Malaysian Journal of Science **21A**: 55-57 (2002)

Preparation of YIG by modified domestic iron oxide

M. Mozaffari and **J. Amighian**

Physics Dept., Isfahan University, Isfahan (81746-73441), Iran.

ABSTRACT Iron oxide by – product of a local steel Complex was modified to use for preparation of Yttrium iron garnet (YIG). The improvement was necessary to reduce impurities, especially the SiO_2 and Cl^- contents, which have deteriorative effects on magnetic properties and equipments used for preparation of the samples. The modified iron oxide was then mixed with Yttrium oxide of Merck Company in appropriate proportion to obtain a stoichiometric single phase YIG, using the conventional ceramic technique. XRD and SEM equipments were used to identify the resulting phases and microstructure respectively. Magnetic parameters were measured by VSM. Curie temperature of the samples was obtained by DTG (M) method. The results were compared with those obtained from samples that made by Merck iron oxide. There are small differences between the results. This was discussed according to extra pores and minute secondary phase in the samples made by domestic iron oxide.

(Raw materials, Yttrium iron garnets, Conventional ceramic technique and microstructure)

INTRODUCTION

In production of magnetic garnets, normally the main constituent is iron oxide, which has a major effect on physical and chemical properties of the prepared samples [1]. Also as cost is related to the price of raw materials, production of suitable iron oxide can have an important effect on the economy of the production [2]. Specific attention has been paid to the impurity contents of the iron oxide, especially to the silica and chlorine contents [3]. This is due to the fact that these have a deteriorative effect on the magnetic properties of the product [4]. The usual source of iron oxide is chemically precipitated powder prepared from sulfate solution or spray- roasted oxides obtained from Ruthner process. In Mobarakeh steel complex the same process is used and a huge amount of iron oxide by-product is obtained. This oxide is suitable for hard ferrite industries, but due to high-level impurities of SiO_2, Mn^{2+} and Cl^-, it is not suitable for soft ferrites, especially garnets.

EXPERIMENTAL

$Y_3Fe_5O_{12}$ polycrystalline samples have been produced by conventional ceramic technique. The starting materials used were improved iron oxide by-product of Mobarakeh steel complex and Y_2O_3 with minimum purity of 99% from Merck company. To improve the quality of the iron oxide, it is necessary to reduce SiO_2 and Cl^- contents to reasonable values. For this purpose, the iron oxide was dissolved in hydrochloride acid and filtered to separate unwanted SiO_2. By adding NaOH to the filtered chloride, a precipitation of hydroxide was obtained. This was washed by deionized distilled water several times and then heated in an electric furnace at about 500°C to obtain an improved iron oxide. The oxide was then washed with deionized distilled water several times again to remove any trace of residual chlorine. The chemical analysis of the refined oxide shows a reasonable reduction of initial SiO_2 and Cl^- contents in comparison with unrefined one. The raw material were wet mixed for 5 hours in the ratio appropriate for the reaction:

$$3\ Y_2O_3 + 5\ Fe_2O_3 \rightarrow 2\ Y_3Fe_5O_{12}$$

The mixture was then dried and calcined in an electric furnace at the temperature range of 1100 to 1250 °C. XRD patterns of the calcined powders were obtained by a Philips diffractometer X'Pert model. The calcined powder was then wet milled in acetone for 3 h, using a Fritsch P6 model planetary mill. A laser particle size analyzer was used to measure the particle size distribution of the powders. By

adding 1% of PVA to the powder, it was possible to press pellets 10 mm in diameter and 3 mm in thickness. The pellets were then sintered in the range 1350-1425 °C for 8 h in oxygen atmosphere. Saturation magnetization of the samples were measured by an Oxford VSM 3001 Model at a maximum field of 5 T. Microstructure and elemental identification of the sintered specimen were examined by a Cambridge S360 SEM, equipped with an energy dispersive spectrum (EDS). A TA 2050 balance was used for derivative thermomagnetometry (DTG/M/) measurements. The temperature program consists of four heating and cooling cycles starting well below the lowest transition and continuing to clearly above the highest one. In the course of measurements, the first heating and cooling runs are to diminish any possible trace of impurities entered in the sample in the course of preparation. Heating rates of 20 and 10 °C/min were employed for the first and second heating cycles respectively. In all measurements free control cooling rates were employed. The measurements were carried out in N_2 atmosphere.

RESULTS AND DISCUSSION

X-ray diffraction patterns of the calcined powders made by refined domestic and Merck iron oxides are shown in figures 1 and 2 respectively. As can be seen both garnets are single phase at calcining temperature of 1200 °C, while at calcining temperature of 1100 °C, there are extra lines of $YFeO_3$, Y_2O_3 and Fe_2O_3. This shows that the calcining temperature of 1100 °C is not sufficient to obtain a single-phase garnet.

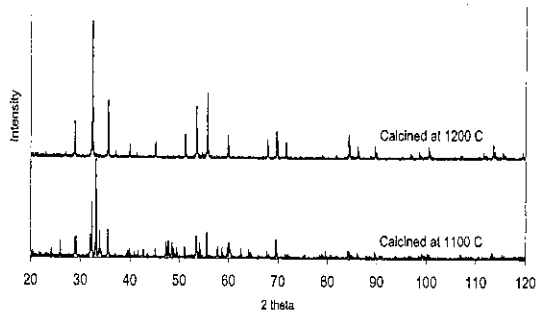

Figure 1. X-ray diffraction pattern of the calcined powders made by refined domestic iron oxide.

