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# The electromagnetic properties of Cu-substituted garnets with low sintering temperature

Jean-Pierre Ganne [a,*], Richard Lebourgeois [a], Michel Paté [a], Daniel Dubreuil [a], Ludovic Pinier [a,b], Hubert Pascard [b]

[a] *Thales Research and Technology, RD 128, 91767 Palaiseau, France*
[b] *Ecole Polytechnique, LSI, 91128 Palaiseau, France*

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**Abstract**

Yttrium iron garnet ceramics are used as non-reciprocal materials in circulators because they present low dielectric and magnetic losses, and their magnetic properties can be engineered in order to optimise the material for specific applications. The main drawbacks of circulators are their size and cost, due to complex mechanical assembling of different materials. A possible solution would be to adapt the different materials to a common LTCC process in order to produce circulators with a multilayer process. We showed that Cu substitutions enable us to decrease considerably the sintering temperatures of garnets, from about 1450 °C to about 1070 °C, and our more recent results show it will be possible to decrease this temperature to 1000 °C, which would be a suitable temperature for co-firing with gold. We present new microwave characterizations (the dielectric constant and losses, the different magnetic line-widths: $\Delta H_{\mathrm{eff}}$, $\Delta H_K$) of Cu-substituted garnets. The important impact of these characteristics on the properties of circulators is explained, and their variations versus composition and microstructure of the materials are shown.


*Keywords:* A. Firing; C. Magnetic properties; C. Dielectric properties; D. Ferrites; Circulator

## 1. Introduction

Microwave components, such as circulators or isolators make use of the non-reciprocity of the EM propagation induced by the ferromagnetic resonance in soft ferrites. When a soft ferrite is magnetized by an external magnetic static field, its internal magnetization vector M is aligned along the resulting internal field $H_0$. The direction of $H_0$ is usually chosen as the $z$ axis. If a microwave field $h$ ($h \ll H_0$, $h$ in the plane $xy$ perpendicular to $H_0$) is propagating through the ferrite, microwave permeability is described by Polder's tensor which accounts for ferromagnetic resonance at $F_0 = \gamma H_0$. The most used ferrite materials are yttrium iron garnet ceramics of composition $Y_3Fe_5O_{12}$ (YIG), with substitutions in order to tailor their electromagnetic characteristics.[1]

In the conventional powder process, the reaction of phase formation occurs at 1250 °C, and the pressed parts are sintered above 1450 °C in order to achieve full densification. We showed that Cu-substitutions enable to decrease sintering temperatures down to 1070 °C, which opens new ways of fabrication and integration of circulators: the objective is to produce them by LTCC technologies.[2] Moreover this decreased temperature produces materials with much finer microstructures than in conventional garnets, with the benefit of a better capacity to transmit microwave power,[3] which in turn will contribute to reduce component sizes.

In this paper, we present new results concerning Cu-substituted garnets of formula $Y_{3-2x/3}Cu_xFe_5O_{12}$ (YIG-Cu($x$)) where $0.02 < x < 0.28$. We measured their magnetic and microwave characteristics at 10 GHz: magnetization, permittivity and dielectric loss factor $\tan\delta$, magnetic effective line-width $\Delta H_{\mathrm{eff}}$, spin-wave line-width $\Delta H_K$. The magnetic effective line-width $\Delta H_{\mathrm{eff}}$ accounts for microwave magnetic losses, and the spin-wave line-width $\Delta H_K$ is the intrinsic material characteristics related to the microwave critical field for non-linearity $h_C$. In a good ferrite material, $\Delta H_{\mathrm{eff}}$ should be as small as possible in order to minimize losses, while $\Delta H_K$ should be as large as possible in order to allow to transmit high microwave power. These characteristics can be engineered in ferrites by doping or microstructure control. However their variations are correlated for reasons of physical mechanisms, so that a trade-off must be found between contradictory wishes:

* Corresponding author. Tel.: +331 69 41 58 91; fax: +331 69 41 57 38.
*E-mail address:* jean-pierre.ganne@thalesgroup.com (J.-P. Ganne).

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low losses, high power transmission capacity. Their values are correlated with the microstructures of our samples. We explain the impact of these material characteristics on the characteristics of a circulator at 10 GHz by two models, a simplified physical analytical model, and a more accurate calculation using a 3-D Finite Element model.

## 2. Experimental procedures

### 2.1. Synthesis

Polycrystalline samples with the chemical composition $Y_{3-2x/3}Cu_xFe_5O_{12}$ $Y_3$ are prepared using a conventional route. Raw materials with industrial grade are used: $Y_2O_3$, $Fe_2O_3$ and CuO. First, the starting powders are calcined during 2 h in air between 950 and 1050 °C. After grinding and pressing, the samples are sintered at 1070 °C for 2 h in an oxygen atmosphere.

