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Preface

The 4th International Conference on Advanced Materials Science and Technology 2016 (ICAMST-2016).

International Conference on Advanced Materials Science and Technology 2016 (ICAMST-2016) is an international forum for sharing knowledge and results in theory, computation, synthesis, characterization in all aspects of advanced materials and its technological application. This ICAMST is designated to promote research interests, knowledge sharing, and transfer, as well as to improve our common ground of science and technology.

The conference has brought together researchers and academicians from both academia as well as industry to meet and share cutting-edge development in the field. The Conference welcomes significant contributions in all major fields of theoritical & analyses, synthesis & Characterization, nanoscience, Functional Materials. This volume is a collection of 100 selected from 212 manuscripts by 503 authors. These papers were presented at the International Conference on Advanced Materials Science and Technology 2016 (ICAMST-2016) which was held in Malang, Indonesia, September 27 - 28, 2016. The selection of papers included in this volume was based on an international peer review procedure. We feel the variety of topics will be of interest to researchers.

I would like to thank:

- The Scientific Committee of ICAMST 2016 for their precious contribution.
- The distinguished keynote speakers and invited speakers for their difficult task and unique lectures Prof. Dr. Takayuki Ishida (Japan); Prof Dong-Sing Wuu (Taiwan), Assoc. Prof. Dr. Graeme R. Blake (The Netherlands), Prof. Dr. Andrivo Rusydi (Singapore), Prof. Dr. Robert Jann (Taiwan), Prof. Hadi Nur, Universiti Teknologi Malaysia (Malaysia), Prof. Dr. Darminto, Indonesia, Dr. Edy Giri Rachman Putra (Indonesia), Dr. Atsushi Okazawa (Japan), Dr. Hendrik Lintang (Indonesia), on their respective fields of expertise.
- The Organizing Committee Prof. Arif Hidayat (UM), Prof. Khairurijal (ITB), Assoc. Dr. Nandang Mufti, Dr. Sunaryono, Dr. Ahmad Taufiq, Assoc. Prof. Kuwat Triyana (UGM), for their dedication.
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I would also like to express my gratitude to all authors and contributing reviewers. Last but not least, my special thanks goes to the respectable the president of KoSaTeM Consortia, Prof. Khairurijal, and his team.

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Modification of Pseudobrookite $Fe_{2-X}Mn_xTiO_5$ with Solid State Reaction Method using a Mechanical Milling

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Abstract. Modification of pseudobrookite $Fe_{2-x}Mn_xTiO_5$ with solid state reaction method using a mechanical milling has been synthesized. Raw materials used to prepare these samples were Fe_2O_3, $MnCO_3$, and TiO_2. Fe_2O_3 and TiO_2 powders (ratio of 1:1) were mixed with $MnCO_3$ powder at various composition of x = 0; 0.1; 0.2; 0.3; 0.4; 0.5; and 1, which each composition was added with 50 ml ethanol and then milled for 5 hours through high energy milling, after that sintered at 1000 °C for 5 hours by using box furnace. The phases of $Fe_{2-x}Mn_xTiO_5$ were measured by using X-ray diffraction (XRD) and then identified by using Match program. The crystal structure was analyzed by using the program of General Structure Analysis System (GSAS). Quality fitting of Rwp and χ^2 (chi-squared) are relatively good because based on the curve of normalized error distribution looks just left background and its normal probability plot shows the value of comparable between observation and expectation. The refinement analyses of X-ray diffraction patterns showed that the samples formed single phase for $x \leq 0.3$. However, the samples of $x > 0.3$ were multi-phases. The single phase of sample had composition of pseudobrookite Fe_2TiO_5 with orthorhombic structure, space group of C m c m (63), the lattice parameters of a = 3.7390 Å, b = 9.7790 Å, and c = 9.9780 Å, $\alpha = \beta = \gamma = 90^o$, V = 364.83 $Å^3$, and $\rho = 4.360$ g.cm^{-3}. Meanwhile, the other phase analysis for the composition of $x > 0.3$ is bixbyite ($FeMnO_3$). The bixbyite has a cubic structure, under the space group of I a -3 (206), the lattice parameters of a = b = c = 9.40 Å, $\alpha = \beta = \gamma = 90^o$, V = 830.58 $Å^3$, and ρ = 5.078 g.cm^{-3}.

