ISSN NO. 2320-5407

Journal homepage: http://www.journalijar.com

INTERNATIONAL JOURNAL OF ADVANCED RESEARCH

RESEARCH ARTICLE

Thermal Analysis of Titanium Doped Manganese Zinc Ferrite, $Mn_{0.8+x}Zn_{0.2}Ti_xFe_{2-2x}O_4$ with x=0.10, 0.15, 0.20

Satheesh D J[1], Reenu Jacob[3],Vasudevan Nair N[2], Jayakumari Isac[3*]

1.Centre for Condensed Matter, Department of Physics, CMS college, Kottayam, India
2.Department of Physics, M. G College, Trivandrum, Kerala.
3.Department of Physics, CMS college, Kottayam,686001,

Manuscript Info

Manuscript History:

Received: 15 July 2015
Final Accepted: 22 August 2015
Published Online: September 2015

Key words:

MnZnTiFeO, Thermogravimetric analysis(TGA), DSC, Differential Thermal Analysis(DTA)

Corresponding Author

Satheesh D J

Abstract

Mn0.8+x Zn0.2TixFe2-2xO4 with x=0.10, 0.15, 0.20 were prepared by conventional solid state reaction technique. Final sintering was done at 1150^0C for 20 hours followed by slow cooling to room temperature. To acquire desired homogeneity and phase formation samples were subjected to calcination. To establish the elemental composition EDX analysis was done. TGA, DSC and DTA were used to study the thermal behaviour of the samples.

INTRODUCTION

Ceramics generally can withstand very high temperatures such as temperatures that range from 1000˚C to 1600˚C (1800˚F to 3000˚F). Conventional solid state reaction method is a common and effective way to fabricate modern ceramics [1]. The need for high resistivity ferrites led to the synthesis of various ferrites. It is found that the additions like Ti, Zn etc considerably change the thermal and electrical properties. Manganese Zinc Ferrite in the spinel structure is a low cost material which is generally useful for microwave devices and memory core applications.

In the present work the authors study the thermal behaviour of **MnZnTiFeO** compound. Thermo Gravimetric Analysis (TGA), DSC and Differential Thermal Analysis(DTA) were used to study the thermodynamical behavior [2-4]. One can get information regarding the long-term thermal of such systems [5]. The EDX spectrum gave the information of the elemental composition of the material [6].

Material and Methods

1. Preparation of the Samples

Titanium doped ferrites with the chemical formula **MnZnTiFeO** were prepared by the conventional solid state reaction method according to their molecular formula in stotiometric proportions. For this AR grade MnO, ZnO, TiO_2 and Fe_2O_3 were used as raw materials. The required powders were mixed mechanically and grounded for two hours using an agate mortar and pestle. The presintered ferrites were again grounded and then pressed into pellets. Final sintering was done at 11500C for 20 hours. Chopper stabilized amplifier is used as temperature control system within the furnace. Temperature is controlled by a Platinum-Rhodium thermocouple

within the furnace. Control of temperature is often necessary to ensure that the desired crystalline phase is formed with optimum particle size [7].

2.TGA Analysis

Thermogravimetric analyses, the mass change of a sample as a function of temperature in the scanning mode or as a function of time was done (Fig.1. a-c) . Factors such as sample mass, volume and physical form, the shape and nature of the sample holder, the nature and pressure of the atmosphere in the sample chamber, and the scanning rate have significant influences on the characteristics of the recorded TG curve.

3.Thermal Analysis

Thermogravimetric Analysis (TG) determines the weight changes of a sample, whereas Differential Thermal Analysis (DTA) measures changes in temperature between a sample and a reference, as a function of temperature or time.TG and DTA curves of the sample were recorded using Perkin Elmer, Diamond TG/DTA with Flexible axial and radial view instrument, with high concentration capabilities. TGA is plotted in figure 1 (a), (b) & (c). DSC is plotted in figure 2 and DTA/DTG is plotted in figure 3. Figure illustrates the typical TGA, DSC and DTA curves obtained during heating up of the ceramic sample **MnZnTiFeO** up to 720oC. Endothermic peaks accompanied by weight loss at 650-7500C can be observed.

