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Dielectric Relaxation Behaviour of Bi:SrTiO$_3$: I. The Low Temperature Permittivity Peak

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Abstract

The dielectric properties of $(Sr_{1-1.5x}Bi_x)TiO_3$ $(0{\cdot}002 \leq x \leq 0{\cdot}167)$ *ceramics were systematically studied. The temperature dependence of the complex-permittivity was measured in the temperature range of 10–300 K. Permittivity peaks with obvious frequency dispersion were observed and with the increase of the Bi content, the temperature of the permittivity maximum of each one is shifted to higher temperatures. In the high temperature side of the permittivity peaks, notable departure from the Curie–Weiss law was observed. The relaxation behaviour is characterised in the present paper. The effect of Bi on the dielectric relaxation behaviour and the physical mechanism giving polarisation are briefly discussed. © 1998 Elsevier Science Limited. All rights reserved*

1 Introduction

As a member of the perovskite family of compounds, strontium titanate is a quantum paraelectric[1,2] and is sensitive to impurities.[3] Much work has been performed on impurity doped strontium titanate, either as a single crystal,[4] as a polycrystalline material[5–7] or as amorphous thin films[8] and many applications have been found, such as varistors,[9] boundary layer capacitors,[10] high voltage capacitors[11] and so on. Among them, strontium-bismuth titanate SrTiO$_3$–Bi$_{2/3}$TiO$_3$ system[12–14] was reported, for the first time, by Skanavi *et al.* A single phase structure was obtained up to 35% Bi$_{2/3}$TiO$_3$, and a cubic perovskite lattice was determined by X-ray diffraction analysis.[13]

Skanavi's pioneer work on the dielectric behaviour of the SrTiO$_3$–Bi$_{2/3}$TiO$_3$ solid solutions showed the existence of a permittivity peak with frequency dispersion[12–14] without observable nonlinearity and ferroelectricity. Skanavi *et al.* discussed the possible physical nature of the dielectric behaviour and attributed it to a polarisation mechanism of 'hopping ions',[13] i.e. the 'relaxational polarisation' originated from relatively large displacements of weakly bound ions (Ti^{4+} ions), which by thermal motion surmounted local potential barriers. Weakly bound Ti^{4+} ions were considered due to the presence of strontium vacancies, which led to the distortion of the oxygen octahedra in the perovskite lattice.[12,13]

Contrary to the Skanavi's point of view, the existence of ferroelectricity was suggested by other authors.[14–16] A slim electric hysteresis loop for the 0·85SrTiO$_3$–0·15Bi$_{2/3}$TiO$_3$ solid solution observed at the low temperature and high electric field was reported by Smolenskii *et al.*[14] and also by Gubkin *et al.*[15,16] for other compositions. After this dispute between the two schools, work on the physical nature of the polarisation and the dielectric relaxation behaviour of Bi:SrTiO$_3$ has not been done, and the mechanism is still uncertain.

On the other hand, relaxor type materials have been extensively studied during the last two decades. Pb(Mg$_{1/3}$Nb$_{2/3}$)O$_3$ (PMN), a typical relaxor, displays a diffuse phase transition (DPT), extending over a finite range of temperatures, which was attributed to a compositional heterogeneity by Smolenskii *et al.*[17] This DPT transition is common in disordered ionic structures, especially in solid solutions or complex compounds. With the increasing technological importance of relaxor materials, to meet the needs of industrial applications, such as electrostrictive components, energy transducers, multilayer capacitors, etc., together with an increasing theoretical interest in dielectric physics, these materials[17–21] have attracted great attention both from the applied as well as the

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fundamental points of view. Different explanations were put forward for these relaxor materials. A concept of 'superparaelectric' and a further 'dipole glass' model has been proposed by Cross and Viehland *et al.*[19,20] while a random-electric-field model was suggested for PMN by Westphal *et al.*[21] To date, a complete understanding of the relaxor mechanism has not been reached yet. A search for new relaxor systems would be desirable in order to get a deeper insight into this phenomenon.

Recently, work done by the present authors in Bi doped $\mathrm{SrTiO_3}$ shows that the essential features of the relaxor behaviour, i.e. (1) a large dielectric constant, (2) a broad relaxation peak with low frequency dispersion, and (3) a hysteresis loop in the polarisation, are all observed in Bi:$\mathrm{SrTiO_3}$.

