

CRYSTALLOGRAPHIC STRUCTURE AND MAGNETIC PROPERTIES OF PSEUDOBROOKITE $Fe_{2-x}Ni_xTiO_5$ SYSTEM ($x = 0, 0.1, 0.2, 0.3, 0.5$ and 1)

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ABSTRACT

CRYSTALLOGRAPHIC STRUCTURE AND MAGNETIC PROPERTIES OF PSEUDOBROOKITE $Fe_{2-x}Ni_xTiO_5$ SYSTEM ($x = 0, 0.1, 0.2, 0.3, 0.5$ AND 1). Crystallographic structure and magnetic properties of pseudobrookite $Fe_{2-x}Ni_xTiO_5$ system ($x = 0, 0.1, 0.2, 0.3, 0.5$ and 1) have been performed through solid state reaction. Pseudobrookite $Fe_{2-x}Ni_xTiO_5$ system was synthesized by mixing of Fe_2O_3 , NiO, and TiO_2 with stoichiometry composition using wet mill. The mixture was milled for 5 hours and sintered in the electric chamber furnace at $1000^\circ C$ in the air at atmosphere pressure for 5 hours. The refinement against of X-ray diffraction data shows that the samples with composition of ($x = 0$) and ($x = 0.1$) have a single phase with Fe_2TiO_5 structure. However the samples with composition of ($x > 0.1$) consist of multiple phases, namely $Fe_{2-x}Ni_xTiO_5$, $FeTiO_3$, Fe_2NiO_4 and NiO. Particle morphologies of the composition $x = 0$ and $x = 0.1$ are homogenous and uniform on the sample surface with a polygonal particle shape and particle size varies. At room temperature, the sample with $x=0$ is paramagnetic and that with $x=0.1$ is ferromagnetic. Magnetic phase transformation of this study is caused by the presence of Ni substituted Fe in the system. Thus substitution Ni into Fe on the system pseudobrookite Fe_2TiO_5 only capable of 0.1 at.% without changing the crystal structure of the material. It means that there is an interaction between the magnetic spin Fe^{3+} on the $3d^5$ configurations and Ni^{2+} on the $3d^3$ configurations through the mechanism of double exchange. Double exchange mechanism is a magnetic type of exchange that appears between the ions Fe^{3+} and Ni^{2+} adjacent in different oxidation states.

Keywords: Pseudobrookite, $Fe_{2-x}Ni_xTiO_5$, Substitution, Crystal structure, Magnetic

ABSTRAK

STRUKTUR KRISTAL DAN SIFAT MAGNETIK DARI BAHAN PSEUDOBROOKITE SISTEM $Fe_{2-x}Ni_xTiO_5$ ($x = 0, 0.1, 0.2, 0.3, 0.5, \text{ DAN } 1$). Analisis struktur kristal dan sifat magnetik dari bahan pseudobrookite sistem $Fe_{2-x}Ni_xTiO_5$ ($x = 0, 0.1, 0.2, 0.3, 0.5, \text{ dan } 1$) hasil reaksi padatan telah dilakukan. Bahan pseudobrookite sistem $Fe_{2-x}Ni_xTiO_5$ disintesis dengan mencampurkan bahan baku Fe_2O_3 , NiO, dan TiO_2 dengan komposisi stoikiometri menggunakan metode *milling* basah. Campuran *dimilling* selama 5 jam dan *disinter* dalam tungku listrik pada $1000^\circ C$ di udara pada tekanan atmosfer selama 5 jam. Hasil *refinement* pola difraksi sinar-X menunjukkan bahwa sampel dengan komposisi ($x = 0$) dan ($x = 0.1$) memiliki fase tunggal dengan struktur Fe_2TiO_5 . Namun sampel dengan komposisi ($x > 0.1$) terdiri dari multi-fase, yaitu $Fe_{2-x}Ni_xTiO_5$, $FeTiO_3$, Fe_2NiO_4 dan NiO. Morfologi partikel komposisi $x = 0$ dan $x = 0.1$ memiliki homogenitas yang cukup baik dan seragam di seluruh permukaan sampel dengan bentuk partikel poligonal dan ukuran partikel bervariasi. Sifat magnetik dari komposisi $x = 0$ adalah paramagnetik pada suhu kamar. Sementara komposisi $x = 0.1$ berubah menjadi ferromagnetik pada suhu kamar. Transformasi fasa magnetik ini disebabkan oleh kehadiran Ni yang telah berhasil mensubstitusi sebagian atom Fe. Namun substitusi Ni ke dalam Fe pada sistem pseudobrookite Fe_2TiO_5 hanya mampu sebesar 0,1 % tanpa mengubah struktur kristal dari bahan ini. Ini berarti bahwa ada interaksi antara *spin* magnetik Fe^{3+} pada konfigurasi $3d^5$ dan Ni^{2+} pada konfigurasi $3d^3$ melalui mekanisme pertukaran ganda. mekanisme pertukaran ganda adalah jenis magnetik pertukaran yang muncul antara ion Fe^{3+} dan Ni^{2+} berdekatan di bilangan oksidasi yang berbeda.