Keywords: Pseudobrookite, $Fe_{2-x}Mn_xTiO_5$, Milling, Modification, Crystal Structure.

1. Introduction

Absorber materials of electromagnetic waves is now becoming one of the interesting topics to be studied and understood for electronic application [1,2]. The main requirement needed as a material absorbing electromagnetic waves is the presence of intrinsic characteristics, namely magnetic loss and dielectric loss on the materials. At first, the absorber materials developed until now are a carbon-based material that has a high dielectric loss [3]. However, recently it is found that many studies of electromagnetic wave absorber materials began to lead to the use of magnetic materials [4-5]. This study has done structure engineering on the paramagnetic material of pseudobrookite Fe_2TiO_5-based. The main reason for choosing this system is the magnetic material has a stable structure up to high temperatures. Pseudobrookite Fe_2TiO_5 may also be obtained from the phase transformation of ilmenite ($FeTiO_3$) [6-7], while ilmenite itself can be obtained from the iron sand where their reserves is very

large in Indonesia. Therefore, this material becomes an excellent product at low price as well as the 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, photocatalyst, photoelectrode, anode batteries, pigments, and a membrane at high temperatures for fuel cell applications [8-10]. 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. Previous research has carried out preliminary studies about the effect of composition on the phase and parameter of crystal structure pseudobrookite $Fe_{2+x}Ti_{1+x}O_5$ [11]. This research conducted modification through structural engineering on the pseudobrookite $Fe_{2-x}Mn_xTiO_5$ materials by solid-state reaction using a wet milling process. The addition of nickel (Mn) 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 Mn^{3+} occur it and can affect the magnetic properties of this material. Thus, the aim of this study is to synthesize and characterize pseudobrookite $Fe_{2-x}Mn_xTiO_5$ material with variations in composition (x = 0.1, 0.2, 0.3, 0.4, 0.5 and 1). The discussion will be focused on the analysis of the crystal structure (phase and parameter structures), the morphology of particles and primary content of the pseudobrookite $Fe_{2-x}Mn_xTiO_5$ due to the addition of Mn atoms in the system.

2. Experimental Method

Pseudobrookite $Fe_{2-x}Mn_xTiO_5$ was synthesized by solid state reaction method with variations in composition (x = 0.1, 0.2, 0.3, 0.4, 0.5, and 1). The raw materials used were Fe_2O_3, TiO_2 (anatase) and $MnCO_3$. The three materials were weighed according to the calculation of stoichiometric composition, then mixed and placed in a media made of stainless steel. Once it was added with ethanol and balls also made of stainless steel with a mass ratio between the balls and the material are 1: 2. The mixture was then milled for 5 hours by using high energy milling equipment of Spex8000. The mixture of milling result was then dried and compacted with a pressure of 5000 psi. Furthermore, the samples were sintered at 1000 °C for 5 hours in a furnace.

Crystalline phase identification was measured by X-ray diffractometer (XRD), Pan Analytical. Measurement of the diffraction pattern by using X-ray tube with a wavelength of λ = 1.5406 Å, mode: continuous scan, step size: 0.02^o, and time per step: 0.5 seconds and qualitative-quantitative phases analysis formed in the sample was used by GSAS (General Structure Analysis System) software. Meanwhile, the particle morphology was observed by using transmission electron microscope (TEM) of JEOL brand.

3. Results and Discussion

Figure 1 shows the results of measurements of X-ray diffraction pattern of the sample pseudobrookite $Fe_{2-x}Mn_xTiO_5$ with variations in composition (x = 0.1, 0.2, 0.3, 0.4, 0.5, and 1). Based on the results of phase identification appears that the reaction successfully formed a single phase $Fe_{2-x}Mn_xTiO_5$ on the composition of x = 0.1 – 0.3, while for the composition of x > 0.3, the sample could not react perfectly so the sample consisted of two phases, namely phases of $Fe_{2-x}Mn_xTiO_5$ and $FeMnO_3$. Results of phase identification are fascinating to understand because for x = 0.1 – 0.3 show that atoms of manganese have succeeded in substituting partially of the atoms Fe in the structure $Fe_{2-x}Mn_xTiO_5$, and for x > 0.3 is thought to occur reaction imbalance when the amount of Fe content was reduced, the Mn content increased. In Figure 1 we can see that the increasing Mn content (x > 0.3) in the sample caused the phases of $FeMnO_3$ increased as well. Thus, it required further analysis to determine the changes in the crystal structure parameters, the amount of mass fraction formed, and cationic distribution on the results of substitution Mn into the Fe atom as shown in Figure 2.

