

## Dielectric and Pyroelectric Properties of Y-doped $\text{Ba}_{0.6}\text{Sr}_{0.3}\text{Ca}_{0.1}\text{TiO}_3$ Ceramics by Solid-state Reaction Technique

CAO Sheng, MAO Chao-Liang, YAO Chun-Hua, CAO Fei, WANG Gen-Shui, DONG Xian-Lin

(Key Laboratory of Inorganic Functional Materials and Devices, Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai 200050, China)

**Abstract:**  $(\text{Ba}_{0.6}\text{Sr}_{0.3}\text{Ca}_{0.1})_{1-x}\text{Y}_x\text{Ti}_{0.999}\text{Mn}_{0.001}\text{O}_3$  ( $0 \leq x \leq 0.007$ ) ceramics were prepared by the solid-state reaction technique. The effects of Y doping on the microstructure, dielectric properties and pyroelectric properties were investigated. The average grain size decreases with the increase of Y concentration. The dielectric and pyroelectric properties measurement results show that the dielectric constant, dielectric loss,  $T_c$  and pyroelectric coefficient all initially increase and then decrease with Y concentration. And the pyroelectric figure of merit  $F_d$  can be improved by the Y doping. The specimen doped with 0.7mol%Y shows a higher maximum  $F_d$  value of  $8.22 \times 10^{-5} \text{ Pa}^{-1/2}$  under 700 V/mm near  $30^\circ\text{C}$  with the smallest average grain size of 3.1  $\mu\text{m}$ , indicating its promising application in infrared thermal imaging arrays devices.

**Key words:** (Ba,Sr,Ca)TiO<sub>3</sub>; ceramics; dielectric properties; pyroelectric properties

Barium strontium calcium titanate ( $\text{Ba,Sr,CaTiO}_3$  (BSCT)), one of the most important ferroelectric solid solution materials, has attracted much attention because of its interesting ferroelectric, dielectric and pyroelectric properties. Due to the high dielectric constant, low dielectric loss, and concentration-dependent phase transition temperature, BSCT shows its potential in dynamics random access memories, tunable microwave devices, and infrared thermal imaging devices<sup>[1-4]</sup>. In particular, the large changes in spontaneous polarization and dielectric constant near the ferroelectric-paraelectric phase transition allow the development of BSCT-based high performance infrared thermal imaging devices<sup>[5]</sup>. To achieve good imaging performance, much effort has been devoted to the technology of fabricating hybrid arrays detectors with thin pixel and large array, *i.e.* 10–20  $\mu\text{m}$  thickness and  $384 \times 288$  elements<sup>[5-7]</sup>. So it is required to use a ceramics with a grain size much smaller than the thickness, typically the grain size in the range of 1–3  $\mu\text{m}$ , to avoid poor mechanical properties when thinning and large pixel to pixel variation in crystallite orientation and pyroelectric response<sup>[6]</sup>.

However, there seems to be a contradiction between high performance and small grain size. It was reported that, for the pure BSCT, the high pyroelectric response cannot

be obtained unless the grain size was greater than 10  $\mu\text{m}$  or so<sup>[6]</sup>. In order to solve this problem, various dopants were introduced. Kulwicki<sup>[6]</sup> doped the material with donor ions such as Nb, Ta, Bi, Sb, Y, La and so on, and obtained the greatly improved pyroelectric response ceramics which had a figure of merit  $F_d$  greater than  $11 \times 10^{-5} \text{ Pa}^{-1/2}$  and a small grain size (less than 5  $\mu\text{m}$ ). Lee, *et al.*<sup>[8-9]</sup> reported the effects of  $\text{Y}_2\text{O}_3$  and  $\text{MnCO}_3$  doping on the microstructure and electrical properties of  $\text{Ba}_{0.6}\text{Sr}_{0.3}\text{Ca}_{0.1}\text{TiO}_3$  ceramics prepared by Sol-Gel method and found that 0.5mol%  $\text{Y}_2\text{O}_3$  and 0.1mol%  $\text{MnCO}_3$  doped BSCT ceramics showed the enhanced figure of merit  $F_d$  about  $5.91 \times 10^{-5} \text{ Pa}^{-1/2}$  and small grain size (1–3  $\mu\text{m}$ ). Because chemical method is more complicated and expensive, it is desirable to prepare the pyroelectric ceramics by conventional solid-state reaction technique, accounting for the easy and low cost fabrication. The present paper describes the Y doping effects on the microstructure, dielectric and the pyroelectric properties of BSCT-based ceramics prepared by solid-state reaction with the goal of assessing the potential applications for infrared detectors.

