

1. Introduction

In recent years, high dielectric materials play an important role in microelectronics and have been utilized in wide range of technological applications. For the development of capacitors, many investigators have tried to synthesize and study the materials that exhibit high dielectric constant [1-3]. However, most high dielectric materials contain lead that inevitably causes environmental problems due to the toxicity of lead oxide. Therefore, it is necessary and urgent to search for lead-free materials with excellent dielectric properties, which should be comparable with those found in the lead-based ceramics.

Recently, a non-lead $\text{Ba}(\text{Fe}_{0.5}\text{Nb}_{0.5})\text{O}_3$ (BFN) ceramic was reported to exhibit a very high value of dielectric constant (giant dielectric material). BFN was first synthesized via solid state reaction by Saha and Sinha in 2002 [3]. After that, many workers have studied properties of the BFN, including Yokosuka [4], Tezuka et al. [5], Raevski et al. [6], and Saha and Sinha [7]. It has been reported that BFN is a relaxor type ferroelectric material characterized by a broad dielectric transitions with frequency dependent [7]. The ferroelectric transition temperature of BFN is around 110 °C. BFN also shows very high values of dielectric constant ($\epsilon_r \approx 10^4$ - 10^5) covering a wide range of temperature [8].

Researchers normally utilize conventional mixed oxide method to synthesize BFN powders. The disadvantage of this method is that it requires rather high temperature for calcinations. It is known that a chemical route can be used to produce fine powders under low-temperature conditions of reaction. In present work, we report a novel chemical method for the synthesis of BFN powders. The characteristics of BFN powder and dielectric properties of BFN ceramic prepared by this route were also investigated.

2. Experimental procedure

The BFN powders were prepared by chemical process using barium chloride $[\text{BaCl}_2 \cdot 2\text{H}_2\text{O}]$, ferric nitrate $[\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}]$, niobium chloride $[\text{NbCl}_5]$ and citric acid anhydrous. Their purities are over 99.9%. The experimental procedure is illustrated in Fig. 1. Citric acid, ferric nitrate and barium chloride were dissolved in deionized water. To form niobium ethoxide, niobium chloride was dissolved in ethanol. All solutions were thoroughly mixed in ambient conditions using magnetic stirrer. The mixed solutions of precursors were transferred into closed microwave-transparent vessel. The sample were irradiated by microwave (240 watts) for 1 minute and incubated for 3 h. The precursors were dried at 110 °C for 10 h. After drying, the obtained powder was calcined at 850 °C for various soaking times to obtain the pure perovskite phase of BFN. In order to study the phase formation of the calcined powders, X-ray diffraction (XRD) analysis was performed using a diffractometer with CuK_α radiation (XRD; Philips PW 3040/60 X'PERT PRO Console). Powders of the optimum calcined condition were then pressed into a pellet and sintered at 1250 °C for 4 h under the heating and cooling rate of 5 °C/min. Microstructure of calcined powders and sintered samples were examined using scanning electron microscopy (LEO 1455 VP). The samples for dielectric measurements were polished and electroded with gold sputtering. The dielectric properties were measured with an HP-4284A LCR meter utilizing an environmental chamber for the temperature measurements.

3. Results and discussion

Fig.2 shows XRD patterns of powder samples calcined at 850 °C for various soaking times. For a soaking time of 7 h, the formation of BFN structure accompanied with second phase was observed. The crystal growth of BFN progressed with soaking time, indicated by the increase of relative intensities of their corresponding diffraction peaks. Pure perovskite phase of the BFN with a cubic symmetry was obtained when the soaking time reached 14 h. The analysis of XRD pattern was carried out based on ICSD database [9]. Our results were consistent with those obtained by Rama et al. who prepared the BFN via solid state reaction [10]. Saha and Sinha obtained monoclinic-type structure of BFN by using a standard computer program (POWD) analysis [3]. Further increasing time of the calcinations led to the formation of small amount of impurity phase. As widely known, Fe^{2+} changes to Fe^{3+} at high temperature causing defect of the crystal growth where the second phase may start to grow.

Morphology of BFN powder calcined at 850 °C for 14 h is illustrated in Fig. 3(a). Submicron-size particles with average diameter of about 0.7 μm were observed. The sample also appeared rather homogeneous. Fig.3 (b) shows microstructure of BFN ceramic sintered at 1250 °C for 4 h. By using intercept method, the average grain size of BFN ceramic was about 2.1 μm . We note that this chemical route can produce a fine grain ceramic with uniform grain size compared to that of the conventional method (>9 μm) [8, 11].

Fig. 4(a) shows the dielectric constant of BFN ceramics measured as a function of temperature up to 350 °C. The dielectric constant increased with increasing temperature and became broad curve from 200 °C onward. The dielectric constant measured at 1 kHz was in the range of 6,000 to 30,000. In addition, the dielectric constant was found to decrease with increasing frequency. This behavior may be due to

the fact that the dipoles are able to follow the applied at low frequency, but they do not follow to the field at higher frequency [12]. This result agrees with the experimental work done on the BFN ceramics prepared from the mixed oxide method. Fig.4(b) shows the variation of dielectric loss as a function of temperature. At room temperature, the dielectric loss of our ceramics was in the range of 0.5 to 0.1. For the temperature below 250 °C, the dielectric loss value of less than 0.6 was observed. Above 250 °C, however, high values of dielectric loss were found for all samples. Compared with the ceramics prepared by mixed oxide method (dielectric loss >1.0), the BFN ceramic in the present work shows a lower dielectric loss which is suitable for use in capacitor applications.

