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MICROWAVE ABSORPTION STUDY OF MANGANASE FERRITE IN X-BAND RANGE PREPARED BY SOLID STATE REACTION METHOD

Mashadi Mashadi, Yunasfi Yunasfi, Ade Mulyawan

Abstract

A research to study the microwave absorption properties of manganase ferrite in the X-band range have been conducted by using high energy milling technique. The synthesis of manganase ferrite ($Mn_{1+x}Fe_{2-x}O_4$) was performed using solid state reaction method with the material composition (x)MnO : (2-x) Fe_2O_3 (x = 0.25; 0.50; 0.75 and 1.0) according to the molar ratio. This powder mixture was being milled for 10 hours then sintered at 1200 °C temperature for 3 hours. Material characterization was done by using FTIR spectroscopy (Fourier Transform Infra Red Spectroscopy) to observe the functional group, XRD (X-ray diffractometer) for phase identification, SEM (Scanning Electron Microscope) for surface morphology observation and VNA (Vector Network Analyzer) to determine the ability of materials to absorb microwaves. Analysis by FTIR showed two absorption peaks in the range of ~ 446 and ~ 557 cm^{-1} were associated with the octahedral and tetrahedral sites in structure of $MnFe_2O_4$. Phase identification by XRD showed that the increasing content of Mn (above x=0.25 composition) caused a single phase of $MnFe_2O_4$ turned into two phases ($MnFe_2O_4$ and Fe_2O_3), this results correspond to the SEM results which showed the morphological structure of those compositions are inhomogenous. The Absorption of microwaves was also decreased along with the increasing of Mn content. The maximum refflection loss was reached in the composition of Mn (x=0.0) which equal to ~ 82%, while for the composition of Mn (x=0.0) only reached ~ 55%.

Keywords

Manganase Ferrite, milling technique, solid state reaction, microwave absorption

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MICROWAVE ABSORPTION STUDY OF MANGANESE FERRITE IN X-BAND RANGE PREPARED BY SOLID STATE REACTION METHOD

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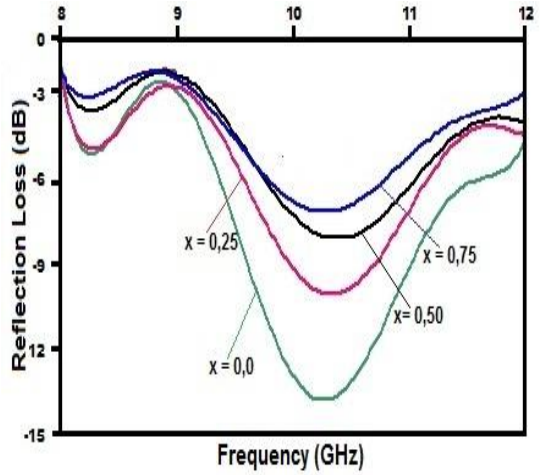

Abstract

A research to study the microwave absorption properties of manganese ferrite in the X-band range have been conducted by using high energy milling technique. The synthesis of manganese ferrite ($Mn_{1+x}Fe_{2-x}O_4$) was performed using solid state reaction method with the material composition (x)MnO : (2-x) Fe_2O_3 (x = 0.25; 0.50; 0.75 and 1.0) according to the molar ratio. This powder mixture was being milled for 10 hours then sintered at 1200 °C temperature for 3 hours. Material characterization was done by using FTIR spectroscopy (Fourier Transform Infra Red Spectroscopy) to observe the functional group, XRD (X-ray diffractometer) for phase identification, SEM (Scanning Electron Microscope) for surface morphology observation and VNA (Vector Network Analyzer) to determine the ability of materials to absorb microwaves. Analysis by FTIR showed two absorption peaks in the range of ~ 446 and ~ 557 cm^{-1} were associated with the octahedral and tetrahedral sites in structure of $MnFe_2O_4$. Phase identification by XRD showed that the increasing content of Mn (above x=0.25 composition) caused a single phase of $MnFe_2O_4$ turned into two phases ($MnFe_2O_4$ and Fe_2O_3), this results correspond to the SEM results which showed the morphological structure of those compositions are inhomogenous. The Absorption of microwaves was also decreased along with the increasing of Mn content. The maximum refflection loss was reached in the composition of Mn (x=0.0) which equal to ~ 82%, while for the composition of Mn (x=0.0) only reached ~ 55%.

