A comparison study of the structural and magnetic properties of pure Ni metal and NiZnMn ferrite

Ali A-K. Hussain, Raad M.S.Al-Haddad, Muthafar F. Al-Hilli, Mazin Salman
Department of Physics, College of Science, University of Baghdad, Baghdad, Iraq
E-mail: mfja72@yahoo.com

Abstract
The magnetic properties of a pure Nickel metal and Nickel-Zinc-Manganese ferrites having the chemical formula Ni_{0.1}(Zn_{0.4}Mn_{0.6})_{0.9}Fe_{2}O_{4} were studied. The phase formation and crystal structure was studied by using x-ray diffraction which confirmed the formation of pure single spinel cubic phase with space group (Fd3m) in the ferrite. The samples microstructure was studied with scanning electron microstructure and EDX. The magnetic properties of the ferrite and nickel metal were characterized by using a laboratory setup with a magnetic field in the range from 0-500 G. The ferrite showed perfect soft spinel phase behavior while the nickel sample showed higher magnetic loss and coercivity.

Key words
Hysteresis loop, magnetic properties, ferrites.

Article info.
Received: Mar. 2019
Accepted: May. 2019
Published: Dec. 2019

NiZnMn
دراسة مقارنة للخواص التركيبية والمغناطيسية لمعدن النتيل النقي و فيرايت
علي عبد الكريم حسين, رعد محمد صالح الحداد, مظهر فؤاد جميل, مازن سلمان
قسم الفيزياء، كلية العلوم، جامعة بغداد، بغداد، العراق

الخلاصة
تم دراسة الخصائص المغناطيسية لمعدن النتيل النقي و فيرايت النتيل- زنك – منغنيز ذو الصيغة الكيميائية Ni_{0.1}(Zn_{0.4}Mn_{0.6})_{0.9}Fe_{2}O_{4}، وذلك دراسة تشكيل الأطوار والتركيب البلوري بواسطة حيود الإشعاع السيني (Fd3m) للمادة الفيرايت. تم تعريف المفردات و تضيح الخصائص المغناطيسية للمادة الفيرايت دون استخدام المجهز الإلكتروني الماسح و EDX. تم تشخيص الخصائص المغناطيسية للمادة الفيرايت ومعدن النتيل باستخدام تجهيزات قياس مختبرية ضمن مدى مجال مغناطيسي 500 G-0. أظهرت عينة الفيرايت سلوك مغناطسي متباين لحالة الفيرايت بطور السبينل اللين، بينما عينة النتيل النقي أظهرت فقان مغناطسي عالي مع قوة مغناطيسية عالية.

Introduction
Ferrites being most important magnetic materials with ferromagnetic properties are extensively studied due to their wide technological importance in magnetic recording media, magnetic storage and contrast agents for MRI. Spinel ferrites being a part of the ferrite family are important ferrite for the field of electronics. Crystal of spinel ferrite possesses the structure of the natural spinels MgAl_{2}O_{4}, determined by Bragg [1].

Nickel ferrite with the formula NiFe_{2}O_{4}, consists of a well-known an inverse spinel possesses 8 units of NiFe_{2}O_{4} in each unit cell in the structure. Half of the Fe^{3+} prefers to occupy the A-sites while the others take up the B-sites. Consequently, the ferrite may be given in the form (Fe^{3+})\text{A}[Ni^{2+}Fe^{3+}]BO_{4} [2, 3].

Fundamental characteristic of any magnetic substance is the relationship between flux density and magnetic field, i.e., the B–H loop. The loop area
is a represents the amount of energy dissipated owing to hysteresis in a unit volume through 1 cycle of magnetization [4]. Ni-Zn is consistently examined owing to its noticeable magnetic characteristics such as high saturation magnetization, low coercitivity and cheap price [5]. Ni-Zn soft ferrite crystallizes as a face-centered cubic lattice of the oxygen ions of the type (Zn$_x$Fe$_{12x}$)[$Ni_{12x}Fe_{11x}$]O$_4$. In this formula the metallic cations in ( ) fill in the A-sites and the metallic cations in [ ] fill in the B-sites. This ferrite is the most famous one employed for many years in the electronic applications. Currently, this substance is largely investigated in view of seeking improved properties for novel applications, particularly in the nano as applications and thin films. Nano Ni-Zn ferrites could be obtained with improved dielectric properties and high performance at relatively lower firing temperature. The substance magnetic properties are greatly influenced when the particle size comes to be smaller, because of the effect of thermal agitation on the magnetic moment arrangement, producing the super-paramagnetic relaxation phenomenon [6, 7].

This work reports a comparison between the magnetic properties of Nickel metal and nickel-zinc-manganese ferrite based upon the investigation of their crystal- and micro-structure.

**Experimental**

Nickel-Zinc ferrites with the formula Ni$_{0.1}$(Zn$_{0.4}$Mn$_{0.6}$)$_{0.9}$Fe$_2$O$_4$ was prepared by standard ceramic technique. A proper ratios of analytical grade materials; Fe$_2$O$_3$, NiO MnO and ZnO were mixed in acetone for 2 hours using a ball mill. The samples were prepared by uniaxial powders pressing technique. The final sintering was carried out at 1100 °C with soaking time of 2 hours using Carbolite furnace. Ni metal laboratory reagent sample was obtained from BDH chemicals limited, England, with purity of 99.9 %. The samples crystal structure was characterized by using x-ray diffraction technique performed on Shimadzu X-Ray Diffractometer XRD 6000. The microstructure of the samples was studies by using scanning electron microscope supplemented with EDX to confirm the material stiochiometry. The SEM was performed on ALS2300 Angstrom.

