

Un-necessity of Milling in Ferrites Preparation

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Abstract

This work is an attempt to prove that the milling stage for some pre-sintered ferrite compounds is unnecessary. Three samples of Ni, Zn and Mg ferrites were prepared by using the traditional ceramic method without milling. The samples were tested by XRD and the analysis of the results showed that Ni and Zn ferrites (in contract of Mg-ferrite) could be prepared without milling. We can make use of this phenomenon to minimize the energy required for preparation process.

الخلاصة

هذا العمل محاولة لاثبات ان اجراء عملية الطحن لعينات الفزايت الملبدة اولياً هي عملية غير ضرورية لبعض مركبات الفزايت. تم تحضير ثلاثة مركبات هي النيكل_فزايت و الزنك_فزايت والمغنيسيوم_فزايت باستخدام الطريقة التقليدية للسراميك وبدون طحن، ثم اجري فحص حيود الاشعة السينية للعينات، وبتن تحليل النتائج المستحصلة انه بالامكان الحصول على النيكل_فزايت و الزنك_فزايت باستخدام هذه الطريقة على العكس من المغنيسيوم_فزايت.و يمكن استغلال ذلك في تقليل الطاقة المصروفة في عملية الانتاج وبالتالي الكلفة الكلية لعملية الانتاج.

Introduction

Ferrites are usually occurred in isometric octahedron crystals and are optically isotropic, similar to the mineral spinel. Ferrites are magnetic materials that have the general chemical formula of (MFe_2O_4) where M represents metallic cations like Ni^{++} , Mn^{++} , Fe^{++} , Co^{++} , Cu^{++} , Zn^{++} , Cd^{++} , and Mg^{++} (Sinka, 1995). Majority of ferrites are stoichiometrically balanced comprising one mole of bivalent oxide and one mole of ferric oxide. There are many techniques that can be used to produce a ferrite ceramic body from its raw materials. The earliest route was the mixed stoichiometric oxides for preparing homogeneous fine powder (Kingery, et al, 1976; and Singer, and Singer, 1997). Sridhar Komarnent et al. commented that, for most technical ferrites, the art developed for these materials has been the control of raw materials and processing. A reproducible preparative route to ceramic powders is to dissolve all constituent elements in the same solvent and process the solution so as to obtain a dry, homogeneous, finely divided, and hence reactive powder (Sridhar, et al, 1988, Prinya, 1988).

The usual commercial production of ferrites components are made by the ceramic technique, in which the compacted powder is pre-sintered at suitable temperature, and then the product is milled to the required fineness of powder and the components are formed normally by compaction or extrusion, then final firing to the sintering temperature up to 1450 °C to produce the desired magnetic materials.

The main purpose of the current research was to prepare ferrite by one cycle of operating ceramic technique, without passing through the milling stage.

Experimental procedures

Magnesium, Nickel, and Zinc ferrites have been prepared from its pure oxides. Mixing of these oxides stoichiometrically with iron oxide are made as a slurry by adding 1% wt. of polyvinyl alcohol. The mixture is well stirred to obtain homogeneous suspension. The resulting solution was filtered, dried, ground, and finally sieved to fine powder (< 38 micron). The prepared powder is placed in steel die of 30 mm diameter, and then pressed at 100 MPa to produce pellets free of

fractures and any layering. The formed green samples are oven-dried for 24 hours at 105°C and the firing procedure is achieved in an electrical furnace to the temperature of 400 °C for a minimum of 4 hours to complete the burn-off the binder, and followed by raising the sintering temperature to 1100 °C. The next step of the firing was the same except the sintering temperature of 1250 °C. All the samples have a heating rate of 3 °C/min and soaking time of one hour. The prepared ferrites of this study are not milled the pre-sintered product as usually make in literatures.

Philips-PW 1965/50 x-ray diffractometer with a nickel filter using monochromatized $Cu\alpha$ radiation at 30 kv and 20 mA was used throughout. The samples were scanned at $2^\circ 2\theta$ per min. from 29° to $39^\circ 2\theta$. The counts were recorded with chart speed of 25 mm/min. CaF_2 is used as internal standard. Diffraction peaks were measured to the nearest $0.01^\circ 2\theta$ and converted to d-observed spacing using Bragg's law ($n\lambda = 2d \sin\theta$) with $\lambda = 1.54178$ ($Cu\alpha$).

Results and discussion

X-ray diffraction data for Mg, Ni, and Zn-ferrites are given in table (1). The unit cell dimension of each ferrite is driven from the formula, $1/d^2 = (h^2+k^2+l^2)/a^2$. Where:

- d = Interplanar spacing of adjacent planes.
- (hkl) = Miller's indices of the plane.
- a° = length of unit cell.

Table (1) x-ray data of prepared ferrites.

Ferrite Type	d-spacing A°	I/I $^\circ$	(hkl)	$a^\circ \text{A}^\circ$	$a^\circ \text{A}^\circ$ (avg.)
Zn	2.983	31	220	8.437	8.439
	2.545	100	311	8.441	
	2.436	10	222	8.439	
Mg	2.978	40	220	8.423	8.411
	2.710	20	n.d.	n.d.	
	2.532	100	311	8.398	
Ni	2.969	30	220	8.398	8.400
	2.532	100	311	8.398	
	2.426	10	222	8.404	

n.d.= not defined

Observed and calculated densities of the specimens were measured using pycnometer and x-ray data respectively (Table 2).