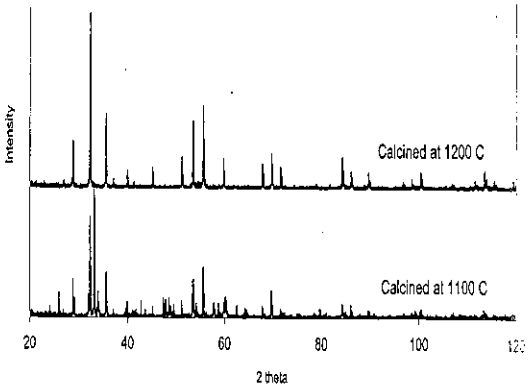

Figure 2. X-ray diffraction pattern of the calcined powders made by Merck iron oxide.

Particle size distribution of the milled powders is shown in the figure 3. As can be seen sizes of more than 75% of the particles are smaller than 1.5 μm. This distribution is quite suitable to achieve a reasonable dense sample in the course of the sintering process.

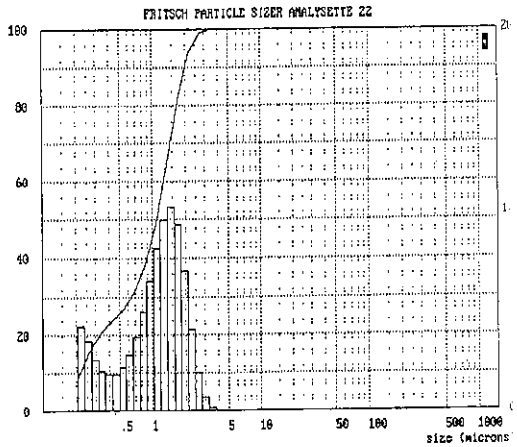

Figure 3. Particle size distribution of the milled powders.

SEM micrographs of the two sintered samples are shown in figures 4 and 5, for the samples made by refined and Merck iron oxides respectively. As can be seen in the figure 4 there are some pores and a secondary phase associated with the garnet made by refined domestic iron oxide. These cannot be seen in figure 5 associated with the garnet made by Merck iron oxide. Using EDS, it was found that the secondary phase in the first micrograph has basically formed by iron and oxygen together with a trace of manganese. This means that the secondary phase is basically iron oxide.

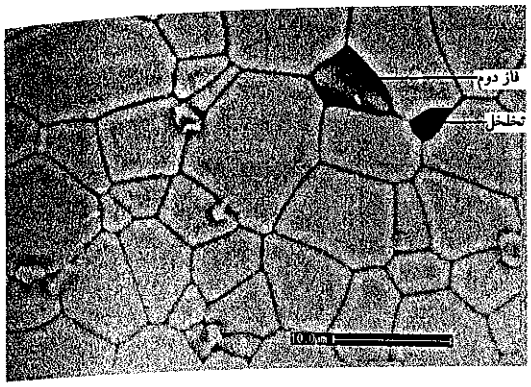

Figure 4. SEM micrograph of the sintered garnet made by refined domestic iron oxide.

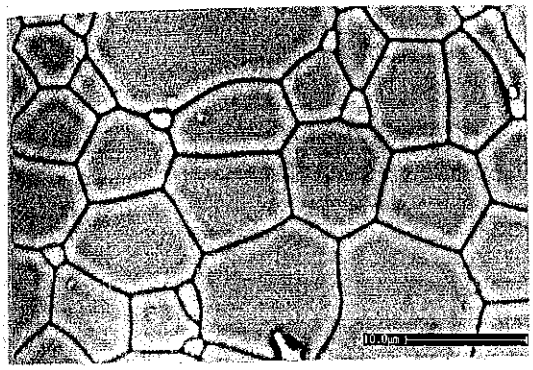

Figure 5. SEM micrograph of the sintered garnet made by Merck iron oxide.

Saturation magnetization of the refined domestic iron oxide garnet was 24.2 emu/g and that related to the Merck iron oxide garnet was 27.1 emu/g. the difference can be due to the extra pores and secondary phase in the garnet made by refined domestic iron oxide.

Curie temperatures of the samples made by refined and Merck iron oxides are 272 and 268 °C respectively, which shows a reasonable agreement with those reported elsewhere [6].

To see the degree of refinement of domestic iron oxide in our work, a sample was made, using the original domestic iron oxide with no refinement. Saturation magnetization and Curie temperature of the sample were also measured with the values of 13.2 emu/g and 235 °C respectively. As can be seen there are big differences between these figures and those obtained in the garnet made by refined domestic iron oxide. This shows the effectiveness of our route to achieve a suitable iron oxide.

REFERENCES

1. Gallagher, P. K. Johnson, D. W. Schrey Jr., F. and Nitti, D. J. (1973). Ceramic Bulletin **52**: 842-849
2. Wagner, U. (1980). *J. Magn. Magn. Mater.* **19**: 99-104
3. Tsuzaki, M. Takaki, K. Yoshikawa, F. Nakamura, M. and Kuriyama, T. (1991). *J. Mater. Eng.* **13:** 135-140
4. Chien, Y. T. and Ko, Y. C. (1990). *J. Mater. Sci.* **25**: 1711-1714
5. Ruthner, J. M. (1989). Proc. ICF5, India, **1**: 23-34
6. Winkler, G. Hansen, P. and Holst, P. (1972). *Philips Res. Repts.* **27**: 151-171