4. Conclusion

In the present work, a novel chemical route is proposed for the synthesis of BFN powder. Pure perovskite phase with cubic symmetry was obtained. The BFN ceramics prepared by our route exhibited high dielectric constant. In addition, low dielectric loss of the BFN ceramics indicated a potential use of this material for capacitor applications.

Acknowledgements

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Figure captions

Fig.1 A schematic illustration for the preparation of BFN powder and BFN ceramic

Fig.2 XRD patterns of BFN powders prepared by using various calcination times at 850 °C

Fig. 3 SEM micrographs of (a) BFN powder and (b) BFN ceramic

Fig.4 (a) Dielectric constant and (b) dielectric loss of BFN ceramic measured at various frequencies over broad temperature range

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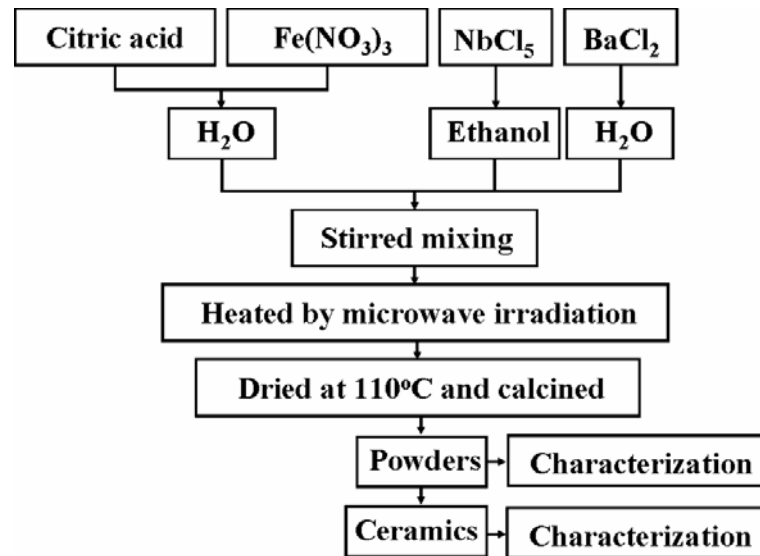


