Ultrafast Microwave Hydrothermal Synthesis of BiFeO₃ Nanoplates

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We report the synthesis of 100 nm–sized single-crystalline BiFeO₃ (BFO) nanoplates, with thickness ranging from 20 to 160 nm and lateral size of submicrometers, via a simple and very rapid (1–2 min) microwave-assisted hydrothermal approach. We show that the microwave treatment gives comparable improvement in crystallinity of BFO nanocrystals with respect to traditional hydrothermal processes while requiring significantly less time and energy. In addition, we show that microwave radiation power, reaction time, and alkali concentration play important roles in the crystallinity and morphology of the products. We discuss a possible formation mechanism of the nanoplates based on our experimental results. Additionally, the BFO nanoplates exhibit weak ferromagnetic properties at room temperature, which we attribute to the size-confinement effect on magnetic ordering. The present microwave hydrothermal method has great potential in large-scale fabrication of BFO nanomaterials as well as other composite functional materials due to significantly reduced time and energy.

I. Introduction

Multiferroic materials that exhibit a coupling of the electrical and magnetic order parameters in the same phase, have attracted increasing interest because of their potential applications in data storage, spintronics, sensors, quantum electromagnets, photonics, and electronics. Typical multiferroics belong to the group of the perovskite transition-metal oxides (e.g., BiFeO₃), and include rare-earth manganites and ferrites (e.g., TbMnO₃, HoMn₂O₅). The recent emergence of new types of multiferroics such as Bi₂FeCrO₆ and Bi₂CoMnO₆, with large polarization and magnetization, has attracted increasing interest because of their potential applications in data storage, spintronics, sensors, quantum electromagnets, photonics, and electronics. Typical multiferroics belong to the group of the perovskite transition-metal oxides (e.g., BiFeO₃), and include rare-earth manganites and ferrites (e.g., TbMnO₃, HoMn₂O₅). We report the synthesis of 100 nm–sized single-crystalline BiFeO₃ (BFO) nanoplates, with thickness ranging from 20 to 160 nm and lateral size of submicrometers, via a simple and very rapid (1–2 min) microwave-assisted hydrothermal approach. We show that the microwave treatment gives comparable improvement in crystallinity of BFO nanocrystals with respect to traditional hydrothermal processes while requiring significantly less time and energy. In addition, we show that microwave radiation power, reaction time, and alkali concentration play important roles in the crystallinity and morphology of the products. We discuss a possible formation mechanism of the nanoplates based on our experimental results. Additionally, the BFO nanoplates exhibit weak ferromagnetic properties at room temperature, which we attribute to the size-confinement effect on magnetic ordering. The present microwave hydrothermal method has great potential in large-scale fabrication of BFO nanomaterials as well as other composite functional materials due to significantly reduced time and energy.

