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**ELEC. & STRUCTURAL PROP. STUDY OF LAYERED DIELECTRIC & MAGNETIC**

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UNIVERSIDADE FEDERAL DO CEARA.**

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Final Report**

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14. ABSTRACT In this work the magnetic and dielectric properties of ceramic-ceramic and ceramic-polymer composites with BiNbO <sub>4</sub> , SrBi <sub>2</sub> Nb <sub>2</sub> O <sub>9</sub> (SBN), BaBi <sub>4</sub> Ti <sub>4</sub> O <sub>15</sub> (BBT), Na <sub>2</sub> Nb <sub>4</sub> O <sub>11</sub> (NNO), Sr <sub>2</sub> CoNbO <sub>6</sub> (SCN) and ferrites BaFe <sub>12</sub> O <sub>19</sub> and Y <sub>3</sub> Fe <sub>5</sub> O <sub>12</sub> (YIG) was studied for RF and microwave applications. New configurations of magneto-dielectric composites and blends structures for high frequency applications was done. A new method for the measurement of the temperature coefficient of resonant frequency (Tf), is presented. The traditional method (based on the Courtney method) present some limitations of measuring the values of Tf, for samples with high dielectric loss.					
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## FINAL TECHNICAL REPORT- FA9550-11-1-0095

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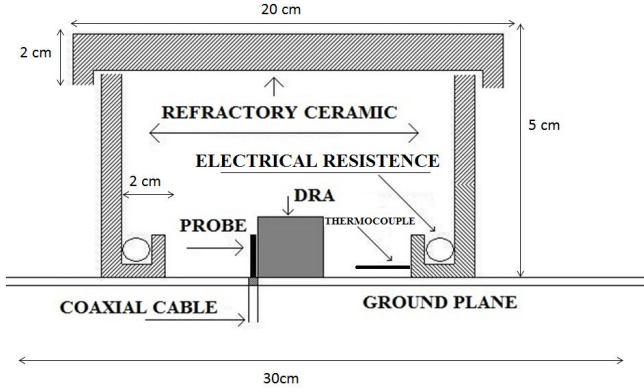
In this work the magnetic and dielectric properties of ceramic-ceramic and ceramic-polymer composites with  $\text{BiNbO}_4$ ,  $\text{SrBi}_2\text{Nb}_2\text{O}_9$  (SBN),  $\text{BaBi}_4\text{Ti}_4\text{O}_{15}$  (BBT),  $\text{Na}_2\text{Nb}_4\text{O}_{11}$  (NNO),  $\text{Sr}_2\text{CoNbO}_6$  (SCN) and ferrites  $\text{BaFe}_{12}\text{O}_{19}$  and  $\text{Y}_3\text{Fe}_5\text{O}_{12}$  (YIG) was studied for RF and microwave applications. New configurations of magneto-dielectric composites and blends structures for high frequency applications was done. The 0-3 type dielectric and magnetic composites with homogenously distributed ceramic inclusions was fabricated in a polymer matrix. Magnetic Yttrium Iron Garnet (YIG) and (SBN) powders were used to enhance the permittivity and permeability of the composites. This group of dielectric and magnetic phases was studied in the RF and microwave region. The microstructure, high frequency dielectric and magnetic properties of individual layers and 2-2 composites was investigated and measured.

A new method for the measurement of the temperature coefficient of resonant frequency ( $\tau_f$ ), is presented. The traditional method (based on the Courtney method) present some limitations of measuring the values of  $\tau_f$ , for samples with high dielectric loss due to their inability to observe clearly the  $\text{TE}_{011}$  mode. The new experimental setup, to measure the  $\tau_f$  value, is based on the variation of the temperature of the dominant mode of a dielectric resonator antenna (DRA).

The study of the thermal stability of magneto-dielectric composites is important for applications at the microwave band and in the millimeter and near millimeter region (100-300GHz) where the thermal stability of the resonators is fundamental.

In this project we are investigating experimentally and numerically this new method to measure the thermal stability of layered dielectric and magnetic composite structures for RF and Microwave Applications .

In the area of communication is important that the devices, responsible for transmitting/receiving data have its characteristics preserved in whatever temperature environment they are submitted. This new method for the measurement of the temperature coefficient of resonant frequency ( $\tau_f$ ), is presented. The traditional Courtney method, present some limitations of measuring the values of  $\tau_f$ , for samples with high dielectric loss due to their inability to observe clearly the  $\text{TE}_{011}$  mode. The new experimental setup (figure below), to measure the  $\tau_f$  value, is based on the variation of the temperature of the dominant mode of a dielectric resonator antenna.



Modified setup, for the measurement of  $\tau_f$

A new method to measure the microwave thermal stability coefficient  $\tau_f$

$$\tau_f = \frac{1}{f_i} * \frac{\Delta f}{\Delta T} * 10^6,$$

To use this new method a group of traditional materials were used to compare the traditional and new method

TABLE I.  $TE_{01\delta}$ ,  $HE_{11\delta}$  e  $TM_{01\delta}$  modes and dielectric parameters of CTO,  $Al_2O_3$ , and BTNO dielectrics.

	$CaTiO_3$	$Al_2O_3$	BTNO
a (mm)	7.48	12.70	7.31
h (mm)	8.04	12.70	7.38
a(mm)/h(mm)	0.93	1	0.99
$\epsilon_R$	92.25	9.80	63.68
$\tan \delta$	$5.81 \times 10^{-4}$	$1.11 \times 10^{-4}$	$5.61 \times 10^{-2}$
$f_{monopole}$ (GHz) measured	1.888	3.089	2.439
$f_{HE11\delta}$ (GHz) calculated	1.837	3.147	2.328
$f_{TE01\delta}$ (GHz) calculated	1.830	3.201	2.288
$f_{TM01\delta}$ (GHz) calculated	2.695	4.527	3.357

Used samples in the measurements

The comparative between the two systems for measurement of  $\tau_f$  values, show excellent agreement, as observed in Figure 4. In the Courtney procedure the obtained value is 621.10 ppm/ $^{\circ}C$  and compares to 624.32 ppm/ $^{\circ}C$  obtained in the DRA procedure. Both measurements exhibit the same linearity and angular coefficient (see TableII and Figure4).

The frequency evolution of the  $HE_{11d}$  mode with increasing temperature for DRA procedure is showed in Figure 5, where the  $HE_{11d}$  mode is isolated and well defined. The decrease in the return loss (in modulus) is associated to impedance matching variation due to volumetric expansion and the change in value of dielectric permittivity the DRA with temperature. The measurement of  $\tau_f$  for the BTNO phase was not reported in the literature. We believe that the reason is the high dielectric loss, which almost do not allows to use the Courtney method. In this case, the resonances are too broad. Considering the Courtney geometry, the quality factor for  $TE_{011}$  mode is low, leading to a broad band. The monitoring of the resonant frequency shift with temperature is quite difficult with the enlargement of this band and a very poor mode visualization, see Figure7.

In the present proposed new method, the measurement of the  $\tau_f$  for BTNO is quite satisfactory. The  $HE_{11d}$  mode is quite strong and well defined . The value of  $\tau_f = -104.19$  ppm/ $^{\circ}C$  (Table II) was obtained for the first time. The linearity for frequency shift with temperature increase is showed in Figure 9, where a good linear agreement of the frequency with temperature was obtained.

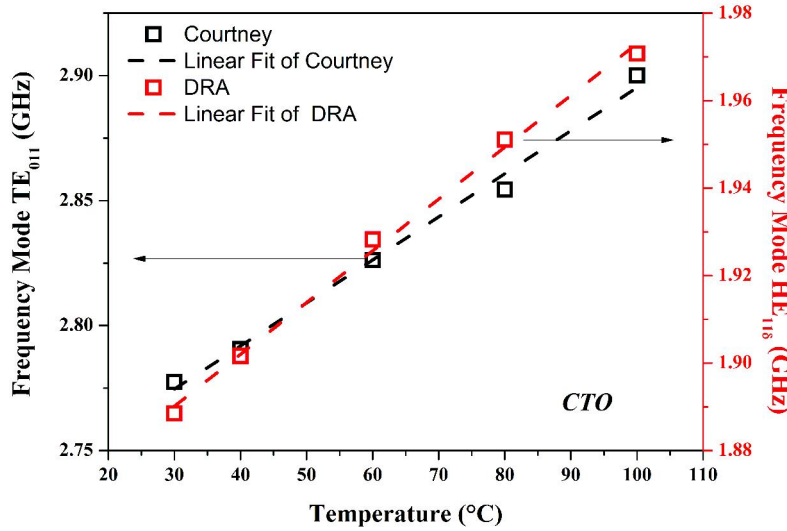


FIG. 4. Measurement of  $\tau_f$  for a DRA based on CTO:  $\square$  alternative method ( $HE_{11d}$ ) and  $\circ$  Courtney method ( $TE_{011}$ ).