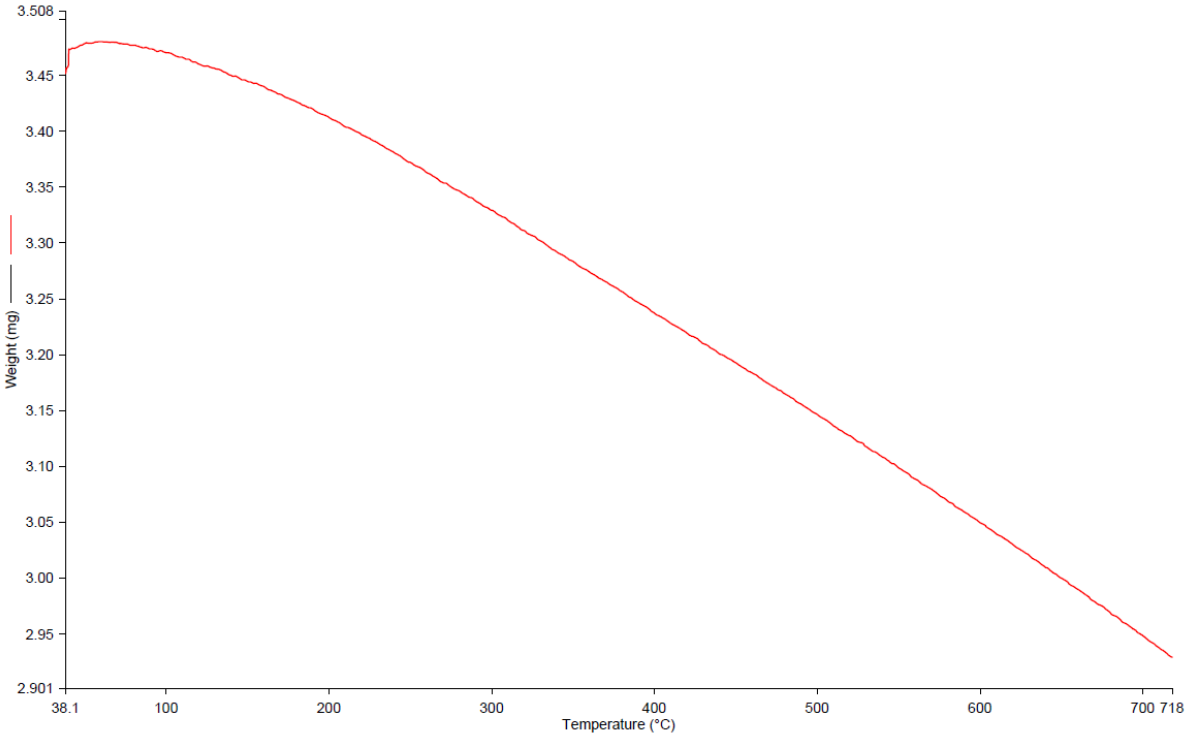
This is according to $5MnO_2 \rightarrow Mn_2O_3+Mn_3O_4+1.5O_2$..

On increasing the temperature Mn_3O_4 is further dissociated to MnO as shown. Also Fe_2O_3 is partially dissociated to Fe_3O_4 .The dissociation reactions of Mn_3O_4 and Fe_2O_3 are as follows.

$$Mn_3O_4 \rightarrow 3MnO+0.5O_2$$

$$3Fe_2O_3 \rightarrow 2Fe_3O_4+0.5O_2$$

At higher temperatures the reactions seems to proceed leading to the formation of single phase spinel structure. Phase determination of the obtained samples was found using XRD analysis as reported [4].