XRD experiments with Co Kα1 radiation are performed to check the crystallographic structure of the resulting samples. Density is measured using the hydrostatic technique. Microstructures are studied by SEM.

### 2.2. Electromagnetic measurements

Permittivity and dielectric loss tangent are measured at 10 GHz in a rectangular cavity by a perturbation technique with a 1 mm diameter and 15 mm length ferrite rod. Effective ferromagnetic line-width $\Delta H_{eff}$ at 10 GHz is determined from the resonant modes excited in metallized cylindrical resonators of ferrite placed in a static field.[4] Two sizes of cylinders are used: diameters of 10 and 6 mm, with respective heights of 7 and 4 mm. Spin-wave line-width $\Delta H_K$ is determined by measuring the electromagnetic power level for reaching the non-linearity threshold by parallel pumping of a 2.5 mm diameter sphere placed inside a dielectric resonator.[5]

### 2.3. Simulation of circulators

The relation between the characteristics of garnet materials and those of X-band circulators is calculated in two ways: by a simple analytical model adapted from a reference text-book by Helszajn,[6] and by using Ansoft HFSS, a commercial 3-D electromagnetic simulator.

## 3. Results and discussions

### 3.1. Material synthesis and microstructures

Several pre-experiments were made between 950 and 1050 °C in air to determine the best calcining and firing conditions. XRD patterns indicate that only cubic garnet phase was present when the powder was fired at 1050 °C. We used the method described by Nelson & Riley to calculate the lattice parameter. The densities of sintered samples were systematically measured and compared with the theoretical densities of the ideal corresponding garnets, assuming that copper is located in yttrium sites. This procedure showed that full densification was achieved for the samples sintered between 1070 and 1080 °C (their relative density was higher than 99%).

EDX analyses showed that copper is uniformly located inside the ferrite grains. The precision was not sufficient to say if copper concentration is higher at the grain boundaries. Nevertheless, it confirms that copper atoms are substituted for yttrium atoms.

TEM show that grain size strongly increases with sintering temperature, from about 3 μm to more than 400 μm, respectively, from 1065 to 1080 °C. This suggests the formation of a melting phase that could promote grain growth. However our recent experiments show that this effect can be controlled by using additives as grain growth inhibitors.

Fig. 1 compares the microstructure of a Cu-substituted garnet sintered at 1070 °C and that of a commercial garnet sintered at 1500 °C. Notice the difference in the scales. The new material present a much finer grain size than the commercial material. These fine microstructures are known to increase the spin-wave line-width $\Delta H_K$ of the materials very effectively, with a moderate increase of magnetic losses $\Delta H_{eff}$. In a microwave ferrite, there is a critical microwave magnetic field $h_C$ above which a non-linear regime of spin-waves is established, and losses increase catastrophically. This is a fundamental limitation for

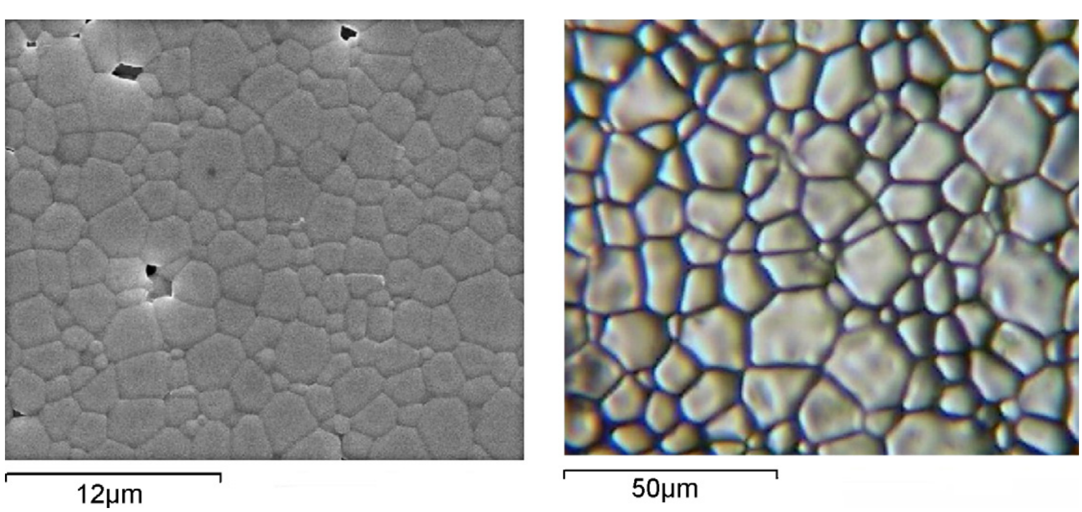


Fig. 1. Microstructures of a Cu-substituted garnet sintered at 1070 °C (left) and of a commercial garnet sintered at 1500 °C (right).

