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PART A

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AND NATURAL
SCIENCES

PROQRES
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Muhammad Aziz Majidi[1], Wisnu Ari Adi[2]

[1]Dept of Physics, Faculty of Mathematics and Natural Sciences, University of Indonesia, Kampus Baru UI, Depok,
[2]Centre for Science and Technology of Advanced Materials – National Nuclear Energy Agency
Kawasan Puspiptek, Setu, Tangerang Selatan, Banten **(INDONESIA)**
E-mails: aziz@ui.ac.id, dwisnuaa@batan.go.id

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ABSTRACT

A nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ was successfully synthesized by solid state reaction through mechanical milling method. Stoichiometric quantities of analytical-grade $MnCO_3$, $BaCO_3$, Fe_2O_3, TiO_2 and $SrCO_3$ precursors with a purity of greater than 99% were mixed. The mixture of all precursors were first mechanically milled for 10 hrs and then sintered at a temperature of 1000 °C for 10 hrs in which a fully crystalline material is ensured. The sintered material was then re-milled for 20 hrs to obtain powder-based nanoparticles. The refinement of x-ray diffraction trace for re-milled materials confirmed a single phase material with a hexagonal structure of lattice parameters: a = b = 5.878(1) Å and c = 23.082(8) Å. The first mechanical milling resulted in powders with mean size 835 nm. The mean size particle size was reduced further to 81 nm in the second mechanically milled powders. Results of mean crystallite size evaluation for respective powder materials showed almost similar mean crystallite size about 15 nm. In addition, the hysteresis curve showed that the sample is ferromagnetic. Results of VNA evaluation indicated that there were three of absorption peaks with reflection loss values ~ -15.0 dB, ~ -10.0 dB, and ~ -10.2 dB at frequency 9.0 GHz, 12.5 GHz, and 15.0 GHz respectively. The study concluded that the $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ material with nanoparticles has been successfully synthesized showing a good candidate as for electromagnetic absorber materials.

Key words: nanoparticle, $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$; crystallite size; particle size; electromagnetic, absorber material

1. INTRODUCTION

Barium-strontium hexa-ferrites with a chemical formula of $Ba_{0.5}Sr_{0.5}Fe_{12}O_{19}$ have a much larger uniaxial anisotropy constant and high saturation magnetization values and hence potential for permanent magnet applications [1-4]. Since the compounds are made of oxide based materials and they have high permeability value, ferrites are the most potential candidates as electromagnetic wave absorbing materials especially at high frequencies range such as radar absorbing material [5-7].

Recently, some magnetic materials including hexa-ferrite based materials have been found to exhibit characteristics of microwave absorption [8-10]. They have been shown to be excellent candidate for microwave absorbing materials due to a high magnetization value. However, hexaferrites such as barium-strontium hexaferrite has a relatively high anisotropy constant value and hence a high coercivity. Thus, more suitable as permanent magnets [11] instead of as absorbing materials because the interaction between magnetic moment and the magnetic field of microwave would not easy to take place. It is therefore some intrinsic properties of its magnetic phase like the anisotropy constant which govern the coercivity value has to be reduced. Reduction in anisotropy constant of barium-strontium hexaferrite modification is primarly required since a substantially low coercivity while the magnetisation remains high are the most properties that required for microwave absorber applications. At this study has shown that the magnetic coercivity was reduced very significantly in Mn and Ti substituted barium-strontium hexaferrite. In addition, the influence of fine particle size especially in nano size regime is also well known to affect both remanence and coercivity in this material [12-13].

The purpose of this study was to investigate the magnetic properties which exhibited by strontium (Sr) substituted barium (Ba) and manganese (Mn)-titanium (Ti) substituted iron (Fe) in $BaFe_{12}O_{19}$ structure. The study included synthesize of nanoparticles for this composition and the microwave absorption performance for this material.

