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## Grain size and lattice compatibility enhanced figure-of-merit in $Ba_{0.95}Ca_{0.05}Ce_{0.005}Zr_{x}Ti_{0.995-x}O_{3}$ material for pyroelectric energy conversion $\bigcirc$

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# Grain size and lattice compatibility enhanced figure-of-merit in $Ba_{0.95}Ca_{0.05}Ce_{0.005}Zr_xTi_{0.995-x}O_3$ material for pyroelectric energy conversion

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#### ABSTRACT

Phase-transforming ferroelectric materials have attracted significant attention due to their potential for energy conversion from waste heat. Here, we explore the impact of grain size and lattice compatibility on the energy conversion figure-of-merit (FOM) of a phase-transforming ferroelectric system  $Ba_{0.95}Ca_{0.05}Ce_{0.005}Zr_xTi_{0.995-x}O_3$  with Zr content ranging from 0.004 to 0.03. The results demonstrate that tuning grain size and lattice compatibility can significantly increase the FOM. The optimal composition  $Zr_{0.006}$  exhibits the highest FOM among its neighboring compositions, with a corresponding peak pyroelectric current density of 5.6  $\mu$ A/cm<sup>2</sup> generated from a temperature fluctuation of 30 °C at a temperature rate of 5 °C/s. This work provides a rational understanding of the effect of grain morphology and crystal structure on the pyroelectric properties for energy conversion.

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Waste heat accounts for up to half of the energy consumed in the past decades.<sup>1</sup> Low-grade waste heat at 80-150 °C dumped as temperature fluctuations rise significantly in electronic devices and supercomputers.<sup>2,3</sup> Reusing these dumped energies has become an emerging environmental task. Ferroelectric materials showing temperature dependent polarization upon applied field enable the pyroelectric effect,<sup>4</sup> which has been widely used in energy conversion from heat to electricity.5-8 The performance of the energy converter is usually characterized by the pyroelectric coefficient (dP/dT). During energy conversion, the pyroelectric property can be enhanced by orders of magnitudes as the ferroelectric materials undergoing first-order phase transformations.<sup>9,10</sup> The nature of this phase transformation is diffusionless and reversible. The transport properties such as polarization, pyroelectric coefficient, and heat capacity undergo an abrupt jump within a narrow temperature range. In contrast to the application in non-transforming regime,<sup>11</sup> many experiments show that the electric energy generation can be improved by two orders of magnitude when shifting the energy conversion regime to the vicinity of transition temperature.<sup>9,12–14</sup> Therefore, the energy conversion by transforming ferroelectrics is of particular interest to the waste heat harvesting from small temperature fluctuations.

The net energy converted from small temperature differences by phase-transforming ferroelectrics can be theorized by the bias-free thermodynamic cycle.<sup>12</sup> Different from conventional Olsen cycles,<sup>15</sup> the energy conversion device is not connected to an external biased electric field between the polar and non-polar phases. Figure-of-merit (FOM)<sup>12</sup> of the phase-transforming pyroelectric device is calculated as

$$FOM = \frac{\llbracket P \rrbracket \kappa}{\ell}, \tag{1}$$

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where  $[\![P]\!]$  is the polarization change before and after the phase transformation,  $\kappa = |dP/dT|_{\rm max}$  is the maximum pyroelectric coefficient during phase transformation, and  $\ell$  is the latent heat. It has been numerically and experimentally verified that the electric energy generated by the phase transformation is linearly proportional to FOM proposed in Eq. (1).<sup>12</sup> This FOM has been proven to be an effective merit to guide the material development in barium titanate and strontium titanate based ferroelectrics.<sup>9,16</sup> The material related transport properties such as polarization and heat capacity are quite sensitive to the grain morphology and phase boundaries of the ferroelectric materials.<sup>14,17,18</sup> The spontaneous polarization usually increases with the grain coarsening for the equiaxial ferroelectric polycrystals,<sup>19,20</sup> while

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the coupled transport properties and corresponding functional fatigue resistance during long-lasting phase transformation cycles closely rely on the lattice compatibility of the phase transformation between polar and non-polar structures.<sup>9,16,21,22</sup>

Here, we study the effect of Zr dopant on grain morphology and lattice compatibility in a lead-free ferroelectric system. By the floating zone method, we can manipulate the grain growth and tune the grain morphology of the material. With a systematic one-parameter doping procedure, the lattice parameters of both ferroelectric and paraelectric phases can be optimized to satisfy the primary condition of compatibility, i.e.,  $\lambda_2 = 1$ , where  $\lambda_2$  is the middle principal stretch of the transformation stretch tensor of the phase transformation. Through a systematic material development, we are able to understand the effect of doping element on figure-of-merit as well as the functional fatigue behaviors for the pyroelectric energy conversion device.

