UNIVERSITI PUTRA MALAYSIA

STRUCTURAL, MAGNETIC AND DIELECTRIC PROPERTIES OF FeTe1-xMx (M = Se, S) SUPERCONDUCTOR PREPARED BY SOLID STATE REACTION AT AMBIENT PRESSURE

EDMUND LIM HUA HANG

FS 2015 8

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By

EDMUND LIM HUA HANG

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the Degree of Master of Science

STRUCTURAL, MAGNETIC AND DIELECTRIC PROPERTIES OF $\mathbf{FeTe_{1-\mathit{x}}M_{\mathit{x}}}$ (M = Se, S) SUPERCONDUCTOR PREPARED BY SOLID STATE REACTION AT AMBIENT PRESSURE

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November 2015

Chair : Associate Professor Chen Soo Kien, PhD
Faculty: Science

The polycrystalline samples with nominal composition $FeTe_{1-x}Se_x$ (x = 0.1 - 0.5) and $FeTe_{1-x}S_x$ (x = 0.1 - 0.3) were prepared via solid-state reaction method. The pellets were sealed inside a stainless steel tube with both ends clamped for sintering at ambient pressure. Argon gas flow was maintained throughout the heat treatment in order to minimize the oxidation. According to the X-ray diffraction (XRD) data, both $FeTe_{1-x}Se_x$ and $FeTe_{1-x}S_x$ were indexed to tetragonal structure with space group of P4/nmm. The lattice parameters *a*- and *c*-axis decrease with the substitution of Se and S. SEM images showed that the samples developed into plate-like grain structure gradually with the increase of Se and S concentration. EDX results show that the ratio of Te substituted by Se and S are gradually increased with increasing of the Se and S concentration.

For $FeTe_{1-x}Se_x$, onset of superconducting transition temperature, T_c was found to increase with Se concentration. The T_c is about 13.8 K and 13.5 K for x = 0.4 and 0.5, respectively compared with 10.6 K for x = 0.1. Substitution of Te with S suppresses the antiferromagnetic transition of the parent compound FeTe as shown by the temperature dependence of magnetic moment measurements. The samples with S content x = 0.25 and 0.30 exhibited diamagnetism suggesting the coexistence of magnetic and superconducting phase in these samples.

Both $FeTe_{1-x}Se_x$ and $FeTe_{1-x}S_x$ exhibited ferromagnetic behaviour as shown by the field dependent magnetization. In general, the values of coercivity, retentivity and magnetization (at 10 kG) decrease with Se and S concentration. The values of these magnetic properties are comparable for both Se and S substitution.

Both $FeTe_{1-x}Se_x$ and $FeTe_{1-x}S_x$ show negative dielectric permittivity (ε_rĐQGWKLVFRXOG be due to the plasma-like behaviour of the electron gas at frequency less than the

plasma frequency. Se substitution decreases the dielectric constants Û$_r$• while increased the Û$_r$•IURP x = 0.0 to x = 0.5. For S substitution, Û$_r$•GHFUHDVed while Û$_r$•LQFUHDVHIURP x = 0.0 to x = 0.1. However, Û$_r$•LQFUHDVHG DQG Û$_r$•GHFUHDVHG ZLWK LQFUHDVLQJ 6 concentration for x = 0.25 and 0.30. $FeTe_{1-x}S_x$ (x = 0.1) has greater magnitude of Û$_r$•DQG Û$_r$•PRUHUDSLGHQHUJ\ORVVDQGKLJKHU ac conductivity (ů$_{ac}$) compared to $FeTe_{1-x}Se_x$ (x = 0.1 - 0.3) at 1 kHz.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

SIFAT STRUKTUR, MAGNET DAN DIELEKTRIK $FeTe_{1-x}M_x$ (M = Se, S) SUPERKONDUKTOR YANG DISEDIAKAN MELALUI TINDAK BALAS PEPEJAL PADA TEKANAN AMBIEN