2. MATERIALS AND METHODS

A $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ composition was prepared by solid state reaction using a conventional milling technique. Stoichiometric quantities of analytical-grade $MnCO_3$, $BaCO_3$, Fe_2O_3, TiO_2 and $SrCO_3$ precursors with a purity of greater than 99% were mixed and milled using a high-energy milling (HEM) type Spex 8000 to a powder weight ratio of 10:1 for 10 hours. The sample powders were then compacted into pellets and sintered in the electric chamber furnace at 1000°C for 10 hours to obtain crystalline materials. The $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ sample was re-milled for 20 hours to obtain nanoparticles powders. The sample was characterised by powder x-ray diffraction (XRD) using Miniflex Rigaku diffractometer with CuK radiation (= 1.5406 Å). Powders obtained after 20 hours of re-milling time were found to be highly deformed materials, as indicated by the absence of Bragg diffraction peaks and the presence of broad and diffuse peaks by XRD analysis. Additional XRD analysis was performed to determine the mean crystallite size. In this case, un-overlapping diffracted peaks were selected, and reliable line broadening data were obtained using the step-scanning mode. Intensity data were recorded for each 0.02° step using a 2-second scanning time, and the mean crystallite size was derived from williamson-hull formula [14] that takes into account the instrumental broadening correction and the correction due to lattice strain. The re-milled powders were then dispersed in distilled water and evaluated using a Helos simpatec particle size analyzer for micron-sized particles and a Zeta nanosizer analyzer for nano-sized particles. Microstructural examination of the samples observated using the transmission electron microscope (TEM). The magnetic properties were evaluated using an Oxford instrument

vibrating sample magnetometer (VSM). Finally, the reflection and transmission of microwave were carried out using the vector network analyzer (VNA) with frequency range of 300 kHz - 20 GHz.

3. RESULTS AND DISCUSSION

The x-ray diffraction profiles of synthesized material is shown in Figure 1 in which respective profiles of $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ before and after milling (nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$) are compared. It is shown that the two diffraction profiles exhibit a pattern similar to that of $BaFe_{12}O_{19}$ phase despite a small shift in the peak positions due to the presence of a partial Sr ion substitution for Ba, in addition to Mn and Ti ion substitutions for Fe. Thus, the material is a single phase [4]. In addition, all XRD traces consistently exhibit diffraction line broadening.

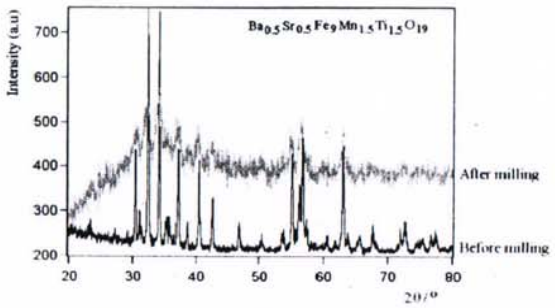


Fig. 1. The XRD profiles of synthesized material.

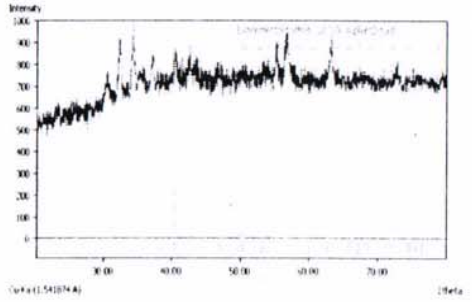


Fig. 2. Phase identification of XRD profile

The phase identification result of XRD profile of a nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ is shown in Figure 2.

The refinement result of XRD profile of a nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ is shown in Figure 3. The resulting profile of the residue shows almost no intensity over the entire diffraction angle range. The quality factors of fitting, R (criteria of fit) and χ^2 (goodness of fit) were considered acceptable. It was found that the χ^2 is less than 1.3 for nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$. Figure 3 showed that the profile is then in a good agreement between observation and calculation. The refinement results of x-ray diffraction pattern confirmed that the nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ is a single phase material with a hexagonal structure, space group of P 63/m m c (194).

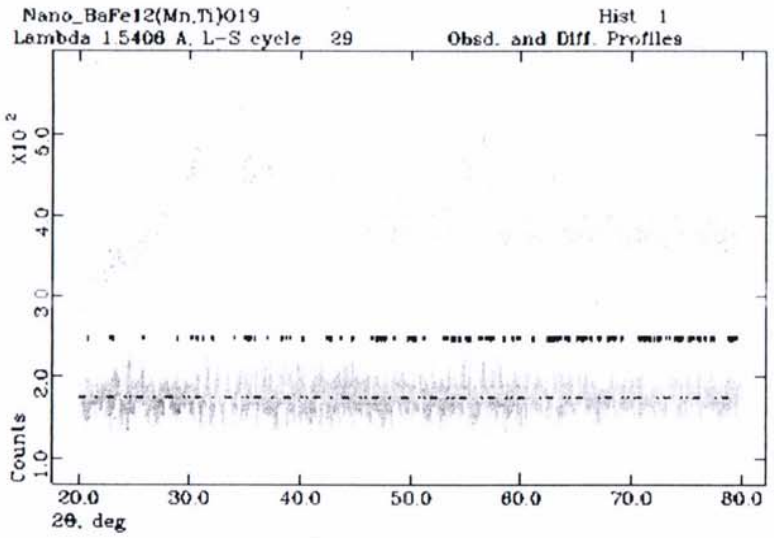


Fig. 3. *The refinement of XRD profile on a nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$*

The unit cell parameters for the phase in nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ sample are summarized as shown in Table 1.

