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Control of conductivity and electric field induced strain in bulk $\text{Bi}_{0.5}\text{K}_{0.5}\text{TiO}_3\text{-BiFeO}_3$ ceramics

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High electrical conductivity is one of the main obstacles for advances of bulk BiFeO_3 ceramics in piezoelectric applications. Here, we demonstrate that the electrical conductivity of BiFeO_3 can be lowered by compositional modification with $\text{Bi}_{0.5}\text{K}_{0.5}\text{TiO}_3$ and further reduced by annealing in oxidizing or reducing atmospheres. These manipulations also allow for tailoring of other functional properties. In particular, we demonstrate that the electric field induced strain performance of bulk bismuth ferrite can be significantly improved by addition of 30% $\text{Bi}_{0.5}\text{K}_{0.5}\text{TiO}_3$ and thermal annealing in an inert atmosphere. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4869976>]

Bismuth ferrite, BiFeO_3 (BFO), possesses the essential prerequisites for high electromechanical performance in a wide temperature range, such as large bipolar strain of up to 0.36% (peak-to-peak value),¹ high spontaneous polarization ($\sim 90 \mu\text{C}/\text{cm}^2$),^{2,3} and high Curie temperature T_C ($\sim 820^\circ\text{C}$).^{3,4} However, practical exploration of these attractive features is limited by several factors including thermodynamic phase instability at the temperature range of ceramics processing,⁵ high conductivity,^{2,3,6} and large coercive field ($\sim 100 \text{ kV}/\text{cm}$) coupled with strong domain wall pinning effects.⁶ Owing to these limitations, application of pure bismuth ferrite in piezoelectric devices is challenging, but as a perovskite modifier it can be very prospective for compositional engineering. Recently, tailoring of BFO-related compositions for high-temperature electromechanical applications has been proposed for a wide range of solid solutions with lead-free $\text{Bi}_{0.5}\text{K}_{0.5}\text{TiO}_3$ (BFO-BKT),⁷⁻¹⁰ and further along morphotropic phase boundary towards lead titanate (BFO-BKT-PT).^{11,12}

The understanding of the origins of the undesired conductivity in BFO has been a topic of several theoretical^{13,14} and experimental¹⁵⁻¹⁷ studies. The first principle calculations^{13,14} based on density functional theory (DFT) considered formation of vacancies as main intrinsic point defects in pure bismuth ferrite. Depending on oxygen partial pressure ($p\text{O}_2$), the dominating point defects were suggested to be Bi and Fe vacancies at oxidizing conditions and Bi and O vacancies at reducing conditions. Thus, the conductivity of BFO was suggested to be of p -type with a tendency to decrease with oxygen partial pressure and become n -type.^{13,14} Recently, the p -type conductivity in BFO has been rationalized by polaron hopping that is strongly influenced by the oxidation state of Fe.^{16,17}

BKT is expected to possess n -type conductivity, analogously to $\text{Bi}_{0.5}\text{Na}_{0.5}\text{TiO}_3$,¹⁸ and therefore, can be an appropriate modifier to suppress p -type conductivity of bismuth ferrite.

In spite of several successful attempts to suppress conductivity and dielectric losses of pure bismuth ferrite by reducing the oxygen partial pressure during synthesis^{19,20} or

by appropriate donor dopants,^{15,21,22} no significant advance in exploration of high electromechanical performance of this material has been reported yet.

In this contribution, we provide an *in-situ* study of the conductivity of BKT-BFO ceramics during thermal cycles in oxidizing and reducing atmospheres. Additionally, we report ferroelectric softening of BFO ceramics by compositional modification with 30% BKT. We also demonstrate the suppression of the electronic conductivity by heat treatment of this ceramic in inert atmosphere, which enabled the investigation of the electric field-strain hysteresis at high electric field amplitudes ($\geq 100 \text{ kV}/\text{cm}$) and various frequencies.

Phase pure and dense ($\sim 98\%$) ceramics of $(1-x)\text{BKT-xBFO}$ or $\text{BKTF-x}(\%)$ were prepared by conventional solid state processing, as described elsewhere.²³ The sintering temperatures varied within $980\text{--}1080^\circ\text{C}$, depending on the composition; and the sintering time was 2 h.