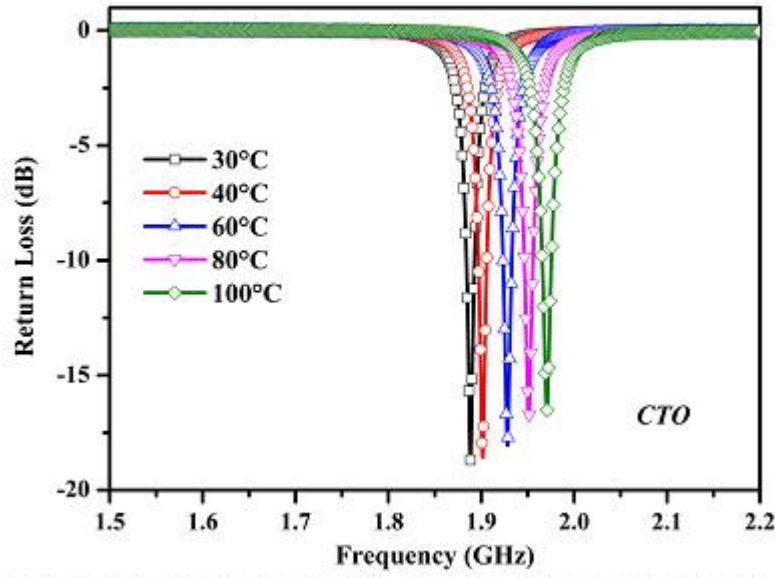


FIG. 5. Frequency variation of the  $HE_{11\delta}$  mode for DRA based on CTO with increasing temperature.

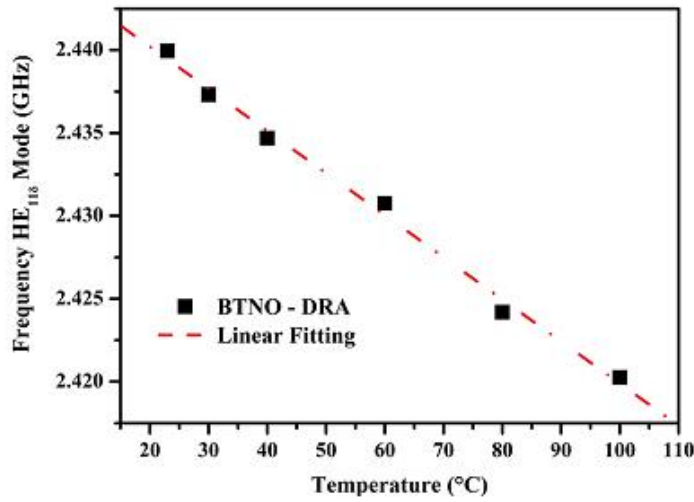


FIG. 9. Measurement of  $\tau_f$  for a DRA based on BTNO by the alternative method ( $HE_{11\delta}$ ).

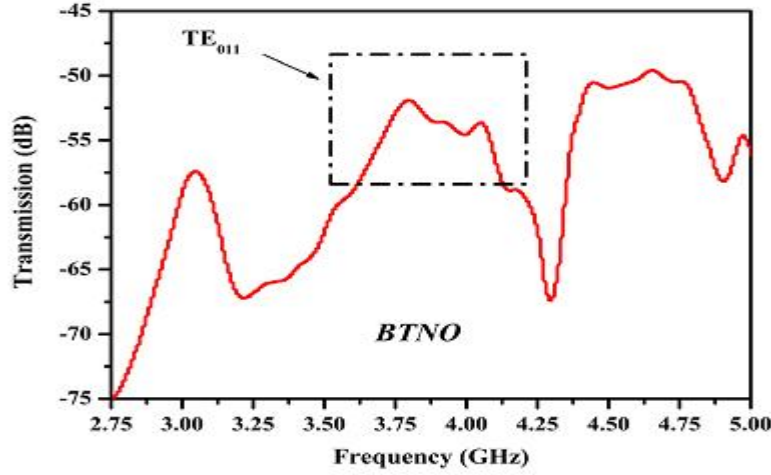


FIG. 7. Measurement of transmission by the Courtney method for the BTNO resonator.

TABLE II. Measurements of  $\tau_f$  of CTO,  $\text{Al}_2\text{O}_3$ , and BTNO from Courtney and DRA methods.

Ceramic	Method					
	Courtney method			Dielectric resonator antenna		
	$\tau_f$ (ppm $^{\circ}\text{C}^{-1}$ )	Error (%)	$\Delta f/\Delta T$ (Angular coefficient)	$\tau_f$ (ppm $^{\circ}\text{C}^{-1}$ )	Error (%)	$\Delta f/\Delta T$ (Angular coefficient)
$\text{CaTiO}_3$	621.16	0.108	$1.72 \times 10^{-3}$	624.32	0.088	$1.18 \times 10^{-3}$
$\text{Al}_2\text{O}_3$	-47.38	0.015	$-2.47 \times 10^{-4}$	-44.20	0.035	$-1.37 \times 10^{-4}$
BTNO	—	—	—	-104.19	0.021	$-2.54 \times 10^{-4}$

In conclusion a new experimental configuration to measure the temperature coefficient of resonant frequency ( $\tau_f$ ) in dielectric resonators was presented. The new experimental setup, to measure the  $\tau_f$  value, is based on the frequency variation with the temperature of the  $\text{HE}_{11d}$  mode of a DRA. The method is quite compatible with the measurement of  $\tau_f$  of the Courtney method. The obtained results by measuring the  $\tau_f$  value of CTO and  $\text{Al}_2\text{O}_3$ , in this proposed method, is presenting excellent agreement when compared to the traditional Courtney method. The dielectric loss is less affected in this method and this is the most important advantage that was obtained. In the tests, the  $\tau_f$  of the sample with higher loss ( $>10^{-2}$ ) was obtained. In this case, the  $\tau_f$  value for the BTNO resonator was  $-104.19 \text{ ppm}^{\circ}\text{C}^{-1}$ . The analysis of the temperature coefficient of resonant frequency ( $\tau_f$ ) in dielectric

resonators is an important property for the development of high frequency electronic devices, considering that this is a fundamental parameter, for the production of new components like filters, oscillators and antennas, with high thermal stability.

REF Journal of Applied Physics 112(7), 074106 (2012) (AIP)

M.A.S. Silva, T.S. M. Fernandes and A.S.B. Sombra

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#### **Supervision of PhD Thesis**

1-STUDY OF THE EFFECTS OF THE ADDITION OF LEAD AND BISMUTH IN THE DIELECTRIC PROPERTIES OF  $\text{BiNbO}_4$  CERAMIC MATRIX AND ITS APPLICATIONS IN RADIO FREQUENCY AND ANTENNAS, José Silva de Almeida Programa de Pós Graduação em Física da UFC (2011)

2-Study of thermal and structural stability of  $\text{Ca}(\text{Nb}_{12}\text{Y}_{12})\text{xTi}_{1-\text{x}}\text{O}_3$  Y Bi, Fe and Ca ( $\text{Nb}_{23}\text{Li}_{13}$ )  $\text{xTi}_{1-\text{x}}\text{O}_{3-\delta}$  and its use in dielectric resonator antennas (DRAs) Antonia Daniele Souza Bruno Costa, Programa de Pós Graduação em Engenharia de Teleinformática (2012)

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10- Ferrimagnetism and Ferroelectricity of the Composite Matrix:  $\text{SrBi}_2\text{Nb}_2\text{O}_9$  (SBN)- $\text{BaFe}_2\text{O}_6$  (BFO)100–X M. J. S. Rocha, M. C. C. Filho, K. R. B. Theophilo, J. C. Denardin, I. F. Vasconcelos, E. B. Araújo, A. S. B. Sombra *Materials Sciences and Applications*, 3(1) (2012) 6-17 (Scientific Research Publishing )  
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18- Experimental and numerical investigation of dielectric resonator antenna based on the BiFeO<sub>3</sub> ceramic matrix added with Bi<sub>2</sub>O<sub>3</sub> or PbO  
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**Changes in research objectives, if any: xxxxx**