Fig.1

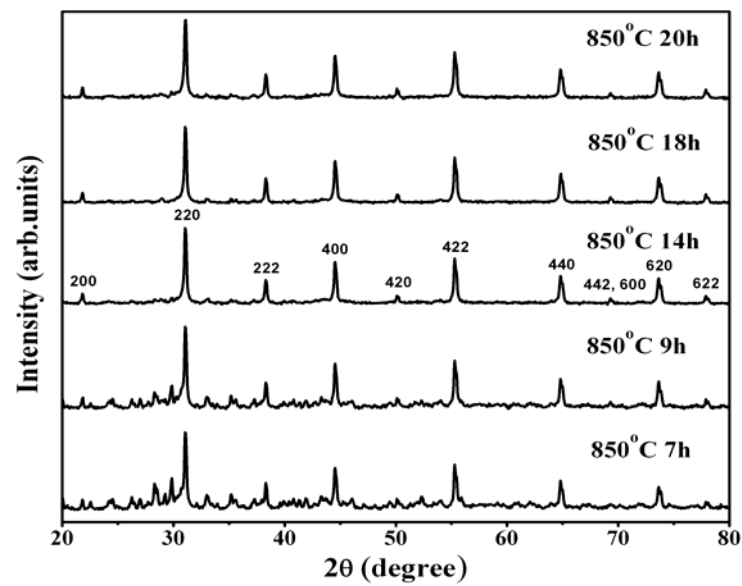


Fig.2

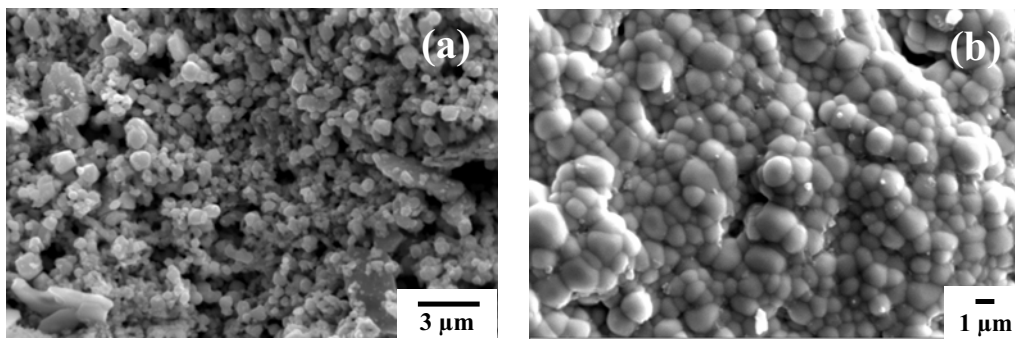


Fig.3

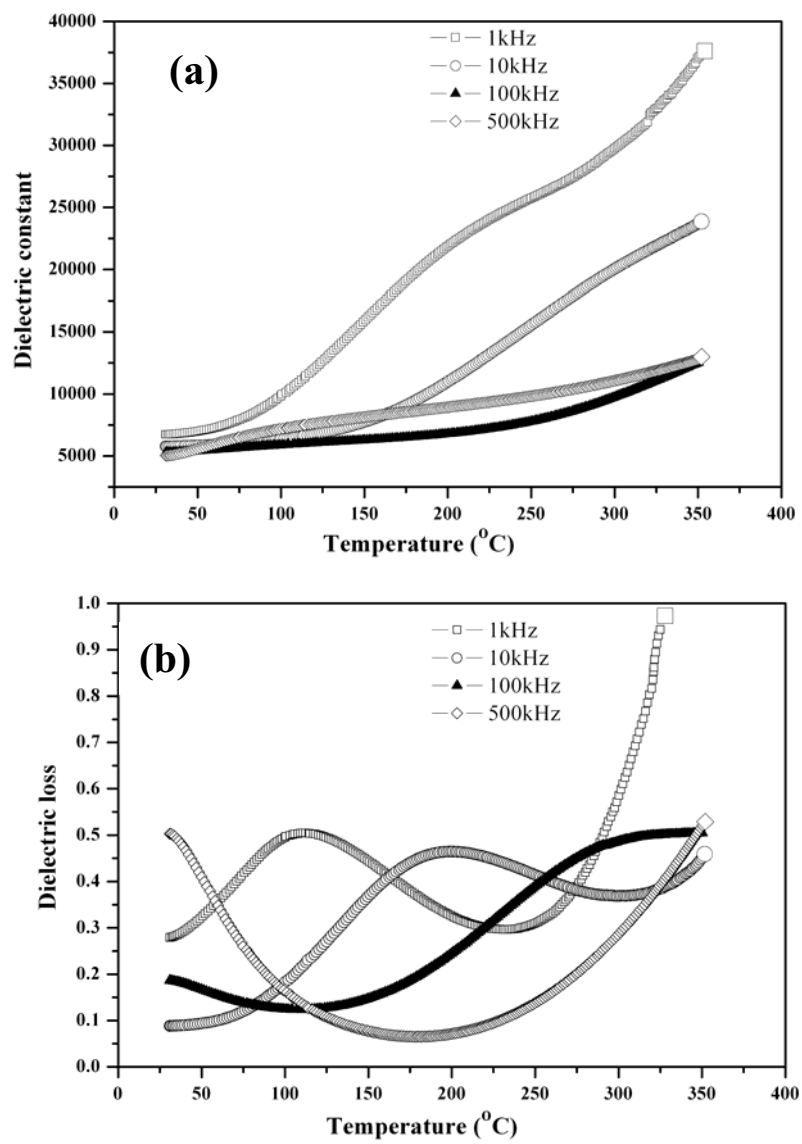


Fig.4

Title Page

Progress in Synthesis of Ba(Fe_{0.5}Nb_{0.5})O₃ Ceramics: A Co-precipitation Method

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Abstracts

$\text{BaFe}_{0.5}\text{Nb}_{0.5}\text{O}_3$ (BFN) is a class of material that exhibits very high value of dielectric constant over wide range of temperature, rendering it a promising candidate for capacitor application. In this contribution, we report a versatile route for synthesis of BFN ceramic via co-precipitation process. The method simply involved mixing of solutions of precursors in ambient conditions. The mixed solution was dried at 110 °C and subsequently calcined at 850 °C for different periods of time. The formation of perovskite structure of BFN was followed by utilizing X-ray diffraction and Fourier transformed infrared spectroscopy. The pure perovskite phase was obtained upon the calcination at 850 °C for 14 h, a much milder conditions compared to normal solid-state reaction. The powder samples were pressed into pellets and sintered at 1250 °C for 4 h. The examination by scanning electron microscopy observed cubic-like crystals. The grain sizes were also found to vary with soaking time of the calcinations. The field dependences of dielectric response and dielectric loss were measured in a frequency range of 20 Hz to 2 MHz at room temperature. The maximum values of dielectric constant and dielectric loss are ~9000 and ~1.1, respectively. In addition, the measured dielectric constants of the samples exhibit frequency dependent, suggesting that the BFN is a relaxor ferroelectric.

Key words: Barium iron niobate, High dielectric constant, Co-precipitation, Ferroelectric

1. Introduction

Complex perovskites such as $\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3}\text{O}_3)$, $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$ are very promising for electroceramic applications. This is mainly due to their high values of the dielectric constant over wide range of temperature [1]. $\text{Ba}(\text{Fe}_{0.5}\text{Nb}_{0.5})\text{O}_3$ (BFN) is one of the materials in this family that has been known to exhibit temperature- and frequency-dependent dielectric values [2-4]. The maximum value of 30,000 was detected at around 170 °C [2,3]. The synthesis of BFN can be achieved via routine solid state reaction, which normally takes place at the temperature above 1000 °C [2,3,5-7]. However, the apparent disadvantage of this method is that it is rather difficult to control stoichiometry and homogeneity of BFN powders. Moreover, this method requires high temperature to obtain complete solid solution and phase-pure BFN powder. Continuous efforts have been made in order to improve the synthetic methods that yield BFN ceramic with, at least, comparable dielectric properties [7-10]. Recent study by Tunkasiri et.al showed that the addition of LiF into system caused the reduction of sintering temperature by about 150-200 °C [8]. However, the values of dielectric constant were found to decrease with increasing amount of the doped LiF. Other studies have shown that the dielectric constant of BFN ceramic can be increased by doping some metals such as bismuth and lanthanum [9,10].