## 1 Experimental

The ceramics were prepared according to the formula

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Biography: CAO Sheng (1985–), male, candidate of PhD. E-mail: caosheng658@126.com

Corresponding author: Dong Xian-Lin, professor. E-mail: xldong@sunm.sh.cn

$(\text{Ba}_{0.6}\text{Sr}_{0.3}\text{Ca}_{0.1})_{1-x}\text{Y}_x\text{Ti}_{0.999}\text{Mn}_{0.001}\text{O}_3$  ( $\text{BSCY}_x\text{TM}$ ,  $0 \leq x \leq 0.007$ ), using the conventional solid-state reaction technique. The raw materials used were 99.0%  $\text{BaCO}_3$ , 99.0%  $\text{SrCO}_3$ , 99.0%  $\text{CaCO}_3$ , 99.44%  $\text{TiO}_2$ , 99.99%  $\text{Y}_2\text{O}_3$  and 92.83%  $\text{MnCO}_3$  powders. They were milled in water with agate balls for 24 h. 0.1 mol%  $\text{MnCO}_3$  was added to maintain sufficiently high resistivity, because the purely Y doped BSCT ceramics may be too conductive. After the mixture was dried, it was calcined at  $1150^\circ\text{C}$  for 2 h in air. Then the powder was mixed with a small amount of poly vinyl acetate (PVA) as adhesive and pressed at 100 MPa in a disk of 15 mm in diameter. The samples were sintered at  $1450^\circ\text{C}$  for 2 h in oxygen after the PVA was burned out at  $800^\circ\text{C}$  for 2 h. The sample disks were machined into 0.2 mm in thickness and silver electrodes were screen printed onto both surfaces of the disks.

The crystallinity and the microstructure of the  $\text{BSCY}_x\text{TM}$  specimens ( $0 \leq x \leq 0.007$ ) were observed using X-ray diffraction (XRD, Rigaku D/MAX-2550V) and scanning electron microscope (SEM, JSM-6700F), respectively. The heat capacity per unit volume  $C_v$  at room temperature was determined by a Perkin-Elmer DSC-2C system. The temperature variation of the dielectric properties as a function of DC bias field at 1 kHz was measured by an automatic measuring system based on HP4284 LCR meter. The pyroelectric properties with variation of temperature and DC bias field were studied with a Keithley 6517A electrometer/high-resistance meter using modified

Byer and Roundy techniques.

## 2 Results and discussions

### 2.1 Phase structure and microstructure

Figure 1 illustrates the XRD patterns of  $\text{BSCY}_x\text{TM}$  ceramics ( $0 \leq x \leq 0.007$ ). All the specimens show a single perovskite structure without second phase. And all peaks move toward high  $2\theta$  with the increase of Y concentration, due to the replacement of  $\text{Ba}^{2+}$ ,  $\text{Sr}^{2+}$  and  $\text{Ca}^{2+}$  with a smaller  $\text{Y}^{3+}$  on the A-site.

Figure 2 shows the surface SEM micrographs of  $\text{BSCY}_x\text{TM}$  ceramics ( $0 \leq x \leq 0.007$ ). It can be seen that all the specimens exhibit a dense grain structure and the average grain size decreases with Y concentration mono-

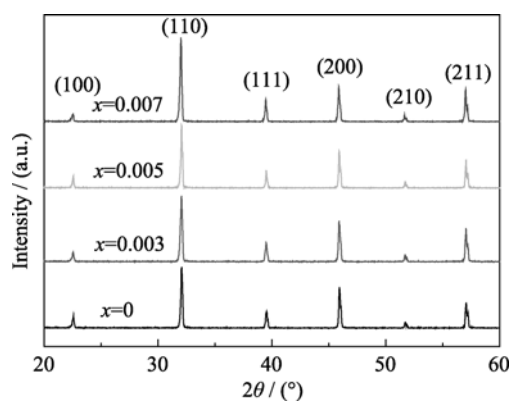


Fig. 1 XRD patterns of  $\text{BSCY}_x\text{TM}$  ceramics ( $0 \leq x \leq 0.007$ )