**Change in AFOSR program manager, if any: xxxxx**

**Extensions granted or milestones slipped, if any: xxxxx**



**The study of layered magneto-dielectric composites structures is important for applications at higher frequencies where the use of metals is leading to higher loss. This kind of component based in a new configuration and using a new group of magneto-dielectric composites and blends is expected to present better bandwidth, low loss, high impedance matching that will open the possibility to be used in radars, communication devices, navigation equipments, and so on.**

**The use of special structures based in composites and blends is important for components operating at high frequencies.**

**In this work the magnetic and dielectric properties of ceramic-ceramic and ceramic-polymer composites with  $\text{BiNbO}_4$ ,  $\text{SrBi}_2\text{Nb}_2\text{O}_9$  (SBN),  $\text{BaBi}_4\text{Ti}_4\text{O}_{15}$  (BBT),  $\text{Na}_2\text{Nb}_4\text{O}_{11}$  (NNO),  $\text{Sr}_2\text{CoNbO}_6$  (SCN),  $\text{FeNbTiO}_6$ ,  $\text{BiFeO}_3$ ,  $\text{CaTi}_{1-x}(\text{Nb}_{1/2}\text{Fe}_{1/2})_x\text{O}_3$  and ferrites  $\text{BaFe}_{12}\text{O}_{19}$ ,  $\text{Ba}_2\text{Co}_2\text{Fe}_{12}\text{O}_{22}$  ( $\text{Co}_2\text{Y}$ ) and  $\text{Y}_3\text{Fe}_5\text{O}_{12}$  (YIG) was studied for RF and microwave applications.**

**The study of the thermal stability of magneto-dielectric composites is important for applications at the microwave band and in the millimeter and near millimeter region (100-300GHz) where the thermal stability of the resonators is fundamental.**

**In this presentation we will discuss**

**--- A study in the structural and microwave properties of the alloy matrix of  $\text{CaTi}_{1-x}(\text{Nb}_{1/2}\text{Fe}_{1/2})_x\text{O}_3$**

**--- Ferrimagnetism and Ferroelectricity of the Composite**

**Matrix:  $\text{SrBi}_2\text{Nb}_2\text{O}_9(\text{SBN})_x\text{-BaFe}_{12}\text{O}_{19}(\text{BFO})_{100-x}$**

**--- A new method to measure the microwave thermal stability coefficient  $\tau_f$  of materials**

# HIGH THERMAL STABILITY OF MICROWAVE DIELECTRIC PROPERTIES OF $\text{CaTi}_{1-x}(\text{Nb}_{1/2}\text{Fe}_{1/2})_x\text{O}_3$ CERAMICS

In this work, we studied and discussed the structural and microwave dielectric properties of the B-site modified calcium titanate ceramics. The compounds were prepared by a new procedure in the conventional solid-state method. They were properly studied, using X-ray diffraction (XRD), Raman Scattering spectroscopy, and microwave dielectric properties. Therefore, the refinement analysis of the XRD was presented and discussed.

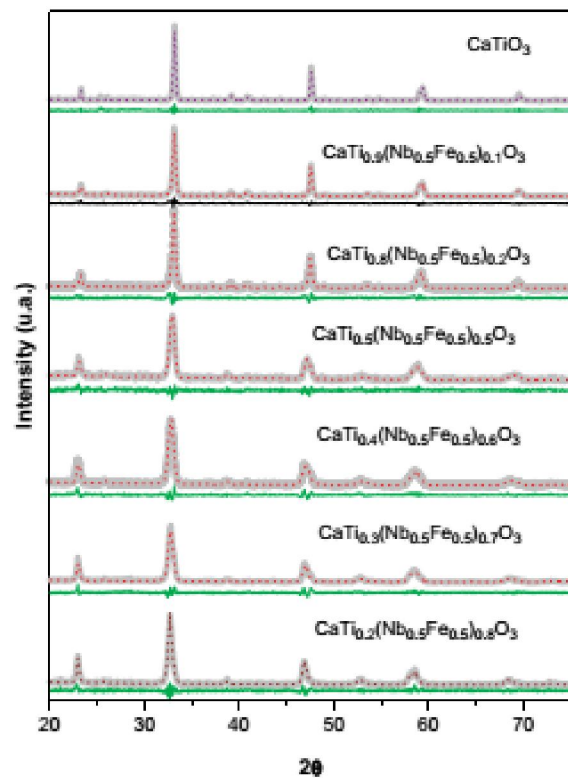


Fig. 1. XRD patterns of CTO and CNFTOX series. The dotted line is experimental pattern and the straight line is the fitted curve. The refined lattice parameters are shown below them.

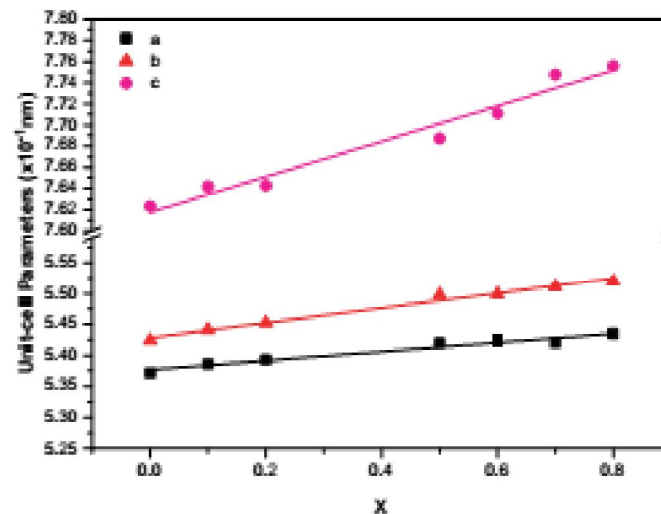
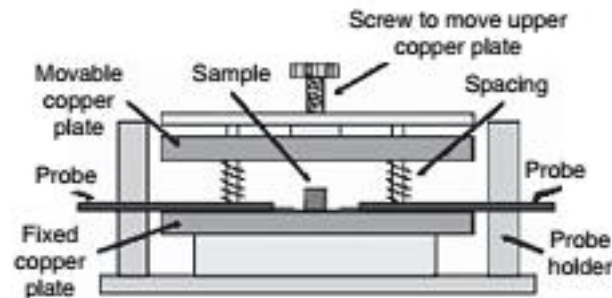


Fig. 2. Unit-cell Parameters ( $10^{-1}$  nm) for CNFTOX series.

Table 5. Microwave Dielectric Properties for samples ball-milled with ratio of 1 ball/g, calcinated at 900°C for 3 and 5 h, and sintered at 1100°C, for 3 h.

Sample	Calcination condition	$f_r$ (GHz)	$\epsilon_r$	$tg\delta$	$Q \times f$ (GHz)
CNFTO1	900°C/3h	4.451	30.42	$6.4 \times 10^{-3}$	681.14
CNFTO1	900°C/5h	3.619	58.00	$3 \times 10^{-3}$	1067.86
CNFTO2	900°C/3h	4.804	25.72	$3 \times 10^{-3}$	1535.70
CNFTO2	900°C/5h	4.365	38.83	$4 \times 10^{-3}$	979.81





**Figure 2.4** Schematic sketch of Courtney setup for measuring the dielectric constant under end shorted condition (after Ref. [12]).

Table 5. Microwave Dielectric Properties for samples ball-milled with ratio of 1 ball/g, calcinated at 900°C for 3 and 5 h, and sintered at 1100°C, for 3 h.

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Table 6. Microwave Dielectric Properties for samples calcinated at 900°C (for 5 h), and sintered at 1100°C (for 3 h).