Keywords: Manganese Ferrite, milling technique, solid state reaction, microwave absorption

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1.0 INTRODUCTION

In this era, designing and developing electronic devices materials which suitable for high frequency range application is still a great challenge for scientist due to the problem of the electromagnetic interference (EMI). The utilization of the Radar Absorbing Materials (RAM) especially for military purposes is a huge command to do, it was meant to reduce radar cross section. RAM is a classification for materials which able to absorb the microwave in the X-band range (8-12 GHz). Almost all of the communication for military purposes worked under this range which made the demand for the development of the RAM either for military purposes or commercial applications increased rapidly in near future [1, 2].

There are several magnetic nanoparticle materials which have been used as main compound of microwave absorbing material such as Manganese ferrite. Manganese ferrite with chemical formula $MnFe_2O_4$ are very interesting to be used as an absorbing material due to several specific characteristics. Manganese ferrite is known have a high resistivity, high magnetization saturation, good chemical stability, and unilateral magnetic anisotropy which made them have a good microwave absorbing ability [3, 4]. Other than that, Mangan ferrite also known to have either dielectric or magnetic loss in radio frequency range which very important for RAM [5, 6].

There are numerous techniques that have been used to synthesize magnetic nanoparticle materials such as sol-gel method [7], milling technique [8], co-precipitation method [9], etc. In previous research, a single phase of nickel ferrite nanoparticle have been successfully synthesized using sol-gel method [10] and solid state reaction method by Yunasfi, *et al*. [11]. In this research, Manganese ferrite ($Mn_{1+x}Fe_{2-x}O_4$) was synthesized by using solid state reaction method by using High energy Milling (HEM). This methods was used because of the efficiency and low cost for massive production compared to other methods. A modified manganese ferrite in the form of $Mn_{1+x}Fe_{2-x}O_4$ was characterized to identify the phase formation and analyze the crystal structure of each modification and also to know the influence of each modification to its microwave absorption properties.

2.0 METHODOLOGY

The synthesize process of mangan ferrite $Mn_{1+x}Fe_{2-x}O_4$ have been done by solid state reaction method using milling technique. Iron oxide (Fe_2O_3) and mangan oxide (MnO_2) from sigma aldrich with purity more than ≥ 99.9% were used as a compound material. The composition of $(2+2x)MnO_2$: $(2-x)Fe_2O_3$ (x = 0,0 ; 0.25 ; 0,50 dan 0,75) was measured in accordance with the molal ratio in total 10 gram. Each composition was poured into stainless steel vial then added by stainless steel ball in 1:5 compound ratio and finally assisted by ethanol to start the milling process for 10 hours. After milling process, the mixture were dried in the oven to evaporate the ethanol then grinded until fine powder was formed, and finally followed by sintering process at 1200 °C for 3 hours.

Fine powder of each composition were characterized by using FTIR (Fourier Transform Infra Red Spectroscopy) do identify the functional group, XRD (X-ray diffraction) technique using Panalytical Phillips to confirm the phase formation and SEM-EDX (Scanning Electron Microscope) HITACHI type SU3500 to define the morphological structure of each composition, and finally all composition were characterized by using VNA (Vector Network Analyzer) machine type Advatest-R3370 in the range of 300 KHz - 20 GHz to define the microwave absorption properties.