Oleh

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Pengerusi : Profesor Madya Chen Soo Kien, PhD
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Sampel polihablur dengan komposisi nominal $FeTe_{1-x}Se_x$ (x = 0.1 - 0.5) dan $FeTe_{1-x}S_x$ (x = 0.1 - 0.3) disediakan melalui kaedah tindak balas keadaan pepejal. Pelet yang siap dimasukkan dalam tiub keluli tahan karat dengan kedua-dua hujungnya diapit untuk pensinteran pada tekanan ambien. Gas argon dialirkan sepanjang proses pemanasan untuk mengurangkan pengoksidaan sampel. Menurut data pembelauan sinar-x (XRD), kedua-dua $FeTe_{1-x}Se_x$ dan $FeTe_{1-x}S_x$ diindeks kepada struktur tetragonal dengan kumpulan ruang P4/nmm. Parameter kekisi paksi *a* dan *c* berkurang dengan penggantian Se dan S. Imej SEM menunjukkan sampel menjadi struktur plat dengan penambahan kepekatan Se dan S. EDX menunjukkan nisbah penggantian Te oleh Se dan S meningkat dengan peningkatan kepekatan Se dan S.

Untuk $FeTe_{1-x}Se_x$, didapati bahawa suhu peralihan superkonduktor, T_c meningkat dengan kepekatan Se. T_c bagi x = 0.4 dan 0.5 ialah ~ 13.8 K dan ~ 13.5 K masing-masing berbanding dengan ~ 10.6 K bagi x = 0.1. Penggantian Te oleh S telah menyekat peralihan antiferomagnet FeTe seperti yang ditunjukkan oleh pengukuran pergantungan momen magnet pada suhu. Sampel dengan penggantian S, x = 0.25 and 0.30 menunjukkan diamagnet dan ini mencadangkan fasa magnet dan superkonduktor wujub bersama dalam sampel ini.

Kedua-dua $FeTe_{1-x}Se_x$ dan $FeTe_{1-x}S_x$ mempamerkan sifat feromagnet seperti yang ditunjukkan oleh pergantungan kemagnetan pada medan. Secara amnya, nilai coercivity, retentivity dan kemagnetan (pada 10 kG) berkurang dengan kepekatan Se dan S. Nilai-nilai sifat magnet ini adalah setanding bagi kedua-dua penggantian Se dan S.

Nilai ketelusan dielektrik (Ů$_{r}$•EDJL NHGXD-dua $FeTe_{1-x}Se_x$ dan $FeTe_{1-x}S_x$ adalah negatif dan ini mungkin disebabkan oleh kelakuan plasma gas elektron pada frekuensi yang

kurang daripada frekuensi plasma. Penggantian Se mengurangkan Ůr• WHWDSL meningkatkan Ůr•GDUL x = 0.0 ke 0.5. Untuk penggantian S, Ůr•EHUNXUDQJDQ Ůr• meningkat daripada x = 0.0 ke 0.1. Penambahan kepekatan S hingga x = 0.25 dan 0.30 telah meningkatkan Ůr•GDQPHQJXUDQJNDQ Ůr•VHFDUDSHUEDQGLQJDQ)H7H$_{1-x}$S$_x$ (x = 0.1) mempunyai magnitud Ůr•GDQ Ůr•DQJOHELKEHsar, kehilangan tenaga yang lebih cepat dan kekonduksian ac yang lebih tinggi berbanding dengan $FeTe_{1-x}Se_x$ (x = 0.1 - 0.3) pada 1 kHz.

ACKNOWLEDGEMENTS

First and foremost, I would like to thank God for giving me courage, wisdom, and healthiness to complete my project.