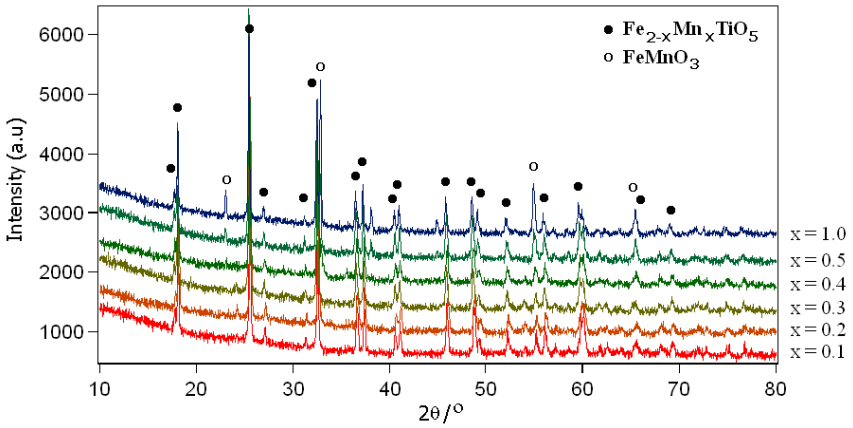
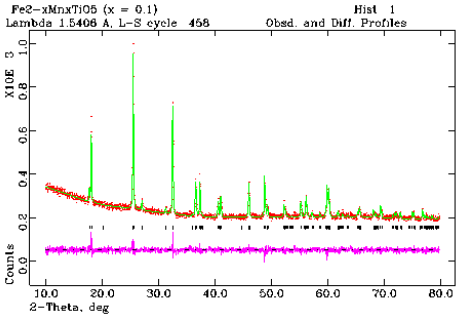
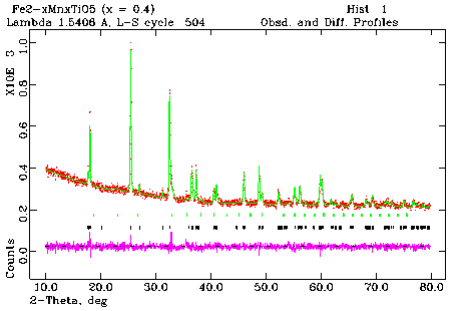


Figure 1. X-ray diffraction pattern of the sample pseudobrookite $Fe_{2-x}Mn_xTiO_5$ with variations in composition (x = 0.1, 0.2, 0.3, 0.4, 0.5, and 1)

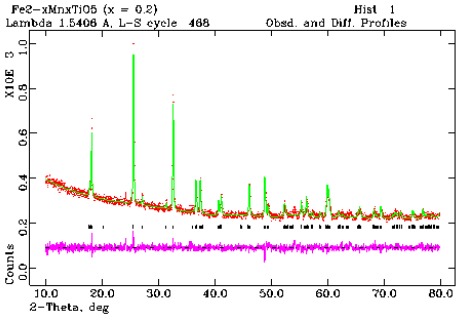
Figure 2 shows the results of refinement X-ray diffraction pattern of the sample pseudobrookite $Fe_{2-x}Mn_xTiO_5$ with the variations in composition (x = 0.1, 0.2, 0.3, 0.4, 0.5, and 1).