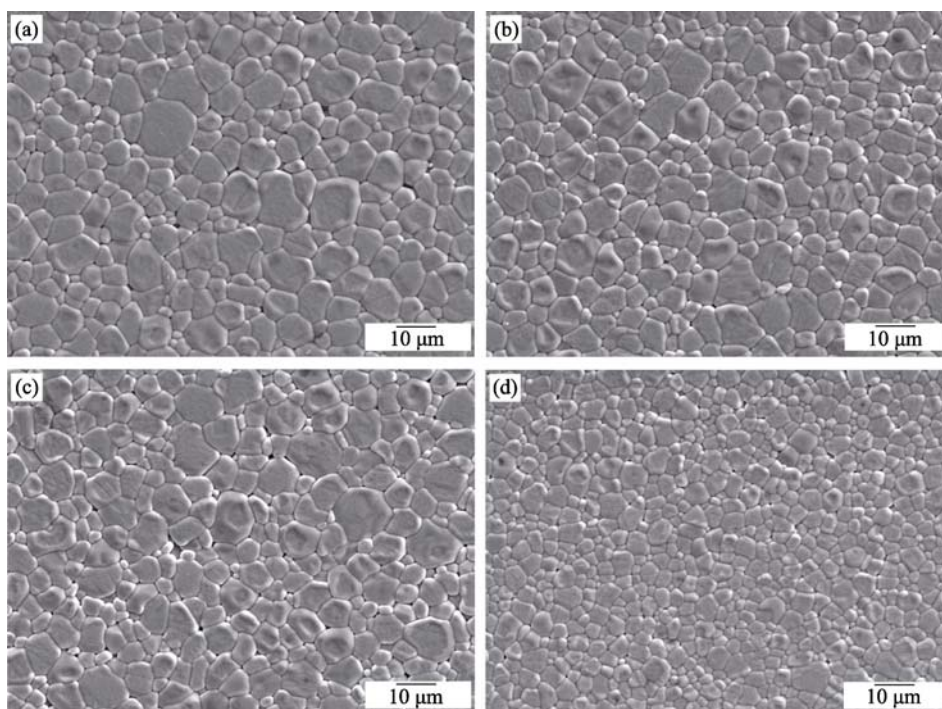


Fig. 2 Surface SEM images of  $\text{BSCY}_x\text{TM}$  ceramics  
(a)  $x=0$ ; (b)  $x=0.003$ ; (c)  $x=0.005$ ; (d)  $x=0.007$

tonically. When the Y concentration is 0.7mol%, the smallest average grain size of 3.1  $\mu\text{m}$  is obtained, compared with that of 8.5  $\mu\text{m}$  for the undoped specimen. The change in grain size could be attributed to the effect of the donor-dopant incorporation. Drofenik<sup>[10]</sup> proposed that the incorporation behavior of donors played an important role in grain growth and suggested a model for the grain size anomaly. According to this model, the driving force for grain growth of donor doped  $\text{BaTiO}_3$  consists of the stored energy released during grain growth and the energy consumed during donor incorporation. The more donor dopants added, the more energy consumed for donor incorporation. Therefore, the driving force for grain growth is annihilated, and the grain size decreases.

## 2.2 Dielectric properties

The dielectric constant and dielectric loss as a function of temperature at zero bias field for  $\text{BSCY}_x\text{TM}$  ceramics ( $0 \leq x \leq 0.007$ ) are shown in Fig. 3. Small concentration of Y ions in the ceramics leads to an increase of the dielectric constant (especially the peak dielectric constant), but a further increase of Y concentration causes a decrease of the dielectric constant. Thus, the highest peak dielectric constant was obtained for  $x=0.005$  sample. There are three reasons for the change of the dielectric constant. Firstly, the Y doping in the ceramics leads to the distortion of the crystal lattice, resulting in an increase of internal stress, which is beneficial for the improvement of dielectric constant<sup>[11]</sup>. Secondly, Y ions can inhibit the grain growth and decrease the grain size. With the decrease of grain size, the total  $90^\circ$  domain walls area per volume will increase, giving more contribution to the dielectric constant, which leads to the increase of the dielectric constant<sup>[12]</sup>. Thirdly, as the grain size decreases, the fraction of grain boundary volume increases. Generally, the dielectric constant of grain is larger than that of grain boundary. Therefore, the smaller the grain size is, the more grain boundaries are, and the lower dielectric constant is. As discussed above, the effects of the internal stresses,  $90^\circ$  domain walls and