Sample	Balls/Mass ratio	$f_r$ (GHz)	$\epsilon_r$	$tg\delta$	$Q \times f$ (GHz)
CNFTO1	1ball/g	3.619	58.00	$3.0 \times 10^{-3}$	1067.86
CNFTO1	2.4balls/g	2.937	78.11	$1.5 \times 10^{-3}$	1794.89
CNFTO2	1ball/g	4.365	38.83	$4.0 \times 10^{-3}$	979.81
CNFTO2	2.4balls/g	3.435	56.05	$9.4 \times 10^{-3}$	358.61

The temperature coefficient of resonant frequency ( $\tau_f$ ) measures, the variation of the resonance frequency of the dielectric resonator with temperature variation, as seen in below equation<sup>1,4</sup>

$$\tau_f = \frac{1}{f_i} * \frac{\Delta f}{\Delta T} * 10^6, \quad (1)$$

Table 7. Microwave Dielectric Properties for samples ball-milled with ratio of 2.4 balls/g, calcinated at 900°C (for 5 h) and sintered 1100°C, for 3 h.

Sample	$f_r$ (GHz)	$\epsilon_r$	$Tg\delta$	$\tau_f$ (ppm/°C)	$Q \times f$ (GHz)
CTO	2.659	101.33	$2.1 \times 10^{-3}$	1022.909	1266.19
CNFTO1	2.937	78.11	$2.3 \times 10^{-3}$	518.676	1275.65
CNFTO2	3.435	56.05	$9.4 \times 10^{-3}$	422.987	365.43
CNFTO3	3.964	40.66	$5.5 \times 10^{-3}$	264.635	720.73
CNFTO4	4.335	34.16	$5.1 \times 10^{-3}$	412.154	850.00
CNFTO5	4.889	26.52	$4.8 \times 10^{-3}$	58.478	1017.92
CNFTO6	4.831	28.35	$4.2 \times 10^{-3}$	2.866	1150.24
CNFTO7	5.280	24.60	$3.6 \times 10^{-3}$	-32.574	1466.94
CNFTO8	5.381	22.62	$4.6 \times 10^{-3}$	-44.744	1169.78
CNFTO9	5.771	22.18	$1.0 \times 10^{-3}$	-71.318	577.10
CNFO	5.723	21.28	$4.8 \times 10^{-3}$	-88.231	1192.29

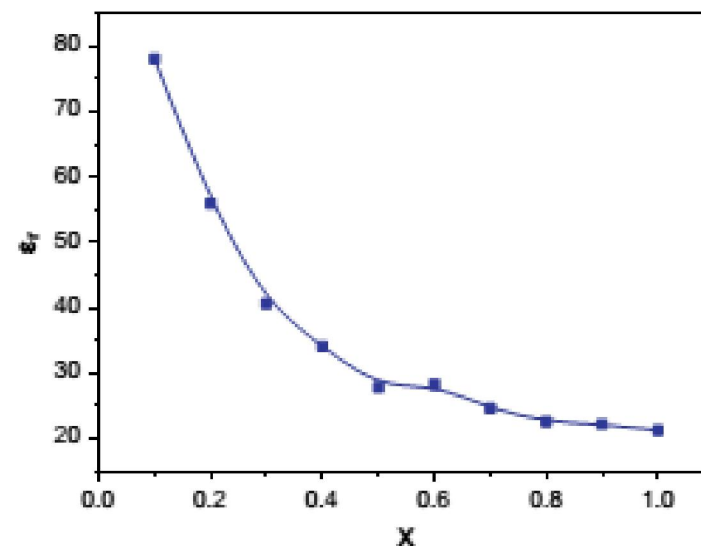


Fig. 4. Dielectric permittivity of CNFTOX ( $0 \leq x \leq 1$ ) for ball milled samples with ball/mass ratio of 2.4, calcinated at 900°C (for 5 h), and sintered at 1100°C (for 3 h).

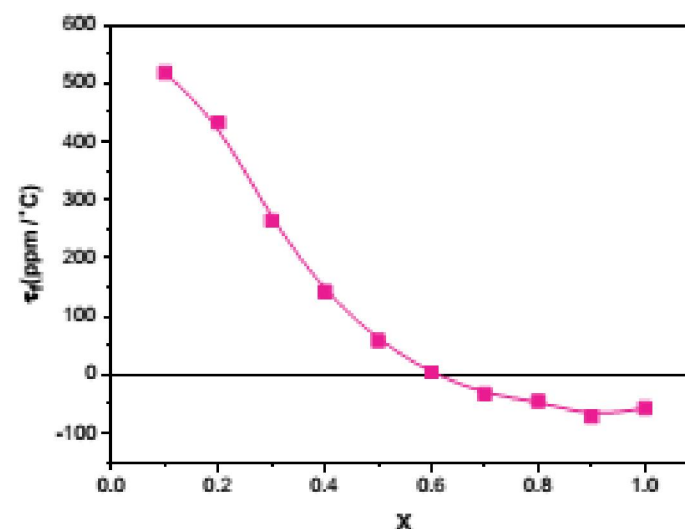


Fig. 6.  $\tau_f$  of CNFTOX ( $0 \leq x \leq 1$ ) for ball milled sample with ball/mass ratio of 2.4, calcinated at 900°C (for 5 h), and sintered at 1100°C (for 3 h).

Results showed that the samples belong to the Pbnm spatial group.

The microwave dielectric properties of the  $\text{Ca}[(\text{Fe}_{1/2}\text{Nb}_{1/2})_x\text{Ti}_{1-x}]\text{O}_3$  for ball-milled samples (with ratios of 1 and 2.4 balls/g), calcinated at  $900^\circ\text{C}$  (with different time of exposure – 3 and 5 h), and sintered at  $1100^\circ\text{C}$  (for 3h) were investigated.

Dielectric permittivity values in the range of 20 to 80 were obtained.

Regarding the studied samples, the quality factor values increased with the decrease of the titanium substitution in the region from  $x = 0.2$  to  $0.7$ . Considering the increase of the  $x$  value (titanium substitution), we observe the decrease of the temperature coefficient of resonant frequency ( $\tau_f$ ). The CNFTO has excellent microwave properties at  $x = 0.6$ , with a temperature coefficient of resonant frequency ( $\tau_f$ ) almost zero ( $\tau_f = 2.8 \text{ ppm}/^\circ\text{C}$ ). At  $x = 0.7$ , the  $\tau_f$  values became negative and  $Q.f$  decreases.

# Ferrimagnetism and Ferroelectricity of the Composite Matrix: $\text{SrBi}_2\text{Nb}_2\text{O}_9$ (SBN) $_x$ - $\text{BaFe}_{12}\text{O}_{19}$ (BFO) $_{100-x}$

In this paper a study of the magnetic and dielectric properties of composites based on M-type barium hexaferrite BFO ( $\text{BaFe}_{12}\text{O}_{19}$ ) and SBN ( $\text{SrBi}_2\text{Nb}_2\text{O}_9$ ) is presented. The magneto-dielectric matrix composite  $(\text{SrBi}_2\text{Nb}_2\text{O}_9)_x$  ( $\text{BaFe}_{12}\text{O}_{19}$ ) $_{100-x}$ , ( $x = 0, 25, 50, 75$  and  $100$  wt%) were prepared by a new procedure using the solid state reaction method.

In this work, our main goal is to develop a dielectric material that is able to respond to both electric and magnetic stimulus, i.e. that is ferroelectric and ferromagnetic.

To do so, we use the Aurivillius ceramic  $\text{SrBi}_2\text{Nb}_2\text{O}_9$  and the Hexaferrite  $\text{BaFe}_{12}\text{O}_{19}$ . Such a material could be applied in the same way that common dielectrics (as dielectric resonator antennas, for example) but opening a wide range of possibilities to make the application of ceramics to electronic devices, memories and telecommunications more useful and powerful.



