

## Analysis of the magnetic properties nanoscale barium hexaferrite (BHF) prepared by milling and ultrasonic method

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**Abstract.** Barium hexaferrite (BHF) is well established material which widely used respectively as permanent magnets. In this research, we report our recent investigation on magnetic properties analysis of barium hexaferrite (BHF) compounds with a ratio of Fe/Ba: 11 prepared by a mechanical alloying process and high power ultrasonic destruction to promote the soft magnetic properties. The investigation carried out by Scanning Electron Microscope (SEM) shows the grain size between 500-1500 nm, it indicates that each grain is composed of several crystallites or polycrystalline. By mean of X-ray diffraction revealed the phase composition and the mean crystallite size <70 nm. The Characterization of the magnetic properties of the effects of downsizing the particle size of ~ 200 nm to ~ 50 nm by the ultasonik method provide saturation value of 0.35 T, remanent 0.24 T and the coercivity is 115 kA / m.

### 1. Introduction

The hexagonal barium hexaferrite ( $\text{BaFe}_{12}\text{O}_{19}$ ) is known as a hard magnetic material and represents one of the mostly used materials in the area of magnetic recording media, which cannot be easily substituted by other magnets [1–3]. From the scientific point of view, as well as from the standpoint of the technological applications, the barium hexaferrite presents interest due to its special properties, such as: high remanence and coercivity, thermal, electrical and chemical stability [4, 5]. The rapid development of information and telecommunications technology provide the effect of increasing the use of microwave-based electronic instrument (300 MHz - 300 GHz)[6]. In this research we have prepared a barium hexaferrite using the mechanical alloying process and high power ultrasonic destruction then treated at different temperatures. The barium hexaferrite monocrystals resulting after the heat treatments were extracted by solving their structural, and magnetic properties were studied.

### 2. Numerical Methods

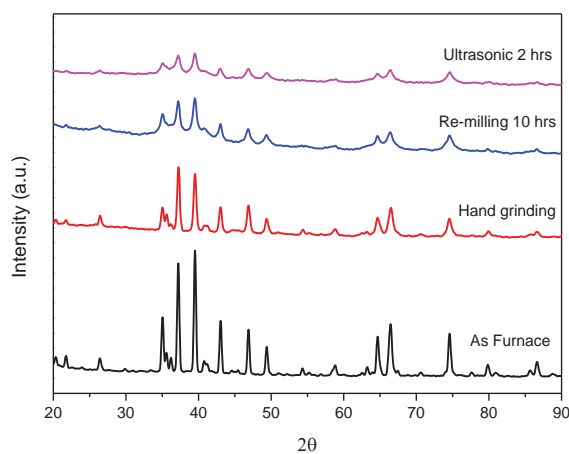
The quantities of powder  $\text{BaCO}_3$  and  $\text{Fe}_2\text{O}_3$  with 99% purity (Merck) were weighted according to the stoichiometric formula. The powder were mixed together with a planetary mill  $\text{Fe}_2\text{O}_3$ (99% purity) and  $\text{BaCO}_3$  (99.8% purity) in proportion of 1:2, 1:1, and 1:0, , reaching a homegenisasi phase, followed by fracturing (embrittlement) which resulted in the particle size reduction of subsequent collisions. The entire amount of the particle size distribution is characterized by a Malvern Zetasizer Ver. 6:20, particle size analyzer (PSA) with the ability to 20 nm. For samples such as BHF magnetic, attractive force between the



magnetic particles are very strong need to be separated by the use of ultrasonic and the addition of surfactant to prevent agglomeration so that the data produced is a true particle size. Morphology characterization of samples with a scanning electron microscope (SEM) type FEI-F50 are able to achieve magnification 200.000 $\times$ . Magnetic characterization of samples was done by using Permagraph EP.3 magnetometer with a maximum induction field in 1700 kA /m (21.5 kOe). Variables were entered in the form of density data acquisition software is done with density meter to generate data that does not depend on the volume or size of the sample. Synthesis samples are taken to form a single phase system BHF by mechanical alloying method with a planetary ball mill. And followed by sonication.