Molecular interaction with high-frequency electromagnetic radiation, which is different from that of conventional thermal treatment by convection current [Fig. 1(a)]. In M-H processing, the precipitate can be rapidly dissolved in aqueous solution to provide a saturated solution, resulting in enhancement of the reaction kinetics by one to two orders of magnitude by high-frequency electromagnetic radiation (2.45 GHz). In addition to the extensively studied BaTiO₃, relevant for designing new multifunctional materials combining magnetic, ferroelectric, and optoelectronic properties. Two-dimensional (2D) nanomaterials such as nanosheets and nanoplates have been studied extensively because their anisotropic shape is advantageous with respect to irregular-shaped nanocrystals for constructing nanodevices. The design and morphological control of crystal facets is a commonly employed strategy to optimize the physical and chemical properties of various crystalline semiconductors. Recent developments in the synthesis of 2D crystalline nanosheets/plates show promising properties for developing a new generation of optoelectronic devices and high-performance catalysts. Recently, Lu et al. reported the synthesis of 2D BFO plates using the surfactant cetylethyltrimethylammonium bromide. However, synthesizing single-crystalline planar BFO nanosheets or nanoplates with controllable crystallographic facets by template- or surfactant-free solution routes is still a major challenge. Numerous reports have described the synthesis of BFO materials by various routes that involve solid-state reactions, rapid molten salt sintering, mechanochemical synthesis, sol–gel method, or wet chemistry. The majority of these processes require long reaction times and high temperatures, therefore involving high-energy consumption and cost. The most commonly used conventional solid-state synthesis of BFO crystals requires a prolonged treatment at considerably high calcination temperatures (800 °C or higher), causing a loss of bismuth and severe impurity contaminations. The hydrothermal technique is becoming one of the most important tools for advanced materials processing, as it is a simple and low-cost route with and low reaction temperature, and also because it is very useful for producing various new types of nanohybrid and nanocomposite materials. Recently, hydrothermal processing methods have been shown to yield highly crystalline BFO products at low temperatures (~200 °C). Although this method can be efficient in the synthesis of materials using relatively low temperatures (100–250 °C), its main disadvantage is related to the long processing times due to slow reaction kinetics at relevant temperatures. Microwave-assisted hydrothermal (M-H) methods are becoming widespread for the synthesis of nanomaterials as they offer a simple, low-cost approach to obtain highly crystalline nanocrystals within a very short reaction time with high yields and good reproducibility. Microwave heating [Fig. 1(b)] provides energy to the reactants by means of molecular interaction with high-frequency electromagnetic radiation, which is different from that of conventional thermal treatment by convection current [Fig. 1(a)]. In M-H processing, the precipitate can be rapidly dissolved in aqueous solution to provide a saturated solution, resulting in enhancement of the reaction kinetics by one to two orders of magnitude by high-frequency electromagnetic radiation (2.45 GHz). In addition to the extensively studied BaTiO₃, relevant for designing new multifunctional materials combining magnetic, ferroelectric, and optoelectronic properties. 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recently the synthesis of several important inorganic materials such as Tellurium (Te), \( \text{TiO}_2 \), and \( \text{Fe}_2\text{O}_3 \) was demonstrated in one step under M-H conditions. Up to now, a few reports have also described the fabrication of BFO nanostructures by the M-H approach, yet the treatment time needed for all these reports is still long (30 min or more).

Here, we report a rapid and simple solution-based method for the synthesis of BFO nanoplates under M-H conditions within a very short reaction time (\( \sim 1-2 \) min). The final products were characterized by X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), high-resolution transmission electron microscopy (HRTEM), and X-ray photoelectron spectroscopy (XPS). Various parameters influencing the final products such as reaction time, radiation power, and alkali concentration were discussed in detail in this study. We further investigated the magnetic properties of the as-synthesized BFO nanoplates.

II. Experimental Procedure

(1) Synthesis

In a typical synthesis, 0.0025 mol Bi(NO\(_3\))\(_3\)-5H\(_2\)O (\( > 99.99\% \)) and 0.0025 mol FeCl\(_3\)-6H\(_2\)O (\( > 98\% \)) were added in 50 mL of ethylene glycol (C\(_2\)H\(_6\)O\(_2\)) solution by stirring until completely dissolved. Secondly, aqueous NH\(_3\)-C\(_2\)H\(_2\)O (0.25 M) solution was slowly introduced into the above solution until the pH value reach 10–11. Subsequently, the aqueous solution was further stirred for 30 min. After filtering and rinsing to remove residual Cl\(_{-}\), NO\(_3\)-, NH\(_3\)-C\(_2\)H\(_6\)O\(_2\), the precipitate was redispersed in 60 mL of pre-prepared NaOH solution. Then 15 mL of the mixture was transferred into a Microwave Acid Digestion Autoclave (Parr Instrument Company, St Moline, IL) with 23 mL capacity. Microwave processing was performed in a Panasonic Inverter Microwave Oven (2.45 GHz) with radiation power varying from 360 to 720 W. (Sample preparation parameters are summarized in Table II.) This microwave oven applies a precise power in contrast to older models, where an average power can be achieved through the maximum power applied over a variable duty cycle. For comparison, a conventional hydrothermal (C-H) experiment was conducted using the same solution with a stainless steel autoclave of the same volume and filling factor. It was placed in an oven at 170°C for 16 h without moving or shaking (the final sample is listed as CH). After the autoclaves were cooled down to RT, the resulting products were collected, washed for several times using ethanol and distilled water, respectively, and dried at 70°C for 4 h.

