INSPIRING EXCELLENCE

Internship Report on

Permeability Measurements of $Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$ Using Impedance Analyzer

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DEPARTMENT OF MATHEMATICS AND NATURAL SCIENCES

By

AFRIN JAHAN SHARA

DEPARTMENT OF MATHEMATICS AND NATURAL SCIENCES

BRAC UNIVERSITY

STUDENT ID : 14115003

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CANDIDATE'S DECLARATION

It is hereby declared that this report or any part of it has not been submitted elsewhere for the award of any degree or diploma.

Afrin Jahan Shara

ID: 14115003

Certified By

Muhammad Lutfor Rahman

Senior Lecturer

Department of Mathematics and Natural Sciences

BRAC University

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ABSTRACT

This internship presents the work of $Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$ sample preparation and its permeability measurements at different sintering temperature using 6500B impedance analyzer. Firstly ''$Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$'' sample were being prepared by using different materials according to their stoichiometric ratio using an electronic balance. The sample were then hand milled for 5 hours to gain a homogenous distribution and to remove impurities as much as possible by using an agate pestle and mortar. After that the sample was calcined at 950ºC using a furnace to eradicate water, oxides. To achieve a more pure and uniform sample, it was hand milled for more 2 hours until the powder is finely mixed. Afterwards the sample was made into three disk and ring shapes for three sintering temperatures (T_s). Once the sintering is finished the disks and rings were ready to be analyzed. The ring shaped samples were examined by the impedance analyzer (Wayne Kerr 6500B Precision Impedance Analyzer) to calculate the permeability of the samples, the frequency to which the sample can operate.

Introduction

All sorts of scientific studies are done to improve and better the technologies we use and invent more advanced technologies that we will use in the near future. Especially in the field of science theoretical and practical knowledge both come hand in hand-one without the other is not sustainable in the world of science. The purpose of this internship is to provide that practical knowledge of how we are relating and applying polycrystalline ferrites to our everyday life. This internship fully focuses on the structure of $Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$ and the change in its magnetic properties in order to enhance their applications.

Overview of $Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$ ferrite

Ferrites are ceramic materials composed of iron oxide and other metallic elements that are different from ferromagnetic materials like iron, cobalt, nickel. It shows a form of magnetism called ferrimagnetism. Ferrites are hard, brittle, and iron-containing. They show such magnetic field that is less strong than that of a ferromagnetic material but has high permeability and high resistivity which make it more desirable to study with. A lot of works have been done on ferrites especially on Ni-Cu-Zn ferrites due to its properties like high permeability, low loss factor, high wire resistance, low switching coefficient and controlled device force etc. It is been a topic of interest since decades both to scientists and manufacturers for its magnetic and electrical properties and great applications for high frequency systems. This ferrite has a spinel structure of the formula AB_2O_4.The compound $Ni_{0.4}Cu_{0.15}Zn_{0.45}$ as a whole represents A-site which is the tetrahedral site in the spinel structure whereas Fe represents B-site, the octahedral site of the crystal. The work is about to observe what happens to the permeability of the ferrite at different sintering temperatures.

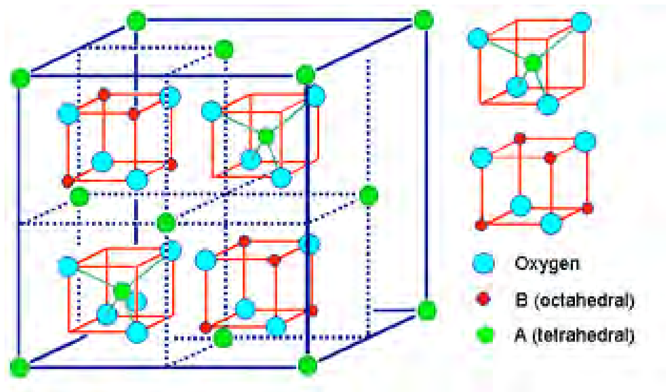


Fig 01: Spinel Structure (A-site and B-site)

Sample preparation

The whole procedure has been done by solid state reaction method. "$Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$" sample is made from different oxides of the required materials that are highly pure. These materials had been weighed according to the stoichiometric ratio that were already calculated for 15 gram sample. Later on they were hand milled for 5 hours and calcined for 5 hours to drive off impurity and oxides.

