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Performance Improvement of S-Band Phase Shifter Using Al, Mn and Gd Doped $Y_3Fe_5O_{12}$ and Sintering Optimization

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The searching for $Y_3Fe_5O_{12}$ Yttrium Iron Garnet (YIG) ferrite base material is a difficult challenge for the microwave phase shifter. Although YIG ferrite is promising for its good microwave characteristics, the adjustment of its corresponding magnetic properties for applied frequency is always a tough challenge. In this work, a study was made to evaluate the impacts of stoichiometry and sintering temperature on hysteresis properties of Mn, Al and Gd substituted YIG ferrites. The Mn, Al substituted YIG ferrite, $(Y_3)(Mn_xAl_{0.83-x}Fe_{4.17})O_{12}$ showed its best properties for application in the 2~4GHz frequency range (i.e. S-band) latching phase shifter at x=0.13. Magnetic properties were further improved by sintering optimization. The increasing sintering temperature increases saturation of magnetization ($4\pi M_s$), remanence (B_r) and Squareness Ratio (SQR), while decreasing the coercivity (H_c). To further investigate the impact of the sintering process, Gd substituted YIG ferrites are also discussed. $(Y_{3-y}Gd_y)(Mn_{0.13}Al_{0.7-0.5y}Fe_{4.17})O_{12}$ with y=0.6 demonstrating its best magnetic properties at 1450°C sintering. Although the $4\pi M_s$, B_r and SQR are comparable to that of previous results, its H_c cannot meet the requirement. $(Y_3)(Mn_xAl_{0.83-x}Fe_{4.17})O_{12}$ ferrites with x=0.13 sintered at 1430°C shows the optimum magnetic properties: $4\pi M_s$ =675G, B_r=575G, H_c=0.45Oe, SQR=0.86. Good Ferromagnetic Resonance (FMR) linewidth (i.e. ΔH) were also realized as 38.6Oe at 3.2GHz. Base on the above results, we reveal Mn, Al substituted YIG ferrite is a good candidate for practical latch phase shifter applications at the S-band frequency range.

Keyword: Sinter, Yttrium Iron Garnet (YIG) ferrites, Phase shifter, S-band, Squareness Ratio (SQR)

1. INTRODUCTION

Yttrium Iron Garnet (YIG) ferrites are presently used in microwave communication devices such as circulator and latching phase shifters due to its good thermal stability, narrow ferromagnetic resonance linewidth and low dielectric loss(1). In order to properly control the microwave properties for the applied devices, the characteristics of the hysteresis loop are very critical(2). Mn, Al substituted YIG possess narrow ΔH with low H_c at relatively low $4\pi M_s$, which could be employed to the latching phase shifter at the S-band frequency range(3-4).

Ferrites are often used in microwave phase shifters for scanning of radar beams(5). However, the need for a phase shifter which could be switched in a few microseconds has caused the recent emphasis on the ferrite. Fast switching characteristic for a phase shifter could not be obtained without low H_c for the hysteresis properties of ferrite(6). In addition, a square hysteresis loop for ferrite is arranged in a closed magnetic path. The two remanent states of magnetization corresponding to the two stable states are critical to a phase shifter. The higher squareness ratio, Squareness Ratio (SQR) (i.e. $B_r/4\pi M_s$), makes for better switching of a phase shifter(6). The bi-static characteristics of a phase shifter make it an attractive component applied in phase-array radar system(6). For our work, the hysteresis characteristics of ferrite, such as $4\pi M_s$ about 700 Gauss, H_c about 0.6Oe, SQR about 0.85 are required for S-band phase shifter applications(1-2, 5-6).