(2) Characterization

The crystal structure of the as-synthesized powder was examined by XRD (D8 Advance; Bruker, Billerica, MA) using CuK\(_\alpha\) radiation (\( \lambda = 1.5418 \) Å). The morphology of the samples was analyzed using FESEM (JEOL JSM740IF, Tokyo, Japan) at an acceleration voltage of 5 kV. HRTEM images and selected-area diffraction pattern (SAED) were taken with a JEOL 2100F microscope operated at an accelerating voltage of 200 kV. The chemical composition of the sample was analyzed using XPS (ESCALAB 2201-XL, East Grinstead, UK) equipped with an AlK\(_\alpha\) (1486.6 eV) monochromatic source. All binding energies of various peaks were calibrated using the binding energy of C1s (284.6 eV). The magnetization as a function of the magnetic field (hysteresis loops) was investigated using a superconducting quantum interference device (SQUID, MPMS-XL7, San Diego, CA) with an applied magnetic field of 1 T. All measurements were performed at RT.

III. Results and Discussion

(1) Characterization of BiFeO\(_3\) Nanoplates

Crystal structure characteristics of samples prepared by M-H (MH-5) and C-H method were investigated by XRD first, as depicted in Fig. 2. For the sample obtained by C-H treatment [Fig. 2(a)], the main phase can be indexed to a rhombohedral lattice of BFO with the space group \( R3c \) (JCPDS card No. 86-1518). However, a trace amount of secondary phase of Bi\(_2\)Fe\(_4\)O\(_9\) (JCPDS card No. 72-1832) is present, in addition to the main phase. On the other hand, for the sample synthesized by M-H treatment [Fig. 2(b)], all the reflection peaks in the pattern belong to the rhombohedral structure of BFO, and no noticeable peaks from secondary phases are detected. The strong and sharp peaks suggest that
both as-synthesized samples are highly crystalline. In addition, the intensity of the peaks of MH-5 is similar to those of sample CH, indicating that the microwave treatment gives comparable improvement in crystallinity with respect to traditional hydrothermal process while requiring significantly less time. To obtain more detailed information on the crystal structure, the patterns collected for both samples have also been refined by the Rietveld method using FULLPROF. The space group \( R3c \) in its hexagonal representation was used as the basis, and the starting values for all Rietveld refinements were \( a_{hex} = 5.63 \text{Å}, c_{hex} = 13.98 \text{Å} \), and atomic position Bi (0,0,0). Fe(0.0,0.224), O(0.441,0.023,0.949).\(^{41}\) The structure parameters have been estimated and are summarized in Table I.

The TEM image of sample prepared by M-H method (MH-5) [Fig. 3(a)] exhibits a 2D platelike morphology with submicrometers in width and about 50–80 nm in thickness. SAED taken from an individual nanoplate [inset of Fig. 3(a)] show patterns the presence of sharp diffraction spots, which are indicative of the formation of well-defined, single-crystal-line BFO nanoplates. Figure 3(b) shows the HRTEM image recorded from the selected area of the nanoplate shown in Fig. 3(a). The fringe spacing of 0.395 and 0.282 nm matches the spacing of the (012) and (104) lattice planes of BFO in agreement with the SAED patterns. The unit cell of BFO can be described in a hexagonal frame of reference with its c-axis parallel to the diagonal of the perovskite cube (i.e., \{001\}_{hex}/\{111\}_c), so the \{012\}_{hex} corresponds to \{100\}_c pseudocubic crystal plane. Therefore, the results demonstrate that the BFO nanoplates are exposed with \{100\}_c planes, similar to the BFO nano/microcubes reported previously.\(^{12,13}\)

