CuO added $Pb_{0.92}Sr_{0.06}Ba_{0.02}(Mg_{1/3}Nb_{2/3})_{0.25}(Ti_{0.53}Zr_{0.47})_{0.75}O_3$ ceramics sintered with Ag electrodes at 900 °C for multilayer piezoelectric actuator*

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CuO added $Pb_{0.92}Sr_{0.06}Ba_{0.02}(Mg_{1/3}Nb_{2/3})_{0.25}(Ti_{0.53}Zr_{0.47})_{0.75}O_3$ ceramics were studied to prepare high-quality multilayer piezoelectric actuators with pure Ag electrodes at 900 °C. CuO addition not only reduced the sintering temperature significantly from 1260 °C to 900 °C but also improved the ceramic density to 7.742 g/cm^3. The 0.7 wt.% CuO added ceramic sintered at 900 °C shows the remnant polarization (P_r) of 40 μC/cm^2, 0.28% strain at 40 kV/cm, and the piezoelectric coefficient (d_{33}) of 630 pC/N. This ceramic shows a strong relaxor characteristic with a Curie temperature of 200 °C. Furthermore, the 0.7 wt.% CuO added ceramic and pure Ag electrodes were co-fired at 900 °C to prepare a high-quality multilayer piezoelectric actuator with a d_{33} of over 450 pC/N per ceramic layer.

Keywords: piezoelectric ceramics, low-temperature co-fired ceramic, multilayer piezoelectric actuators

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1. Introduction

Piezoelectric actuators convert electrical energy directly into linear motion with high speed, force, and virtually unlimited resolution. These actuators are used in every modern high technology field from semiconductor test and inspection to super-resolution microscopy, bio-nanotechnology, and astronomy/aerospace technology.[1,2] The actuators require high-quality piezoelectric ceramics which possess a high piezoelectric constant (d_{33}) and a large strain under an external electric field.[3] Extensive theoretical and practical interest has been taken in Pb-based complex perovskite solid solutions consisting of ferroelectric and relaxor materials, due to their excellent electrical and piezoelectric properties.[4] Researchers have tried immensely hard to discover Pb-free materials with good piezoelectric property comparable to that of Pb-based ones but have failed.[5–7] Among these Pb-based materials, the $(1-x)PbMg_{1/3}Nb_{2/3}O_3$–$xPbZr_{0.52}Ti_{0.48}O_3$ (PMN-PZT) system has been extensively studied for actuators because of its high piezoelectricity and large strain. 0.25PMN-0.75PZT exhibits a morphotropic phase boundary (MPB) with better dielectric and piezoelectric properties than the single-phase.[8,9] The MPB of $Pb(Zr_{0.53}Ti_{0.47})O_3$ and ferroelectric relaxation characteristics of $Pb(Mg_{1/3}Nb_{2/3})O_3$ (PMN) are the reasons for their excellent properties, such as a large dielectric constant and a broad diffuse transition.[10] Wang *et al.*[8] reported that $Pb_{0.92}Sr_{0.06}Ba_{0.02}(Mg_{1/3}Nb_{2/3})_{0.25}(Ti_{0.53}Zr_{0.47})_{0.75}O_3$ ceramic sintered at 1260 °C shows excellent piezoelectric and dielectric properties. Such phase boundary-induced enhancements in a material's properties have also been observed in other systems such as $BiFeO_3$.[11,12]

Generally, multilayer piezoelectric actuators require co-firing with an internal electrode such as Ag–Pd. However, the sintering temperature of PZT-based ceramic materials is at about 1200 °C.[7] Mostly Ag–Pd alloy is used in multilayered actuators as the inner electrodes, but Pd is expensive, which increases the fabrication cost.[13,14] Another disadvantage of the high sintering temperature is the volatility of PbO during sintering, which pollutes the environment. In addition, the decrease of the sintering temperature can reduce energy consumption and environmental pollution. The multilayer piezoelectric actuator with pure Ag electrodes should be prepared at ∼900 °C, since Ag evaporates rapidly above 930 °C. Therefore, low-temperature processing is one of the most important techniques for the fabrication of multilayer piezoelectric actuators.

