

反应闪烧技术制备 $\text{BiFeO}_3\text{--BaTiO}_3$ 陶瓷

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摘 要: $\text{BiFeO}_3\text{--BaTiO}_3$ 陶瓷的低温快速烧结致密化有助于抑制 Bi 元素的挥发, 降低能耗以及快速筛选出具有优异电学性能的 $\text{BiFeO}_3\text{--BaTiO}_3$ 基陶瓷材料。在 200 V/cm 的直流电场和 280 mA 的限制电流作用下, 通过反应闪烧 Bi_2O_3 、 Fe_2O_3 、 TiO_2 和 BaCO_3 混合粉体, 能够在 415 °C 的炉温下, 30 s 内获得具有良好铁电及压电性能的致密 $\text{BiFeO}_3\text{--BaTiO}_3$ 陶瓷。反应闪烧过程中, 固相化学反应和烧结致密化同时发生; 限制电流值的大小对样品的相变和致密化程度有重要影响。反应闪烧技术可通过一步闪烧陶瓷材料的前驱体混合粉末而获得单相且致密的陶瓷材料, 为陶瓷材料的快速制备提供了一个新的途径。

关键词: 铁酸铋-钛酸钡; 无铅压电陶瓷; 反应闪烧; 相变; 致密化

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Reactive Flash Sintering of $\text{BiFeO}_3\text{--BaTiO}_3$ Ceramics

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Abstract: Rapid preparation of dense $\text{BiFeO}_3\text{--BaTiO}_3$ lead-free piezoelectric ceramics at low temperatures is beneficial to preventing the Bi volatilization, reducing the energy consumption and developing novel materials with some unique properties. In this paper, dense $\text{BiFeO}_3\text{--BaTiO}_3$ ceramics with good ferroelectric and piezoelectric properties were prepared in an electric field of 200 V/cm with a current limit of 280 mA via reactive flash sintering of multiphase precursor powders (*i.e.*, Bi_2O_3 , Fe_2O_3 , TiO_2 and BaCO_3) at 415 °C for 30 s. The solid-state reaction in sintering occurs during the flash event. The current limit has an effect on the phase transformation and densification. It is indicated that the reactive flash sintering technology could prepare the single-phase and dense ceramic material through flash sintering of starting precursors in one step, thus providing an effective approach for the rapid production of ceramic materials.

Keywords: bismuth ferrite-Barium titanate; lead-free piezoelectric ceramics; reactive flash sintering; phase transformation; densification

1 Introduction

Lead-based piezoelectric ceramics, such as $\text{Pb}(\text{Zr,Ti})\text{O}_3$, $\text{Pb}(\text{Mg,Nb})\text{O}_3\text{--PbTiO}_3$, *etc.*, are widely used in various electromechanical devices due to their superior piezoelectric properties and high Curie temperatures^[1-3]. However, lead-based materials are restricted because of the toxicity of lead oxide. Many countries have enacted the legislation to restrict the use of lead containing compounds and alloys in commercial products^[4]. It is thus necessary to develop lead-free piezoelectric ceramics with biocompatibility and friendly environment

instead of the lead-based ones. Some lead-free piezoelectric ceramics, such as BaTiO_3 , $(\text{K,Na})\text{NbO}_3$, $(\text{Bi,Na})\text{TiO}_3$ and $\text{BiFeO}_3\text{--BaTiO}_3$ are investigated in recent years^[5-7].

As one of the lead-free piezoelectric ceramics, $\text{BiFeO}_3\text{--BaTiO}_3$ solid solutions have been attracted recent attentions as promising candidates for high-temperature piezoelectric applications due to their high Curie temperature (even over 580 °C) and good piezoelectric properties^[8-10]. However, it is difficult to prepare dense and pure-phase $\text{BiFeO}_3\text{--BaTiO}_3$ ceramics by conventional sintering methods because Bi element is

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easily evaporated at high sintering temperatures, resulting in the formation of second phases like $\text{Bi}_2\text{Fe}_4\text{O}_9$ and $\text{Bi}_{25}\text{FeO}_{39}$ ^[11–13]. The excessive amount of Bi should be added to compensate the loss of Bi, thus complicating the preparation process. Hence, it is essential to develop the effective methods available for rapid preparation of BiFeO_3 – BaTiO_3 ceramics at low temperatures for a short time.