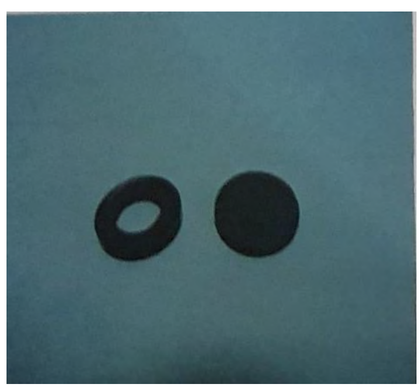


Fig 02: ring and disk for $Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$

Once the samples were cooled down, they were again hand milled for more 2 hours. Three disks and rings were made at a pressure of 6000 Pa. Lastly they were sintered at 1100, 1150 and 1200 degree Celsius for 5 hours to perform various analyses like X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) etc. Disks are used for XRD, SEM and EDS experiments and rings are used for permeability measurements.

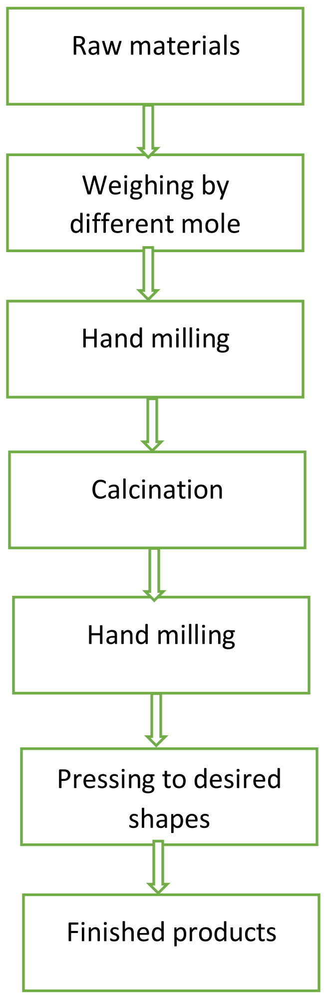


Fig 03: Flow chart of the stages in preparation of spinel ferrite

Stoichiometric Ratio Calculation

Stoichiometric ratio is the calculation of relative quantities of reactants and products in chemical reactions. It is founded on the law of conservation of mass where the total mass of the reactants equals the total mass of the products leading to the insight that the relations among quantities of reactants and products typically form a ratio of positive integers. This means that if the amounts of the separate reactants are known, then the amount of the product can be calculated. Conversely, if one reactant has a known quantity and the quantity of product can be empirically determined, then the amount of the other reactants can also be calculated. For the samples used in this work, each was of a total of 15 gram and each pellet and ring were made of 1 gram. The amount used for sample preparation is given below.

Table 01: atomic mass of the compounds

Raw materials	Ni (g/mole)	Cu (g/mole)	Zn (g/mole)	Fe (g/mole)	O (g/mole)	Total (g/mole)
NiO	58.6934				15.9994	74.6928
Cu_2O		63.546			15.9994	143.0914
ZnO			65.38		15.9994	81.3794
Fe_2O_3				55.845	15.9994	159.6882

Table02: total mass of the sample

Composition	Mass of the sample (g/mole)
$Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$	58.6934*0.4+63.546*0.15+65.38*0.45+ 55.845*2+15.9994*4=238.11786

Table 03: Calculation for the need of raw materials

Composition	Need of	Amount (g/mole)
$Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$	NiO	74.6928*0.4*15/238.11786 =1.8821
	Cu_2O	143.0914*0.15*15/238.11786 =1.3521
	ZnO	81.3794*0.45*15/238.11786 =2.3069
	Fe_2O_3	159.6882*15/238.11786 =10.0594

Working Principle of 6500B Precision Impedance Analyzer

1. Power on-After connecting the instrument with correct AC power supply, pressing the POWER button will switch on the instrument. The power indicator will display light, the bias indicator will flash. After running the startup routine, the instrument will show the analysis mode.