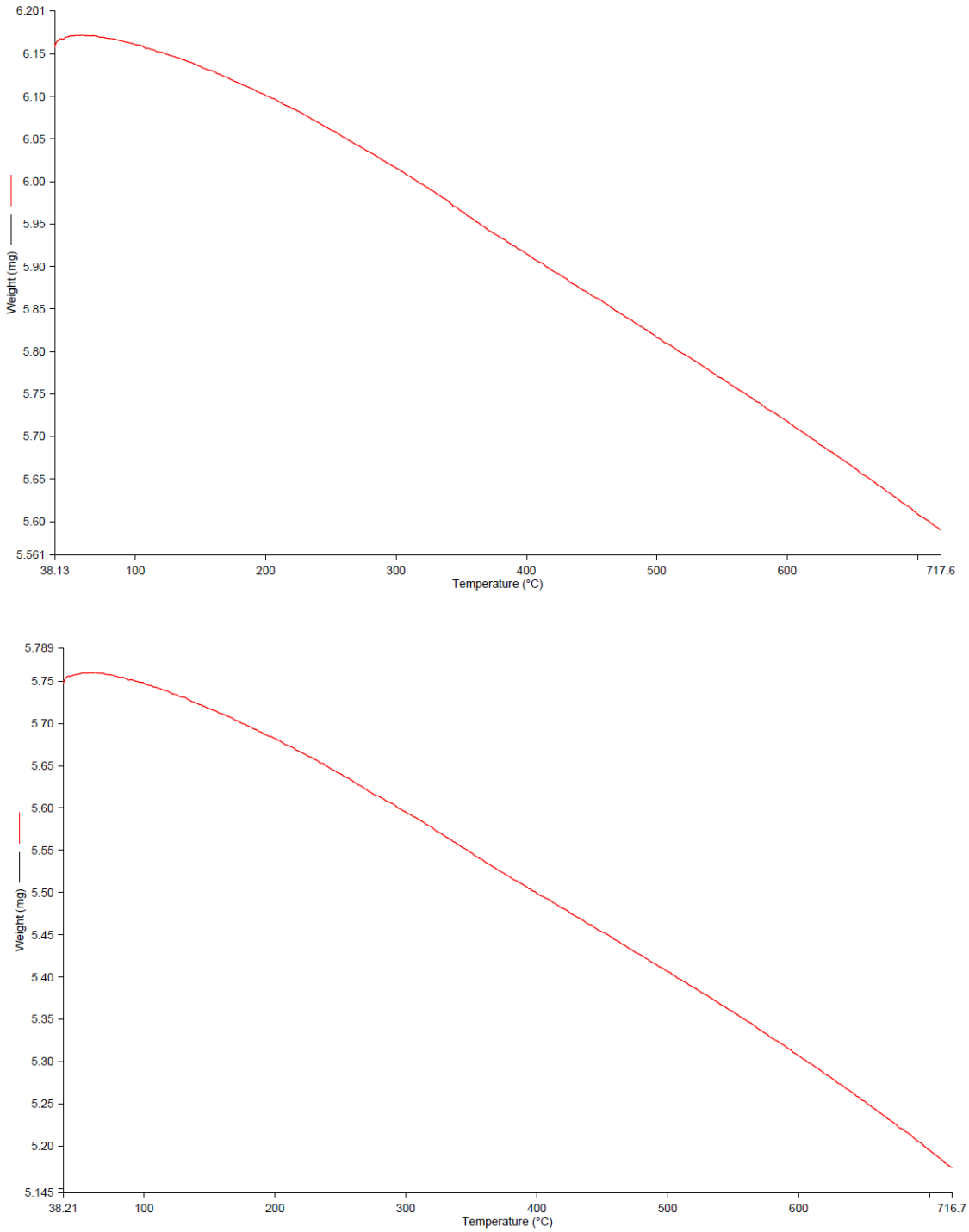
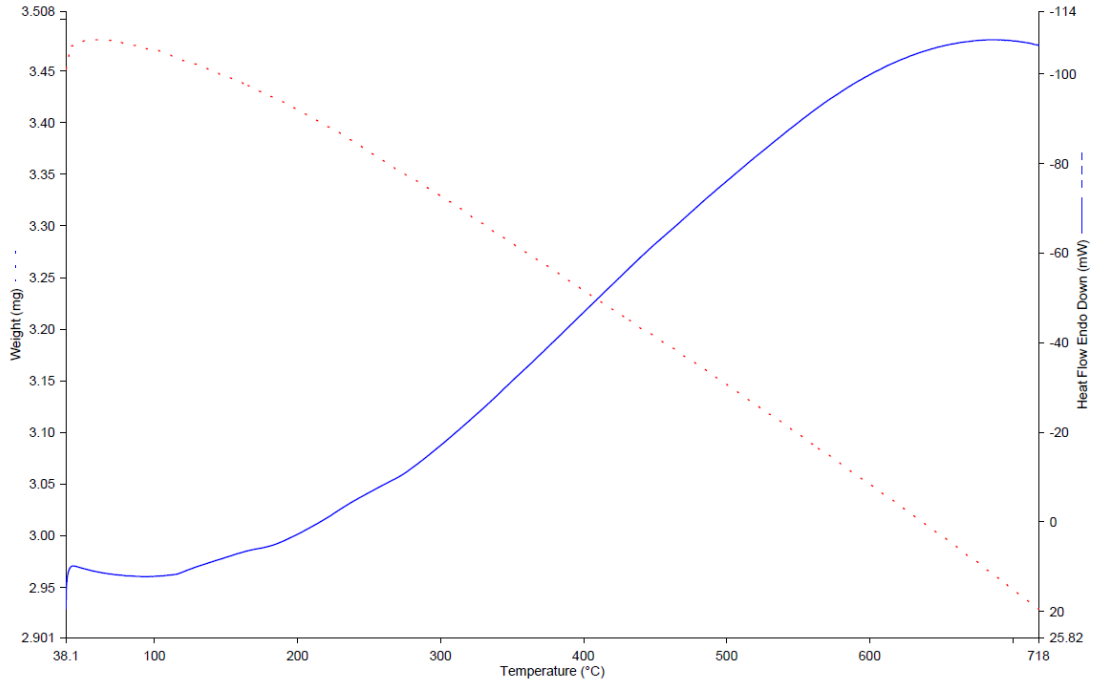
