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THE EFFECT OF SINTERING TIME ON THE ELECTRICAL PROPERTIES OF $CaTiO_3$ CERAMICS

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In this paper the effect of sintering time on microstructure and dielectric constant of calcium titanate have been reported. CaTiO3 ceramics was pressure sintered starting from mixed oxide powders. The results indicate that as the sintering time increase the grain size, density, dielectric constant and curie temperature increases. The change of dielectric constant by temperature shows some fluctuation, but for low temperature the samples with large sintering time are more stable.

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Keyword: Sintering time, Calcium titanate, Dielectric constant, Loss tangent

1. Introduction

There are great number of application for ceramic materials due to their high relative permittivity, electrical insulating and electrical conducting properties. Nowadays, advanced ceramics became the key of success for the development of integrated circuits in microelectronic industry. Calcium titanate (CaTiO3) is well known for the treatment of radioactive wastes since this properties form a vast number of solid solutions with rare earth metals [1,2]. Recently, CaTiO3 based dielectric ceramics have been extensively investigated for their microwave dielectric properties. Calcium titanate exhibits a combination of high permittivity and modest dielectric loss on the other hand, CaTiO3 based materials are used as catalysts for partial oxidation of light hydrocarbons. [3]. CaTiO3 could be of great usefulness for the development of integrated circuits in microelectronic industry in the future. The aim of this paper is to study the effect of sintering time on the microstructure and dielectric properties of calcium titanate. For this purpose three set of specimens have been prepared at different sintering times.

2. Experimental procedure

The fabrication of most bulk ceramics starts with powder preparation [4,5]. The powder then prossed to the required shapes and sizes, and the green shapes are in turn processed to mechanically strong and dense ceramics. The more important processes that influences the product characteristics and properties are powder preparation, powder calcining and sintering. In thids study the calcium titanate ceramics were fabricated by powder mixing method and the starting products are CaCo3 and TiO2. Calcium titanate have been mixed and calcinated at 1000 ^oC for 1 h. The samples are sintered at temperature of 1350 ^oC with heating rate of 5 ^oC/min and soaking time held for three sets of specimens for a period of 3, 7 and 11 h. Careful temperature control is maintained through the calcinations and sintering cycle with a microprocessor controlled furnace model labotherm HT04/17. The specimens were poled at 120 ^oC in a bath of silicon oil. The poling was carried out for 30 min using an applied field of 20 kV/cm and then the specimens cooled to room temperature with the field applied. The temperature measured with a high resolution (0.1^oC) microprocessor-based digital thermometer model Fluke-51. An external k-type thermocouple with a tolerance ±0.1^oC over the range of 0 to 250_C is used as temperature sensor.

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3. Results and discussion

Fig1 shows the FTIR spectra of the specimens that have been sintered for 11 h.

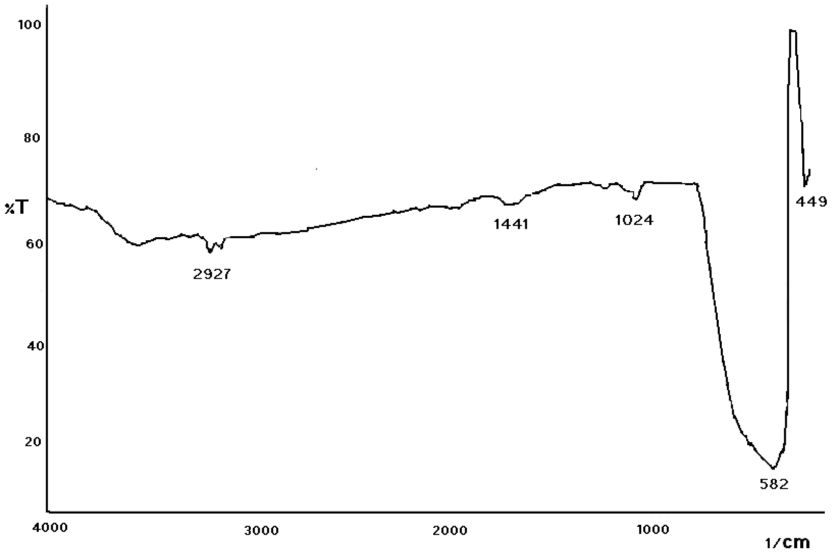

Fig 1. FTIR spectra of the CaTiO3 sintered at 11 h

It is observed that a broad peak is at 583 cm-1 which is due to the Ti-O vibration and other at 450 cm-1, which is a characteristic feature of CaTiO3 (4 fig4).