For the compositional variable  $x \in [0.004, 0.030]$  at%, a series of ferroelectric Ba<sub>0.95</sub>Ca<sub>0.05</sub>Ce<sub>0.005</sub>Zr<sub>x</sub>Ti<sub>0.995-x</sub>O<sub>3</sub> powder compound were prepared by the conventional ball milling and calcination, following the same procedure as our previous works.<sup>14,16</sup> Under 30 MPa hydrostatic pressure for 30 min, the fine powder was cold-pressed into a rod of 6 mm in diameter and 30 mm in longitudinal length for the floating zone process. We used the optical Infrared (IR) furnace (Quantum Design IRF11-001-00) to conduct the floating zone synthesis and microstructure engineering. The green body rods were vertically aligned and hung in the furnace chamber, locally melted by focused infrared light (nearly reaching 1700 °C in the specimen), followed by a gradual solidification through a linear pulling motion at 10 mm/h. This process ensures the completion of sintering with nearly equiaxial grains in the cross section area. We selected the same synthesis parameters for all tested compositions to reveal the effect of Zr dopant on grain growth by the floating zone method. After sintering and grain growth process, we obtained a one-parameter family of ferroelectric compounds with varying Zr composition. The chemical homogeneity was examined by the Energy-dispersive X-ray Spectroscopy analysis (SEM JOEL 6390).

In ferroelectric phase, the grain reflectivity is orientation dependent, characterized as the optical gradients by the polarized lightreflected differential interference microscope.<sup>23</sup> To reveal the grains and their boundaries, the specimen was polished and etched with 37% hydrochloric acid for 10 min. Figures 1(a)-1(d) show the grain morphology of the material system for compositions Zr<sub>0.004</sub>, Zr<sub>0.006</sub>, Zr<sub>0.010</sub>, and Zr<sub>0.030</sub>, corresponding to the statistical histogram of gain size in Figs. 1(e)-1(h). For each of samples, we took several images from various surface locations to obtain a sufficient sampling of grains. We observe that grain coarsening is achieved in all compositions with similar textures. The histograms in Figs. 1(e) and 1(g) exhibit a certain degree of asymmetry. In order to better capture the distribution of grain sizes, we use a lognormal distribution function to fit the histograms. This allowed us to determine the mean grain size as the expected value of the grain fineness for each composition. Figure 2(a) shows the trend of the mean grain size as the Zr content varies from 0.005 at. % to 0.03 at. %. The mean grain size exhibits high sensitivity to Zr compositions within the range of 0.005-0.015 at. %. As the Zr content increases, the mean grain size reduces rapidly and asymptotically converges to 60–70  $\mu$ m. As a comparison, the grains of ferroelectrics are usually below  $10 \,\mu m$  obtained by conventional solid-state reaction synthesis.<sup>24,25</sup> The floating zone method is widely used not only in single crystal growth of functional materials<sup>26,27</sup> but also to increase the grain size of ferroelectric materials.<sup>14,28</sup> By the grain morphology study, this work confirms that the floating zone method is an effective sintering approach to achieve the coarse equiaxial polycrystal lead-free ferroelectric oxides.



FIG. 1. Grain morphology analysis of ferroelectric Ba<sub>0.95</sub>Ce<sub>0.005</sub>Ce<sub>0.005</sub>Cr<sub>x</sub>Ti<sub>0.995</sub>-xO<sub>3</sub> system. Optical micrographs of (a) Zr<sub>0.004</sub>, (b) Zr<sub>0.006</sub>, (c) Zr<sub>0.10</sub>, and (d) Zr<sub>0.030</sub>, corresponding to (e)–(h) the statistical histogram of grain size. The solid line is the lognormal fitting, and the dashed line is the mean grain size.

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**FIG. 2.** Effect of Zr composition on (a) grain size, (b) pyroelectric coefficient and jump of polarization, and (c) figure-of-merit in the ferroelectric  $Ba_{0.95}Ca_{0.05}Ce_{0.005}Zr_xTi_{0.995-x}O_3$  system.

