as-cast and annealed samples (a) with different amplitudes of current annealing; 5 mA, 10 mA, and 20 mA for 20 min, and (b) with different annealed times; 10 min, 20 min, and 30 min for 5 mA, respectively. Frequency dependence of (c) field sensitivity ($\eta$) and (d) anisotropy field ($H_k$) of as-cast and the annealed samples which improve GMI ratio. The inset plots shown in (c) are field dependent $\eta$ at 40 MHz. (d) The anisotropy field for the annealed microwires in comparison with the as-cast sample. The inset in (d) shows the low frequency portion of the $H_k$.

As mentioned previously, the sharpness of the dip in the MI (Fig. 4.3) at near-zero $H_{dc}$ is a crucial component to consider for a weak-field sensing applications. The $\eta$ and $H_k$ are shown in Fig. 4.4 (c) and (d), respectively. As seen from Fig. 4.4 (c), the maximum $\eta$ of the annealed
microwires rapidly increases up to 375 %/Oe (10 min, 20 mA) and 500 %/Oe (20 min, 5 mA), which are 1.7, and 2.2 times of 225%/Oe (as-cast state) at $f \approx 40$ MHz, respectively. The peak in the sensitivity at $f \approx 40$ MHz indicates the sensor applied region (SAR) for sensor applications should be between 20-60 MHz, which is desirable for high frequency magnetic sensors.

An additional remark about Fig. 4.4 (c) is that while $\eta$ of the microwire annealed at 5 mA for 20 min (red circle) improves over the entire measured frequency spectrum, the microwire annealed at 20 mA for 10 min only improves $\eta$ up to 350 MHz. The deteriorated GMI effect above 350 MHz for the latter case (20 mA, 10 min) relative to the as-cast microwire may be caused by the decreasing circumferential permeability owing to partial surface crystallization. This can be associated with the frequency dependence of $H_k$ of the annealed microwires as compared to the as-cast sample (see Fig. 4.4 (d)). For the optimum annealing condition (5 mA, 20 min), the $H_k$ is roughly comparable to that of the as-cast one, but with higher current annealing (20 mA, 10 min) the $H_k$ has noticeably increased. This shows that the magneto-crystalline anisotropy has increased comparing with the as-cast microwire, possibly due to the partial crystallization of Co-rich microwires leading to a decreased GMI effect [20,39,40]. It is worth mentioning here that relative to the as-cast microwire, the optimally annealed microwire (5 mA, 20 min) possesses a much larger peak-depth of the GMI-response (Fig. 4.3) while a comparable $H_k$ (Fig. 4.4 (d)), resulting in the much greater $\eta$ for this sample (Fig. 4.4 (c)). This result suggests that this Joule annealing did not significantly alter the amorphous state and the circumferential domain structure of the as-cast microwire but partially released residual stresses leading to the enhanced response of circumferential permeability to a dc magnetic field.
4.1.3.3 Surface magnetic domain structure (SMDS). To verify this hypothesis, the evolution of outer shell domain structure by low current heating process has been studied, and the MFM images are shown in Fig. 4.5. As one can see in this figure, both as-cast and annealed microwires (5 mA, 20 min) possess a typical circumferential domain structure. In case of the annealed microwire, however, the circumferential domain structure is more uniform resulting from the residual-stress relief. The similar domain patterns observed for both the as-cast and annealed samples are fully consistent with their above reported similar values of $H_k$ (Fig. 4.4 (d)). As a result, this domain adjustment plays a crucial role in improving GMI ratio and $\eta$. The amorphous microwire structures retain intrinsic soft ferromagnetic properties without any major modification to the domain configuration as a result of low current Joule heating [27,29]. Therefore, Joule-heating method is a suitable alternative technique to promote high-frequency GMI effect in the amorphous microwires for low field sensing applications.

Fig. 4.5 The MFM images showing surface circumferential domain structures of (a) as-cast and (b) annealed samples (5 mA, 20 min) for two and three dimensions, respectively.
4.2 Multi-step Joule Annealing Effect

4.2.1. Introduction

As shown in the previous section, Joule annealing Co-rich microwires as a post-processing treatment has revealed improvement of GMI properties \([41,42]\). In a simple Joule annealing scheme, the sudden application of dc current intensity of \(\sim 100 \text{ mA}\) for an appropriate time showed an overall improvement of the GMI response \([43]\). This is due partially to the relief of quenched-in stresses, structural relaxation, and microstructure evolution during Joule heating \([3]\). In addition, the circumferential magnetic domain structure was observed to be enhanced by the self-induced magnetic field of the annealing current \([19,27]\). Furthermore, the formation of nanocrystals in the amorphous matrix caused by the heat treatment increases the electrical conductivity \([43]\). The Joule heating technique has more advantages than conventional annealing methods and promotes the high frequency GMI effect in Co-rich microwires. However, an asymmetric peak in the GMI response, attributed to induced helical domains, was observed when the current intensity is greater than 100 mA. To combat this, a stepped approach to the highest current intensity has shown a GMI enhancement up to 360\% and reduced the undesired asymmetric GMI peak \([29]\). Furthermore, this multi-step Joule-heating markedly promote the field sensitivity for the weak field regime, \(H < 2 \text{ Oe}\). However, most results were explored for the excitation frequency below 20 MHz \([27,29,43]\). Despite recent investigations have shown great promotion in the GMI-ratio, a clear evident between GMI-promotion through multi-step current annealing and their surface properties for high frequency GMI-response is lacking.

In this experiment, the high frequency GMI response in melt-extracted amorphous Co\(_{69.25}\)Fe\(_{4.25}\)Si\(_{13}\)B\(_{12.5}\)Nb\(_1\) microwires over a high frequency range (1 MHz -1 GHz) is studied. The comparative study of single- and multi-step Joule-heating in the Co-rich microwires is performed.
Moreover, the structural characterization of the microwire is performed using x-ray diffraction (XRD), Scanning electron microscopy (SEM), and high-resolution transmission electron microscopy (HRTEM). The surface magnetization of the as-quenched and post-treatment microwires were investigated through magneto-optical Kerr effect (MOKE). We found that tailoring the GMI effect through the multi-step annealing technique enhanced GMI-ratio and field sensitivity to 750% and 820%/Oe, respectively. We demonstrate the multi-step current annealing successfully promote the low magnetic field sensitivity. The results are potentially applicable to the development of highly sensitive, high operating frequency GMI-based sensors.

4.2.2 Experimental

The high quality amorphous magnetic microwires with a nominal composition of Co69.25Fe4.25Si13B12.5 Nb1 (in wt%) were fabricated by a melt-extraction technique as previously described [18]. The microstructure of the as-prepared and annealed microwires was characterized by high-resolution transmission electron microscopy (HRTEM) (Tecnai G2F30). The surface morphology and the nominal elemental composition of the samples were investigated by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) (JEOL JSM-6390LV), respectively. Surface magnetic properties of the samples were characterized using Magneto-optical Kerr effect (MOKE) as previously described in our study [44]. The as-quenched microwires with diameter ~55 μm and 10 mm in length (l) were selected from a strand of Co69.25Fe4.25Si13B12.5 Nb1 microwire. Identical samples (d~55 μm, l ~10 mm), as determined by an initial magneto-impedance measurement, were chosen for the comparative post-heating treatments. The selected microwire was soldered to SMA ports with copper ground plane. The mounted microwires were subjected to a constant dc current of $i_{dc} = 100$ mA for $t = 10$ min. This annealing scheme is denoted as single-step annealing (SSA). For comparison, a multi-step annealing scheme (MSA) is applied
to the second microwire consisting of an initial current intensity $i_{dc} = 20$ mA and then increasing the current intensity by $i_{step} = 20$ mA every $t_{step} = 10$ min until $i_{dc} = 300$ mA. The schematic setup for the experiment is given previously in the section 3.2. The GMI response over the high frequency range ($f_{ac} = 1$ MHz-1 GHz) was measured using a HP4191A impedance analyzer through a transmission line [37]. Standard calibration procedures (short, open, and 50 $\Omega$) were performed before the measurement. A pair of Helmholtz coils was employed to generate the external field ($H_{dc}$) along the longitudinal direction of the microwires. All measurements were performed at room temperature. The figure of merit of the Co-rich microwire is determined by the GMI-ratio and magnetic field sensitivity ($\eta$), which are previously defined in the chapter 3.

4.2.3 Results and discussion

4.2.3.1 Structural characterization. The surface microstructure of a soft magnetic microwire plays an essential role in high frequency GMI properties due to the large skin effect [42,45]. To explore the surface morphology and microstructure, SEM micrograph, EDS spectra, XRD pattern, and HRTEM image of as-prepared Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{13.5}$Nb$_1$ microwires are shown in Fig. 4.6 (a)-(d), respectively. As illustrated, the high quality microwire has a smooth surface and cylindrical shape over most of the wire. The composed elements of the microwire are presented in the EDS spectra. The EDS spectra show 69.3 wt% of Co (used as a normalized element), 4.6 wt% of Fe (Sd. = 0.10), 14.4 wt% of Si (Sd. = 1.1), and 1.0 wt% of Nb (Sd. = 1.10). The XRD pattern (Fig. 4.6 (c)) has a broad peak feature which indicates $\alpha$-CoFe-phase embedded in an amorphous matrix with center near $2\theta = 45$ deg. The high-resolution TEM images exhibits the presence of nanocrystal-like phase embedded in the amorphous matrix. The corresponding
electron diffraction pattern (Fig. 4.6 (d) inset) exhibits a diffused broad ring indicating random distribution of short-range order in the as-prepared microwires.

Fig. 4.6 (a) The SEM micrograph, (b) EDS spectra, (c) XRD pattern, and (d) HRTEM image of as-prepared Co$_{69.25}$Fe$_{4.25}$Si$_{13}B_{13.5}$Nb$_{1}$ microwires. Insets in (d) shows the corresponding electron diffraction pattern corresponding to the HRTEM.

The surface morphology of the as-prepared wires at different stages of the annealing schemes is shown in Fig. 4.7 (a)-(d). A smooth surface is observed in the as-prepared, SSA@100mA, and MSA@100mA annealed samples. However, crystalline pieces (>100 nm) on the surface are clearly observed after the MSA@200mA (Fig.4.7 (d)). This is due to the
temperature experienced during current intensity exceeding the crystallization temperature of the Co-rich microwire, which is typically ~ 753 K for the Co-rich microwires [46, 47].

**Fig. 4.7** (a) The SEM micrograph of the as-prepared the Joule annealing microwires; (b) SSA@100mA, (c) MSA@100mA, and (d) MSA@200mA.

It is well known that applying small current ($i_{dc} < 100$ mA) through amorphous microwires does not change the composition of their composed elements [29, 43]. In contrast, subjecting a high current ($i_{dc} \sim 100-200$ mA), thereby heating the microwire to above the glass transition temperature ($T_g$), could introduce crystallization into the microwire [48]. Table 1 shows the EDS measurement from the as-prepared, SSA@100mA, and MSA@100mA microwire. As can be seen, the standard deviation of elemental composition is less than 1%. This conforms that the heat
treatment (SSA@100mA, and MSA@100mA) does not change the nominal composition at the sample surface. It is worth mentioning that the composed elements become significantly alter when the application of dc annealed current is high up to 200 mA.