In addition to the low temperature relaxor behaviour previously described,[12,14] other dielectric permittivity peaks with a frequency dispersion, appearing in different temperature ranges, were also found. So a systematic study of Bi doped $\mathrm{SrTiO_3}$ was seen as a mean to explore the mechanism of the complex dielectric relaxation phenomena. Such a study could, not only settle the previous controversy over polarisation mechanism but also make a contribution to an understanding of relaxor behaviour.

In this paper, the dielectric dispersion behaviour of $(\mathrm{Sr}_{1-1\cdot5x}\mathrm{Bi}_x)\mathrm{TiO_3}$ as a dielectric relaxor in the low temperature range of 10–300 K is mainly characterised. The dielectric relaxation behaviour in the high temperature range will be dealt with in subsequent articles.[22,23]

2 Experimental Procedure

Ceramic samples were prepared by solid state reaction. Raw materials ($\mathrm{SrCO_3}$, $\mathrm{Bi_2O_3}$ and $\mathrm{TiO_2}$) were weighed according to the composition $(\mathrm{Sr}_{1-1\cdot5x}\mathrm{Bi}_x)\mathrm{TiO_3}$, where x = 0, 0·002, 0·0033, 0·0053, 0·0067, 0·0133, 0·0267, 0·04, 0·0533, 0·08, 0·10, 0·133 and 0·167, respectively. The weighed batches were wet milled in an agate ball mill of a planetary type for 5 h. After drying, they were calcined from 1000 to 1150°C for 2 h. The calcined powders were milled again, dried and pressed into disks. Finally, the samples were sintered from 1300 to 1380°C for 2 h in air and furnace-cooled. The lower the Bi content, the higher the sintering temperature. After sintering, the samples with $x \leq 0\cdot08$ were annealed at 1000°C for 10–100 h in air to eliminate the second set of peaks which will be discussed in Part II of this study.[22]

The density of the samples was measured by the Archimedes method. Gold electrodes were used for dielectric measurements. Silver electrodes were also used for comparison. The electrical behaviour of the samples did not depend on the type of electrodes.

X-ray powder diffraction (XRD) (Rigaku, Cu K_α radiation) was carried out for the sintered samples at room temperature and the lattice parameter was precisely calculated.

The dielectric permittivity and dissipation factor of $(\mathrm{Sr}_{1-1\cdot5x}\mathrm{Bi}_x)\mathrm{TiO_3}$ system were measured with a Solartron 1260 Impedance Gain-Phase Analyser from 10 to 300 K in a frequency range from 1 Hz to 1 MHz.

The temperature dependence of dielectric properties was measured in a cryogenic system while the temperature of the specimen was being heated up at a rate of 0·5 or 1°C min^{-1}, and readings were taken every 2°C.

3 Results

The XRD results indicate that all samples are cubic with the lattice parameter increasing with rising Bi content (Fig. 1).

3.1 Dielectric relaxation behaviour and composition dependence of relaxation

The temperature dependences of the real and imaginary parts of the dielectric complex-permittivity were measured. Typical curves of the real part of the permittivity (ε) are shown for x = 0·0033, 0·0133, 0·08, and 0·167 in Fig. 2. It can be seen that the dielectric behaviour varies strongly with the amount of the bismuth doping. The intensity of the permittivity peak decreases with increasing Bi concentration. The permittivity peaks are quite diffuse with a frequency dispersion present even for the sample with the smallest amount of Bi.

Figure 3 is the plot of the variation of the temperature of the permittivity maximum (T_m) versus bismuth content x at 1 kHz. It can be seen that this temperature increases with the increase in the Bi content.

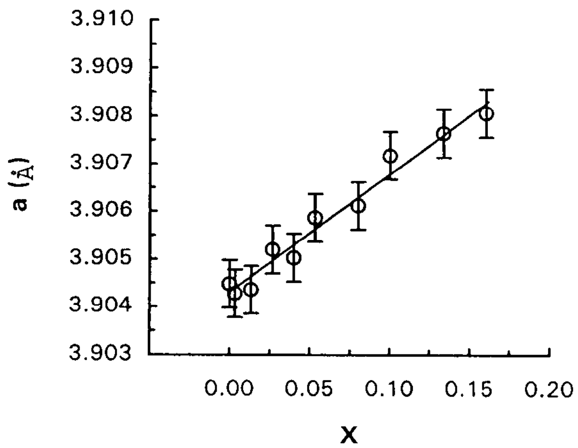

Fig. 1. The lattice parameter a as a function of x.

