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Structural analysis of Mn-Zn ferrite powder prepared from indigenous and imported Mn_2O_3 powders

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ABSTRACT

Mn-Zn ferrite samples have been prepared from Mn_2O_3 powders supplied by two different suppliers from India and abroad. Despite of high purity of both the powders, the magnetic properties of prepared Mn-Zn ferrite samples were different. The X-Ray diffraction analysis of the Mn_2O_3 powders, pre-sintered and sintered Mn-Zn ferrites prepared from indigenous and imported Mn_2O_3 powders have been done to identify the phases. It was found that basic causes of variation in properties are the existence of other non magnetic phases in the indigenously processed sample.

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KEYWORDS

Mn_2O_3;
Ferrite;
X-ray fluorescence;
X-ray diffraction.

INTRODUCTION

Mn-Zn ferrite materials are the most broadly known category of soft electromagnetic materials. These are polycrystalline materials crystallized in the cubic structure similar to that of the mineral "spinel". Their magnetic properties arise from interactions between the magnetic dipoles of the metallic ions(i.e. Fe, Mn), that have uncompensated spin electrons and therefore net magnetic moments, occupying certain positions in relation to the oxygen ions in the crystal lattice[1]. Mn-Zn ferrites find various applications in devices that can be basically characterized as inductors, transformers and absorbers and can be found almost in all broad consumption in electric, electronic or telecommunication equipments.

Analogous to the mineral spinal the magnetic spinal($MgAl_2O_4$ or $MgO.Al_2O_3$) have general formula $MO.Fe_2O_3$, where M is divalent metal ion. The trivalent aluminum is usually replaced by Fe^{+++} or Fe^{+++} in combination with any other trivalent ion. Although the great majority of ferrites contain iron oxide as name implies, but there are some "ferrites" based on Cr, Mn, and other elements. Mn, Cr are not ferromagnetic elements but in combination with other elements such as oxygen and other metal ions, they can behave as magnetic ions[2].

The spinal lattice is composed of a close packed oxygen arrangement in which 32 oxygen ions form a unit cell which is the smallest repeating unit in crystal network. Between the layers of oxygen ions, there are the interstices that may accommodate the metal ions. Not all the interstices are same; some which would be called A sites are surrounded with four nearest neighboring oxygen ions and are called tetrahedral sites. The other type of sites (B sites) is coordinated by six neighboring oxygen ions whose centre connecting lines describe an octahedron. The B sites are called octahedral sites. In the unit cell of 32 oxygen ions there are 64 tetrahedral sites and 32 octahedral sites. If all the sites are filled with metal ions, of either +2 or +3 valence, the positive charge would be much greater than the negative charge and so the structure will be much greater than negative charge. Due to which the structure will be electrically neutral. It turns out that out of the 64 tetrahedral sites, only 8 are occupied and out of the 32 octahedral sites, only 8 are occupied. If , as in the mineral spinal the tetrahedral are occupied by divalent ions and the octahedral sites are occupied by the trivalent ions, the total positive charge will be 8×(+2)= +16 plus the 16×(+3) = +48, or a total of +64 which is needed to balance the 32×(-2)=-64 for the oxygen ions. Then there would be eight formula units of $MO.Fe_2O_4$.

Two exceptions are found in Zn^{++} and Cd^{++} which prefer tetrahedral sites because electronic configuration is favorable for tetrahedral bonding to oxygen ions. In most commercially important Mn-Zn ferrite materials, the starting mix may contain slightly more than 50 mole percent of Fe_2O_3. The purpose of the extra ion is to improve the magnetic properties by the formation of Fe^{2+} ions. One such basic property is the magnetostriction which is defined as change in the length of a material when it is subjected to a magnetic field.

In spinal ferrites the divalent ions can be Mn^{2+}, Ni^{2+}, Co^{2+}, Mg^{2+}, Cu^{2+}, Zn^{2+}, and Fe^{2+}. The choice is determined by the specific applications. For materials where large magnetic moments are needed, such as in power applications, the magnetic metal ions with a most unpaired spins are chosen. This is one reason why $Mn(5\mu_B)$ ferrite and ferrites that contain uncompensated $Fe^{3+}(5\mu_B)$ ions are useful. Although Ni^{2+} has a lower moment$(2\mu_B)$ than Mn^{2+}, $NiFe_2O_4$ has higher resistivity for high-frequency operation and, because of its higher T_c, it can function at higher temperature. Most commercially important low frequency ferrites contain Zn. Zinc ion substitution for other divalent ion can increase effective magnetic moment. It also contributes for an increase in magnetic permeability. Very often, it is the ratio of ZnO to the other divalent oxides as well as the degree of divalent Fe substitution that gives ferrite material developers greatest latitude in optimizing the properties of a specific ferrite. In the sintered ferrite the zinc content will depend on the amount that went in originally minus that which was lost in the sintering process. Since Zn is a rather volatile ion, incorrect firing will cause its loss which will lead to a gradient in Zn content across the thickness of the ferrite. In addition to this, the loss of Zn may further create vacancies. Because of this strain will be produced which may further deteriorate the properties of ferrites[2-3]. If the proper vacancies are not compensated in Mn-Zn ferrites the required magnetic properties can not be achieved. In this paper, effort has been made to identify the basic cause of variation in permeability for both categories of samples made from indigenously and imported powders.