the grain boundary are responsible for the change of the dielectric constant with Y concentration.

The dielectric loss shows the similar dependence on Y concentration as that of the dielectric constant. The dielectric loss initially increases and then decreases with the increase of Y concentration. The highest dielectric loss was obtained for  $x=0.003$  sample. Obviously, Y doped samples show higher dielectric loss than undoped sample, especially below the transition temperature, which could be attributed to the contributing of the defects to the dielectric loss such as  $\text{Y}_{\text{Ba}}$ .

Moreover, it can be seen that  $T_c$  initially increases with Y concentration up to a maximum value of  $22.1^\circ\text{C}$  for  $x=0.003$  before decreasing with increasing Y concentration to a lower value of  $20.4^\circ\text{C}$  for  $x=0.007$ . The initial rise in  $T_c$  could be attributed to Y, Mn co-doping effect. For  $x=0$  sample, only  $\text{Mn}^{2+}$  (acceptor) is introduced into the lattice. The replacement of  $\text{Ti}^{4+}$  with  $\text{Mn}^{2+}$  causes the creation of oxygen vacancy leading to a “break” of the cooperative vibration of the Ti–O chains, and therefore results in the suppression of  $T_c$  for the Mn-doped system<sup>[13-14]</sup>. While for  $x=0.003$  sample, the replacement of  $\text{Ba}^{2+}$ ,  $\text{Sr}^{2+}$  and  $\text{Ca}^{2+}$  with  $\text{Y}^{3+}$  (donor) inhibits oxygen vacancy formation, neutralizing the  $\text{Mn}^{2+}$  doping effect on  $T_c$ , and therefore results in the initial rise in  $T_c$ . Obviously, the decrease in  $T_c$  with increasing  $x$  from 0.003 to 0.007 can be understood by the A-site cation size effect, in which the smaller Y ion causes a reduction in the average radius of the A-cation size and thereby stabilizes the high temperature cubic structure to lower temperature.

The dielectric constant and dielectric loss as a function of temperature under different DC bias fields for  $\text{BSCY}_x\text{TM}$  ceramics ( $x=0.007$ ) are illustrated in Fig. 4. With the increase of bias field, the curves display the broadness of peak and the reduction in the dielectric constant and loss, which can be attributed to the domain wall motion under DC bias field<sup>[15]</sup>. Moreover,  $T_c$  shifts to a

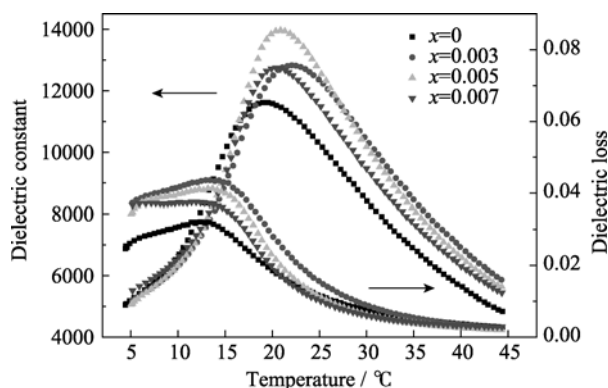


Fig. 3 Dielectric constant and dielectric loss as a function of temperature at zero bias field for  $\text{BSCY}_x\text{TM}$  ceramics ( $0 \leq x \leq 0.007$ )