Table 1. Structure parameter, factor R and *goodness of fit* (χ^2).

Nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{15}Ti_{1.5}O_{19}$		
Space group : P 63/m m c (194), crystal system : Hexagonal a = 5.878(1) Å, b = 5.878(1) Å and c = 23.082(8) Å, α = β = 90° and γ = 120°. V = 690.9(4) Å^3 and ρ = 5.890 gr.cm^{-3}		
R factor	wRp = 3.92 Rp = 3.13	χ^2 (chi-squared) = 1.071

Further confirmation was measuring the elemental analysis and observation of surface morphology on the samples to determine the particle distribution, homogenous, and its composition by using SEM-EDS equipment.

The elemental analysis and observation of surface morphology on the nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ showed that the sample has been well established as shown in Figure 4.

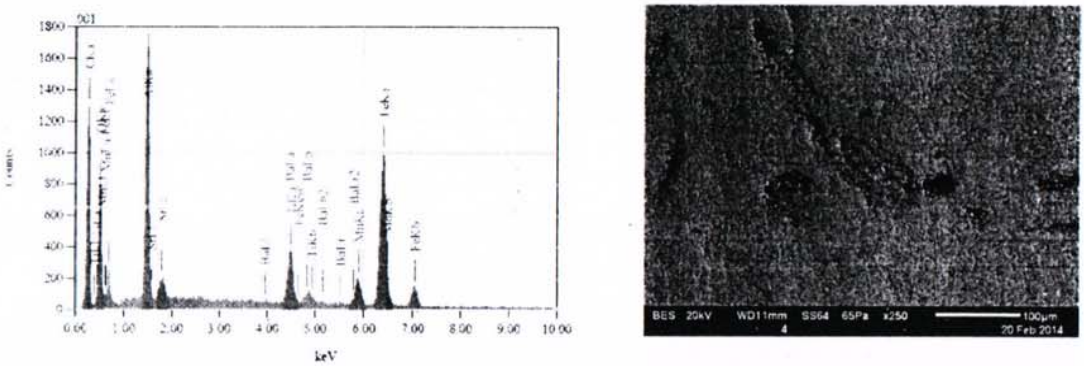


Fig. 4. *Surface morphology and elemental analysis of the nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$*

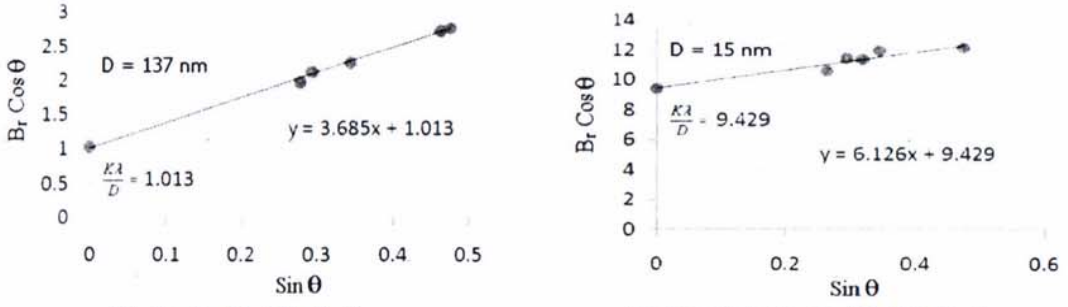
The microstructure analyses showed that the particle shapes was aggregates with the varied particle sizes, uniform and evenly distributed on the surface of the sample. So that required further analysis of the element content in the samples use energy dispersive spectroscopy. The elements content of the nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ was shown in Table 2.

Tabel 2. The results of element analysis by using energy dispersive spectroscopy

No.	Unsure	Content	
		(wt.%)	(at.%)
1.	Barium (Ba)	6,21 ± 0,38	1,71
2.	Strontium (Sr)	3,57 ± 0,26	1,25
3.	Iron (Fe)	46,79 ± 0,26	26,17
4.	Manganese (Mn)	8,27 ± 0,25	4,48
5.	Titanium (Ti)	6,07 ± 0,16	4,23
6.	Oxygen (O)	29,09 ± 0,08	62,16

Energy dispersive spectroscopy (EDS) spectra shows that the sample had composition in accordance to stochiometry composition.