2. Control panel-The instrument can be controlled in three different ways. Firstly it can be control using the LCD display touch panel interface, or using the front control panel. Other way is to attach extra key board and mouse to control the functions of the instruments. Using the LCD display or front control panel or external control panel of giving command, one can select instrument modes, functions and measurement parameters.

3. Power off-The power can be switched off at any time without damaging the instrument however it is recommended that, the power should be turned off when it is in quiescent sate rather than when it is running a routine.

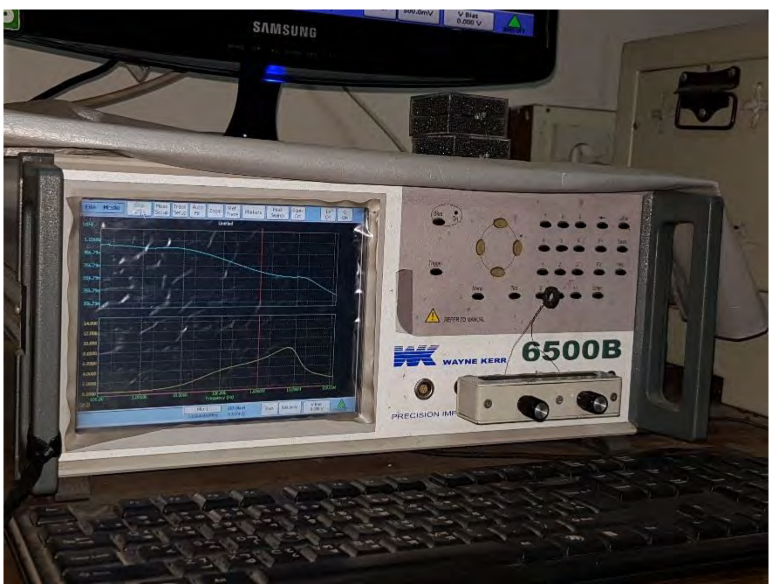


Fig 04: 6500B Precision Impedance Analyzer

The control board consists of major functional buttons with which every routine can be performed by the impedance analyzer. Their functions are given below-

Fig 05: Control Board of 6500B Precision Impedance Analyzer

a) Power button is to switch on/off the instrument.

b) Navigation keys are used to move cursor in the display screen like a mouse does.

c) BIAS and TRIGGER button both work as the measurement keys. Trigger key initiates a routine. If it is held, the analyzer will continue to measure until it is released. Bias key is used to apply DC voltage to the AC measurement signals if a DC bias option is fitted.

d) Control keys consist of the ‘Save’, ‘Local’, ‘Help’, ‘Enter’, ‘Back key’, ‘Tab’ and ‘Menu’ buttons. MENU displays or hides file, mode or display menu items in Meter or Analysis mode. TAB selects the next menu, parameter or option. Function keys (F1, F2) specify menu functions. ENTER confirms data input. SAVE button saves the instrument setup. HELP displays guideline regarding software and instrument types. LOCAL restores control to the front panel when external control is activated.

e) Data entry keys allow to input data.

f) Measurement terminals are to connect component fixture. Fixture interface is for further expansion if necessary.