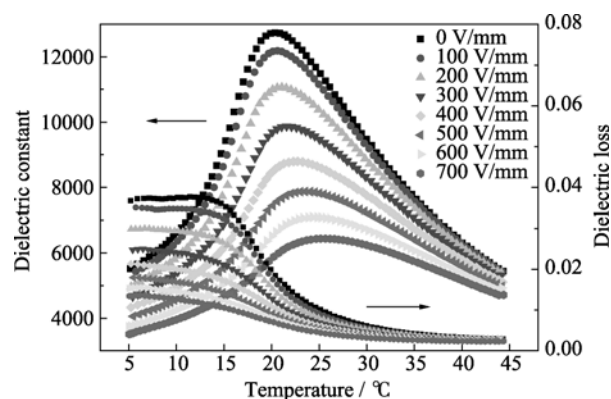


Fig. 4 The dielectric constant and dielectric loss as a function of temperature under different DC bias fields for  $\text{BSCY}_x\text{TM}$  ceramics ( $x=0.007$ )

higher temperature as DC bias field increases, which confirms that FE phase can be stabilized by the electric field.

### 2.3 Pyroelectric properties

Figure 5 shows the pyroelectric coefficient as a function of temperature under different DC bias fields for BSCY<sub>x</sub>TM ceramics ( $0 \leq x \leq 0.007$ ). For each specimen, the maximum pyroelectric coefficient first increases and then decreases with the increase of DC bias field from 0 to 700 V/mm. Generally, the pyroelectric coefficient under DC bias can be obtained as following<sup>[5]</sup>:

$$p = \left( \frac{\partial D}{\partial T} \right)_E = \left( \frac{\partial P}{\partial T} \right)_E + \epsilon_0 \int_0^E \left( \frac{\partial \epsilon_r}{\partial T} \right)_E dE \quad (1)$$

where  $D$  is the displacement,  $T$  is temperature,  $E$  is the bias field,  $P$  is the polarization, and  $\epsilon_r$  is dielectric constant. According to equation 1, the total pyroelectric coefficient  $p$  consists of the spontaneous polarization contribution (the first item on the right side) and the dielectric contribution (the second item). Under zero field, the net decrease of the spontaneous polarization is zero because of the random orientation of dipoles and the dielectric contribution is also zero, and then no pyroelectric current can be detected. As the bias field increases, larger and larger pyroelectric current can be obtained, mainly because more and more dipoles are orientated along the bias field direction. When the bias field continues to increase, the polari-

zation becomes saturated, but the pyroelectric current decreases due to the dielectric contribution. It implies that the polarization plays a more important role in pyroelectric response than dielectric contribution.

Y doped samples show higher pyroelectric coefficient than undoped sample. The highest pyroelectric coefficient peak of  $37.8 \times 10^{-8} \text{ C}/(\text{cm}^2 \cdot \text{K})$  is obtained for  $x=0.005$  sample, which is almost twice than that of undoped sample. The pyroelectric coefficient is mainly dependent on the polarization. Therefore, the enhanced pyroelectric response is mainly due to the enhanced polarization by the Y doping, which could be confirmed by the enhanced dielectric constant as shown in Fig. 3.

The pyroelectric figure of merit  $F_d$  which describes signal to noise can be obtained as following:

$$F_d = \frac{p}{C_v (\epsilon_0 \epsilon_r \tan \delta)^{1/2}} \quad (2)$$

where  $p$  is pyroelectric coefficient,  $C_v$  the heat capacity per unit volume, approximately  $2.38 \text{ J}/(\text{cm}^3 \cdot \text{K})$ ,  $\epsilon_r$  dielectric constant and  $\tan \delta$  dielectric loss. Figure 6 shows the figure of merit  $F_d$  as a function of temperature and DC bias field for BSCY<sub>x</sub>TM ceramics ( $0 \leq x \leq 0.007$ ). Since the dielectric constant and loss are both suppressed by the DC bias field, the figure of merit  $F_d$  increases as the DC bias field increases for all specimens. For  $x=0.007$