Among all developed compositions, we discovered that the maximum grain size is achieved at  $Zr_{0.006}$ , i.e., about 400  $\mu$ m. Figure 2(a) shows that the grain size of  $Zr_{0.006}$  is approximately 4 times larger than its neighboring compositions and appears visually coarse and isotropic. Interestingly, the ferroelectric properties and figure of merit (FOM) exhibit a similar trend with the grain size in this material system, as demonstrated in Figs. 2(b) and 2(c). Through the microstructure engineering, we enhance the FOM by a factor of 7, i.e.,  $3.5 \ \mu$ C<sup>2</sup>/J cm/°C, approaching to the maximum FOM value reported in ferroelectric single crystal.<sup>16,29</sup>

Figures 3(a) and 3(b) show the temperature dependent remnant polarization and pyroelectric coefficient, characterized by the ferroelectric analyzer (aix ACCT TF 2000E). The reversible phase transformation was measured by differential scanning calorimetry (TA DSC 250) in Fig. 3(c). We observed significant heat exchanges at 110 °C during both heating and cooling path. The transformation involves the changes of crystal structure accompanied by discontinuities in transport properties. The pyroelectric coefficient at the transition temperature is nearly 1.0  $\mu$ C/cm<sup>2</sup>K, an order of magnitude higher than that within the non-transforming regime. The remnant polarization in ferroelectric phase reaches 18  $\mu$ C/cm<sup>2</sup>, which is the highest reported value



FIG. 3. Temperature dependent (a) remnant polarization, (b) pyroelectric coefficient, and (c) heat flow in Ba<sub>0.95</sub>Ca<sub>0.05</sub>Ce<sub>0.005</sub>Zr<sub>0.006</sub>Ti<sub>0.989</sub>O<sub>3</sub>.

among BaTiO<sub>3</sub> based ferroelectric polycrystals.<sup>30–33</sup> This value almost approaches to the spontaneous polarization in single crystal BaTiO<sub>3</sub>.<sup>29</sup> In addition, the polarization abruptly jumps 17  $\mu$ C/cm<sup>2</sup> from ferroelectric to paraelectric phase at 120 °C, which makes this material a good candidate for energy conversion working below 150 °C. Other ferroelectric materials such as LiNbO<sub>3</sub> and LiTaO<sub>3</sub> have high transition temperature,<sup>34,35</sup> which can be used for energy conversion from high temperature environment.

We explored the crystallographic compatibility between ferroelectric and paraelectric phases for the optimized composition at  $Zr_{0.006}$ . The crystal structures are characterized to be tetragonal (*P4mm*) for ferroelectric phase at 90 °C and cubic (*Pm3m*) for paraelectric phase 120 °C by using X-ray powder diffraction (PAnalytical Diffractometer, Model X'per Pro). The lattice parameters were determined as a = 3.998 94 and c = 4.020 21 Å for tetragonal lattice and  $a_0 = 4.007$  26 Å for cubic lattice. The stretch tensor that distorts the cubic lattice to tetragonal lattice is calculated as

$$\mathbf{U} = \text{Diag}[(\alpha, \alpha, \beta)]. \tag{2}$$

Here, "Diag" denotes a 3 × 3 diagonal matrix generated by the diagonal components  $\alpha = a/a_0$  and  $\beta = c/a_0$ . In addition to the variant in Eq. (2), the ferroelectric martensite has 2 more variants, calculated as

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**FIG. 4.** Effect of Zr doping on lattice distortion. The parameters  $\alpha$  and  $\beta$  are the relative stretch components of the transformation stretch tensor in Eq. (2).

Diag[ $(\beta, \alpha, \alpha)$ ] and Diag[ $(\alpha, \beta, \alpha)$ ] corresponding to the twofold symmetry axes [101] and [011], respectively. The symmetry related variants form twins that should be compatible with the cubic austenite through an elastic transition layer. During a phase transformation, the elastic transition layer between two phases causes the accumulation of non-transforming defects, adversely affects the functionality of the transforming materials. This elastic layer can be completely eliminated if the Cofactor Conditions are satisfied by the transformation stretch tensor and its symmetry related twin variants.<sup>36</sup> In this case, we can explicitly calculate the Cofactor Conditions as

$$\text{CC1: } \alpha = 1, \tag{3}$$

CC2: 
$$\frac{(\alpha^2 - 1)(\alpha^2 - \beta^2)}{\alpha^2 + \beta^2} = 0.$$
 (4)

When the CC1 condition is met, the CC2 condition is satisfied simultaneously. For the cubic to tetragonal transformation, the relative lattice distortion can be measured as the relative length change between the polar ([001]) and non-polar ([100] or [010]) axes. The difference between  $\alpha$  and  $\beta$  in Eq. (2) underlies the degree of tetragonality in the polar phase. Literally, we expect the material possesses high tetragonality while maintaining good phase compatibility during the structural transformation. As shown in Fig. 4, the tetragonality presented as  $|\alpha - \beta|$  is maximized at the composition Zr<sub>0.006</sub>, while the CC1 is closest to 1. It shows that the crystal structure is also optimized for this material system.

