MICROSCOPIC OBSERVATION OF THE TEMPERATURE COEFFICIENT DISTRIBUTION OF DIELECTRIC MATERIAL FOR MICROWAVE APPLICATION USING SCANNING PHOTOTHERMAL DIELECTRIC MICROSCOPE

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ABSTRACT

Two dimensional images of the temperature coefficient of the distribution of the dielectric constant of a two phases composite ceramics composed of TiO_2 and $Bi_2Ti_4O_{11}$ are observed using scanning photothermal dielectric microscope. The obtained images clearly show that the each grain of TiO_2 and $Bi_2Ti_4O_{11}$ in the ceramics has a negative and a positive temperature coefficient, respectively and that the macroscopic averaged temperature coefficient of the ceramics is relatively low due to the cancellation of the coefficients with the opposite signs.

INTRODUCTION

Numerous dielectric materials have been developed with the progress of microwave telecommunication and satellite broadcasting, and they have various dielectric properties according to the respective microwave application. Among such properties to be considered in designing a dielectric material, one of the most important and general properties is temperature characteristics of dielectric constant. Normally, temperature coefficients of dielectric constant have been determined from the temperature response of the resonant frequency using the resonant cavity method,[1] so that the obtained coefficient by this method has the macroscopic average value of the coefficients distributing in the dielectric material. However, it is easily understood that microscopic measurement for the distribution of the temperature coefficients of dielectric constant provides more precise information for designing the material than that obtained by the macroscopic measurement. Especially in the composite ceramics composed of the grains with different dielectric temperature coefficients (for example, a positive and a negative coefficients) for the temperature compensation, such microscopic technique for the distribution of the temperature coefficient of dielectric constant is very effective to the precise characterization of the material.

On the other hand, recently, we have developed a new photothermal techniques for the thermal properties of dielectric materials and for the microscopic distribution of the temperature coefficients of the dielectric constants by using photothermal dielectric (PTD) effect.[2] This is based on the temperature characteristic of the dielectric constant of light-irradiated material. When chopped light is absorbed in a dielectric material, an alternating variation of capacitance is caused by the heat produced due to light absorption and is detected with sufficient dynamic range and sensitivity by using a frequency demodulation technique. Since the principle is based on the temperature characteristic of the dielectric constant, the measurement of the microscopic distribution of temperature coefficient of the dielectric constant for dielectric materials is possible.

To demonstrate the usefulness of this technique for the area distribution of temperature coefficient of dielectric constants, we measured the two dimensional image of the two phases composite ceramics composed of TiO_2 and $Bi_2Ti_4O_{11}$ which have a positive and a negative dielectric temperature coefficient, respectively.

MEASUREMENT SYSTEM

To measure the capacitance variation with chopped light with a frequency fc, we used the coaxial cavity resonator with the capacitor C made of the material to be measured, as shown in Fig.1. This probe has the resonance frequency of 1.3GHz. As the front electrode

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of the capacitor, we used a Cr film with a thickness of 200nm. This electrode is completely opaque so that the incident light can not penetrate the electrode. The thickness of this Cr electrode must be sufficiently less than its thermal diffusion length (12.7μm at fc=40KHz), so we can obtain the capacitor with a surface heat source facing it. The localized alternating temperature change due to the absorption of the focused chopped light causes the localized alternating variation of the capacitance because of its temperature characteristics.

In the one-dimensional analysis for the thermally thick material, the alternating capacitance variation C_{ac} is given by [2]

$$C_{ac}=C(T_0)\frac{\varepsilon'(T_0)}{\varepsilon(T_0)}\frac{I_0}{2l\rho_s C_s(1+g)}\frac{1}{\omega_c}\cos\left(\omega_c t-\frac{\pi}{2}\right) \quad (1)$$

with

$$g=\left(\frac{k_g\rho_g C_g}{k_s\rho_s C_s}\right)^{\frac{1}{2}}$$

where $\varepsilon(T_0)$, $\varepsilon'(T_0)$ and $C(T_0)$ are the dielectric constant, its first-order temperature coefficient and the capacitance at room temperature T_0. ρ_s, C_s and k_s are the density, the specific heat and the thermal conductivity of the specimen, respectively and ρ_g, C_g and k_g are the corresponding parameters of the air. l, ω_c and I_0 show the thickness of the specimen, the chopping angular frequency of the incident light beam and the light intensity, respectively.

A schematic diagram of the system is shown in Fig.2. The probe, combined with a translation stage, is the basis of a scanning photothermal dielectric microscope system. It measures the microscopic area distribution of temperature coefficient of materials. The probe is connected to the oscillator tuned to the resonance frequency of the probe, so that the oscillating frequency is modulated by the change of capacitance. As a result, a frequency modulation (FM) signal comes from this oscillator. By detecting this FM signal using FM demodulator and lock-in amplifier, we obtain a voltage signal proportional to the capacitance variation.