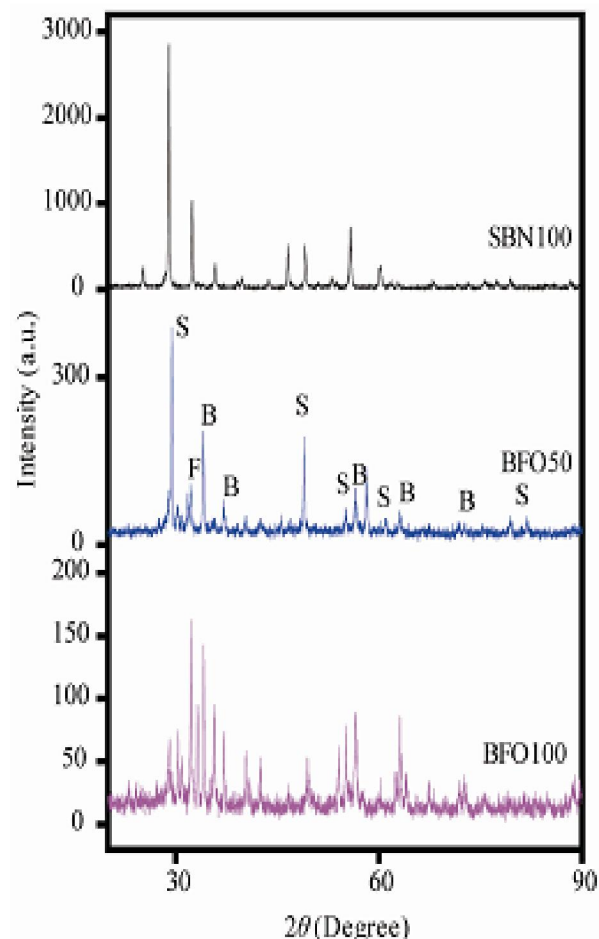


Figure 1. X-Ray diffractograms for SBN100, BFO50, BFO100 samples.

Table 1. Rietveld refinement parameters.

Sample	SBN 100	BFO100
a (nm)	0.5515	0.5868
b(nm)	0.5513	0.5868
C(nm)	2.5024	2.3106
Density (g/cm <sup>3</sup> )	7.293	5.358
Volume (nm <sup>3</sup> )	0.761062	0.689074
R <sub>p</sub>	10.74%	27.43%
R <sub>wp</sub>	14.7%	34.96%
R <sub>exp</sub>	11.6%	22.73%
S	1.27	1.54

Table 2. Relative density of the samples obtained from the Archimedes method.

Binder	Sample	Relative Density
TEOS	BFO100 T	93.83%
	BFO75 T	83.09%
	BFO50 T	91.88%
	BFO25 T	87.19%
	SBN100 T	82.70%
	BFO100 P	83.09%
PVA	BFO75 P	80.77%
	BFO50 P	85.60%
	BFO25 P	78.11%
	SBN100 P	67.92%
Glycerin	BFO100 G	66.04%
	BFO75 G	81.02%
	BFO50 G	83.61%
	BFO25 G	83.32%
	SBN100 G	80.30%

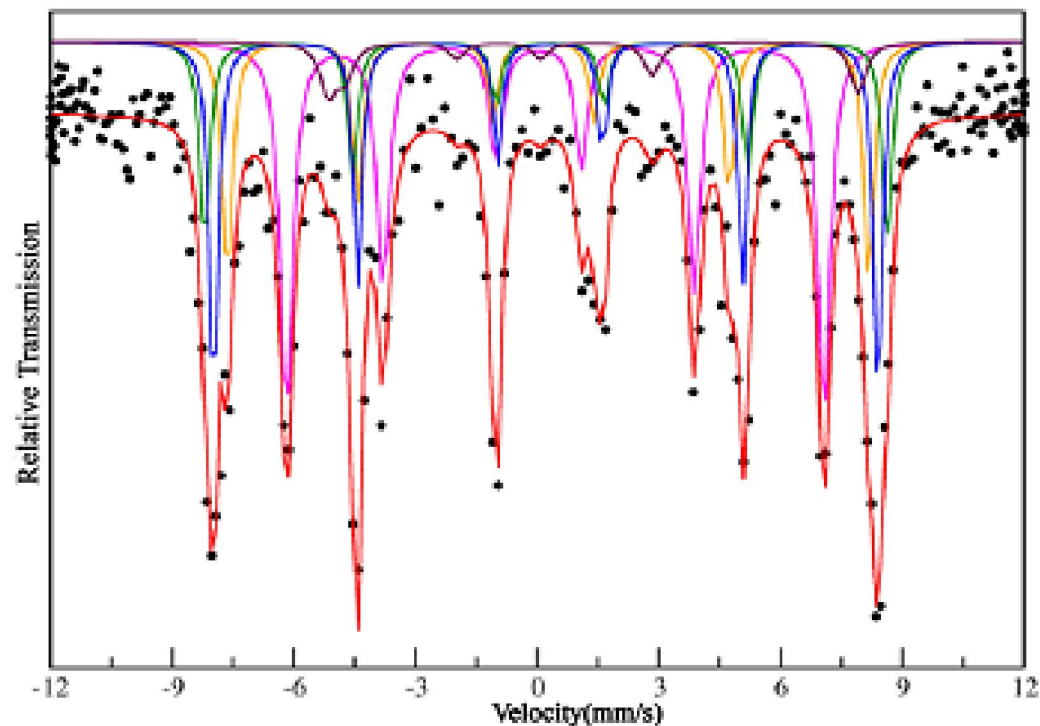


Figure 4. Mössbauer Spectrum for the BFO100 sample.

Table 3. Hyperfine parameters of the Mössbauer measurements.

Sample	Sites	Coordination	IS (mm/s)	QS (mm/s)	$H_{\text{eff}}$ (T)	$R_A$ (%)
BFO100	12k	octahedral	0.351	0.401	41.09	36%
	4f1	tetrahedral	0.334	0.097	49.00	18%
	4f2	octahedral	0.372	-0.099	52.16	15%
	2a	octahedral	0.371	-0.098	50.79	24%
	2b	trigonal bipyramidal	0.326	2.310	40.65	6%



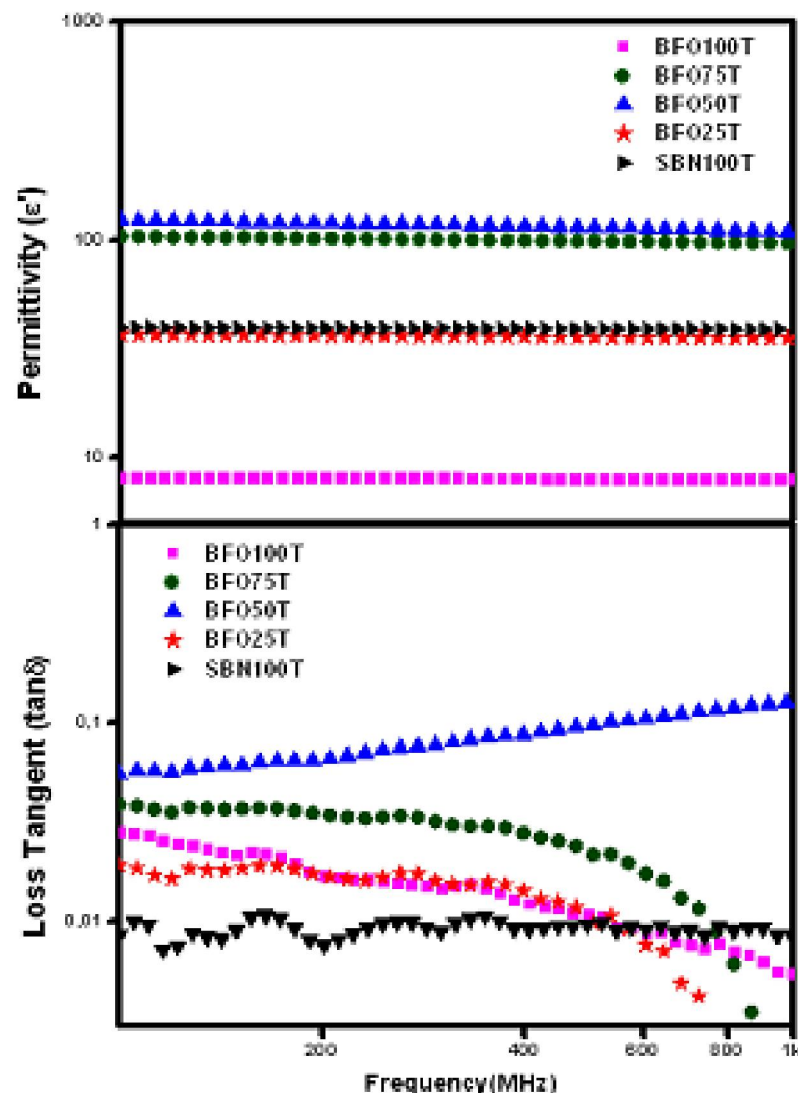


Figure 6. RF measurements of (a) Permittivity and (b) Loss tangent for TEOS samples.