Cylindrical samples with diameter $\sim 8 \text{ mm}$ and thickness $1\text{--}4 \text{ mm}$ with gold sputtered electrodes were subjected to impedance analysis using an Alpha-A analyzer (Novocontrol, Germany). The oxygen partial pressure and temperature were controlled using external gas sources and a Probostat (Norecs, Norway) testing chamber. The chamber was flushed with the controlled atmosphere with a flow rate of $\sim 20 \text{ ml}/\text{min}$. The impedance measurements were performed every 30 s during heating and cooling with a ramping of $2^\circ\text{C}/\text{min}$.

Estimation of the dc conductivity was performed at “low enough” frequencies (mainly at 1 Hz or below, depending on temperature), where the dispersion of the real part of ac conductivity forms a so-called “dc plateau.”

Prior to the measurements the samples were thermally annealed in dry synthetic air to eliminate the barrier-type Maxwell-Wagner relaxation recently reported for these materials.²⁴ The measurements along with the heating-cooling cycles were performed in the following order: first in nitrogen ($5.0, p\text{O}_2 \sim 10^{-5} \text{ atm}$), then in pure oxygen (5.0) atmospheres.

Characterization of the electric field-induced strain was performed on a BKTF-70 ceramic sample of 0.4 mm thickness, using an aixPES-Piezoelectric Evaluation System (aixACCT, Germany). The electroded sample was thermally annealed in nitrogen at 580°C for 2 h.

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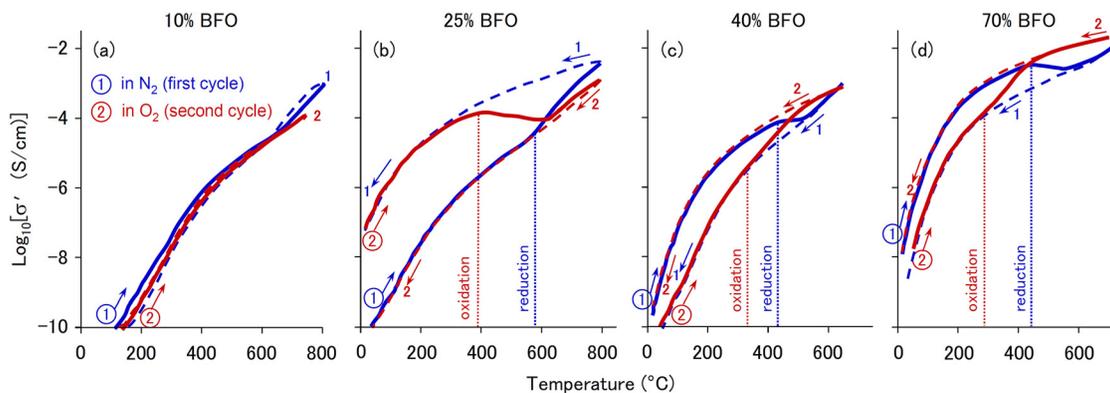


FIG. 1. Real part of ac conductivity (1 Hz) for BKTf- x ceramics during the sequence of thermal cycles in (1) nitrogen and in (2) oxygen: (a) $x = 10\%$; (b) $x = 25\%$; (c) $x = 40\%$; and (d) $x = 70\%$. The arrows show the heating and cooling directions.

The temperature dependencies of the conductivity during the sequence of heating-cooling cycles, first in nitrogen and then in oxygen, are shown in Fig. 1 for several BKTf-BFO compositions. The results demonstrate distinctive tendencies for changes in the concentration of the major charge carriers caused by the redox processes related to the partial pressure of oxygen (pO_2) in these two atmospheres.

All the ceramics examined in N_2 showed the rise of conductivity as the temperature reached $600^\circ C$. After the annealing cycles, the conductivity of BKTf-10 ceramics showed no significant change with respect to variation of temperature and pO_2 (Fig. 1(a)). The other materials, with a higher concentration of iron, showed notable and reversible variation of the conductivity $\sigma(pO_2)$ within the reduction-oxidation cycles (Figs. 1(b)–1(d)).