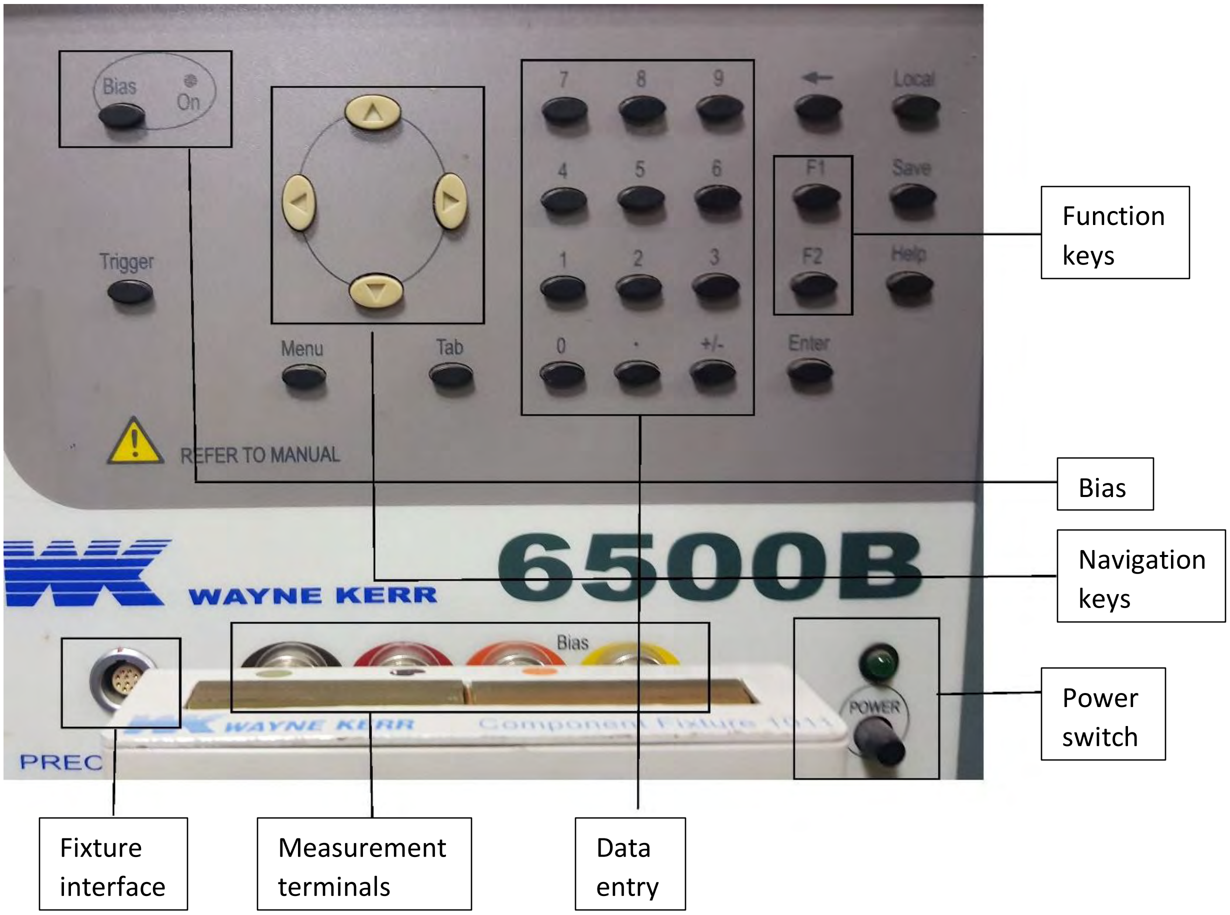


Fig 06: Keys to control & command

Measuring Complex Initial Permeability Using 6500B Precision Impedance Analyzer

Before proceeding to the procedures for preparing rings for permeability measurements, it is important to know what permeability is and why it is important. For ferrites and small scale measurements, the term 'complex initial permeability' is used instead of permeability. Permeability is a quantity that defines the change in self-inductance of a coil in the presence of a magnetic core. The core is taken as a toroid shape to avoid demagnetizing effects.