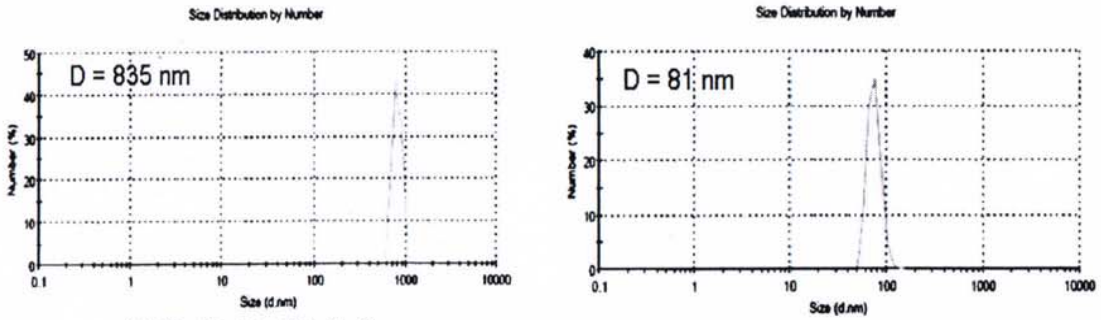
The mean crystallite size of $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ and nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ was calculated by the analysis method williamson hull of x-ray diffraction pattern [14]. The plot between values of B_r Cos θ and Sin θ for $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ and nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ were given in Figure 5. The plot was a lineir graph from which the mean crystallite size for respective samples was calculated. It is found that the mean crystallite size for $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ and nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ are 137 nm and 15 nm respectively. Thus, it is confirmed that both samples have a similar mean crystallite size value and re-milling of sintered powders would not change the size of crystallites containing in the particles.



(a) $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ (b) Nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$

Fig. 5. *The crystallite size of the samples*

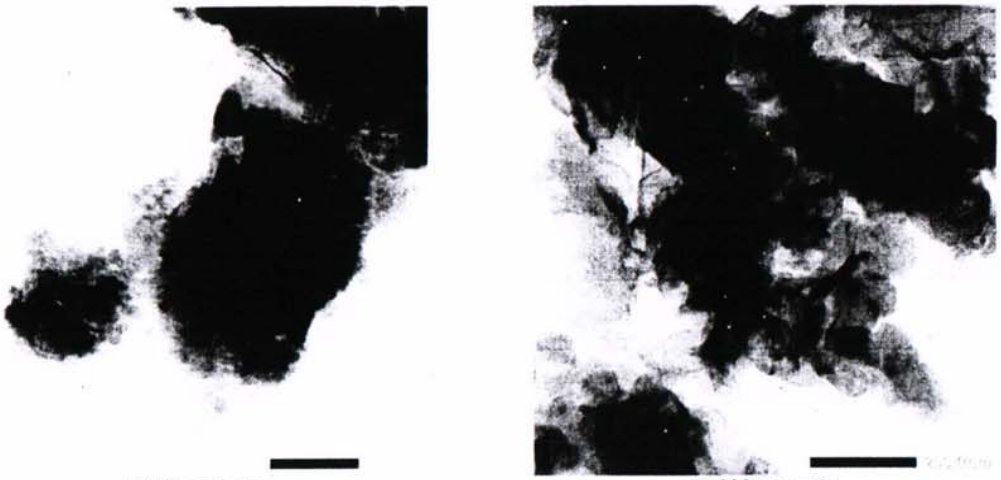
In addition to the mean crystallite by x-ray line broadening analysis, the typical particle size distribution for $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ and nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ subject to nano sizer evaluation is demonstrated in Figure 6. The mono modal curves for the two samples indicated that the suspended particles in the dispersant media are homogeneous. However, the powders of nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ showed a narrower particle size distribution with the mean particle size of ~ 81 nm which is clearly much smaller than that of $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ (~ 835 nm). It is then concluded that re-milled $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ powders with an extended milling time up to 20 hours has refined the particle sizes from ~ 835 nm to ~ 81 nm that is one tenth of the original size. Re-milling the sintered powders is to refine the particles toward nanoparticles that is the particles containing nanocrystallites.



(a) $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ (b) Nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$

Fig. 6. *Particle size distribution of the samples*

A TEM micrograph of a synthesized nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ powders is shown in Figure 7.