In previous reports, few methods have been proposed for adjusting the ferrite properties to achieve the system requirements. Recently, Gd substituted YIG structure has been demonstrated to tune the hysteresis properties of ferrite; however, the Gd substituted ferrites cost appropriately twice the price of Mn, Al substituted YIG and is thus more uneconomical to use(3). We are the first to demonstrate that the substitution both of Mn and Al irons into YIG structure, which could effectively tune the magnetic characteristics. By the substitutions of elements, the interaction between and within sub-lattices of YIG structure would lead to modification of property(7-8). In this respect, it is desirable to reduce $4\pi M_s$ to achieve the requirement of a S-band phase shifter by Al substitution while maintaining high SQR. However, an effort to obtain the required $4\pi M_s$, results in sacrifice of the SQR(9). We deal with the property modification of high SQR through Mn substitution, i.e. by increasing Mn^{x+} content(3). Replacing part of the proportion of Al by Mn was introduced in order to keep the value of B_r while achieving required ($4\pi M_s$) at the same time. In order to further improve magnetic properties, different sintering temperatures were conducted(10). The higher sintering temperature of the ferrite can lead to a change in grain size, thus affecting $4\pi M_s$, H_c and SQR, etc. Using the principles and approach mentioned above, SQR could be maintained while H_c decreases to meet a device's target.

Furthermore, Gd is usually used to substitute the Y iron in YIG structure to adjust magnetic properties(3). In order to confirm the magnetic properties of Gd substituted YIG ferrite, Mn, Al and Gd substituted YIG ferrites were also demonstrated to evaluate its performance at different sintering temperatures. Mn, Al substituted YIG ferrite had better magnetic properties than that of Mn, Al and Gd substituted YIG ferrites were also clarified by the results in our work. From the above discussion, the desired property modification of $(Y_3)(Mn_xAl_{0.83-x}Fe_{4.17})O_{12}$ garnet ferrites which are of interest in order to obtain a suitable ferrite for the S-band latching phase shifter application were successfully realized in this paper.

2. EXPERIMENTAL METHOD

The raw materials Fe_2O_3, Y_2O_3, Mn_3O_4, and Al_2O_3 were prepared. The raw materials should be weighed with an accuracy of 0.0001g according to the chosen stoichiometry of $(Y_3)(Mn_xAl_{0.83-x}Fe_{4.17})O_{12}$ with x=0.09 (sample A), 0.13 (sample B), 0.23 (sample C), and then mixed in a ball mill for 3hrs with Alcohol and ZrO_2 balls. The slurry was dried and then calcined at 1200°C for 2hrs in an air atmosphere furnace with a heating rate of 5°C/min. The calcined powder was re-milled by vibration mill for 2hrs. After being milled, the powder was pressed into cylindrical molds. Cold isostatic pressing technology was used to improve uniformity and density of the green bodies. Then, the green bodies were sintering at 1350~1450°C for 6hrs in an air atmosphere furnace with a heating rate of 5°C/min and then furnace cooled to room temperature. Finally, the sintered bodies were mechanically processed into rectangular toroid, rod, and pellet with specific size for measuring the magnetic characteristics of ferrites according to IEC60556. The specific size samples will be polished before testing. For further discussion of the impact to sintering temperature of Gd substituted YIG ferrite, $(Y_{3-y}Gd_y)(Mn_{0.13}Al_{0.7-0.5y}Fe_{4.17})O_{12}$ with y=0.4 and 0.6 were also prepared for the above processes.

Hysteresis loop properties ($4\pi M_s$, B_r, H_c, SQR) of the toroids with 10 turns wound were measured by means of Yokogawa SK-130 *B-H* tracer analyzer. The relative dielectric constant and dielectric loss were detected by the modified Hakki and Coleman's approach(11-12) in the TE_{011} mode using network analyzer (Agilent E5071B) at the frequency range between 2GHz and 4GHz. The microstructure of sintered specimens were investigated by a Scanning Electron Microscopy (SEM). The crystalline structures of ferrites were confirmed by X-Ray powder Diffraction (XRD) technique.

