CHAPTER III
EXPERIMENT

3.1 Materials

Glacial acetic acid and methyl alcohol were purchased from Italmar. The Polyvinylidene (PVDF) samples was provided by Solvay (solef 1008). Barium acetate and Dimetylformamide was purchased from Lab Scan. Strontium acetate (99.995%) and titanium tetra-n-butoxide (97%) were purchased from S.M. Chemical. All chemicals were used without further purification.

3.2 Equipment

3.2.1 Fourier Transformation Infared Spectroscopy (FTIR)

The Crystalline of PVDF were measured by a Fourier Transformation Infrared Spectrophotometer (FTIR). The measurements were made in absorbance mode using a Bruker FTIR Spectrometer, model Vector 3.0, using 32 scans per resolution.

3.2.2 Thermogravimetric Analysis (TGA)

Thermal degradation of PVDF, barium strontium titanate (BST) and composite was performed by a high resolution TG-DTA Pyris Diamond (Perkin Elmer). Samples were loaded on a the alumina pan heated from 50°C to 1150°C with a heating rate of 20°C/min under N_2 flow

3.2.3 Differential Scanning Calorimeter (DSC7)

Heating profiles of PVDF film and PVDF composites were performed by a differential scanning calorimeter 7, DSC 7 (Perkin Elmer) at a heating rate of 20 °C/min. The samples were heated from 30°C to 300°C.

3.2.4 X-ray Diffraction (XRD)

A crystal phase and structure of PVDF ,BST and composites were analyzed by X-ray diffraction (Rigaku, model Dmax 2002) with Ni-filtered CuKα radiation operated at 40 kV and 30 mA.

3.2.5 Scanning Electron Microscope (SEM)

Microstructures and surface morphology of BT/BST were performed by a scanning electron microscope (JEOL, model JSM 2590).

3.2.6 Pycnometer

The apparent density of BST powder and PVDF/BST composites were measured by pycnometer (Quantachrome, Ultrapycnometer 1000) under helium purge at pressure of 20 psi.

3.2.7 Transmission Electron Microscope (TEM)

Particle size of barium strontium titanate was measured by a transmission electron microscope TEM 100 keV (JEOL, model MJEM-1230).

3.2.8 Impedance/Gain-Phase Analyzer

Dielectric properties of BST ceramics, PVDF and PVDF/BST composites were measured by impedance/gain-phase analyzer (Hewlett Packard., model 4194A) in parallel capacitance (C_p) mode, with frequency from 1 kHz to 10 MHz at room temperature. The specimens were prepared by sputtering gold as a electrode on both sides of the specimens. Before electroding, the major faces of specimens were polished parallel with silicon carbide powders size of 13 and 5 μm in water slurry, respectively, and geometries were recorded. The dielectric constant of materials was calculated from the capcaitance by using the following equation:

$$\varepsilon = \frac{Cd}{\varepsilon_0 A}$$

where C is the capacitance (F), ε_0 the free space dielctric constant value (8.85×10^{-12} F/m), A the capacitor area (m^2), and d the thickness of specimens.

3.2.9 Ferroelectric Measurement Test System

The polarization and electric field characteristics (Hysteresis loop) were measured by RT66A: standardized ferroelectric measurement test system. Voltage in the range of 1000-4000 Volts was applied to the specimens, which were immersed in silicone oil at room temperature to observed hysteresis loops.

3.2.10 d_{33} Meter

Stress piezoelectric coefficients (d_{33}) of the polarized films were obtained from d_{33} meter (APC Int. Ltd., model 8000) operating at frequency of 1000 Hz and a time interval of 24 h after film polarization.

3.2.11 Compression Molding Machine

PVDF and PVDF/BST composite samples were preformed by a compression press (Wabash, model V50H-18-CX).

3.3 Methodology

3.3.1 PVDF Film Preparation

Poly (vinylidene fluoride) powder manufactured from Solvay Company (Belgium) (Solef 1008) were used. Two methods were utilized to fabricate PVDF thin film. First was solution casting which PVDF powder was dissolved in DMF solution which ratio was 10g/100ml. The viscous solution is then cast on glass substrate. After the solvent evaporation in a stove at 60 ° C for 20 minutes under vacuum the sample films of 50-100μm thickness were obtained. Another method was compression molding which PVDF films were prepared by Wabash compression. From this method, PVDF powder was compressed at 174 ° C for 20 minutes under pressure 15 tons. The thickness of the prepared films ranged 100-200 μm. Film produced from compression molding was stretched by stretching instrument to obtain film at different ratio as constant stretching rate of 5 mm/min.

3.3.2 Barium Strontium Titante Preparation

3.3.2.1 Sol-gel process of Barium Strontium Titanate

$(Ba_{1-x}Sr_x)TiO_3$ was prepared by using x=0, and 0.3 respectively. Equal moles of barium acetate and strontium acetate were dissolved separately into methyl alcohol in the presence of glacial acetic acid. The solutions were then mixed and stirred. The prescribed amount of titanium-n-butoxide was added into the mixture. All the materials mentioned above were thoroughly mixed to prepare a stable solution with uniform composition. After that the solution was poured into an alumina crucible and heated by using a 2-step thermal decomposition method which is shown in Figure 3.1 in order to decompose the precursors and to crystallize the barium strontium titanate.

