

[1, 2], the adherence  $\gamma_{IC}$  values obtained in reducing firing conditions should be indicative of the lack of a strongly interpenetrating thick film glass-metal interface. This is confirmed by the separation (fracture) surface analysis. The locus of separation of thick film-96 wt% substrate samples fired in Ar-H<sub>2</sub> to 850 and 950°C primarily involves separation at the smooth glass-metal interface together with fracture of the limited number of glass fibrils that penetrate into the conductor metal. The samples fired to 1050°C separated entirely along the smooth glass-metal interface which is devoid of interlocking glass fibrils.

In comparison to the data for the 96 wt% alumina, adherence is negligible after firing at 850°C in the reducing atmosphere when the 99+ wt% alumina and 0° sapphire substrates are used. The locus of thick film separation is along the glass/metal interface for both the 99+ wt% alumina and sapphire substrates. This behaviour is similar to the loss of adherence in air with the purer substrates where the loss of an interpenetrating glass-metal interface is further promoted by greater glass-to-substrate wetting [2].

Thus, the adherence of the fired thick film conductor is significantly degraded by the introduction of reducing versus oxidizing firing conditions. This is due to the decrease in the interpenetrating microstructure of the metal-glass interface brought on primarily by the poor glass-to-metal wetting and in part by the poor metal sintering. It is felt that residual carbonaceous contamination from incomplete binder pyrolysis in Ar-H<sub>2</sub> is responsible for the poor glass-to-metal wetting. Such effects of carbon contamination of the metal on wetting have been noted [4] and

could also result in the observed decrease in metal sintering. On the other hand, loss of metal surface oxide film, while it might cause poor glass-to-metal wetting, would lead to enhanced metal sintering. Finally, loss of thick film glass or flux constituents (notably B<sub>2</sub>O<sub>3</sub>, CdO, or Bi<sub>2</sub>O<sub>3</sub>) are not felt to be factors in the degraded adherence for reducing atmosphere firings as the glass still wets and flows on the alumina substrates and the TGA data does not indicate any related weight loss until temperatures > 900°C.

### Acknowledgement

It is a pleasure to recognize the work of L. A. Mann in the specimen preparation, T. J. Dohanue in the testing, and S. M. Morey in the screening electron microscopy. This study was supported by the U.S. Naval Air Systems Command, with J. W. Willis as Contract Monitor.

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Received 30 May  
and accepted 22 July 1977.

P. F. BECHER  
Naval Research Laboratory,  
Washington, D.C. 20375, USA

### Control of stress sensitivity in gadolinium yttrium iron garnets

Y<sub>2.01</sub>Gd<sub>0.99</sub>Fe<sub>4.925</sub>O<sub>12</sub> has been shown to have good magnetic properties for microwave applications, but the remanent magnetization is small [1]. Remanent magnetization and coercive force in garnets can be altered by stress developed in the specimens [2], and stress in the garnet toroid can result in a serious degradation in performance of

remnant devices [3]. It is intended to show in this letter that the remanent magnetization, coercive force and sintered density are improved by a small addition of manganese ion.

A study was undertaken to investigate the effect of manganese ion concentration on properties of Y<sub>2.01</sub>Gd<sub>0.99</sub>Fe<sub>4.925</sub>O<sub>12</sub> Y<sub>2.01</sub>Gd<sub>0.99</sub>Fe<sub>4.925-x</sub>Mn<sub>x</sub>O<sub>12</sub>, where  $x = 0, 0.03, 0.05, 0.07$  and  $0.09$ . Samples were prepared by spray-drying precursory solutions followed by rotary calcining,

a preparation technique which has recently been discussed [4]. Thin-walled toroids formed by pressing the calcined powders were sintered at 1460°C for 20h in an oxygen atmosphere. Sintered density and magnetic properties were measured. Fig. 1 shows the effect of manganese concentration on remanent magnetization, coercive force and sintered density of the sintered garnets. The results shows that a small addition of manganese ion improves the remanent magnetization, coercive force and sintered density. The best remanent magnetization and coercive force were obtained with a manganese concentration ( $x$ ) of 0.05 ions per unit formula. This agrees with Dionne and Goodenough, who found that the magnetostriction constant,  $\lambda_{100}$ , for single crystal  $Y_3Fe_{5-y}Mn_yO_{12}$  was zero with a manganese concentration of 0.05 ions per unit formula [5].