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LIST OF ABBREVIATIONS AND SYMBOLS

a, *b*, *c*	lattice parameter
AC	alternating current
AF	audio frequency
Al	Aluminum
AISI	American Iron and Steel Institute
B	magnetic induction
B_c	critical field
B_{c1}	lower critical field
B_{c2}	upper critical field
BSCCO	Bi-Sr-Ca-Cu-O superconductor
C	capacitance
Cd	Cadmium
DC	direct current
E	electric field
$\mathring{U}_o$	permittivity of free space
$\mathring{U}_r$ •	dielectric constant
$\mathring{U}_r$ • •	dielectric loss
FeS	Iron Sulphide
FeSe	Iron Selenide
FeTe	Iron Telluride
FCL	Fault Current Limiters
F_L	Lorentz force
G	conductance
Hg	Mercury
HTS	high temperature superconductor

I_c	critical current
ICDD	International Centre for Diffraction Data
J	electrical current density
J_c	critical current density
Maglev	magnetic levitated trains
MgB_2	Magnesium Diboride
MPMS	magnetic property measurements system
MRI	Magnetic Resonance Imaging
Nb	Niobium
NbTi	Niobium-Titanium
R	resistance
RF	radio frequency
S	Sulphur
Se	Selenium
SEM	scanning electron microscopy
SMES	superconducting magnetic energy storage
SQUID	superconducting quantum interference device
TBCO	Tl-Ba-Cu-O superconductor
TCBCO	Tl-Ca-Ba-Cu-O superconductor
T_c	superconducting transition temperature
Te	Tellurium
V	potential difference
VSM	vibrating sample magnetometer
XRD	x-ray diffraction
YBCO	Y-Ba-Cu-O superconductor
[illegible]	dielectric loss tangent
$\mathring{u}_{ac}$	ac-conductivity

ρ	resistivity
κ	Ginzburg-Landau Coefficient
ΔT_c	superconducting transition breadth
λ	penetration depth
ξ	coherence length
ω	angular frequency
χ	magnetic susceptibility

CHAPTER 1

INTRODUCTION

1.1 A Brief History of Superconductor

The discovery of superconductivity is widely regarded as one of the enormous scientific findings in the 20th century. This unique property causes certain material to flow current without resistance at low temperatures and enables a wide range of innovative technology applications. In 1911, Heike Kamerlingh Onnes, a Dutch physicist of Leiden University was managed to liquefy helium. Soon after that, he discovered mercury, Hg showed a dramatic drop in resistance to zero by cooling it with liquid helium down to 4.2 K (Figure 1.1). That was the first superconducting element found (Cyrot and Pavuna, 1992). Following this phenomenon, a number of superconductors were discovered for examples Aluminum (Al), Lead (Ld), Niobium (Nb), Niobium-Titanium (NbTi) and several alloys. These superconductors are known as conventional superconductor which superconduct at very low temperatures (Roslan, 2004). In the 1960s, NbTi has been chosen for commercial superconducting**.**

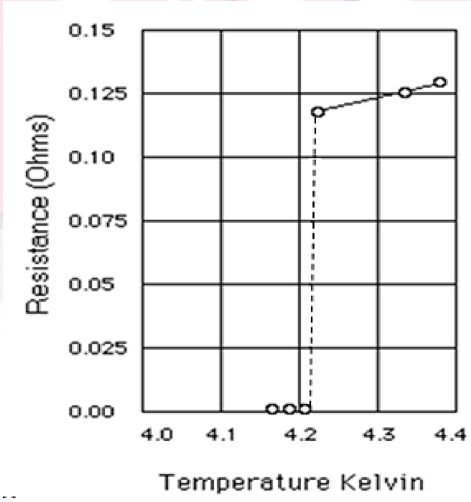

Figure 1.1. Zero resistance of Hg at 4.2 K as reported by H. K. Onnes in 1911 (Kittel, 2005)

Soon after the discovery of zero resistance in mercury, it has led to an intensive research on finding new superconductors. Figure 1.2 shows the timeline of superconductors with critical transition temperature, T_c according to the year of discovery.