**Table 1** the EDS qualitative data for the nominal elements in the Co-rich magnetic microwires.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Si_{13}</th>
<th>Fe_{4.25}</th>
<th>Co_{69.25}</th>
<th>Nb_{1}</th>
<th>B_{12.5}</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>14.42</td>
<td>4.41</td>
<td>69.25</td>
<td>1.21</td>
<td>10.7</td>
</tr>
<tr>
<td>SSA</td>
<td>15.05</td>
<td>4.47</td>
<td>69.25</td>
<td>1.31</td>
<td>9.92</td>
</tr>
<tr>
<td>MSA</td>
<td>15.19</td>
<td>4.49</td>
<td>69.25</td>
<td>1.32</td>
<td>9.75</td>
</tr>
<tr>
<td>Ave.</td>
<td>14.89</td>
<td>4.46</td>
<td>69.25</td>
<td>1.28</td>
<td>10.12</td>
</tr>
<tr>
<td>Sd.</td>
<td>0.41</td>
<td>0.04</td>
<td>0.00</td>
<td>0.06</td>
<td>0.51</td>
</tr>
</tbody>
</table>

The microstructure of the as-prepared (Fig. 4.8 (b)) and Joule heated microwires, investigated using HRTEM is presented in Fig. 4.8. The HRTEM image of the as-prepared wire, Fig. 4.8 (b), is typical for this material, which is characterized as containing randomly oriented “nanocrystallites” or crystal nuclei within an amorphous matrix. No long-range ordering of the nanocrystallites is observed in the as-prepared wire, which is confirmed by the main blurry halo of the SAED pattern (Fig. 4.8 (b) inset). Similar results have been observed in Co-rich microwires with different compositions as well [21,29,43,49]. After the SSA@100mA annealing protocol, the TEM image (Figure 4.8 (c)) shows a similar random distribution of nanocrystallites however the SAED shows some additional diffuse rings, indicating nucleation of crystallites at the surface due to the SSA@100mA procedure. However after the MSA@100mA treatment, the electron diffraction pattern of Fig. 4.8 (d) inset produced crystalline diffraction bright spots on top of the diffused broad ring pattern and in addition some outer rings in Fig. 4.8 (c) inset do not appear. Therefore, the MSA treatment produces a nanocrystalline-embedded matrix with greater degree of uniformity in phase and orientation of the formed nanocrystals [29, 43].
Fig. 4.8 The HRTEM images of (a) a prepared sample, (b) as-prepared, (c) SSA@100mA, (d) MSA@100mA, (e) MSA@140mA and (f) MSA@200mA microwires. Insets show the corresponding electron diffraction patterns (b-d) and selected area electron diffraction (SAED) (e-f) of the investigated samples.
The formation of significant nanocrystalline size is presented when the applied current intensity reaches $i_{dc} = 140$ mA. As can be seen in Fig. 4.8 (e), the TEM image shows large nanocrystalline size (> 5 nm) in the amorphous matrix. The electron diffraction displays high ordering of poly-nanocrystalline phases in the annealed sample (MSA@140mA). Thus, this annealing parameter can introduce large nanocrystalline size and increase the volume fraction of the nanocrystalline phases into the magnetic microwires. More importantly, the application of the current up to 140 mA has reached the crystallization temperature of the Co-rich microwire [18]. In addition, the clear crystalline phase is observed in the MSA@200mA (T~1000 K), which confirms the result obtained from the SEM. This introduced large crystalline phase in amorphous magnetic materials could have great impact on their magnetic and GMI properties [19].

4.2.3.2 Surface magnetic properties. Hysteresis loops obtained from MOKE is a powerful tool to characterize the surface magnetic properties of materials. To understand the surface magnetic properties of the Co-rich microwire, longitudinal MOKE was performed. In this measurement, the magnetic field ($H_{ex}$) was applied in axial direction, therefore, the M-H loop behavior represents the magnetization along the length of the wire [50-52]. Figure 4.9 (a) demonstrates the MOKE hysteresis loops obtained from the as-prepared microwire. The surface hysteresis loop show rectangular-like shape with coercivity of $H_c = 0.25$. The small $H_c$ suggests that the rapid change in the surface magnetization occurred by domain wall movement. A similar experimental result has been reported by Chizhik et al. [53]. The square M-H loop indicates the surface domain structures of the microwires are irregular oriented along the axial direction of the microwires [53]. As a result of the SSA@100mA protocol (Figure 4.9 (a)), the coercivity increased to $H_c = 0.75$ Oe of in the SSA@100mA microwire and it became harder to magnetize in the axial direction. The increase of coercivity may come from the induced magnetic nanocrystalline phases.
into the amorphous matrix. It is worth to mention that the effective anisotropy field \( (H_k) \) estimated by the saturation point in the hysteresis loop increases about two times that of the as-prepared microwires.

**Fig. 4.9** The measured Hysteresis loops using longitudinal MOKE for (a) the as-prepared, (b) SSA@100mA, (c) MSA@100mA, and (d) MSA@200mA microwires.

Furthermore, recent studies have shown that the application of dc currents to a magnetic microwire promotes the surface magnetic properties of the microwires [29,43,54]. As seen from **Fig. 4.9** (c), the dramatic change of the MOKE hysteresis loop was observed from the MSA@100mA sample. This hard axis loop of the surface magnetization suggests that the surface magnetic domain structures (SMDS) are mostly oriented in the circumferential direction.
The hard-axis surface magnetization process is associated with the rotation of surface magnetic domains into the axial field direction [50,51,56]. In this case, the coercivity is absent but larger $H_k$ is observed, which is due to the strong circumferential anisotropy induced by the MSA@100mA treatment. It is worth mentioning that, the application of high dc current exceeded $T_x$ to the Co-rich microwires will induce crystalline phase and deteriorate the circumferential magnetic domain structures. As a result, the magnetic hardening would be observed as shown in Fig. 4.9 (d).

4.2.3.3 High-frequency GMI effect. It is established that the GMI-ratio and its field sensitivity of magnetic microwires strongly depends on their excitation frequencies [2,42]. To compare the impacts of suddenly (SSA) and gradually (MSA) heating to the microwires, the enhanced GMI ratio and $\eta$ between the SSA and MSA are shown in Fig. 4.10. As can be seen, the MSA@100mA greatly promotes the GMI ratio and $\eta$ over the high frequency range as well as overcome the SSA@100mA does. In our previous studies, the single-step Joule annealing (SSA) by applying current 5 mA for 20 minutes promotes the GMI ratio and $\eta$ in Co-rich microwires to 610% and 500%/Oe (at $f_{ac}$~40 MHz ), respectively [54]. In this experiment the maximum GMI ratios were found at $f_{ac}$ = 20 MHz, which are 500% (as-prepared), 650% (SSA@100mA) and 760% (MSA@100mA). The maximum $\eta$ for the as-prepared microwire is 179.55 %/Oe for $f_{ac}$~80 MHz. The SSA@100mA and MSA@100mA samples reach their maximum $\eta$ at $f_{ac}$~20 MHz, which are 619 %/Oe and 925%/Oe, respectively. As mentioned earlier, the application of dc currents (80-100 mA) for a suitable time promotes the GMI-ratio and the filed sensitivity in the Co-rich microwires [29,43].
Fig. 4.10 The field dependence of MI response and its field sensitivity over wide frequency (1 MHz-1 GHz). (a), (d) as-prepared, (b), (e) SSA@100mA, (c),(f) MSA@100mA, respectively.

In appreciation of the low field enhancement of the GMI ratio, the sharpness of the dip in the GMI-response at near-zero field at 20 MHz for the as-prepared and annealed samples...
(SSA@100mA, MSA@100mA) are shown in Fig. 4.11 (a)-(b). As can be seen, the MSA@100mA shows significant improvement in the GMI ratio at 20 MHz. The maximum GMI ratio is 760%, which is 1.75 times of 500% (as-prepared) and 1.17 times of 650% (SSA@100mA), respectively. More importantly, there is a remarkable improvement of $\eta$ for the MSA@100mA to 925%/Oe, which is 17.92 times of 51.61%/Oe (as-prepared) and 1.49 times of 619%/Oe (SSA@100mA), respectively. A similar approach has been employed to improve the GMI-ratio in Co$_{68.15}$Fe$_{4.35}$Si$_{12.25}$B$_{13.75}$Nb$_1$Cu$_{0.5}$ and Co$_{68.15}$Fe$_{4.35}$Si$_{12.25}$B$_{12.75}$Zr$_3$ microwires [29,43]. However, the studies focused on $f_{ac}$ up to 20 MHz and the reported GMI ratio and $\eta$ are 364% ($\eta \sim 585.71$%/Oe) and 256% ($\eta \sim 372.57$%/Oe), respectively. Therefore, this multi-step current annealing method is more effective in promoting the high frequency GMI effect in Co-rich microwires than the single step current annealing technique.

![Fig. 4.11](a) The field dependence of the MI and (b) field sensitivity of as-prepared (black) and annealed samples; SSA@100mA (blue), MSA@100mA (red) at 20 MHz.

In order to correlate the GMI effect and the microstructure evolution owing to the multi-step Joule annealing, the maximum GMI ratio, $[\Delta Z/Z]_{max}$ and field sensitivity ($\eta$) of the annealed
microwires as a function of annealing current (I) are shown in Fig. 4.12 (a). As can be seen, the GMI-ratio gradually increases with increasing current I up to 80 mA. Then, the ratio drastically changes to the highest value (760%) at I ~100 mA. A similar development of the η is also observed. As mentioned previously, the annealing current below 80 mA affects the structural relaxation and reduces the structural stress from the as-prepared state. This structural relief may contribute to the increases of the effective permeability and electrical conductivity of the microwires [43]. Thus, the GMI-ratio is promoted. Furthermore, once the annealing current is close to 100 mA, the nanocrystalline becomes significant and rational to enhance the electrical and soft magnetic properties of the microwires, which is mentioned previously [29,49]. The circumferential self-induced magnetic field promotes well define SMDs [27,29]. Nonetheless, the thermal energy due to Joule heating promotes nano-crystalline in the amorphous matrix. Therefore, the magnetic permeability of the microwires becomes larger (softer) and the electrical conductivity becomes smaller in comparing to the as-quenched microwires. Therefore, the GMI-ratio and η simultaneously reach the maximum value at this point.

The decrease in GMI-ratio and η is observed when annealing current is higher than 100 mA. This is due to the nanocrystalline size and its volume fraction become large and deteriorate soft magnetic properties of the microwire [43,49]. In particular, the annealing current up to 140 mA introduced poly-nanocrystalline phase in to the microwire structure shown previously in the Fig. 4.8 (e). Furthermore, the large crystalline sizes are formed when the annealed current generating heat above the Tx (I ~200 mA) shown in Fig. 4.8 (f). The effective magnetic permeability of the microwires decreases due to the introduction of large magnetic crystalline anisotropy into the microwire [19,43]. Therefore, the GMI-ratio and η markedly deteriorate.
The effective magnetic anisotropy field \( (H_{k,\text{eff}}) \) estimated by GMI of the microwires as a function of annealing current is shown in Fig. 4.12 (a). The \( H_{k,\text{eff}} \) does not change until \( i_{dc} = 140 \) mA. The jump in \( H_k \) corresponds to the large volume fraction of nanocrystalline phase is introduced into the microwire as seen in the TEM image and electron diffraction. The \( H_k \) further increases up to 6 Oe when \( i_{dc} = 200 \) mA \( (T \sim 1000 \text{ K}) \). At this current intensity, the microwire’s temperature exceeds the glass transition temperature of a Co-rich microwire and it becomes primarily crystalline and the deterioration of GMI properties is clearly observed.