Table 1
Electromagnetic characteristics at 10 GHz of Cu-substituted garnets sintered at 1070 °C

| $x$ | $\varepsilon$ at 10 GHz | $\tan\delta_\varepsilon$ at 10 GHz ($\times 10^{-3}$) | $\Delta H_{\text{eff}}$ (Oe) ($\pm$15%) |
|---|---|---|---|
| 0.28 | 15.1 | 3.8 | 40 |
| 0.25 | 14.9 | 3.7 | 35 |
| 0.15 | 14.7 | 1.8 | 25 |
| 0.10 | 15.3 | 1.4 | 20 |
| 0.05 | 15.3 | 0.4 | 12 |
| 0 (commercial) | 15.5 | 0.2 | 8 |

The last line concerns a commercial material sintered at 1500 °C.

Table 2
Effective line-widths and spin-wave line-widths of Cu-substituted garnets as a function of Cu concentrations

| $x$ | $\Delta H_{\text{eff}}$ at 10 GHz | $\Delta H_K$ at 10 GHz |
|---|---|---|
| 0.05 | 10 Oe ± 2 | 1.3 Oe ± 0.1 |
| 0.04 | 15 Oe ± 5 | 1.6 Oe ± 0.1 |
| 0.03 | 16 Oe ± 6 | 1.6 Oe ± 0.1 |
| 0.02 | 9 Oe ± 2 | 2.1 Oe ± 0.1 |
| 0 (YIG) | 6 Oe ± 1 | 0.4 Oe ± 0.1 |

Materials sintered at 1070 °C. Bottom line: commercial material sintered at 1500 °C.

the microwave power that can be transmitted in a circulator. This critical field $h_C$ is proportional to the spin-wave line-width $\Delta H_K$, which is an intrinsic characteristics of microwave ferrites. Consequently we expect that these new materials with fine grains will present higher power transmission capacity than classical materials, for comparable magnetic losses. Conversely, for the same power transmission capacity (same $\Delta H_K$), the new material will present lower magnetic losses ($\Delta H_{\text{eff}}$) than classical materials.

### 3.2. Electromagnetic characteristics of materials

Table 1 shows the results of measurements of dielectric constant and losses and of magnetic effective line-width made on a first series of materials of formula $Y_{3-2x/3}Cu_xFe_5O_{12}$ with $0.05 < x < 0.28$, and sintered at 1070 °C. The characteristics of a commercial non-substituted garnet ($x = 0$) are included for reference in the bottom line.

The dielectric constant does not change much. The lower values for $x = 0.15$ and 0.25 correspond to samples slightly less densified than others. The main observations are that both dielectric and magnetic losses increase linearly with Cu concentration, and that for the lower Cu concentration, material characteristics tend to be equal to those of the reference material. Consequently we decided to realize new samples with lower Cu concentrations, with $x$ between 0.02 and 0.05, using the same conditions for calcining and sintering. All samples were well densified at 1070 and 1080 °C.

We measured the effective line-widths $\Delta H_{\text{eff}}$ which characterize the magnetic losses of the materials, and the spin-wave line-widths $\Delta H_K$ which characterize their power transmission capacity. The results are shown in Table 2. As in Table 1, the bottom line concerns a classical commercial reference material. Clearly reducing Cu concentration is beneficial as the decrease of $\Delta H_{\text{eff}}$ is confirmed, when comparing with values in Table 1. On the contrary $\Delta H_K$ tend to increase for lower Cu concentration, which can be due to a microstructure effect. The overall result is that there is an optimum for $x = 0.02$. At this Cu concentration, the garnet present magnetic losses $\Delta H_{\text{eff}}$ similar to those of the classical material (slightly higher) with a power capacity $\Delta H_K$ higher by a factor 4 to 5.

### 3.3. Correlation between microstructures and $\Delta H_K$

During this research, we found some inconsistent results in measurements of $\Delta H_K$ made on different spherical samples extracted of the same sintered parts. We got very different values, for instance 1.1 and 4.5 Oe. The differences cannot be ascribed to the measurement technique: we checked that uncertainty and reproducibility are of about 0.1 Oe in this range of