The synthetic method of BFN can be reduced to milder conditions by utilizing a wet chemical process. The method also provides a route for thin film fabrication of the ceramic and has a better control over final composition. It was demonstrated by Chang et.al that the synthesis of BFN can be accomplished via sol-gel route [11]. The calcination temperature required for obtaining pure perovskite BFN was much lower than that of the solid state reaction. It was found that the crystallization into perovskite BFN occurred at 550 °C and eventually completed at 850 °C. In this study, a co-

precipitation route for fabrication of BFN ceramic was explored. It was recently shown that this method was successfully used to prepare $\text{Pb}(\text{Fe}_{0.5}\text{Nb}_{0.5})\text{O}_3$ [12]. The procedure simply involved the mixing of precursor solutions in atmospheric conditions. The pure perovskite powder was obtained by using mild conditions for calcinations comparable to those of the sol-gel method.

2. Experimental procedure

The $\text{BaFe}_{0.5}\text{Nb}_{0.5}\text{O}_3$ powders were prepared by co-precipitation process using barium chloride [$\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$], ferric nitrate [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$], niobium chloride [NbCl_5] and citric acid anhydrous. The purities of all chemicals are over 99.9%. The diagram depicting the details of experimental procedure is shown in Fig. 1. The citric acid, ferric nitrate and barium chloride were dissolved in deionized water while the niobium chloride was dissolved in ethanol. All solutions were thoroughly mixed in an ambient atmosphere using magnetic stirrer. The precipitates of precursors were obtained after continuous stirring for 3 h. The solutions containing the co-precipitates were dried at 110 °C for 10 h. The samples were subsequently calcined at 850 °C for various soaking time. The progress of reaction was followed by utilizing X-ray diffraction (XRD) with Cu K_α ($\lambda=1.54 \text{ \AA}$) radiation (XRD; Philips PW 3040/60 X'PERT PRO Console) and Fourier transformed infrared spectroscopy (FT-IR; Perkin-Elmer). The powder samples were pressed into pellets and sintered at 1250 °C for 4 h under the heating and cooling rate of 5 °C/min. Microstructures of the ceramics were examined by scanning electron microscopy (LEO 1455 VP). The sintered samples were polished to obtain parallel surfaces and electroded with gold sputtering for dielectric measurements. The dielectric properties of the pellet sample were measured

at room temperature and various frequencies ranging between 20 Hz-2 MHz by using an LCR meter (Hioki model 3532-50).

3. Results and discussion

The preparation of BFN powders by sol-gel route in previous study showed that pure perovskite phase was obtained when calcined at 850 °C [11]. Therefore, we selected this temperature for calcinations of our sample. Structural evolution of the sample was then followed by utilizing XRD and FT-IR. Fig. 2 illustrates XRD patterns of powder samples obtained from the calcinations at 850 °C for different soaking times. A crystallization of BFN into perovskite structure is observed when the soaking time is 9 h. However, a considerable amount of residual phases still remains. The crystallization progresses with increasing of the soaking time. It is clear that nearly single perovskite phase is obtained when time of the calcinations reaches 14 h. Further increasing time of the calcinations to 20 h hardly affects structure of the powder sample. Our XRD pattern is consistent with previous works, reporting a monoclinic structure of BFN with $\beta = 90.11^\circ$, $a = 4.0743 \text{ \AA}$, $b = 4.0388 \text{ \AA}$ and $c = 2.876 \text{ \AA}$ [2,3].

Fig. 3 shows FT-IR spectra of BFN powders prepared by using different conditions for calcinations. The intense broad band detected at approximately 600 cm^{-1} is attributed to the absorption of metal oxide bonds in the perovskite structure of BFN. The existence of some H_2O and hydroxyl group in the system contributes to the broad band at $2700\text{-}3400 \text{ cm}^{-1}$ while the peaks at about 1633 cm^{-1} are due to asymmetric stretching vibration of COO^- group. The measurements by FT-IR are in agreement with the XRD results. The broad absorption band at low frequency, mainly associated with the vibrations of metal-oxygen bonds, is detected at 9 h of the calcinations. However, the spectrum also constitutes of other absorption bands of residual organic fraction. Its

concentrations indicated by the intensity of their corresponding absorption bands decrease with increasing soaking time of the calcinations. Only small fraction of the organic residuals remains in the samples when soaking times of the calcinations reach 14 h and 20 h.

Microstructure of the BFN powder revealed by scanning electron microscopy is illustrated in Fig. 4. The powder consists of fine particles with averaged grain size of about 0.99 μm . It also shows a good homogeneity of the sample. Fig. 5 shows surface micrograph of the BFN ceramics sintered at 1250 $^{\circ}\text{C}$ for 4 h. The pellets were prepared by using powder samples obtained from different soaking times of the calcinations. The crystals of all samples exhibit a cubic-like structure. However, the grain size of each sample is found to vary significantly. The average grain sizes are about 4.83 μm , 1.53 μm and 1.28 μm when the soaking times of calcinations are 9 h, 14 h and 20 h, respectively.

The field dependences of the dielectric response and dielectric loss were measured in a frequency range from 20 Hz to 2 MHz at room temperature as shown in Fig. 6. The maximum value of dielectric constant detected at 20 Hz is $\sim 9,000$. The value decreases significantly upon increasing frequency and reaches about 2,500 at 1 kHz. This behavior is due to the fact that the dipoles are able to follow the applied field at low frequency while it becomes more difficult at higher frequency. This frequency-dependent of dielectric constants of the samples suggests that the BFN is a relaxor ferroelectric. The values of dielectric loss ($\tan\delta$) are in the range of 1.1 to 0.1. It is important to note that the dielectric response and dielectric loss of our samples are comparable to the values of BFN ceramics prepared by solid state synthesis [9].