Flash sintering is a novel sintering technology, by which ceramic green compacts can be densified in several seconds or minutes at low temperatures^[14]. This technology is utilized to prepare some ceramic materials containing highly volatile species, such as KNbO_3 , NaNbO_3 and $(\text{K},\text{Na})\text{NbO}_3$ due to the rapid densification process and low sintering temperatures. The evaporation of K and Na elements is found to be greatly reduced, thus maintaining the stoichiometry of the ceramics^[15–17]. Recent work demonstrates that the chemical reaction and

sintering both occur concurrently during flash sintering process, which is known as reactive flash sintering^[18]. As a result, dense and single-phase ceramics can be prepared in one step by reactive flash sintering of their elemental constituents, skipping the preparation process for starting powders. Some ceramics have been fabricated by reactive flash sintering, such as MgAl_2O_4 ^[19], $(\text{K},\text{Na})\text{NbO}_3$ ^[17], $\text{Pb}(\text{Zr},\text{Ti})\text{O}_3$ ^[20], $\text{Li}_{0.5}\text{La}_{0.5}\text{TiO}_3$ ^[21], $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ ^[22], $\text{Mg}_{0.2}\text{Ni}_{0.2}\text{Co}_{0.2}\text{Cu}_{0.2}\text{Zn}_{0.2}\text{O}$ ^[23] and $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$ ^[24].

In this paper, dense BiFeO_3 – BaTiO_3 ceramics were synthesized by a reactive flash sintering method with multiphase precursor powders. The phase transformation and densification behaviors during the reactive flash sintering in the DC electric fields of 150–300 V/cm were investigated. In addition, the ferroelectric and piezoelectric properties of reactive flash-sintered BiFeO_3 – BaTiO_3 ceramics were also analyzed.

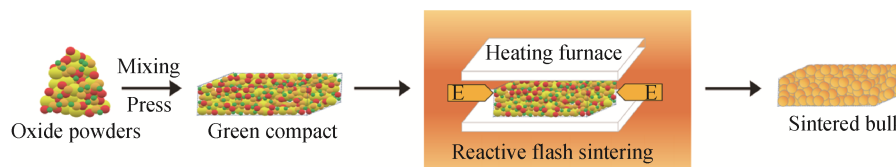


Fig. 1 Schematic of reactive flash sintering process for preparation of BiFeO_3 – BaTiO_3 ceramics

2 Experimental

2.1 Materials

Analytical grade (Macklin Chemical, China) bismuth oxide (Bi_2O_3 , 99.9%), iron oxide (Fe_2O_3 , 99%), titanium dioxide (TiO_2 , 99.9%) and barium carbonate (BaCO_3 , 99.9%) powders were used as starting materials.

2.2 Synthesis procedure

These powders corresponding to the 0.7BiFeO_3 – 0.3BaTiO_3 compositions were mixed by ball milling for 24 h with zirconia balls in absolute ethyl alcohol. After drying at 80 °C, the multiphase precursor powders were pressed into dog-bone shaped green compacts with a gage length of 20 mm, a width of 3 mm and a thickness of about 1 mm using uniaxial cold press at 300 MPa. The experiments for reactive flash sintering were performed in a tubular furnace at a ramp rate of 10 °C/min. An electric field was applied by connecting a DC power supply (HPS 0614, Lanyi, China) to the green compacts with platinum wires. A digital multimeter (DMM 4040, Tektronix, USA) was used to measure the current flowing through the sample. When flash event occurred, the current increased rapidly to a preset maximum value and then was maintained for 30 s. Afterwards, the power supply and furnace were turned off. The ceramic product was finally obtained.