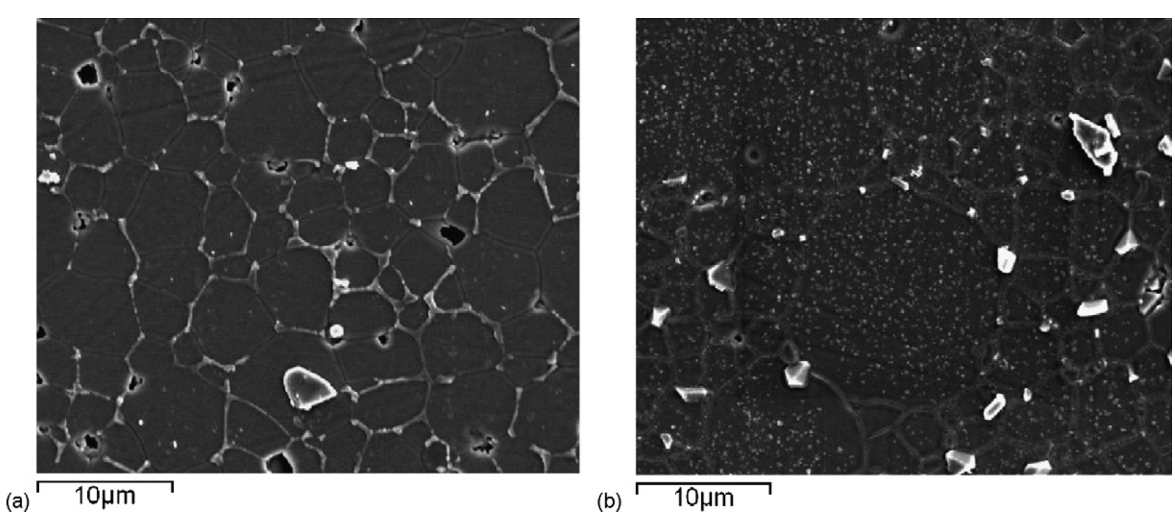


Fig. 2. Microstructures of two different spherical samples (a) and (b) used to measure $\Delta H_K$. Both spheres are machined from the same sintered part. $\Delta H_K$ (a) = 4.5 Oe > $\Delta H_K$ (b) = 1.1 Oe.

Table 3
Values of $\Delta H_K$ measured on different spheric samples machined from the same parts

| $T_{sintering}$ (°C) | $\Delta H_k$ (measurements of different spheres) (Oe) | | | | | Fluctuation |
|---|---|---|---|---|---|---|
| 1070 | 0.8 | 4.9 | 3.3 | 0.8 | 1.0 | 3.1 |
| 1080 | 0.8 | 0.7 | 0.7 | 0.7 | | 0.1 |

Materials $x = 0.05$ sintered at 1070 and 1080 °C. Measurement incertitude ± 0.1 Oe.

values. Microstructure observations made on the very spheres used for measurements gave the key, revealing that different spheres present different microstructures.

Fig. 2 compares the microstructures of two spheres made from the same material ($x = 0.1$, sintering temperature 1070 °C). The microstructure of the sphere which presents the higher value of $\Delta H_K$ (4.5 Oe) is fine and homogeneous (Fig. 2a, left), while the microstructure of the sphere with the lower value (1.1 Oe) is coarse and heterogeneous (Fig. 2b, right).

These observations were made on materials sintered at 1070 °C, which tend to present fine grains and high values of $\Delta H_K$. They show the importance of controlling grain growth in these materials. On the contrary for materials sintered at higher temperatures, grain sizes are much larger and the values of $\Delta H_K$ are consistently much lower, and much more reproducible. Table 3 shows a comparison of the dispersion of the values of $\Delta H_K$ for different spherical samples of the same material composition ($x = 0.05$), sintered at 1070 and 1080 °C. The dispersion is comparable to measurement uncertainties for samples sintered at 1080 °C, while it is much higher for samples sintered at 1070 °C.

### 3.4. Circulator simulations

In order to study the relations between material characteristics and circulator performance we built a simple 3-D model of strip-line circulator using HFSS.

Fig. 3(a) gives a schematic view of the model of an S-band circulator. The microwave ferrite discs are 0.75 mm thick and 14.5 mm in diameter.

The ferrite material is defined by its characteristics which are used to calculate Polder's tensor (in a magnetic static field supposed to be homogeneous, a strong approximation). Not shown are the magnets and the soft material armature. Their characteristics and design determine the value of the static magnetic field inside microwave ferrite. The purpose was not to make an exact model, but rather to study the impact of the improvements in microwave ferrites on circulator performances. The model enables us to calculate the S parameters of the circulator as a function of material characteristics and to visualize the intensities of microwave fields inside the circulator for a given microwave power. An example is shown on Fig. 3(b) for the same S-band circulator.

We studied the effect of materials losses on circulator insertion losses through a simple model of X-band circulator.

The main causes of losses are

- metallic (ohmic) losses in lines, ground planes and walls due to resistivity. As lines and planes are much thicker than skin depth, ohmic losses vary proportionally to the square root of resistivity
- dielectric losses in dielectric and ferrite materials: tan $\delta$
- magnetic losses in ferrite, included in Polder's tensor as the effective line-width $\Delta H_{eff}$.

Fig. 4 shows the absolute values of the S parameters in dB versus frequency between 8 and 12 GHz, for our ideal model circulator, when materials present no losses. The circulator works well between 9 and 10.4 GHz. In this range insertion losses are about 0.02 dB, which is due to non-ideal matching and isolation, a fraction of power is reflected and another fraction is transmitted to the isolated port.