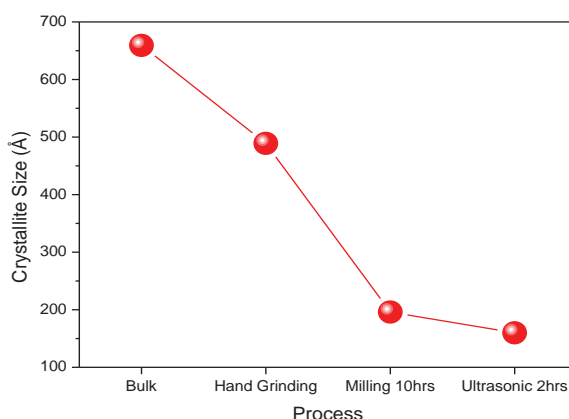
### 3. Results and Dscussion

The BHF literature review of phase formation does not follow strictly stoichiometric calculations, but follow the ratio of Fe/Ba dependent on the process [7,8]. In the observed to the ratio of Fe/Ba =11 to the mechanical alloying process, with variations of milling time 20, 40, 60 hours are shown in Figure 1. The diffraction pattern BHF which uses the ratio of Fe/ Ba: 11 after the sintering process is then performed manually destruction process by using a mortar and then re-milling for 10 hours, followed by sonication process for 2 hours. BHF seen that the single phase can be generated by using the ratio of Fe /Ba: 11 and the process after forming BHF shows no impurities [9].



**Figure 1.** The diffraction pattern in Fe/Ba: 11 with a variety of milling time

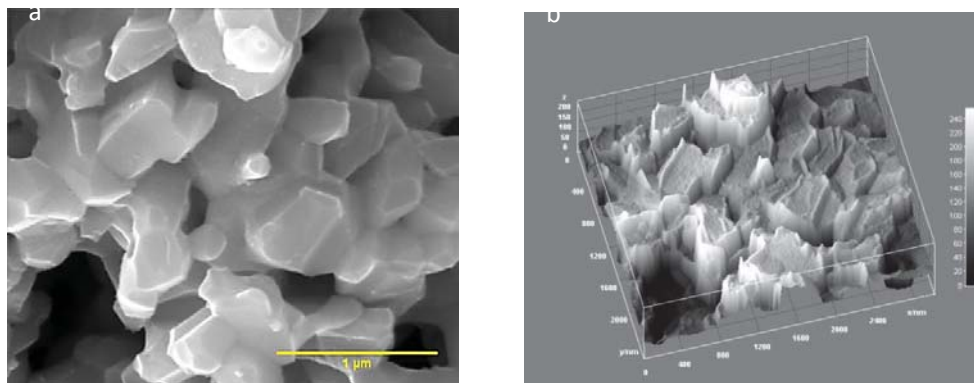
This shows that the impurity phase  $\text{Fe}_2\text{O}_3$  is growing with increasing milling time occurs only at the beginning of the process before the phase BHF formed. It is also seen that a decline in the intensity and widening on the entire diffraction pattern peak as compared with the sample produced after sintering process (as-furnace). The particle size reduction by the process and the size of the smallest one is in the process of sonication.