4. Conclusions

Our study demonstrated a co-precipitation method for synthesis of $\text{BaFe}_{0.5}\text{Nb}_{0.5}\text{O}_3$ ceramics. This novel route is rather versatile and requires milder conditions compared to common solid-state reaction. The single phase of perovskite BFN was obtained when calcined at 850 °C for 14 h. The variation of soaking time of the calcinations is also found to affect the grain size of BFN ceramics.

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Figure captions

Fig.1 Illustration of the preparation of the BFN powders and BFN ceramic via co-precipitation process

Fig.2 XRD patterns of BFN powders calcined at 850 °C for various soaking times

Fig.3 FT-IR spectra of BFN powders calcined at 850 °C for various soaking times

Fig.4 SEM micrographs of BFN powder calcined at 850 °C for 14 h

Fig.5 SEM micrographs of BFN ceramic sintered at 1250 °C for 4 h. The pellets were prepared from powders calcined at 850 °C for (a) 9 h (b) 14 h and (c) 20 h.

Fig.6 Dielectric properties at room temperature of BFN ceramics sintered at 1250 °C for 4 h. The pellets were prepared from powders calcined at 850 °C for 14 h.

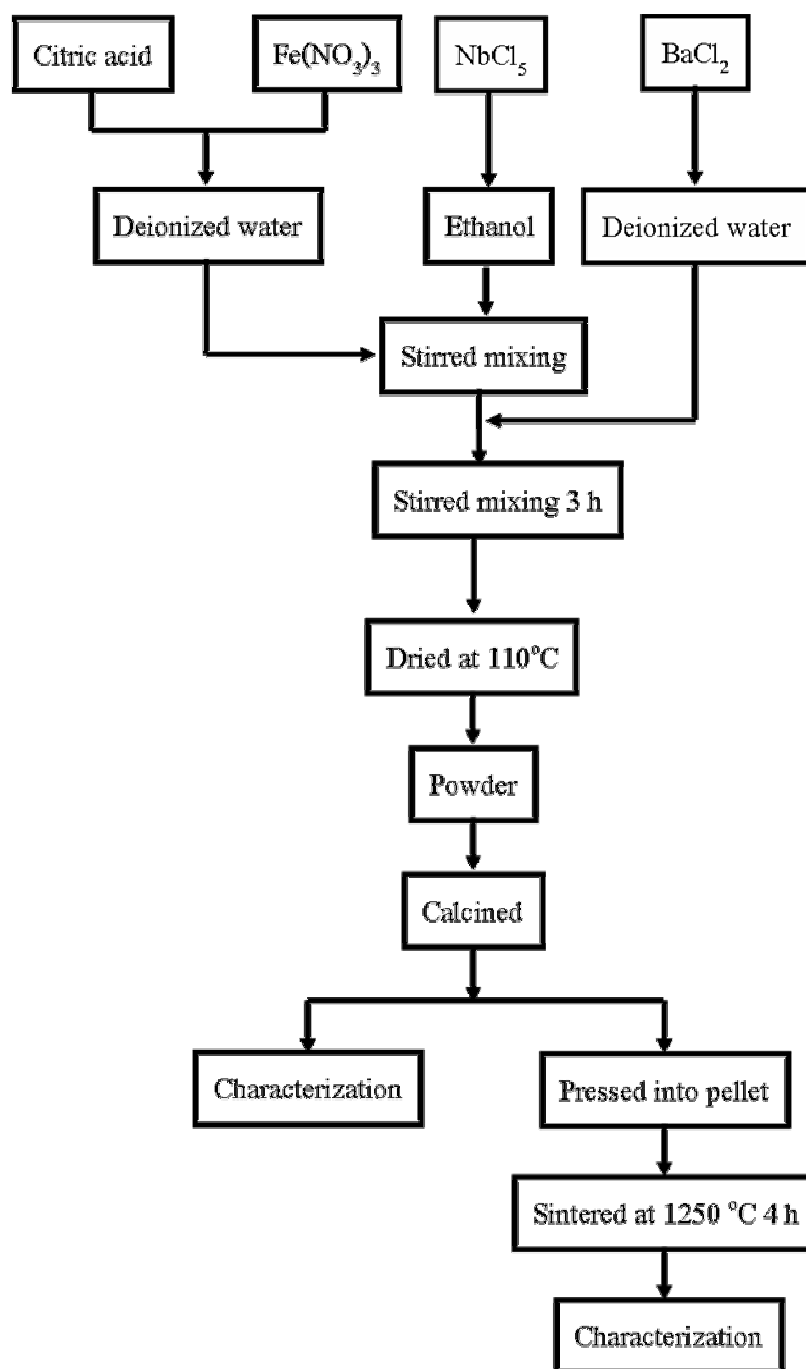


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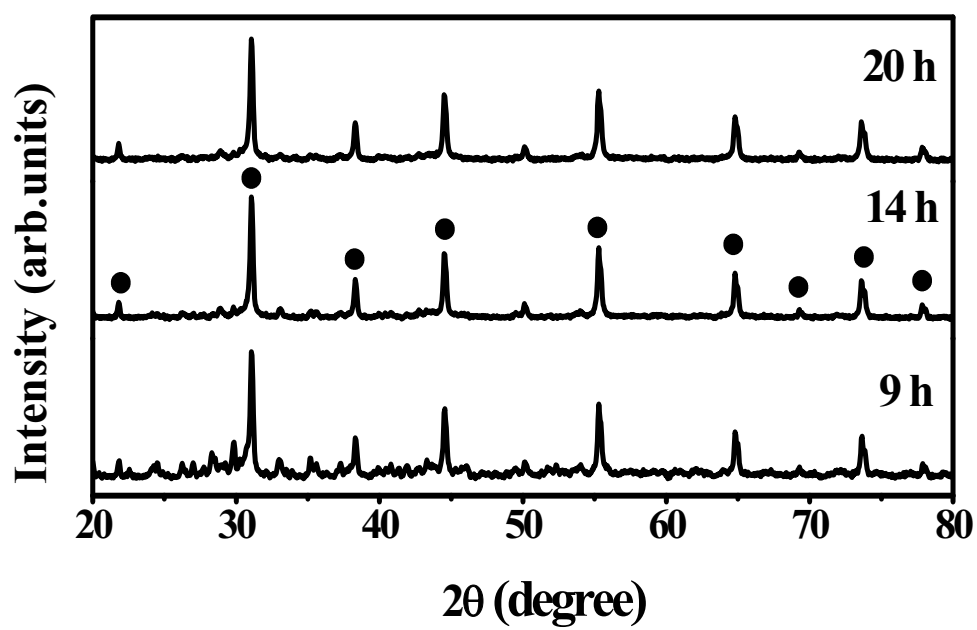


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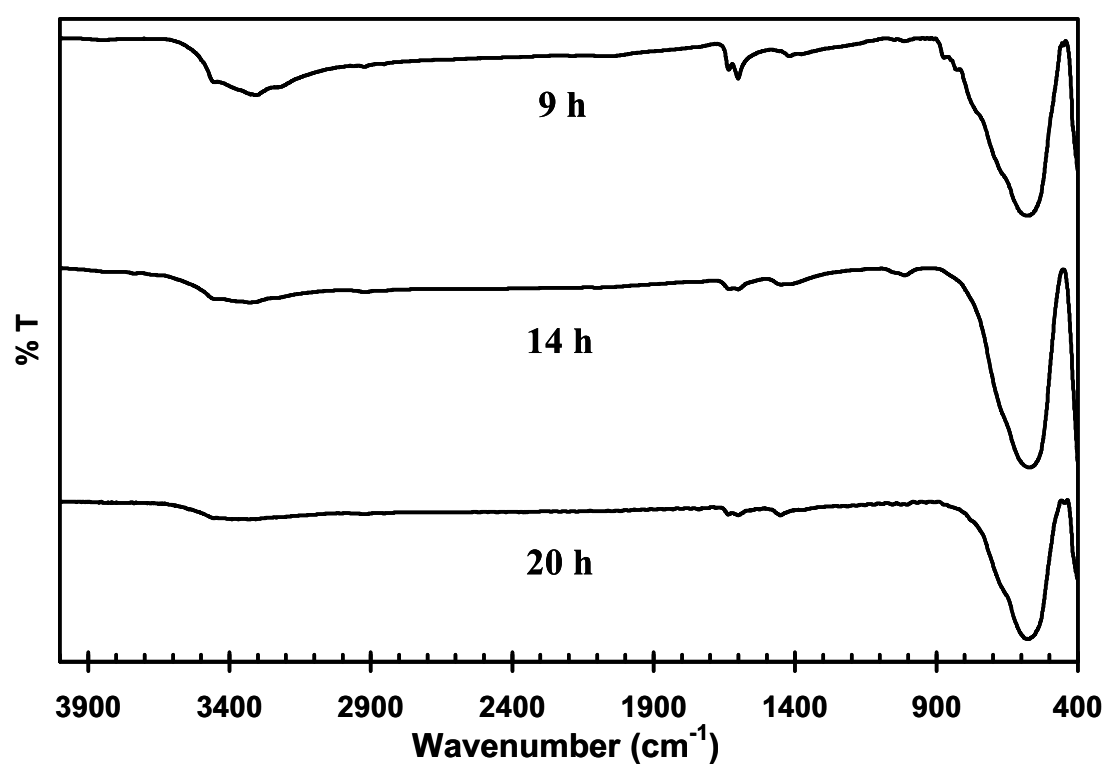