Fig. 4.12 (a) Annealed currents dependence of GMI-ratio (black square) and field sensitivity of as-cast and annealed samples; SSA@100 mA (green and blue squares), MSA@20-280 mA (red circle). (b) Annealed currents and anisotropy field \( (H_k) \) of the annealed samples at 20 MHz. (b) SSA@100mA, (c) MSA@100mA and (d) MSA@200mA.

4.3 Summary

We have systematically studied the high frequency GMI response in melt-extracted amorphous Co\(_{69.25}\)Fe\(_{4.25}\)Si\(_{13}\)B\(_{13.5}\) microwires using Joule-heating technique. We found that the application of a small dc current for a suitable time to the amorphous microwires promoted the GMI effect and magnetic field sensitivity. The optimum annealed parameters of 5 mA for 20 min
remarkably improved GMI ratio, and its field sensitivity up to 610% (1.7 time of as-cast state) at $f \approx 20$ MHz, and 500%/Oe (more than two times of the as-cast) at $f \approx 40$ MHz, respectively. Low current annealing relieved the residual stress generated during the fabrication process and enhanced circular outer shell domain structure without major modifications of the amorphous microstructures. This alternative tailoring method improves GMI effect for amorphous microwires and can be practically applied for GMI-based sensors in the high-frequency range.

The optimization of the high frequency GMI-effect in melt-extracted Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{12.5}$ Nb$_{1}$, through the single-step (SSA) and multi-step (MSA) Joule annealing techniques was systematically studied. Increasing the $i_{dc}$ from 20 mA to 100 mA for 10 minutes remarkably improved the GMI ratio and its field sensitivity up to 760% (1.75 time of that of the as-prepared), and 925%/Oe (more than 17.92 times of that of the as-cast) at $f \approx 20$ MHz, respectively. The employment of the MSA technique successfully enhances the microstructures and the surface magnetic domain structures (SMDS) of the Co-rich magnetic microwires. This tailoring method is suitable for improving the magnetic field GMI sensitivity at small magnetic fields. The high sensitivity response of GMI to a weak magnetic field is highly promising for biomedical sensing applications.
4.4 References


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CHAPTER FIVE

NOVEL HIGH-FREQUENCY MAGNETOIMPEDANCE SENSORS FOR INDUSTRIAL APPLICATIONS

Note to the reader.

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In this chapter, we present the design and fabrication of a new class of GMI-based sensors. We have employed the optimized microwire, which is described in the previous chapter, as a sensing element for a new sensor. The magneto-impedance response of the sensor is characterized over a wide high frequency range (1MHz-1GHz). A comparison between the field sensitivity of the GMI-based sensor and a commercially available product is demonstrated. The practical utility of the linear response and high field sensitivity for the real-time monitoring of position and gear rotation are described. Reliable and accurate measurements of position and speed of a moving object by the sensor are performed. This GMI-based sensor is highly promising for a real-time position detection; and monitoring of oscillatory motion, and gear rotation.

5.1 High-frequency GMI Sensor for Real-time Position Detection and Oscillation Monitoring

5.1.1. Introduction
Real-time position and speed monitoring of a non-stationary object finds wide ranging applications in robotics, industrial manufacturing and processing, collision prevention assistance, and autonomous vehicles, etc. [1-5]. In particular, the real-time monitoring of a moving object is crucial for a feedback loop process and safety compliance [2,5]. Magnetic sensors play an essential role in these technologies and also have superior advantages to other types of sensors [6-9]. For instance, they provide precise, contactless measurements and are able to operate in dirty, high temperature, and/or non-transparent environments. A variety of magnetic sensors, such as those based on magnetoresistance (MR) [10], the Hall effect [11], induction [12], and superconducting quantum interference device (SQUID) [7] have been developed for magnetic field detection. Among them, sensors based on the Hall effect [5], giant magnetoresistance (GMR) [8], and inductive proximity [1] effects have been extensively used for position and speed detection owing to their robustness and cost effectiveness [1,2,6,13]. However, the signals become diminished and the noise disturbance increases when these sensors are located at far-off distances from a weak field source [6,14]. Therefore, there is a pressing need for developing new magnetic sensors that can sense weak fields from far working distances.

In recent decades, the giant magneto-impedance (GMI) effect in soft ferromagnetic microwires has been extensively studied to promote the GMI response at high working frequency [15-18]. The GMI effect in soft ferromagnetic microwires refers to a large change in the complex impedance when the wires are subjected to an external magnetic field along their axis [19,20]. Recently, a large and pronounced GMI response and field sensitivity in Co-rich microwires at RF excitation frequencies have been developed through a Joule heating technique [15,21,22]. When the exciting frequency increases, the ac excitation field tends to concentrate near the surface of the microwire due to the skin effect [23]. As a result, the circumferential magnetic anisotropy
attributed to the outer shell domain structure becomes significant and a double peak feature of the complex impedance is observed [17,24]. With the Joule annealing treatment, the magnetic microwires possess an ultra-high sensitivity to small magnetic fields (below the anisotropy field, \( H_k \), of the microwire), which is highly promising for weak magnetic field sensing at room temperature. In addition, the excellent mechanical properties and cost effectiveness of this metallic glass microwire make them attractive for the industrial applications [25,26]. Therefore, a GMI-based sensor employing a Co-rich microwire is a suitable candidate for active position and speed detection from a far-off distance [27,28].

In this study, a contactless GMI-based sensor is constructed with a Joule-annealed Co-rich microwire. The high frequency magneto-impedance response of the GMI-based sensor is characterized. The potential sensor’s sensitivity, stability and reliability are shown. A comparison between the field sensitivity of the GMI-based sensor and a commercial Gaussmeter is performed. Then, the GMI-based sensor is employed for real-time position and oscillatory motion monitoring from a test source. A thorough discussion on existing sensing technologies and the promise of GMI-sensor for an active position and speed detection is provided.

5.1.2. Experimental

5.1.2.1 Optimization of melt-extracted microwires. Co-rich magnetic microwires with a nominal composition \( \text{Co}_{69.25}\text{Fe}_{4.25}\text{Si}_{13}\text{B}_{12.5}\text{Nb}_1 \) were fabricated by a melt-extraction technique described elsewhere [25]. The obtained magnetic microwires are typically 30-60 microns in diameter and 10-50 cm in length. After rapid quenching, the microwires have a cylindrical shape and possess excellent mechanical properties. The surface morphology and the nominal elemental composition were investigated using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS), respectively. The EDS spectra shows 69.3 wt% of Co (used as the normalized
element), 4.6 wt% of Fe (sd = 0.2 wt%), 14.4 wt% of Si (sd = 0.4 wt%), and 1.0 wt% of Nb (sd = 0.1 wt%). The amorphous nature of the as quenched microwires was characterized by high-resolution transmission electron microscopy (HRTEM) and an x-ray diffractometer (XRD) previously described in Ref. [22,25], respectively.

In this experiment, an as quenched microwire with 50 µm diameter and 7 mm in length was selected and cut from a long microwire strand. The sample was then soldered to SMA ports, which are amounted to a micro-strip Cu ground plane (see Fig. 5.1). The multi-step Joule heating procedure used to tailor the magnetic and mechanical properties of the microwire is given here: the mounted sample was subjected to increasing current intensity from 20 mA to 100 mA in steps of 20 mA. During each step, the microwire is subjected to a constant current for 10 min and then stopped for 10 min to reach ambient temperature. This multi-step current annealing process has been shown to optimize the GMI effect in melt-extracted microwires in previous studies [21,25].

5.1.2.2 High frequency impedance spectroscopy. The impedance spectrum of the annealed microwire was measured over the frequency range (1 MHz - 1 GHz) using an Agilent 4191A RF-impedance analyzer through transmission line methods [29]. In this measurement, the standard calibration using short-, open-circuits, and 50-ohm standard was performed, respectively. A fixed 50 cm coaxial cable and the 50-ohm terminator were employed to facilitate and match the input impedance of the analyzer. The 4191A determines the complex reflection coefficient (Γ) of a measurement frequency test signal applied to the terminated transmission line. The figure of merit of the Co-rich sensing element is determined by the GMI-ratio [30] mentioned previously in the Chapter 3.
5.1.2.3 Measurement of position and oscillatory motion in real-time. To explore the field sensitivity of the GMI-based sensor, a cylindrical 8 mm wide x 4 mm thick Neodymium magnet was attached to a small homemade crane as seen in Fig. 5.1. The magnet is positioned such that the stray magnetic field from the face of the magnet is parallel to the wire axis to induce a longitudinal GMI response. Then, a stepper motor, which is controlled by an Arduino UNO board, moves the magnet collinearly to the GMI sensor. The longitudinal GMI response and its corresponding distance \((d)\) were measured with an impedance analyzer. In addition, the magneto-impedance was measured with the stray magnetic field from the magnet perpendicular to the microwire axis. Finally, a commercial Gaussmeter (Lakeshore model 410) was used to measure the stray magnetic field at the same distance, \(d\), from the magnet in order to compare the magnetic field sensitivity to the GMI sensor. The field source varied from 0.2 - 0.6 Oe at \(d = 14\) cm. The impedance response to the object positions for selected frequencies (100, 200, 400, and 600 MHz) was measured.

To demonstrate the real-time position monitoring of the GMI-based sensor, the test magnet was set up at \(d \sim 20.0\) cm above the sensor. Then, the magnet was stepwise moved downward 2.0 cm every 30 s until the magnet reached \(d \sim 12.0\) cm. In order to explore the stability of the sensor, the impedance response and the test position was continuously measured. In a second experiment, the cylindrical magnet was moved down toward the GMI sensor at various speeds, \(v_1 = 1.76\) cm/s and \(v_2 = 0.95\) cm/s, respectively. Then, the amplitude of the oscillatory motion was varied by the stepper motor at amplitudes 24.0, 22.5, and 21.5 cm. Finally, the stepper motor was replaced by a vibrator at \(d \sim 10.0\) cm. The magnet was oscillated with sinusoidal, square, and triangular patterns, at amplitudes of frequencies of 0.2, 0.1, 0.05 Hz, respectively.
5.1.3. Results and Discussion

5.1.3.1 High-frequency impedance spectrum of the GMI-based sensor. The high frequency GMI response in Co-rich microwires has two peaks at $H_{dc} = \pm H_k$, on either side of $H = 0$ Oe, and typically possesses a high magnetic field sensitivity at magnetic fields below $H_k$ [26,27]. The magneto-impedance effect significantly depends on its excitation frequency as shown in Fig. 5.2 (a). With increasing excitation frequency, the impedance increases due to a strong skin effect [23]. In the low field region ($H_{dc} \leq H_k$), the GMI response shows a gradual increase with the increase of the excitation frequency until $f_{ac} \sim 400$ MHz. Then, the GMI response decreases for higher frequencies. This finding indicates that there is a large modification of the skin depth and ac magnetic permeability in the microwire [31] when $f_{ac} \sim 400$ MHz. The $H_k$ values of the
optimized wire over the frequency range measured are shown Fig. 5.2 (b). In order to achieve high sensitivity, the operating frequency of the GMI sensor should be selected so that $H_k$ is small. As can be seen in Fig. 5.2 (b)-inset, the low field GMI response shows a large change in the impedance resulting from the external magnetic field for $f_{ac} \sim 400$ MHz. Consequently, the optimal magnetic field sensitivity occurs when the $f_{ac} \sim 300-400$ MHz. Therefore, the GMI-microwire based sensor should be operated at this frequency range in order to attain an optimal magnetic field sensing ability.