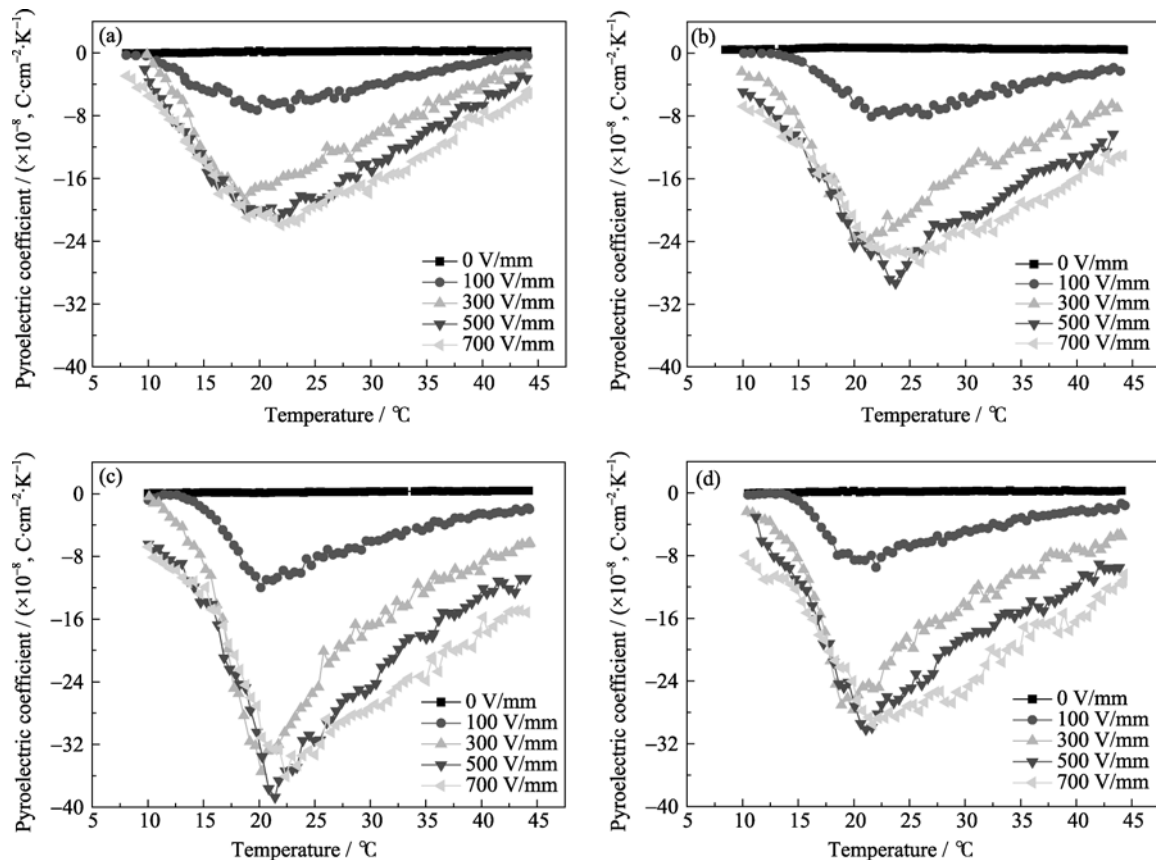


Fig. 5 Pyroelectric coefficient as a function of temperature under different DC bias fields for BSCY<sub>x</sub>TM ceramics (a)  $x=0$ ; (b)  $x=0.003$ ; (c)  $x=0.005$ ; (d)  $x=0.007$