The formula of ($\rho_{calc.} = (MW*Z*1.66) / V$) is considered here to calculate density, in which:

- $\rho_{calc.}$ = calculated density (g/cc).
- MW = molecular weight. (g/mole)
- Z = number of atoms per unit cell.
- 1.66 = reciprocal of Avogadro number.
- V = volume of unit cell (A°)³.

Table (2) Observed and calculated densities of the prepared ferrites.

Ferrite type Density (g/cc)	Zn-ferrite	Mg-ferrite	Ni-ferrite
Observed density	4.18	2.68	4.32
Calculated density	5.34	4.47	5.25
Theoretical density	5.324	4.522	5.368

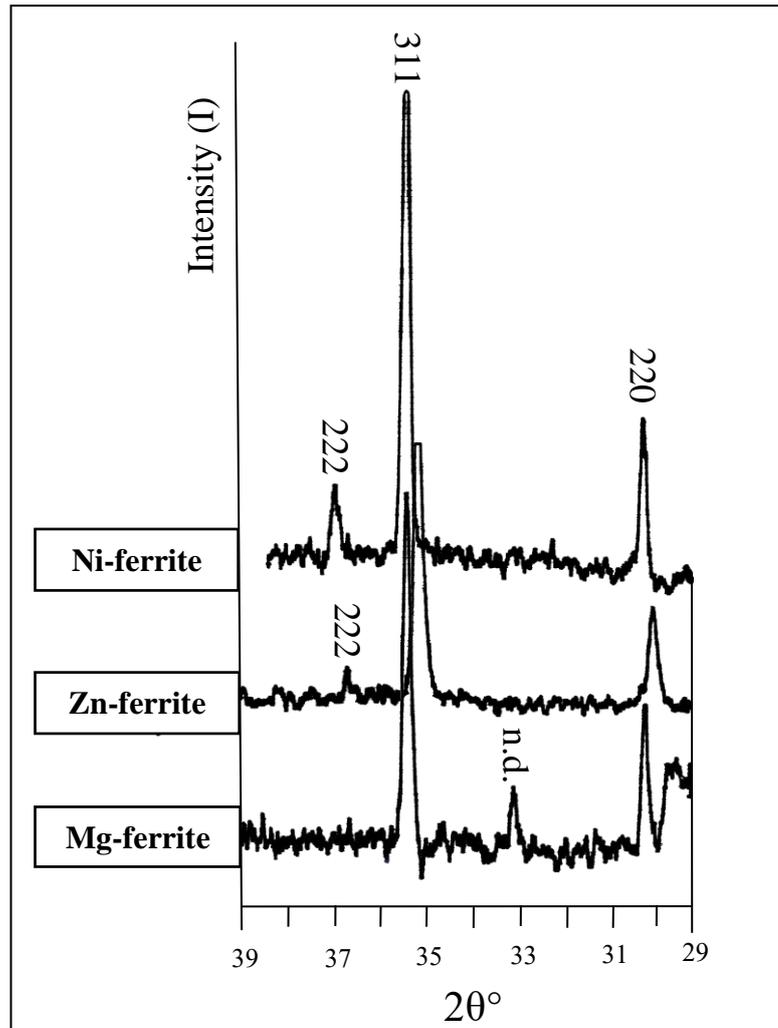


Fig (1) x-ray analysis of the prepared ferrites.

Figure (1) shows the peaks diffracted from the principal planes (311), (220), and (222) of the prepared ferrites. These peaks are coincidence with those analogues of the joint committee on powder diffraction standards (JCPDS), for Ni-ferrite and Zn-ferrite.

Mg-ferrite shows some deviation by presence of undefined peak, table (1) and fig (1). This additional peak is generated as a result of high diffusion coefficient for Mg cations in the O anions of the ferrite body in comparison with those of Ni and Zn cations. The Kirkendall effect explains this phenomena as the Fe_2O_3 and MgO blocks (in Mg-ferrite) will move away from each other because the same amount of oxygen is transported from a shell with a large radius ($r_{Fe} = 0.74 \text{ \AA}$) to a shell with a smaller

radius ($r_{Mg} = 0.64A^\circ$) (Michel, 1997, and Jastrzebski, 1977). Voids growth will thus occur in this situation, and affected directly upon the structure of Mg-ferrite, in which the peak (222) is gradually faded. Hence, there is some expansion happened in its structure and caused dropping in observed density (2.68 g/cc) table (2).

We know that all the ferrites are ferromagnetic materials, in which the ferromagnetism is detected when the magnetic dipoles are aligned with the external magnetic field. This alignment was achieved when the samples have high grade ordering (no centre) in the unit cell structure (James, and Cornelis, 2002). This ordering was observed in the samples of Ni and Zn ferrite, as shown from x-ray analysis fig. (1) & table (2), therefore they can reflect the magnetic property while they didn't milled after the pre-sintering stage.

Conclusions

The main conclusion of this study is that some of ferrite compounds can be prepared without needing to mill treatment of pre-sintering samples, as shown for Ni-ferrite and Zn-ferrite, and we can make use of this phenomenon to minimize the energy required for preparation process and then decreasing the total cost.

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