![Fig. 5.2](image)

**Fig. 5.2** (a) The field dependent response of magneto-impedance and (b) effective anisotropy field ($H_k$) of the Co-rich microwire over wide frequency range (1 MHz- 1 GHz), respectively. The inset shows the GMI-ratio for $f_{ac} \sim 400$ MHz.

**5.1.3.2 Detection regime and sensor sensitivity.** Fig. 5.3 (a) shows the impedance change in the GMI based sensor as a function of operating frequency and distance, $d$. As can be seen from Fig. 5.3 (a), the impedance change, $\Delta Z$, over a wide frequency range increases with decreasing a distance. Not surprisingly, the maximum change in the impedance occurs at distance $d \sim 4.5$ cm,
which indicates that the external field ($H_{dc}$) strength reaches the $H_k = 4.2$ Oe value at this distance. Then, a decrease in the impedance is observed after the magnet crosses this point.

A comparison of the magnetic field read by the commercial Gaussmeter and change in impedance from the GMI-based sensor as a function of magnet distance $d$ for $f_{ac} = 400$ MHz is shown in Fig. 5.3 (b). The Gaussmeter was set to DC mode. The minimum measured stray field from the test magnet by the commercial Gaussmeter was found to be 0.2 Oe at a distance of $d \sim 14.0$ cm. In contrast, a significant change in the impedance of the microwire is observed due to the same test magnet at twice the distance, $d \sim 28.0$ cm. It can be seen from Fig. 5.3 (b) that at $d \sim 28.0$ cm, the stray magnetic from the test magnet cannot be measured by the commercial Gaussmeter. This is due to the fact that the magnetic field sensing technology of the commercial Gaussmeter is based on the Hall effect, with the smallest field detection typically in the few micro-Tesla, or 0.1 Oe, range [11,14]. Fig. 5.3 (b)-inset shows an enlargement of the sensor responses for the Gaussmeter (red-sphere), transverse (green-sphere) GMI, and longitudinal (blue-sphere) GMI sensors, respectively. It is noticeable that the longitudinal GMI effect is more suitable for the sensing applications because of its greater field response than the transverse effect [32,33]. Furthermore, the change in impedance measured in the longitudinal geometry is $\sim 12.15$ $\Omega$ greater than the transverse geometry at a farther distance shown in Fig. 5.3 (b). The larger impedance change is due to the high field sensitivity of the longitudinal GMI effect due to the circumferential magnetic anisotropy of the outer shell domain structure. The angular dependence of the GMI of a Co-rich wire in magnetic field has been reported in Ref. [34]. The field-dependent GMI response showed broadened peaks as the wire orientation angle changed from longitudinal (parallel to the field) to transverse (perpendicular to the field). A broad and flat transverse GMI response implies
low magnetic field sensitivity; therefore, the longitudinal GMI response is utilized for all further experiments.

It should be mentioned that Fig. 5.3 (b) shows a non-monotonous variation in the impedance with magnetic field strength/distance was observed for the transverse and longitudinal GMI responses. While in general a linear sensor response is favorable due to simplicity of implementation, it is possible to create a look-up table with a calibration curve in order to utilize the non-linear output.

![Graph](image)

**Fig. 5.3** (a) The position dependent response of the impedance change for selected frequency range. (b) The comparison between the GMI-based sensor (transverse, green-sphere and longitudinal, blue-sphere) and a commercially available Gaussmeter for $f_{ac} \sim 400$ MHz. The inset shows the enlargement of the small portion of the sensor response.

Another crucial characteristic for any sensor operation is large frequency sensitivity. **Fig. 5.4** (a-d) shows a large signal increase when the GMI based sensor experiences higher applied magnetic fields. In this measurement, the several test cylindrical magnets were added to increase the field strength of the test field source; using up to five magnets. The magnitude of the stray field
for the additional magnets are 0.2, 0.3, 0.4, 0.5, and 0.6 Oe, respectively, as measured at the sensor position from a distance \(d\sim14.0\) cm away. It can be observed from Fig. 5.4 (a-d) that the impedance change can be enhanced by tuning the field strength of the source. This finding suggests that the sensing distance for the GMI-based sensor can be extended. In comparison, the working distance for current technologies such as GMR and variable reluctance is quite limited. For example, in Ref. [8], the amplitude of the measured GMR signal markedly decreases when the sensing distance reaches \(d \sim 4\) cm or \(H_{dc} \sim 10\) Oe. In the GMI-sensor proposed in this work, the GMI signal decreases at \(d \sim 12\) cm or \(H_{dc} \sim 0.2\) Oe. Since the mentioned GMR effect in this case is at most \(\Delta R/R \sim 5\%\) at \(d \sim 1\) cm, there is a limitation for applying this technology for weak-field detection. In the GMI-based sensor studied here, \(\Delta Z \sim 55\) \(\Omega\) at \(d = 4\) cm \((H_{dc} = 6.2\) Oe\). Therefore, having a larger sensing distance makes the GMI-based sensor more suitable for long-distance, real-time position monitoring than the GMR-based sensor.

5.1.3.3. Sensing stability, reliability, and accuracy. The sensor stability and reliability of the optimized GMI-based sensor was performed. The test magnet was located at distance \(d \sim 20\) cm above the sensor. Then, it was moved downward 2 cm every 30 s until the distance \(d\) reached 12 cm. Fig. 5.5 displays the change in the impedance due to the various test magnet positions and is consistent and reliable for each step. Once the test magnet moves closer to the sensor, the impedance response becomes larger. This is due to the increase of the stray field magnitude experienced by the microwire from the test magnet. Fig. 5.5-inset shows the plots of real-time position monitoring of the magnet and its corresponding impedance alteration in the GMI-based sensor. It is worth mentioning that this nonlinear sensor response can be extracted by using spline interpolation in Matlab [35]. In this experiment, the measured impedance and position shown in Fig. 5.3 (b) were used as known data points to predict new data points using interpolation. After
interpolation of the data in Matlab, the position and speed of a moving object can be accurately monitored through the GMI sensor. As mentioned earlier, the state-of-the-art position sensor based on MI effect was previously reported [27,28], however, this new finding is to focus on the utility of the highly sensitive, low magnetic field detection for cost effectiveness and sensor miniaturization.

**Fig. 5.4** The position dependent response of the enhanced impedance response for selected frequencies of (a) 100, (b) 200, (c) 400 and (c) 600 MHz, respectively.
5.1.3.4 Real-time position and oscillatory motion monitoring. The real-time position monitoring of a moving object (a cylindrical magnet) with different speeds was carried out. As can be seen in Fig. 5.6 (a), the impedance changes attributed to the stray field of the object with moving speeds, $v_1 = 1.76$ cm/s and $v_2 = 0.95$ cm/s, are consistent for three cycles. The extracted object positions were retrieved from the measured impedance as shown in Fig. 5.6 (b). As can be seen in the position graphs (red triangle), the object position shows a linear change, which is consistent with the driven speed from the stepper-motor. This finding can be applied for precise position and speed detection of a moving object at excitation frequencies in the 100s of MHz. The corresponding change in impedance, $\Delta Z/Z$, at $d \sim 20$ is 50%, which is greater than typical GMR.
based sensors ($\Delta R/R \sim 5\%$) [8]. The employment of a similar technology based on the GMI effect at $f_{ac} < 3$ kHz to control an autonomous car was demonstrated by Aichi Steel Corporation [36].

Fig. 5.6. (a) The real-time position monitoring of a cylindrical magnet with different speeds $v_1 = 1.76$ cm/s and $v_2 = 0.95$ cm/s, respectively. (b) The extracted position of the magnet in (a) using measured impedance. (c) and (d) The measured impedance of the oscillatory motion amplitudes and the small vibrations, respectively.

The oscillatory motion and small vibration of the target magnet were captured by the GMI-based sensor in Fig. 5.6 (c) and (d), respectively. The oscillatory amplitudes of the driven magnet are 24.0, 22.5, and 21.5 cm, respectively. As can be seen in the Fig. 5.6 (c), a reliable pattern and accurate period of the oscillatory motion were observed. The period for three oscillations is
consistent over the observation period. This result is highly promising for an oscillation or vibration system monitoring that is essential in industrial machinery. For example, if a machine keeps repeating the same process or pattern to manufacture goods or products, machine conditions can be observed and controlled by detecting fault states through the GMI sensor. Furthermore, the small vibrations of the magnet were observed in Fig. 5.6 (d). Different wave patterns (sine, square, and triangle) and frequencies (0.2, 0.1, and 0.05 Hz) of small vibration amplitudes (2.0, 1.1, 0.8 and 0.6 mm) were monitored via the GMI sensor. The precision position detection demonstrated here can be applied to noise pattern discrimination or small vibration monitoring. For instance, parking facility vibrations cause by vehicles have been observed through an integrated tank circuit and solenoid with ferromagnetic core [37]. The small detected position change ~ 83.0-83.7 mm is comparable to the present GMI-based sensor. Therefore, the GMI-based sensor is suitable for real-time machine diagnostics, the prevention of system failure, and small vibration detections.

5.2 Real-time Monitoring and Failure Prediction of Gear Rotation with a GMI Sensor

5.2.1 Introduction

Real-time condition monitoring of machinery in non-stationary operations is essential to industrial applications such as manufacturing quality control and automotive safety [1,4,13,38,39]. In particular, rotational speed monitoring and rotor position detection of a rotating gear are industrial applications that require high precision. Currently, optical reflection and magnetic field variation are the two key methods used to capture the motion of moving parts in order to actively monitor high speed machinery. So far, several technologies have been developed to achieve this, for example, the Hall-effect sensor [11], giant magneto-resistance (GMR) sensor [40,41], inductive proximity sensors [42,43], and optical photoelectric sensors [44].
The photoelectric sensor, which operates based on an electrical signal resulting from the received light intensity after reflection, has been implemented in existing systems \[38,44\]. However, potential blockages in the optical path, such as dust particles or oil-drops, greatly decrease the optical intensity and the signal quality \[38,40\]. In contrast, the magnetic-based sensor, which operates by sensing a magnetic field that varies in strength with distance from the sensor, is not hindered by dust or non-magnetic materials. Therefore, a giant magneto-impedance (GMI) based sensor \[27,45-47\], which is capable of detecting weak magnetic fields, is an ideal candidate for industrial applications that focus on rotational gear speed detection. For example, a magnetically labeled gear can produce a changing magnetic field or a magnetic flux when in operation and this can be detected by taking advantage of the field sensitivity of the GMI effect. Consequently, the gear speed and tilt angle can be observed through the generated electrical signal. This inexpensive technology is more robust and cost effective for industrial application when compared to the photoelectric sensors \[27,38,40,47\].

The GMI-based sensor has the benefit of ultra-sensitivity to small magnetic fields, capable of achieving high magnetic field sensitivity comparable to that of a superconducting quantum interference device (SQUID) at room temperature \[15,47\]. The highly sensitive GMI effect refers to a large change in the impedance of a high permeability conductor upon application of a small external magnetic field \[19,20,47\]. At operating frequencies in the MHz range, the penetration or skin depth of the ac current has a strong and sensitive dependence on an externally applied field \(H_{dc}\) due to the increase in ac permeability during dc magnetization reorientation. This aspect comes from the high circumferential permeability \(\mu_\phi\) of these near zero magnetostrictive Co-rich microwires \[17,20,48-50\]. The penetration depth \(\delta\), electrical resistivity \(\rho\), circumferential permeability \(\mu_\phi\), and excitation frequency \(f\) are related by \(\delta = \sqrt{\frac{\rho}{\pi \mu_\phi f}}\) \[23, 48\]. The figure
of merit of the Co-rich microwire is determined by the GMI-ratio and magnetic field sensitivity ($\eta$), which are previously defined in the chapter 3.