X-ray photoelectron spectroscopy measurements were performed to further indentify the chemical composition of the as-obtained BFO nanoplates (MH-5). The wide-scan XPS spectrum shown in Fig. 4(a) confirms that the BFO nanoplates consist of Bi, Fe, and O. Double Bi 4f peaks at binding energies of 164.0 and 158.6 eV are corresponding to Bi\(^{3+}\), shown in Fig. 4(b). In addition, the peaks at 710.6 and 724.3 eV [Fig. 4(c)] correspond to Fe\(^{3+}\), whereas no peak of Fe\(^{2+}\) can be detected, which demonstrates that the oxidation state of Fe is only 3+ in the BFO nanoplates. The XPS peak of O1s [Fig. 4(d)] shows only one symmetrical peak at binding energies of 529.8 eV, verifying that the BFO nanoplates are single phase, which is consistent with reports on BFO nanoparticles obtained by the rapid molten salt sintering method.\(^{25}\)

Several studies have investigated the role of microwave radiation in the synthesis of inorganic materials.\(^{34,42,43}\) Our results show that through the use of the M-H treatment, the reaction time needed for synthesizing BFO nanocrystals with comparable crystal quality can be reduced from 16 h when using the C-H method to only 1–2 min. Microwave heating is significantly faster due to the high penetration depth of microwave radiation, which enhances the reaction kinetics. As a result, the enhancement of crystallinity of BFO performed in short periods via microwave treatment could be attributed to very rapid (within minutes) heating of the precursor and fewer thermal gradients compared with C-H treatment (Fig. 1). Moreover, in the present case, the use of an inverter microwave oven that provides a higher intensity in the center of the oven, also contributes to the very rapid reaction of obtaining BFO nanocrystals.

### Table I. Relevant Parameters from Rietveld Refinement XRD Patterns of BiFeO\(_3\) Samples Prepared by C-H and M-H (MH-5) Method; Space Group \( R3c \)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Lattice parameters</th>
<th>Atom coordinates</th>
</tr>
</thead>
<tbody>
<tr>
<td>CH</td>
<td>( a = 5.577(1) \text{Å} ) Bi ( 6a )</td>
<td>( x ) ( y ) ( z ) ( \chi^2 )</td>
</tr>
<tr>
<td></td>
<td>( c = 13.863(4) \text{Å} ) Fe ( 6a ) O ( 18b )</td>
<td>0 0 0 1.68</td>
</tr>
<tr>
<td>MH-5</td>
<td>( a = 5.584(2) \text{Å} ) Bi ( 6a ) Fe ( 6a ) O ( 18b )</td>
<td>0.453(9) 0.02(1) 0.951(3) 2.04</td>
</tr>
<tr>
<td></td>
<td>( c = 13.876(6) \text{Å} )</td>
<td>0.480(2) 0.040(1) 0.959(5)</td>
</tr>
</tbody>
</table>

**Fig. 3.** (a) TEM image of the BiFeO\(_3\) nanoplate (MH-5), and inset shows typical SAED patterns taken from the nanoplate; (b) HRTEM image taken from the selected area of the nanoplate in (a).
powers between 360 and 720 W (MH-1, MH-2, and MH-5), all the diffraction peaks can be indexed to the rhombohedral structure of BFO with the literature value of JCPDS No. 86-1518, except a trace amount of Bi$_2$Fe$_4$O$_9$ which appears as a secondary phase in MH-1. The intensities of the peaks increase correspondingly as the radiation power increases from 360 to 600 W. However, the peaks decrease in intensity when 720 W was used. These results indicate that BFO crystals can be synthesized under different radiation output powers.