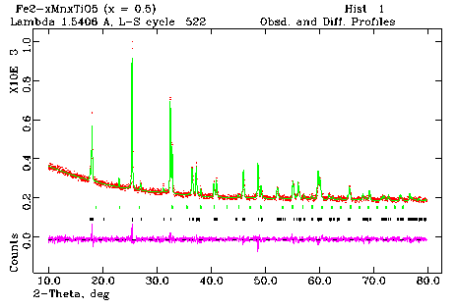
(a) x = 0.1



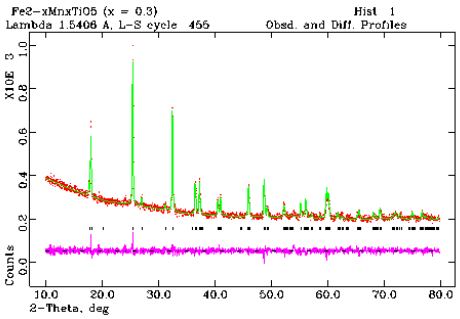
(d) x = 0.4



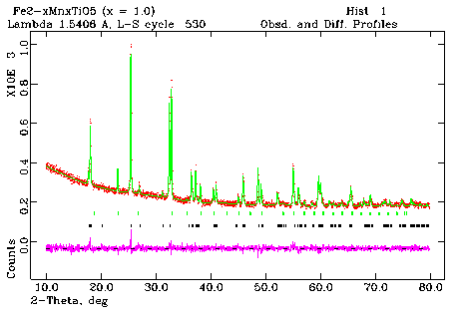
(b) x = 0.2



(e) x = 0.5



(c) x = 0.3



(f) x = 1

Figure 2. The refinement results of XRD pattern of the sample pseudobrookite $Fe_{2-x}Ni_xTiO_5$

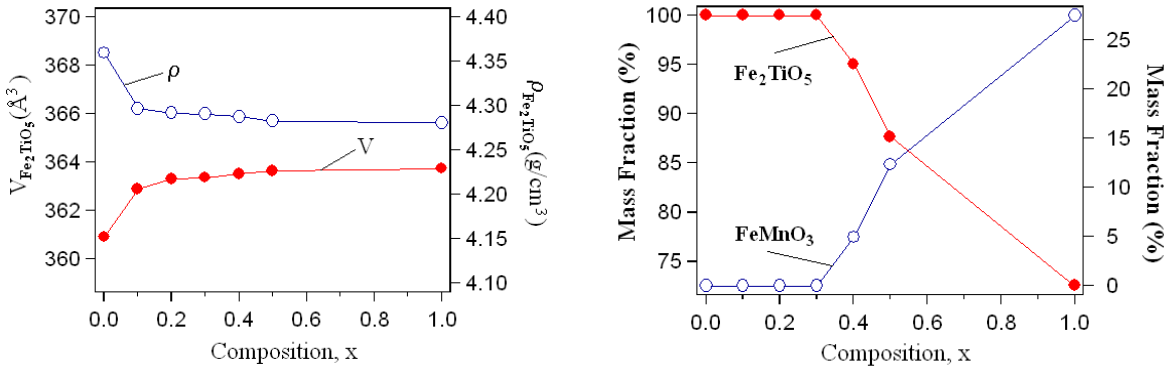
Figure 2(a – c) show the results of refinement of the XRD patterns for x = 0.1 – 0.3 that formed Bragg diffraction peaks with a single phase following the Fe_2TiO_5 structure. Figure 2(d – f) show the results of refinement of the XRD patterns for x = 0.4 – 1.0 that formed Bragg diffraction peaks with a multi-phases, which follows the structure of Fe_2TiO_5 and $FeMnO_3$. Qualitative and quantitative analysis refers to the Crystallography Open Database with the card number (COD: 1011342) and (COD: 9008068) respective for phases of Fe_2TiO_5 and $FeMnO_3$.

Refinement complete summary of the X-ray diffraction pattern results of the pseudobrookite $Fe_{2-x}Mn_xTiO_5$ sample with variations in composition (x = 0.1, 0.2, 0.3, 0.4, 0.5, and 1) for all of samples is shown in Table 1.

Table 1. The value of structure parameters, criteria of fit (R_{wp}), the goodness of fit ($\chi 2$) and the mass fraction of phase formed in the pseudobrookite $Fe_{2-x}Mn_xTiO_5$ sample with the variations in composition (x = 0.1, 0.2, 0.3, 0.4, 0.5, and 1).