In this study, we propose using the low-field region of the high frequency GMI response in a Co-rich microwire for sensitive magnetic field detection in safety control systems and industrial applications. A rotating gear was constructed and a small magnetic label was attached to the gear. The impedance variation due to the rotating external magnetic field from the gear was measured. The gear was tilted off-axis during operation to test the ability of the GMI sensor to detect the change. This is the first study that demonstrates the excellent capacity of an optimized low-field high frequency GMI sensor for real-time condition monitoring and predictive failure of a rotating object.

### 5.2.2 Experimental

The sensing element of the GMI sensor was made with a high quality melt-extracted Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{2.5}$Nb$_{1}$ microwire, which is ~ 50 microns in diameter and 7 mm in length. The surface morphology, and surface magnetic domain structures were investigated by scanning electron microscopy (SEM), and magnetic force microscopy (MFM), respectively. The magnetic field dependence of the GMI-effect was optimized by annealing through low intensity Joule-heating technique [15,26] under the multi-step current annealing (20 mA up to 100 mA, with increasing 20 mA for each 10 min). Then, the microwire was mounted to an SMA-port attached to a ground plane holder for a high-frequency impedance measurement using HP Agilent 4191A impedance analyzer via a transmission line technique [29,51]. The external field ($H_{dc}$) in the range ±114 Oe was generated by a pair of Helmholtz coils and applied along the longitudinal direction of the microwire.
To demonstrate the ability of the GMI sensor to detect a fast moving gear, a small cylindrical 8 mm x 4 mm Neodymium magnet was attached to a homemade gear as seen in Fig. 5.7 (a). Consequently, the optimized field sensitivity was determined to be ~14.0 cm (0.25 Oe) away from the sensing element. Then, the nonmagnetic circular gear which is 6.00 cm in diameter was implemented at the optimized distance. The cylindrical magnet was attached to the edge of the circular gear which produced a small magnetic field ~0.25 Oe towards the magnetic sensing element. The gear speeds were controlled using a stepper motor controller and an Arduino UNO board current driver. The experimental set up, sensing element, and the SMDs are shown in Fig. 5.7 (a)-(c), respectively.

**Fig. 5.7** (a) The schematic of experimental setup, (b) sensing element, (c) and the SEM image and its selected area showing SMDs.
5.2.3 Results and Discussion

5.2.3.1 High-frequency GMI response. The field dependence of the GMI response over the frequency range 100-1000 MHz is shown in Fig. 5.8 (a). The largest GMI change relative to zero applied magnetic field \( H_{\text{ref}} \) for this magnetic microwire is about 300\% (red area in Fig. 5.8 (a)) and occurs at excitation frequencies in the range of \( f_{\text{ac}} \sim 200-400 \) MHz. In Fig. 5.8 (b), the GMI response of the microwire sensor at 400 MHz is presented. At an excitation frequency of 400 MHz, the well-known double peak characteristic of high frequency GMI can be seen. The corresponding field location of the peak, called the \( H_k \) field or anisotropy field, is at \( \sim 4.5 \text{ Oe} \) and shows a linear response to fields less than \( H_k \). Due to the relatively small \( H_k \) field value, the double peak feature of the MI response in the microwire is attributed to the switching field of the microwire core \([17,52]\). Fig. 5.8 (c) shows remarkably \( \eta \) high up to 230\%/Oe at the low field region, which is highly promising for small varying magnetic field detection \([7,32,53]\). The ac resistance from the impedance measurement was used to estimate the skin depth, \( \delta \), and effective permeability, \( \mu_\phi \), using the approach from Ref. 33. The estimated \( \delta \) in Fig. 5.8 (d) (red, left axis) shows that the majority of the current is distributed in the top 1-2 \( \mu \text{m} \) of the microwire. The corresponding permeability variation is less than one order of magnitude over the magnetic fields measured. Typically, the most sensitive region occurs just below \( H_k \) and its value can be tuned by the strength of a field source or selecting a different operation frequency.

5.2.3.2 Real-time speed monitoring of a gear rotation. Fig. 5.9 (a) shows the time dependent impedance change during rotation of the sample gear driven at angular speeds of \( \omega_1 = 0.39 \text{ rad/s} \), \( \omega_2 = 0.26 \text{ rad/s} \), and \( \omega_3 = 0.17 \text{ rad/s} \). Fig. 5.9 (b) shows the period of rotation extracted from the obtained signals, which are 16.26, 24.39, and 37.25 sec., respectively. Since period (T) and angular frequency (\( \omega \)) are related by \( \omega = \frac{2\pi}{T} \); therefore, the corresponding angular
speeds can be determined as 0.39, 0.26, and 0.175 rad/s, respectively. These retrieved speeds are perfectly matched with the driven angular speeds controlled by the stepper motor controller. Similar current technologies, for example, GMR and Hall sensors have been used to measure ferromagnetic wheel speed. However, the measured signals significantly decrease when the sensing distance is larger than 4 cm ($H_{\text{ext}} \sim 10 \text{ Oe}$) [8]. In contrast, the GMI sensor shown an excellent response for the sensing distance up to 12.0 cm ($H_{\text{ext}} \sim 2.5 \text{ Oe}$); therefore, the GMI based sensor is more suitable for the weak-field detection than the GMR and Hall sensors.

Fig. 5.8 The field dependent of magneto-impedance response at high frequency (100 MHz-1 GHz) (a), at selected exciting frequency ~400 MHz, its inset shows an enlarged area of the large change in GMI ratio (b). High sensitive at the low-field region, its inset shows an enlarged area and the operating point $H_{\text{dc}} \sim 0.25 \text{ Oe}$ (c). Estimated skin-depth and magnetic permeability from the experimental data (d).
The virtual simulation of unknown rotating speeds was further explored as seen in Fig. 5.9 (c). The retrieved angular speeds are 0.08, 0.10, and 0.15 rad/s, which are perfectly agreed with the trend of previous known-speed measurement (see Fig. 5.9 (d)). Due to the high accuracy and reproducible measurement, the optimized GMI sensor is highly promising for rotation speed sensing at room temperature.

**Fig. 5.9** Time-dependence of the impedance response of a rotating gear with different speeds (a), measured periods of the gear rotation from the real-time impedance monitoring (b), Unknown angular speeds and their measured periods (c), and plots of the known and unknown retrieval vs angular speeds (d).

### 5.2.3.3 Real-time detecting of a fault rotating gear.
To assess the ability of the sensor to detect a faulty gear, the MI was measured rotating gear was tilted off axis by angles ranging between 1.0 and 7.0 deg. as seen in Fig. 5.10 (a). The maximum impedance gradually decreases
as a function of the tilt angle resulting from the direction of the stray field turning away from the longitudinal direction (wire axis) [34]. This nonlinear relation between the tilted angles and the impedance changes can be used to determine an unknown-tilted angle of the rotating gear. In this procedure, the known tilt angles and impedance changes were employed as known data points to construct new data points by the means of interpolation in Matlab. The unknown-tilted angle retrieval (blue) of rotating gears Fig. 5.10 (b) shows excellent agreement with the measured impedance changes as a function of known (red) angles. In fact this study has been focused on an earlier anomalous rotating gear for the predictive maintenance and prior safety. Therefore, having detected small tilted angle or not being observed by naked eyes for the anomalous rotating gear will be advantages for the real-time monitoring and predictive maintenance. For instance, this sensor technology can be employed for real-time monitoring of anomalous vibrational behaviors of a steam turbine driving a centrifugal compressor.

Fig. 5.10 (a) The time-dependence of the impedance response of normal, and tilted rotating gears, (b) measured impedance changes as a function of known (red) and unknown-tilted angle retrieval (blue) of the rotating gears.
Using the field-dependent GMI curve (Fig. 5.8 (b)), the small magnetic field change due to the tilted gear can be determined. By fitting the $Z(H)$ curve in the low field region, as seen in Fig. 5.11 (a), the small change in stray magnetic field ($\Delta H_{dc}$) due to the off-axis tilted gear can be determined. The results show that the difference in field experienced by the sensing element due to tilt angles of 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 7.0 deg were 0.4, 1.2, 1.9, 3.4, 5.2, and 7.7 mOe, respectively. The reciprocal value of the slope in Fig. 5.11 (b) represents the operating field sensitivity ($\eta$) $\approx$ 86 %/Oe, which shows good agreement with the selected operating point shown in Fig. 5.8 (c).

![Fig. 5.11](image)

**Fig. 5.11 (a)** The reversed plot of measured $Z$ and $H_{dc}$ from the GMI curve at a low field region, Its inset shows an enlarged area of the small $H_{dc}$, (b) measured $\Delta Z_o$ and the predicted $\Delta H_{dc}$ owing to the tilted gear.

### 5.3 Summary

We have fabricated a GMI-based sensor made of Co-rich magnetic microwire for real-time monitoring of position, and vibration. The fabricated GMI sensor response was explored over a high frequency range. The impedance spectrum showed a high GMI ratio and great field sensitivity response. We have shown that the GMI sensor based on longitudinal effect is more sensitive than
the transverse-based case and a commercial Gaussmeter. The practical utility of the high field sensitivity for a position real-time monitoring was demonstrated. The reliable and accurate measurement of position and speed of a moving object by the sensor was observed. This GMI-based sensor is highly promising for real-time position detection and oscillatory motion monitoring.

We have also demonstrated the utility of the low-field, high frequency response in a magneto-impedance based sensor for real-time monitoring of gear rotation. The impedance change attributed to a position-varying small magnetic field has been applied for rotation speed sensing. Small changes in the magnetic field, down to mOe level, were detected by tilting the gear, thereby simulating faulty gear detection. The high-frequency GMI-based sensor can be used for real-time condition monitoring and predictive failure of a rotating gear.

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CHAPTER SIX

NEW MAGNETO LC-RESONANT SENSOR TECHNOLOGY FOR BIOMEDICAL DEVICE APPLICATIONS

Note to Reader

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In this chapter, we present a new sensor based on a magnetic coil made of Co-rich melt-extracted microwire for the detection of small magnetic fields. The operating principle based on magneto-LC resonance is employed for the magnetic microwire coil (MMC). The changes in the effective resistance ($R$), reactance ($X$), and impedance ($Z$) due to a small external magnetic field are described. In addition, simulated respiratory patterns with various changes in amplitudes, frequencies, and waveforms are demonstrated. Through actual tests on a human volunteer, we demonstrate the high capabilities of the non-contact and non-invasive real-time monitoring for the respiratory motion and eye movement monitoring.

6.1 Introduction

Respiration is an essential mechanism to sustain life in an organism by ensuring an adequate oxygen supply and carbon dioxide removal from the body. Therefore, monitoring the
rate of respiration and its pattern can be used as crucial parameter to assess an individual’s health or progression of an illness [1-3]. In a normal state, the coordination of ventilation organs and the cardiovascular system perform a consistent respiratory rate and periodic rhythm or pattern. On the other hand, a change in rate or rhythmic pattern corresponding to an effort in breathing is found in abnormal states such as serious personal illnesses, obstructive sleep-apnea [2, 4, 5], cardiovascular disease [6-10], Cheyne-stroke [9, 11], or heart failure [3, 12-14]. In addition, the respiratory patterns of many breathing disorders have been observed and documented for diagnostic and therapeutic purposes [15, 16]. Therefore, a reliable and accurate measurement of respiratory rate and pattern is crucial to ongoing efforts of diagnosing and monitoring illness in human patients.