3. RESULTS AND DISCUSSION

3.1 Effect of substitution of Mn and Al to magnetic properties

Figure 1 shows the XRD patterns of the samples with different Mn and Al substitutions. From the identified patterns, the well formation of YIG phase without extra phases was confirmed for all the samples. As the ionic radius of Mn^{x+} (0.066~0.067*nm*) and Al^{3+} (0.0675*nm*) were very close to that of Fe^{3+} (0.064*nm*), thus, the incorporation of Mn^{x+} and Al^{3+} could not result in obvious variation in the lattice parameter, which results

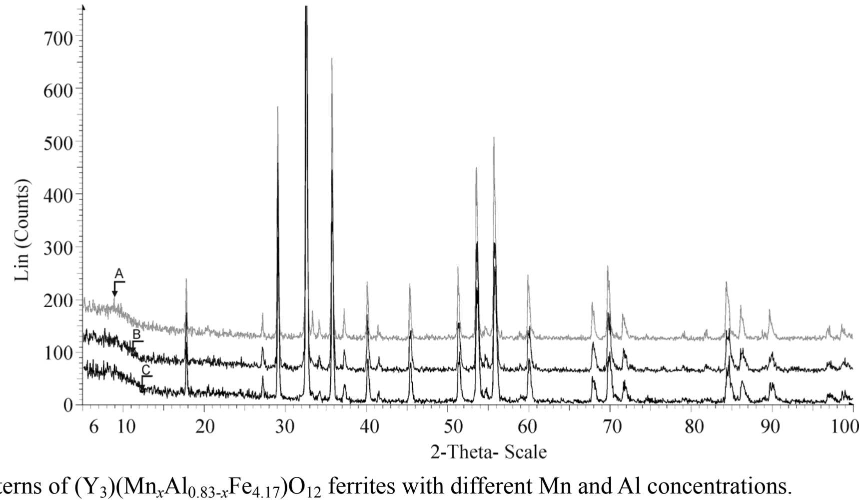


Fig.1. XRD patterns of $(Y_3)(Mn_xAl_{0.83-x}Fe_{4.17})O_{12}$ ferrites with different Mn and Al concentrations.

in the formation of the second phase like $YFeO_3$. In addition, sample A, B, C all obtained a high sintering density. The bulk densities were measured by Archimedes method at around 5.07~5.08g/cm^3 for this work, which suggests that all samples were well densified and the substitution of Mn, Al has no disadvantageous impact to the densification of YIG ferrite.

For phase shifter design, an optimum ferrite can be selected for a frequency with the relationship(13) in Eq. as follow:

$$\gamma 4\pi M_s / \varpi \approx 0.6$$

Where γ = 2.8MHz/Oe is the gyromagnetic ratio of the ferrite, and ω is the operation frequency. For 3.2GHz application, a low saturation magnetization ($4\pi M_s$) less than 685Gauss with a low coercivity (H_c) was desired. Figure 2-3 show the variation in saturation of magnetization ($4\pi M_s$) and coercivity (H_c) with Mn and Al concentrations for the (Y_3) ($Mn_xAl_{0.83-x}Fe_{4.17}$)O_{12} ferrites prepared in our work. As can be seen in these figures, with Mn% increasing and Al% decreasing, the $4\pi M_s$ increases. As to coercivity, it exhibits a sharp drop once Mn% increases and Al% decreases. In the best conditions for device purpose, when x=0.13 (i.e. sample B), the results of $4\pi M_s$=654G and H_c=0.54Oe could be satisfied with a commercial latch S-band phase shifter. For phase shifter, the SQR has very important influence on the signal switching. It is well known that the Mn substitution of YIG is very effective to increase the remanence (B_r) due to its smaller magnetostriction constant (λ_{111}) (14). The smaller the λ_{111} is, the higher B_r will be; thus, the SQR could be risen with B_r. The SQR results with different Mn, Al concentrations are presented in Fig.4. It implies that with Mn% increasing and Al% decreasing, the SQR increases. However, the SQR decreases sharply when x rises to 0.23 (Sample C) due to the increasing rate of $4\pi M_s$ is not able to follow that of B_r. In short, the substitution of Mn and Al could adjust the magnetic properties to commercial S-band phase shifter demands effectively, and the proportion with x=0.13, i.e. (Y_3) ($Mn_{0.13}Al_{0.7}Fe_{4.17}$)$O_{12}$ ferrites, shows the optimum properties: $4\pi M_s$=654G, B_r=557G, H_c=0.54Oe, SQR=0.85.