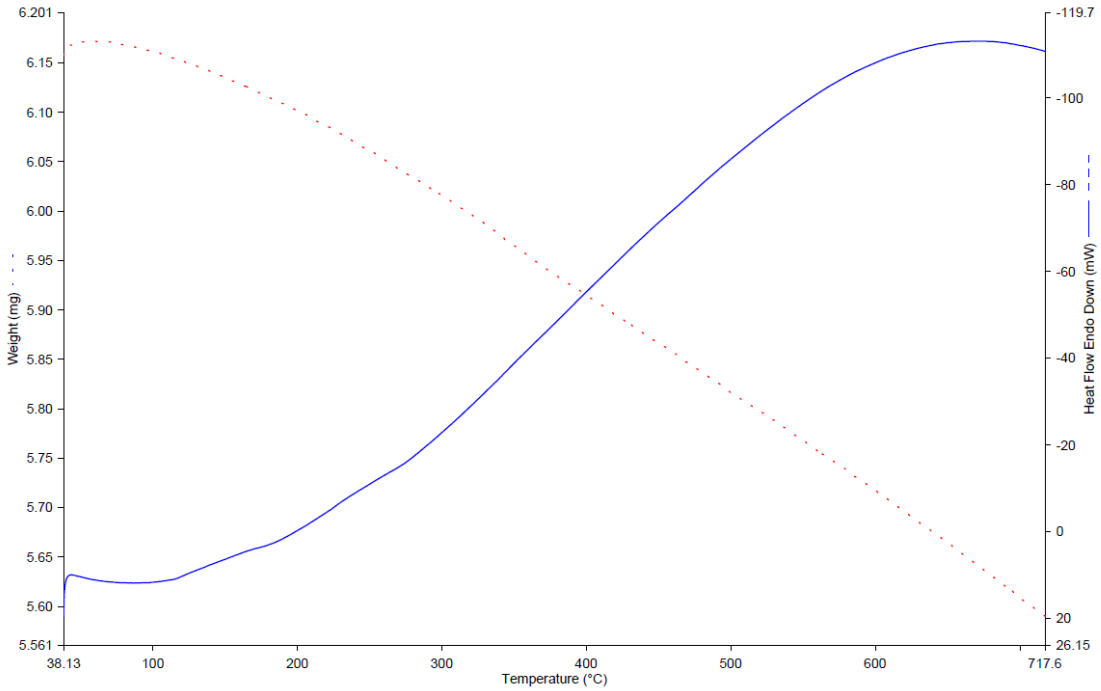