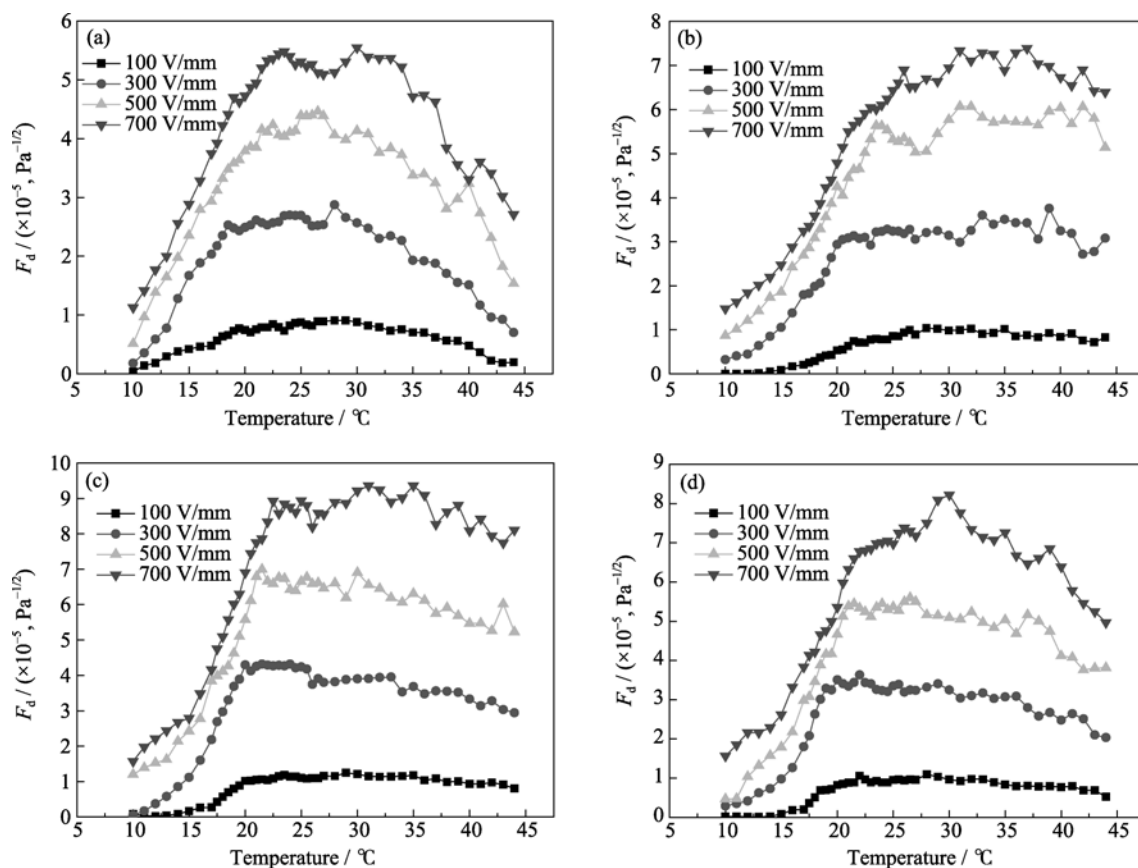


Fig. 6 The figures of merit  $F_d$  as a function of temperature and DC bias field for  $\text{BSCY}_x\text{TM}$  ceramics  
(a)  $x=0$ ; (b)  $x=0.003$ ; (c)  $x=0.005$ ; (d)  $x=0.007$

sample, it shows a maximum  $F_d$  value of  $8.22 \times 10^{-5} \text{ Pa}^{-1/2}$  under 700 V/mm near 30 °C with the smallest average grain size of 3.1  $\mu\text{m}$ , while the undoped sample exhibits a maximum  $F_d$  value of  $5.54 \times 10^{-5} \text{ Pa}^{-1/2}$  with the largest average grain size of 8.5  $\mu\text{m}$ , which indicates that microstructure and pyroelectric properties of the sample are both improved by the Y doping.

Pyroelectric properties of  $\text{BSCY}_x\text{TM}$  ceramics prepared by solid-state reaction and Sol-Gel method are given in Table 1. Typical properties of  $\text{Ba}_{0.67}\text{Sr}_{0.33}\text{TiO}_3$  ceramics listed are approximate and broadly published. Obviously,  $\text{BSCY}_x\text{TM}$  ceramics prepared by solid-state reaction technique exhibit the highest  $F_d$  value. And for  $x=0.007$  sam-

ple in this work, its maximum  $F_d$  is higher than that of ceramics prepared by Sol-Gel method<sup>[8-9]</sup>.