3.0 RESULTS AND DISCUSSION

Diffraction patterns of all varied $Mn_{(1+x)}Fe_{(2-x)}O_4$ (x = 0; 0.25; 0.50; dan 0.75) which have been synthesized by using milling technique is shown in the Figure 1. In the composition of x=0.0 ($MnFe_2O_4$) and x = 0.25 ($Mn_{1,25}Fe_{1,7}O_4$), it can be noticed that a single phase of $MnFe_2O_4$, which had spinel structure with lattice parameters $a = b = c$ = 0,58836 nm (space group Fd3m), has successfully formed. The most intense peak in the position of 2θ = 35° is correspond to the crystal plane of [311] which is the main characteristic of spinel ferrite structure.

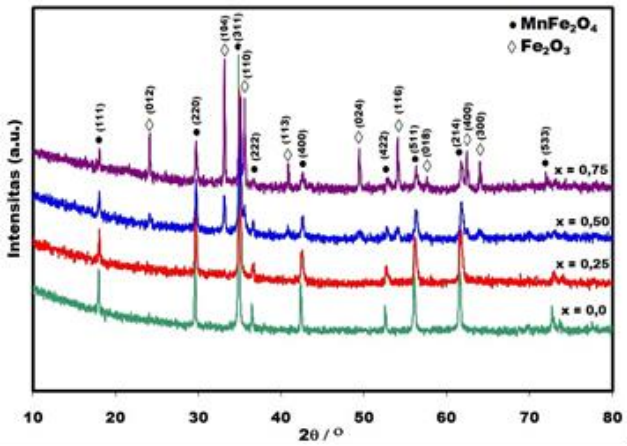

Figure 1 X-ray diffraction pattern for all variation of $Mn_{(1+x)}Fe_{(2-x)}O_4$ through milling process for 10 hours

The refinement results of all varied $Mn_{(1+x)}Fe_{(2-x)}O_4$ are shown in the Figure 2. All refinement results were figured out by It was also supported by several diffraction peaks which also correspond to crystal plane of $MnFe_2O_4$ the peaks at 2θ = 18°, 30°, 37°, 42°, 53°, 56°, 62° correspond to [111], [222], [400], [422], [511], [440], [533] respectively. All of this peaks are suitable to the diffraction database from ICDD 96-

100-6117 using match! software. These results are also correspond to other study which have been done by Pardhan who also studied about the formation of the $MnFe_2O_4$ [12].