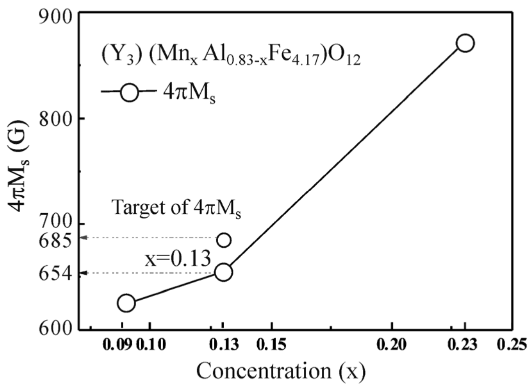


Fig.2. Magnetic properties $4\pi M_s$ of (Y_3)($Mn_xAl_{0.83-x}Fe_{4.17}$)O_{12} ferrites with different Mn, Al concentrations.

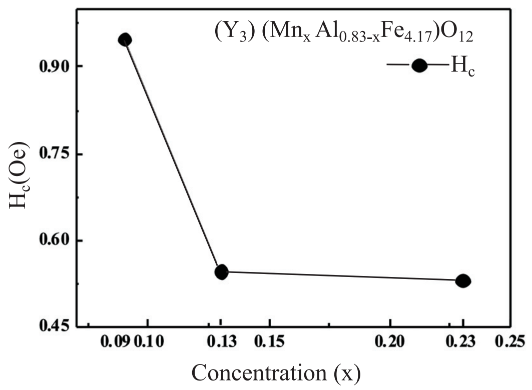


Fig.3. Magnetic properties H_c of (Y_3)($Mn_xAl_{0.83-x}Fe_{4.17}$)O_{12} ferrites with different Mn, Al concentrations.

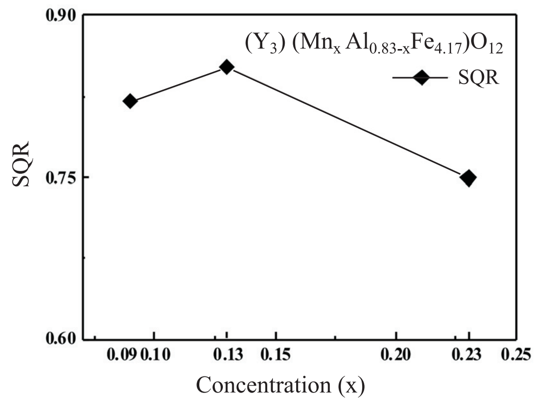


Fig.4. Magnetic properties SQR of (Y_3)($Mn_xAl_{0.83-x}Fe_{4.17}$)O_{12} ferrites with different Mn, Al concentrations.

3.2 Impact of sintering temperature

In order to further improve the magnetic properties of (Y_3) ($Mn_xAl_{0.83-x}Fe_{4.17}$)O_{12} ferrites. The sample with x=0.13 were prepared and sintered at 1350°C, 1400°C, 1430°C, 1450°C, respectively. Figure 5 shows the SEM micrograph of the polished surface of the above samples after hot corrosion. As can be observed in this figure, the average grain size for higher sintering temperatures is larger than that for lower sintering temperatures. This phenomenon is beneficial for the increase of $4\pi M_s$, B_r and decrease of H_c, which lead to achieve a relative high SQR at the same time. As shown in Fig.6, $4\pi M_s$ and B_r increases with the increasing sintering

