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Internship on $(Ni_{0.4}Cu_{0.15}Zn_{0.45})_{1-x}Mn_xFe_2O_4$ Sample Preparation and Calcination

An Internship Report Submitted To
the Department of Mathematics and Natural Sciences, BRAC University,
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Bachelors of Applied Physics and Electronics

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CANDIDATE'S DECLARATION

It is hereby declared that this report titled "Internship on $(Ni_{0.4}Cu_{0.15}Zn_{0.45})_{1-x}Mn_xFe_2O_4$ Sample Preparation and Calcination" is submitted to the Department of Mathematics and Natural Sciences of BRAC University in partial fulfilment of the requirements for the degree of Bachelor of Science in Applied Physics and Electronics. This report is the very own work of my own and has not been submitted elsewhere. Every work that has been used as reference has been cited properly.

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The Author

Zarrin Tasnim Mridula

Abstract

The sole purpose of this internship is to provide us hands on experience on how to measure, prepare and characterise a sample of the $(Ni_{0.4}Cu_{0.15}Zn_{0.45})_{1-x}Mn_xFe_2O_4$ ferrites (x=0.3, x=0.45) which was further hand milled before and after calcination to achieve a homogenous distribution. In order to prepare the sample, the stoichiometric ratio was found, and then the exact amount of each required compound was measured separately using an AX120 electronic balance as it provides a high precision of 10 micrograms. It has a full range tare to allow unladed weight measurement. It was hand milled with a pestle and mortar and finally calcined at 950°C and sintered at 1100°C in air for 5 hours at 1100, 1150 and 1200^0C using a Nabertherm p330 to drive off water, carbon dioxide, and other volatile constituents and to oxidise the whole composite. Structural and magnetic properties of were investigated and it was prepared by solid state reaction methods.

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Introduction

The sole purpose of this internship is to provide us hands on practical experience in experimental solid state physics laboratories. Once we step out of our academic atmosphere it very important for us to implement what we have learned so far, or else the basis of our knowledge will have no value at all. We shall not only fall backwards but also lack in understanding the overall impact of our learnings in the ever changing world of science. In the period of the last few months, I have been given a very vital opportunity to have some valuable experiences in the Solid State Physics Laboratory in Bangladesh University of Engineering and Technology.

The target of this report is to analyse and understand the very core concepts of sample sample preparation using solid state techniques, how characterisations of these materials are done and how to improve the procedures for further progress in technology.

Laboratory Facilities Available in the Department of Physics in Bangladesh University of Engineering and Technology:

- Ferroelectric measurements
- UV visible Spectrometer
- High power Xenon Lamp for Photocatalytic experiment
- Gas Chromatograph
- High Temparature Inert Gas Furnace
- Close Cycle Cryocooler
- Torque Magnetometer
- Vibrating Sample Magnetometer
- Plasma polymerization

- Thin Film Deposition
- High Temperature Furnace
- Hydraulic Pressing Machine
- Optical Polarising Microscope
- Crystal Growth
- Spray Pyrolysis Deposition Unit
- Modelling and Simulation Facilities
- Scanning Electron Microscope (SEM) & X-ray Diffractometer

Solid State Physics Laboratory's Research Areas:

This part of the institution has their fair activity set mostly on different fields of Material Science like :

- Multiferroics
- Nanocrystalline Ferrites
- Nanocrystalline Alloys and
- Colossal Magnetoresistance

Example of Specific Research Projects :

- Preparation and characterization of nanostructured spinel type ferrites
- Preparation and characterization of perovskite type material
- Preparation and characterization of nanocrystalline alloys
- Manganites for colossal magneto resistance/ giant magneto resistance study

- Ferroelectric and dielectric (high k) material
- Multiferroic materials

Sample Preparation of $(Ni_{0.4}Cu_{0.15}Zn_{0.45})_{1-x}Mn_xFe_2O_4$

In this present work, the samples of Mn^{2+} doped Ni-Cu-Zn ferrite are synthesized and investigated. The samples are: $(Ni_{0.4}Cu_{0.15}Zn_{0.45})_{1-x}Mn_xFe_2O_4$ (with x=0.30, 0.45) which is a spinel ferrite. Spinel ferrites have the general molecular formula of $(A^{2+})[B_2^{3+}]\ O_4^{2-}$. A ferrite is a ceramic-like material with magnetic properties that are useful in many types of electronic devices. Ferrites are hard, brittle, iron containing and generally grey or black. They are polycrystalline, i.e., made up of a large number of small crystals. They are composed of iron oxide and one or more other metals in chemical combination. Ferrites exhibit a form of magnetism named ferrimagnetism, which is distinguished from the ferromagnetism of materials like iron, cobalt, and nickel. In ferrites, the magnetic moments of constituent atoms align themselves in two or three different directions. A partial cancellation of the magnetic field results in this, and the ferrite is left with an overall magnetic field that is less strong than that of a ferromagnetic material.