We then applied different values of loss parameters, for the different causes of losses.

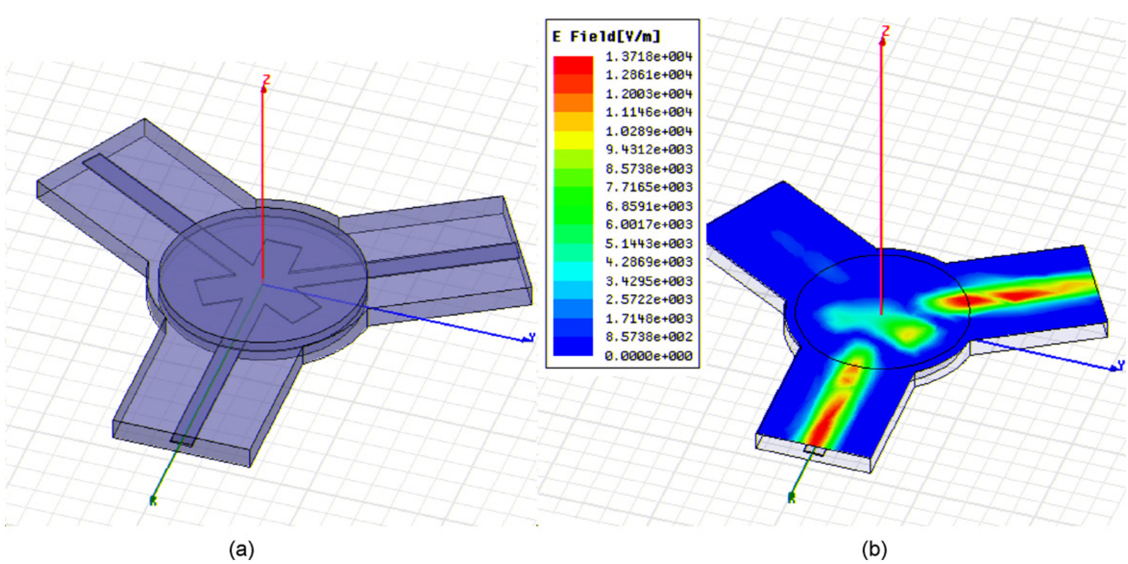


Fig. 3. Schematic views of a model of S-band circulator, (a) showing metal lines and ferrite discs (left), and (b) the intensity of microwave E field (right).

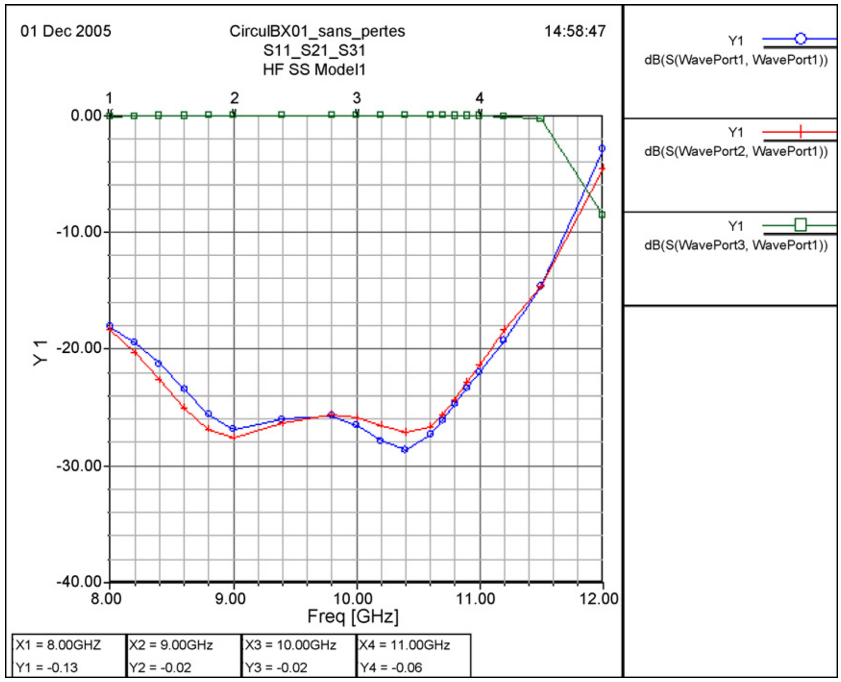


Fig. 4. S-parameters in dB of an ideal X-band circulator model, with no material losses. Green curve: insertion losses nearly confused with the reference 0 dB axis. Red and blue curves: isolation and reflection in dB, respectively.