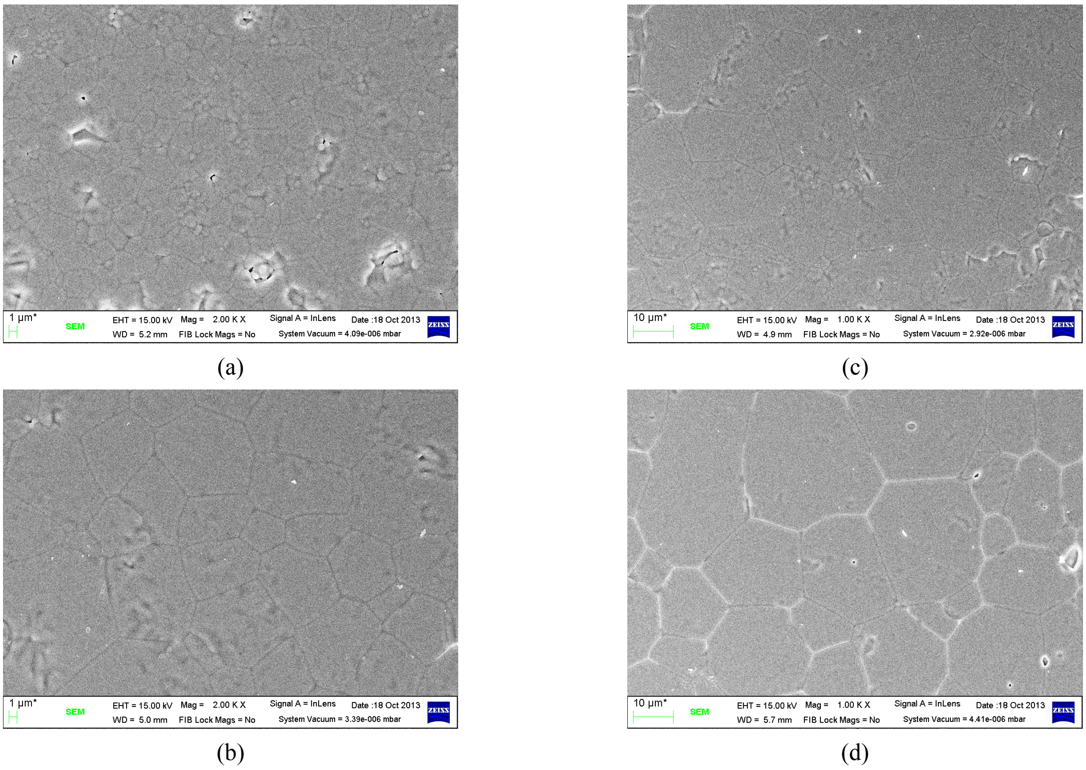


Fig.5. SEM micrograph (×2K) of $(Y_3)(Mn_{0.13}Al_{0.7}Fe_{4.17})O_{12}$ ferrites sintered at (a)1350°C; (b) 1400°C; (c) 1430°C; and (d) 1450°C (×1K).

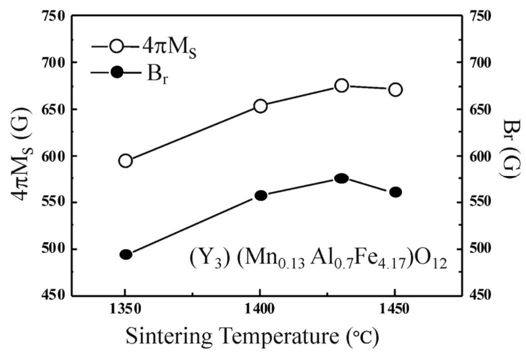


Fig.6. Magnetic properties $4\pi M_s$ of $(Y_3)(Mn_{0.13}Al_{0.7}Fe_{4.17})O_{12}$ ferrites versus sintering temperature.