Several methodologies, including contact and non-contact methods, are currently being used to monitor the respiration rate and pattern of patients [2,15-19]. Of the non-contact methods, radar signal monitoring, optical based instruments, and thermal imaging analysis have been employed [20,21]. While these non-contact methods are advantageous, for example in child respiration monitoring, sophisticated technology, high error and time consuming analysis is required for implementation. On the other hand, electrical impedance-based methods, known as impedance pneumography and respiratory inductance plethysmography (RIP), have been established and widely employed in contact-based respiratory rate monitoring [1,2,22]. While these methods are more accurate and easier to use in respiration activity monitoring than the previously mentioned non-contact methods, they suffer from the downfalls of typical contact-based methods [2].

The working principle of a conventional magneto-inductive coil is based on an induced voltage attributed to the change of magnetic flux and is widely used to detect certain magnetic
fields [23]. The coupling between a pickup coil and a magnetic microwire provides a prominent signal [24-25]. However, the magneto-inductive coil based device is reliable and accurate only for a dc field measurement or low working frequencies. In recent decades, the giant magneto-impedance (GMI) effect in a ferromagnetic conductor has demonstrated promise for sensitive magnetic field sensing applications [26-28]. In particular, Co-rich magnetic microwires have exhibited extremely high GMI ratios and magnetic field sensitivities. Both of these properties make the magnetic microwires suitable for applications in long distance, non-contact sensing of small magnetic fields [29-31]. Unlike copper wires used in inductive coils, Co-rich amorphous microwires demonstrate excellent field sensitivity for high working frequency measurements [32]. Furthermore, the prototype of LC resonance sensor [33, 34], wireless magnetic probe [35-37], magneto-impedance sensor [38,39] and magnetic markers [40] have already been demonstrated for a non-contact, small magnetic field sensor in applications outside of the biomedical field.

Therefore, the development of a small magnetic microwire coil (MMC) sensor is promising for a new type of real-time, non-contact respiratory monitor and other bio-mechanical movement monitoring.

In this study, we present a new sensor based on MMC for the detection of small magnetic fields, which can be applied to real-time, non-contact respiratory monitoring. The operating principle of the MMC is discussed and the effective resistance ($R$), reactance ($X$), and impedance ($Z$) due to a small ambient magnetic field are described. In addition, simulated respiratory patterns with various changes in amplitudes, frequencies, and waveforms are demonstrated. Through actual tests on a voluntary person, we demonstrate the high capabilities of the non-contact and non-invasive real-time measurement of the MMC sensor for respiratory rate and eye movement monitoring.
6.2 Experimental

6.2.1 Sensor Design and Operating Principle

First we review the basic operating principle of the currently accepted contact-based impedance methods and compare it with the proposed MMC sensor. In impedance pneumography, two or four electrodes are attached to the patient’s chest wall and the impedance across the probes is measured [2]. Chest expansion and contraction change the impedance, thus recording a respiration pattern. In respiratory inductance plethysmography (RIP), an inductive coil is embedded in an elastic belt and fastened around the chest or abdomen [2]. The coil displacement caused by the chest expansion results in a change in inductance, thus a change in impedance. There are two major differences between the aforementioned inductance methods and the new MMC sensor proposed in this work. First, in contrast with the inductance coil in RIP, which is made from copper-based materials (non-magnetic), the MMC sensor is constructed from an amorphous magnetic microwire. This allows small external magnetic fields to significantly modify the inductance, hence the impedance. In essence, the advantage of this method, namely the substitution of a non-magnetic material with a magnetic material, allows the MMC sensor to be a non-contact impedance/inductance method since the expansion/contraction of the coil is not required.

Although the cause of the impedance change differs between the MMC and copper coil, both can be understood through fundamental lumped-element circuit theory. Similar to other solenoids, the MMC coil is an inductor, which creates a magnetic field when an electric current is flowing through it. A simplified model of a non-ideal inductor consists of the series inductance \( L \) and resistance \( R_L \) of the coil in parallel with the parasitic capacitance \( C_L \), as shown in Fig. 1(a) [41-43]. The complex impedance \( Z_{\text{coil}} \) of the coil has two components, the first from the series combination of \( L \) and \( R_L \) and the second from \( C_L \) in parallel. By applying the definitions of
inductive and capacitive reactance, the impedance of the coil can be expressed in terms of $L$, $R_L$, and $C_L$:

$$Z_{\text{coil}} = Z_{R_L} + Z_{C_L}$$ \hspace{1cm} (6.1)

$$Z_{\text{coil}} = \frac{1}{\frac{1}{R_L} + \frac{1}{\frac{1}{\omega L} + i/\omega C_L}}$$ \hspace{1cm} (6.2)

$$Z_{\text{coil}} \approx \frac{R_L + i\omega[L(1+\omega^2LCL)L/R^2] - CLR^2}{(1-\omega^2LCL)^2 + (\omega C_L R_L)^2}, \hspace{1cm} (6.3)$$

where $Z_{\text{coil}}$ is the coil impedance, $\omega$ is the angular frequency, and $i$ is the imaginary unit. For a small loop of wire with high conductivity, the $R_L$ would be very small and negligible. The coil impedance for the highly conducting limitation recovers:

$$Z_{\text{coil}} \approx \frac{\omega L}{1-\omega^2LC_L}, \hspace{1cm} (6.4)$$

An interesting phenomenon occurs when the inductive reactance ($X_L$) is equal to the capacitive reactance ($X_C$). In this case, the two branch currents are equal in magnitude but 180 degrees out of phase with each other. As a result, the total current turns out to be nearly zero. Then, the coil impedance becomes very large and exhibits a self-resonance phenomenon. At this moment, very little current flows into the loops which is known as anti-resonance [44, 45]. The resonance frequency is given as

$$f_r = \frac{1-(R^2_L C_L/L)}{2\pi \sqrt{LCL}}, \hspace{1cm} (5)$$

where $f_r$ is the resonance frequency. At high operating frequencies, $R_L$, $L$, and $C_L$ have significant frequency dependencies and the $Z$ should be measured with an impedance or network analyzer devices [42,46,47]. For the MMC-LC resonator, the greater impedance change can be achieved by
applying an external magnetic field to the LC resonator. The nature of resonance effect attributed
from MMC and GMI effect results a significant change to the measured impedance. The working
principle of the MMC magneto-LC resonance sensor is therefore different from a typical inductive
solenoid (made of non-magnetic wires) and from those using magnetic microwires as a sensing
core [24,30]. Fig. 6.1 (b) presents the setup of the impedance measurement using a mechanical
vibrator to simulate the chest movements of a human patient during respiration. Fig. 6.1 (c) shows
the potential application of the MMC for respiratory rate monitoring.

**Fig. 6.1** (a) An equivalent circuit, (b) experimental set up for a magnetic coil sensor, and (c) a
principle operation of MMC for a noncontact respiration rate monitoring device.
6.2.2. MMC Sensor Fabrication

The MMC sensor was constructed from a high quality melt-extracted amorphous microwire with nominal composition Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{12.5}$Nb$_{1}$. The diameter of the microwire is \(~60 \mu \text{m}\). The amorphous nature of the Co-rich microwires was characterized by an x-ray diffractometer (XRD). The atomic weight percent of the elements present in the amorphous microwires was investigated by energy dispersive spectroscopy (EDS). The fabrication details and material characterization (see Fig. 6.2) of the microwires can be found elsewhere [48, 49]. The constructed magnetic coil has 10 turns, is 7.0 mm in length, has a 3 mm internal diameter, and was wound around a quartz tube. A conventional inductive coil was also constructed for a comparative study with a commercial copper wire with 86.36 \mu \text{m} diameter.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig6_2.png}
\caption{(a) An XRD pattern of amorphous Co$_{69.25}$Fe$_{4.25}$Si$_{13}$B$_{12.5}$Nb$_{1}$ microwires; (b) Optical and (c) SEM images of the microwire; (d) EDS spectra of the microwire.}
\end{figure}
6.2.3. High-frequency Impedance Measurement

The frequency dependence of the effective impedance ($Z$), resistance ($R$), and reactance ($X$) of the magnetic coil was measured over high frequency range (1 MHz-1GHz) using a HP Agilent 4191A RF impedance analyzer interfaced with a LABVIEW program. The MMC was mounted onto a board with copper cladding on the opposite side. A coaxial cable of about 50 cm length was connected to the board via an SMA port, which was connected to the impedance analyzer shown in Fig. 6.1 (b). The HP 4191A was calibrated at the end of the 50 cm SMA cable using typical open, short, load (50 $\Omega$) standards. A frequency sweeping measurement of $R$, $X$, and $Z$ was performed for the MMC and copper coil. To introduce the source of the small magnetic field, a cylindrical 8 mm x 4 mm Neodymium magnet was fixed to a non-magnetic arm attached to a mechanical vibrator. The average distance between the coils and the permanent magnet was 3.0 cm, which can be extended up to 15 cm. The magnetic field was determined to be $\sim 10$ Oe at the center of the magnetic coil by a Gauss meter. Due to the source of the position-varying magnetic field, further study should be conducted to assess the applicability of this method to individuals with pacemakers. In principle stray magnetic fields from other sources, such as a cell phone or computer, should be minimized to reduce influence from the environment just as one would practice for procedures such as x-rays or magnetic resonance imaging (MRI).

6.2.4. Simulation of Real-time Respiratory Motion and Eye Movement Monitoring

A function generator was implemented to drive the mechanical vibrator and create a position-varying source of the small magnetic field. This portion of the setup was built to simulate the chest movements of a human patient during respiration as well as eyelid movements in human. The mechanical vibrator was driven at various frequencies of $f_{vib} \sim 0.02-0.2$ Hz and vibrational amplitudes ranging from 0.12-1.51 mm representing different respiration rates and depths,
respectively. In order to investigate the sensing discrimination, three different waveforms (square, sine, triangle wave) were used.

6.3. Results and discussion

6.3.1. Resonance Frequency of the MMC Resonator

The impedance of the MMC and copper coil was measured over the frequency range (1 MHz-1GHz) in the absence and presence of an external magnetic field aligned perpendicularly to the coil axis. Fig. 6.3 shows the self-resonance frequency of the copper and MMC without an external magnetic field occurs around 340 MHz (red), and 440 MHz (green), respectively. The frequency shift between these two impedance maxima occurs due to the different $R_L$ and $C_L$ from the coil material. The magnitude of $Z_{\text{Cu coil}}$ is much larger than $Z_{\text{MMC}}$ due to the high conductivity approximation given in Equation 6.4. The negligible $R_L$ of the copper coil allows the denominator of Equation 4 to reach a smaller value, hence a more pronounced peak in the impedance, when a excitation frequency approaches the self-resonance frequency, $f_{\text{ac}} \sim 340$ MHz \([43,44]\). Unlike the copper coil, the MMC has a higher resistivity, thus a lower magnitude of the impedance at its self-resonance frequency, since $R_L$ cannot be neglected.