(a) 50 nm scale (b) 200 nm scale

Fig. 7. *The TEM photographs of nanoparticle* $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$.

The image reveals the morphology and size of nanocrystallites and the presence of particles that exist as aggregates of fine grains. It is clear that the additional milling of sintered mechanically alloyed powders is quite effective, not only in reducing the particle size, but also in changing the shape of the particles and producing a significant improvement in their size distribution. The average particle size which estimated from the TEM micrograph of Figure 7 was around ~80 nm which very close to the mean particle size subject to a nano sizer evaluation (~81 nm). The mechanical alloying of material precursors has resulted in laminated powders containing embryos of $BaFe_{12}O_{19}$ phase. An additional treatment in form of sintering at a temperature 1000 ^{o}C to the mechanically alloyed powders has promoted the formation of crystalline particles with the mean particle size ~ 835 nm. The particles contained crystallites of mean size ~81 nm. A further milling of sintered nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ powders during 20 hrs has refined further the mean particle size to ~ 137 nm which is very close to the size of mean crystallite sizes (~ 15 nm).

One of research topic in this area is the materials that can absorb electromagnetic waves that are can effectively reduced the reflection of electromagnetic signals. There are at least two important conditions must meet the requirements for a suitable and high performance absorbing material. The first is the so called matched characteristics impedance as shown in Figure 8. Basically, the intrinsic impedance of the material must be equal to the intrinsic impedance of the free space. Second, the interaction between electromagnetic energy and material should results in a rapid attenuation of the incident electromagnetic, thus reducing the emerging wave to an acceptably low magnitude. It is therefore believed that materials with poseses dielectric and magnetic behavior would be the most potential candidates as absorbing materials.

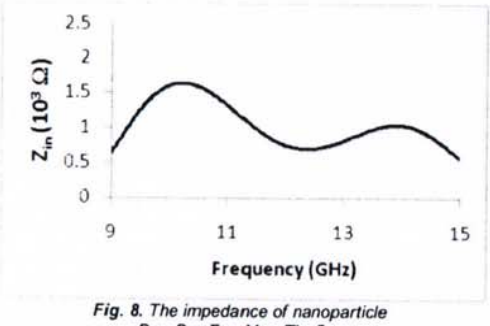


Fig. 8. The impedance of nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$

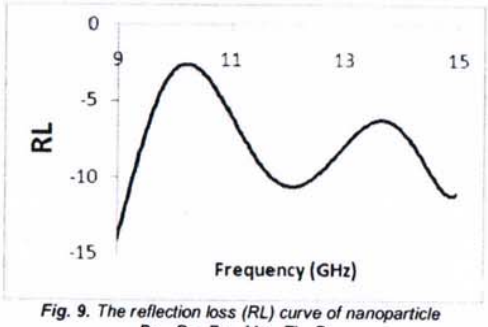


Fig. 9. The reflection loss (RL) curve of nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$

Figure 9 shows the relation between the reflectance (RL) of nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ and the microwave frequency in range of 9-15 GHz when the thickness of sample is 15 mm. There are at least four absorption peaks observed within the frequency range. However, RL increases from 9.0 GHz to 10.2 GHz, decreases from 10.2 GHz to 12.5 GHz, then increases from 12.5 GHz to 13.5 GHz, and decreases further from 13.5 GHz to 15.0 GHz. This indicates that nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ has certain microwave absorbing properties in the frequency range of 9- 15 GHz, whose value of the absorbing peaks are -15 dB at 9.0 GHz; -10.0 dB at 12.5 GHz and -10.2 dB at 15.0 GHz.

4. CONCLUSIONS

The synthesized of $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ has successfully been made. Nanoparticles formation was confirmed by results of particle and crystallite analysis which confirmed by TEM images. The sintering treatment at a temperature 1000 °C to the mechanically alloyed powders has promoted the formation of crystalline particles with the mean particle size ~ 835 nm. The particles contained crystallites of mean size ~137 nm. A further milling of sintered $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ powders during 20 hours has refined further the mean particle size to ~ 81 nm which is very close to the size of mean crystallite sizes (~ 15 nm). As to microwave properties the nanoparticle $Ba_{0.5}Sr_{0.5}Fe_{9.0}Mn_{1.5}Ti_{1.5}O_{19}$ has indicated certain microwave absorbing properties in the frequency range of 9- 15 GHz, whose value of the absorbing peaks are -15 dB at 9.0 GHz; -10.0 dB at 12.5 GHz and -10.2 dB at 15.0 GHz.

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