Table 4. Values of permittivity and loss in the RF range.

Samples	100 MHz		500 MHz		1 GHz	
	$\epsilon'$	$\tan \delta$	$\epsilon'$	$\tan \delta$	$\epsilon'$	$\tan \delta$
BFO 100 G	12.38	0.0072	12.58	0.02	13.15	0.040
BFO 75 G	23.17	0.0374	22.80	0.023	23.56	0.032
BFO 50 G	69.75	0.167	48.71	0.212	45.340	0.235
BFO 25 G	36.12	0.004	36.84	0.117	41.81	0.202
SBN 100 G	35.46	0.048	37.51	0.246	43.03	0.428
BFO 100 P	13.19	0.0061	13.43	0.036	14.13	0.050
BFO 75 P	77.04	0.0484	78.03	0.068	96.91	0.117
BFO 50 P	33.10	0.0854	31.07	0.0217	33.15	0.00052
BFO 25 P	42.42	0.0115	44.02	0.174	51.70	0.308
SBN 100 P	27.89	0.0037	28.03	0.0027	28.90	0.0007
BFO 100 T	8.04	0.0055	8.05	0.002	8.10	0.003
BFO 75 T	96.68	0.0046	104.8	0.238	143.3	0.490
BFO 50 T	108.94	0.128	91.44	0.231	88.94	0.282
BFO 25 T	35.35	0.0055	36.47	0.146	42.16	0.255
SBN 100 T	39.05	0.0086	39.19	0.008	41.18	0.006

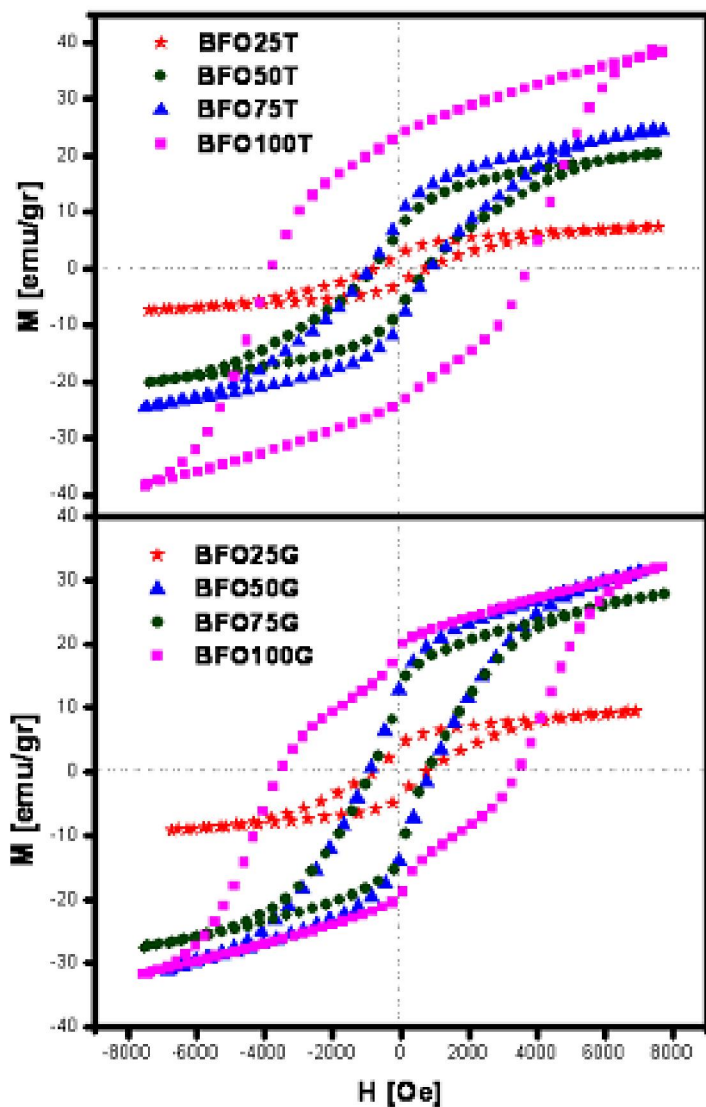


Figure 7. Magnetic hysteresis loops for (a) TEOS and (b) Glycerin samples.

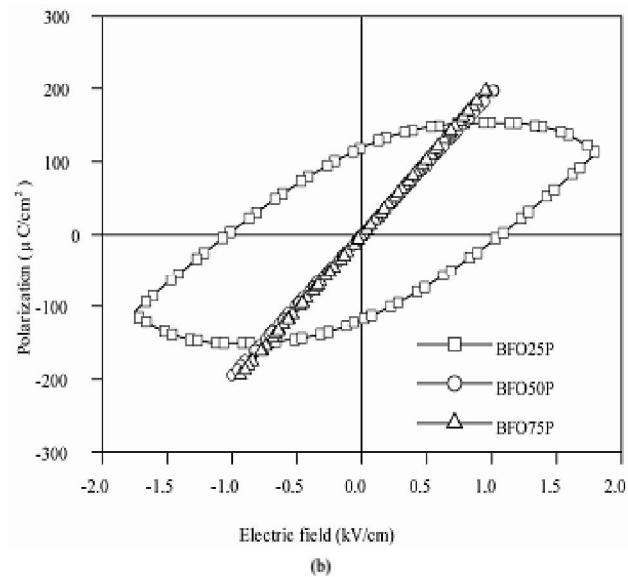
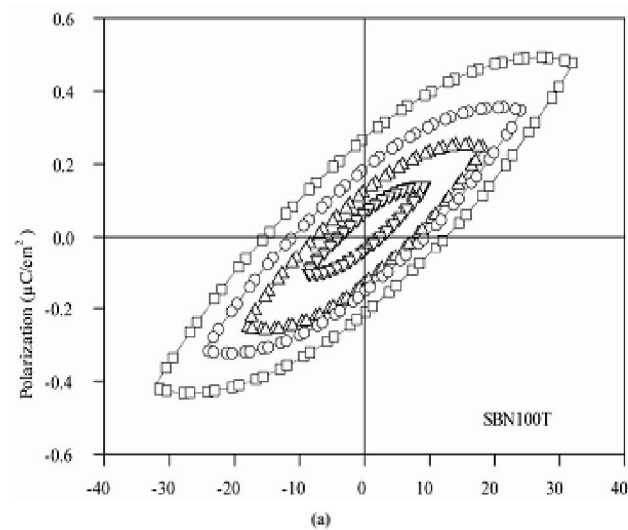


Figure 8. Electric hysteresis loops recorded at 1 Hz frequency for (a) SBN100T sample and (b) BFO25P, BFO50P and BFO75P composites.



**Table 5. Magnetic Hysteresis parameters of the samples.**

Samples	Remanent Magnetization (emu/g)	Coercive Field (Oe)	Saturation Magnetization (emu/g)	Maximum Field (Oe)
<b>BFO 100 T</b>	23.98	3744.4	34.97	7680
<b>BFO 75 T</b>	10.07	932.8	20.39	7676.4
<b>BFO 50 T</b>	7.60	813.6	17.39	7539
<b>BFO 25 T</b>	2.34	725	5.99	7388
<b>BFO 100 P</b>	21.13	3699.4	31.33	7578
<b>BFO 75 P</b>	15.84	1110	27.34	7418.4
<b>BFO 50 P</b>	8.73	633.8	18.13	7629
<b>BFO50 P</b>	3.76	923	6.46	7648.6
<b>BFO 100 G</b>	19.99	3460.4	28.90	7692.8
<b>BFO 75 G</b>	13.86	833	26.24	7731.6
<b>BFO 50 G</b>	14.10	868	22.31	7010.4
<b>BFO 25 G</b>	4.01	747.4	7.33	6888.2

## Conclusions

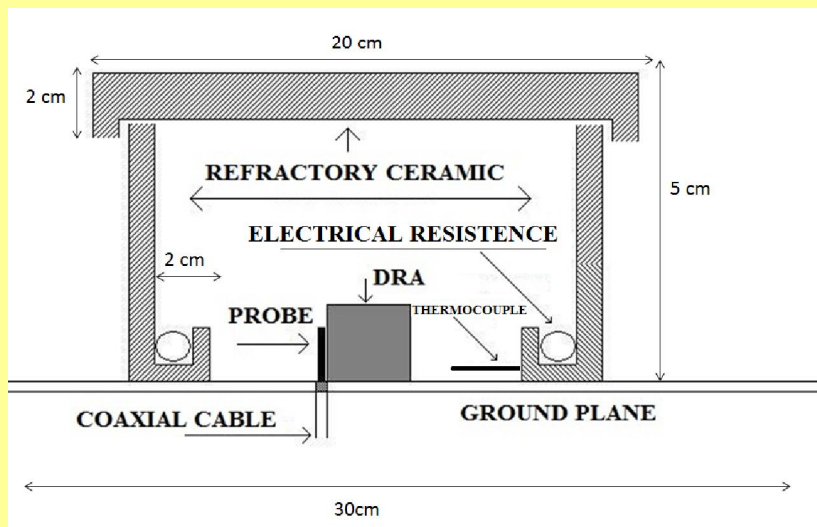
A complex behavior was observed for the loss tangent over the radio frequency range, which means that less lossy samples could not help keeping this characteristic over entire frequency range. The magnetic hysteresis loops showed that composite samples preserve the ferrimagnetism for hexaferrite when SBN is added to the composite, although they become less coercive. For electric hysteresis the density of the samples are not high enough to define the true behavior of ferroelectricity in composite samples.