The BKTf-40 and BKTf-70 ceramics, along with the other BFO-rich compositions (not shown) demonstrated another distinctive feature—a local drop of conductivity when treated in nitrogen in the temperature range of 400 – $500^\circ C$ (Figs. 1(c) and 1(d)). This drop was followed by the rise of the conductivity above $600^\circ C$, as observed for all the other BKTf-BFO compositions.

Thermal treatment in pure oxygen returned the conductivity to the original value, demonstrating the complete reversibility of the $\sigma(pO_2)$ in the selected temperature ranges. Broad band dielectric spectroscopy performed at room temperature after each of the thermal cycles showed no barrier type of Maxwell-Wagner relaxation; thus, demonstrating

equilibration of the bulk material with respect to the partial pressure of oxygen above $\sim 700^\circ C$.²⁴

In order to elucidate the conditions required for establishing the equilibrium state in these ceramics, the kinetics of the dc conductivity relaxation has been investigated for the case of BKTf-70 ceramics. The conductivity relaxation under reducing atmosphere is shown in Fig. 2(a), while the reverse oxidation kinetics is shown in Fig. 2(b). Both processes appeared to be reasonably fast at temperatures above $500^\circ C$. Furthermore, the dc conductivity data measured upon cooling from $550^\circ C$ down to $400^\circ C$ in nitrogen in both dynamic ($2^\circ C/min$) and static (equilibrium) regimes did not show any significant divergence (Fig. 2(c)). Thus, the observed changes of conductivity resulting from thermal cycles with amplitudes above $600^\circ C$ in the reducing and oxidizing atmospheres are justified to ensure nearly equilibrium conditions for the experimental data illustrated in Fig. 1.

Fig. 2(d) illustrates the potential of the dc conductivity suppression in bulk BKTf-70 ceramics by thermal annealing under low pO_2 . At room temperature, this suppression may count almost 4 orders of magnitude. Similar variations of dc conductivity upon treatments at oxidation and reduction conditions have been reported for BFO thin films²⁵ and for barrier layers at the surface of BKTf- x ceramics ($x \geq 40\%$).²⁴

The observed changes of the conductivity can be rationalized by point defect equilibria. In the most general case for pure BFO, the principle of electroneutrality yields

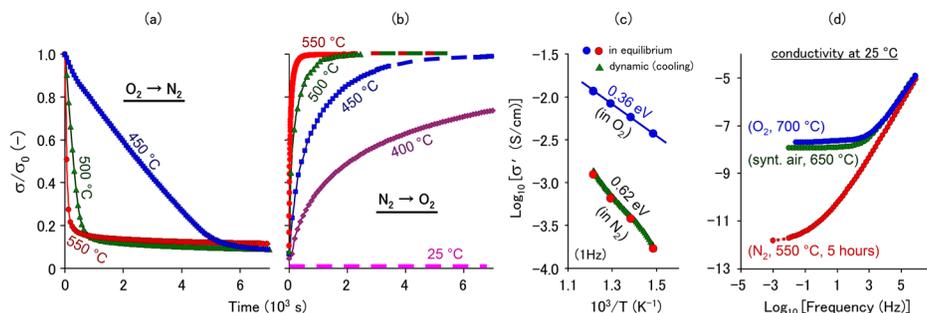


FIG. 2. (a)–(b) Electrical dc conductivity relaxation of BKTf-70 due to changes in the partial pressure of oxygen (σ_0 designates the dc conductivity in the equilibrium state with $pO_2 = 1$): (a) $O_2 \rightarrow N_2$ and (b) $N_2 \rightarrow O_2$. (c) Arrhenius plot of the bulk dc conductivity (σ' at 1 Hz) in the temperature range of 400 – $550^\circ C$. (d) The frequency dependence of the real part of the ac conductivity after annealing in various atmospheres (measurements performed at room temperature in dry synthetic air).