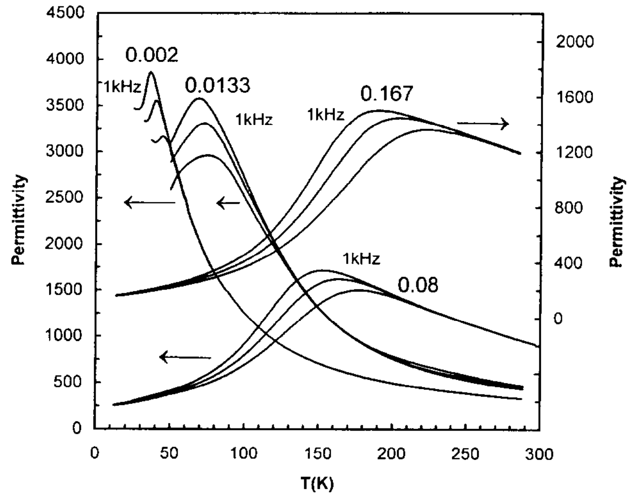

Fig. 2. Temperature dependence of the permittivity for the $(Sr_{1-1.5x}Bi_x)TiO_3$ samples with $x = 0.002$, 0.0133, 0.08 and 0.167 at 1, 10 and 100 kHz.

3.2 Hysteresis loop

Conventional hysteresis loops have been observed under smaller electric fields than those reported by Smolenskii *et al.*[12–14] Typical hysteresis loops for $x = 0.0067$ at 11, 15, 20, 40, 60, 70 K and at 50 Hz with electric fields lower than ~20 kV cm^{-1} are shown in Fig. 4. At 11 K, a remanent polarisation of ~0.83 μC cm^{-2} was obtained. This value is about one order of magnitude higher than that of Ca:SrTiO$_3$,[22] and is much smaller than that of the normal ferroelectric BaTiO$_3$.[1]

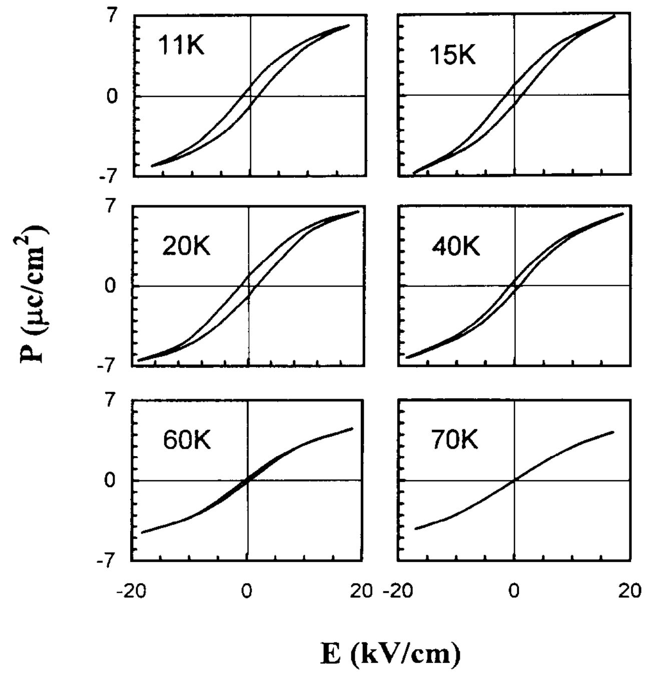

Fig. 4. Hysteresis loops of the $(Sr_{1-1.5x}Bi_x)TiO_3$ sample with $x = 0.0067$ at 50 Hz and at 11, 15, 20, 40, 60 and 70 K, respectively.