Kata kunci: Pseudobrookite, $Fe_{2-x}Ni_xTiO_5$, Substitusi, Struktur kristal, Magnetik

INTRODUCTION

Absorber material of electromagnetic wave is now becoming one of the interesting topics to be studied and understood. The electromagnetic wave absorber materials began to attract attention since the emergence of the phenomenon of electromagnetic wave interference (EMI) on several electronic components that work at high frequencies along with the development of telecommunications technology. This EMI can degrade the performance of the electronic components. Characteristics intrinsic and extrinsic of electromagnetic wave absorber material make a very unique phenomenon to be studied. Because the main requirement is needed as a material absorbing electromagnetic waves is the presence of intrinsic characteristics (properties of magnetic loss and dielectric loss) on the material in addition to the extrinsic characteristics (factors of the geometry of the particles).

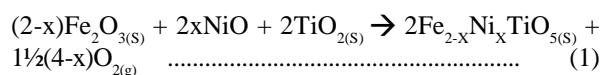
At first the absorber materials developed until now are a carbon-based material that has a high dielectric loss [1]. However recently too is found many studies of electromagnetic wave absorber materials began to lead to the use of magnetic materials, especially ferromagnetic [2-4]. Ferromagnetic material is believed to have a high permeability which is expected through the structure engineering may have high magnetic loss. But it is very interesting to study if these materials are paramagnetic at room temperature. It means that the paramagnetic material is necessary a structure engineering in order to transform into a ferromagnetic phase [5]. This study has been done structure engineering on the paramagnetic material of pseudobrookite Fe_2TiO_5 -based. Main reason for choosing this system is the magnetic material of pseudobrookite Fe_2TiO_5 has a stable structure up to high temperatures. Pseudobrookite Fe_2TiO_5 may also be obtained from the phase transformation of ilmenite (FeTiO_3) [6], while ilmenite itself can be obtained from iron sand where their reserves were very large in Indonesia. Therefore, this material becomes excellent products at low prices as well as raw materials are easily obtained.

Pseudobrookite Fe_2TiO_5 previously can be used for multiple applications such as microelectronics materials, gas sensors, non-linear optics, magnetic applications, filter optics [7], photocatalyst [8], photo electrode, anode batteries [9], pigments, and a membrane at high temperatures for fuel cell applications [10]. Thus the application of Pseudobrookite Fe_2TiO_5 material is very wide and makes this material is one of the multi-functional materials so the further understanding of this material is very interesting to study. Several methods have been developed to synthesize this compound is by using a hydrothermal method [11], ball-milled [9], sol-gel processes [8], solid state [12], and chemical vapor deposition [13]. However, one of the simplest methods is to use a solid state reaction method through mechanical milling.

Pseudobrookite Fe_2TiO_5 is a semiconductor material and has the crystal structure of orthorhombic with space group Cmc. Pseudobrookite Fe_2TiO_5 has magnetic spin glass transition. Magnetic properties of pseudobrookite Fe_2TiO_5 material is paramagnetic phase at room temperature [14]. This research will be conducted preliminary studies for structural engineering on the pseudobrookite $\text{Fe}_{2-x}\text{Ni}_x\text{TiO}_5$ materials by solid state reaction using a wet milling process. The addition of nickel (Ni) atoms is expected able to replace most of the position of the iron (Fe) atoms so that the interaction between the magnetic spin Fe^{3+} ions with Ni^{2+} occur it and can affect the magnetic properties of this material. Thus the aim of this study is to synthesize and characterize pseudobrookite $\text{Fe}_{2-x}\text{Ni}_x\text{TiO}_5$ material with variations in composition ($x = 0, 0.1, 0.2, 0.3, 0.5$ and 1). The discussion will be focused on the analysis of the crystal structure (phase and parameter structures), morphology of particles and magnetic properties of the pseudobrookite $\text{Fe}_{2-x}\text{Ni}_x\text{TiO}_5$ due to the addition of Ni atoms in the system.