temperature of up to 1430°C, and then drops while temperature rose to 1450°C. From Fig.7, the SQR was found to be also upgraded since the rates of increase of $4\pi M_s$ and B_r were different. H_c was also improved as shown in Fig.8. Although an excessive decrease in H_c at 1450°C was advantageous for the phase shifter; however, SQR at 1450°C is detrimental to the device purpose. From the above results, the sintering temperature impact to the ferrites sintered at 1430°C is noticeably larger. Figure 9 shows the hysteresis loops of the (Y_3) $(Mn_{0.13}Al_{0.7}Fe_{4.17})O_{12}$ ferrites at different sintering temperatures. The changes of the above, due to the adjustment of the sintering temperature, yield an improvement in the magnetic properties of ferrites for the applications of the S-band phase shifter. In general, the sample with x=0.13, i.e. (Y_3) $(Mn_{0.13}Al_{0.7}Fe_{4.17})O_{12}$, sintered at 1430°C shows the optimum magnetic properties: $4\pi M_s$ =675G, B_r=575G, H_c=0.45Oe, SQR=0.86 in our work. Besides, ε_r = 14.77 and $\tan\delta_e$ = 6×10^{-4} were also obtained at 3.2GHz.

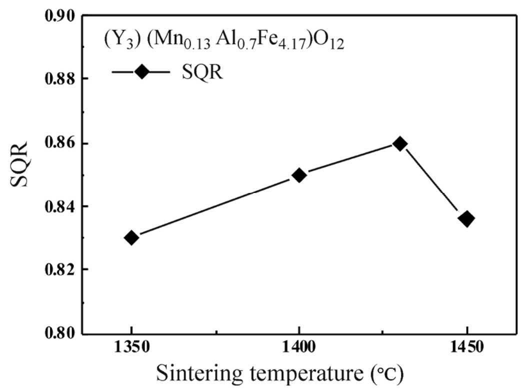


Fig.7. Magnetic properties SQR of $(Y_3)(Mn_{0.13}Al_{0.7}Fe_{4.17})O_{12}$ ferrites versus sintering temperature.

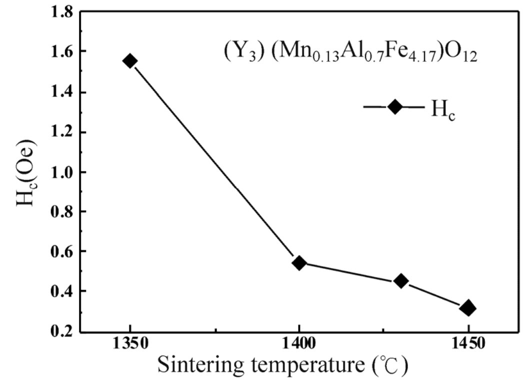


Fig.8. Magnetic properties H_c of $(Y_3)(Mn_{0.13}Al_{0.7}Fe_{4.17})O_{12}$ ferrites versus sintering temperature.

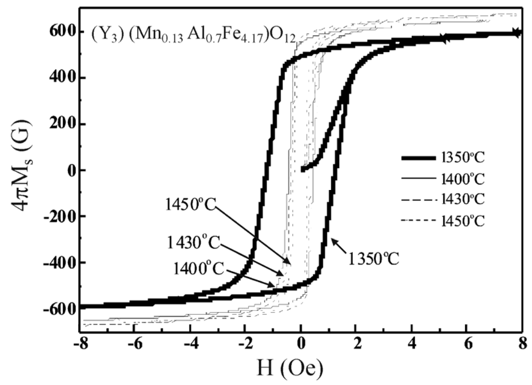


Fig.9. The hysteresis loop of $(Y_3)(Mn_{0.13}Al_{0.7}Fe_{4.17})O_{12}$ ferrites sintered at 1350°C, 1400°C, 1430°C and 1450°C.