Fig. 5 shows the values in dB of circulator insertion losses as a function of the values of metallic losses, dielectric losses and magnetic losses, respectively, all other causes of losses being set to zero. The curves are corrected of 0.02 dB in order to eliminate initial losses due to non-ideal matching and isolation. In Fig. 5(a), metallic losses are quantified as the square root of the resistivity of metals normalized to the resistivity of pure copper. In Fig. 5(b), dielectric losses are quantified as $\tan\delta$, and in Fig. 5(c) magnetic losses are quantified as $\Delta H_{\mathrm{eff}}$ in Oerstedts. Notice that we get linear behaviours, insertion losses are proportional to the different loss factors.

We checked the total insertion losses combining the effects of the different causes of losses: it is a simple linear combination of the insertion losses due to the different contributions of materials losses. Fig. 6 shows a typical good case, with low loss materials. Table 4 gives the values of the contributions of material losses to circulator insertion losses for two cases: a typical worst case, with lossy materials, a typical good case, same as in Fig. 6.

### 3.5. Circulator analytical model

A simple analytical model gives a better insight in the relation between circulator insertion losses and materials characteristics and enables us to estimate the contributions of the different kinds of materials losses to insertion losses. The model is derived from the approach by Helszajn in his text-book on circulators.[6] Metallic losses are not accounted for in Helszajn's model, we add their contribution using a classical result derived in Moreno's text-book on microwave lines and cavities.[7]

We consider an ideal circulator as a cavity, with three ports. We use the following classical notations: $P_{\mathrm{i}}$, $P_{\mathrm{t}}$, $P_{\mathrm{r}}$ are the incident power, transmitted power and reflected power, respectively, $P_{\mathrm{d}}$ is the power dissipated in the cavity (due to materials losses), $Q_{\mathrm{L}}$ and $Q_0$ the loaded and unloaded quality factors, $E$ the reactive energy stored in the cavity, $\omega$ the pulsation, $\omega = 2 \times \pi \times F$ where $F$ is the frequency.

The circulator is supposed to be perfectly matched for reflection and isolation: $P_{\mathrm{r}} = P_{\mathrm{t3}} = 0$.

Energy conservation gives: $P_{\mathrm{i}} = P_{\mathrm{t}} + P_{\mathrm{d}}$

By definition, we can write: $P_{\mathrm{i}}/\omega E = 1/Q_{\mathrm{L}}$ and $P_{\mathrm{d}}/\omega E = 1/Q_0$

Losses are supposed to be very small, so $P_{\mathrm{d}} \ll P_{\mathrm{i}}$ and $Q_0 \gg Q_{\mathrm{L}}$

They may be of different origins: magnetic, dielectric, or ohmic so we can write:

Total losses = magnetic losses + dielectric losses + ohmic (conductor) losses.

Table 4
Contributions of material losses to circulator insertion losses at 10 GHz for two cases: a typical case, same as in Fig. 6, and a bad case, with lossy materials

| Kind of losses | Typical case, materials | Typical case, insertion losses | Bad case, materials | Bad case, insertion losses |
|---|---|---|---|---|
| Resistivity | Pure copper resistivity | 0.10 dB | 10 × copper resistivity | 0.27 dB |
| Magnetic | 50 Oe | 0.09 dB | 50 Oe | 0.09 dB |
| Dielectric | $2 \times 10^{-4}$ | 0.02 dB | $3.8 \times 10^{-3}$ | 0.13 dB |
| Total | | 0.21 dB | | 0.49 dB |