In table 1 the measured grain size, density, curie temperature, dielectric constant and loss tengent at room temperature are shown.

Table 1. The sintering time and density for three set of specimens

	Sintering time (h)	Density(gr/cm3)	Gran size(μm)	Dielectric constant	Loss tangent	Curie temperature(^{o}C)
A1	3	3.07	2	146	0.047	170
A2	7	369	5	178	0.037	178
A3	11	3.97	7	190	0.033	197

The curie temperatures were measured from depolarization. It is clear that the density, grain size, dielectric constant, loss tangent and curie temperature are depend on sintering time. With the increase of sintering time, density, dielectric constant and curie temperature increased and loss tangent decreased.

Fig 2 shows the dielectric constant as a function of sintering time at different frequency. The dielectric constant of the samples investigated are found to be decreasing with the increasing frequency and increasing with the increasing sintering time.

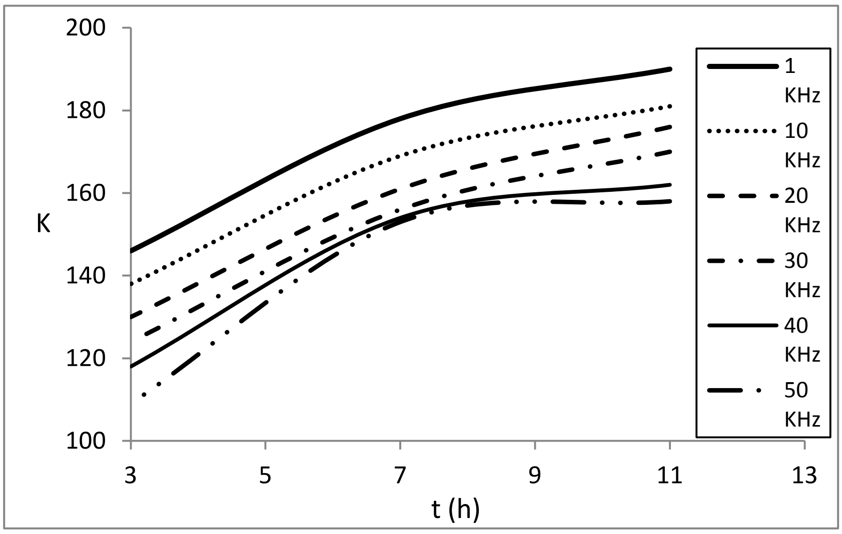

Fig 2 Dielectric constant as a function of sintering time at different frequency

The decreasing dielectric constant with the increasing frequency can be attributed to lagging of the dipoles present in the material, which is a typical Debye type behavior exhibited by most of the dielectric materials.

Frequency dependence of loss tangent of the three set of specimens are shown in Fig 3.

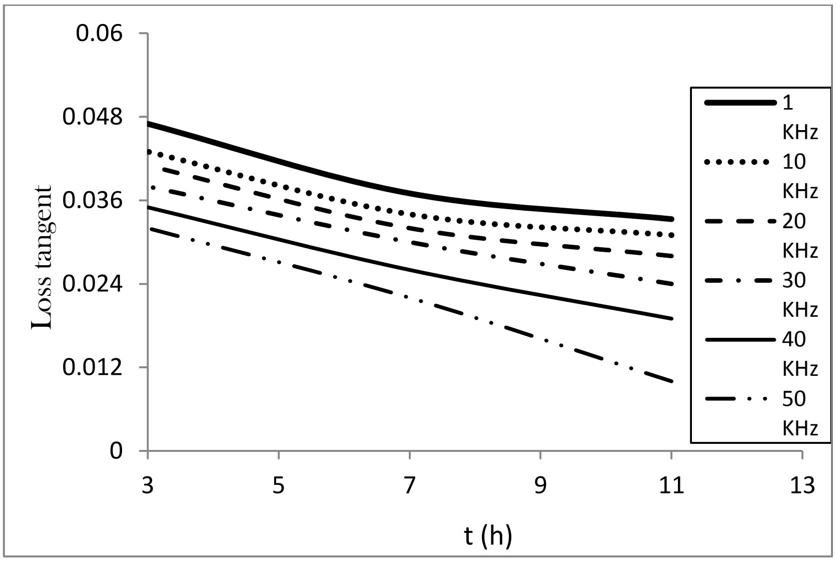

Fig 3 Frequency dependence of loss tangent of the three set of specimens

The loss tangent of the specimens is found to be decreasing with increasing frequency and as shown in table 1 with increasing sintering, its value have been decreased.