The morphological and size changes in the as-prepared BFO samples also strongly depend on the radiation power. Figures 5(b)–(d) present typical SEM images of the obtained BFO samples synthesized under different radiation output powers. The morphological and size changes in the as-prepared BFO samples also strongly depend on the radiation power. All the resulting products consist predominantly of platelike nanostructures. At higher magnification, we can clearly see that the thickness of the nanoplates increases while increasing the radiation output power from 360 to 600 W, and decreases when further increasing the power to 720 W. This is an interesting phenomenon observed in our experiment that the mean crystal size and morphology of the BFO nanoplates can be tuned by adjusting the irradiation power when keeping the total energy constant. As listed in Table II, a statistic investigation on crystalline sizes through SEM also confirms that the mean grain size increases with power, to decrease above a threshold of 600 W at a constant energy. The results demonstrate that irradiation power is very important to attain the crystallinity of the final products during the rapid synthesis process of M–H treatment. Further systematic studies are underway to better understand the influence of irradiation power on the crystallization behavior of the final products during the M–H treatment process and the involved mechanism.

**Table II. Synthesis Parameters, Phases, and Mean Grain Sizes of Different Samples Prepared by M–H Method**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Concentration of NaOH (M)</th>
<th>Radiation power (W)</th>
<th>Time (s)</th>
<th>Phase</th>
<th>Mean size (nm)</th>
<th>Thickness of the nanoplates (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MH-1</td>
<td>0.07</td>
<td>360</td>
<td>150</td>
<td>BiFeO$_3$, Bi$_2$Fe$_4$O$_9$</td>
<td>~580</td>
<td>40–100</td>
</tr>
<tr>
<td>MH-2</td>
<td>0.07</td>
<td>600</td>
<td>90</td>
<td>BiFeO$_3$</td>
<td>~720</td>
<td>60–150</td>
</tr>
<tr>
<td>MH-3</td>
<td>0.07</td>
<td>720</td>
<td>45</td>
<td>Amorphous, Bi$<em>{23}$FeO$</em>{40}$</td>
<td>~420</td>
<td>20–40</td>
</tr>
<tr>
<td>MH-4</td>
<td>0.07</td>
<td>720</td>
<td>60</td>
<td>BiFeO$<em>3$, Bi$</em>{23}$FeO$_{40}$</td>
<td>~470</td>
<td>50–80</td>
</tr>
<tr>
<td>MH-5</td>
<td>0.07</td>
<td>720</td>
<td>75</td>
<td>BiFeO$_3$</td>
<td>~470</td>
<td>50–80</td>
</tr>
<tr>
<td>MH-6</td>
<td>0.03</td>
<td>720</td>
<td>65</td>
<td>BiFeO$<em>3$, Bi$</em>{23}$FeO$_{40}$</td>
<td>~480</td>
<td>40–80</td>
</tr>
<tr>
<td>MH-7</td>
<td>0.07</td>
<td>720</td>
<td>65</td>
<td>BiFeO$_3$</td>
<td>~850</td>
<td>60–160</td>
</tr>
<tr>
<td>MH-8</td>
<td>0.15</td>
<td>720</td>
<td>65</td>
<td>BiFeO$<em>3$, Bi$</em>{23}$FeO$_{40}$</td>
<td>~850</td>
<td>60–160</td>
</tr>
<tr>
<td>MH-9</td>
<td>0.50</td>
<td>720</td>
<td>65</td>
<td>BiFeO$<em>3$, Bi$</em>{23}$FeO$_{40}$</td>
<td>~850</td>
<td>60–160</td>
</tr>
</tbody>
</table>