EXPERIMENTAL

The Mn_2O_3 powder samples were taken from two different suppliers, and their chemical analysis was done using X-ray fluorescence technique to check the composition difference. In order to study the phase changes in the Mn-Zn ferrites through different stages of their processing the X-ray diffraction analysis of pre-sintered and sintered ferrites has been done using Rigaku X-ray diffractometer(Model Rigaku D-Max IIIC)

Raw materials used in the present investigation are oxides having high purity. The required quantity of oxides for each set of experiment was pot milled. The mixture was calcined at 950^0C for 90min. After calcinations the mixture was ball milled using 30mm balls for 6 hours to achieve a specific surface area(SSA) of $4200cm^2/gm$. Very small amount of Bi_2O_3(0.02wt %) was added which acts as grain growth accelerator which evaporates at high temperature and therefore does not participate in the composition variation in final sintered

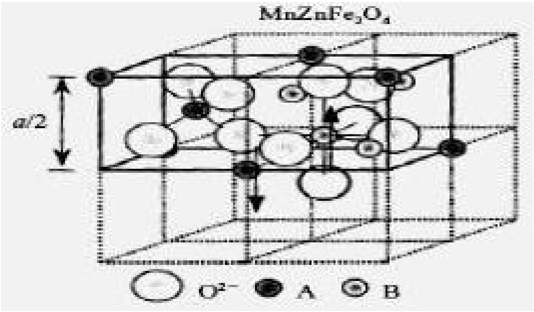

Figure 1: Crystal structure of Mn-Zn Ferrite

product[4]. The slurry obtained during this process was dried in an oven and then granulated, by adding polyvinyl acetate (PVA) and polyethylene glycol (PEG), till granules of size between 200 and 500μm are obtained. These were then mechanically pressed to form torroids of external diameter of 29.13 to 29.20mm with internal diameter of 17.38 to 17.45mm and height of 14.17 to 14.25 mm, with a variation in green density of 3 to 3.2gm/mm^3. In the final stage of production the mechanically pressed samples were sintered in a step up furnace of 900^{0}C to 1360^{0}C. The systematic chart of procedure followed for the preparation of Mn-Zn ferrite sample is shown in figure 2.

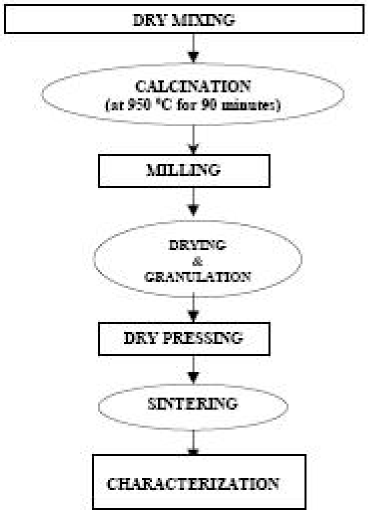

Figure 2: Schematic diagram of the experimental procedure followed for the synthesis of Mn-Zn ferrite

RESULTS AND DISCUSSION

Five sets of ferrite samples of same composition were prepared using both categories of Mn_2O_3 powders. In each case amount of ZnO was same. Since Mn-Zn ferrites find their applications for high magnetic permeability and low losses, all the prepared samples were checked for their permeability. It was found that the samples prepared from indigenous Mn_2O_3 powders failed to show promising results as comparison to the Mn-Zn ferrite prepared from imported Mn_2O_3 powders. For same zinc content the permeability for Mn-Zn ferrite produced from indigenous Mn_2O_3 powder came out to be 2500 while from imported Mn_2O_3 powder it was 6500, which is a big difference. Because of technical reason for the undertaken work, amount of Zn content is not mentioned here.

In order to investigate the cause both physical and chemical properties of powders was checked. It was observed that both powders are not of same color. The indigenously processed was of blackish red color whereas imported powder was of light brown color. During course of the study it was observed that the bulk density of the both the powders were found to be same. The compositional difference of the powders was done by X-ray florescence technique. The results obtained from above mentioned procedures is shown in TABLE 1.