Temperature dependence of dielectric constant of three set of specimens are shown in Fig 4.

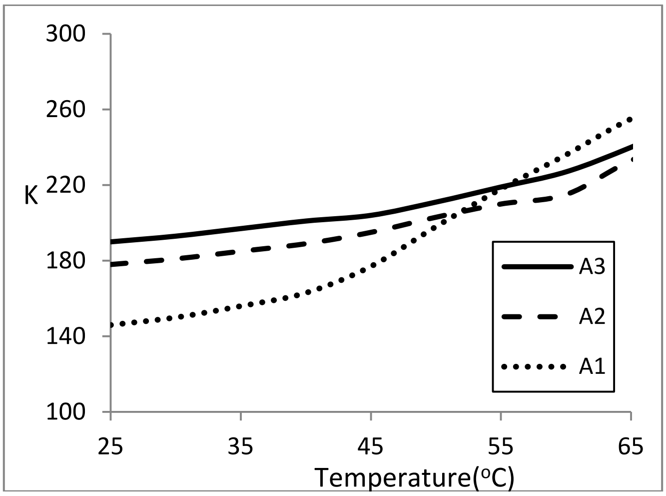

Fig 4 Temperature dependence of dielectric constant of three set of specimens

It is observed that the dielectric constant is increasing slightly with the increasing temperature which is already known to be characteristic behavior of materials with provskite structure. The dielectric constant shows some fluctuation by the temperature but at low temperatures the samples which have been prepared at large sintering time are more stable.

The change of dielectric constant at a wide range of temperature has been shown in Fig 5 for three set of specimens. There are three distinguishable and significant regions that associated with different phenomenon's. In region 1 (between 25 and 65 oC (A3 group)) the changes in depolarization with temperature are small hence the dielectric constant is small. As the temperature rise, region 2, (between 65 and 200 oC (A3 group)) the dipoles get enough energy to move and change the polarization and dielectric constant increase. The maximum dielectric constant in this region vary with temperature which for sample A1, A2 and A3 that occurs at temperatures 170, 178 and 197 oC respectively. If we take this peak as Curie temperature, the measured Curie temperatures with other parameters are shown in Table 1. By increasing temperature further, the dielectric constant will decrease (region 3).

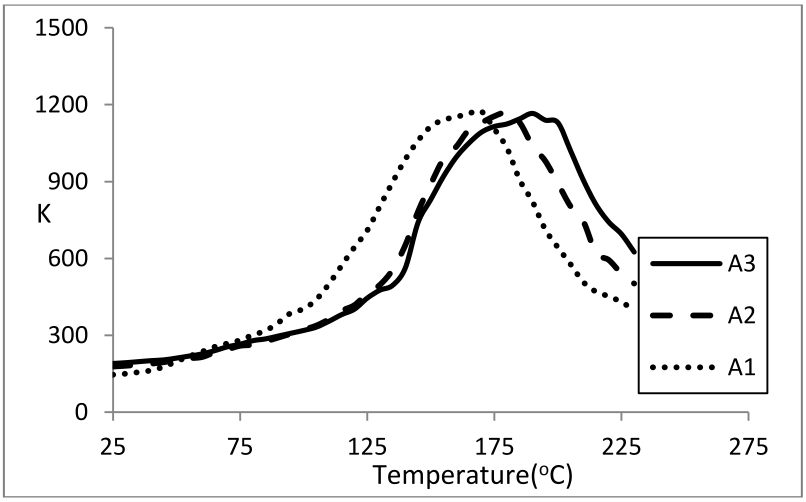

Fig 5 Dielectric constant as a function of temperature

4. Conclusion

Calcium titanate ceramics were fabricated at three set with different sintering time. It is found that with the increase of sintering time, density, grain size and dielectric constant increased. By depoling the samples the measured curie temperatures and dielectric constant increase. The loss tangent of the specimens is found to be decreasing with increasing frequency and sintering time. There are three significant regions that associated with different phenomenon's in variation of dielectric constant by temperature.

Acknowledgment

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