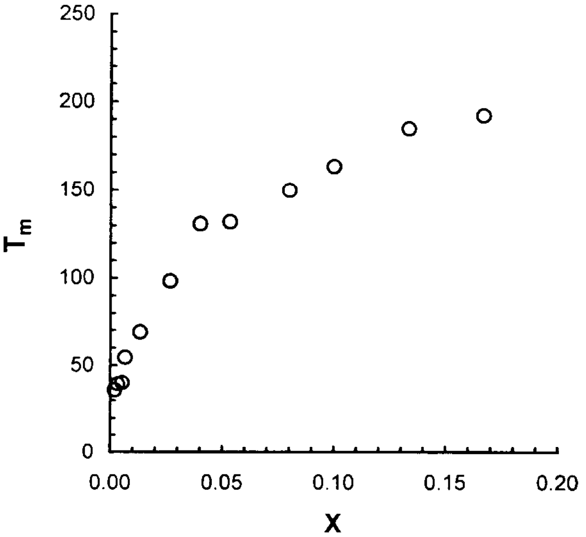

Fig. 3. The compositional dependence of the permittivity maximum temperature (T_m) at 1 kHz for the $(Sr_{1-1.5x}Bi_x)TiO_3$ samples.

3.3 Fitting to the Curie–Weiss law

The high temperature side of the curve of the reciprocal permittivity ($1/\varepsilon$) versus temperature (T) was fitted to the Curie–Weiss law. Typical curves of $1/\varepsilon$ versus T at 100 kHz are shown for the samples with $x = 0.002$, 0.0133, 0.10 and 0.167 in Fig. 5(a)–(d), respectively. The fitting Curie temperature (Θ), Curie constant (C) and ($T_{dev} - T_m$) for all compositions are summarised in Table 1, where ($T_{dev} - T_m$) is the temperature difference between the temperature T_{dev} at which there is a departure from the Curie–Weiss law and the temperature T_m at which the permittivity maximum occurs.

From Table 1, it can be seen that, Curie constants (C) and Curie temperatures (Θ) of 0.91×10^5 and 41.1 K for $x = 0.0033$, 0.9×10^5 K and 82 K for $x = 0.0133$, and 2.5×10^5 K and 150 K for $x = 0.167$ were obtained. The Curie constants are similar to those of pure SrTiO$_3$ ceramics.[2] For all the samples, an obvious deviation from the Curie–Weiss behaviour was observed, and the temperature difference ($T_{dev} - T_m$) increases with the Bi content. This implies that the temperature range, where the Curie–Weiss law is valid, narrows as the Bi content increases. Rather than following the Curie–Weiss law for temperatures higher than T_m, the temperature dependence of the reciprocal dielectric permittivity of the solid solutions with frequency dispersion exhibits a more complex behaviour. In what follows, this behaviour is characterised by using some empirical parameters that were used in previous works.[24,25]

3.4 Dielectric relaxation degree and diffuse degree

In order to characterise the dielectric dispersion and diffuseness of the phase transition, an empirical expression[24,25] was proposed:

$$1/\varepsilon - 1/\varepsilon_m = (T - T_m)^\gamma / C_1 \qquad (1)$$

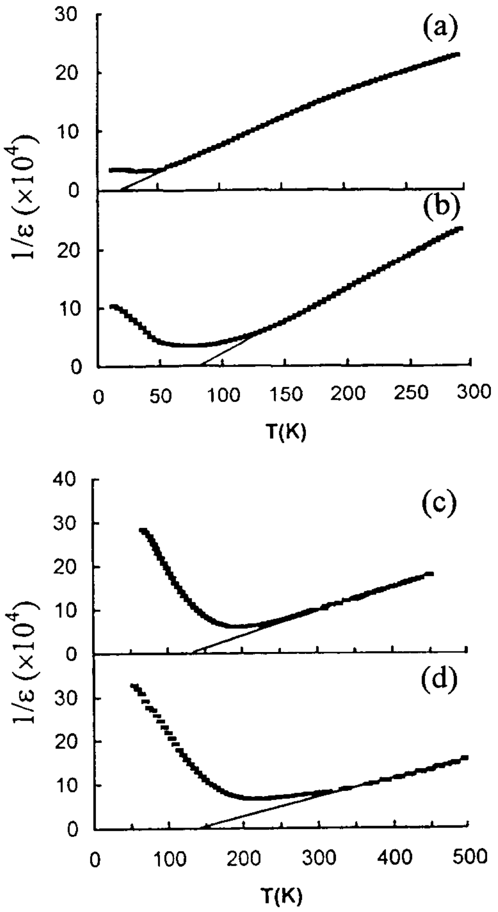

Fig. 5. The curve of the reciprocal permittivity versus temperature at 100 kHz for (a) $x=0{\cdot}002$, (b) $x=0{\cdot}0133$, (c) $x=0{\cdot}1$ and (d) $x=0{\cdot}167$. Solid lines: fitting curves: small bars, experimental data.