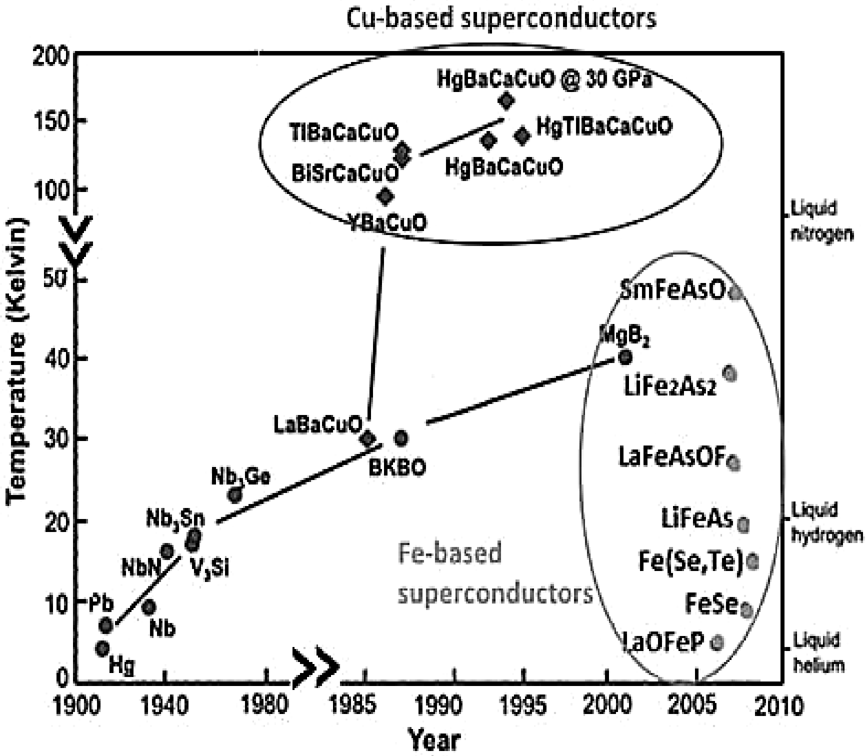

Figure 1.2. Timeline of superconductors with superconducting transition temperature, T_c according to the year of discovery (Mousavi et al., 2014)

In 1933, Meissner and Ochsenfeld discovered another unique property of superconductor where it expels magnetic flux from its interior when cooled below its T_c. This is known as Meissner effect. After the discovery of Meissner effect, the London Model was proposed in 1935 by taking account the Meissner effect to predict the penetration depth of magnetic flux into a superconductor (Cyrot and Pavuna, 1992). *LQ]EXUJ DQG /DQGDX SURSRVHG D ³PDFURVFRSLF WKHRU Ginzburg-Landau theory in 1950 to explain the macroscopic properties of superconductor. In 1957, Abriskosov managed to classify superconductors into two types i.e. type I and type II based on their magnetic properties. In the same year, a complete microscopic theory of superconductivity was proposed by the three American physicists, Bardeen, Cooper and Schrieffer. This theory is named after the first letter of the three physicists which is known as Bardeen-Cooper-Schrieffer (BCS) theory. BCS theory explained the interactions of gas of conduction electrons with elastic waves of the crystal lattice. In 1962, Josephson postulated fascinating quantum tunnelling effects that could occur when superconducting electron pairs tunnel through an extremely thin layer (~ 10 Å) of insulator into another superconductor. After all, this effect was known as Josephson effects which can be used to explain the basic of the applications of superconducting electronic devices (Kittel, 2005; Roslan, 2004; Cyrot and Pavuna, 1992). In 1986, Bednorz and Muller, scientists of IBM Laboratory at Switzerland, discovered superconductivity in perovskite La-Ba-Cu-O (LBCO) with T_c up to 35 K (Bednorz and Muller, 1986). This discovery had triggered active research on finding high temperature superconductors (HTS). In the following year, Wu and his co-workers found Y-Ba-Cu-O (YBCO) superconductor with T_c = 93 K (Wu et al., 1987), above the boiling point of liquid nitrogen (77 K). This finding leads to the usage of liquid nitrogen as a cooling medium which is much cheaper than liquid helium. In the following year, Maeda et al. (1988) discovered the Bi-Sr-Ca-Cu-O (BSCCO) superconductor with T_c = 105 K while Ti-Ba-Cu-O (TBCO) and Ti-Ca-Ba-Cu-O (TCBCO) were discovered to be superconducting at 90 K (Sheng and Hermann, 1988a) and 120 K (Sheng and Hermann, 1988b), respectively in the same year. The Hg system of cooper oxide high temperature was discovered in 1993 with T_c at 94 K (Hg-Ba-Cu-O) (Putilin et al., 1993) and 120 K (Hg-Ba-Ca-Cu-O) (Schilling et al., 1993). In 2001, Nagamatsu et al. (2001) discovered MgB_2 with T_c a 39 K. Iron-based superconductors were first reported in 2008. La-Fe-As-O and Fe-Se were discovered to have T_c at 26 K (Kamihara et al., 2008) and 8 K (Hsu et al., 2008), respectively. Since then, enormous effort has been put forward in investigating the iron-based superconductors.