For further works, the properties over microwave frequency range, thermal influences on the dielectric properties will be investigated for possible applications of the composite.

# A NEW METHOD FOR THE MEASUREMENT OF THE MICROWAVE TEMPERATURE COEFFICIENT OF RESONANT FREQUENCY ( $\tau_f$ ).

A.S.B.Sombra, Federal University of Ceará – *BRAZIL*

The study of the thermal stability of magneto-dielectric composites is important for applications at the microwave band and in the millimeter and near millimeter region (100-300GHz) where the thermal stability of the resonators is fundamental.



Modified setup, for the measurement of  $\tau_f$

## Objectives and Approach

A new method to measure the microwave thermal stability coefficient  $\tau_f$

$$\tau_f = \frac{1}{f_i} * \frac{\Delta f}{\Delta T} * 10^6,$$

TABLE I.  $TE_{01\delta}$ ,  $HE_{11\delta}$  e  $TM_{01\delta}$  modes and dielectric parameters of CTO,  $Al_2O_3$ , and BTNO dielectrics.

	CaTiO <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	BTNO
a (mm)	7.48	12.70	7.31
h (mm)	8.04	12.70	7.38
a(mm)/h(mm)	0.93	1	0.99
$\epsilon_R$	92.25	9.80	63.68
$\tan \delta$	$5.81 \times 10^{-4}$	$1.11 \times 10^{-4}$	$5.61 \times 10^{-2}$
$f_{\text{monopole}}$ (GHz) measured	1.888	3.089	2.439
$f_{HE_{11\delta}}$ (GHz) calculated	1.837	3.147	2.328
$f_{TE_{01\delta}}$ (GHz) calculated	1.830	3.201	2.288
$f_{TM_{01\delta}}$ (GHz) calculated	2.695	4.527	3.357

Used samples in the measurements

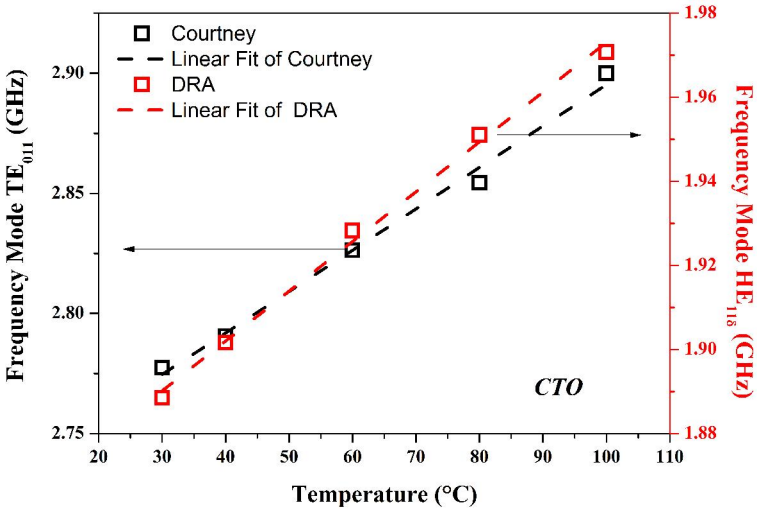


FIG. 4. Measurement of  $\tau_f$  for a DRA based on CTO:  $\square$  alternative method ( $HE_{116}$ ) and  $\circ$  Courtney method ( $TE_{011}$ ).

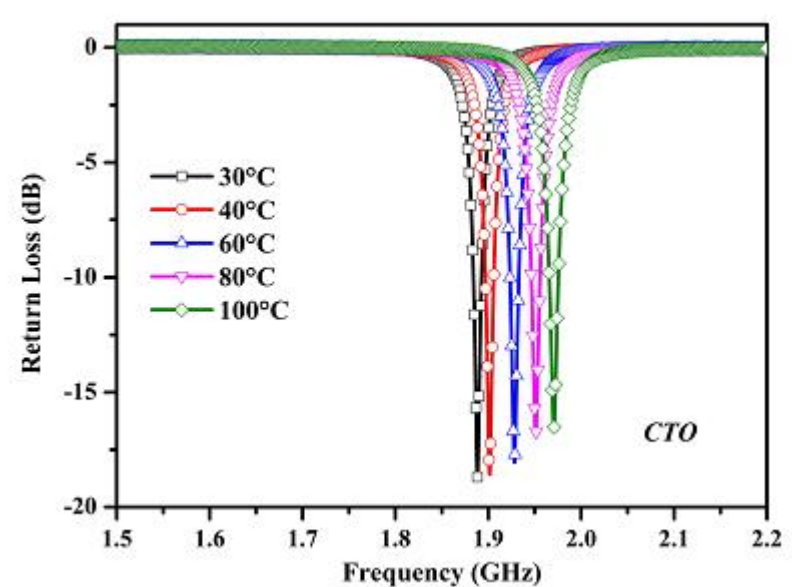


FIG. 5. Frequency variation of the  $HE_{116}$  mode for DRA based on CTO with increasing temperature.

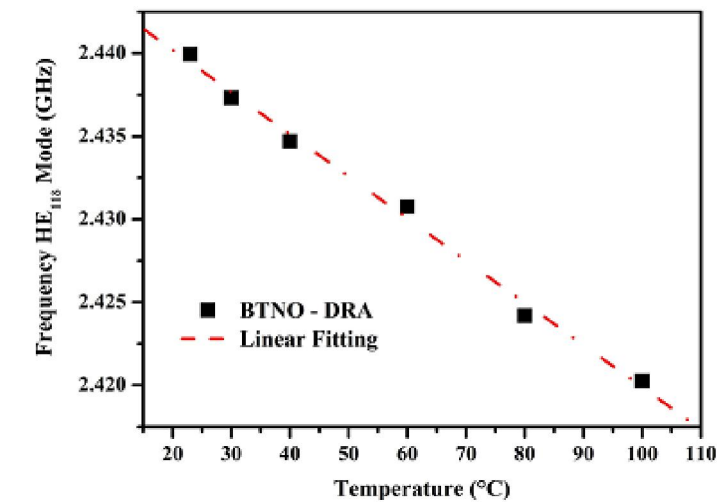


FIG. 9. Measurement of  $\tau_f$  for a DRA based on BTNO by the alternative method ( $HE_{116}$ ).

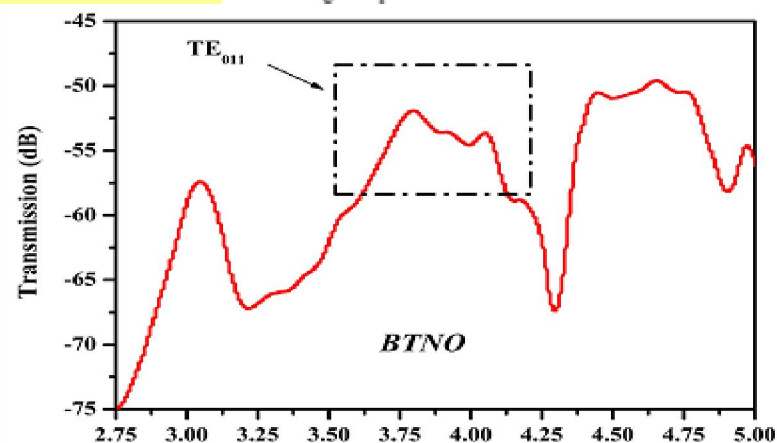


FIG. 7. Measurement of transmission by the Courtney method for the BTNO resonator.