2.3 Characterizations

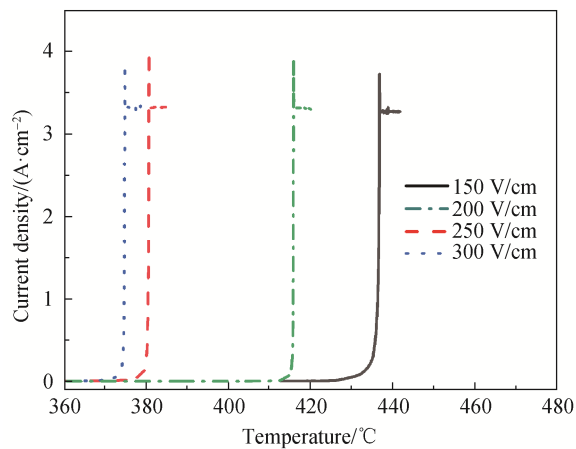
The phase compositions of the samples were characterized by a model Bruker D8 X-ray diffractometer (XRD, Davinci Co., Germany) with Cu-K radiation (40 kV, 60 mA). The scan was performed at a scan rate of 5 (°)/min with a step size of 0.02°. The microstructures of

samples were examined by a model S-4800 field emission scanning electron microscope (FESEM, Hitachi Co. Ltd., Japan) with an acceleration voltage of 15 kV. The element composition analysis of samples was performed by a model XRF-1800 X-ray fluorescence spectroscope (XRF, Shimadzu Co. Ltd., Japan). The densities of the flash-sintered bulks were measured by Archimedes' method. The relative density was calculated with respect to the theoretical density of 0.7BiFeO_3 – 0.3BaTiO_3 ceramics, which was calculated based on the rule of mixture of BiFeO_3 (theoretical density of 8.42 g/cm³) and BaTiO_3 (theoretical density of 6.02 g/cm³). The average grain sizes of samples were determined from the SEM images using a software named Nano Measurer 1.2, and more than 100 grains randomly selected were analyzed. To measure the electrical properties, the flash-sintered samples were polished, coated with silver paste, and heated at 600 °C for 15 min to form electrodes. The polarization hysteresis loops were measured using a model aixACCT TF Analyzer 3000 ferroelectric analyzer unit (aixACCT, Germany) at 1 Hz. The piezoelectric properties were measured using a model ZJ-3A quasi-static piezoelectric coefficient testing meter (Institute of Acoustics, Chinese Academy of Sciences, China).

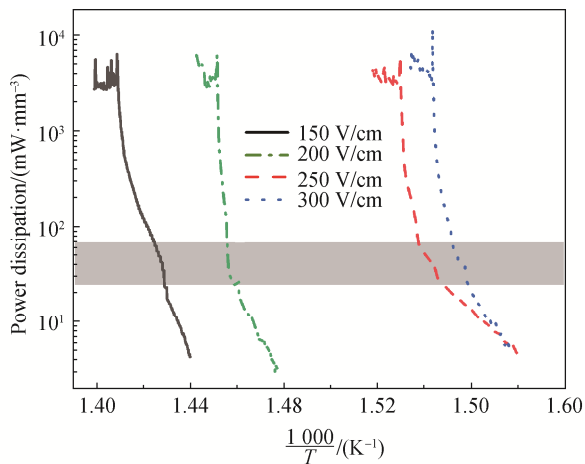
3 Results and discussion

Fig. 2 shows the current density and Arrhenius plot of the power density as a function of furnace temperature for green compacts processed in electric fields of 150–300 V/cm with a current limit of 100 mA. For all the samples, the current density increases rapidly to a peak

value of approximately 3.33 A/cm^2 as the furnace temperature increases to a certain value, as shown in Fig. 2(a), which signals the flash event^[14]. The furnace temperature for onset of flash is 434, 415, 379 and 373°C



(a) Current density



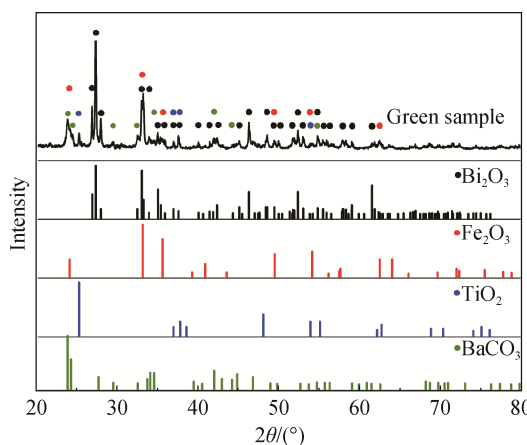
(b) Power dissipation

Fig. 2 Current density and Arrhenius plot of the power dissipation as a function of furnace temperature for samples in various electric fields with a current limit of 100 mA