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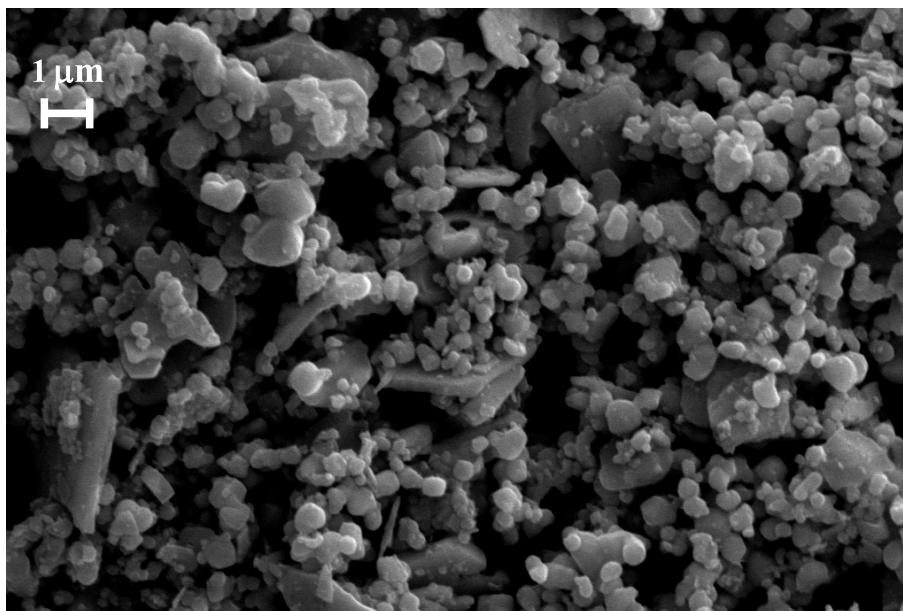
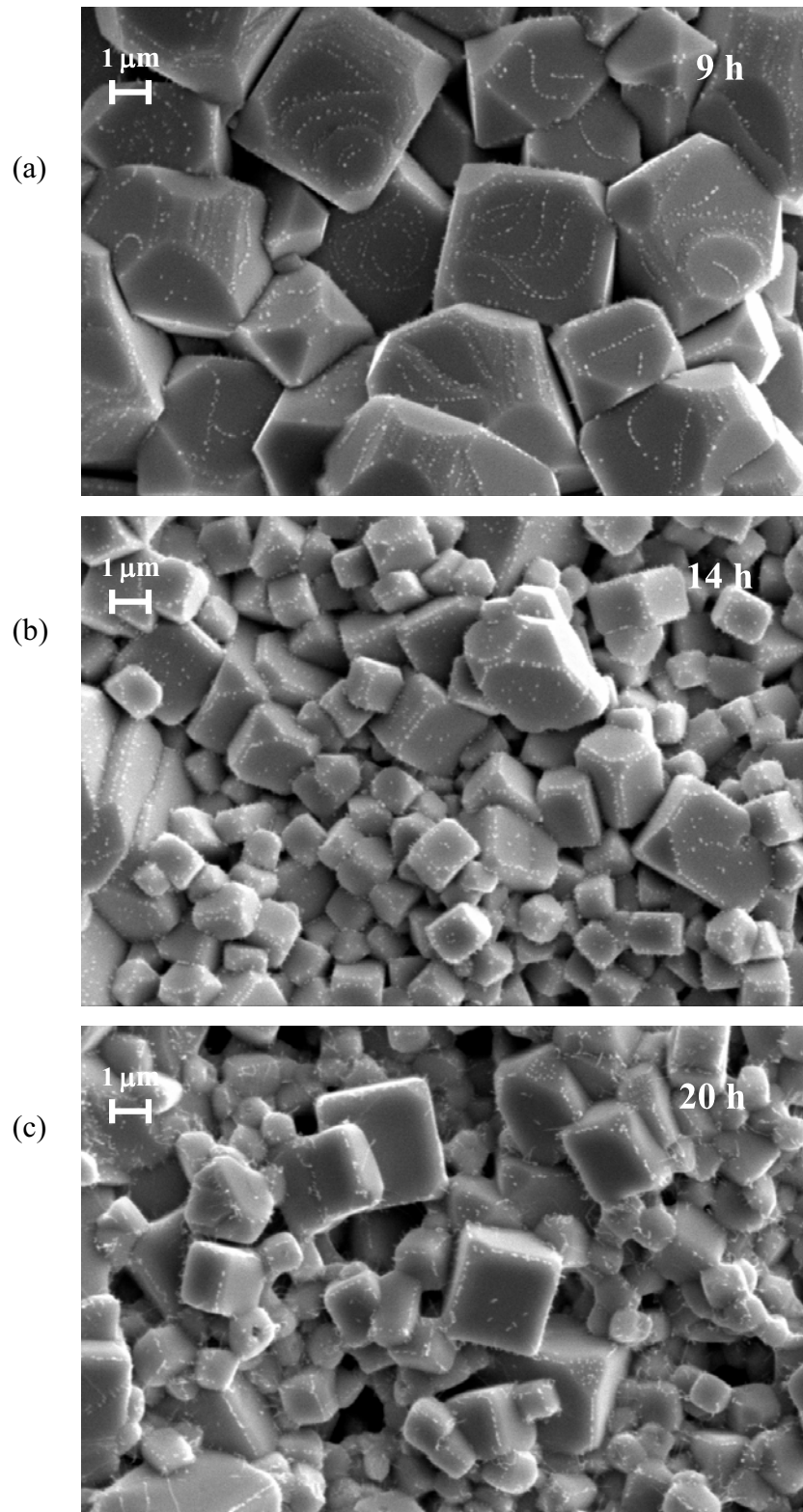