Fig.1. TGA curve of $Mn_{0.8+x}Zn_{0.2}Ti_xFe_{2-2x}O_4$ with x=0.10, 0.15, 0.20 (in weight vs. Temp.)

From the observations (Fig.1) the sample Mn0.8+x Zn0.2TixFe2-2xO4 with (a) x=0.10 initially with mass of 3.452mg reduces to 2.94 mg upon heating to 7200C (Fig1a). The loss in mass percentage is nearly 14.832% Fig.4a).
(b) x= 0.15, the sample initially with mass of 6.158mg reduces to 5.58mg upon heating(Fig1b). The loss in mass percentage is nearly 9.386% (Fig.4b).
(c) x= 0.20, the sample initially with mass of 5.748mg reduces to 5.185mg upon heating to 7200C(Fig1c). The loss in mass percentage is nearly 9.794% (Fig.4c).

From the Differential scanning calorimetry (DSC) measurements, enthalpy changes for phase transitions can easily be determined. The applications of DSC are numerous, either for routine quality control measurements or in research, where high sensitivity and flexibility are important aspects. DSC curve was plotted using Mettler Toledo DSC 822e which is shown in figure 2.





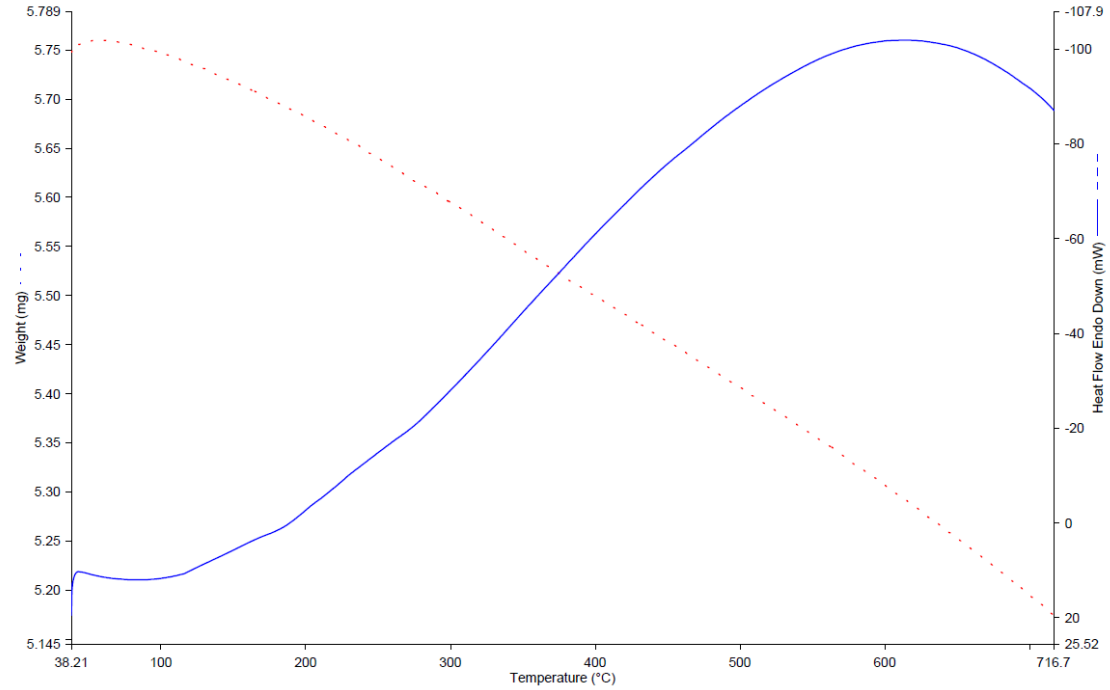
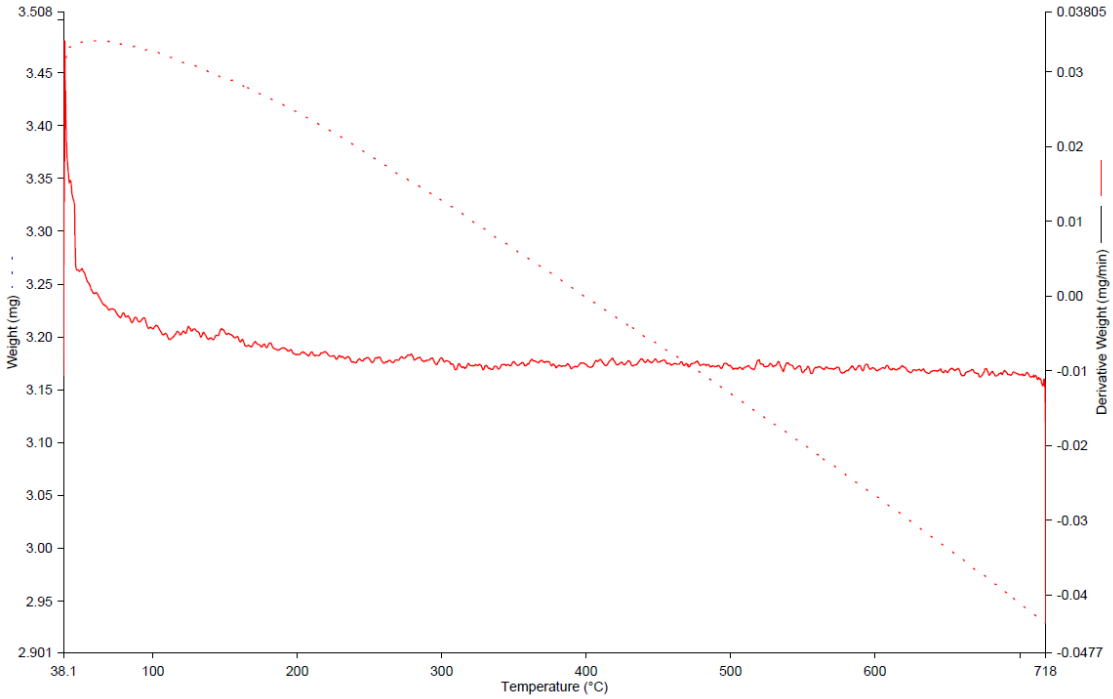