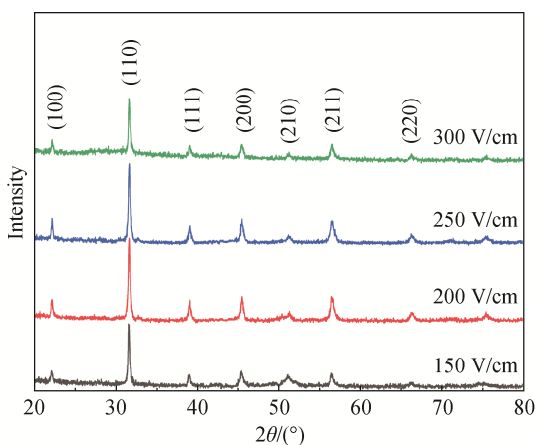
Fig. 2(b) shows the Arrhenius plots of the power dissipation in samples at various electric fields. There is an evident transition from linearity to highly nonlinearity in power dissipation, further confirming the flash event under these experimental conditions^[21]. Moreover, the power dissipation transition occurs in the range of $16\text{--}48 \text{ mW/mm}^3$, which is exactly consistent with the results for previous literature^[28–29]. This phenomenon indicates that

the Joule heating plays a critical role in initiating the flash event^[29].

Fig. 3(a) shows the XRD patterns of the green compact. Clearly, there are the diffraction peaks of Bi_2O_3 (PDF#41–1449), Fe_2O_3 (PDF#33–0664), TiO_2 (PDF#21–1272) and BaCO_3 (PDF#05–0378), without any trace of other phases. This indicates that green compact consists of Bi_2O_3 , Fe_2O_3 , TiO_2 and BaCO_3 .



(a) Green compact



(b) Samples obtained by reactive flash sintering

Fig. 3 XRD patterns of green compact and samples obtained by reactive flash sintering in different electric fields with a current limit of 100 mA

Fig. 3(b) shows the XRD patterns of the samples obtained after the flash event in different electric fields of 150–300 V/cm with a current limit of 100 mA. All the samples exhibit a typical pseudo-cubic perovskite phase (ABO_3) without any impurity phases. It is indicated that the starting precursors react completely to form a stable solid solution of $\text{BiFeO}_3\text{--BaTiO}_3$ during the flash event. According to the literature^[12–30], the formation of single

perovskite phase of $\text{BiFeO}_3\text{--BaTiO}_3$ always needs high calcination temperature of 800°C and long holding time of 5–6 h as using the conventional solid-state method. In contrast, upon the application of the electric field of 150–300 V/cm, a pure $\text{BiFeO}_3\text{--BaTiO}_3$ solution can be obtained at low temperatures in the range of $373\text{--}434^\circ\text{C}$. Also, the holding time tremendously decreases from several hours to 30 s. Hence, the phase transformation is

greatly accelerated in the flash event.

Fig. 4(b) shows the current density as a function of furnace temperature in a constant electric field of 200 V/cm with different current limits (50–200 mA). Clearly, the flash event occurs at 413, 415, 411 and

412 °C when the current limit is 50, 100, 150 and 200 mA, respectively. The flash sintering temperature changes slightly at different current limits. These trivial differences could be due to the sample-to-sample variation.