The following flowchart below shows the different stages in preparation of spinel ferrite.

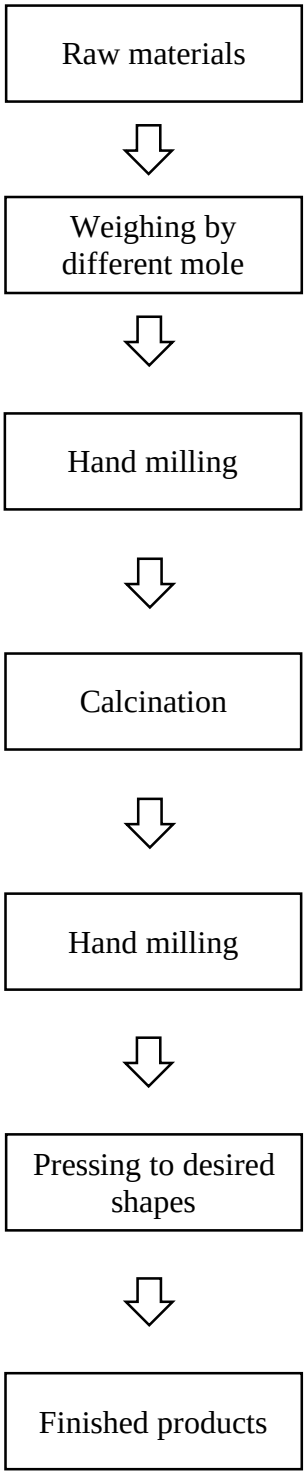

Figure 1: Flowchart of the stages in preparation of spinel ferrite

Stoichiometric Ratio Calculation

The following tables show the calculation of different amount of raw materials that was taken to make the samples. For the samples used in this work, each was of a total of 15 grams and each pellet and ring were made of 1 gram.

Table 1.0: atomic mass of the compounds

Raw materials	**Ni (g/mole)**	**Cu (g/mole)**	**Zn (g/mole)**	**Mn (g/mole)**	**C (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
$MnCO_3$				54.938	12.0107		15.9994	114.9469
Fe_2O_3						55.845	15.9994	159.6882

Table 1.1: total molecular mass of the sample

X	Composition	Mass of the sample (g/mole)
0.3	$Ni_{0.4}Cu_{0.15}Zn_{0.45}Fe_2O_4$	(58.6934*0.4) +(63.546*0.15) +(65.38*0.45) + (55.845*2)+(15.9994*4)=238.11786

0.45	$Ni_{0.22}Cu_{0.0825}Zn_{0.2475}Mn_{0.45}Fe_2O_4$	58.6934*0.22+63.546*0.0825+65.38*0.2475+54.938*0.45+55.84*2+15.9994*4=234.746343

Table 1.2: 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
	$MnCO_3$	0
	Fe_2O_3	159.6882*15/238.11786 =10.0594
$Ni_{0.22}Cu_{0.0825}Zn_{0.2475}Mn_{0.45}Fe_2O_4$	NiO	74.6928*0.22*15/234.746343 =1.0500
	Cu_2O	143.0914*0.0825*15/234.746343 =0.7543
	ZnO	81.3794*0.2475*15/234.746343 =1.28720
	$MnCO_3$	114.9469*0.45*15/234.746343 =3.3052

	Fe_2O_3	159.6882*15/234. 746343 =10.2039

Measuring the required amount of raw materials using AX120 electronic balance

For setting the measuring mode on the power key of the balance is pressed. When zero is displayed the glass door is opened and aluminum foil is placed inside. It is very important to clean the foil with acetone to avoid any sort of contamination. All fans should be kept turned off to avoid any sort of misreading.

When the arrow appears on the scale suggesting the total weight of the foil has been registered the TAR button is pressed to nullify it. Small amount of each compounds were then scooped onto the foil using a clean spatula and after that it is to be waited till the reading matched up to the fourth decimal place of the calculated required amount. After repeating the process for all the five samples they were finally put in a jar which was cleaned beforehand with acetone for hand milling.