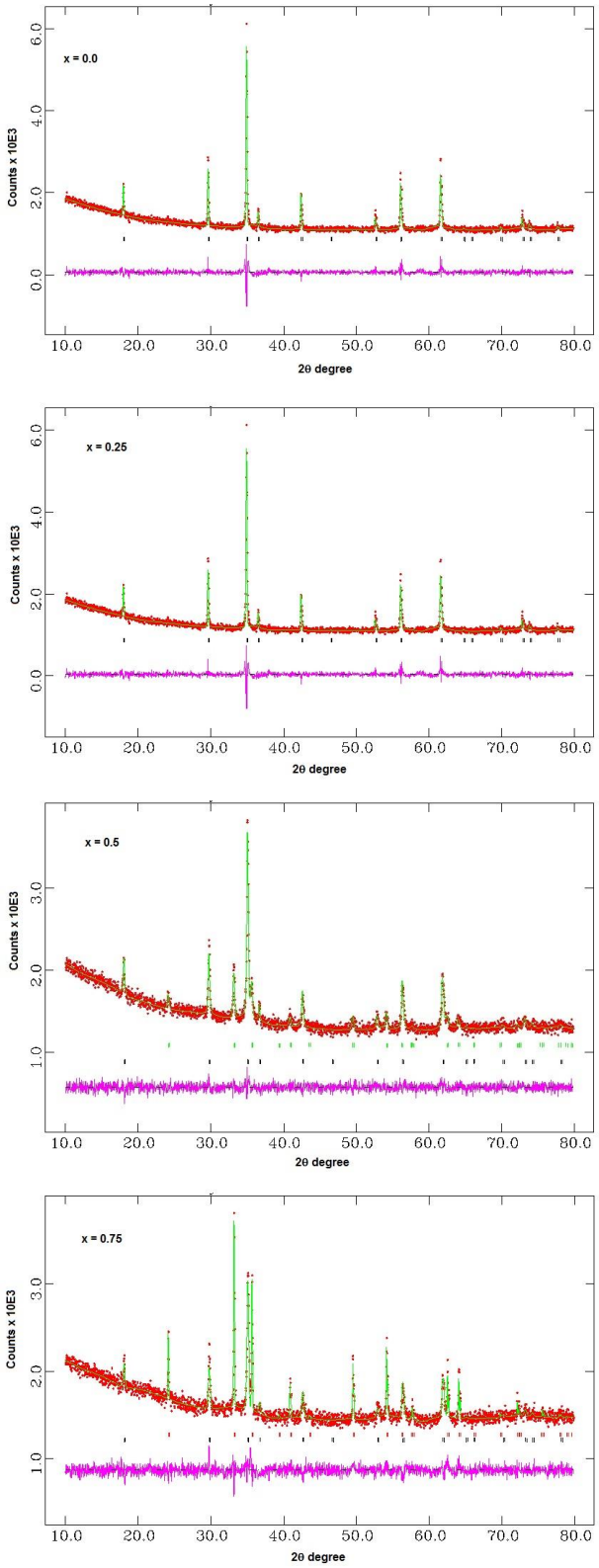

Figure 2 Refinement results for all variation of $Mn_{(1+x)}Fe_{(2-x)}O_4$ through milling process for 10 hours

As the result of the increasing contain of Mn^{2+} in the composition of x = 0.5 and 0.75, there are several additional peaks which appeared in the diffraction pattern, it means the composition are no longer in a single phase form, it also can be noticed from the intensity of the $MnFe_2O_4$ phase which gradually decreased along with the appearance of other phase. Referring to the results of the phase identification using Match! Software, it is known that the other phase which appeared in the diffraction pattern for composition x = 0.5 and 0.75 is Fe_2O_3 phase with diffraction database number ICDD 96-100-6117. All of the additional peaks in the position of 2θ = 24°, 33°, 36°, 41°, 49°, 54°, 58°, 61°, 64° are correspond to the crystal plane of Fe_2O_3 which are [012], [104], [110], [113], [024], [116], [018], [214], and [300] respectively.

The refinement results of all varied $Mn_{(1+x)}Fe_{(2-x)}O_4$ are shown in the Figure 3. All refinement results were figured out by using GSAS (General System Analysis System) which based on the rietveld method for fitting all of the diffraction patterns.

Tabel 1 Detailed refinement results for all variation of $Mn_{(1+x)}Fe_{(2-x)}O_4$ as the result of GSAS refinement

	X = 0	**X=0.25**	**X = 0.50**	**X = 0.75**
	#$MnFe_2O_4$ Phase			
Crystal structure	Cubic			
Space group	F d -3 m (227)			
Lattice parameters (Å)	$a = b = c = 8.5052$ $\alpha = \beta = \gamma = 90^o$	$a = b = c = 8.5039$ $\alpha = \beta = \gamma = 90^o$	$a = b = c = 8.4731$ $\alpha = \beta = \gamma = 90^o$	$a = b = c = 8.4749$ $\alpha = \beta = \gamma = 90^o$
V (Å^3)	615.273	614.965	608.313	608.715
% phase	100	100	93,1	50,1
	#Fe_2O_3 Phase			
Crystal structure			Trigonal	Trigonal
Space group			R – 3c (167)	R -3 c (167)
Lattice parameters (Å)			$a = b = 5.031$ $c = 13.718$, $\alpha = \beta = 90^o$ $\gamma = 120^o$	$a = b = 5.0288$ $c = 13.7170$ $\alpha = \beta = 90^o$ $\gamma = 120^o$
V (Å^3)			300.675	300.409
% phase	100	100	6,9	49,9
R factor				
Rwp	0.0358	0.036	0.0273	0.038
Rp	0.0263	0.0263	0.0217	0.0251
χ^2	1.6	1.698	1.105	1.382

From this fitting process we could exactly define the percentage of each phase which contained in those compositions with detailed lattice parameter as shown in the Table 1.