In this study, we used a diode laser with the power of 5.3mW and the wavelength of λ=785nm. The beam diameter of the focused light was less than a few micron. Thus, we can obtain the two dimensional image of the distribution of the dielectric temperature coefficient by scanning the focused light source.

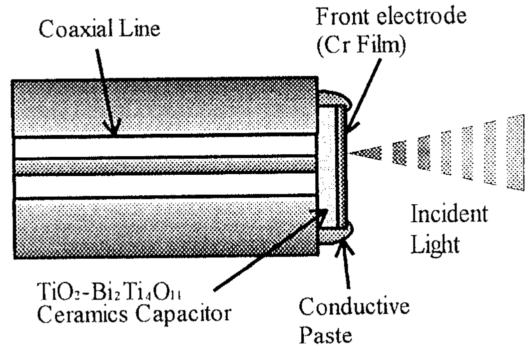

Fig.1 Coaxial cavity resonator for the probe of scanning photothermal dielectric microscope and the capacitance sample made of the TiO_2-$Bi_2Ti_4O_{11}$ ceramics.

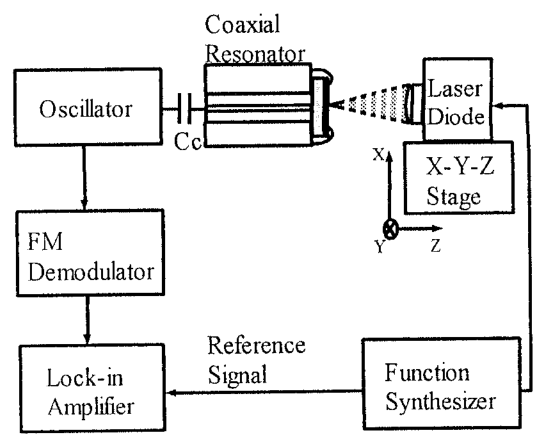

Fig.2 Schematic diagram of the scanning photothermal dielectric microscope.

MICROSTRUCTURE OF TiO_2-$Bi_2Ti_4O_{11}$ CERAMICS

Before describing the measurement of two dimensional images of the temperature coefficient distribution, we will mention about the specimen. The backscattering electron image (SEM image) of this ceramics is shown in Fig.3. In this figure, the grain shown by the black part is composed of only TiO_2 and the white part is composed of only $Bi_2Ti_4O_{11}$. Table **I** summarizes the compositions and the microwave characteristics of the binary-system TiO_2-Bi_2O_3 ceramics. TiO_2 has a large

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negative temperature coefficient of dielectric constant of τ_ε=-876ppm/°C, whereas $Bi_2Ti_4O_{11}$ has a large positive coefficient of τ_ε=+1034ppm/°C . The macroscopic average value of temperature coefficient of this ceramics is τ_ε=-34ppm/°C because of the cancellation of the coefficients with the opposite signs.

Table I Microwave characteristics of TiO_2-$Bi_2Ti_4O_{11}$ ceramics.

Composition(mol %)		Chemical formula	Dielectric properties		
TiO_2	Bi_2O_3		ε_r	Q(at5GHz)	τ_ε (ppm/°C)
100	0	TiO_2	105.0	9200	-879
91.9	8.1	TiO_2-$Bi_2Ti_4O_{11}$	80.0	1800	-34
80.0	20.0	$Bi_2Ti_4O_{11}$	53.2	900	+1034

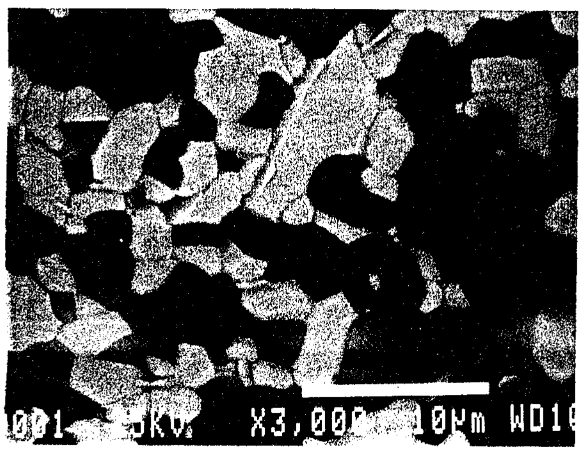

Fig.3 Backscattering electron image of the grains of TiO_2-$Bi_2Ti_4O_{11}$ ceramics sample.

MICROSCOPIC OBSERVATION OF THE TEMPERATURE COEFFICIENT DISTRIBUTION

Using the scanning photothermal dielectric microscope system, we measured the two dimensional images of dielectric constant of the above mentioned two phases composite ceramics composed of TiO_2 and $Bi_2Ti_4O_{11}$. In the measurement for the two dimensional image of temperature coefficient of dielectric constant, we chose the light chopping frequency of fc=40, 4 and 0.4KHz. The results are shown in Fig.4. The clear temperature coefficient image corresponding to the two different grains was obtained only at the light chopping frequency of fc=40KHz and the image becomes ambiguous and homogeneous according to the decrease of the light chopping frequency. This can be explained qualitatively as follows.