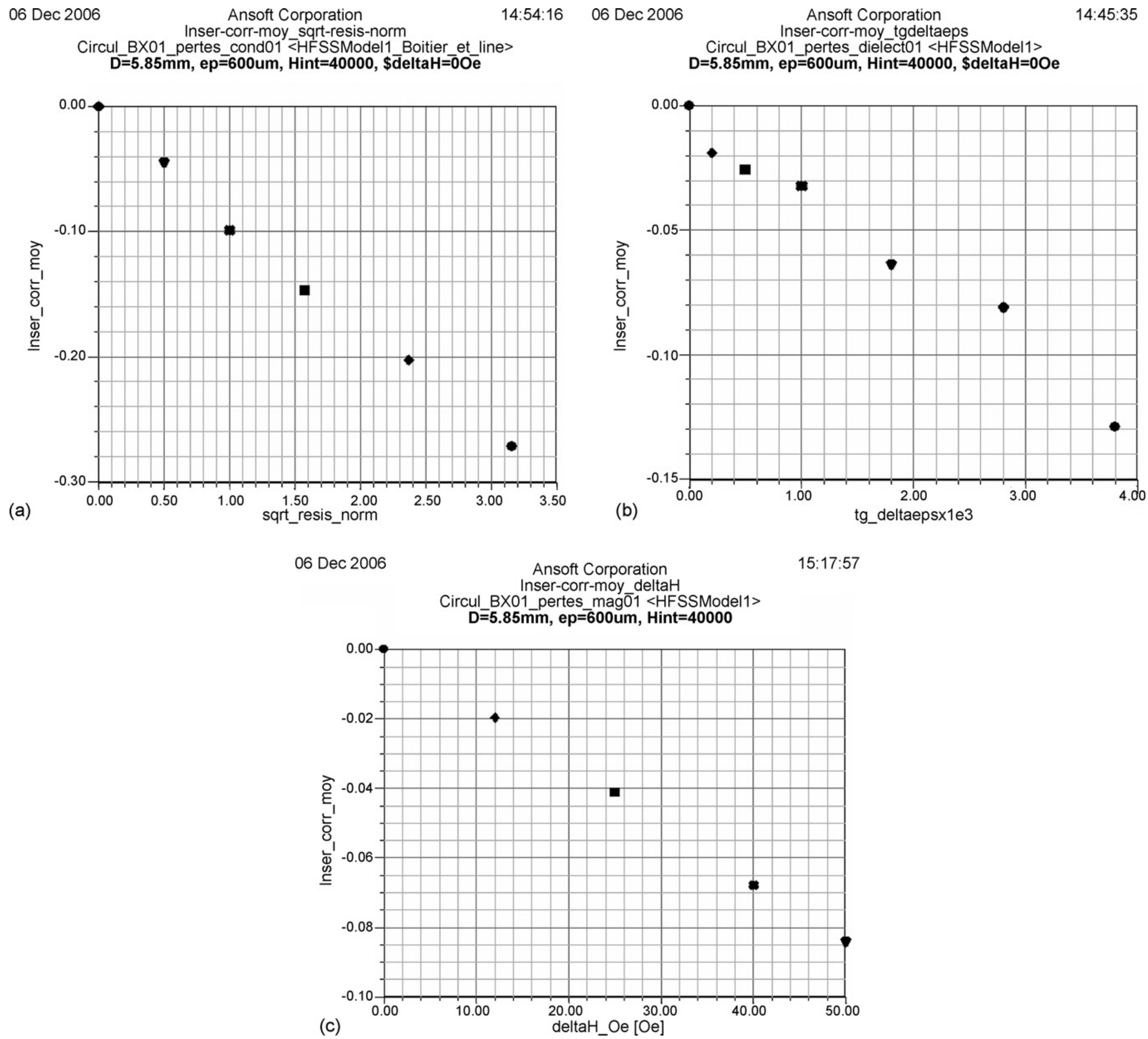


Fig. 5. Influence of the different materials loss contributions on the insertion losses (dB) of an X-band model circulator: (a) effect of metallic losses, represented as the square root of resistivity normalized to that of pure copper (b) effect of dielectric losses, as $\tan\delta$ in $10^{-3}$, (c) effect of magnetic losses, as $\Delta H_{\mathrm{eff}}$ in Oerstedts.

For every kind of loss we define a $Q$ factor: loss/$\omega E = 1/Q$

These $Q$ factors can be explicitly related to materials loss factors:

For magnetic losses : $$\frac{1}{Q_{\mathrm{magnetic}}} = \frac{\gamma \Delta H_{\mathrm{eff}}}{2\omega}$$

For dielectric losses : $$\frac{1}{Q_{\mathrm{dielectric}}} = \tan\delta$$

For conductor losses : $$\frac{1}{Q_{\mathrm{conductor}}} = \frac{\text{Distance between metal surface}}{\text{Skin depth}} = \frac{\text{Ferrite thickness}}{\text{Skin depth}}$$

Total losses = magnetic losses + dielectric losses + conductor losses

So : $$\frac{1}{Q_0} = \frac{1}{Q_{\mathrm{magnetic}}} + \frac{1}{Q_{\mathrm{dielectric}}} + \frac{1}{Q_{\mathrm{conductor}}}$$

Now we derive the relation between Insertion Losses and total losses in the cavity. From energy conservation:

$$P_{\mathrm{t}} = P_{\mathrm{i}} - P_{\mathrm{d}}, \quad \text{whence} \quad \frac{P_{\mathrm{t}}}{P_{\mathrm{i}}} = 1 - \frac{P_{\mathrm{d}}}{P_{\mathrm{i}}} = 1 - \frac{Q_{\mathrm{L}}}{Q_0}$$

We have to calculate insertion losses (IL) in dB:

$$\mathrm{IL} = -10\log\frac{P_{\mathrm{t}}}{P_{\mathrm{i}}} = -20\log\frac{E_{\mathrm{t}}}{E_{\mathrm{i}}} \quad \text{(in voltages)}$$