1.2 Fundamentals of Superconductivity

The materials must have these two unique properties in order to be classified as superconductors:

, =HUR UHVLVWLYLW\ !

Superconductivity is the phenomenon in which the electrical resistivity of certain material completely varnishes at a temperature below critical temperature, T_c as shown in Figure 1.3. For a non-superconductor such as metal, achieving zero resistivity at low temperature is impossible (Figure 1.3). As the temperature of a material decreases below T_c, the cooper pairs are able to move without resistance or loss. These highly correlated pairs of electrons are formed by electron-phonon interaction according to BCS theory (Cyrot and Pavuna, 1992). These phenomena will be further discussed in chapter 3. Once this flow is formed, the flow will move constantly and there is no scattering of electron pairs, therefore no resistance is produced. Superconductors can carry large amount of current with little or no loss of energy.

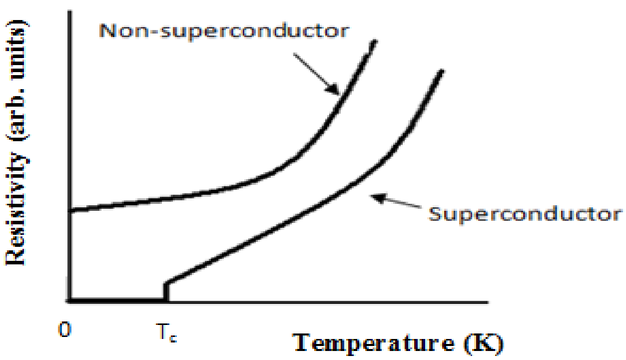

Figure 1.3. Temperature dependent resistivity for a normal metal and superconductor

II) No magnetic inductance (B_{inside} = 0)

Another unique property of superconductors is the absence of magnetic inductance. The magnetic inductance becomes zero inside a superconductor when it is cooled below Tc under a weak external magnetic field. This is also known as Meissner effect where magnetic flux will be excluded from inside of the superconductors as shown in Figure 1.4. Superconductivity will be destroyed if an applied magnetic field is larger than thermodynamic critical field, B_c in superconductors.

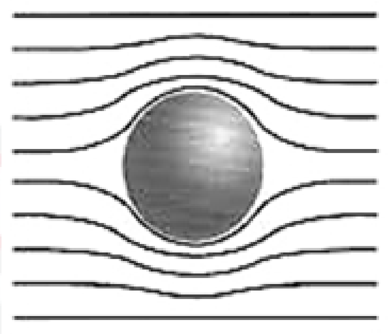

Figure 1.4. Expulsion of magnetic flux from the inside of a superconductor at temperature below T_c

1.3 Applications of Superconductors

The two unique properties of superconductors, namely zero resistivity and perfect diamagnetism have a major impact on the performance of many electronic devices. The miraculous property of superconductors which allows resistanceless flow of current at low temperature has led to a wide range of innovative technology application. The advancement of superconductors as the materials of the future is promising. Indeed, most of the applications as follow would not be viable without superconducting technology. Examples of the applications of superconductors are magnetically levitated trains (Maglev), magnetic resonance imaging (MRI), transformers, superconducting energy storage devices (SMES) and fault current limiter.