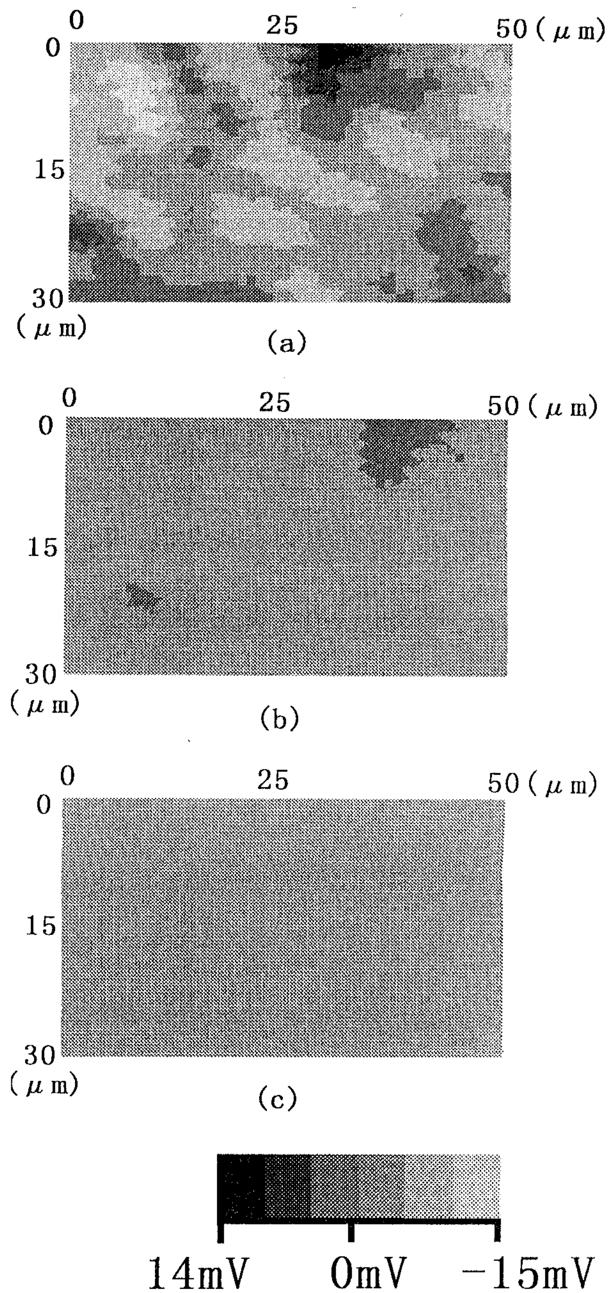

Fig.4 Dielectric temperature coefficient images of TiO_2-$Bi_2Ti_4O_{11}$ ceramics, with a light chopping frequency of fc=40kHz(a), 4kHz(b) and 0.4kHz(c).

As is well known, the thermal diffusion length μ of the material is inversely proportional to the square root of the light chopping frequency f_c. That is

$$\mu_s=\sqrt{\frac{k_s}{\pi \rho_s C_s f_c}} \quad .$$

Therefore, we can obtain a good resolved image corresponding to the grains with the opposite sign of the temperature coefficient when we use a high chopping frequency of 40KHz at which the thermal diffusion length is smaller than the grain size. With the decrease of light chopping frequency, μ_s becomes longer and the area with the radius of μ_s is alternately heated simultaneously. Thus we obtained the averaged temperature coefficient image corresponding to the macroscopic value of the temperature coefficient of τ_ε=-34ppm/°C of the TiO_2-$Bi_2Ti_4O_{11}$ two phases ceramics at the low light chopping frequency of 0.4KHz. From the above mentioned experimental results, we find that this scanning photothermal dielectric technique is very useful for the temperature coefficient of dielectric constant of each grain in the composite ceramics as well as for the macroscopic average value of it.

CONCLUSION

We have proposed a new photothermal technique (scanning photothermal dielectric microscope) for the area distribution of the temperature coefficient of dielectric materials for microwave application. Using this technique, we successfully measured the two dimensional temperature coefficient image of the two phases composite ceramics composed of TiO_2 and $Bi_2Ti_4O_{11}$. The obtained images show that the each grain of TiO_2 and $Bi_2Ti_4O_{11}$ has a negative and positive temperature coefficient respectively. This scanning photothermal dielectric microscope is very useful for the temperature coefficient of dielectric constant of each grain in a composite ceramics as well as for the macroscopic average value of them without using the other expensive, complicated and destructive techniques.

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REFERENCES

[1]S.Nishigaki, H.Kato, S.Yano and R.Kamimura:Am. Ceram. Soc. Bull.,**66**,1405(1987)

[2]Y.Cho and T.Kumamaru: "Photothermal Dielectric Spectroscopic Microscope", Rev. Sci. Instrum.,**67**, 19(1996)