**Figure 2.** The crystallites of BHF after sintering 1100° C for 2 hours

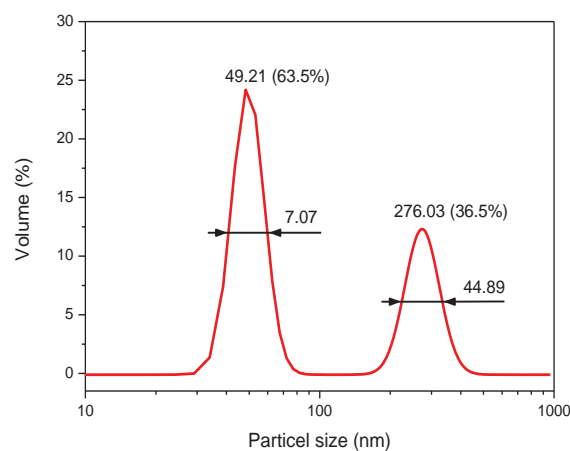
Therefore, based on a search for the formation of BHF through the process of mechanical alloying with the ratio of Fe/Ba : 11, by milling process 20 hours and sintering 1100° C for 2 hours to produce a single phase BHF.

Figure 2. The size of the crystallites of the processes carried out after sintering. It turned out that the destruction process can manually reduce the size of the crystallites to about 150 nm followed by milling for 10 hours to reduce the size to about 200 nm and a subsequent process which sonication can reduce the crystallite size to about 100 nm. This process is the basis for the production of nano-particles on the method of top-down processing.



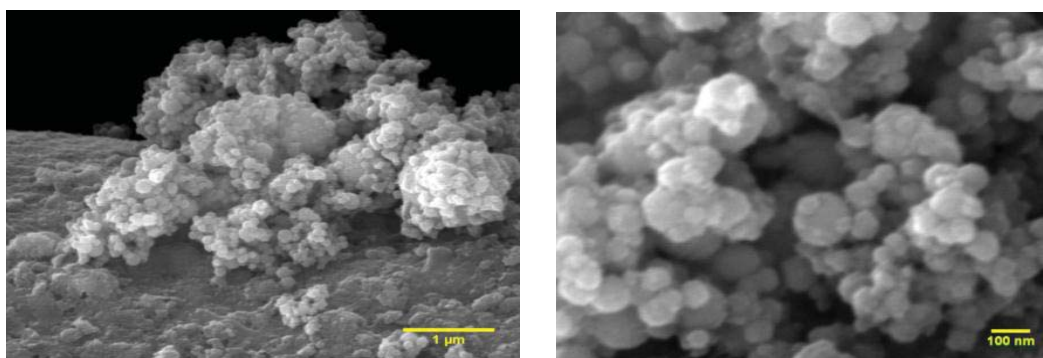
**Figure 3.** SEM photos and topographic after sintering process 1100 °C

Figure 3. SEM morphology of the results of the samples after sintering showed a particle size between 500-1500 nm, while the crystallite size XRD analysis resulted in an average diameter of 65 nm which indicates the sample polycrystalline.



**Figure 4.** The particle size of 5 hours of sonication with deposition method

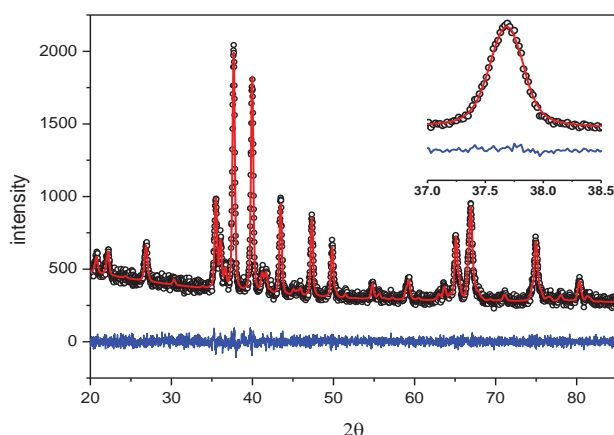
Samples that have undergone sonication was deposited for 48 hours, after which the particles do not settle taken and that settles reprocessed. This method provides control with homogeneous results as shown in Figure 4 where the 5 hours of sonication resulting particle diameter of 49.2 nm with a deviation of 7.1 nm and 276 nm with a deviation of 44.9 nm. These results prove that this method is effective and can be used to control the particle size after sonication process. Figure 5 the size of the particles at a magnification of 50000 times (adzan 200000 times (b) produced by the process of sonication for 5 hours. It is seen that the particles produced by the process of ultrasonic spherical with an average diameter ranging between 42-90 nm and a particle size of 276 nm is a measure of the agglomeration of the magnetic particles BHF. To strengthen the results of the PSA and SEM analysis, the crystallite size (crystallite size) on the XRD data by the method of Whole Powder Pattern Modeling (WPPM).