EXPERIMENTAL METHOD

Pseudobrookite $\text{Fe}_{2-x}\text{Ni}_x\text{TiO}_5$ was synthesized by solid state reaction method with variations in composition ($x = 0, 0.1, 0.2, 0.3, 0.5$, and 1). The raw materials are used $\alpha\text{-Fe}_2\text{O}_3$, TiO_2 (anatase) and NiO, by following Equation (1).



The three materials were weighed according to the calculation of stoichiometric composition, then mixed and placed in a media that is made of stainless steel. Once it is added ethanol and balls are also made of stainless steel with a mass ratio between the balls and the material are 1: 2. The mixture is then milled for 5 hours using high energy milling equipment of Spex8000. The mixture of milling result is then dried and compacted with a pressure of 5000 psi. Furthermore, samples were sintered at 1000 °C for 5 hours in a furnace.

Crystalline phase identification was measured by X-Ray Diffractometer (XRD), Shimadzu type XD610. Measurement of the diffraction pattern using X-ray tube with a wavelength of $\lambda = 1.5406 \text{ \AA}$, mode: continuous scan, step size: 0.02°, and time per step: 0.5 seconds, and qualitative-quantitative phases analysis formed in the sample was used by GSAS software (General Structure Analysis System). While the particle morphologies were observed by using Scanning Electron Microscope (SEM) of JEOL type JED 2300. The magnetic properties were measured by using a Vibrating Sample Magnetometer (VSM) of Oxford type VSM1.2H instrument.

RESULTS AND DISCUSSION

Figure 1 shows the results of measurements of X-ray diffraction pattern of the sample pseudobrookite $Fe_{2-x}Ni_xTiO_5$ with variations in composition ($x = 0, 0.1, 0.2, 0.3, 0.5, \text{ and } 1$).