were γ and C_1 are assumed to be constant with $1<\gamma<2$. The limiting values $\gamma=1$ and $\gamma=2$ reduce the expression to the Curie–Weiss law valid, for the case of a normal ferroelectric and to the quadratic dependence valid for an idealised relaxor, respectively.

Typical fitting curves using expression (1) are shown for the samples with $x=0{\cdot}002$, $0{\cdot}0267$, $0{\cdot}08$ and $0{\cdot}167$ in Fig. 6(a)–(d), respectively. For example, with $x=0{\cdot}0267$, in the temperature range of $T-T_m=3$–100 K, the best fitting gives to γ the value $1{\cdot}94\pm0{\cdot}1$.

The diffuseness of the phase transition can be described by an empirical parameter ΔT, defined as

$$\Delta T = T_{0{\cdot}9\varepsilon m(100\ \mathrm{Hz})} - T_{\varepsilon m(100\ \mathrm{Hz})} \tag{2}$$

i.e. the difference between $T_{0{\cdot}9\varepsilon m(100\ \mathrm{Hz})}$ (the temperature corresponding to 90% of the permittivity maximum (ε_m) in the high temperature side) and $T_{m(100\ \mathrm{Hz})}$.

Moreover, the parameter ΔT^* defined as

$$\Delta T^* = T_{m(100\ \mathrm{kHz})} - T_{m(100\ \mathrm{Hz})} \tag{3}$$

has also been used to quantify the degree of relaxation behaviour.

The fitting data of γ, ΔT and ΔT^* obtained for all the samples are presented in Table 1. The compositional dependence of the parameters γ, ΔT and ΔT^* is shown in Fig. 7.

4 Discussion

For the quantum paraelectric $SrTiO_3$, its dielectric permittivity increased continuously with the decrease in temperature, until near 0 K with no evidence of a ferroelectric phase.[1,2] However, ferroelectricity can be induced by application of a dc bias or of an uniaxial pressure.[26] On the contrary, $BaTiO_3$ shows ferroelectric instability. Since they have similar crystal structure but $BaTiO_3$ has an ion of larger ionic radius (Ba^{2+}), the difference is probably due to an increase in the space allowed for the movement of Ti^{4+} ions. For Bi doped $SrTiO_3$, the increase in the lattice parameter with the increase of Bi content favours a similar situation. The appearance of a permittivity peak and of an obvious hysteresis loop indicates that ferroelectric instability also occurs in Bi doped $SrTiO_3$.

The diffuse nature of the dielectric permittivity peak of Bi doped $SrTiO_3$ and the dependence of its