The magnetic properties were studied by using lab setup with a magnetic field in the range from 0-500 Gauss.

**Results and discussion**

Fig.1 illustrates the XRD pattern of Nickel-Zinc ferrite with the formula Ni$_{0.1}$(Zn$_{0.4}$Mn$_{0.6}$)$_{0.9}$Fe$_2$O$_4$. It confirms the formation of pure single spinel cubic phase with space group (Fd3m) in the ferrite. The sharp peaks indicate that all specimens are pure phase crystalline structure [8]. The diffraction peaks and their correspondent (hkl) results agree perfectly with ICDD card number 00-023-1119. The existence of (220), (311), (400), (422), (511), and (440) planes in the patterns assured crystallization of a pure cubic phase [9, 10]. Fig.2 shows the XRD pattern of a pure nickel metal characterized by the three peaks with hkl identified as (111), (200) and (220) indicating that the resulting powders are face-centered cubic (fcc) nickel [11].
Fig. 1: XRD pattern of Ni ferrite.

Fig. 2: XRD pattern of Ni metal.

Fig. 3 shows the SEM micrograph of the ferrite sample. It shows very well dense surface with the existence of some closed pore. The particles size seems to be less than 10 μm. Fig. 4. shows the micrograph the nickel sample which illustrates a laminar surface of the metal with no flaws exists on the surface.
Fig. 3: SEM micro image of Ni ferrite.

Fig. 4: SEM micro image of Ni metal.
Fig. 5 shows the EDX spectrum of the ferrite sample which confirms the stoichoimetry of the ferrite sample as given by elements analysis in Table 1. There is a small deviation from the exact stoichoimetry due to the loss of material during pressing and sintering processes. Fig.6 shows the EDX spectrum of the pure nickel metal sample. The sample purity seems to be less than that given by the supplier as given by the elements analysis of the sample in Table 2. This is may be due to the surface contaminations and/or the pressing of the round–shaped sample into pellet shape. So, the contamination of the dies might cause the existence of some elements e.g. C and Fe.

![EDX spectrum of Ni ferrite.](image)

**Table 1: Elements analysis of ferrite sample based on the EDX spectrum.**

<table>
<thead>
<tr>
<th>Elements</th>
<th>Weight%</th>
<th>Atomic%</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>1.95</td>
<td>6.00</td>
</tr>
<tr>
<td>O</td>
<td>18.15</td>
<td>41.97</td>
</tr>
<tr>
<td>Si</td>
<td>0.14</td>
<td>0.19</td>
</tr>
<tr>
<td>Ca</td>
<td>0.15</td>
<td>0.14</td>
</tr>
<tr>
<td>Mn</td>
<td>13.57</td>
<td>9.14</td>
</tr>
<tr>
<td>Fe</td>
<td>53.06</td>
<td>35.15</td>
</tr>
<tr>
<td>Ni</td>
<td>1.09</td>
<td>0.69</td>
</tr>
<tr>
<td>Zn</td>
<td>11.89</td>
<td>6.73</td>
</tr>
<tr>
<td>Total</td>
<td>100.00</td>
<td></td>
</tr>
</tbody>
</table>
Fig. 6: EDX spectrum of Ni metal.

Table 2: Elements analysis of the nickel sample base on the EDX spectrum.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Weight%</th>
<th>Atomic%</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>3.25</td>
<td>13.87</td>
</tr>
<tr>
<td>O</td>
<td>0.66</td>
<td>2.11</td>
</tr>
<tr>
<td>Fe</td>
<td>1.34</td>
<td>1.23</td>
</tr>
<tr>
<td>Ni</td>
<td>94.75</td>
<td>82.78</td>
</tr>
<tr>
<td>Total</td>
<td>100.00</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 7 shows the hysteresis behavior of the ferrite sample. It illustrates a typical soft magnetic material behavior with very narrow hysteresis loop. This is due to the very low magnetic loss of the ferrites sample with very low coercively and relatively high saturation magnetization. The saturation magnetization of the specimens followed the modified Bloch’s law for ferromagnetic materials based on the modified spins wave spectrum of the particles owing to their finite size effects [12]. Fig. 8 shows the hysteresis behavior of pure nickel sample. It illustrates soft-like magnetic material behavior with a wider hysteresis loop. This is due to the higher magnetic loss of the nickel sample with higher coercivity and relatively higher saturation magnetization.
**Fig.7: Hysteresis loop of NiZnMn ferrite.**

**Fig.8: Hysteresis loop of Ni metal.**

**Conclusions**
A pure nickel metal sample and nickel-Zinc-Manganese ferrite sample could be obtained and then its structural and magnetic properties were studied for comparison. The
nickel metal showed an FCC structure, while the ferrite sample showed pure simple cubic spinel phase. The magnetic properties of the samples could be measured by using lab setup with an adequate accuracy. The magnetic properties of the metal showed a higher saturation and magnetic loss than that of the ferrite.

Acknowledgement
The authors would like to thank the ministry of higher education and scientific research for the financial support.

References