Figure 2: AX120 Electronic Balance

Figure 3: Control Fields and Display of AX120 Electronic Balance

Figure 4: Ferrite Sample

Advantages of AX120 Electronic Balance:

- Extremely high precision balance, around 10 microgram
- Draftshield for preventing for preventing the influence of convection and other factors
- Can check weighing, parts counting, and percent weighing
- Over checking protection
- Full range tare

Some procedures followed for hand milling:

- Thorough grinding is necessary to achieve a homogenous mixture of reactants.
- The number of crystallites in contact may be increased by pelletizing the powders using a hydraulic press.
- The reaction mixture is typically removed and reground to bring fresh surfaces in contact, which speeds up the reaction.
- Furnaces use resistance heating with metal, SiC, or MoSi2 heating elements
- Containers for the reaction (crucibles) must be able to withstand high temperatures and be sufficiently inert to the reactants. Common crucibles are silica (to 1430 K), alumina (to 2200 K), zirconia (to 2300 K), or magnesia (2700 K). Platinum (m.p. 2045 K) and silver (m.p. 1235 K) are also used for some reactions.
- Reaction times are sometimes hours, but may range into several days or weeks for a complete reaction, with intermediate grinding.

Calcination of the sample using Nabertherm P330

Calcination is a process of subjecting a substance to the action of heat, but without fusion, for the purpose of causing some change in its physical or chemical constitution. For Calcination, its is very important to keep the temparataure used below the melting or fusing point.

Calcination is an important step because hand milling the composite cannot cause completion of the chemical fusion in between the different compounds. Calcinating the sample ensures total sealing of the composite and wards off all sorts of impurities.

The objectives of Calcination are usually:

- To drive off water
- To drive off carbon dioxide or other volatile constituents
- To oxidize a part or the whole of the substance

To start the process of Calcination, crucibles were first cleaned with detergent followed by distilled water. It was then further cleaned with acetone, left to dry for a while, filled with the composite and placed in the furnace to be calcined at 950^0C for five hours. The crucibles in question were made of alumina. This is because alumina is the hardest of the oxides, has great heat conductivity, large thermal expansion co-efficient, low impact resistance and is chemically stable which means it will not react with the composite. The temperature was gradually raised in increments of 10^0C for 5 hours until 950^0C was reached. The final product was again hand milled to get rid of clusters and turned into powder thereby completing the preparation of the ferrite sample.

The calcined powders were granulated using polyvinyl acid a binder and pressed in to desired toroid and pellet shapes. In the final stage they were sintered at various temperatures from 1050^0C to 1200^0C in air for 5 hours. The temperature ramps for sintering are 5^0C /min for heating and 10^0C /min for cooling.

Nabertherm P330 Operation

First the power is switched to “I” Position. The controller first displays the controller type and version number and then the temperature display. If the temperature is displayed, the controller is ready to operate.

Figure 5: Nabertherm P330

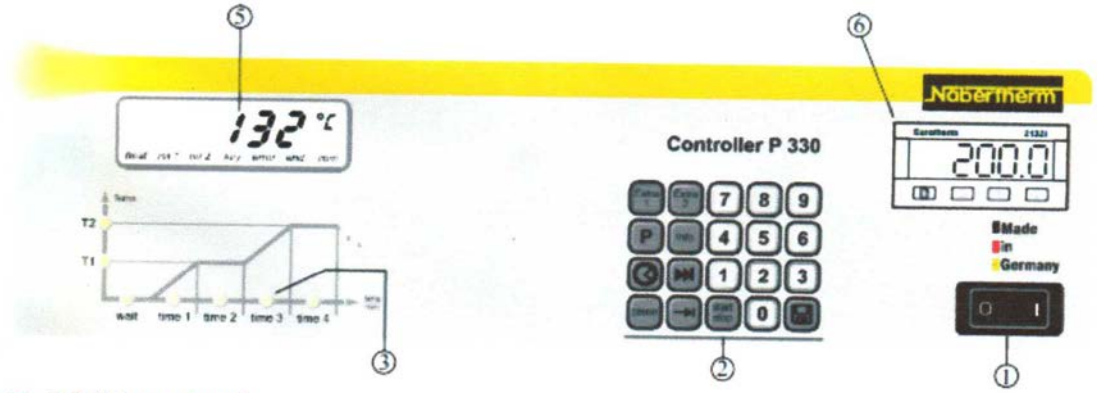

Fig. 2: P 330 control panel

1 = Power switch
2 = Keyboard block
3 = Program LED
4 = - - -
5 = Display
6 = Over-temperature limit controller (optional)

Figure 6: Control Fields and Display of Nabertherm P330)

