Textured Z-type Hexaferrites $\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$ Ceramics with High Permeability by Reactive Templated Grain Growth Method

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Abstract:

The c-axis oriented $\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$ (Co_2Z) ceramic with high permeability was successfully prepared by reactive templated grain growth (RTGG) method using reactive template $\text{BaFe}_{12}\text{O}_{19}$ (BaM) and complementary $\text{Ba}_2\text{Co}_2\text{Fe}_{12}\text{O}_{22}$ (Co_2Y). The microstructure and magnetic property of the textured ceramic sample in different orientations were investigated. The sample presents both structural and magnetic anisotropic characteristics. The obtained (00h) textured Co_2Z ceramic exhibits a favorable combination of easy magnetization plane orientation, high initial permeability of 19.0 ± 1.2 (much higher than that from conventional ceramic method (CCM) ($\mu_i=12$)), and high cut-off frequency ($f_r > 1\text{GHz}$), indicating the potential for high frequency application in novel electronic devices.

Keywords: Z-type hexaferrite; Textured ceramics; Magnetic anisotropic; Reactive templated grain growth

1. Introduction

The rapid development of communication and electronic technology has given significant demand for improved performance and miniaturization of high frequency devices. Thus, the materials with improved properties, especially in high frequency range, exhibit a wide variety of practical applications and attract tremendous attentions. The Z-Type hexaferrite Co_2Z , whose easy magnetization direction lies in the crystallographic basal plane (a-b plane) of the hexagonal structure, possesses excellent magnetic properties with a high cut-off frequency in the gigahertz region.¹⁻³

It is considered to be a good candidate for high frequency applications, such as

antennae substrates, chip inductors, inductor-capacitor filters and microwave absorbers.^{4, 5} Also, it is reported that the c-axis oriented Co_2Z has excellent performance in tunable devices and wideband electromagnetic band gap (EBG) metamaterials.⁶⁻⁹ It is noteworthy that improved properties can be obtained if polycrystalline ceramics are textured and exhibit the anisotropic characteristics as that of single crystals.¹⁰ For non-magnetic particles, the textured ceramics are often prepared by templated grain growth (TGG) method and reactive templated grain growth (RTGG) method. A textured microstructure can be evolved by grain growth in the oriented anisotropic template.^{11, 12} For the magnetic particles, it is common that they can be textured by the magnetic orientation process. In this work, it is the first time to use the RTGG method to prepare textured Z-type hexaferrite Co_2Z ceramics. It is reported that Co_2Z can be synthesized at high temperature above 1200°C even using the molten salt method.^{13, 14} In this way, the Co_2Z powders prepared by molten salt method possess poor phase purity and inhomogeneous microstructure, which are not suitable to act as the seed particles for the TGG method. As we know, the Co_2Z can be obtained when the BaM reactive with complementary Co_2Y at high temperature.¹⁵ Also, it is well known that large average grain size favors high permeability of the ferrites¹⁶ and the high permeability will enable the fabrication of miniaturized antennas, filters and electromagnetic interference (EMI) devices, etc.¹⁷ Thus, if the large, anisotropic BaM templates can be obtained, the textured Z-type hexaferrite Co_2Z ceramics with higher permeability can be obtained by RTGG method using reactive templates BaM and complementary Co_2Y .

1 In this study, BaM templates were prepared and the RTGG method was firstly
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3 employed for the preparation of the textured Co₂Z ceramics successfully. Different
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5 amounts of prepared BaM templates (20wt%, 50wt%, 80wt%, 100wt% of the needed
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7 BaM amount) were utilized to act as oriented anisotropic templates. The
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9 microstructures and magnetic properties of the textured Co₂Z samples in different
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11 directions were investigated.
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16 2. Experimental