The peak shift and magnitude change of $Z_{\text{MMC}}$ with the application of a small magnetic field, as featured in Fig. 6.3, is significant for sensor development. The obtained measurement ensures the advantages of the MMC magneto-LC resonance sensor for a small field detection. It can be seen from Fig. 6.3 that the significant increase in the $Z$ after introducing the small magnetic field to the MI coil was observed at working frequencies less than 340 MHz. On the other hand, the measured $Z$ noticeably decreases from the initial value at higher frequencies. Therefore, the working frequency certainly plays an essential role for a magnetic field sensing design. The
magnitude change and peak location shift, owing to the interaction of the external magnetic field and the magnetic microwire, are highly promising characteristics for the development of a respiration rate monitor using a magnetic marker, such as a small permanent magnet, on the patient body. To further explore the sensing capability of the MMC, the proceeding sections will be devoted to examining the potential of frequencies around (and including) the self-resonance peak location of $Z_{MMC}$ as the driving frequency for the sensor.

![Graph showing frequency dependence of the effective impedance ($Z$) of a copper coil and MMC (10 turns, $D = 3$ mm.) before and after introducing H-field $\sim 10$ Oe. The inset shows an enlarged region around the resonance frequency of the copper coil.](image)

**Fig. 6.3** Frequency dependence of the effective impedance ($Z$) of a copper coil and MMC (10 turns, $D = 3$ mm.) before and after introducing H-field $\sim 10$ Oe. The inset shows an enlarged region around the resonance frequency of the copper coil.

6.3.2. High-frequency MMC Response

As mentioned previously, the constructed coils are composed of the inductance, $L$, and the parasitic elements $R_l$ and $C_l$. The frequency dependent circuit characteristics for the coil can be
split into three regimes: resistive, inductive, and capacitive, as defined in Fig. 6.4(a). Taking a closer look into the resistive ($f_{ac} \leq 200$ MHz) and inductive regimes ($200 < f_{ac} \leq 360$ MHz), the measured $R$ contributes to $Z$ more than $X$ does. However, $R$ and $X$ both significantly contribute to $Z$ in the capacitive regime for the higher frequency range ($f_{ac} > 360$ MHz).

To get more insight into the impedance response owing to a position-varying magnetic field, the time dependence of the impedance was measured at several working frequencies as seen in Fig. 6.4(b): $f = 200$ (A), 280 (B), 320 (C), 340 (D), 480 (E), and 600 MHz (F). The working frequencies were chosen where $R$, $X$, and $Z$ exhibit a maximum: $f_{ac} \sim 200$ (A), 320 (C), and 340 (D) MHz, respectively. Fig. 6.4(b) shows the change in the effective impedance ($\Delta Z_{peak}$) detected from the position-varying permanent magnet at a distance $d \sim 3$ cm away from the MMC. The mechanical vibrator was operated at frequency $f_{vib} = 0.05$ Hz with a displacement of $\sim 0.87$ mm. It can be seen from Fig. 6.4(b) that the large changes in $Z$ occur at $f_{ac} \sim 200$, 280, and 480 MHz which are located on the resistive, inductive, and capacitive regimes, respectively. Therefore, any of these points are suitable for the field sensing operation. Conversely, there is no significant change in $Z$ observed at the LC-resonance frequency $f_{ac} \sim 360$ MHz (see Fig. 6.4(b)). At LC-resonance, there is a superposition of opposite flowing currents and as a consequence the current across the coil will be minimum for this frequency [44, 47]. As a result, the change in the impedance of the MMC is less pronounced at the resonance frequency.
Fig. 6.4 (a) The frequency dependence of the effective impedance ($Z$), resistance ($R$), and reactance ($X$) of the MMC coil with no magnetic field present. (b) time dependence of $\Delta Z$ of the MMC coil under the influence of a small permanent magnet vibrating at 0.05 Hz with a displacement of 0.87 mm at different working frequencies.

6.3.3. Sensing Reliability, Sensitivity, and Accuracy

The coil sensing reliability was investigated for the working frequency of $f_{ac} \sim 200$ MHz. Fig. 4(a) shows the change in impedance resulting from a square waveform applied to the mechanical vibrator and attached magnet at $f_{vib} = 0.1$, 0.05, and 0.02 Hz and amplitude $\sim 0.87$ mm. As seen from the period extracted from the impedance change in Fig. 4 (a), the original oscillatory pattern applied to the mechanical vibrator and attached magnet is recovered. Unsurprisingly, the copper coil shows no significant change in $Z$ over time since there is no interaction with a small magnetic field. In general, conventional solenoids are sensitive only to the ambient field perpendicular to the cross-sectional coil area since the only way a voltage can be induced in this coil is by Faraday’s law. For all cases presented in this work, the majority of the field lines emanating from the permanent magnet are parallel to the cross-sectional area of the coils, which is parallel to the coil wound direction. In this configuration, the magnetic microwire of the MMC exhibits a significant and sensitive response to small magnetic fields due its magneto-
impedance properties, which have been characterized and described elsewhere [32]. As compared to a single magnetic microwire, the impedance response of the MMC increases by 250% (see Fig. 6.5).

![Fig. 6.5](image)

**Fig. 6.5** (a) The comparison of impedance changes between MMC, and (b) a single sensing element of the amorphous magnetic microwire under the vibrating magnet.

**Fig. 6.6(b)** shows the $R$ and $X$ components of $Z$ at different $f_{\text{vib}}$. At an excitation frequency of $f_{\text{ac}} = 200$ MHz, the $R$ component contributes most to $Z$ as expected in the resistive regime. What can be noticed in this Fig. is that monitoring either $R$ or $X$ of the MMC coil is promising for a magnetic field sensor. As mentioned previously, the large change in resistance and reactance due to the small position-varying magnetic field is a direct result of the giant magneto-impedance effect.

By applying a small permanent magnet to the patient’s chest wall, small movements of varying frequency can be detected by the MMC coil. To simulate this effect, several different driving frequencies (0.02 to 0.2 Hz) have been applied to the function generator to simulate different breathing rates as seen in Fig. 6.6(b). It has been found that during a normal rest state,
adult breathing occurs at a consistent rate of 12-20 breaths per minute or 0.2-0.3 Hz [1,21,50], while a rapid breathing rate of ~40 breaths per minute or 0.7 Hz has been found in an excited state [17,18]. Moreover, apnea from patients with respiratory abnormalities [4,7,22] can be easily detected as a flat line in the impedance response. Overall, the impedance responses illustrated in Fig. 6.6(b) demonstrate great promise for breathing state detection.

Fig. 6.6 (a) The time dependence of the change in impedance (Z) of the MMC due to the position-varying magnet at different \( f_{\text{vib}} \). The impedance change of the copper coil under the vibrating magnet at \( f_{\text{vib}} \) is shown for comparison. (b) The time dependence of \( R \), \( X \), and \( Z \) of the MMC coil at different vibrational frequencies.

In order to demonstrate the ability of the MMC coil to detect the depth of breathing, the vibration amplitude of the function generator with vibrational magnitudes was performed. As can be seen from Fig. 6.7, the \( R \), \( X \), and \( Z \) show marked response to small changes in vibration amplitudes, 1.15, 1.07, 0.65, 0.22, and 0.12 mm over time for the working frequency ~200 MHz. It is clearly seen that the \( R \) contributes to the \( Z \) more than \( X \) does. The measured \( Z \) increases when the magnetic marker moves from the lowest to the highest position, and exhibits a similar feature to the \( R \) value. Unlike the \( R \) and \( Z \) responses; however, \( X \) shows the reverse response. As mentioned
previously, this is due to the impedance responses of the ferromagnetic microwires at high stimulating frequency. This field sensitive response can be used for the real-time respiratory monitoring. Furthermore, it has been demonstrated that sleep-disordered breathing patterns in Cheyne-stoke, sleep apnea, and chronic heart failure patients have similar features to Fig. 6.7 [5, 15,51]. Moreover, the respiratory rate measuring from the abdomen and chest wall movements exhibits similar characteristics described elsewhere. [2,4,50,51]. Therefore, the incorporated distinct sensing characteristics of the MMC coil shown in Figs. 6.6 and 6.7 are potentially detected and recognized for the respiratory rate and patterns.

Fig. 6.7 The time dependence of the measured impedance (Z), resistance (R) and reactance (X) from the vibrating magnet with different amplitudes.

The discrimination of the input field signals from various oscillatory patterns such as square, sine, and triangle waves was investigated. An excellent response from the input-modulated signals was observed (see Fig. 6.8). The retrieved amplitude and $f_{\text{ vib}}$ are consistent and reliable over the time measurement. Similar trend to the preceding section, the coil responses to the external field for the $R$, and $Z$ is comparable in magnitudes, but the measured $X$ has reverse change attributed to the magnetic field as expected. As mentioned earlier, common respiratory patterns, for example, sinusoidal wave type, triangle waves, square-like shape, varying periods, increasing
depth *etc.* has been recognized and may be observable via the MMC. Interestingly, the sensing characteristics demonstrated in Fig. 6.8 absolutely promote the coil for the real-time respiratory measurement.

![Fig. 6.8](image)

**Fig. 6.8** The time dependence of the measured impedance (Z) from the vibrating magnet with different types of waveforms.

### 6.3.4 Measurement of Real-time Respiratory in a Human Patient

Monitoring the real-time respiration of human patient is useful for personal healthcare assessment. We have explored a practical applicability of the MMC magneto-LC resonance technology for active respiratory monitoring. Fig. 6.9 illustrates the signals obtained from the trial session using the working frequency of 200 MHz. The acquired signal displays a precise, real-time respiratory pattern of a healthy man, who voluntarily participated in the test, from the normal to controlled breathing modes (Fig. 6.9 (a)). It is clearly seen from the recorded pattern, that normal
breathing rate can be determined at 17 cycles or 17 breaths per minute. The controlled breathing was recorded and illustrated in Fig. 6.9(b).

![Breathing patterns](image)

**Fig. 6.9** The breathing patterns (impedance vs. time) of a healthy man in (a) normal and (b) controlled (three different amplitudes) modes.

The person was instructed to perform regular inhale per breath corresponding to the first three peaks (between 0th and 9th second, Stage I). The person was then instructed perform deeper inhale per breath (aimed at increasing the amplitude of breathing) which corresponded to the next three highest peaks (between 10th and 24th second, Stage II), and to slightly reduce his breathing amplitude which corresponded to the next three lower peaks (between 25th and 36th second, Stage III). Finally, the person was instructed to return to Stage I and II (between 37th and 60th second). In this trial, the person controlled his breath for ~3 seconds per cycle and performed three consecutive breathings for an identical breathing amplitude. All these results demonstrate the excellent capability of the MMC magneto-LC resonance technology for active respiratory motion monitoring of a human patient.
6.3.5 Measurement of Human Eye Movement

Real-time eye movement monitoring can provide useful information pertaining to sleep behavior and non-verbal communication [52,53]. In this section, the MMC magneto-LC resonance sensor is employed to detect a magnetic signal from a magnetic marker attached to a human eyelid. In this measurement, the MMC sensor is attached to the left temple of a pair of glasses. The volunteer subject is instructed to sit on a comfortable chair. A rare earth neodymium cylindrical magnet of which the diameter and thickness are 3 mm and 2 mm, respectively, is vertically attached to the left eyelid of the human test subject employing a Band-Aid. The magnetic field strength at the MMC sensor location is approximately 0.2 Oe. For further practical applications, the neodymium magnet is replaced by 15 milligrams of supermagnetic nanoparticles (Fe₃O₄ nanorods), which are embedded in Band-Aid [54]. The average length and width of the Fe₃O₄ nanorod are 41 nm and 7 nm, respectively. The nanorod sample was provided by our synthesis lab in the Physics Department at the University of South Florida (USF).