TABLE 1: Physical and chemical analysis of Mn_2O_3powders

IMPORTED Mn_2O_3 powder		INDIGENIOUS Mn_2O_3 powder
Physial		
(1) Color: Light brown		(1) Color: Blackish red
(2) Bulk density: 1.4gm/cm^3		(2) Bulk density: 1.4 gm/cm^3
Chemical analysis		
XRF- analysis in Kcps		
Elements	Imported (A)	Indigenious (B)
SiO_2	0.6084	0.6179
CaO	0.4691	0.4921
Al_2O_3	0.6652	0.6875
Fe_2O_3	3.6747	3.6781
ZnO	0.4923	0.495
Cr_2O_3	0.8252	0.8213
CoO	0.4676	0.5251
K_2O	0.1247	0.156
Na_2O	0.0138	0.0154
Cl	0.0654	0.0713
MnO	153.0072	153.0389
SO_4	0.3594	0.1864
MgO	0.3641	0.3895
P_2O_5	0.0043	0.0204
SnO_2	0.0026	0.0033
CuO	0.3342	0.3454
NiO	0.3712	0.38
ZrO_2	0.0056	0.0052
V_2O_5	0.671	0.6482
TiO_2	0.6485	0.6784
Sb_2O_3	0.004	0.0024
Se_2O_3	0.026	0.0329
Bi_2O_3	0.0134	0.0175
CdO	0.0031	0.0022
Nb_2O_5	0.0039	0.0037
PbO	0.0102	0.013
BaO	0.5145	0.5442

The results show that there is not much difference in composition of the two powders which may lead to big difference in permeability of the derived product.

In order to check the phase difference of the com-

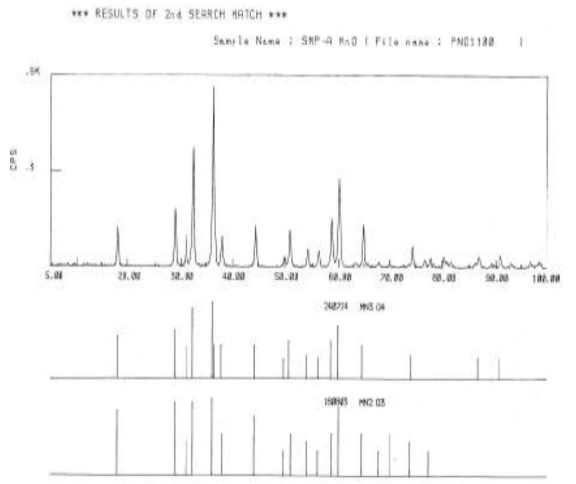

Figure 3: X-Ray diffraction pattern of imported Mn_2O_3 powder

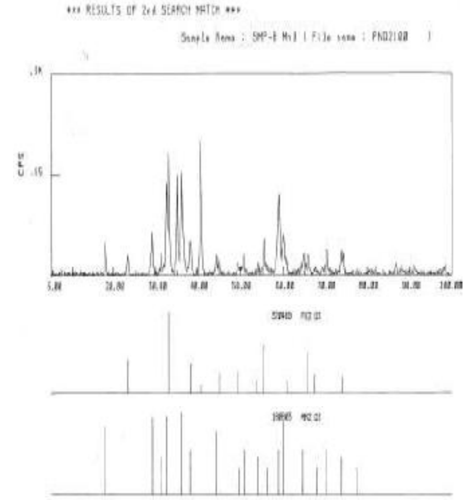

Figure 4: X-Ray diffraction pattern of Indigenous Mn_2O_3 powder

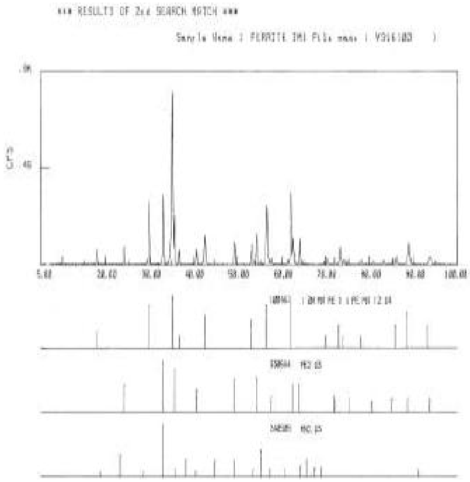

Figure 5: X-Ray diffraction pattern of pre-sintered Mn-Zn ferrite prepared from imported Mn_2O_3 powder