(3) **Effect of Alkali Concentration**

The concentration of alkali of the precursor solution also plays a critical role on the formation of BFO nanoplates. The XRD patterns [Fig. 6(a)] indicate that the BFO crystal can be obtained under a range of NaOH concentrations. SEM imaging [Figs. 6(b)–(e)] shows that when the NaOH concentration is either too low (0.03 M) or too high (0.5 M), the BFO crystals are composed of irregular-shaped particles. Only the samples prepared at concentrations between 0.07 and 0.15 M are observed to self-organize into 2D nanostructures.
In the hydrothermal process for synthesizing oxide crystals, OH\(^{-}\) ions are found to be one of the dominating anions that affect the nucleation and growth behavior of oxide materials. It has been reported that the OH\(^{-}\) ions can serve as capping agents and adsorb on certain faces of oxide crystals, which may create growth anisotropy and direct the crystal growth as well.\(^{44-47}\) Some cluster facets have a preference to absorb OH\(^{-}\) due to the difference of surface energy associated with certain crystallite facets before crystal nuclei are assembled together, which strongly depends on reaction conditions such as the precursor, concentration of mineralizer or surfactant, temperature, and so on. As a result, the shielding effect of OH\(^{-}\) ions on the interface will control the growth rate of the OH\(^{-}\)-absorbed facets. A great number of 1D and 2D nanostructures have been fabricated applying such a control approach.\(^{20,44-50}\) Previous reports confirmed that uniform BFO nano/micromaterials with highly exposed crystal facets such as \{111\}_c exposed nanorods,\(^{15}\) and \{100\}_c enclosing nanocubes\(^{12,13}\) can be obtained by adding NaOH under different reaction environment. In our case, we consider that under the microwave treatment, OH\(^{-}\) may adsorb onto surfaces of the \{100\}_c crystal facets of nucleating BFO to regulate the lamellar growth and suppress the aggregation tendency of the growing plates consequently. To obtain BFO nanoplates with controllable crystallographic facets, nucleation kinetics should be carefully controlled by choosing the favorable alkali concentration of the reaction solution. Further studies are needed to better understand the adsorption behaviors of OH\(^{-}\) ions with certain BFO crystal facet under the M-H condition.

(4) Effect of Reaction Time
To develop a better understanding of the evolution process of the BFO nanoplates, we trapped intermediate states of the product by varying the reaction time at a fixed output power of 720 W. XRD patterns of the samples obtained at different M-H radiation times were recorded [Figs. 7(a)–(c)]. The sample MH-3 treated for 45 s does not show a crystallized BFO phase [Fig. 7(a)]. The 60 s irradiated sample (MH-4), on the other hand, gives peaks corresponding to perovskite BFO phase [Fig. 7(b)]. The peaks show a consistent yet remarkable rise in intensity compared to that of MH-4 upon increasing the radiation time up to 75 s [Fig. 7(c)].

The time-dependent morphological evolution process was followed by SEM at various stages of the M-H process, providing valuable information regarding the formation of BFO nanoplates. Figures 7(d)–(f) presents SEM images of samples prepared by radiating the solutions between 45 and 75 s. A large amount of small crystallites nucleated without any particular orientation when the reaction time was 45 s [Fig. 7(d)]. The sample prepared with 60 s of radiation [Fig. 7(e)], however, shows the presence of BFO nanoplates, and a similar morphology is retained up to 75 s [Fig. 7(f)] of radiation. The crystalline size of sample MH-5 slightly increases when the treatment time increased from 60 to 75 s (Table II), as investigated from the SEM images. This means that extended periods of microwave hydrothermal treatment greatly enhance the crystallinity, yet have little influence on the lateral crystalline size.