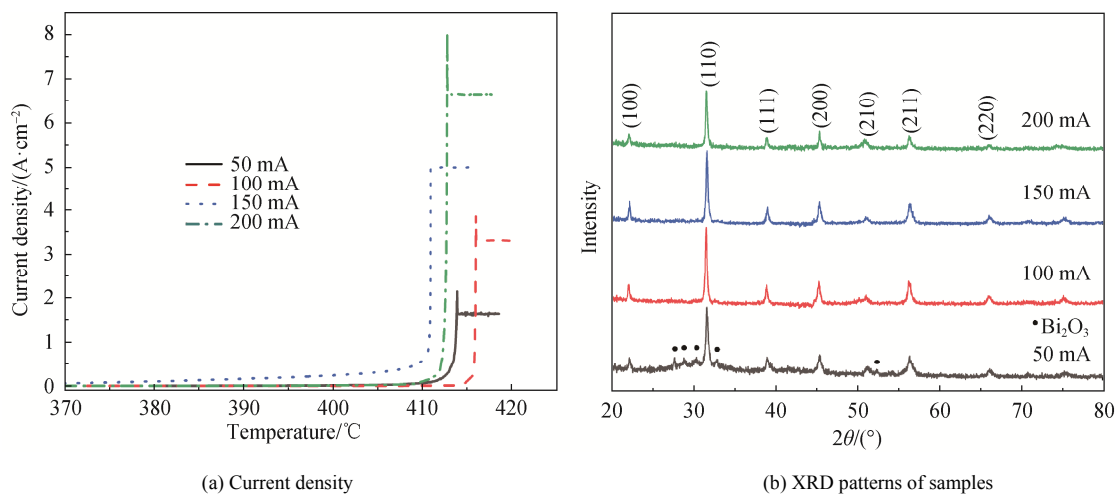


Fig. 4 Current density as a function of furnace temperature for samples in an electric field of 200 V/cm with different current limits, the corresponding XRD patterns of resulted samples

Fig. 5 shows the SEM images of flash-sintered samples under different experiment conditions. Clearly, the samples obtained in different electric fields of 150–300 V/cm with a current limit of 100 mA shows some porosity areas, indicating that these samples are not well densified under these experiment conditions. However, the microstructure of samples becomes denser with the increase of current limit. The relative density

of samples is 90.8%, 96.3%, and 98.2% when the current limit increases to 150, 200, and 280 mA, respectively. Meanwhile, the grain size of samples increases (see Table 1). It is indicated that the densification and grain growth both depend strongly on the current limit in the flash event. The higher the current limit is, the higher the density and the larger the grain size will be.