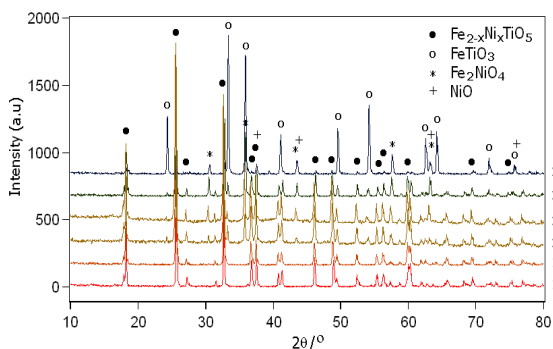
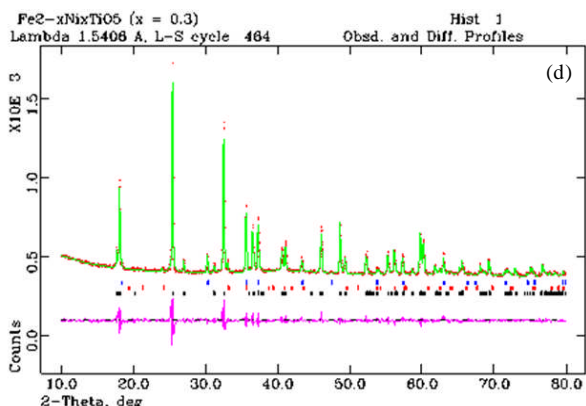
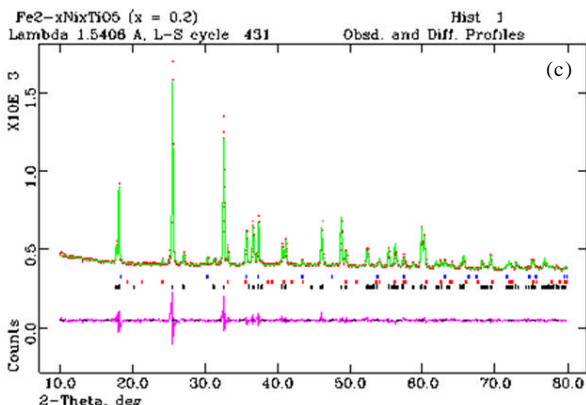
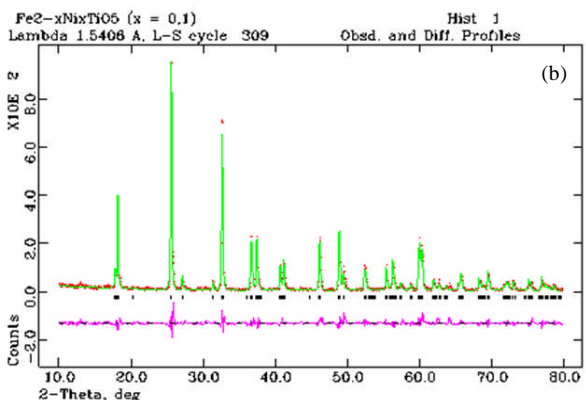
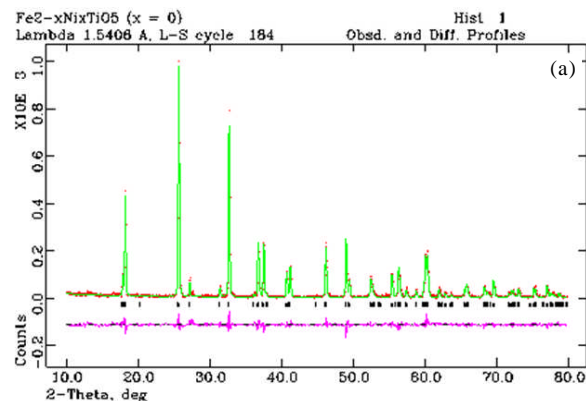


Figure 1. X-ray diffraction pattern of the sample pseudobrookite $Fe_{2-x}Ni_xTiO_5$ with variations in composition ($x = 0, 0.1, 0.2, 0.3, 0.5, \text{ and } 1$)

Based on the results of phase identification appears that the reaction has been successfully formed a single phase $Fe_{2-x}Ni_xTiO_5$ is on the composition of $x = 0$ and $x = 0.1$, while for the composition of $0.1 < x < 0.5$, the sample can not react perfectly so the sample consists of three phases, namely phases of $Fe_{2-x}Ni_xTiO_5$, $FeTiO_3$, and Fe_2NiO_4 . More reaction unperfectly occurs on the composition $x = 1$, which results in four phases, namely phases of $Fe_{2-x}Ni_xTiO_5$, $FeTiO_3$, Fe_2NiO_4 and NiO . Results of phase identification is very interesting to understand because for $x = 0.1$ shows that atoms of nickel has succeeded in substituting partially of the atoms Fe in the structure $Fe_{2-x}Ni_xTiO_5$, and for $x > 0.1$ is thought to occur reaction inbalance when the amount of Fe content is reduced meanwhile the Ni content increased. In Figure 3 appears too that with increasing Ni content ($x > 0.1$) in the sample, cause the phases of $FeTiO_3$, Fe_2NiO_4 and NiO increasing. Thus required further analysis to determine the changes of the crystal structure parameters, the amount of mass fraction formed, and cationic distribution on the results of substitution Ni into the Fe atom.

In Figure 2 shows the results of refinement X-ray diffraction data of the sample pseudobrookite $Fe_{2-x}Ni_xTiO_5$ with variations in composition ($x = 0, 0.1, 0.2, 0.3, 0.5, \text{ and } 1$).