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18 A schematic of the approach employing RTGG method for the preparation of
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20 textured Co₂Z is shown in Figure 1. To synthesize BaM and Co₂Y powders, raw
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22 materials, analytical grade oxide powders (≥ 99 , Sinopharm Chemical Reagent,
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24 Shanghai, China) BaCO₃, Fe₂O₃ and CoO were weighed according to the
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26 stoichiometric proportion. Powders were mixed and calcined at 1100 °C in air for 2h.
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28 The SEM micrographs of the synthesized BaM and Co₂Y powders are shown in
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30 Figure 2(a) and 2(b), respectively. The BaM templates were synthesized by a two-step
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32 molten salt method (MSM). Firstly, the raw materials, analytical grade oxide powders
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34 (≥ 99 , Sinopharm Chemical Reagent, Shanghai, China) BaCO₃, Fe₂O₃ were mixed
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36 according to the stoichiometric ratio required by BaM. The mixed powders were dried
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38 and sintered at 1000 °C for 2h together with KCl at the weight ratio of 1:1. The molten
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40 salt was washed with hot deionized water. Plate shaped particles approximately 1 μ m
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42 in diameter were obtained, the microstructure of the particles was shown in Figure
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44 2(c). Secondly, to coarsen the plate shaped BaM powders obtained from the MSM, the
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46 obtained powders were mixed with BaCl₂ and Fe₂O₃. The BaCl₂: Fe₂O₃ ratio was 2:1
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by weight and the Fe_2O_3 : BaM ratio was 500:1 by mole.¹⁸ In our experiment, the amount of Fe_2O_3 and BaM is 0.05 mole and 0.0001 mole, respectively. The mixtures were heated at 1150 °C for 8h to synthesize plate shaped particles approximately 5-20 μm in diameter and 1 μm in thickness, the microstructure of the coarsened BaM particles was shown in Figure 2(d). To prepare the homogeneous slurry for the tape casting, the Co_2Y powders (together with the BaM powders) and different amounts of BaM templates (20wt%, 50wt%, 80wt%, 100wt% of the needed BaM amount) were mixed according to the stoichiometric ratio required by Co_2Z , together with the solvent (alcohol, 60wt% of the powders, and butanone, 90wt% of the powders), dispersant (glycerol trioleate, 2wt% of the powders), binder (Polyvinyl butyral (PVB), 5wt% of the powders) and plasticizer (dibutyl phthalate (DBP), 3wt% of the powders), the details of mixing process were shown in Figure 1. Then the tape casting was carried out by the doctor blade technique at a speed of 30 cm/min. The thickness of the obtained green tape was 15-20 μm . The green tape cutouts were stacked and laminated under 30 MPa at 70 °C followed by a cold isostatic pressing at 200 MPa to produce green compacts with thickness of 1.5-2 mm. The green compacts were heated to 600 °C for 4h to burn out the organics with the heating rate of 0.3 °C/min. Final sintering were carried out at 1100 °C-1275 °C for 4h with the heating rate of 3 °C/min. The fractured section of 1250 °C/4h sintered sample was polished and etched at 1150 °C for 30min for the observation of the interior microstructure.

Phase compositions of all the sintered samples were detected by X-ray diffraction (XRD) with $\text{CuK}\alpha$ radiation (D/MAX-2400 X-ray diffractometer, Rigaku,

Tokyo, Japan) in the range of $2\theta=10^{\circ}$ - 70° . The microstructures of the magnetic bulks were observed using scanning electron microscope (SEM) (Quanta250FEG, FEI, Hillsboro, USA), and in order to avoid the adverse influence on the field emission microscope, the microstructures of the magnetic powders were observed using SEM (JSM-6360, JEOL, Tokyo, Japan). The frequency dependence of the complex permeability of the torus-shape specimen was measured by the one-turn coil method using an impedance analyzer (4291B, Hewlett Packard, Palo Alto, CA) with a 16454L magnetic test fixture in the frequency range from 10 MHz to 1 GHz. The magnetic hysteresis loops of the samples were detected using a Physical Property Measurement System (PPMS) (Quantum design model 6000, San Diego, California, USA). The bulk density of the textured sample was determined by the Archimedes method. The preferred orientation of the textured sample was quantified using Lotgering factor:¹⁹

$$f = (P - P_0) / (1 - P_0), \text{ where } P = \sum I_{(00l)} / \sum I_{(hkl)} \text{ for oriented samples, } P_0 = \sum I_{0(00l)} / \sum I_{0(hkl)} \text{ for the standard random powders using the published diffraction file ICDD, \#19-0097.}$$

3. Results and discussions

Figure 3 shows the XRD spectra of the BaM powders and templates. It can be seen that the coarsened BaM particles exhibit obvious increase in (00h) peaks, in accordance with the anisotropic microstructure as shown in Figure 2(d). Such anisotropic BaM particles can be the desired templates for the RTGG process. Figure 4 presents the XRD spectra of textured Co₂Z ceramics with different amounts of BaM templates. Compared with the Co₂Z ceramic sample prepared by CCM, all the textured Co₂Z ceramic samples exhibit typical phase of the Z-type hexaferrites.