(5) Proposed Formation Mechanism
The theory of dissolution/recrystallization can be applied to interpret the present M-H reaction process. Bismuth oxide nitrate (BiONO\(_3\))\(^{15,51,52}\) and iron hydroxides (Fe(OH)\(_3\)) are readily formed as thermodynamically stable materials at an initial stage during the synthesis process. The involved reactions during this process are as follows:

\[
\text{Bi}^{3+} + \text{NO}_3^- + 2\text{NH}_3(\text{aq}) + \text{H}_2\text{O} \rightarrow \text{BiONO}_3 + 2\text{NH}_4^+ \quad (1)
\]

\[
\text{Fe}^{3+} + 3\text{NH}_3(\text{aq}) + 3\text{H}_2\text{O} \rightarrow \text{Fe(OH)}_3 + 3\text{NH}_4^+ \quad (2)
\]
after running the microwave treatment, these precursor compounds experience a dissolution process under the present reaction environment. Next, the precursor recrystallize to BFO nanocrystal gradually through a slow aggregation and crystallization of primary colloids, depending on the degree of supersaturation and the chemical potential of OH/CO₂, by the following reactions:

\[
\text{BiONO}_3 + \text{H}_2\text{O} \rightarrow \text{Bi}^{3+} + 2\text{OH}^- + \text{NO}_3^- \quad (3)
\]

\[
\text{Fe(OH)}_3 \rightarrow \text{FeO}_3^{2-} + 3\text{H}^+ \quad (4)
\]

\[
\text{Bi}^{3+} + \text{FeO}_3^{2-} \rightarrow \text{BiFeO}_3 \quad (5)
\]

On the basis of the above time-dependent transformation process, the formation of BFO nanoplates can be achieved by the following procedure [Fig. 7(g)]: (i) The precursor nucleated to form a large amount of tiny crystal nuclei in the initial reaction process; (ii) With prolonged time, the newly formed BFO crystal nuclei aggregated with the formerly formed ones, and began to grow into two-dimensionally thin platelike structures due to the adsorption of OH⁻ ions on (001) faces; and (iii) The primary BFO particles continued to adsorb onto these growing plates and then gradually ripened into single-crystal nanoplates. In addition, we observed that fine particles adhered to the large ones from SEM imaging [Figs. 7(e)], indicating that the growth of BFO nanoplates follows an Ostwald ripening mechanism.

(6) Magnetic Properties

The magnetization hysteresis loops of the as-synthesized BFO nanoplates (MH-1, MH-2, and MH-5) are shown in Fig. 8. From the enlarged figure inset of Fig. 8, we can clearly see that all the samples exhibit weak ferromagnetic behavior at RT. It is well-known that bulk BFO exhibits a linear magnetic response when a magnetic field is applied at RT because of the antiferromagnetic arrangement of Fe³⁺ magnetic moments. From the XPS analysis, we know that the oxidation state of elemental Fe in the BFO nanoplates is 3⁺ [Fig. 4(c)]. In addition, these BFO samples consist of 2D platelike morphologies with thickness ranging from 20 to
150 nm, which are comparable with the wavelength period of the spiral spin arrangement of 62 nm of bulk BFO. Therefore, we infer that the cycloid structure of bulk BFO is partially destroyed in the as-prepared BFO nanoplates, which leads to the weak ferromagnetic behavior observed at RT. Similar observations have been reported in BFO nanoparticles and one-dimensional nanowires. In addition, the saturation magnetization of the nanoplates decreases with increasing crystal size, which is also consistent with previous reports on BFO nanoparticles.

IV. Conclusions

In summary, we report a simple and inexpensive method to synthesize 2D BFO nanocrystals with exposed {100} facets by microwave hydrothermal (M-H) reaction without any surfactant. We found that plate-shape BFO single crystal can be obtained within a very short time of 60–150 s, much less than that of conventional and other M-H process reported before. Moreover, we demonstrated that the radiation power, reaction time, and concentration of alkali are important parameters for the crystallization process of BFO during M-H treatment. In addition, a weak ferromagnetism of the BFO nanoplates was observed at RT due to the nanoscale size effect, which is different from that of bulk BFO. The rapid reaction involved with this M-H process of obtaining BFO nanostructures has the potential to lower the manufacturing cost with significant time and energy savings. Further work is under way to investigate more chemical and physical properties of 2D BFO nanostructures.

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