Fig.2. DSC curve of $Mn_{0.8+x}Zn_{0.2}Ti_xFe_{2-2x}O_4$ with x=0.10, 0.15, 0.20



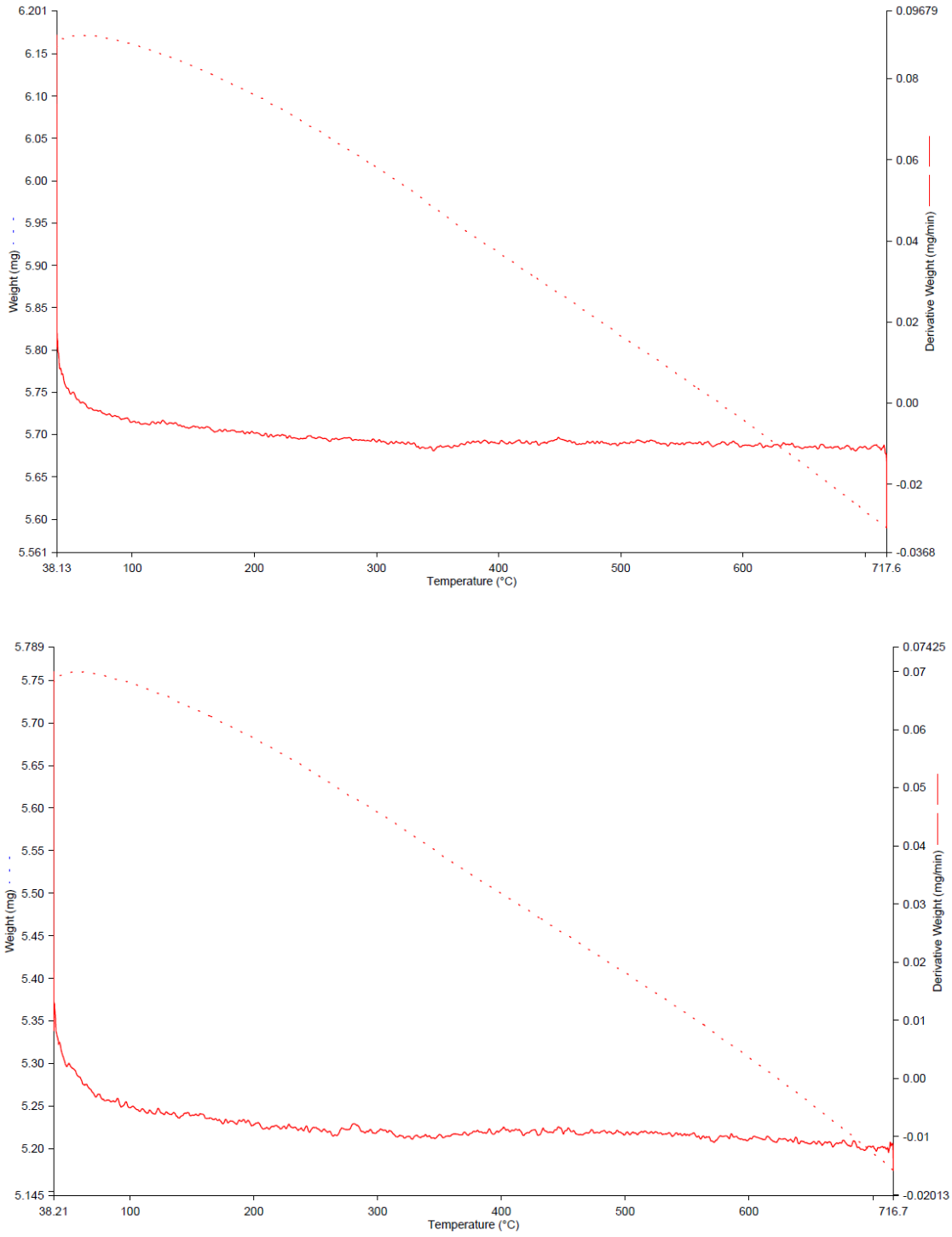


Fig.3. TG/DTA /DTG curve of the sample $Mn_{0.8+x}Zn_{0.2}Ti_xFe_{2-2x}O_4$ with x=0.10, 0.15, 0.20

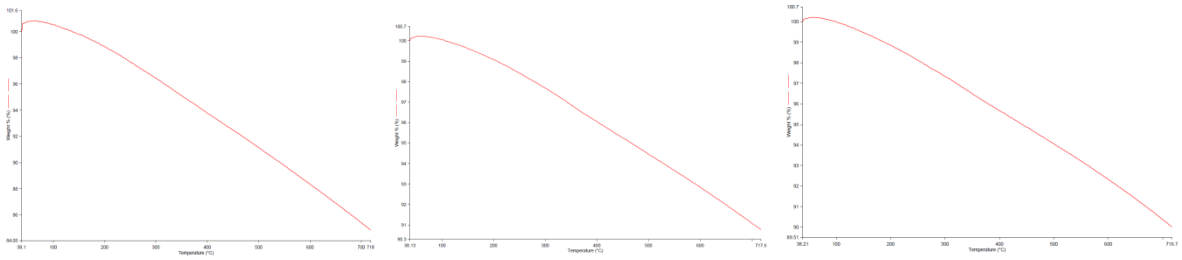


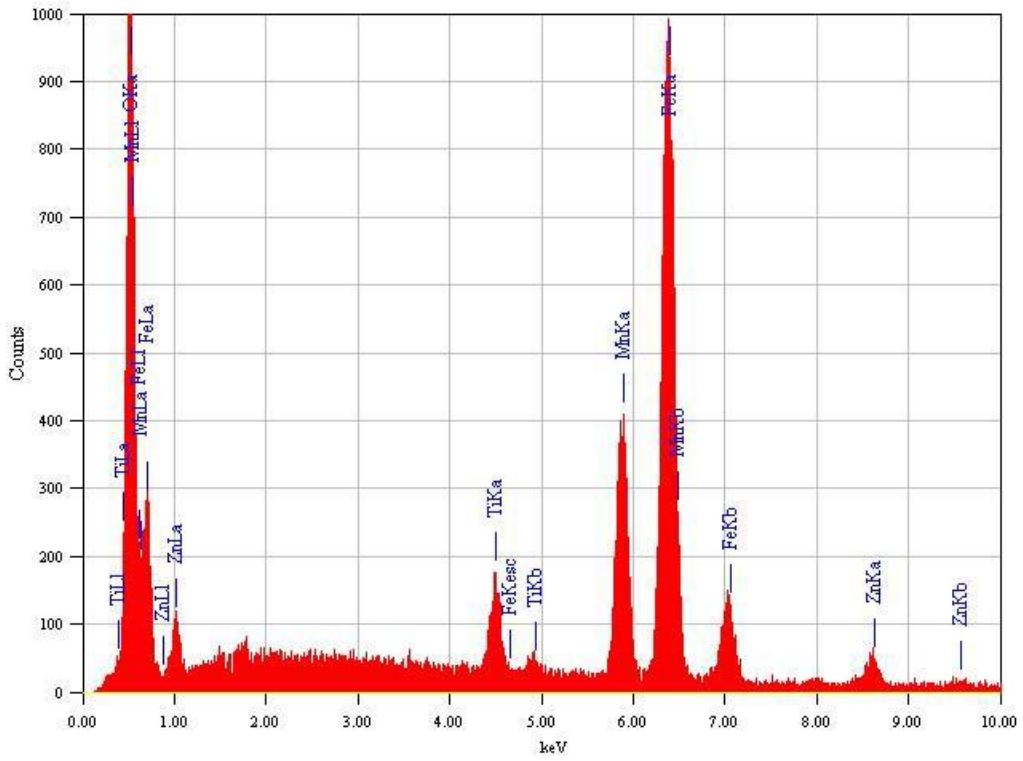
Fig.4: Percentage of mass loss on heating the sample $Mn_{0.8+x}Zn_{0.2}Ti_xFe_{2-2x}O_4$ with x=0.10, 0.15, 0.20