According to the result of the refinement by using GSAS, the fitting results between the actual data and the diffraction database shows a good and reliable match. It can be seen from the value of the goodness of fit (χ^2) which have a value between 1.3-1.8 and small different between Rwp and Rp as shown in Table 1. It have been confirmed that in the composition of x = 0 and 0.1 have formed a single phase of $MnFe_2O_4$, even so there is a slight difference of its volume, It happened attributed to the shrinkage of the lattice parameters because of the substitution in the composition of x=0.25. In the composition of x = 0.5 and 0.75, it can be confirmed that the percentage of Fe_2O_3 as an additional phase is increased along with the increasing contain of the substitution.

The results of IR spectra for all variation of $Mn_{(1+x)}Fe_{(2-x)}O_4$ are shown in Figure 3, all of the graphics were carried out by using FTIR with KBr method in the range of 400 - 1200 cm^{-1}. According to the Figure 3, all of the $Mn_{(1+x)}Fe_{(2-x)}O_4$ composition have 2 absorbance peaks in the range of 446 and 557 cm^{-1}, these two peaks are the main peaks which appeared as the functional group of metal ion and oxygen in the structure of the spinel ferrite structure, each peak correspond to the octahedral and tertrahedral site of the spinel ferrite structure respectively [13]. In $MnFe_2O_4$ phase, the Mn^{2+} cations are located in the octahedral site and the Fe^{3+} cations are located in the tetrahedral and oktahedral site. As the increasing contain of the substitution in the form of $Mn_{(1+x)}Fe_{(2-x)}O_4$, there is a slight shifting for the absorbance peaks which attributed to the formation of the additional phase of Fe_2O_3 referring to the diffraction pattern data.

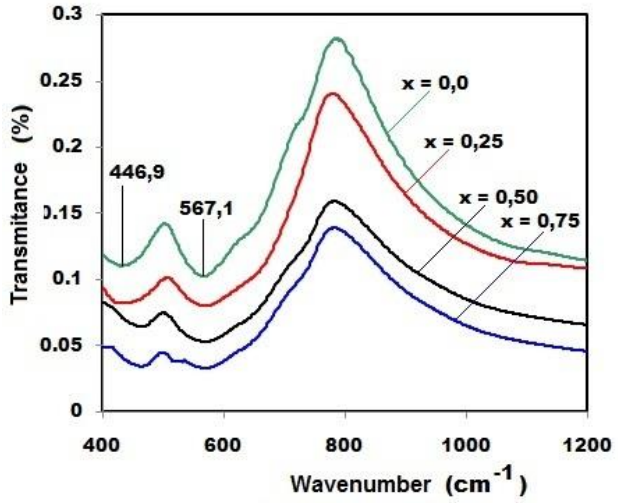

Figure 3 IR spectra for all variation of $Mn_{(1+x)}Fe_{(2-x)}O_4$ through milling process for 10 hours

The morphological surface for all variation of $Mn_{(1+x)}Fe_{(2-x)}O_4$ is shown in the Figure 4 with 5000x magnification to observe the dispersion and agglomeration of the particle. In the single phase composition which are x = 0 and 0.5, all of the particle are homogen with particle size around 200 nm. In the composition of x = 0.25 and 0.75 which are no longer in a single phase form, it can be seen that the particle were not in same size and tend to be agglomerated (varying from 200-500 nm), these results also supported the fact that the maximum amount to perform a single phase of $MnFe_2O_4$ supposed to be below x<0.25.