TABLE II. Measurements of  $\tau_f$  of CTO,  $\text{Al}_2\text{O}_3$ , and BTNO from Courtney and DRA methods.

	Method					
	Courtney method			Dielectric resonator antenna		
	$\tau_f$ (ppm °C <sup>-1</sup> )	Error (%)	$\Delta f/\Delta T$ (Angular coefficient)	$\tau_f$ (ppm °C <sup>-1</sup> )	Error (%)	$\Delta f/\Delta T$ (Angular coefficient)
Ceramic						
CaTiO <sub>3</sub>	621.16	0.108	$1.72 \times 10^{-3}$	624.32	0.088	$1.18 \times 10^{-3}$
Al <sub>2</sub> O <sub>3</sub>	-47.38	0.015	$-2.47 \times 10^{-4}$	-44.20	0.035	$-1.37 \times 10^{-4}$
BTNO	—	—	—	-104.19	0.021	$-2.54 \times 10^{-4}$

In this work a new experimental configuration to measure the temperature coefficient of resonant frequency ( $\tau_f$ ) in dielectric resonators was presented. The new experimental setup, to measure the  $\tau_f$  value, is based on the frequency variation with the temperature of the HE<sub>11</sub>dmode of a DRA. The method is quite compatible with the measurement of  $\tau_f$  of the Courtney method. The obtained results by measuring the  $\tau_f$  value of CTO and Al<sub>2</sub>O<sub>3</sub>, in this proposed method, is presenting excellent agreement when compared to the traditional Courtney method. The dielectric loss is less affected in this method and this is the most important advantage that was obtained. In the tests, the  $\tau_f$  of the sample with higher loss ( $>10^{-2}$ ) was obtained. In this case, the  $\tau_f$  value for the BTNO resonator was -104.19 ppm. C<sup>-1</sup>. The analysis of the temperature coefficient of resonant frequency ( $\tau_f$ ) in dielectric resonators is an important property for the development of high frequency electronic devices, considering that this is a fundamental parameter, for the production of new components like filters, oscillators and antennas, with high thermal stability.

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M.A.S. Silva, T.S. M. Fernandes and A.S.B. Sombra

doi:10.1063/1.4755799

# AFOSR Deliverables Submission Survey

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The full title of the funded effort.

ELECTRICAL AND STRUCTURAL PROPERTIES STUDY OF LAYERED DIELECTRIC AND MAGNETIC COMPOSITES AND BLENDS STRUCTURES FOR RF AND MICROWAVE APPLICATIONS

## Grant/Contract Number

AFOSR assigned control number. It must begin with "FA9550" or "F49620" or "FA2386".

FA9550-11-1-0095

## Principal Investigator Name

The full name of the principal investigator on the grant or contract.

Antonio sergio Bezerra Sombra

## Program Manager

The AFOSR Program Manager currently assigned to the award

James Fillerup

## Reporting Period Start Date

05/15/2011

## Reporting Period End Date

06/05/2014

## Abstract

In this work the magnetic and dielectric properties of ceramic-ceramic and ceramic-polymer composites with BiNbO<sub>4</sub>, SrBi<sub>2</sub>Nb<sub>2</sub>O<sub>9</sub> (SBN), BaBi<sub>4</sub>Ti<sub>4</sub>O<sub>15</sub> (BBT), Na<sub>2</sub>Nb<sub>4</sub>O<sub>11</sub>(NNO), Sr<sub>2</sub>CoNbO<sub>6</sub> (SCN) and ferrites BaFe<sub>12</sub>O<sub>19</sub> and Y<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub> (YIG) was studied for RF and microwave applications. New configurations of magneto-dielectric composites and blends structures for high frequency applications was done. The 0-3 type dielectric and magnetic composites with homogenously distributed ceramic inclusions was fabricated in a polymer matrix. Magnetic Yttrium Iron Garnet (YIG) and (SBN) powders were used to enhance the permittivity and permeability of the composites. This group of dielectric and magnetic phases was studied in the RF and microwave region. The microstructure, high frequency dielectric and magnetic properties of individual layers and 2-2 composites was investigated and measured.

A new method for the measurement of the temperature coefficient of resonant frequency ( $\tau_f$ ), is presented. The traditional method (based on the Courtney method) present some limitations of measuring the values of  $\tau_f$ , for samples with high dielectric loss due to their inability to observe clearly the TE<sub>011</sub> mode. The new experimental setup, to measure the  $\tau_f$  value, is based on the variation of the temperature of the dominant mode of a dielectric resonator antenna (DRA).

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#### Archival Publications (published) during reporting period:

- 1- BiFeO<sub>3</sub> CERAMIC MATRIX WITH Bi<sub>2</sub>O<sub>3</sub> OR PbO ADDED: MÖSSBAUER, RAMAN AND DIELECTRIC SPECTROSCOPY STUDIES H. O. Rodrigues, G. F. M. Pires Junior, A. J. M. Sales, P. M. O. Silva, B. F. O. Costa, P. Alcantara Jr, S. G. C. Moreira and A. S. B. Sombra Physica B 406(13)(2011)2532-2539(Elsevier) doi: 10.1016/j.physb.2011.03.050
- 2- Impedance and Modulus Studies of Magnetic Ceramic Oxide Ba<sub>2</sub>Co<sub>2</sub>Fe<sub>12</sub>O<sub>22</sub> (Co<sub>2</sub>Y) doped with Bi<sub>2</sub>O<sub>3</sub> M. M Costa, G. F. M. Pires Júnior, A. J. Terezo, M. P. F. Graça and A. S. B. Sombra Journal of Applied Physics 110(3), 034107 (2011) AIP doi: 10.1063/1.3615935
- 3 - Study of the temperature and organic bindings effects in the dielectric and structural properties of the lithium ferrite ceramic matrix (LiFe<sub>5</sub>O<sub>8</sub>) M. M. Costa, R. S. T. M. Sohn, A. A. M. Macêdo, S. E. Mazzetto, M. P. F. Graça, A. S. B. Sombra Journal of Alloys and Compounds, 509(39)(2011)9466-9471(Elsevier) doi: 10.1016/j.jallcom.2011.07.038
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- 6- HIGH THERMAL STABILITY OF MICROWAVE DIELECTRIC PROPERTIES OF CaTi<sub>1-x</sub>(Nb<sup>2/3</sup>Li<sup>1/3</sup>)XO<sub>3-δ</sub> (CNLTO) ALLOYS A. D. S. Bruno Costa, D. G. Sousa, R. C. S. Costa, F. W. de O. Amarante, T. S. M. Fernandes, G. D. Saraiva, M. A. S. da Silva, and A. S. B. Sombra Physica Scripta 84 (2011) 055701-055707 (IOP) doi: 10.1088/0031-8949/84/05/055701
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- 8- Morphological, structural, optical and dielectric properties of 91SiO<sub>2</sub>:4Li<sub>2</sub>O:4Nb<sub>2</sub>O<sub>5</sub>:1Dy<sub>2</sub>O<sub>3</sub> (% mole) glass prepared by sol-gel M.A. Valente, M. Peres, C. Nico , T. Monteiro, M.P.F. Graça, A.S.B. Sombra, C.C. Silva  
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H.O. Rodrigues, A.J.M. Sales, G.F.M. Pires Junior , J.S. Almeida, M.A.S. Silva , A.S.B. Sombra Journal of Alloys and Compounds 576 (2013) 324–331 ELSEVIER  
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Changes in research objectives (if any):

Change in AFOSR Program Manager, if any:

Extensions granted or milestones slipped, if any:

AFOSR LRIR Number

LRIR Title

Reporting Period

Laboratory Task Manager

Program Officer

Research Objectives

Technical Summary

Funding Summary by Cost Category (by FY, \$K)

	Starting FY	FY+1	FY+2
Non-Military Government Personnel Costs			
In-house Contractor Costs			
Travel (Be Specific)			
Training (Be Specific)			
Supplies			
Other Expenses (Be Specific)			
Total Resource Requirements			

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