It is well clear from the curves given above that reduction in the mass percentage remains almost same/constant when the temperature is increased. Till 700˚C there is no major loss/reduction in mass and hence we can conclude that complete phase transformation has not attained till 700˚C or the material will have its phase formation only at a high temperature as mentioned earlier.

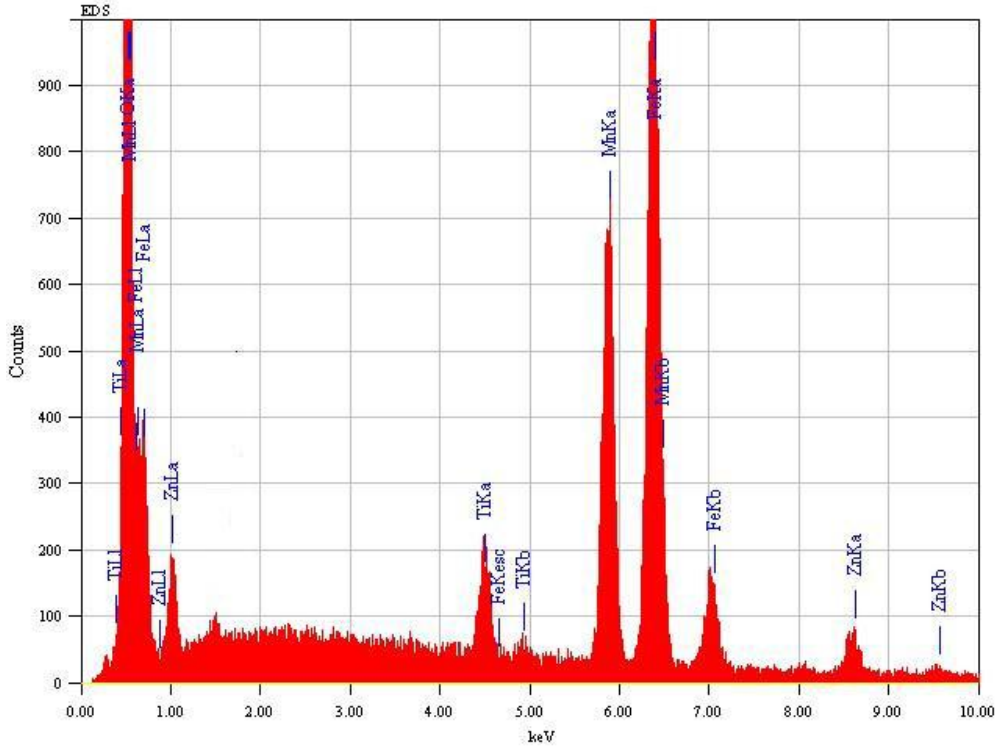
4.EDX Analysis

The instrument used for EDX measurement is ISIS Link Oxford Instrument UK. EDX shows the composition details of the prepared powder. Here an electron beam of 10-20 kev strikes the surface of a sample which causes X-ray to be emitted from point of incidence. The energy of the X-ray from different elements is different and gives the presence of a particular element. When an X-ray strikes the detector, photoelectrons are emitted which in turn generates electron-hole pairs. In this method elements with low atomic number are difficult to be detected. Figure (5) shows the EDX of three samples. Table 1 presents the material content of the samples.