3.3.2.2 Ceramic Processing

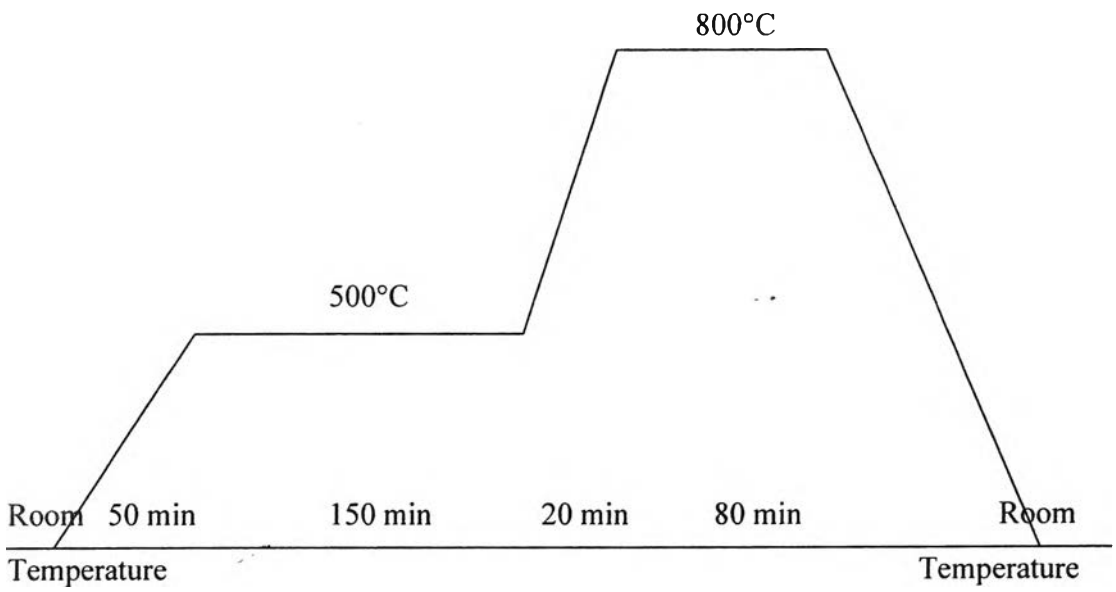

Figure 3.1 Temperature program for the 2-step thermal decomposition method.

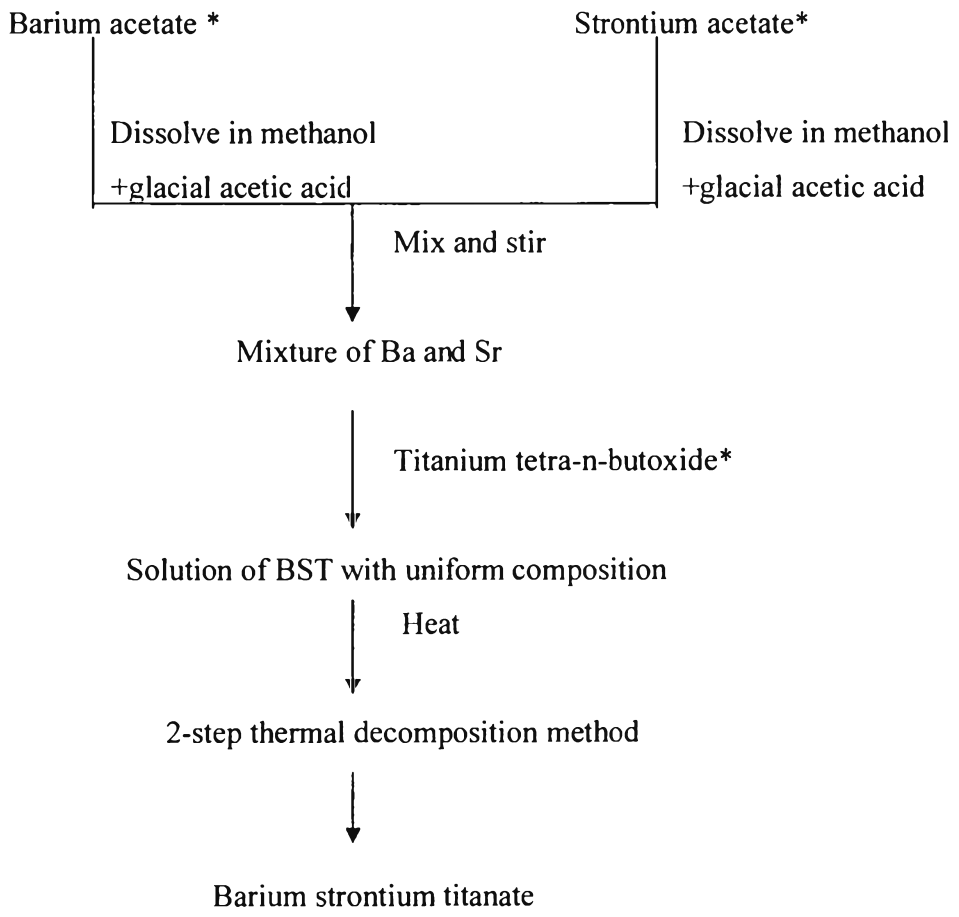

Figure 3.2 Barium Strontium Titanate Preparation.