Mn	Phase	Lattice parameter (Å)			V	ρ	Fraction	R_{wp}	χ^2
(*x*)		*a*	*b*	*c*	(Å^3)	(g/cm^3)	wt%	(%)	
0 [11]	Fe_2TiO_5	3.7188(4)	9.7569(1)	9.9466(1)	360.90(2)	4.360	100.00	-	-
0.1	Fe_2TiO_5	3.7254(1)	9.7766(4)	9.9636(4)	362.89(3)	4.297	100.00	3.29	1.25
0.2	Fe_2TiO_5	3.7287(2)	9.7790(6)	9.9634(6)	363.30(5)	4.292	100.00	3.42	1.29
0.3	Fe_2TiO_5	3.7292(2)	9.7791(6)	9.9636(5)	363.36(4)	4.291	100.00	3.45	1.31
0.4	Fe_2TiO_5	3.7304(2)	9.7795(6)	9.9647(6)	363.52(5)	4.288	95.04	3.45	1.32
	$FeMnO_3$	9.4310(1)	9.4310(1)	9.4310(1)	838.84(1)	5.030	4.96		
0.5	Fe_2TiO_5	3.7311(1)	9.7805(5)	9.9649(5)	363.63(4)	4.283	87.64	3.54	1.30
	$FeMnO_3$	9.4372(1)	9.4372(1)	9.4372(1)	840.48(4)	5.035	12.36		
1	Fe_2TiO_5	3.7318(9)	9.7811(5)	9.9654(5)	363.74(4)	4.281	72.54	3.65	1.31
	$FeMnO_3$	9.4379(7)	9.4379(7)	9.4379(7)	840.69(2)	5.018	27.46		

Figure 2 and Table 1 show that the refinement results of X-ray diffraction pattern have a very good fitting quality based on the criteria of fit (R_{wp}) and the goodness of fit ($\chi 2$) in accordance with the agreement [12]. Table 2 shows that based on the refinement pattern of X-ray diffraction pseudobrookite $Fe_{2-x}Mn_xTiO_5$ samples with variations in composition (x = 0.1, 0.2, 0.3, 0.4, 0.5, and 1), the samples having a single phase are in the composition of x = 0.1 – 0.3. Data supporting the other is from the analysis of changes in the volume of the unit cell as shown in Figure 3.

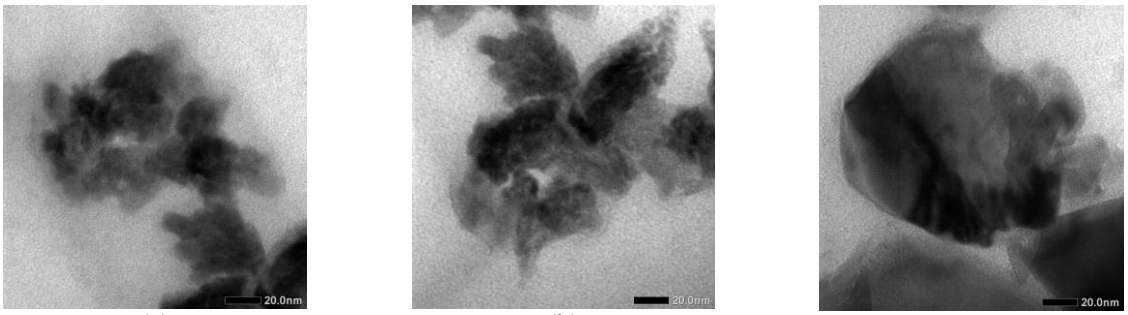


(a) Volume and density of unit cell (b) Mass fraction

Figure 3. The volume of unit cells as a function of composition

In Figure 3(a) it appears that when composition x increased, the expansion of unit cell volume occurred up to x = 0.3. After that, the composition (x > 0.3) under net saturation. This phenomenon was due to the atomic radius of Mn (r = 1.79 Å) was longer than the atomic radius of Fe (r = 1.72 Å). Thus it resulted in lattice parameter which also increased for the third axis. Then, for the composition x > 0.3, the unit cell volume gradually saturated caused by the lattice distortion and by the presence of another phase formed. The addition of Mn excess (x > 0.3) resulted in an imbalance of the reaction so that Mn preferred to bind Fe to form Ferromanganite since the composition of these compounds was relatively stable compared with $Fe_{2-x}Mn_xTiO_5$. Meanwhile, another Fe will bind to Mn forming $FeMnO_3$. Figure 3 (b) shows that on the composition of x > 0.3, there was no change in the unit cell volume on the phases of $FeMnO_3$. It means that Mn had reacted with Fe to form $FeMnO_3$. This phenomenon can be explained accordance to the results of the analysis of mass fractions as shown in Figure 4.

