Microwave Treatment of Hexagonal Barium Ferrite Prepared by Sol Gel Technique and its Application in Radar Wave Absorption

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Abstract: Application of microwave radiation for the treatment of two different series of prepared hexagonal barium ferrite was done. The treated hexagonal barium ferrite was prepared by sol gel autocombusion technique. The first series was calcinated at 850° C for 4 hours while the other was calcinated at 950° C for 5 hours. Both series were irradiated at different microwave power levels in a domestic microwave oven. The power was varied from 200 to 800 W. The prepared samples were characterized by XRD and TEM. The reflections loss of the prepared samples in the X and Ku band frequency range (8-18 GHz) were measured using the reflectometer (PR-17).

Keywords: Hexagonal barium ferrite, anti-radar, radar wave absorption.

1. Introduction

The reduction of the radar cross-section (RCS) of military platforms has been a major challenge since the second-world war. Microwave absorbers have been widely used to prevent or minimize electromagnetic reflections from large structures such as aircraft, ships and tanks and also to cover the walls of anechoic chambers. It has been found that ferrimagnetic metallic oxides are especially suitable for use as RAM, in the form of cubic spinels, garnets or hexagonal magnetoplumbite structured powders in polymeric matrices [1].

Hexaferrites are classified into five types depending on the chemical formula and the crystal structure. These include M-type ($BaFe_{12}O_{19}$), W-type ($BaMe_2Fe_{16}O_{27}$), X-type ($Ba_2Me_2Fe_{28}O_{46}$), Y-type ($Ba_2Me_2Fe_{12}O_{22}$) and Z-type ($Ba_3Me_2Fe_{24}O_{41}$), where the Me is a transition cation or a combination of cations as in spinel ferrites [2].

M type barium ferrite with a hexagonal molecular structure (BaO.6Fe₂O₃) is a well-known permanent magnet. It is widely used in magnetic recording media and in microwave devices [3]. It can be used as a good microwave absorber in the frequency range of 1 to more than 20 GHz [4]. Barium ferrite is characterized by a relatively high Curie temperature ($450\pm$ C), high intrinsic coercivity (6700 Oe), chemical stability and corrosion resistance [5].

Synthesis of barium ferrite has been reported by using the sol-gel auto combustion technique with barium nitrate and ferric nitrate as the sources for cations and citric acid as fuel [6, 7].

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Rahul Sharma showed that increasing microwave power irradiation on barium ferrite leads to systematic growth of nano crystals that attribute to sharp planes of single crystal. Spherical nano crystals formed a system initially that slowly grows into larger one, at the expense of the smaller crystals. The nano crystals act as "nutrients" for the bigger crystals combined with uniform morphological transformation from spherical to pyramidal-faced nano crystals. Nano crystals show a significant improvement in bandwidth during irradiation in [8].

The microwave absorption improvement of the prepared barium ferrite was studied in this work using the microwave irradiation at different powers.

2. Experimental Procedure

Two samples of hexagonal barium ferrite were prepared using citrate sol gel autocombusion technique (cations to citric acid molar ratio equal 1:2) with two different calcination treatments. The first sample was calcinated at 850 for 4 hours and the second was calcinated at 950 for 5 hours [7].

Each of the two prepared barium ferrite powders is mixed and manually cruched with solid sodium chloride with weight ratio of (1:2) before the irradiation process because the NaCl layer was functioning as flux between microwave radiation and nano particles that contributed to a fast and controlled growth of nano particles [8]. The microwave treatments were done to the same weights of the two prepared samples using different microwave powers (from zero up to 800 W) and for constant time for all samples.

The barium ferrite microwave treating influence on structure and size was examined using TEM and XRD. The microwave absorption in the X and Ku band was studied by mixing the prepared powders with polyurethane polymer with 10% weight percent ratio. The produced coating was casted on iron sheet using iron die with internal dimensions of (18 cm * 18 cm) and 1.5 mm thickness, followed by drying at 50°C for 3 h and completing the drying at normal temperature for 24 h, giving iron sheet coated with barium ferrite composite with thickness of 1.5 mm. The reflection losses of all prepared barium ferrite composites were measured using PR-17 reflectometer and the average loss was calculated for each sample in the X and Ku band.

