

# Sintesis dan karakterisasi material sistem nanokomposit BaFe<sub>12</sub>O<sub>19</sub>/SrTiO<sub>3</sub> melalui proses pemaduan mekanik dan sonikasi daya tinggi = Synthesis and characterization of BaFe<sub>12</sub>O<sub>19</sub>/SrTiO<sub>3</sub> nanocomposite prepared by mechanical alloying and high power ultrasonic destruction processes

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## Abstrak

Barium hexaferrite (BaFe<sub>12</sub>O<sub>19</sub>) dan strontium titanate (SrTiO<sub>3</sub>) telah luas dikenal masing masing sebagai material magnet permanen dan piezoelektrik. Kedua jenis material tersebut sangat potensial untuk diaplikasikan pada pembuatan komponen produk magnet dan elektronik. Sifat ekstrinsik kedua jenis material tergantung kepada mikrostrukturnya yang sangat ditentukan pula oleh metode sintesis material yang diterapkan. Kajian literatur menunjukkan bahwa telah banyak dikembangkan berbagai macam metode sintesis senyawa magnetik BaFe<sub>12</sub>O<sub>19</sub> dan dielektrik SrTiO<sub>3</sub> dalam bentuk partikel halus dengan ukuran berskala nanometer. Kegiatan penelitian ini lebih difokuskan kepada sintesis dan karakterisasi material sistem nanokomposit BaFe<sub>12</sub>O<sub>19</sub>/SrTiO<sub>3</sub>, dimana senyawa BaFe<sub>12</sub>O<sub>19</sub> (kode BHF) memiliki fasa feromagnetik dan senyawa SrTiO<sub>3</sub> (kode STO) memiliki fasa feroelektrik dipersiapkan melalui metode pemaduan mekanik (mechanical alloying). Sedangkan pembuatan nanopartikel kedua senyawa diperoleh melalui penghalusan mekanik dan destruksi ultrasonik daya tinggi.

Material BHF dipersiapkan dari campuran prekursornya berupa serbuk BaCO<sub>3</sub> dan Fe<sub>2</sub>O<sub>3</sub>. Sedangkan material STO menggunakan prekursor SrCO<sub>3</sub> dan TiO<sub>2</sub>. Aparatus yang digunakan adalah planetary ball mill dengan perbandingan berat antara material dan ball mill adalah 1 : 10. Ukuran rata-rata partikel dievaluasi menggunakan particle-size analyzer (PSA) dan ukuran rata-rata kristalit dihitung menggunakan metode Williamson Hall Plot dengan software High Score Plus dari data pola difraksi x-ray masing-masing senyawa. Adapun sampel berupa material kristalin diperoleh setelah kompaksi serbuk hasil pemaduan mekanik dan pemanasan pada temperatur 1100°C selama 3 jam dimana kemudian sampel material kristalin tersebut dihaluskan kembali menggunakan ball mill selama 20 jam. Serbuk halus BHF dan STO tersebut masing-masing menjalani destruksi lanjut secara ultrasonik daya tinggi untuk menghasilkan nanopartikel. Hasil evaluasi dengan PSA dan Williamson Hall Plot data XRD terhadap material BHF memperlihatkan nanopartikel dicapai setelah destruksi ultrasonik selama 14 jam. Dalam hal ini hasil PSA menunjukkan ukuran partikel rata-rata BHF adalah 28 nm sedangkan hasil evaluasi ukuran rata-rata kristalit adalah 26 nm. Untuk STO diperoleh hasil evaluasi ukuran rata-rata partikel sebesar 144 nm dan ukuran rata-rata kristalit adalah 30 nm. Kedua jenis material dalam bentuk nanopartikel ini digunakan sebagai komponen nanokomposit sistem BHF-STO.

Berdasarkan hasil karakterisasi material komposit baik melalui pengujian XRD maupun permagraph bahwa sampel komposit tersusun dari dua fasa yaitu BaO.<sub>6</sub>(Fe<sub>2</sub>O<sub>3</sub>) dan SrTiO<sub>3</sub> yang dipastikan dari pola difraksi dan sifat kemagnetannya. Dari kajian efek destruksi ultrasonik terhadap partikel STO dapat disimpulkan bahwa ukuran partikel rata rata dapat direduksi 8 kalinya yaitu dari ukuran 797 nm menjadi 144 nm setelah durasi waktu destruksi 14 jam. Sedangkan untuk partikel BHF tereduksi 100 kalinya yaitu dari 2931 nm menjadi 26 nm pasca durasi waktu destruksi yang sama.