Figure 2(a) and 2(b) are the result of refinement of the XRD patterns for $x = 0$ and 0.1 that have been formed Bragg diffraction peaks with a single phase following the Fe_2TiO_5 structure. Figure 2(c) to (e) are the result of refinement of the XRD patterns for $x = 0.2, 0.3, \text{ and } 0.5$ have been formed bragg diffraction peaks with a multi-phase, which follows the structure Fe_2TiO_5 , $FeTiO_3$ and Fe_2NiO_4 . While Figure 2(f) is the result of refinement of the XRD patterns for $x = 1$ which has been formed bragg diffraction peaks with a multi-phase, which



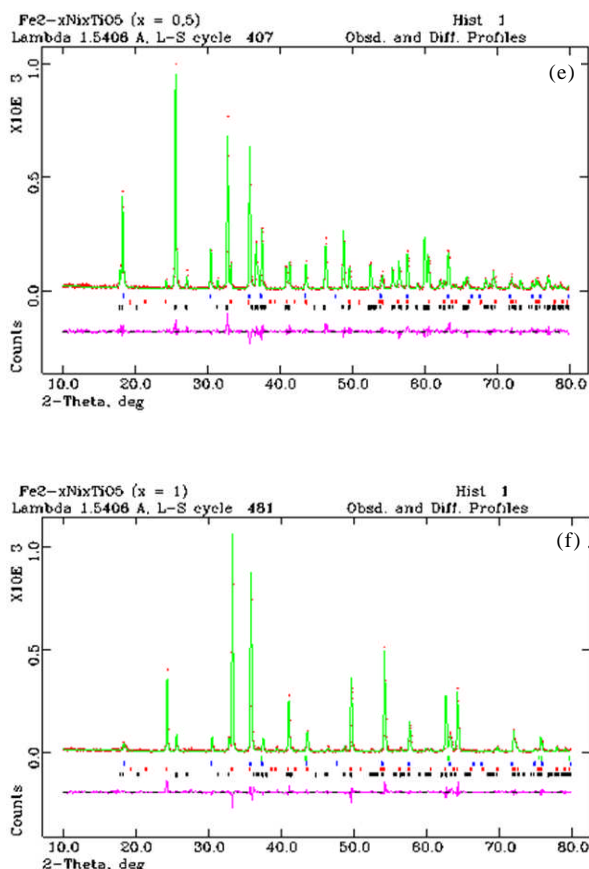


Figure 2. The results of refinement X-ray diffraction pattern of the sample pseudobrookite $Fe_{2-x}Ni_xTiO_5$, (a). $x = 0$, (b). $x = 0,1$, (c). $x = 0,2$, (d). $x = 0,3$, (e). $x = 0,5$, (f). $x = 1$.

follows the structure Fe_2TiO_5 , $FeTiO_3$, Fe_2NiO_4 and NiO . Qualitative and quantitative analysis refers to the Crystallography Open Database with the card number (COD: 1011342), (COD: 9010915), (COD: 2300295) and (9008693) respective for phases of Fe_2TiO_5 , $FeTiO_3$, Fe_2NiO_4 and NiO .

Refinement complete summary of the results of X-ray diffraction pattern of the sample pseudobrookite $Fe_{2-x}Ni_xTiO_5$ with variations in composition ($x = 0, 0.1, 0.2, 0.3, 0.5$, and 1) for all of samples are shown in Table 1.

Figure 2 and Table 1 show that the refinement results of X-ray diffraction pattern has a very good fitting quality based on the criteria of fit (R_{wp}) and goodness of fit (χ^2) in accordance with the agreement [15]. R_{wp} is the weight ratio of the difference between the XRD pattern of observation and calculation (ideal value of $R_{wp} < 10\%$). While χ^2 (chi-squared) is the ratio of the XRD pattern of observation results comparable with expectations (ideal value of $1 < \chi^2 < 1.3$).

In Table 2 shows that based on the refinement pattern of X-ray diffraction samples pseudobrookite $Fe_{2-x}Ni_xTiO_5$ with variations in composition ($x = 0, 0.1, 0.2, 0.3, 0.5$, and 1), the sample has a single phase is in the composition $x = 0$ (Fe_2TiO_5) and $x = 0.1$ ($Fe_{1.9}Ni_{0.1}TiO_5$). This refinement results are also supported by observations of the surface morphology of particles for both single phase using SEM as shown in Figure 3.

In Figure 3 shows that the particle morphologies of the composition $x = 0$ has a good particle homogeneity and uniform in across the sample surface with a polygonal particle shape and the particle size varies from $2 \mu m$ to $10 \mu m$. The similar with the particle morphologies of the composition $x = 0.1$ has a good homogeneity and uniform too with polygonal particle shape and particle size varies, but relatively more small compared with the composition $x = 0$ from $1 \mu m$ to $5 \mu m$. In general a single phase characteristics of polycrystalline samples by observation of SEM image is homogeneity and uniformity particle shape in across the sample surface.