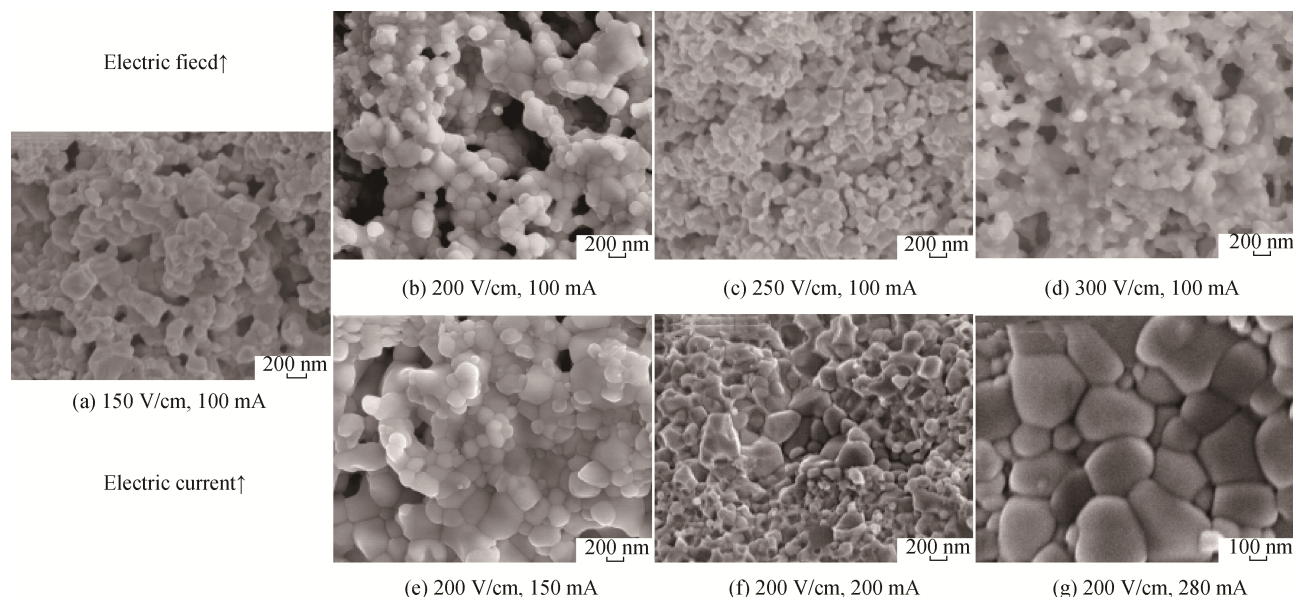


Fig. 5 SEM images of samples prepared under different experiment conditions

X-ray fluorescence (XRF) was performed to determine the elemental composition of samples. For the green samples, the Bi/Fe atomic ratio is 1/0.99. For the bulks obtained after the flash event under electric field of

200 V/cm and current limit of 280 mA, the Bi/Fe atomic ratio remains at 1/0.99, indicating that there is no Bi loss after the flash. According to the above results, BiFeO₃–BaTiO₃ ceramics can be rapidly prepared at low

temperatures by reactive flash sintering of multiphase precursor powders, and skipping the process for synthesis of BiFeO₃-BaTiO₃ powders. The ultrafast synthesis and densification at low temperatures are beneficial to preventing the Bi volatilization and reducing the energy consumption. Moreover, the ability of rapidly producing BiFeO₃-BaTiO₃ ceramics will enable the acceleration of materials screening rate, in favor of discovery of BiFeO₃-BaTiO₃ based ceramics with some unique performances.

Table 1 Relative densities and average grain sizes of samples prepared by reactive flash sintering at 200 V/cm and different current limits

Current limit /mA	Relative density /%	Average grain size/nm
100	86.5	169
150	90.8	247
200	96.3	264
280	98.2	306

It is intriguing that the solid-state reaction and densification occur concurrently in reactive flash sintering. Since the sample temperature is higher than the furnace temperature in flash event due to the power dissipation in sample, we consider that the Joule heating could play an effective role in reactive flash sintering. The real sample temperature during the flash event is estimated by^[31]

$$T = \left[T_0^4 + \frac{W}{A\varepsilon\sigma} \right]^{1/4} \quad (1)$$

where T is the real sample temperature; T_0 is the furnace temperature for flash; W is the steady-state power

dissipation; A is the surface area of sample ($1.66 \times 10^{-4} \text{ m}^2$); ε is the emissivity (assumed to be in the range of 0.8–1.0); σ is Stefan's constant ($5.67 \times 10^{-8} \text{ W/m}^2 \cdot \text{K}^4$). For the samples flash sintered at 200 V/cm with various current limits, the real temperatures are much higher than the furnace temperature for flash (around 415 °C), as shown in Table 2. Moreover, the real sample temperature increases with the increase of the current limit. This can explain the increased density and grain size with the current limit (see Table 1). The real sample temperature at 200 V/cm and 200 mA is 971 °C ($\varepsilon=1$) or 1 036 °C ($\varepsilon=0.8$), which is comparable to the conventionally used sintering temperatures^[8–32]. Although this temperature is high enough to densify BiFeO₃-BaTiO₃ ceramics, the holding time is rather short (*i.e.*, only 30 s) to produce the dense products. In addition, for the synthesis of BiFeO₃-BaTiO₃ powder, a mixture of Bi₂O₃, Fe₂O₃, TiO₂ and BaCO₃ should be calcined at 800 °C for 4 h^[32]. In contrast, the synthesis and densification of BiFeO₃-BaTiO₃ ceramics are accomplished for just 30 s. We believe that the Joule heating alone cannot account for the rapid solid-state reaction and ultrafast densification in the reactive flash sintering. Chaim^[33] claimed that liquid film is formed locally at the particle contacts due to the local Joule heating. The ionic diffusion kinetics was greatly enhanced through the liquid film, thus leading to the fast chemical reaction and sintering. However, the in-depth investigation of fundamental mechanism in reactive flash sintering should be needed in a future work.