3.3 Effect of substitution of Gd to magnetic properties

Due to the sensitivity of Gd substitution to $4\pi M_s$, B_r and H_c were smaller than that of Al substitution(3), we used Gd substitution to replace part of Al in $(Y_3)(Mn_{0.13}Al_{0.7}Fe_{4.17})O_{12}$ ferrite. Figure 10 shows the hysteresis loops of $(Y_{3-y}Gd_y)$ $(Mn_{0.13}Al_{0.7-0.5y}Fe_{4.17})O_{12}$ with y=0.4 (sample D) and 0.6 (sample E) sintered at 1400°C. As can be seen in the figure, Gd% increases to substitute for Y with part of Al% decreases. When y=0.6 (i.e. sample E), the results of $4\pi M_s$, H_c and SQR were better than that of y=0.4; however, its magnetic properties were still not satisfied with a commercial latch S-band phase shifter. To further promote the magnetic properties of $(Y_{2.4}Gd_{0.6})$ $(Mn_{0.13}Al_{0.4}Fe_{4.17})O_{12}$ ferrite, sample E was sintered at different temperatures of 1450°C and 1480°C, respectively. Figure 11 illustrates that the magnetic results of Sample E sintered at 1450°C was the closest to the requirement of a commercial latch S-band phase shifter. However, these results were still poorer than those of (Y_3) $(Mn_{0.13}Al_{0.7}Fe_{4.17})O_{12}$ ferrites sintered at 1430°C. The SEM micrograph results as shown in Fig.12 explain larger grain sizes were observed on the samples sintered at higher temperature. About the phenomenon of different sintering temperature could be also correlated with aforesaid SEM results as shown in Fig.5. Over the large grain size of sample E sintered at 1480°C cause its $4\pi M_s$ to be too high to meet with the requirement of a commercial latch S-band phase shifter.

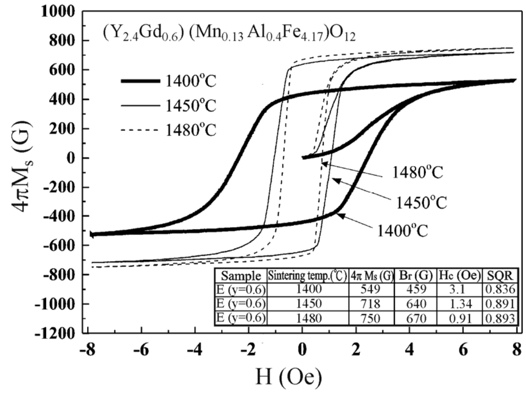


Fig.10. Hysteresis loop of $(Y_{3-y}Gd_y)$ $(Mn_{0.13}Al_{0.7-0.5y}Fe_{4.17})O_{12}$ with y=0.4 and 0.6 sintered at 1400°C.