Complex initial permeability is given by

$$Z = R + jX = j\omega L_o \mu = j\omega L_o(\mu^{/} - j\mu^{//})$$

Where the resistive part is,

$$R = \omega L_o \mu^{//}$$

And the reactive part is,

$$X = \omega L_o \mu^{/}$$

Here, L_o is the inductance of the winding coil in air that is without loss.

μ is the permeability of the magnetic core

On the above expressions, the real part and the imaginary part are calculated by $\mu_i^{/} = L_s/L_o$ and $\mu_i^{//} = \mu_i^{/} tan\delta$, where L_s is the self-inductance of the sample core sample core and $L_o = \mu_o N^2 S/\pi \bar{d}$ derived geometrically.

N is the number of turns of the coil (N = 4), S is the area of cross section of the toroidal sample as given below:

$$S = d \times h,$$

Where $$d = \frac{d_2 - d_1}{2},$$

$d_1 =$ Inner diameter,

$d_2 =$ Outer diameter,

$h =$ Height

And $\bar{d}$ is the mean diameter of the toroidal sample as given below:

$$\bar{d} = \frac{d_1 + d_2}{2}$$

The relative quality factor is determined from the ratio $\frac{\mu_i^{/}}{\tan\delta}$.

Once the toroid shapes are prepared using the samples, they are being sintered. In this research three rings for value of x=0.00 are taken and sintered at 1100, 1150 and 1200^{0}C. After they are cooled down in the furnace, sand papers of different grits (in this case P600, P800 and P1200) have been used to smooth the surface of the rings by polishing them. They have to be handled with care because the rings are very brittle. So if they fall down, they can be broken easily. The next step is to measure the diameter of inner and outer radius and the height of the samples which is later used for the calculation of L_o..

Table 04: Core inductance, L_o calculation

Composition	T_s (^{0}C)	$\bar{d}$ (m)	h(m)	S(m^2)	N	L_o(nH)
$Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$	1100	$.4745*10^{-2}$	$.1976*10^{-2}$	$.027268*10^{-4}$	4	3.68
	1150	$.4702*10^{-2}$	$.2046*10^{-2}$	$.028388*10^{-4}$	4	3.86
	1200	$.4725*10^{-2}$	$.2718*10^{-2}$	$.037100*10^{-4}$	4	4.19

After the calculation of L_o, very thin low resistive copper wires are used to twirl it around the rings and the end points of each wires must be scratched off to uncover the conductive portion of the wire to conduct AC current. Permeability measurements have been done using the 6500B precision impedance analyzer.

The impedance analyzer provides a wide range and variety of options to analyze different tasks and applications with high accuracy as well as frequency. It comes along with a 1011 component fixture with four front panel connectors to ensure great performance while measuring several of components and devices. This component fixture is connected to the four terminals of the analyzer. Two knobs are used to tighten the connection when the wires of the rings are put in between the components.

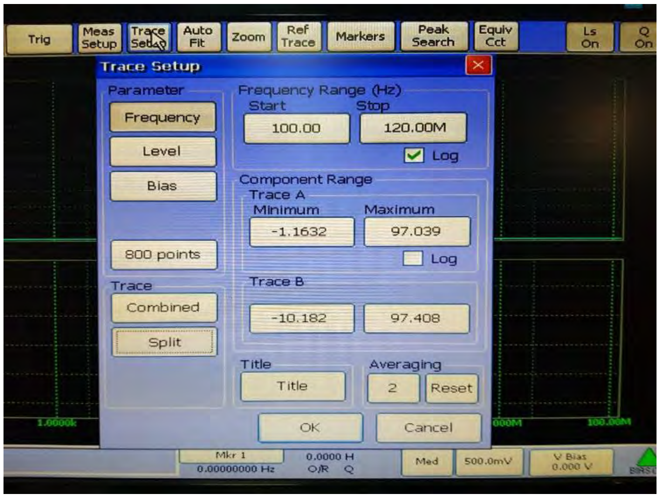
Fig 07: 1011 Component Fixture

After completing connection and starting the analyzer machine the following steps are undertaken to measure permeability as given in Fig 06.