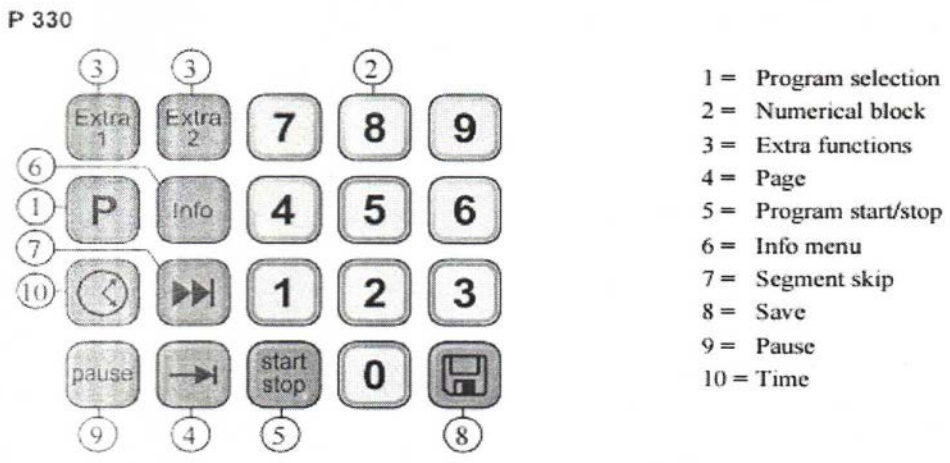

Figure 7: P330 Keyboard Block

Setting or changing program/waiting time

For the automatic operation of the furnace, before starting the controller, a temperature characteristic must be configured which describes the desired temperature behavior. This is known as the heating program.

Each bearing program has two heating ramps, one holding time, and one cooling ramp.

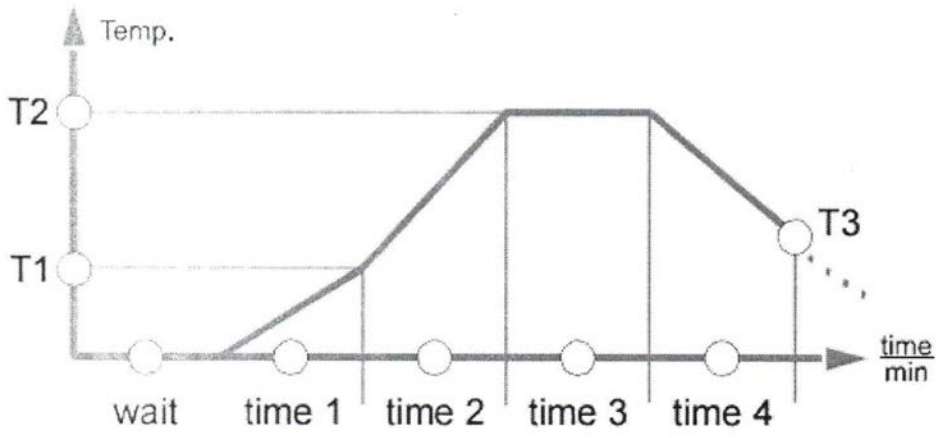

Figure 8: Program Graphic

- In the ramps a segment temperature “T” and a segment time, “Time 1” and “Time 2” define a linear temperature increase which is slow heating.
- In the holding time, “time 3” determines how long the temperature value configured in “T 2” should be held.
- In the cooling time, the natural cooling can be slowed can be sowed using the rate set in “T 3” and “time 4”

The page key is used to:

- Set temperature values with $^0C/^0F$
- Set time specifications with hr:min
- Set gradient specifications with ^0C/hr:min or ^0F/hr:min

To turn it off main switch is switched at position “O”