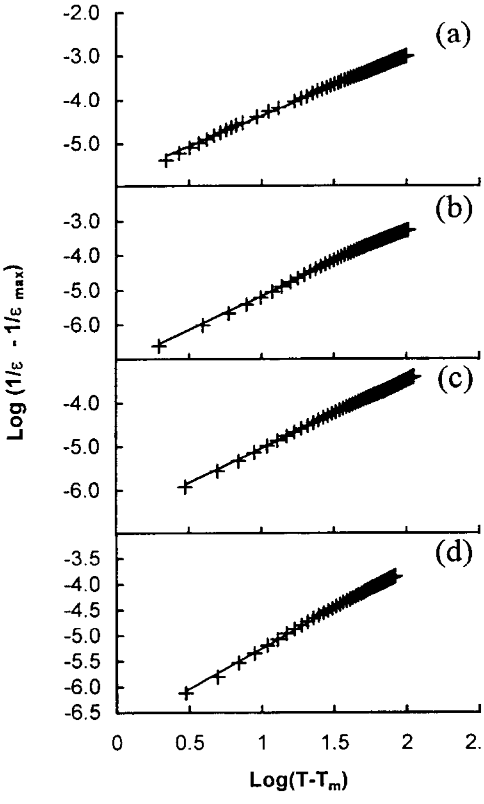

Fig. 6. The curve of the reciprocal permittivity versus temperature fitted by eqn (1) in the temperature range of ($T-T_m$) from ~3–5 to 100 K (T_m is temperature corresponding to the permittivity maximum) for $x=0{\cdot}002$, $0{\cdot}02$, $0{\cdot}1$ and $0{\cdot}167$. Solid lines: fitting curves; dots: experimental data.

Table 1. The Curie temperature (Θ), Curie constant (*C*) and the parameters $T_{dev} - T_m$, T_m, T_{dev}, ΔT, ΔT^* and γ for $0{\cdot}002 \leq x \leq 0{\cdot}167$

Composition		*x* = *0.002*	*x* = *0.0033*	*x* = *0.0053*	*x* = *0.0067*	*x* = *0.0133*	*x* = *0.0267*	*x* = *0.04*	*x* = *0.0533*	*x* = *0.08*	*x* = *0.1*	*x* = *0.133*	*x* = *0.167*
Curie–Weiss Law	Θ	26.9 K	41.1 K	51.4 K	66.9 K	82 K	100.2 K	114 K	113 K	115 K	117 K	140 K	150 K
	C	0·86×10^5 K	0.91×10^5 K	0·77×10^5 K	0·99×10^5 K	0·9×10^5 K	1·14×10^5 K	1·4×10^5 K	1·25×10^5 K	1·5×10^5 K	1·67×10^5 K	2·10^5 K	2·5×10^5 K
	$T_{dev} - T_m$	35·2 K	36·7 K	55 K	54·5 K	76 K	88·9 K	101 K	116·6 K	112 K	125 K	109 K	122 K
T_m(100 kHz)		44·8 K	48·3 K	49·9 K	58·5 K	74·3 K	106·1 K	154 K	153·4 K	166 K	190·9 K	221·6 K	228·8 K
T_{dev}(100 kHz)		80 K	85 K	105 K	113 K	150 K	195 K	255 K	270 K	280 K	300 K	310 K	340 K
ΔT(100 Hz)		7·4 K	10·7 K	11·4 K	12·8 K	16·7 K	19·3 K	30·1 K	27·8 K	35·3 K	39·7 K	47 K	55·2 K
ΔT^*		12 K	10·9 K	11·3 K	11·6 K	12·5 K	11·7 K	29·4 K	27·8 K	33·7 K	37·5 K	37 K	44·4 K
γ (10 kHz)		1·39	1·58	1·56	1·83	1·72	1·94	1·76	1·69	1·70	1·56	—[a]	1·55

[a] The fitting data is unstable.

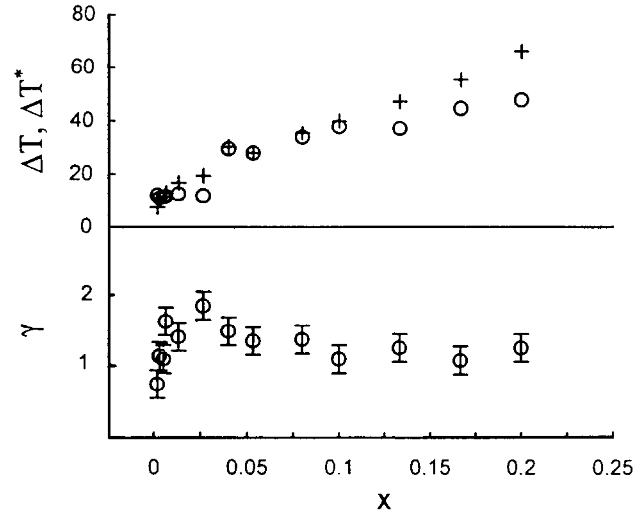

Fig. 7. The compositional dependence of the fitting parameters $\Delta T(+)$, ΔT^* (○) and γ from Table 1.