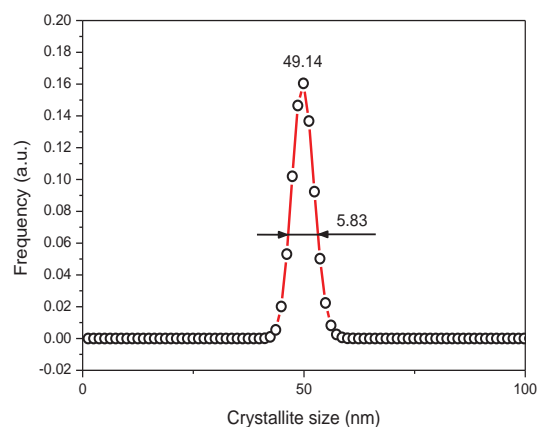
**Figure 5.** SEM photos after sonication for 5 hours

Input parameters are analyzed by wppm will be directly modeled into forms of diffraction peaks so that the fitting is a real physical properties of materials [10]

Figure 6 shows the results of refinement to the software PM2K[10] that implements the methods wppm. The parameters of the instruments kept constant by using a Si-standard, whereas the parameters direfine is intensity, background, displacement, zero offset and crystallite size distribution

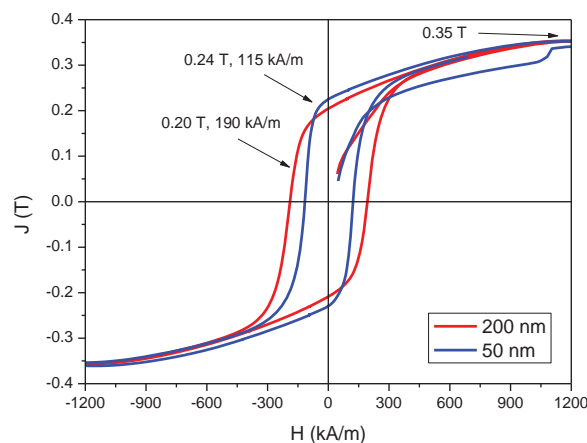


**Figure 6.** Results of refinement using the WPM



**Figure 7.** Distribution of particle size analysis results wppm

Figure 7 shows the distribution of crystallite size (crystallite size) at BHF nano particles produced through the process of sonication for 5 hours. Seen that the crystallite sizes were obtained from analysis WPPM (49.79 nm) is almost equal to the PSA (49.28 nm) in figure 6 peaks 2 and smaller than the result of SEM (~60 nm) in Figure 5.



**Figure 8.** Hysteresis curve with variations in particle size

Figure 8 the sample with a smaller particle size has a higher remanence value but lower coercivity. This is consistent with the theory that particle size reduction over the limit will decrease the single domain limit its coercivity [11]. Saturation value of 0.35 T indicates that the particle size reduction reduces the amount of porosity.

#### 4. Conclusions

Retrieved BHF single phase with a ratio of Fe/Ba = 11 through mechanical alloying process and via sonication can be scaled down to 50 nm particle sizes and narrow distributions (FWHMPSA = 7.07 nm, FWHMWPPM = 5.83 nm). And provide a change of magnetic properties to the value of saturation, remanence and coercivity for a result of the downsizing of the crystals obtained saturation 0.38 T, remanence 0.24 T and coercivity 115 kA/m.

#### 5. References

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