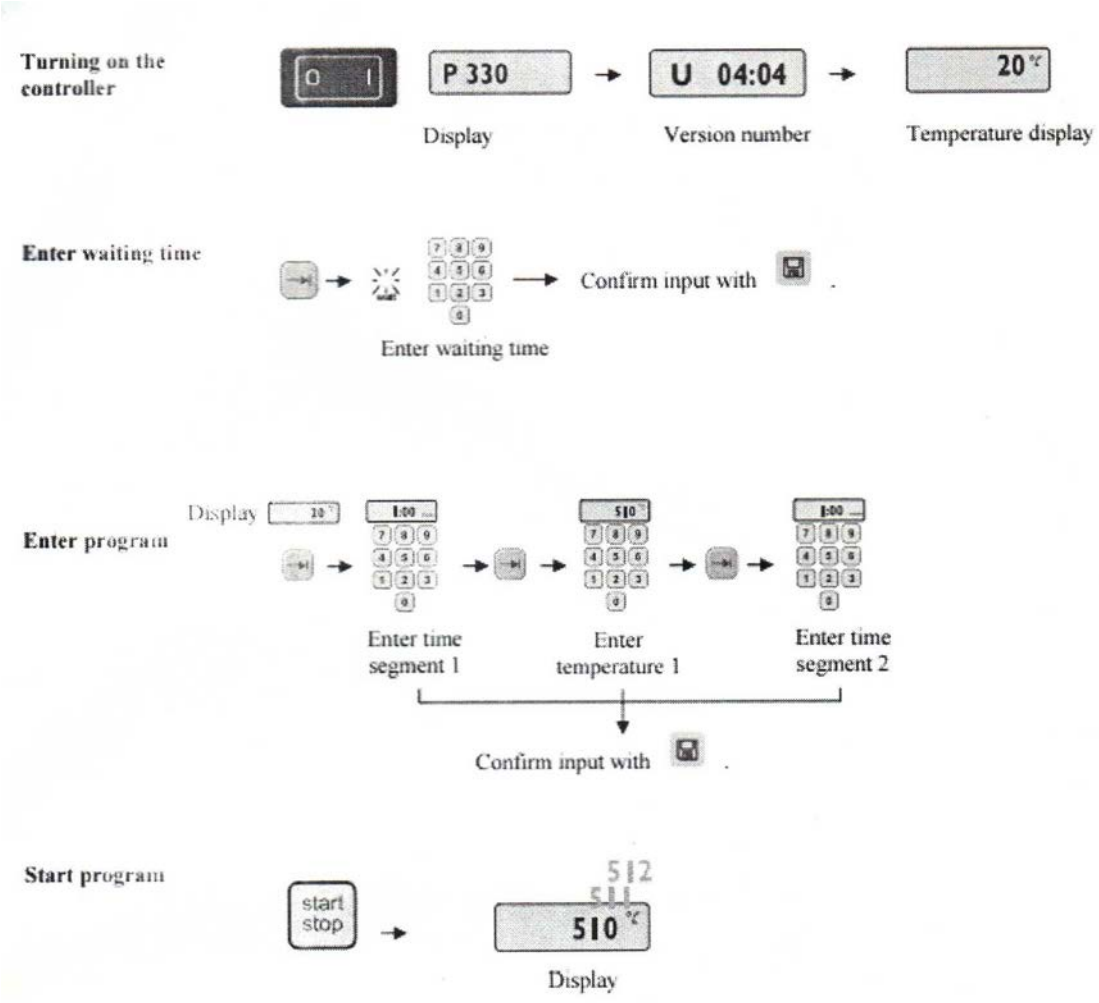

Figure 9: Operating Instructions Summary diagram

Hazard Summary and Safety Precautions:

The process mentioned earlier each have their own list of both acute and diminutive hazards which must be kept in mind to steer clear of accidents. The list includes:

- Exposure to zinc oxide, copper oxide and ferric oxide can cause metal fume fever when breathed in
- Gadolinium oxide can cause serious eye irritation
- Nickel oxide is a carcinogen and may cause skin allergy. It can also affect once inhaled.
- Gas released during calcination may be harmful as well
- There is a risk of getting burned if the crucibles after calcination are not handled using tongs

To carry out all the procedures risk and error free the following steps must be followed:

- Usage of safety goggles
- Usage of nose mask
- Usage of gloves
- Usage of tongs
- Turning off the fans while measuring the weight
- Keeping the compounds in sealed jar to avoid contact with humidity as this might change their weight. So while measuring we will always get less amount of the amount of the original compounds and the overall composition will be faulty
- Cleaning the jars, spatula, foil and every other thing that is supposed to come in contact with the compounds to avoid contamination
- Avoid carrying out the process in dusty rooms to avoid contamination

- Finally, once the sample has been prepared X-ray diffraction can be used to determine the desired crystal structure has been achieved.

Conclusion

The internship at Bangladesh University of Engineering and Technology's solid state physics laboratory has allowed me to learn new skills and added to my knowledge base while making me feel confident in my abilities. Due to this experience I now know how to apply some of the ideas learned in my academic career as it provides a bridge between academic and the professional world. It has made me gain some insight on how the world beyond the book's pages actually function or what kind of approach one must adopt to perpetuate research. The target of this report is to analyse and understand the very core concepts of sample sample preparation using solid state techniques, how characterisations of these materials are done and how to improve the procedures for further progress in technology.

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