$$2[V_{O}^{**}] + p^* = 3[V_{Bi}^{***}] + 3[V_{Fe}^{***}] + n',$$

where $[V_{O}^{**}]$, $[V_{Bi}^{***}]$, and $[V_{Fe}^{***}]$ are the concentrations of O, Bi, and Fe vacancies. The origin of p^* and n' major charge carrier concentrations depends on the charge localization within Fe 3d and O 2p bond covalency. In iron containing perovskites p^* and n' have traditionally been associated with the $[Fe_{Fe}^{\bullet}]$ and $[Fe_{Fe}']$ minor point defects, whose plausible presence has been established for this class of materials.^{26–28} Thus, in the case of localized charge carriers, the electrical conduction can be realized through the polaron hopping mechanism.^{16,17,27,28} Alternatively, in recent DFT studies^{14,15,29} the possibility of band-like semiconductor behavior has been suggested for the same class of materials in which the case of delocalized charge carriers can also be feasible.²⁹

In our experiments, the strong dependence of the conductivity with respect to partial pressure of oxygen was observed for $x > 10\%$ BFO, see Figs. 1(b)–1(d). This gives support to the polaron hopping since the scenario assumes a marginal limit of iron concentration sufficient for electron percolation, and this margin is seemingly achieved only at $x > 10\%$.

The apparent increase of conductivity in all the ceramics under study above 600 °C in nitrogen is possibly a result of enhanced ionic mobility, as evidenced by Figs. 2(a) and 2(b). Only in the case of BKTF-25 this increase of conductivity was irreversible during the nitrogen annealing cycle, see Fig. 1(b), while at higher BFO content the conductivity decreased after processing in nitrogen up to 700 °C, see Figs. 1(c) and 1(d). These observations suggest that p -type conductivity is dominating in ceramics with higher BFO content ($x \geq 40\%$) and there is a tendency towards n -type conductivity under low pO_2 and lower BFO content. In a recent report,²⁴ we have demonstrated that the n -type conductivity may be induced in BKTF-70 ceramics by heat treatment in significantly lower pO_2 . The origin of the p - and n -types of conductivity can therefore be rationalized by the polaron hopping mechanism involving the $[Fe_{Fe}^{\bullet}]$ and $[Fe_{Fe}']$ minor point defects controlled by oxygen partial pressure. As mentioned before, the tendency of increasing n -type conductivity associated with $[Fe_{Fe}']$ along with decreasing the BFO content is principally limited by the marginal content of Fe sufficient for

charge percolation, and seemingly does not cover the case of BKTF-10 ceramics.

The activation energies of conductivity in BKTF-70 ceramics measured in oxygen and nitrogen are found to be 0.36 eV and 0.62 eV (Fig. 2(c)) and consistent with the values and tendencies previously reported for Ca-doped BFO ceramics and attributed to the p -type and ionic conduction mechanisms.¹⁶

The suppression of conductivity in the BKTF-70 ceramic was essential to enable characterization of the ferroelectric switching properties under high coercive fields. In our previous work,⁹ the electric field–strain hysteresis loops were reported only for the relaxor part of the system ($x < 70\%$), while no notable strain was induced in BKTF-70 ceramics at electric field amplitude of 50 kV/cm. In other works,^{7,10} relatively low piezoelectric strain was reported for BKTF-70 ceramics at amplitudes of electric field 80–100 kV/cm. It is worth mentioning that relatively weak strain responses were also reported for bulk BFO ceramics under such relatively low electric fields.^{6,19} In this study, we were able to characterize the field-induced strain in BKTF-70 ceramics at electric field amplitude of 130 kV/cm. The piezoelectric performance of this ceramics under this field amplitude is comparable and even superior with respect to pure BFO,¹ as shown in Figs. 3(a) and 3(b). Compared to BFO ceramics, the electric field induced stain in BKTF-70 ceramics has more regular shape with sharp edges and lower coercive value ($E_C \sim 75$ kV/cm, in good agreement with the previous report by Matsuo *et al.*⁷).