Fig.4

**Fig.5**

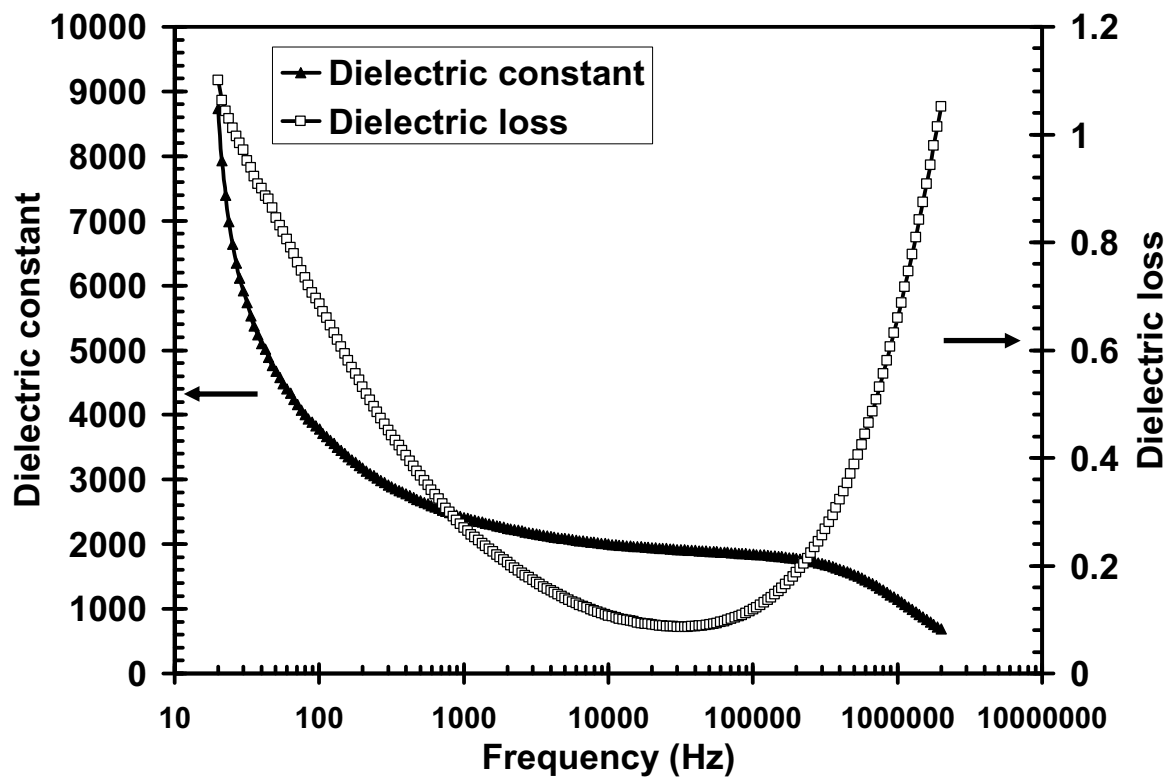


Fig.6

Novel Route for Preparation of Barium Iron Niobate ($\text{BaFe}_{0.5}\text{Nb}_{0.5}\text{O}_3$) Ceramic: Microwave Assisted

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Abstract

$\text{BaFe}_{0.5}\text{Nb}_{0.5}\text{O}_3$ (BFN) ceramic is a ferroelectric relaxor material, exhibiting very high value of dielectric constant over wide range of temperature. Therefore, it has been frequently used in high voltage capacitors and other applications. In this research, the new route to prepare BFN ceramic is explored. The reaction to obtain the BFN compound proceeds by wet chemical process of the precursors in a microwave digestion container. The solutions of precursors are heated by microwave irradiation. Various experimental parameters including reaction time in microwave vessel and calcined temperature are investigated. The progress of reaction is followed by utilizing X-ray diffraction and Fourier transformed infrared spectrometry. It can be seen that the structure of BFN starts to form when calcined at 750 °C for 5 hrs. Increasing the temperature to 850 °C and 950 °C accelerates the reaction process. For the calcination at 850 °C, the pure BFN is obtained at 18 hrs. We also observe that the reaction time of precursors in microwave vessel is an important parameter for obtaining the BFN with high purity.

Keywords : ferroelectric relaxor, microwave assisted synthesis, BFN

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Poster Presentation

Microstructure of BaFe_{0.5}Nb_{0.5}O₃ Ceramic Prepared by Chemical Process with Microwave Irradiation

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BaFe_{0.5}Nb_{0.5}O₃ (BFN) ceramic is a perovskite-type compound that exhibits very high value of dielectric constant over wide range of temperature. The high dielectric property of this material makes it a promising candidate for capacitor application. In this research, the BFN ceramic is prepared by wet chemical process accompanied with microwave irradiation. Various experimental parameters including reaction time in microwave vessel and calcined temperature are investigated. The phase identification of calcined powders is performed by utilizing X-ray diffraction and Fourier transformed infrared spectrometry. The structure of BFN starts to form when calcined at 750 °C for 5 h. Increasing the temperature to 850 °C and 950 °C accelerates the reaction process. For the calcination at 850 °C, the pure BFN is obtained at 18 h. We also observe that the reaction time of precursors in microwave vessel is an important parameter for obtaining the BFN with high purity. The powders is then pressed into pellet and sintered at 1250 °C for 4 h. The microstructure of the ceramics such as grain size and shape, examined by the scanning electron microscopy, is found to vary with experimental parameters. The calcination at high temperature causes higher crystallinity. Moreover, the grain size decreases with increasing calcination time.

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Microstructure of BaFe_{0.5}Nb_{0.5}O₃ Ceramic Prepared by Chemical Process with Microwave Irradiation

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N. Sriprang and G. Rujijanagul*

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**Characterization of $\text{BaFe}_{0.5}\text{Nb}_{0.5}\text{O}_3$ prepared by
a simple new chemical route**
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Abstract

The ferroelectric $\text{BaFe}_{0.5}\text{Nb}_{0.5}\text{O}_3$ (BFN) ceramic is a perovskite-type compound which exhibits very high dielectric constant over wide range of temperature. The high dielectric property of this material makes it a promising candidate for capacitor applications. However, most researchers have synthesized BFN by solid-state reaction which requires high-temperature conditions. Therefore, in this research, we proposed a new chemical route to prepared BFN fine powder. Various processing parameters of the new method were studied in order to obtain the pure phase of BFN. Properties of the BFN powders were also investigated.