A further study of stress properties was made on samples of composition  $Y_{2.01}Gd_{0.99}Fe_{4.875}Mn_{0.05}O_{12}$  and  $Y_{2.01}Gd_{0.99}Fe_{4.925}O_{12}$ . Stress was introduced into the samples by grinding, and the

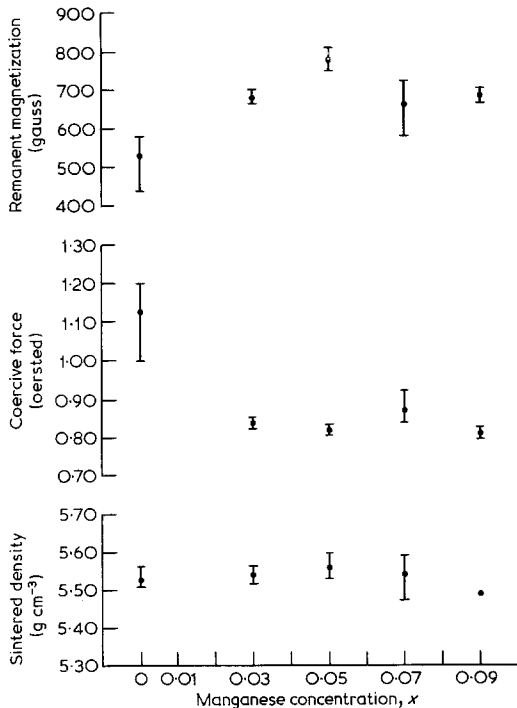


Figure 1 Effect of manganese concentration on remanent magnetization, coercive force and sintered density of  $Y_{2.01}Gd_{0.99}Fe_{4.925-x}Mn_xO_{12}$ .

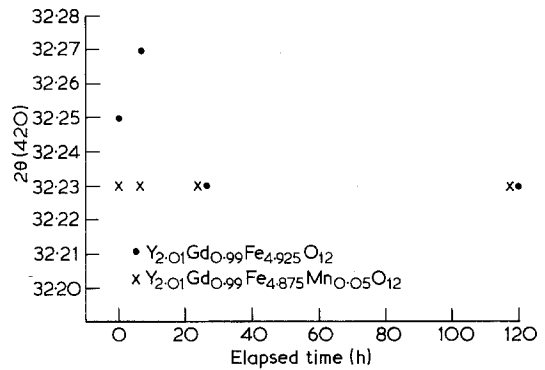


Figure 2 X-ray diffraction angle  $2\theta$  of the (4 2 0) plane versus elapsed time after grinding samples of  $Y_{2.01}Gd_{0.99}Fe_{4.925}O_{12}$  and  $Y_{2.01}Gd_{0.99}Fe_{4.875}Mn_{0.05}O_{12}$ .

ground samples were then X-rayed at various times after grinding. The variation in X-ray diffraction angle of the (4 2 0) plane as a function of elapsed time is shown in Fig. 2. Results show that the  $2\theta$  angle of the manganese-doped garnet stays the same, while the  $2\theta$  angle of the undoped garnet shifted until reaching a constant value 26 h after grinding. Stress induced by compression of the garnet was done by Stern *et al.* [2] and Epstein *et al.* [3]. It appears that manganese-doped garnets are less stress-sensitive than undoped garnets. The improvement of the magnetic properties of the manganese ion-incorporated garnets is attributed to a reduction in the stress sensitivity of the garnets.

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Received 22 June and accepted 22 July 1977.

B. W. JONG\*  
Georgia Institute of Technology  
Atlanta, Georgia, USA

\* Present address: U.S. Bureau of Mines, Tuscaloosa Metallurgy Research Center, University, Alabama, USA