The developed phase-transforming materials were fabricated to the pyroelectric capacitors, which were connected to an external pickup circuit for energy conversion. We use this experiment to demonstrate the pyroelectric current generation from temperature fluctuations in the vicinity of phase transformation regime. Through the demonstration, we show that the pyroelectric current is maximized at the martensitic transformation, which strongly correlates with the FOM, given in Fig. 5. It suggests that the FOM is a good indexer underlying the performance of such an energy conversion method. The energy conversion performance is demonstrated through the biasfree thermodynamic cycle, elaborated in our previous theoretical study.<sup>12</sup> The external electric circuit for picking-up the pyroelectric current follows the reference.9 In experiment, the pyroelectric capacitor is connected to a load resistor and a non-transforming capacitor. The system is initially charged by an external battery, and after fully charged, the external battery is removed. The electricity generation is measured by the recorded voltage on the load resistor. In our experiment, we maintained a relatively stable and steady heating/cooling rate, which we measured as dT/dt for all of the tested samples. The temperature profile and its fluctuation rate are shown in Figs. 5(a) and 5(b), respectively, which induce the pyroelectric current by phase transformations in Zr<sub>0.006</sub> and Zr<sub>0.020</sub>, during both heating/cooling paths. Under a similar heat exchange rate (i.e., 4 - 6 °C) between 100 and 130 °C, the Zr<sub>0.006</sub> device generated approximately twice as much electricity as the  $Zr_{0.020}$  sample. Figure 5(c) shows the linear correlation between the normalized pyroelectric current density and their figureof-merit for all tested compositions. Among all of the Zr-doped BaTiO<sub>3</sub> ferroelectric materials that were developed, the energy conversion capacitor with the  $\mathrm{Zr}_{0.006}$  dielectric layer demonstrated the highest FOM, as illustrated in Fig. 5(c). This capacitor generated a pyroelectric current density of 5.6  $\mu$ A/cm<sup>2</sup>, which is the highest among all of the tested devices developed in our work under identical thermal exchange conditions. The above result confirms that the FOM is an effective index to guide the material development for such an energy conversion technique.

In this study, we investigate the impact of Zr doping on grain morphology and phase compatibility in the  $Ba_{0.95}Ca_{0.05}Ce_{0.005}Zr_xTi_{0.995-x}O_3$  material system. Our findings reveal that the size of grains varies with Zr



**FIG. 5.** Demonstration of pyroelectric energy conversion in (a)  $Zr_{0.006}$  and (b)  $Zr_{0.020}$ . The time dependent heating and cooling rate is presented, and the heating/cooling rate corresponding to the peak of electricity generation is labeled. (c) Correlation between figure-of-merit and generated pyroelectric current density in the ferroelectric Ba<sub>0.95</sub>Ca<sub>0.05</sub>Ce<sub>0.005</sub>Zr<sub>x</sub>Ti<sub>0.995-x</sub>O<sub>3</sub> system. The dashed line is the linear fitting between normalized peak pyrocurrent density and figure-of-merit.

content, with an increased grain size observed in the  $Zr_{0.006}$  composition. This may explain why previous works can easily grow a single crystal for this complex composition. Additionally, we discovered that  $Zr_{0.006}$  exhibits the most compatible structural transformation from tetragonal phase to cubic phase among the series of compositions. As a result, the figure of merit (FOM) is significantly improved at both optimized grain size and lattice compatibility, which makes  $Zr_{0.006}$  an ideal candidate for the energy conversion directly from heat fluctuations near 110 °C. This work not only identifies a promising material with enhanced energy conversion performance but also provides a rational approach to developing high-performance phase-transforming ferroelectric materials for electricity generation from waste heat with minimal temperature fluctuations.

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#### AUTHOR DECLARATIONS

#### **Conflict of Interest**

The authors have no conflicts to disclose.

#### Author Contributions

Ruhao Huang: Investigation (equal); Validation (equal). Zeyuan Zhu: Validation (equal). Ruiheng Geng: Validation (equal). Ka Hung Chan: Visualization (equal). Xian Chen: Funding acquisition (equal); Validation (equal); Writing – review & editing (equal). Chenbo Zhang: Conceptualization (equal); Funding acquisition (equal); Methodology (equal); Visualization (equal); Writing – original draft (equal).

#### DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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