Moreover, the textured ceramics, especially the sample with 100wt% BaM templates, shows the radical increase in the intensity of (00h) peaks, indicating that the large, anisotropic BaM templates benefit the orientation of the Z-type hexaferrite during the process of RTGG method, the more templates introduced, the higher orientation obtained. For the textured Co₂Z prepared by the magnetic orientation process, the reported Lotgering factor varies from 0.3 to 0.5.⁶ For the sample with 100wt% BaM templates prepared by RTGG method, the X-ray diffraction analysis indicates the Lotgering factor of the textured sample is about 0.39 ± 0.05 . It can be seen that the method described in this work is an effective and reliable way to prepare textured Z-type hexaferrite.

The SEM micrograph of the surface of the sintered sample with 100wt% BaM templates is shown in Figure 5. It was well sintered and the bulk density of the textured sample is 4.728 ± 0.029 g/cm³, the relative density is $(88.37 \pm 0.55)\%$. It can be seen that the textured hexaferrite ceramic prepared by RTGG method possesses two types of surface microstructure. In order to understand the formation of the two types of microstructure during RTGG process at high temperature, the samples were sintered at different temperatures and the high magnified SEM micrographs were shown in Figure 6. During the reaction at high temperature, the BaM templates react with the closest Co₂Y powders and form the compact platelike microstructure, named A type as marked in the Figure 5. The high magnified SEM micrographs of the A type are shown in Figure 6(a) and 6(b). At the same time, another type surface microstructure with hexangular grains has also been observed, named B type as

1 marked in the Figure 5. The high magnified SEM micrographs of the B type are
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3 shown in Figure 6(c), 6(d), 6(e), 6(f), which represent the formation of the B type
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5 microstructure during the high temperature at 1200 °C, 1225 °C, 1250 °C, 1275 °C,
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8 repectively. It can be seen that such grains grow with the increasing of sintering
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10 temperature and form platelike microstructure eventually. Both the two types of grains
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12 align in the plane perpendicular to the c-axis, which can be defined as textured plane.
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17 Also, in order to understand the process of texturing in the interior of the sample
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19 at high temperature, the SEM micrographs of the cross section of the sintered samples
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21 are shown in Figure 7. The final sintering was carried out at 1100 °C, 1150 °C, 1200 °C,
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23 1250 °C for 4h, respectively. It can be seen from Figure 7(a) that no reaction occurs
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25 between the BaM templates and Co₂Y matrix at 1100 °C, the BaM templates are well
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27 aligned in the Co₂Y matrix by the tape casting process. Inevitably, while most of the
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29 grains are aligned in the tape casting direction, some remain unaligned. When the
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31 samples are sintered at 1150 °C, the temperature is also not high enough to promote
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33 the reaction between the BaM templates and Co₂Y matrix, meanwhile, relatively thick
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35 BaM templates are observed at this temperature, as shown in Figure 7(b). The BaM
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37 templates begin to react with the closest Co₂Y powders at 1200 °C, it can be seen from
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39 Figure 7(c) that BaM templates begin to grow at the expense of small Co₂Y matrix
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41 grains. When the samples are sintered at 1250 °C, Co₂Y matrix grains are completely
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43 consumed by the extensive network of the anistropic BaM grains and finally form the
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45 well oriented grains, as shown in Figure 7(d).
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textured Co_2Z ceramic with 100wt% BaM templates. It is reported that the permeability will be affected by the demagnetizing field arising from the specimen's geometry, the permeability of the slab is often smaller than that of the torus.^{20, 21} So, in this work, the permeability was measured using torus-shape specimen, thus the values are not affected by the demagnetizing field. The permeability of textured Co_2Z was affected by not only the demagnetizing field, but also the thickness of the textured samples, the permeability of the textured sample increases with the decreasing thicknesses of the measurement sample.²¹ For textured Co_2Z ceramics prepared by the magnetic orientation process with different geometries and thicknesses, the reported permeability was as low as 12 and as high as 31.^{8, 20, 21} In our experiment, the thickness of all the torus-shape samples is about 1.34mm. It can be seen from Figure 8(a) that the RTGG samples possesses higher permeability ($\mu_i > 17$) than that of the CCM sample ($\mu_i = 12$). On one hand, the magnetic path of the measured specimen lies in the textured plane of Z-type hexagonal ferrites, which is the preferred plane of magnetization.²⁰ On the other hand, the large, anisotropic BaM templates, which are dispersed in the compact complementary powders, give rise to the larger average grain size of RTGG sample during the sintering process, thus the larger permeability can be obtained. Moreover, for the planar hexaferrite, the enhancement of permeability will be greatly affected by the Lotgering factors. During the RTGG process in our experiment, while most of the grains are well aligned in the tape casting direction, some remain unaligned inevitably as shown in Figure 7(a), thus different Lotgering factors f will be obtained from different RTGG samples ($f = 0.39 \pm$