As can be seen from Fig. 6.10 (a), the generated magnetic signal due to the eyelid moving up and down is clearly detectable. The change in the impedance ~400 mΩ corresponding to the small variation of the magnetic field is sensed. In this experiment, the test subject was instructed to blink his eye at a normal setting for the first 60 seconds (12 cycles per minute), and then blink faster for another 40 seconds (16 cycles per minute). It is worth mentioning that this small field change cannot be detected by a commercially available Gauss meter (dc-mode), which was mentioned in the Chapter 5.

Moreover, the volunteer person was instructed to close his eyelid and moved his eyeballs at three different positions (left, center, and right). Interestingly, the obvious pattern is observed, as shown in Fig. 6.10 (b). As can be seen, the impedance signal increased (t ~ 7 seconds) when the
The eye moved to the left (closer to the sensor). The change in the impedance ~250 mΩ was detected. Then the signal was reduced to the base line when the eye moved back to the center (t ~ 14 seconds). On the other hand, the signal decreased (~250 mΩ) from the base line (center) when the eye moved to the right (t ~ 21 seconds). It is worth noticing that the small kinks that are observed in Figs. 6.10 (a)-(d) correspond to the blinking eye.

**Fig. 6.10** The impedance change vs. time of a human test subject from the eye blinking patterns integrating with (a) permanent magnet and (c) magnetic nanoparticles. The observed patterns due to the eye movement in different positions (left, center, and right) integrating with (b) permanent magnet, and (d) magnetic nanoparticles.
In addition, the duplicated experiment was performed by replacing the neodymium magnet with magnetic nanoparticles that are embedded in Band-Aid. In this configuration, the neodymium magnet is attached to the left nose pad of the glasses to magnetize the magnetic nanoparticles ($H_{ex} \sim 0.2 \text{ Oe}$) and tune the sensor sensitivity. Patterns that are similar to those shown in Fig. 6.10 (a) and (b) are observable as can be seen in Fig. 6.10 (c) and (d). This method, which employs magnetic nanoparticles can developed further, e.g. magnetic nanoparticle gel or cosmetics with magnetic nanoparticles for a practical application. Therefore, the MMC magneto-LC resonance technology is promising for a real-time eye motion monitoring.

6.4 Summary

In summary, we have demonstrated the MMC based sensor made from the Co-rich magnetic microwire for a small magnetic field sensing. The reliable and sensitive responses in the measured $R$, $X$, and $Z$ of MMC to an applied external magnetic field were investigated. We observed the change in the impedance when MMC experiences the small magnetic field with various amplitudes, frequencies, and waveform-types of oscillations. The MI response to a small oscillatory magnet, which is virtually simulated physiological movements corresponding to human-respiratory activities, was observed. This newly developed MMC magneto-LC resonance technology is very promising for active, non-contact respiratory monitoring of a human patient, real-time eye movement monitoring, and for other biomedical field sensing applications.
6.5 References


[40] E. Henin, M. Bergstrand, W. Weitschies, M.O. Karlsson, Meta-analysis of magnetic marker monitoring data to characterize the movement of single unit dosage forms though the gastrointestinal tract under fed and fasting conditions, Pharm. Res. 33 (2016) 751-762.


CHAPTER SEVEN

CONCLUSIONS AND OUTLOOK

7.1 Summary

As were mentioned in the previous chapters, the major results obtained from this dissertation are: (i) the optimization of the GMI effect in Co-rich magnetic microwires employing the Joule annealing techniques, (ii) the fabrication of a new class of GMI based sensor using the optimal Co-rich microwires for real-time monitoring of position, vibration, and gear rotation, and (iii) the development of the novel magneto LC resonance sensor technology for biomedical sensing applications. These findings are summarized below.

The high frequency GMI response in melt-extracted Co_{69.25}Fe_{4.25}Si_{13}B_{13.5} microwires was enhanced using the Joule-heating technique. The application of a small dc current at a suitable time period to the amorphous microwires promoted the GMI effect and magnetic field sensitivity. The optimum annealed parameters of 5 mA for 20 minutes remarkably improved the GMI ratio, and its field sensitivity up to 610% (1.7 time of that of the as-cast state) at $f \approx 20$ MHz, and 500%/Oe (more than two times of that of the as-cast) at $f \approx 40$ MHz, respectively. Low current annealing relieved a residual stress, which was generated during the fabrication process and enhanced the circular outer shell domain structure without major modifications of the amorphous microstructures. This method improves the GMI response of melt-extracted magnetic microwires and can be practically applied to GMI-based sensors in the high-frequency range.
The optimization of the high frequency GMI-effect in melt-extracted Co-rich microwires, which was subjected to the single-step anneal (SSA) and multi-step anneal (MSA) techniques, was systematically studied. Increasing the MSA annealed parameters from 20 mA to 100 mA for 10 minutes remarkably improved the GMI ratio, and its field sensitivity up to 760% (1.75 time of that of the as-prepared), and 925%/Oe (more than 17.92 times of that of the as-cast) at \( f \approx 20 \text{ MHz} \), respectively. The employment of the MSA technique successfully enhances the surface domain structures of the Co-rich magnetic microwires. This alternative tailoring method is suitable for improving the magnetic field the GMI sensitivity for a small field response. The high sensitive response of the GMI to a weak magnetic field is highly promising for biomedical sensing applications.

Based on the aforementioned results, the engineered magnetic microwires have been employed to fabricate the new class of GMI based sensor for real-time monitoring of position, vibration, and gear rotation.

A position sensor based on the high frequency the GMI effect was fabricated using the Co-rich magnetic microwire. The fabricated GMI sensor response was explored over a high frequency range up to 1 GHz. The impedance spectrum showed a high GMI ratio and great field sensitivity response. We have shown that the GMI sensor based on the longitudinal effect is more sensitive than the transverse-based case and a commercially available Gaussmeter (dc mode). The practical utility of the high field sensitivity for a position real-time monitoring was demonstrated. Reliable and accurate measurements of position and speed of a moving object by the sensor were carried out. This GMI-based sensor is highly promising for real-time position detection and oscillatory motion monitoring.
The utility of the low-field, high frequency response in a magneto-impedance based sensor for real-time monitoring of gear rotation was demonstrated. The impedance change attributed to a position-varying small magnetic field has been applied for rotation speed sensing. Small changes in the magnetic field, down to mOe level, were detected by tilting the gear, thereby simulating faulty gear detection. The high-frequency GMI-based sensor can be used for real-time condition monitoring and predictive failure of a rotating gear. In particular, the GMI-based sensor is well suitable for rotational speed monitoring at a remote distance, which is commonly used for product packaging, conveying, and tracking in industries.

Finally, we have successfully fabricated a new, high sensitive magneto LC resonance sensor for a small field sensing applications. The reliable and sensitive responses of the measured $R$, $X$, and $Z$ of the magneto LC resonance sensor to an applied external magnetic field were investigated. We observed the change in the impedance when the magneto LC resonance sensor experiences the small magnetic field with various amplitudes, frequencies, and waveform-types of oscillations. The MI response to a small oscillatory magnet, which is virtually simulated physiological movements corresponding to human-respiratory activities, was observed. The actual tests on the voluntary person were performed, demonstrating the excellent performance of the sensor. This newly developed magneto-LC resonance technology is very promising for active, non-contact respiratory monitoring of a human patient, real-time eye movement monitoring, and for other biomedical field sensing applications.

7.2 Future Research

The results of this dissertation also open up exciting opportunities for applying the highly sensitive GMI-based sensors to determine the stray field of magnetic nanoparticles. In particular,
the contactless measurement of the magnetic field from functionalized magnetic nanoparticles with
very low concentration (micro, nano grams) is essential for magnetic biomarker, magnetic particle
imaging, magnetic hyperthermia treatment of cancer, and modern drug delivery. To clarify this
aspect, previous and on-going studies have employed contact method to measure the impedance
response when a sensing element experiences the stray field of magnetic nanoparticles. The contact
area between the test sample and the physical motion of the magnetic nanoparticles during the
sensor operation greatly affect the consistency of experimental data. Moreover, the sensing
element or samples become contaminated after a contact measurement, which is not practical for
a large scale application. Thus, the alternative contactless technique is more suitable. The new
class of high frequency GMI-based sensors are capable of measuring a small external magnetic
field without making a direct contact with the test sample. The size of the GMI sensing element
can be miniaturized and integrated into micro-magnetic sensors. However, in order to attain this
aspect, the interference between a sample that is being examined and the environmental field
during the measurement should be optimized.

The superior field sensitivity of the new class of the GMI based sensors is also highly
promising for a sensitive, cost-effective on-chip biosensing platform. In particular, that is true for
the detection of bio-functionalized magnetically sub-micron particles passing through a
microfluidic channel. This approach allows a scaleable microsensor for biomedicine and
environmental monitoring. To clarify this aspect, typically iron oxide magnetic nanoparticles
possess superparamagnetic properties and can be magnetized in the presence of an external
magnetic field. These nanometer-sized particles can be coated with hydrophobic ligands,
biological species, or antibodies for any biological applications. The magnetization of bio-
functionalized magnetic nanoparticles is different when they bind to the proteins, antigens, viruses
or other biological entities in the presence of an external field. Therefore, the magnetic signals from magnetically tagged biological entities are useful for the detection of magnetic carriers for biomedical applications and microfluidic diagnostic technology for point-of-care testing.

A combination of the new magneto LC resonance sensor and the magnetic labeling provides a novel platform for continued studies on biomedical device applications. The practical approaches on the diagnose and management of tremor in patients owing to Parkinson disease as well as other movement disorders can be carried out using magnetically labelling sensors. More importantly, the quantitative assessment of the tremor’s frequencies and their magnitudes will facilitate physicians to classify the symptom types and the progress of medical treatment in the patients. Moreover, real-time monitoring of breathing patterns in a human patient, e.g., sleep apnea, cardiac disease is useful for patient screening for early medical analysis. The data base of the breathing patterns in patients will facilitate non-specialist to classify the early stage of an abnormal condition. With ventilation monitoring system, a hospital will ensure better care with fewer staff members.
Appendix A: List of Publications


Appendix B: Conference Presentations


8. C. Albrecht, T. Eggers, **O. Thiabgoh**, and M. Phan, Detecting superparamagnetic nanoparticles with high frequency giant magneto-impedance microwires, REU-RET 2017 Poster Symposium, July, 26, 2017, Tampa FL, USA.

9. V. Ortiz, J. Dietz, **O. Thiabgoh**, T. Eggers, and M. H. Phan, the 10th annual USF Graduate Research Symposium 2017, March, 21, 2018, Tampa FL, USA (Poster).
Appendix C: Patents Application

1. File No. 210112-9008-US01


    Title: “MAGNETO-LC RESONANCE TECHNOLOGY FOR REAL-TIME
    RESPIRATORY MOTION MONITORING”

    Inventors: **Ongard Thiabgoh**, Tatiana Marie Eggers, and Manh-Huong Phan

    MBF File No. 210112-9008-US01

2. Patent Application

    Title: “DETECTING MAGNETIC NANOBIOMARKERS USING A SINGLE GIANT
    MAGNETO-IMPEDANCE MICROWIRE”

    Inventors: **Ongard Thiabgoh**, Tatiana Marie Eggers, and Manh-Huong Phan

    Status (in preparation)
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