Penelitian ini juga mempelajari perilaku kinetika pertumbuhan ukuran kristalit fasa-fasa material penyusun material komposit dalam sistem komposit yang mengikuti persamaan Avrami. Berdasarkan kajian kinetika dapat diketahui energi aktivasi pertumbuhan kristalit fasa material STO dan BHF masing masing adalah 16 kJ.mol<sup>-1</sup> dan 4 kJ.mol<sup>-1</sup>.

Dapat disimpulkan bahwa kombinasi antara teknik penghalusan mekanik dan destruksi sonikasi daya tinggi terhadap partikel kristalin BHF dan STO dapat dijadikan metode alternatif yang efektif untuk menghasilkan nanopartikel.

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Barium hexaferrite (BaFe<sub>12</sub>O<sub>19</sub>) and strontium titanate (SrTiO<sub>3</sub>) are well established permanent magnet and piezoelectric materials which are technologically and scientifically attractive due to their potential for various applications in the field of magnetic electronics functional materials. The extrinsic properties of these materials depend largely on the microstructure, which in turn depends on the method of synthesis. Different methods have been developed for the preparation of ultrafine BaFe<sub>12</sub>O<sub>19</sub> and SrTiO<sub>3</sub> particles in nanometer scale. In this work, research activities were focused on synthesis and characterization of BaFe<sub>12</sub>O<sub>19</sub>/SrTiO<sub>3</sub> nanocomposites in which ferromagnetic materials of BaFe<sub>12</sub>O<sub>19</sub> phase (coded BHF) and a ferroelectric materials of SrTiO<sub>3</sub> phase (coded STO) were prepared by a mechanical alloying technique. In addition, nanoparticles of BHF and STO were obtained by physical destruction through a combined method between mechanical milling and high power ultrasonication.

BHF materials were made of their precursors which consisted of the mixture between BaCO<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. Whereas for STO materials the precursors were SrCO<sub>3</sub> and TiO<sub>2</sub>. The process of mixing and alloying was carried out under the used of a planetary ball mill apparatus with a weight ratio between mixture and ball mill was 1:10. The mean particle size of milled powders was further characterized by Particle Size Analyzer (PSA). Whereas the mean crystallite size was derived from Williamson Hall Plots using the High Score Plus software to evaluate data of x-ray diffraction patterns for each materials. The crystalline materials were obtained after sintering step at 1100°C for 3 hours to the green compact samples which further followed by remilling the sintered samples for 20 hours. Further refining the powders for BHF and STO was carried out under the use of a high power sonicator for 14 hours to produce nanoparticles.

Results of evaluation indicated that the mean particle size of BHF and STO was respectively 28 nm and 144 nm which refer to results of particles characterization by PSA whereas for their mean crystallite size were respectively 26 nm and 30 nm. Those nanoparticles of BHF and STO were then used as component materials in BHF-STO nanocomposite system. According to results of characterization for composite materials by XRD and permagraph, it was found that the composites consisted of two phases which were identified as BaO.<sub>6</sub>(Fe<sub>2</sub>O<sub>3</sub>) and SrTiO<sub>3</sub> based on their diffraction pattern and magnetic properties.

Further to the characterization results, it was also found that the mean particle size of STO was reduced 8 times in which the mean size of 797 nm was brought down to 144 after ultrasonically destruction for 14 hours. However, much larger reduction in particle sizes were obtained in BHF in which the initial mean particle size of 2931 nm was reduced 100 times downed to 26 nm after the same duration periode of ultrasonic destruction.

Crystallite growth kinetics behavior of BHF and STO phases in the composite system was also studied in which data of mean crystallite sizes at different sintering temperatures and time were fitted into the Avrami equation. It was found that the activation energy for crystallite growth kinetics of BHF and STO is 16 kJ.mol<sup>-1</sup> and 4 kJ.mol<sup>-1</sup> respectively.

We conclude that mechanical alloying coupled with ultrasonication can be used as an effective alternative tools for the preparation of fine and homogeneous powder materials leading to nanoparticle-based materials.