0.05). So, as shown in Figure 8(a), the permeability of the different RTGG samples varies from 17.5 to 20.4 (@50MHz) with the high cut-off frequency $f_r > 1\text{GHz}$. The hysteresis loops of the textured Co_2Z sample in different directions are shown in Figure 8(b). It is known that for the Z-type hexaferrite, the easy magnetization direction lies in the crystallographic basal plane (a-b plane) of the hexagonal structure. It can be seen from the Figure 8(b) that the prepared samples measured with the external magnetic field H in and out of textured plane exhibit obviously different characteristic in hysteresis loops. When the external magnetic field H applies in parallel to the textured plane, it requires lower H value to saturate the sample, indicating that the textured plane is “made” much easier to be magnetized due to the preferred orientation of grains during the RTGG method.

4. Conclusion

In this work, a new process based on RTGG method is presented for the preparation of textured Z-type hexaferrites Co_2Z ceramic with high cut-off frequency. The prepared samples exhibit both structural and magnetic anisotropy, indicating that the RTGG method can be an effective and reliable method to prepare textured hexaferrites. A textured microstructure of Co_2Z was evolved by grain growth in the oriented anisotropic BaM template, the preferred orientation of grains can be obtained during the RTGG process, thus, the textured plane is “made” much easier to be magnetized, high permeability about 19.0 ± 1.2 can be obtained in the prepared (00h) textured Co_2Z samples with high cut-off frequency $f_r > 1\text{GHz}$. Moreover, the method described in this work points out a novel approach for preparing the textured ferrites

with both oriented grains and enhanced permeability. The results could benefit both the miniaturization and high frequency application of the electronic devices.

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Figure captions:

Figure 1. A schematic of the RTGG method for preparing textured Co_2Z ceramics.

Figure 2. The SEM micrographs of (a) BaM powders (CCM). (b) Co_2Y powders (CCM). (c) BaM powders (MSM). (d) BaM powders (coarsened).

Figure 3. The XRD spectra of the BaM powders and templates.

Figure 4. The XRD spectra of the textured Co_2Z ceramics with different amounts of BaM templates. The (00h) peaks are marked with *.

Figure 5. The SEM micrograph (surface) of the textured Co_2Z ceramic with 100wt% BaM templates sintered at $1275^\circ\text{C}/4\text{h}$.

Figure 6. High magnified SEM micrographs of A type microstructure. The sample was sintered at (a) $1200^\circ\text{C}/4\text{h}$, (b) $1275^\circ\text{C}/4\text{h}$. High magnified SEM micrographs of B type microstructure. The sample was sintered at (c) $1200^\circ\text{C}/4\text{h}$, (d) $1225^\circ\text{C}/4\text{h}$, (e) $1250^\circ\text{C}/4\text{h}$, (f) $1275^\circ\text{C}/4\text{h}$.

Figure 7. The SEM micrographs of the cross section of the samples with 100wt% BaM templates sintered at (a) $1100^\circ\text{C}/4\text{h}$, (b) $1150^\circ\text{C}/4\text{h}$, (c) $1200^\circ\text{C}/4\text{h}$, (d) $1250^\circ\text{C}/4\text{h}$. The $1250^\circ\text{C}/4\text{h}$ sintered sample was polished and etched at 1150°C for 30min.

Figure 8. (a) The frequency dependency of the complex permeability of CCM ceramic and different textured Co_2Z ceramics with 100wt% BaM templates. (b) The hysteresis loops in different directions of the textured Co_2Z ceramic with 100% BaM templates.

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