The strain–electric field hysteresis loops of the BKTF-70 ceramic were also characterized at various frequencies (1–20 Hz) and a fixed electric field amplitude of 100 kV/cm. The result is shown in Fig. 3(c). This ceramic demonstrated better frequency stability of the electric field induced strain compared to BFO (Fig. 3(a)). With respect to frequency of the driving field, the amplitude of negative strain in BKTF-70 ceramics appeared to be more stable than the positive maxima. This suggests that the strain relaxation is likely determined by domain wall motion dynamics, rather than by mechanisms of polarization switching. This is in contrast to the case of soft lead-based ferroelectrics demonstrating the opposite effect.^{30,31}

The origin of the strong frequency dependence and general instability of strain response in BFO was suggested to be

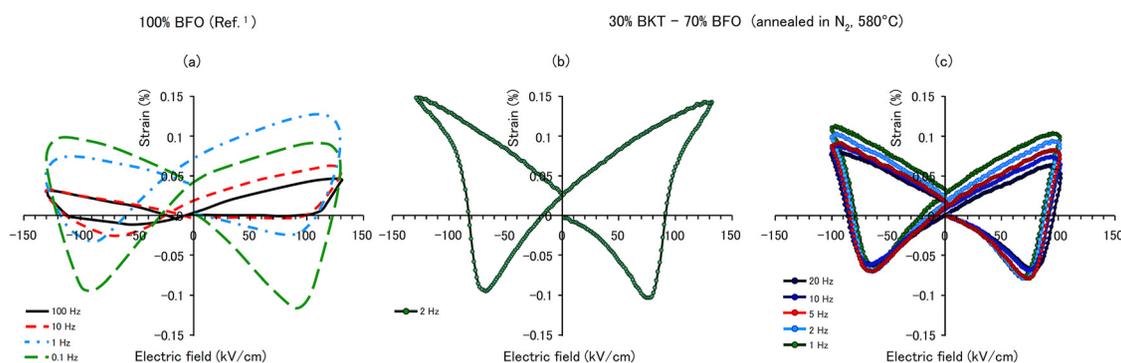


FIG. 3. Comparison of strain–electric field hysteresis loops for pure and modified BFO ceramics: (a) BFO ceramics. Reprinted with permission from T. Rojacz *et al.*, J. Am. Ceram. Soc. **94**, 4108 (2011). Copyright 2011 American Ceramics Society.¹ (b)–(c) BKTF-70 ceramics annealed in nitrogen at 580 °C characterized at 130 kV/cm and 2 Hz (b); and 100 kV/cm with various frequencies (c).

strong domain wall pinning by charged point defects,^{1,6} whose electrostatic rearrangement and collective pinning ability may be affected by application of strong ac electric field. This is a common feature of so-called “hard” ferroelectrics,^{32,33} among which BFO seemingly demonstrates a superior position in the “hardness” ranking. Our preliminary study indicates that hardening-softening of ferroelastic domains may occur in BKTF ceramics as well.

Ferroelectric softening achieved by modification of bismuth ferrite with 30% of BKT can presumably be caused by randomization of pinning centers and/or reduction of their concentration. Partial substitution of trivalent ions by aliovalent K and Ti causes structural disorder that is proposed to induce softening, i.e. enhancing the irreversibility of domain wall motion in ferroelectrics.^{34,35} In addition, the decrease of potentially mobile charge species associated with $[V_O^{\bullet\bullet}]$ and $[Fe_{Fe}^{\bullet}]$ along with BKT concentration implies reducing of domain wall pinning centers responsible for hardening. We shall note, however, that the role of Fe_{Fe}^{\bullet} in ferroelectric hardening has never been addressed and is presently not known.

In summary, a systematic study of the conductivity of BKT-BFO ceramics with respect to variation of the composition, temperature, and oxygen partial pressure has been performed. The ceramics showed a tendency of increasing p -type conductivity along with increasing BFO content. The behavior of the conductivity in ceramics with a relatively high BFO content ($x \geq 25\%$) was consistent with the hypothesis of polaron hopping mechanism, previously suggested for bismuth ferrite and related materials.^{16,17} Annealing of the BKTF-70 ceramics in nitrogen was shown to suppress the electrical conductivity, facilitating characterization of the large bipolar strain at high electric fields. The ceramics demonstrated better stability of the bipolar strain response compared to undoped bismuth ferrite. The improved electric field induced strain performance by modification of BFO ceramics with BKT was discussed in relation to ferroelectric “softening” of the material.

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