1.3.1 Magnetically Levitated Trains (Maglev)

The magnetically levitation transport system is considered as one of the most promising applications of superconductors. Maglev utilizes superconducting magnets as their guideway or track, offering a way for trains to float to their destination. By that, it can eliminate the friction between the static track allowing trains to travel at ultra-high speed at 400 Km/hr (Roslan, 2004). Superconductor magnets are essential in this application due to their lightweight and lower power requirements. Conventional electromagnets have high energy dissipation in the form of heat and physically much larger as compared to superconducting magnet. In present, maglev trains are operating in country like Japan, Germany and China.

1.3.2 Magnetic Resonance Imaging (MRI)

In biomagnetism technology, superconductors can be used as a life-saving tool. MRI has resulted in substantial benefits in medical field providing an enormous increase in diagnostic ability by showing the soft tissue features clearly which are not visible by using x-ray imaging. Moreover, MRI can eliminate the need of risky operation in order to diagnose the patients. MRIs use the small magnets inside the nuclei of the human body atoms to visualize what surrounds the organ i.e. brain by applying magnetic field. The nuclei of most of the atoms behave like a small spinning magnet. Superconducting magnet is used to generate high magnetic field to align the nuclei in this system. In particular, hydrogen atoms in human body can give a strong magnetic resonance signal. This can be used to map the soft tissue due to different water content in human body. The accuracy of the image in MRI is depends on the strength of the magnetic field. In general, magnetic field of 0.5 T or higher is used in MRI in order to obtain high resolution images (Roslan, 2004). MRIs are powerful and safe and have been routinely used in many hospitals as one of the widest applications of superconductor.

1.3.3 Transformers

Superconductor plays an effective role in power generation such as transformer. The superconducting wires used to make the transformer windings are more efficient compared to the conventional copper wires. With superconducting transformers, losses of about 30 % can be reduced. A lower weight of about 70 % and smaller volume size of about 50 % can be easily achieved as compared to the conventional transformers (Chen et al., 2004).

1.3.4 Superconducting Magnetic Energy Storage (SMES)

Electrical energy is one of the high demand forms of energy. The superconducting magnetic energy storage is used to store energy in the form of magnetic field in order to save energy. Direct current is used to flow through the superconducting coil which is placed in a coolant bath. The energy which is stored in the form of magnetic field can be restored when it is needed. The SMES system has 90 - 95 % efficiency and there is energy loss in this system due to the conversion from dc to ac source and heat loss during cooling (Roslan, 2004). So far, SMES is used for short energy storage to improve power quality due to the high cost of superconducting system and cooling.

1.3.5 Fault Current Limiters (FCL)

Fault current limiter (FCL), is a unique device that automatically limits or reduces current when it exceeds the predetermined value. Its working principles are similar to a S H U P D Q H Q W V X S H U F R Q G X F W L Q J I X V H V L Q F H D I W H U D automatically reset in order to protect the appliances. Inside the FCL, a circuit breaker is added to the superconducting element for a complete insulation when there is a fault current. Most of the high temperature superconductor fault current limiter (HTSFCL) exploits the sharp transition of superconductors from zero resistance, at normal currents, to a finite resistance at higher current densities (Paul and Chen, 1998). The current situation is not satisfying and faces a rising risk of high power surges results

IURP ³IDXOWV´ 7KHUHIRUH VXSHUFRQGXFWLQJ)&/ LV enables a better quality and security for electric energy.

1.4 Problem Statement

So far, polycrystalline $FeTe_{1-x}M_x$ (M = Se, S) has been synthesized under vacuum condition by sealing the materials inside evacuated quartz tube using solid state reaction, self-flux and Bridgeman methods (Sklyarova et al., 2014; Hacisalihoglu and Yanmaz., 2012; Mizuguchi et al., 2009a; Yeh et al., 2008). These methods are expensive and not feasible for large scale production. Hence, it is prospected that a simple and low cost method could be established without resort to vacuum condition. This offers tremendous opportunities to investigate the mechanism of superconductivity in this iron-chalcogenide compound, which has the simplest structure among the iron-based superconductors.

