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STUDIES ON SOLID STATE REACTIONS OF FERRIMAGNETIC MATERIALS

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ABSTRACT

Thermogravimetry in a constant magnetic field was applied to detect magnetizable phases formed in the pre-sintering step of yttrium iron garnet preparation. The method was found suitable to follow the solid state reactions concerned and to characterize the effect of substituents.

INTRODUCTION

Polycrystalline ferrites - prepared from oxide powder mixtures - are widely used in telecommunication apparatus. The particular ferrites have different characteristics depending on the intended application.Their quality is usually determined by the saturation magnetization, dielectric loss /tg δ/ and Curie temperature /T_c/.In the case of yttrium iron garnet /YIG/ studied in the present work, the parameters mentioned above may be changed by the substitution of yttrium ions in the lattice [1]. For this purpose, vanadium, zirconium, aluminium and other metal substances are added to the starting mixture.

The optimization of the technology of ferrite production requires a test method of high sensitivity to detect ferrimagnetic phases formed during pre-sintering. X-ray powder diffraction techniques - usually applied for this purpose - have disadvantages: on the one hand, certain magnetizable and non-magnetizable phases cannot be discerned, on the other hand, the sensitivity of x-ray diffraction is not high enough to detect phases constituting small fractions of pre-sintered material. Thermogravimetry in a constant magnetic field TG/M/ offered a solution to this problem.

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EXPERIMENTAL METHOD

A Du Pont 951 thermobalance was applied. The magnetic field exerting a force on the sample was provided by a permanent magnet placed above the sample arm, outside the furnace. When the sample reaches the Curie temperature of a particular magnetizable phase, the balance detects a virtual mass increase caused by the significant decrease of the attracting force. Ferro- and ferrimagnetic phases may be detected and identified on the basis of the temperature of the step or the peak temperature of the derivative signal DTG/M/.

RESULTS AND DISCUSSION

The curves of Fig.1 show the formation of pure YIG from Y_2O_3 and Fe_2O_3. The samples were pre-sintered for equal times at different temperatures. /X-ray diffraction and IR spectroscopic characteristics of the samples were published elsewhere [2]. The garnet phase is responsible for the DTG/M/ peak near 300 °C; the peak at about 385°C detects the Curie temperature of ortoferrite. Visibly, the solid state reaction could start at 700°C, and ortoferrite occurred as intermediate of garnet formation. The highest ortoferrite content was observed in the sample treated at 1200°C; the product obtained at 1400° was free of ortoferrite and the sharp peak at 302°C shows that this was the most homogeneous garnet phase formed in the series of experiments discussed here.

Substitution reactions influencing the technical parameters of the product could also be followed with TG/M/ measurements. The behaviour of a ferric oxide - yttrium oxide - zirconium oxide mixture is demonstrated by Fig.2. Similarly to the basic mixture /Fig.1/ starting of the solid phase reactions was observed at 700°C; the Curie temperature of the garnet formed at this temperature is practically identical with the one measured in the basic mixture. However, the Curie temperatures of samples treated at 1100°C or above were 10°C lower, owing to the beginning incorporation of zirconium ions into the lattice. In the case of some other compositions, a similar trend of the ortoferrite Curie temperature was observed [3].

Our experience has shown that the solid state reactions of garnet formation and subtitution can be sensitively followed with

