CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Chemicals:

- Barium acetate (Aldrich)
- Strontium acetate hydrate 99.995% (Aldrich)
- Magnesium acetate tetrahydrate 99% (Aldrich)
- Titanium tetra-n-butoxide 97% (Aldrich)
- Poly(butylene succinate) grade FZ71PD

3.1.2 Solvents:

- Glacial acetic acid (Lab scan)
- Chloroform (Lab scan)
- Methyl alcohol (Lab scan)

3.2 Equipment

3.2.1 X-Ray Diffraction (XRD)

The crystal phases and structure of sol-gel magnesium-doped barium strontium titanate ceramic powder, $Ba_{1-x-y}Sr_xMg_yTiO_3$ were analyzed by X-Ray Diffraction with Ni-filter CuKα radiation operated at 40 kV and 30 mA with scan speed 5.00 deg/min from the 2Θ range of 20.00 - 80.00 degree.

3.2.2 Scanning Electron Microscope (SEM)

Microstructure and dispersion fractured-surface morphology of BST powders in PBS-composite were observed by a scanning electron microscope (SEM) at voltage of 15 kV.

3.2.3 Agilent E4980A LCR Meter

Dielectric measurement of the sol-gel magnesium-doped barium strontium titanate powder, $Ba_{1-x-y}Sr_xMg_yTiO_3$ and the composites were measured by Agilent E4980A Impedance/Gain-Phase Analyzer in parallel capacitance (C_p) mode, with frequency from 20 Hz to 2 MHz at room temperature. The dielectric constant (ε) of the composites were calculated from the sample thickness and capacitance by using the following equation:

$$\varepsilon = \frac{Cd}{\varepsilon_0 A} \qquad (3.1)$$

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

3.2.4 Agilent E4991A RF Impedance/Material Analyzer

Microwave dielectric properties of the sol-gel magnesium-doped barium strontium titanate powder, $Ba_{1-x-y}Sr_xMg_yTiO_3$ and the PBS/BST composites thin-film were measured by Agilent E4911A Network Analyzers with frequency from 1 MHz to 1 GHz inside the chamber with variable temperature ranging from -60℃ to 150℃ with SU-261 Bench-top Type temperature and humidity chamber.

3.2.5 Compression Molding Machine

Poly(butylene succinate) and composites samples were produced by a Compression Press (Lab Tech, model LP 20) at the temperature about 150℃ and the pressure about 2500 psi.

3.2.6 Thermal Gravimetric Analysis (TGA)

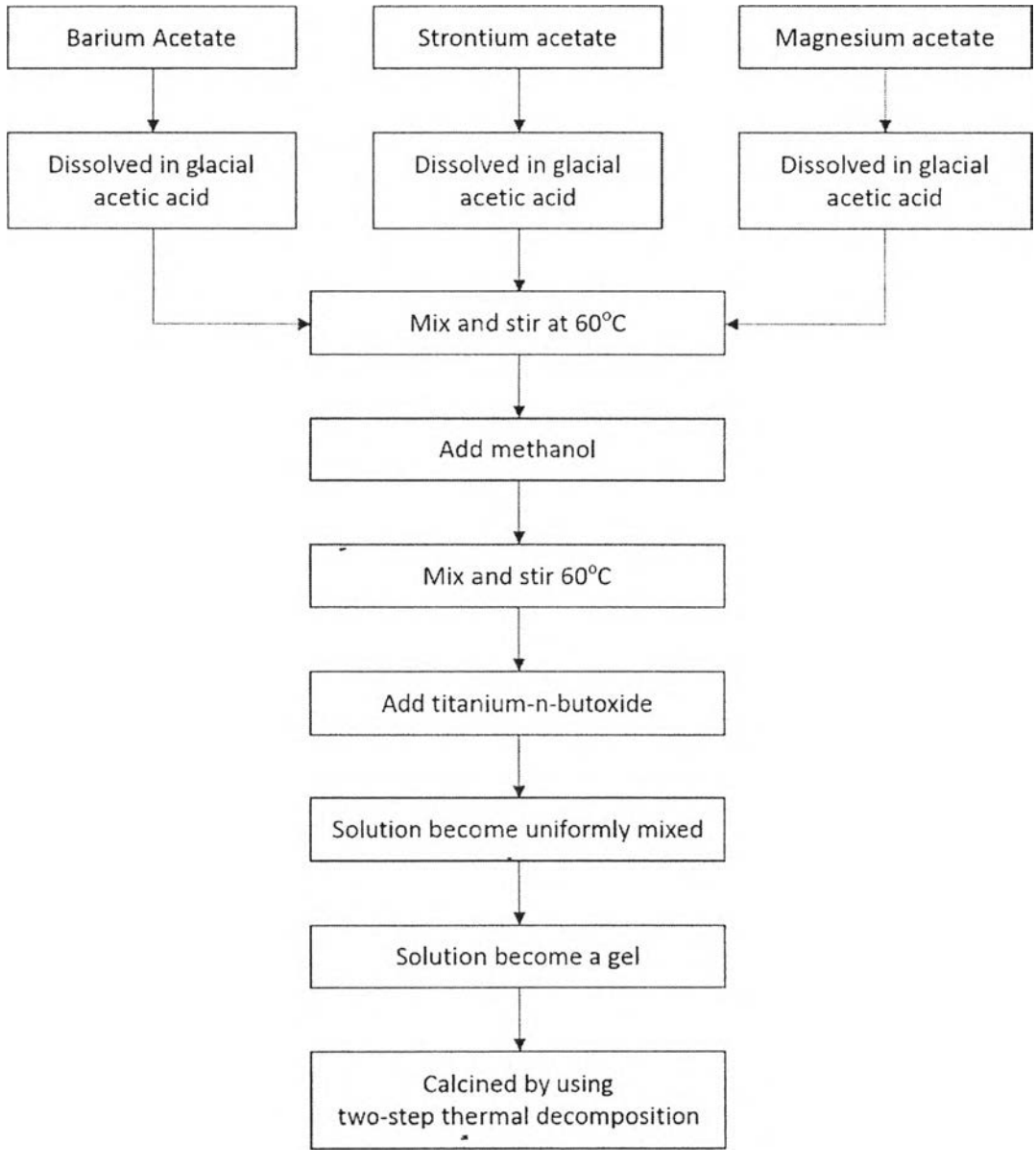
Residual weight and decomposition temperature of the poly(butylene succinate) and the composites were measured by Perkin Elmer Pyris Diamond TG/DTA instrument by using the platinum pan for reference and operating at the heat from 25℃ to 600℃ and heating rate of 10℃/min. The environment condition will be flow under O_2 with flow rate of 10 ml/min.