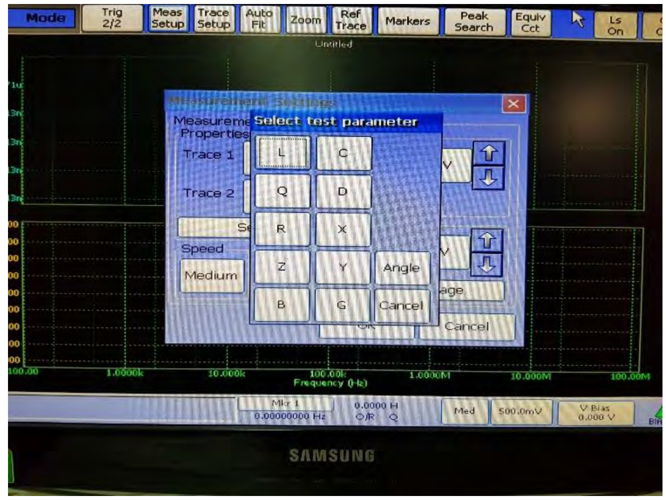
1. Opening up ‘Measurement Setup’ dialogue box to set parameters for L and Q and the voltage to 500mV.

2. Opening up ‘Trace Setup’ dialogue box from the menu bar and resetting all the variables.

3. Setting up frequency range from 100Hz to 120 MHz in log scale.



(a)



(b)

Fig 08: (a) Trace setup and (b) Measurement setup of the Impedance Analyzer

4. Measurement is taken for 400 points and the two parameters are set as ‘split’ so that they can be traced separately. These setups can be selected in the same dialogue box.

5. Speed range is selected as ‘medium’.

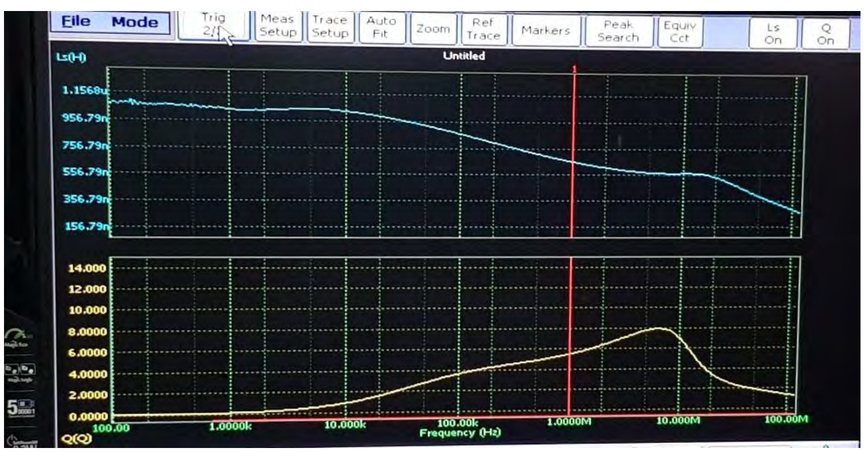


Fig 09: Frequency vs L and Q graphs on the same screen

6. When the setups are done, trigger button is pushed to start the operation.

8. Once the averaging is done, an auto fit option is there on the menu bar to fit the graph accordingly in the screen with given range of frequency to provide a better comparison between the parameters as given in Fig 07.