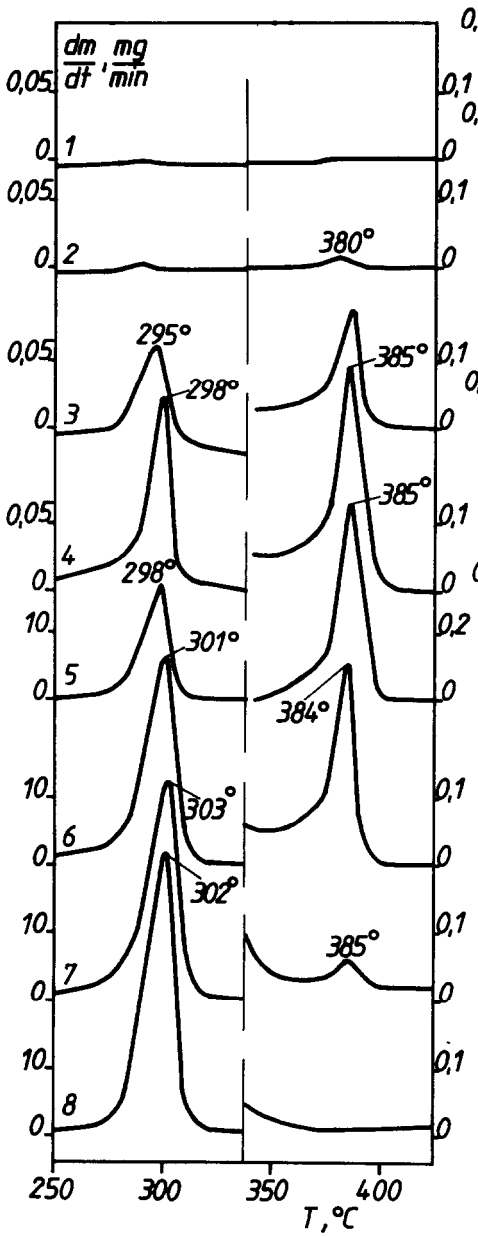

Figure 1. DTG/M/ curves of yttrium oxid-ferric oxide mixtures

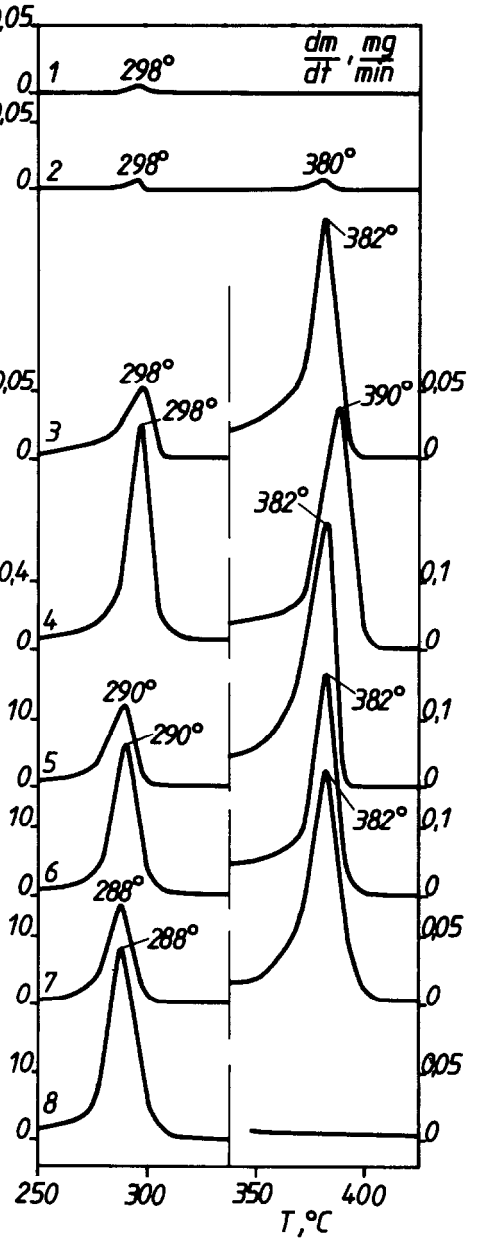

Figure 2. DTG/M/ curves of yttrium oxide-ferric oxide-zirconium oxide mixtures,

pre-sintered at different temperatures for 6 hours; 1-700°C; 2-800°C; 3-900°C; 4-1000°C; 5-1100°C; 6-1200°C; 7-1300°C; 8-1400°C.

thermogravimetry in a magnetic field. The further goal of our studies is to work out quantitative determination of magnetizable phases. From this point of view, the main problem is that several intermediate and product phases are not available in a pure form, and that structural differences of particular phases have an influence on the height of virtual mass change steps.

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