These refinement results are also supported by observations of the surface morphology of particles for both single phases using transmission electron microscope (TEM) as shown in Figure 3. Figure 3 shows that the particle morphology of the composition of x = 0.3 had a good particle homogeneity and uniform in across the sample surface with a polygonal particle shape and the particle size varied from 20 – 100 nm. In general, a single phase characteristic of polycrystalline samples by observation of TEM image is homogeneity and uniformity particle shape in across the sample surface.



(a) (b) (c)

Figure 4. The particle morphology of the pseudobrookite $Fe_{2-x}Mn_xTiO_5$ (x = 0.3) by TEM

The presence of this Mn can be proven based on the result of EDS (Energy Dispersive Spectroscopy) data as shown in Figure 5 and Table 2.

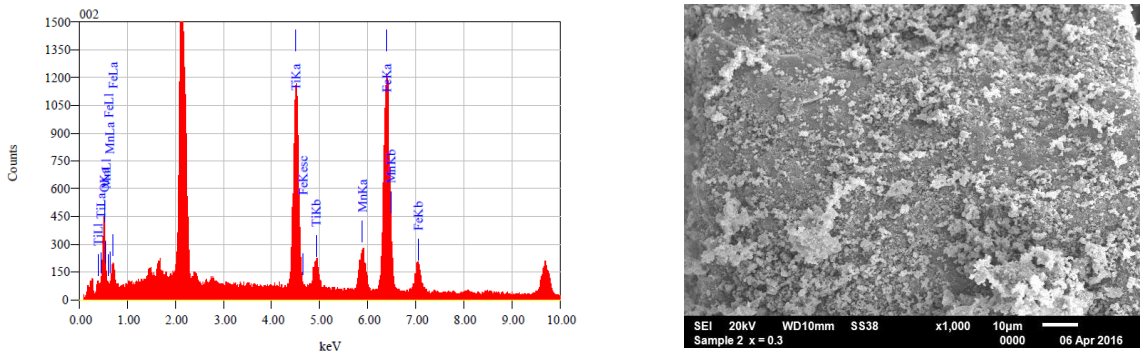


Figure 5. EDS spectrum of the pseudobrookite $Fe_{2-x}Mn_xTiO_5$ (x = 0.3)

The interesting thing is both materials have a single phase with the same structure, but their compositions are different. On the composition of x = 0.3, there was a 0.3 % Mn atoms succeeded substituting partially Fe atoms. Thus, the substitution of Mn into Fe on pseudobrookite Fe_2TiO_5 system was only capable of 0.3 at.% without changing the crystal structure of the material.

Table 2. Element distribution on the $Fe_{2-x}Mn_xTiO_5$ (x = 0.3) measured by EDS.

Element	Energy (keV)	Mass fraction (%)	
		wt.%	at.%
O	0.525	17.63	41.61
Ti	4.508	23.35	18.40
Mn	5.894	7.61	5.23
Fe	6.398	52.41	34.75

4. Conclusion

Synthesis of pseudobrookite $Fe_{2-x}Mn_xTiO_5$ with variations in composition (x = 0.1, 0.2, 0.3, 0.4, 0.5, and 1) has been successfully carried out. The refinement pattern of X-ray diffraction samples of pseudobrookite $Fe_{2-x}Mn_xTiO_5$ shows that the sample has a single phase in the composition of x = 0.1 – 0.3. The particle morphology of the composition of x = 0.1 – 0.3 has a good particle homogeneity and is uniform in across the sample surface with a polygonal particle shape, and the particle size varies. Thus, substitution of Mn into Fe on the pseudobrookite $Fe_{2-x}Mn_xTiO_5$ is only capable of 0.3 at.% without changing the crystal structure of the material.

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