3.3 Experimental Procedures

3.3.1 Preparation Of Magnesium-doped Barium Strontium Titanate Powder ($Ba_{1-x-y}Sr_xMg_yTiO_3$)

Magnesium-doped barium strontium titanate, $Ba_{1-x-y}Sr_xMg_yTiO_3$ ceramic powder specimens were prepared by dissolving the stoichiometric amount of barium acetate, strontium acetate and magnesium acetate in 50 ml of acetic acid. Following by mixing of those three precursor solution and adding 50 ml of methyl alcohol. The solution were mixed and stirred to obtain a clear solution. Then an equimolar amount of titanium-n-butoxide was added into the mixture under vigorous stirring. The atomic ratio of Ba:Sr:Ti = 0.7:0.3:1.0, 0.6:0.4:1.0 and 0.5:0.5:1.0 will be

used in the addition of magnesium the atomic ratio of (Ba:Sr:Ti):Mg = 1.00:0.00 (0 mol%), 0.995:0.005 (0.5 mol%), 0.990:0.010 (1.0 mol%) and 0.980:0.020 (2.0 mol%). When the solution became a white gel, the magnesium-doped barium strontium titanate gels were taken to calcination process by using "2-step thermal decomposition" method in order to decompose of the organic compounds and crystallize the $Ba_{1-x-y}Sr_xMg_yTiO_3$ powders, the overall sol-gel process schematic is shown in Schematic 3.1 and the temperature profile in 2-step thermal decomposition is shown in Figure 3.1.



Schematic 3.1 Preparation of magnesium-doped barium strontium titanate powders.

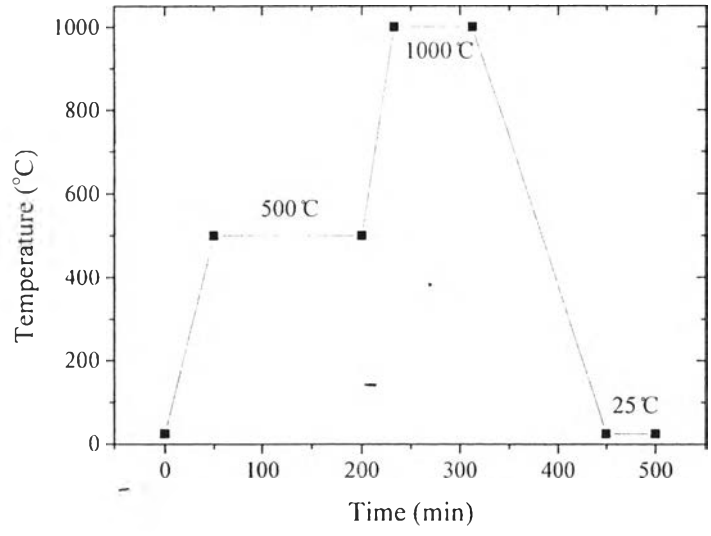


Figure 3.1 Two-step thermal decomposition temperature profile.

3.3.2 Preparation of BST-PBS Composite

The composite of poly(butylene succinate) or PBS and barium strontium titanate powder, $Ba_{0.7}Sr_{0.3}TiO_3$ were prepared by mixing two component using solution mixing method. Firstly, PBS pellets were dissolved in the chloroform solvent. Secondly, $Ba_{0.7}Sr_{0.3}TiO_3$ powder were added to the PBS solution in the composition of 0, 10, 20, 30, 40, and 50 wt%. Then, the PBS-BST mixtures were casted in the aluminum tray, dried in the oven at 60℃ for 5 hours and cut into small flakes. Finally, the composite flakes were fabricated as thin-film composite specimens with the thickness of 0.2 mm by using compression molding at the temperature about 150℃ and the pressure about 2500 psi.