The interesting thing to studied are that both have a single phase with the same structure, but their composition are different. On the composition $x = 0.1$, there is a 0.1% Ni atoms have succeeded substituting

Table 1. The value of structure parameters, criteria of fit (R_{wp}), goodness of fit (χ^2) and the mass fraction of phase formed in the sample pseudobrookite $Fe_{2-x}Ni_xTiO_5$ with variations in composition ($x = 0, 0.1, 0.2, 0.3, 0.5$, and 1).

Lattice parameter (Å)			V (Å ³)	ρ (g/cm ³)	Fraction wt%	R_{wp} (%)	χ^2
a	b	c					
3.7348(2)	9.7785(5)	9.9683(5)	364.05(4)	4.382	100.00	2.22	1.26
3.7262(2)	9.7639(6)	9.9516(6)	362.07(5)	4.274	100.00	2.19	1.24
3.7332(1)	9.7713(4)	9.9629(3)	363.43(2)	4.290	91.73		
5.0354(9)	5.0354(9)	13.766(4)	302.2(1)	5.001	4.09	2.21	1.21
8.334(2)	8.334(2)	8.334(2)	579.0(4)	5.312	4.18		
3.7368(1)	9.7664(3)	9.9595(3)	363.48(2)	4.290	82.64		
5.031(1)	5.031(1)	13.695(7)	302.0(3)	5.035	4.43	2.33	1.23
8.3356(9)	8.3356(9)	8.3356(9)	579.1(1)	5.311	12.93		
3.7413(1)	9.7519(5)	9.9564(5)	363.26(4)	4.196	68.58		
5.030(1)	5.030(1)	13.775(5)	301.8(1)	5.008	5.82	2.92	1.22
8.3300(7)	8.3300(7)	8.3300(7)	578.0(1)	5.321	25.59		
3.7462(9)	9.737(2)	9.936(2)	362.4(2)	3.969	8.79		
5.0315(1)	5.0315(1)	13.7762(7)	302.04(3)	5.005	75.30		
8.329(1)	8.329(1)	8.329(1)	577.8(3)	5.323	3.56	2.86	1.29
4.173(2)	4.173(2)	4.173(2)	72.6(1)	6.827	12.35		

Table 2. Cationic distribution on the $Fe_{2-x}Ni_xTiO_5$ ($x = 0.1$) was calculated from XRD data.

Atom	Site	Point symmetry	$Fe_{2-x}Ni_xTiO_5$ ($x = 0.1$)	
			Fe	Ni
Fe	$8f_1$	m	0.94 (94 at.%)	0.06 (6 at.%)
Composition by GSAS result			$Fe_{1.88}Ni_{0.12}TiO_5$	

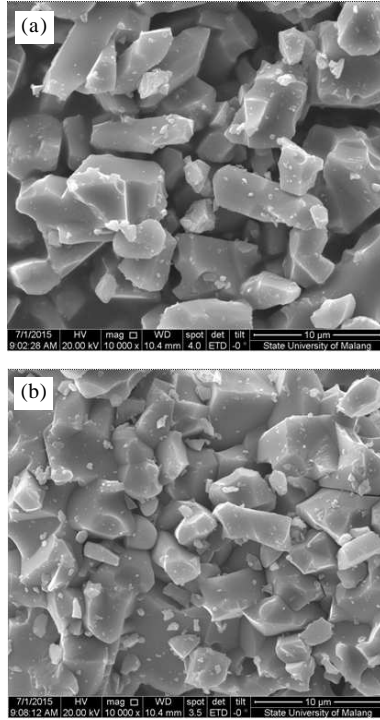


Figure 3. (a). The particle morphology of the pseudobrookite $Fe_{2-x}Ni_xTiO_5$ ($x = 0$); (b). The particle morphology of the pseudobrookite $Fe_{2-x}Ni_xTiO_5$ ($x = 0.1$).