In this work, magnetization hysteresis loops and dielectric properties were measured in room temperature in order to study the potential of $FeTe_{1-x}M_x$ (M = Se, S) for applications at room temperature. Thus, the operation power required for cooling can be reduced. To date, there is no report on dielectric properties of $FeTe_{1-x}M_x$ (M = Se, S). However, there have been studies of dielectric properties in high-T_c oxide superconductors such as $Cu_{0.5}Ti_{0.5}Ba_2Ca_3(Cu_{-y}Cd_y)O_{-\delta}$ (Mumtaz et al., 2013), TiBaCaCuO (Cavdar et al., 2005) and $Bi_2Sr_2Ca_1Cu_2O_{\delta}$ (Cavdar et al., 2011). These samples were reported to show negative capacitance. The negative capacitance of these materials can be used to store information. The information can then be retrieved by applying a high electric field so as to bring the value of capacitance approaching zero (Khan et al., 2008).

Hence, this work is attempted to establish a simple and inexpensive method for synthesizing polycrystalline $FeTe_{1-x}M_x$ (M = Se, S) and to study their crystal structure, magnetic and dielectric properties. By doing so, these properties can be utilized to increase the usage of the materials for a wider range of electronic and electrical applications apart from investigating the mechanism of superconductivity.

1.5 Objective of the Research

The main purpose of the study is to synthesize polycrystalline $FeTe_{1-x}M_x$ (M = Se, S) in a simple way at ambient pressure over vacuum condition and to investigate the structural, magnetic, as well as dielectric properties of the materials. Hence, the objectives of this work are:

I. To establish a simple and low cost method in preparing $FeTe_{1-x}M_x$ (M = Se, S) without resort to vacuum condition.
II. To investigate and compare the phase formation, morphology and magnetic properties of FeTe by selective substitution at Te site with Se and S, respectively.
III. To study the change of dielectric properties of $FeTe_{1-x}M_x$ (M = Se, S) at room temperature by impedance measurement.

1.6 Scope of the Research

This work is about the study of structural, magnetic and dielectric properties of the polycrystalline bulk $FeTe_{1-x}M_x$ (M = Se, S) with the x ranging from 0.0 to 0.5 and 0.0 to 0.3, for Se and S substitution, respectively. The polycrystalline samples of $FeTe_{1-x}Se_x$ and $FeTe_{1-x}S_x$ were prepared by solid state reaction which is one of the most common methods in synthesizing various kinds of ceramics. The sintering of both the samples was done by sealing them inside stainless steel tube with both ends clamped in argon gas flow. Structural properties of the samples were checked by x-ray diffraction (XRD), scanning electron microscope (SEM) and energy-dispersive x-ray spectroscopy (EDX). Temperature dependence of magnetic moment was measured using the magnetic property measurement system (MPMS, Quantum Design) at the temperature range 2 - 300 K with an applied field of 10 Oe. No resistivity measurement was undertaken because of the low superconducting transition temperature, T_c (~ 9 - 15 K) of the samples below the cooling limit of the existing four point probe system (20 - 300 K) available at the Department of Physics, Faculty of Science, Universiti Putra Malaysia. The magnetization versus field of the samples was investigated using a vibrating sample magnetometer (VSM) at room temperature in the applied magnetic field ranges 0 kG - 10 kG. The dielectric properties were measured using an ac impedance analyzer at room temperature. Finally, the experimental data were analysed and discussed.

1.7 Thesis Outline

This thesis is consists of 6 chapters. Chapter 1 begins with a brief history of superconductivity and related applications. In addition, problem statement, research objective and research scope are also given here. Chapter 2 is a brief introduction of iron-based superconductors, FeSe, FeTe and FeS. Extensive literature review on $FeTe_{1-x}Se_x$ and $FeTe_{1-x}S_x$ superconductor are given in this chapter. Chapter 3 covers the fundamental phenomena of superconductivity and introduction to dielectric. The Meissner effect, types of superconductors and BCS theory are discussed. The dielectric properties and dielectric polarization are also explained in this chapter. Chapter 4 explains the experimental details throughout the project. The details of the characterization techniques and data collections are given here. Chapter 5 presents the overall experimental results obtained in this work with detailed discussion. Last but not least, this thesis is concluded in Chapter 6 together with recommendation for future work.

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