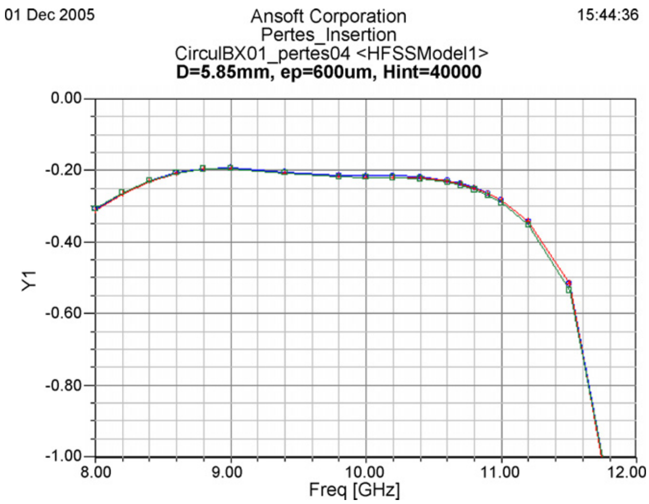


Fig. 6. Insertion losses versus frequency for an ideal circulator model, using low loss materials. Materials characteristics are given in Table 4, in columns "typical case".

$$\mathrm{IL} = -\frac{20}{\ln(10)} \times \ln\left(\frac{1 - Q_\mathrm{L}}{Q_0}\right) = +8.686 \times \frac{Q_\mathrm{L}}{Q_0}$$

(We use $Q_\mathrm{L}/Q_0 \ll 1$ and $\ln(1+x) = x$ for $x \ll 1$).

The estimation of $Q_\mathrm{L}$ is not so easy, but in most circulators its value is between 2 and 5, so that IL (dB) is between $20/Q_0$ and $40/Q_0$. We choose a mean point: $Q_\mathrm{L} = 3.5$, so that

$$\mathrm{IL(dB)} = \frac{30}{Q_0} = 30 * \left(\frac{1}{Q_\mathrm{magnetic}} + \frac{1}{Q_\mathrm{dielectric}} + \frac{1}{Q_\mathrm{conductor}}\right)$$

Using the expressions for the various contributions, we get additive losses, and we can write:

$$\mathrm{IL(dB)} = A \times \Delta H_\mathrm{eff} + B \times \tan\delta + C \times \text{square root (resistivity)}$$

where $A$, $B$ and $C$ are constant.

Now we can estimate the different contributions to IL(dB):

Magnetic losses:

$$\text{Suppose} \quad \Delta H_\mathrm{eff} = 50\,\mathrm{Oe}, \quad F = 10\,\mathrm{GHz},$$

$$\gamma \text{ is a constant} = 2.8\,\mathrm{MHz/Oe}$$

$$\text{We get :} \quad \frac{1}{Q_\mathrm{mag}} = 0.007 \quad \text{and} \quad \mathrm{IL(mag)} = 30 \times 0.007 = 0.2\,\mathrm{dB}$$

Dielectric losses:

$$\text{Suppose} \quad \tan\delta = 4 \times 10^{-3}$$

$$\text{We get} \quad \frac{1}{Q_\mathrm{diel.}} = 0.004 \text{ and IL (diel)} = 0.12\,\mathrm{dB}$$

Conductor losses:

Suppose dielectric thickness = 600 μ, resistivity = 10 × resistivity (Cu), skin depth of Cu is 0.66 μ at 10 GHz, here it is about three times larger, about 2 μm

$$\text{We get} \quad \frac{1}{Q_\mathrm{cond.}} = \frac{2}{600} = \frac{1}{300} \text{ et IL (conductor)} = \frac{30}{300} = 0.1\,\mathrm{dB}$$

Comparing these values with those given by the 3-D full calculation, we observe some discrepancies: the analytical model underestimates metallic losses (this can be explained by the fact that the 3-D model takes the access lines and the lateral walls into account, which is not the case in the analytical model) and it overestimates magnetic losses (a possible explanation is that it overestimates the filling factor). We see that we get the same linear relation as found with the 3-D model but our analytical model is far from giving exact results, which can be explained by the fact that filling factors are not taken into account. Then the analytical model must be considered as a heuristical approach which gives physical insights on the causes of losses and their orders of magnitude, while the 3-D model enables us to make more exact estimates, and compare the relative values of different kinds of losses.

## 4. Conclusions

New compositions of garnet material with Cu substitutions were studied. They can be sintered at 1070 °C, a temperature 400 °C lower than non-substituted commercial materials. Their microstructure is much finer, which improves considerably the microwave power density that can be transmitted. Recent experiments showed that we can decrease sintering temperatures further, in order to make this material co-firable with metal pastes, gold or silver, and produce stacks including conducting lines and ground planes by LTCC technology. Two simple models of circulator enabled us to explain the relations between circulator characteristics and material characteristics. These models will be used in order to optimise simultaneously the design of circulators and the properties of garnet materials.

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