partially Fe atoms that are expected to have an impact on other properties, especially on their magnetic properties. In this case needs to be carried out analysis of the cationic distribution on site of Fe atoms by means refine occupation factor for each Fe site that has been added Ni. The structure of Fe_2TiO_5 is an orthorhombic with space group (C mcm) and point symmetry (mmm). The position of Fe atoms occupy the Wyckoff position on the $8f_1$ site, while the position of Ti atoms occupy the Wyckoff position on the $4c_1$ site. A unit cell of Fe_2TiO_5 contains 20 atoms O, where 4 atoms O(1) occupy the same Wyckoff position on the $4c_2$ site, 8 atoms O(2) and 8 atoms O(3) respective occupy the Wyckoff positions on the sites of $8f_2$ and $8f_3$. The Ni atoms will occupy partially the Wyckoff position of Fe atoms on the $8f_1$ site. Cationic distribution of refinement results are shown in Table 2.

Because Ni has a valence of 2+, is thought to affect on the oxygen content. Since XRD can not refine the oxygen content, it is necessary a structure analysis using neutron diffraction facilities, but in this study has

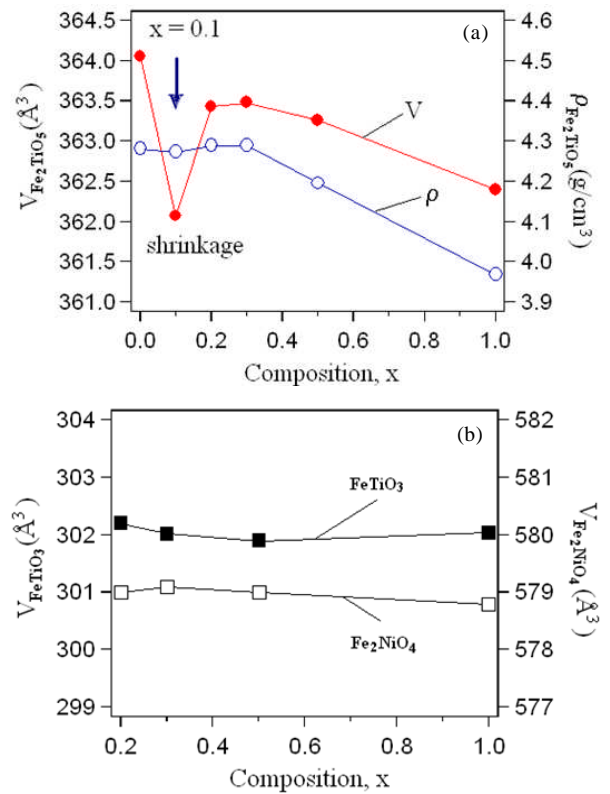


Figure 4. The volume of unit cells as a function of composition (a). Fe_2TiO_5 (b). $FeTiO_3$ and Fe_2NiO_4

not been conducted. Data supporting the other is from the analysis of changes in the volume of the unit cell as shown in Figure 4.

In Figure 4(a) it appears that the composition $x = 0.1$ occurs shrinkage of unit cell volume. This is due to the atomic radius Ni ($r = 1.62 \text{ \AA}$) is shorter than the atomic radius of Fe ($r = 1.72 \text{ \AA}$), thus resulting in lattice parameter is also reduced for the third axis. Then for the composition $x = 0.2$ and $x = 0.3$, the unit cell volume increased again and gradually declined allegedly caused the lattice distortion by the presence of another phase is formed. The addition of Ni excess ($x > 0.1$) results in an imbalance of the reaction so that Ni prefers to bind Fe to form Ferronickel, since the composition of these compounds is relatively stable compared with $Fe_{2-x}Ni_xTiO_5$. While another Fe will bind to Ti forming $FeTiO_3$ and Fe_2NiO_4 . It can be described as in Figure 4 (b) that on the composition of $x > 0.1$, no change in the unit cell volume on the phases of $FeTiO_3$ and Fe_2NiO_4 . It means that Ni has reacted with Fe to form Fe_2NiO_4 .

Besides, the presence of Ni can affect the magnetic properties of these systems. In Figure 5 is shown the results of measurement of magnetic properties by using VSM.