maximum value on the measuring frequency points to a behaviour similar to that of ferroelectric relaxors. As Bi content increases, the increase of temperature T_{dev} (Table 1), also points to larger departures from classic ferroelectric behaviour. The observed deviations from the Curie–Weiss law (Fig. 5) and the existence of a non-zero remanent polarisation (Fig. 4) far above the temperature of the maximum of dielectric permittivity, could indicate the existence of some polar clusters, in the so-called paraelectric regions. This phenomenon was also observed in several relaxors, namely, PMN, $Pb_{1-x}La_xZr_{1-y}Ti_yO_3$[27,28] and $(Sr_{1-x}Ca_x)TiO_3$,[4] where polar clusters were suggested to be a consequence of domain states induced by random fields.[4,21]

The similarities of the Bi doped $SrTiO_3$ system to ferroelectric relaxor systems are also apparent from the analysis of the ΔT, ΔT^* and γ parameters (Table 1 and Fig. 7). With the increase of Bi content, ΔT and ΔT^* increase, indicating a more diffuse and relaxed behaviour. The γ value is about 1·4 for $x = 0{\cdot}002$, showing the closest behaviour to the Curie–Weiss law. As Bi content increases, γ values rised quickly, reaching about 2 for $x = 0{\cdot}0267$, the value that describes the ideal relaxor ferroelectric behaviour.

It is seen from Fig. 7 that the variations of the parameters γ, ΔT and ΔT^* with Bi content show different tendencies for low and high Bi content regions. The crossover boundary is situated around $x = 0{\cdot}0267$. In fact, below about $x = 0{\cdot}0267$, the Bi doped $SrTiO_3$ system has been characterised as a quantum ferroelectric.[29]

The observed high permittivity, sharp transition and clear hysteresis loop confirm the appearance of ferroelectric instability in $SrTiO_3$ by doping with Bi; these observations support the viewpoint of Smolenskii *et al.*[14] However, the viewpoint of Skanavi *et al.*,[13] attributing the high dielectric permittivity peak to relatively large displacements of weakly bound ions (Ti^{4+} ions), is not unreasonable. In fact, the favourable crystal structure in Bi:$SrTiO_3$ with the Bi replacing Sr sites with its high polarizability and small ionic radius, the existence of Sr vacancies, and the increase in the lattice parameter, could make 'active' Ti ions more easy to displace, originating the ferroelectric instability observed.

On the other hand, with further increase in the Bi concentration, the distortion of the lattice would increase and the random electric field would be enhanced originating the crossover from a quantum ferroelectric to a ferroelectric relaxor. For example, the sample with $x = 0{\cdot}15$ displays a strong ferroelectric relaxor behaviour. This was why in the work of Smolenskii *et al.*[14] and of Gubkin *et al.*,[15,16] only a slim hysteresis loop was observed and correspondingly a very high electric field was needed.

5 Conclusions

The results of the present work can be summarised as follows:

1. Well defined permittivity peaks were detected in Bi doped $SrTiO_3$ with clear hysteresis loops even for small amounts of Bi doping.
2. (a) Significant departure from the Curie–Weiss law was detected above the temperature of the permittivity maximum (T_{m}). (b) Both the temperature (T_{dev}) at which there is a departure from the Curie–Weiss law and the difference ($T_{\mathrm{dev}} - T_{\mathrm{m}}$) increase with the increase of the Bi content.
3. (a) The parameter $\Delta T = T_{0{\cdot}9\varepsilon_{\mathrm{m}}(100\,\mathrm{Hz})} - T_{\varepsilon_{\mathrm{m}}(100\,\mathrm{Hz})}$ characterising the diffuseness of the phase transition and the parameter $\Delta T^* = T_{\mathrm{m}(100\,\mathrm{kHz})} - T_{\mathrm{m}(100\,\mathrm{Hz})}$ characterising the relaxation degree both increase with the Bi content. (b) The parameter γ characterising the diffuseness of the dielectric permittivity first increased quickly from ~1·4 for $x = 0{\cdot}002$ to 1·94 for $x = 0{\cdot}0267$, subsequently decreasing.

All these points indicate that the incorporation of Bi into the $SrTiO_3$ lattice induces the quantum paraelectric state of pure $SrTiO_3$ into a ferroelectric state. With further increase in Bi concentration, a ferroelectric relaxor state occurs.

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