3. Results and discussion

The barium ferrite calcinated at 950°C for 5 hr shows crystalline phase content and magnetic properties better than barium ferrite calcinated at 850°C for 4 hr (contains little amount of other amorphous phases) [7].

3.1 Microwave treatment of barium ferrite calcinated at 850°C for 4 hr

The X ray diffraction analysis (Fig. 1) shows that untreated barium ferrite contains approximately 94% hexagonal barium ferrite with some other contents like spinel barium ferrite (BaFe₂O₄), BaO and Fe₂O₃. All microwave treated samples give the similar XRD, which shows the formation of single hexagonal phase with very little content (1-2%) of BaO as impurities, so that just treatment with power of 386 W for 30 min is sufficient to form single hexagonal phase.



Fig. 1 The X ray diffractions of barium ferrite treated with different microwave powers (a) as prepared (b) 386 W (c) 552 W (d) 717 W (e) 800 W

Figure 2 shows the TEM study of the microwave treating of barium ferrite; untreated barium ferrite contains some amorphous phase which disappeared in the treated samples. The particle size diameter increases with the increase of the treating power (from about 10-25 nm in the untreated sample to about 60-80 nm in the sample treated with 800 W for 30 min), no transformation to the pyramidal shape was detected.



Fig.2.The TEM study and diffraction patterns of barium ferrite treated with different microwave powers (a) as prepared (b) 386 W (c) 552 W (d) 717 W (e) 800 W

Figure 3 shows that average reflection loss in X and Ku band was enhanced by treating barium ferrite with power of 386 W and this could be due to the complete transformation of the other phases to the hexagonal barium ferrite phase. Further treatment leads to a decrease in the average reflection losses until power of 717 W and this could be due to the increase in the particle size diameter. Average loss is enhanced again when treating barium ferrite with power of 800 W and this could be explained by supposing that the particle size diameter reaches a critical diameter which provides the best magnetic properties required for the absorption in 8-18 GHz.

The average losses of the barium ferrite treated at power of 800 W (60-80 nm) are 27.65 % in X band and 42.95 % in Ku band, it is near to the average losses of the barium ferrite calcinated at 950°C for 5 h (40-70 nm.) which are 37.15 % in X band and 43 % in Ku band.



Fig.3 Effect of microwave treatment at different microwave powers on the reflection loss of barium ferrite calcinated at 850°C for 4 hr

3.2 Microwave Treatment of Barium Ferrite Calcinated at 950°C for 5 hr

Figure 4 shows The TEM study of the microwave treating of barium ferrite, untreated barium ferrite highly contain crystalline phase. The particle size diameter increases with the increase of the treating power (from about 40-70 nm in the untreated sample to about 150-200 nm in the sample treated with 717 W for 30 min). Little particles found after treating with power more than 552 W could have a pyramidal shape.

Figure 5 shows that average reflection loss in X and Ku band decreased with the treating of barium ferrite until power of 386 W and this could be due to the increase in the particle size diameter which reached 90-130 nm. Further treating leads to approximately constant average reflection losses. This could be explained by considering that the particles size diameters exceed a critical diameter and then magnetic properties change does not significantly affect the reflection loss.



Fig.4.The TEM study and diffraction patterns of barium ferrite treated with different microwave powers (a) as prepared (b) 220 W (c) 386 W (d) 552 W (e) 717 W



Fig.5. Effect of microwave treatment at different microwave powers on the reflection loss of barium ferrite calcinated at 950°C for 5 hr

4. Conclusion

The microwave treatment of the barium ferrite shows the importance of the particle size diameter and its effect on the reflection loss. There is a critical diameter (40-70 nm) after which the change obtained in magnetic properties decreases the reflection loss to a minimum value, then it does not significantly affect the reflection loss. Also, there is no great effect on changing particles shape to the pyramidal shape; it would require power greater than 800 W for 30 min.

The microwave treatment using power of 800 W for 30 min to barium ferrite calcinated at 850° C for 4 h gives barium ferrite with single hexagonal phase, particles size diameter, particles shape and reflection loss (magnetic properties) approximately near to barium ferrite calcinated at 950° C for 5 h.

5. References

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