From the hysteresis curve shows that samples with composition $x = 0$ (Fe_2TiO_5) has paramagnetic behavior and marked with the magnetization response on the external magnetic field is a linear. While the composition $x = 0.1$, the magnetic properties of materials

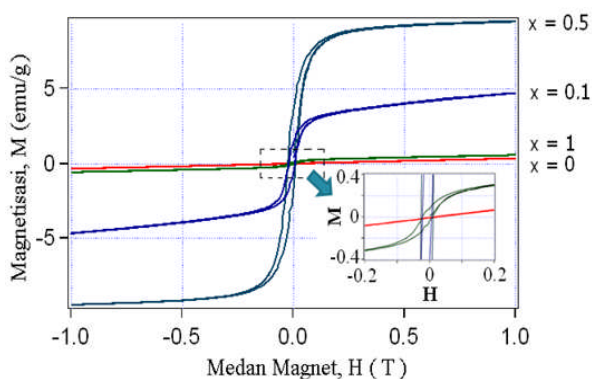


Figure 5. Hysteresis curve of the pseudobrookite $Fe_{2-x}Ni_xTiO_5$

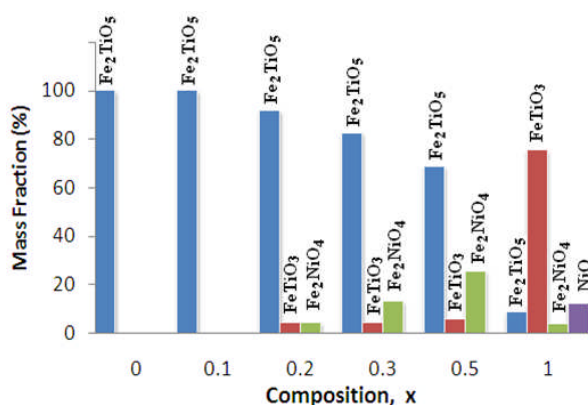


Figure 6. Mass fraction as a function of composition

transformed into ferromagnetic behavior. It is thought to have come from the presence of Ni in the structure Fe_2TiO_5 . It means that there is an interaction between the magnetic spin Fe^{3+} on the $3d^5$ configuration and Ni^{2+} on the $3d^8$ configuration through the mechanism of double exchange. Double exchange mechanism is a magnetic type of exchange that appears between the ions Fe^{3+} and Ni^{2+} adjacent in different oxidation states. It means is the displacement of the electron spins are parallel on the nearest neighbor by performing twice hopping simultaneously from ions of Fe^{3+} to Ni^{2+} through O^{2-} . The increase Ni furthermore ($x = 0.5$) result ferromagnetic behavior more high. Looks on the value of the saturation magnetization increased in the sample $x = 0.5$. This can be explained accordance to the results of the analysis of mass fractions as shown in Figure 6. Increasing the value of magnetic saturation on the composition $x = 0.5$ is contribution of phase of Fe_2NiO_4 around 25.59 wt%. In the composition $x = 1$, the value of magnetic saturation decreased dramatically, since the amount of mass fraction of phase Fe_2NiO_4 also reduced up to 3.56 wt%, meanwhile the phase of $FeTiO_3$ and Fe_2TiO_5 have paramagnetic behavior at room temperature. Thus the substitution of Ni into Fe on pseudobrookite Fe_2TiO_5 system is only capable of 0.1 at.% without changing the crystal structure of the material. The this result different with previously

study in which the substitution of Mn into Fe on pseudobrookite Fe_2TiO_5 system is still capable of up to 0.3 at.% without changing the crystal structure of the material [16].

CONCLUSION

Synthesis of pseudobrookite $Fe_{2-x}Ni_xTiO_5$ with variations in composition ($x = 0, 0.1, 0.2, 0.3, 0.5, \text{ and } 1$) have been successfully carried out. The refinement pattern of X-ray diffraction samples pseudobrookite $Fe_{2-x}Ni_xTiO_5$ shows that the samples has a single phase is in the composition $x = 0$ (Fe_2TiO_5) and $x = 0.1$ ($Fe_{1.9}Ni_{0.1}TiO_5$). The particle morphologies of the composition $x = 0$ and $x = 0.1$ are homogenous and uniform in across the sample surface with a polygonal particle shape and the particle size varies. Thus substitution Ni into Fe on the pseudobrookite $Fe_{2-x}Ni_xTiO_5$ only capable of 0.1 at.% without changing the crystal structure of the material and can alter the magnetic properties of the paramagnetic behavior becomes ferromagnetic at room temperature.

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