### 3 Conclusions

$(\text{Ba}_{0.6}\text{Sr}_{0.3}\text{Ca}_{0.1})_{1-x}\text{Y}_x\text{Ti}_{0.999}\text{Mn}_{0.001}\text{O}_3$  ( $0 \leq x \leq 0.007$ ) ceramics were prepared by the solid-state reaction technique. The average grain size decreased from 8.5  $\mu\text{m}$  to 3.1  $\mu\text{m}$  with the increase of  $x$  from 0 to 0.007, due to the effect of the donor-dopant incorporation. The dielectric and pyroelectric properties measurement results show that the dielectric constant, dielectric loss,  $T_c$  and pyroelectric coefficient all initially increase and then decrease with Y

Table 1 Pyroelectric properties of  $\text{BSCY}_x\text{TM}$  ceramics prepared by solid-state reaction and Sol-Gel method

Material	Bias $/(kV \cdot cm^{-1})$	$C_v$ $/(J \cdot cm^{-3} \cdot K^{-1})$	$P$ $/( \times 10^{-9}, C \cdot cm^{-2} \cdot K^{-1})$	$\epsilon_r$	$\tan \delta$	$F_d / Pa^{-1/2}$	Reference
$\text{Ba}_{0.67}\text{Sr}_{0.33}\text{TiO}_3$	—	2.55	350	5000	0.01	$6.52 \times 10^{-5}$	[16]
$\text{BSCY}_x\text{TM}$ ( $x=0.005$ )	7	2.38	269	6287	0.002	$9.36 \times 10^{-5}$	This work
$\text{BSCY}_x\text{TM}$ ( $x=0.007$ )	7	2.38	256	6200	0.003	$8.22 \times 10^{-5}$	This work
$(\text{Ba}_{0.6}\text{Sr}_{0.3}\text{Ca}_{0.1})\text{TiO}_3 +$ 0.1mol%MnCO <sub>3</sub> +0.5mol% Y <sub>2</sub> O <sub>3</sub>	4	3.2	~270	~8900	~0.01	$2.99 \times 10^{-5}$	(Sol-Gel) <sup>[8]</sup>
$(\text{Ba}_{0.6}\text{Sr}_{0.3}\text{Ca}_{0.1})\text{TiO}_3 +$ 0.1mol%MnCO <sub>3</sub> +0.5mol% Y <sub>2</sub> O <sub>3</sub>	8	3.2	~300	~6000	~0.005	$5.91 \times 10^{-5}$	(Sol-Gel) <sup>[9]</sup>

concentration. The specimen doped with 0.7mol% Y shows a improved maximum  $F_d$  value of  $8.22 \times 10^{-5} \text{ Pa}^{-1/2}$  under 700 V/mm near 30°C with the smallest average grain size of 3.1  $\mu\text{m}$ , indicating that the smaller grain size and higher pyroelectric properties can be both achieved by Y doping. Thus, the specimen doped with 0.7mol% Y prepared by solid-state technique is very promising for practical use in uncooled infrared imaging arrays detectors.

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# 固相法制备钇掺杂的钛酸钡锶钙陶瓷的介电和热释电性能

曹 盛, 毛朝梁, 姚春华, 曹 菲, 王根水, 董显林

(中国科学院 上海硅酸盐研究所, 中国科学院无机功能材料与器件重点实验室, 上海 200050)

**摘 要:** 采用固相反应法制备了 Y 掺杂  $(\text{Ba}_{0.6}\text{Sr}_{0.3}\text{Ca}_{0.1})_{1-x}\text{Y}_x\text{Ti}_{0.999}\text{Mn}_{0.001}\text{O}_3$  ( $0 \leq x \leq 0.007$ ) 陶瓷, 重点研究了 Y 含量对 BSCT 基陶瓷的显微结构、介电性能和热释电性能的影响。结果表明: 随着 Y 含量的增加,  $\text{BSCY}_x\text{TM}$  陶瓷的平均晶粒尺寸逐渐减小, 介电常数、介电损耗、居里温度和热释电系数均呈现先增加后减小的趋势。当 Y 掺杂量为 0.7mol% 时,  $\text{BSCY}_x\text{TM}$  陶瓷的平均晶粒尺寸最小为 3.1  $\mu\text{m}$ , 且探测优值  $F_d$  较大, 最大值可达  $8.22 \times 10^{-5} \text{ Pa}^{-1/2}$  (700 V/mm, 30°C), 高于采用溶胶-凝胶法制备的同组分陶瓷的探测优值  $5.91 \times 10^{-5} \text{ Pa}^{-1/2}$ 。

**关 键 词:**  $(\text{Ba,Sr,Ca})\text{TiO}_3$ ; 陶瓷; 介电性能; 热释电性能

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