Fig. 6(a) shows the room-temperature polarization versus electric field (P - E) hysteresis loop of the reactive

Table 2 Real sample temperatures in reactive flash sintering at 200 V/cm and different current limits

Current limit/mA	Power dissipation at steady state/(mW·mm ⁻³)	Furnace temperature for flash/°C	Sample temperature/°C	
			$\varepsilon=1$	$\varepsilon=0.8$
50	186	413	816	869
100	243	415	881	939
150	321	411	954	1 018
200	341	412	971	1 036

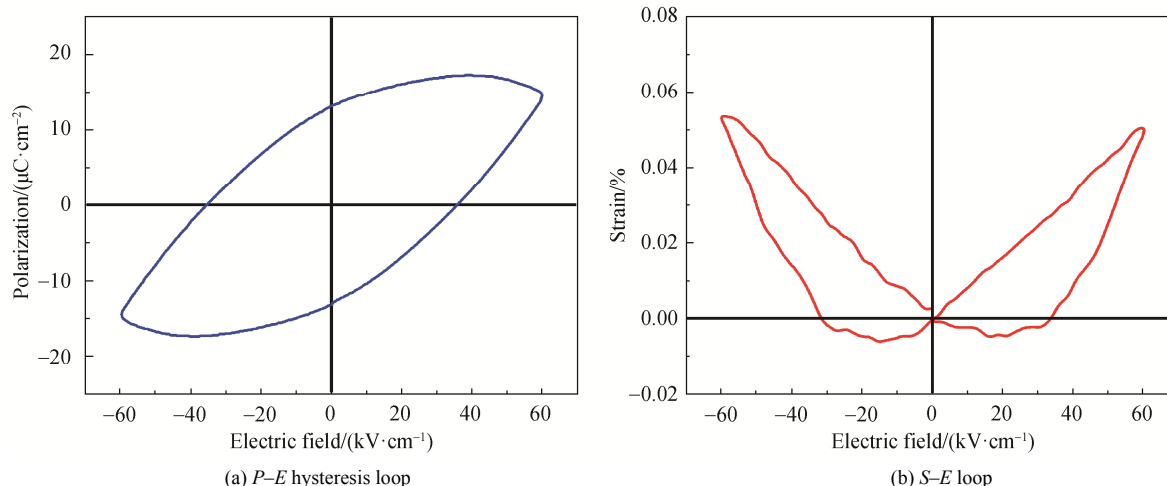


Fig. 6 P - E hysteresis loop (a) and S - E loop (b) for BiFeO₃-BaTiO₃ ceramics flash-sintered at 200 V/cm and 280 mA

flash-sintered bulk at 200 V/cm and 280 mA, which is measured at 60 kV/cm and 1 Hz. The sintered bulk exhibits the classic ferroelectric hysteresis, implying a good ferroelectricity. The maximum polarization P_m , remanent polarization P_r and coercive field E_c are 17.78 $\mu\text{C}/\text{cm}^2$, 13.07 $\mu\text{C}/\text{cm}^2$ and 35.85 kV/cm, respectively. Fig. 6(b) shows the bipolar strain *versus* electric field (S – E) curve of the reactive flash-sintered bulk at 200 V/cm and 280 mA. Clearly, the symmetric butterfly-shaped hysteresis loop characteristic of ferroelectricity appears. A maximum strain of 0.055% is obtained. In addition, the piezoelectric coefficient d_{33} is 148 pC/N for this flash-sintered bulk. Compared to BiFeO₃–BaTiO₃ ceramics prepared by conventional methods, the reactive flash-sintered BiFeO₃–BaTiO₃ ceramics have the slightly weaker ferroelectric and lower piezoelectric properties.

4 Conclusions

Dense BiFeO₃–BaTiO₃ ceramics were prepared by reactive flash sintering of multiphase precursor powders (*i.e.*, Bi₂O₃, Fe₂O₃, TiO₂ and BaCO₃) at low temperatures for a short holding time of 30 s. The solid-state reaction in sintering occurs in flash event. The current limit had an influence on the phase transformation and densification in the reactive flash sintering. The results obtained indicated that the reactive flash sintering could be utilized to rapidly fabricate the ceramics containing volatile ingredients from the elemental powders, thus skipping the synthesis process for stoichiometric starting powders, reducing the energy consumption, and developing the novel materials with some unique properties.

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