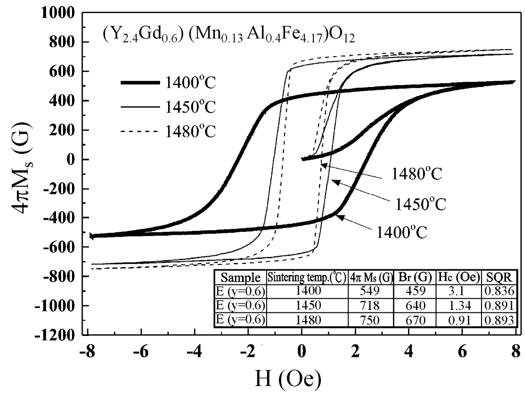
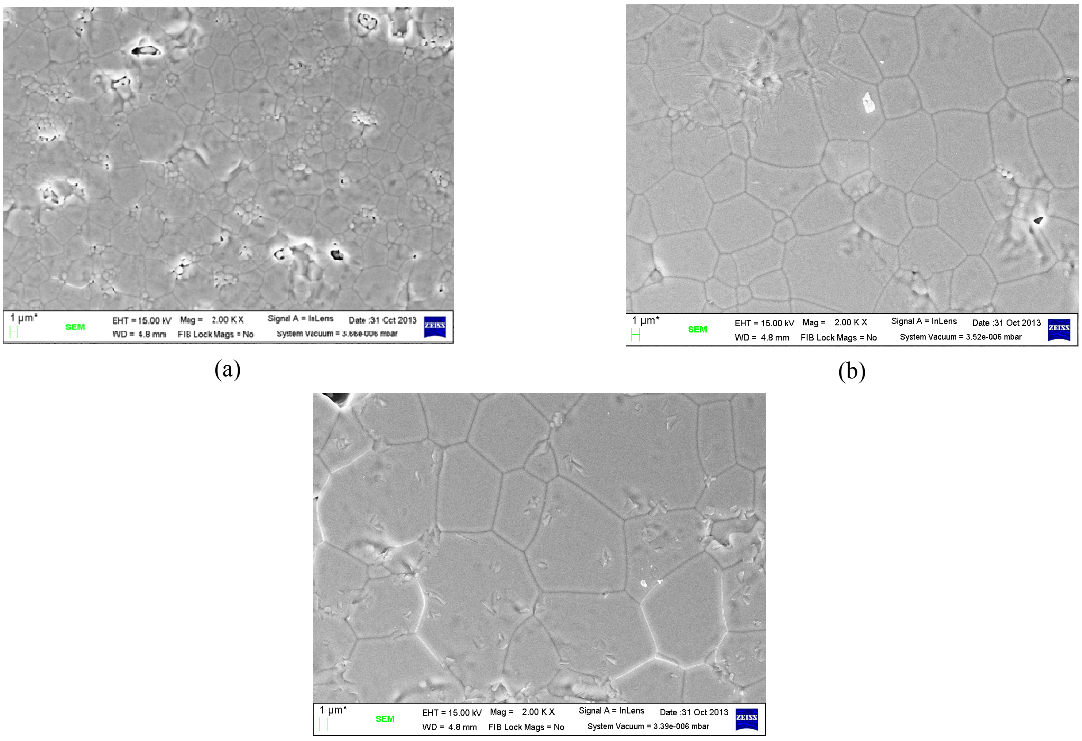


Fig.11. The hysteresis loop of $(Y_{2.4}Gd_{0.6})$ $(Mn_{0.13}Al_{0.4}Fe_{4.17})O_{12}$ sintered at 1400°C, 1450°C and 1480°C, respectively.

4. CONCLUSIONS

The adjustment of the magnetic properties of YIG ferrites are suitable for a S-band latching phase shifter. The substituting of Mn, Al can be improved by a proper physical process. The impact of a materials' sintering temperature is also discussed. By increasing the sintering temperature of Mn and Al substituted ferrites are obviously improved $4\pi M_s$, B_r, H_c and SQR more than that required. As the (Y_3) $(Mn_xAl_{0.83-x}Fe_{4.17})O_{12}$ ferrites with x=0.13 are sintered at 1430°C they show the optimum magnetic properties: $4\pi M_s$ =675G, B_r=575G, H_c=0.45Oe, SQR=0.86. The linewidth of ferromag-



(a) (b)

(c)

Fig.12. SEM micrograph (×2K) of $(Y_{2.4}Gd_{0.6})(Mn_{0.13}Al_{0.4}Fe_{4.17})O_{12}$ ferrites sintered at (a)1400°C; (b) 1450°C; (c) 1480°C.

netic resonance, ΔH, was also measured to be 38.6Oe at 3.2GHz. From our work, the same principles could be extended to ferrites and applied to a wide range of low frequency devices. If one chooses an appropriate initial composition of the ferrites and processed them with a proper sintering temperature. The results indicate that the formula for ferrite and the method mentioned above could be applied to ferromagnetic material to achieve similar property modifications.

5. ACKNOWLEDGEMENTS

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