Sample-1



Sample-2



Sample-3

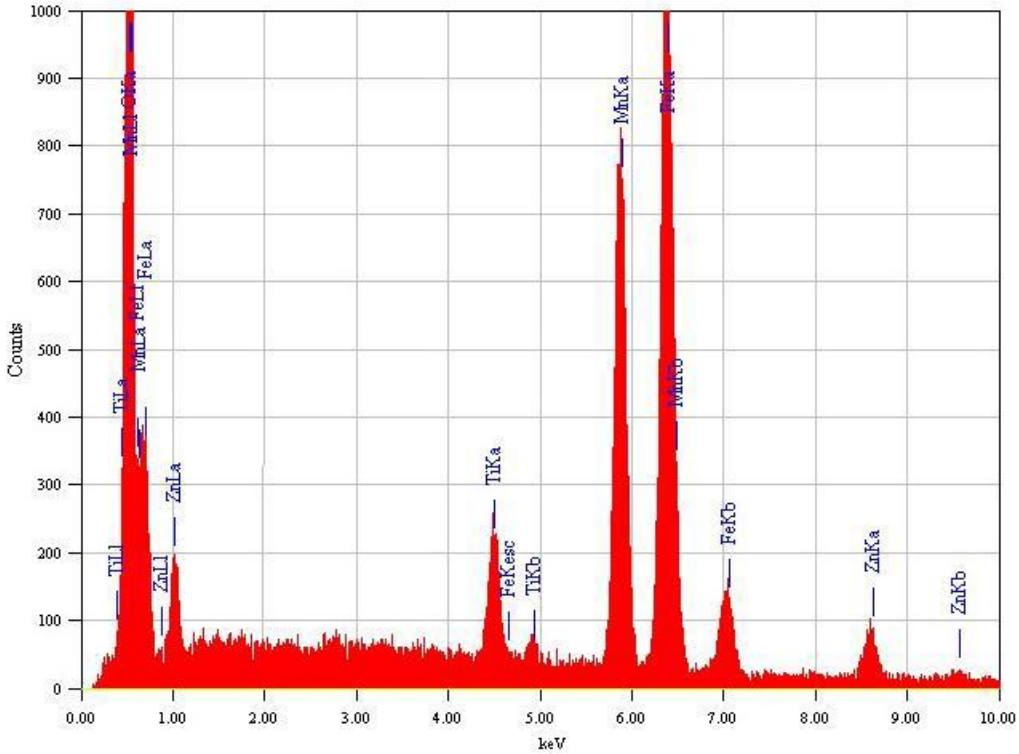


Fig 5. EDX of the samples (1-3) $Mn_{0.8+x}Zn_{0.2}Ti_xFe_{2-2x}O_4$ with x=0.10, 0.15, 0.20

Table 1. Material content of the samples (EDX)

Material	Sample 1	Sample 2	Sample 3
%	%	%	
O	36.4	39.49	37.98
Ti	2.99	3.46	4.55
Mn	13.35	17.21	19
Fe	43.27	35.49	33.19
Zn	4	4.36	5.28

The elemental compositions agree with the stochiometric relations of the prepared compound. EDX spectrum of MnZnTiFeO gave the information on the elemental composition of the material. The EDX spectrums Fig (5) obtained give the material under investigation. From the EDX spectrum of the samples peak positions at 0.637 kev and 5.894 kev correspond to Lα and Kα lines of Mn as reported in the EDAX International chart. Similarly at 1.012 kev and 8.630 kev we get Lα and Kα lines of Zn. At 0.452 kev and 4.508 kev we get Lα and Kα lines of Ti. At 0.705 kev and 6.398 kev we get Lα and Kα lines of Fe. Also at 0.52 kev we get Kα line of Oxygen atom. Hence the dominant peak positions correspond quite well to the energy pattern of the corresponding materials such as Mn,Zn,Fe,Ti and O reported in the EDAX International chart. Table 1 shows the percentage of elements in the prepared samples.

Result and Discussion

TGA / DSC / DTA of the samples indicated that the reaction starting in the range of temperature 900-10000C after the decomposition of MnO2. At 11000C cubic spinel was formed as proved by XRD and SEM analysis. The amplitude of thermal vibrations increases with the increase of temperature. As a result the intensity of the diffracted beam also increases. Hence the reinforcement of waves scattered at Bragg angle differs from a crystal with fixed atoms. The thickness of the planes is 2u where u is the average displacement of atom from its mean position. As the temperature increases, u also increases and hence the intensity of a diffracted beam decreases [8,9]. EDX data supported all these findings.

The free energy inside the interface regions of the ceramic materials affects the phase transitions. The thermal study of the sample clearly shows that the phase transition is taking at a very high temperature. The DTA curves are in conformity with these observations. Changes in lattice imperfections also arise due to the miniature size of the particles. Hence ceramic materials have a different or modified behaviour than that of the other materials. Polymorphism in crystalline structure also can be observed. Thus the thermal stability of the sample can be confirmed from the TGA, DTA & DSC analysis.

Conclusion

Each of these thermal techniques provides unique information that can be used to evaluate the thermal and mechanical properties of the end product. In this work TGA, DTA and DSC analysis was carried out and confirmed that much loss on ignition is not observed, characteristic of a good ceramic material. The EDX analysis indicates the presence and percentage of the elements existing in the sample and it agree with the stoichiometric relations of the prepared compound.

ACKNOWLEDGMENTS

The authors are thankful to SAIF, Kochi for providing the instrumental data and to the Principal, CMS College, Kottayam, Kerala for providing the facilities.

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