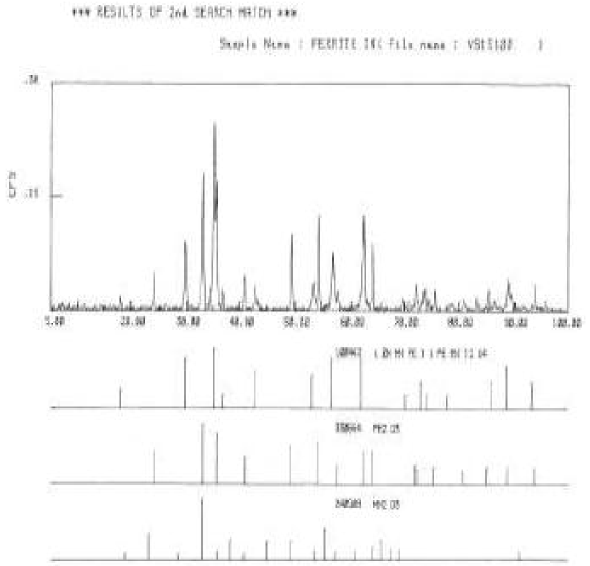

Figure 6: X-Ray diffraction pattern of pre-sintered Mn-Zn ferrite prepared from indigenous Mn_2O_3 powder

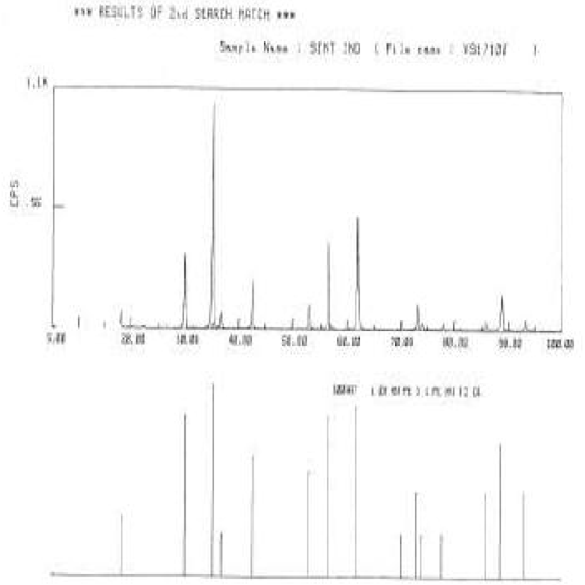

Figure 7: X-Ray diffraction pattern of sintered Mn-Zn ferrite prepared from indigenous Mn_2O_3 powder

position during ferrite manufacturing process, the X-ray diffraction analysis of Mn_2O_3 powders, pre-sintered and sintered Mn-Zn ferrites prepared from these powders was done. The analysis has shown a remarkable difference in between both categories.

The imported Mn_2O_3 powder was having pure manganese phase while indigenously Mn_2O_3 powder was having ferrite phase already present in it as shown in figure 3 and 4 respectively. Further the X-ray diffraction analysis of pre-sintered and sintered ferrites (fig-

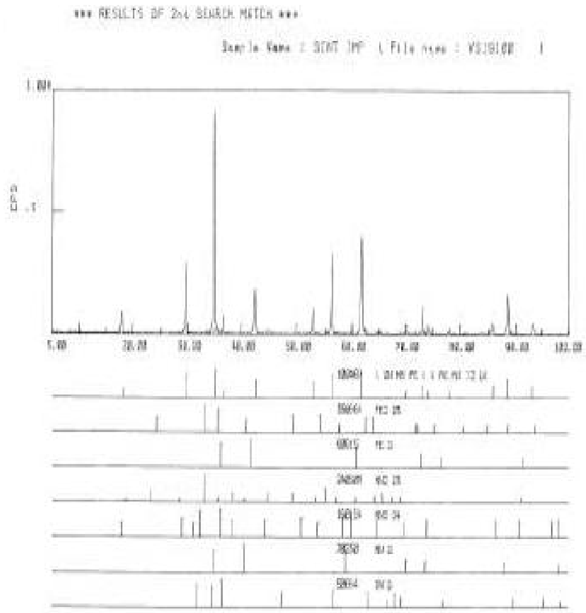

Figure 8: X-Ray diffraction pattern of sintered Mn-Zn ferrite prepared from imported Mn_2O_3 powder

ure 5-8) shows that in ferrite formed from indigenous Mn_2O_3 powder the transformation of Mn^{+++} state to Mn^{++} state is not complete, while this is complete in case of ferrite produced from imported Mn_2O_3 powder. Since Mn^{++} state is responsible for magnetic moments in Mn-Zn ferrites therefore ferrite samples made from indigenous Mn_2O_3 powders shows weak magnetic properties[5].

CONCLUSION

We have investigated the phases present in prepared samples. The following conclusions can be drawn.

1. The complete phase transformation of Mn^{+++} to Mn^{++} phase is important to acquire magnetic properties in Mn-Zn ferrites as in the absence of this; the net magnetic moments would be very less.
2. The quality of Mn-Zn ferrites is not only dependent on purity of substance but also depends on its processing factors. In this study the need of proper sintering parameters is seen to get complete phase formation.

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