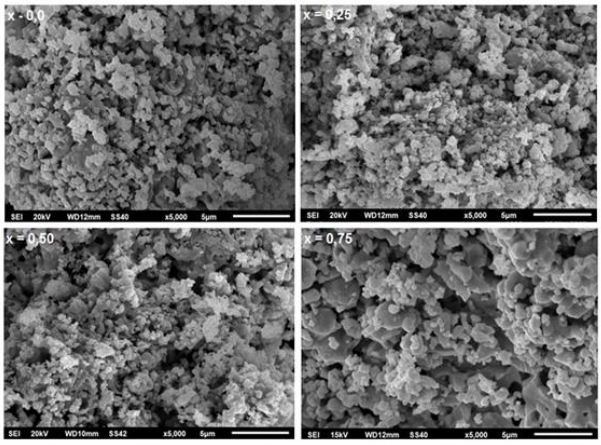

Figure 4 the mophological surface for all variation of $Mn_{(1+x)}Fe_{(2-x)}O_4$

The absorption of the microwave in X-band range (8-12 GHz) for all variation of $Mn_{(1+x)}Fe_{(2-x)}O_4$ in the Refflection loss curve is shown in Figure 5. Reflection loss shows the spin magnetic resonance between microwave and the magnetic materials, the depth of the reflection loss actually depends to some specific frequency, the thickness of the material, permittivity, permeability value and microstructure [5].

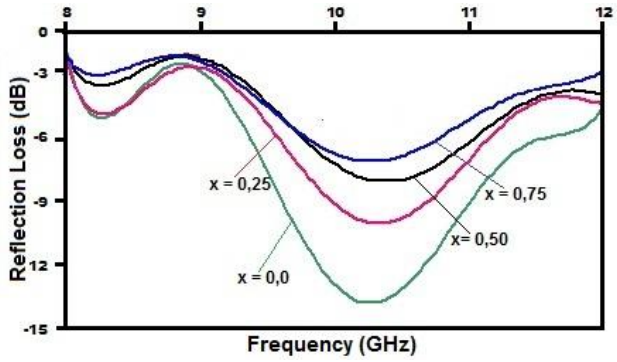

Figure 5 The refflection loss for all variation of the $Mn_{(1+x)}Fe_{(2-x)}O_4$

In Figure 5. it can be noticed that along with increasing contain of the substitution the reflection loss curve became more shallow. with detailed value in Table 2.

Table 2 The refflection loss value for all variation of the $Mn_{(1+x)}Fe_{(2-x)}O_4$

Composition (x)	Sample	Frquency (GHz)	Refflection Loss (dB)	Micro wave absorption (%)
0.0	$MnFe_2O_4$	10.20	-14.77	~82
0.25	$Mn_{1.25}Fe_{1.75}O_4$	10.18	-11.11	~72
0.5	$Mn_{1.5}Fe_{1.5}O_4$	10.15	-7.97	~60
0.75	Mn_2FeO_4	10.12	-7.15	~55

It happened because of the phase formation of the composition in a single phase form in which only $MnFe_2O_4$ phase made initial microwave is trapped due to the spin magnetic interaction mechanism with the material and made the curve to have a deep reflection loss. Unlike those single phase compositions. in the composition with the substitution abve x>0.25 which contained Fe_2O_3 phase have a shallos reflection value. It happened because Fe_2O_3 have an antiferromagnetic behavior and made the magnetic properties became weaker than those single phase form composition. this result is suitable with the result research that have been done by Muflihatun. *et al.* [9].

4.0 CONCLUSION

According to the results of this study, it can be concluded that magnetic nanoparticle of $MnFe_2O_4$ with each variation in the form of $Mn_{(1+x)}Fe_{(2-x)}O_4$ (x = 0; 0.25 ; 0.50 ; dan 0.75) have been successfully synthesized by using solid state reaction method with milling technique for 10 hours. It was known that a single phase form of $MnFe_2O_4$ was formed in the composition of x = 0 and 0.5. In the composition above x>0.25, the additional phase of Fe_2O_3 was formed. The results of the IR spectra showed that two absorbance in the range of ~446 and ~557 cm^{-1} peak are attributed to the octahedral site and tetrahedral site of $MnFe_2O_4$. In this study, it was known that the microwave absorption properties were decreased along with the increasing substitution. The deepest microwave absorption curve was belong to the composition of x = 0 in the value of ~82% and the composition of x = 0.6 only reached ~55%.

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