9. Repeating the same steps every time for each ring's permeability measurements.

10. Lastly, the results of each sample must be saved by using the 'save file' option or icon on the menu bar.

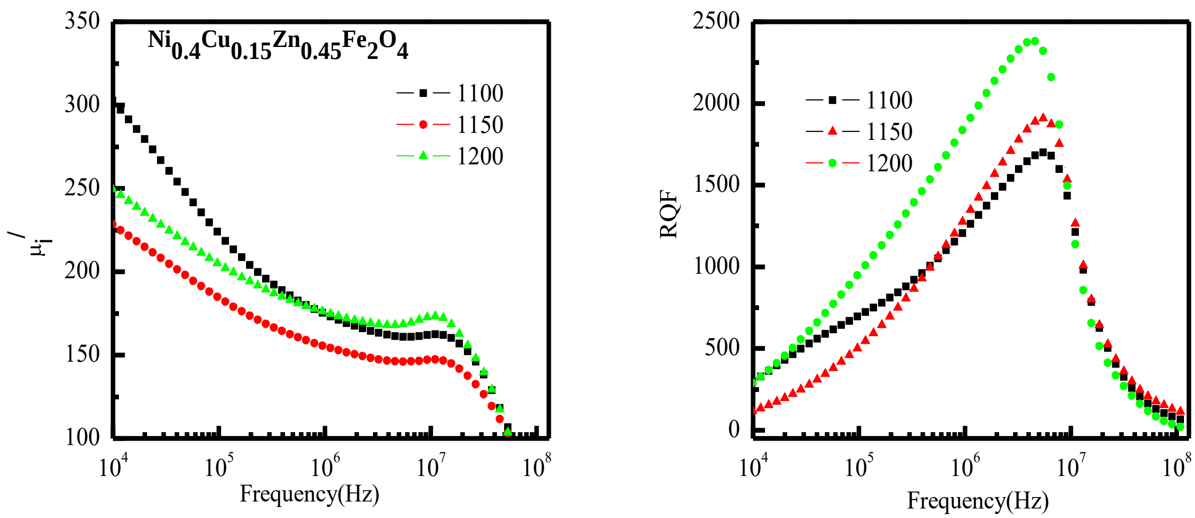


Fig 10: The real part and relative quality factor of permeability spectrum for $Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$

Using the results provided by the impendence analyzer, the real and imaginary part of permeability, quality factor and loss factor can achieved which is already mentioned above. Fig 09 shows the graphs of real part and relative quality factor of permeability spectrum for $Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$.

Advantages of 6500B Impedance Analyzer

1. Different tasks perform and applications with 4 connecting terminals

2. Fast testing of components at frequencies up to 120MHz.

3. Inductance and quality factor accuracy is ± 0.05%.

4. Provides graphical Sweep of two measured parameters and displays clear, large and colored.

5. AC drive levels given up to 1v and 20mA.

6. External projector or monitor, keyboard and mouse can be connected.

7. Data storage is available using USB port.

8. Temperature dependent electrical and magnetic properties can be determined.

Hazard Summary and Precautions

Solid state experiments always involve chemical substances that can affect our health if not handled carefully.

1. Zinc, copper, iron all of them are metal and their oxides can cause metal fume fever when breathed in.

2. Repeated exposure to iron oxide dust or fume can cause pneumoconiosis with cough, shortness of breath.

3. Hydrochloric acid should be handled with care when cleansing mortar and pestle otherwise it can cause serious burn on the skin.

4. During calcination and sintering, crucibles should be handled with tongs otherwise hands can get burned.

To avoid such risks one can follow the measures. It effectively reduces errors and threats while doing experiments.

1. Use nose mask, safety googles.

2. Use of gloves and tongs.

3. Compounds should be kept in sealed jar to avoid contact with air, loss of the compound and getting expose to environment.

4. Using every instruments like mortar, spatula, jar, foils –they must be cleaned by spirit or acetone to ensure a decontamination of the each and every apparatus.

6. Before weighing, fans must be turned off otherwise atmospheric pressure can cause errors to the weight of the compounds.

7. Disks and rings must be made handled with care, otherwise can be broken.

Conclusion

This internship has helped me with not only applying my theoretical knowledge to practical life but it has also developed confidence in me and provided experience about dealing with an experimental science lab environment. For this work in Bangladesh University of Engineering and Technology I got know about procedures and systems and also came to experience different working methods of experiments with professionals working there. I got to know a life that works beyond academic studies and also that helped me to improve my way of approach towards research and scientific studies.

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