Table 3.1 Weight of each chemicals in preparing barium titanate/barium strontium titanate

	Barium acetate	Srontium acetate	Titanium tetra-n-butoxide
$BaTiO_3$	5.3800g	-	7.20ml
$(Ba_{0.70}Sr_{0.30})TiO_3$	4.4600g	1.5400g	8.52ml

3.3.2.3 Ceramic Pellet Preparation

Polyvinyl alcohol (PVA) was weighed (4% by weight of BST powder) then dissolved it into acetone. After that, this solution was poured into ceramic powders and stired until a homogeneous slurry material was obtained. Then this slurry material was left overnight under air, followed by grinding and sieving to obtained the powder. Then the powders were pressed in a press form by using a force of around 7-8 tons and a sintering process was performed by putting the ceramic pellets into an oven by using the following temperature program as shown in Figure 3.3 in order to obtain a sintered product for electrical measurement.

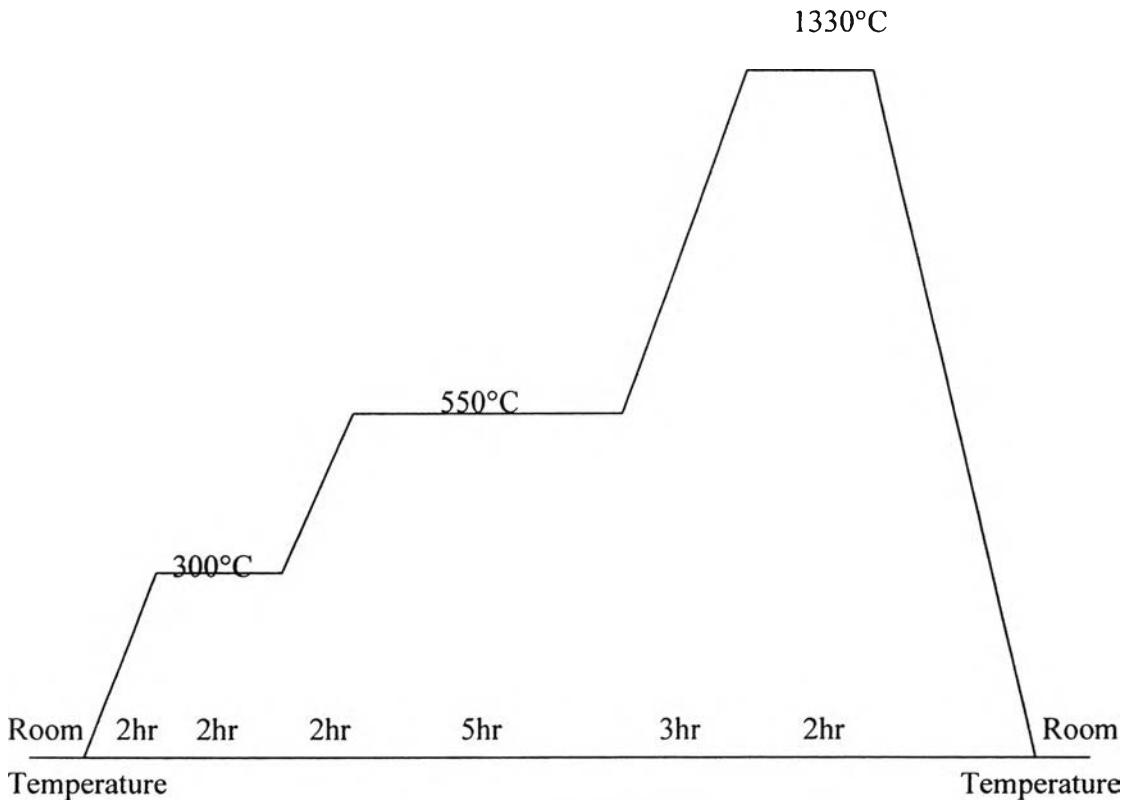

Figure 3.3 Temperature program for the sintering process of ceramic pellet.

3.3.3 Composite Preparation

PVDF powder supplied by Solef 1008 was dissolved in dimethly formamide (DMF) at 60°C. For composite preparation the polymer/solvent ratio was 10g/100 ml .Proportionate quantity of $Ba_{0.7}Sr_{0.3}TiO_3$ powder at different calcine temperature was added in the polymer solution. It was homogenized by magnetic stirrer. Addition mixing by ultra sonic was used to guarantee that the powder agglomerated were broken. The solution was dried by heating at 100 ° C. The composite film was prepared by a Wabash compression by pressing dried solution at 174 °C for 20 minutes under pressure of 10 tons. The thickness of the prepared films was ranged between 100-200 μm. Follows the above method, the composite of 30, 50 and 70% by ceramic volume were fabricated.