A lot of attempts have been made to attain low temperature sintering including the fine powder approach, hot pressing and similar approaches. One such technique is the addition of materials with low melting point, such as metal oxides, for liquid-phase sintering.[15–17] Some of the oxides and compounds are employed in assisting the liquid-phase sintering to reduce the sintering temperature of PZT-based materials. Hayashi *et al.*[18] reported the addition of $LiBiO_2$ in $Pb_{0.95}Ba_{0.05}(Mg_{1/3}Nb_{2/3})_{0.125}Zr_{0.445}Ti_{0.43}O_3$ ceramics sin-

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tered at 950 °C and calculated a d_{33} value of 467 pm/V. Chao *et al.*[19] reported that PMN-PZN-PZT ceramics sintered at 995 °C with 0. 01 wt.% ZnO-Li_2CO_3 and 0.10 wt.% Pb_3O_4 contents showed $d_{33} = 256$ pC/N. Similarly, Gio *et al.*[20] reported $d_{31} = 112$ pC/N and a density of 7.91 g/cm^3 with CuO additive in $0.8Pb(Zr_{0.48}Ti_{0.52})O_3$-$0.125Pb(Zn_{1/3}Nb_{2/3})O_3$-$0.075Pb(Mn_{1/3}Nb_{2/3})O_3$ ceramics sintered at 930 °C. The CuO component has shown a promising influence for the densification compared to many other additives.

Here 0.7 wt.% CuO added $Pb_{0.92}Sr_{0.06}Ba_{0.02}(Mg_{1/3}Nb_{2/3})_{0.25}(Ti_{0.53}Zr_{0.47})_{0.75}O_3$ ceramic was prepared at 900 °C and it shows the remnant polarization of 40 μC/cm^2, 0.28% strain at 40 kV/cm, and d_{33} of 630 pC/N. Furthermore, its multilayer piezoelectric actuator with pure Ag inner electrodes was sintered at 900 °C and each 60 μm layer shows d_{33} of over 450 pC/N.

2. Experiment

The specimens were manufactured using a conventional mixed oxide process. The compositions used in this study are as follow: $Pb_{0.92}Sr_{0.06}Ba_{0.02}(Mg_{1/3}Nb_{2/3})_{0.25}(Ti_{0.53}Zr_{0.47})_{0.75}O_3 + x$CuO ($x = 0.1$–0.9 wt.%). The raw materials such as PbO, ZrO_2, TiO_2, Nb_2O_5, MgO, $SrCO_3$, and $BaCO_3$ for the given composition were weighted by molar ratio and the powders were ball-milled for 12 h. After drying, they were calcined at 800 °C for 3 h. Thereafter, x CuO was added, ball-milled, and dried again. Polyvinyl alcohol (PVA: 5 wt.% aqueous solution) was added to the dried powders. The powders were molded by the pressure of 1000 kg/cm^2 in a mold, which has a diameter of 13 mm, burned out at 600 °C for 3 h, and then sintered at 850–1050 °C for 2 h. Here, the x CuO added ceramics sintered at y temperature is named as x–y ceramic in the following.

For measuring the piezoelectric characteristics, the specimens were polished to 1 mm thickness and then electrodes were pasted with Ag paste. Poling was carried out at 120 °C in a silicon oil bath by 30 kV/cm for 30 min. All samples were aged for 24 h prior to measuring the piezoelectric and dielectric properties. The crystal structure and microstructure of the specimens were analyzed through x-ray diffraction (XRD, Brucker advanced D8) and scanning electron microscopy (SEM, FEI Quanta 250F), respectively. For investigating the dielectric properties, the capacitance was measured at 1 kHz using an impedance analyzer (Agilent 4294A), and the dielectric constant was calculated. Then, the piezoelectric coefficient d_{33} was measured by a piezo-d_{33} meter (IAAS ZJ–30), the resonant and anti-resonant frequencies were measured by the impedance analyzer, and then the planar electromechanical coupling factor k_P and its mechanical quality factor Q_m were calculated according to IEEE standard. Polarization versus electric field (P–E) and longitudinal strain versus electric field (S–E) loops were measured at 1 Hz with a Radiant multiferroic tester.

3. Results and discussion

The XRD patterns of the x-850 ceramics ($x = 0.3$ wt.%, 0.5 wt.%, 0.7 wt.%, and 0.9 wt.%) show perovskite structure without pyrochlore phase (Fig. 1). The diffraction peaks at $2\theta = 37°$–$40°$ and $2\theta = 43°$–$46°$ are amplified in two insets. The single peak of (2 0 0) occurs for the rhombohedral phase, whereas it splits into two peaks of (0 0 2) and (2 0 0) for the tetragonal phase.[21] The (200) reflection is obviously broadened and can clearly be fitted with two peaks of $\{200\}_T$ and $\{002\}_R$. It is due to the well-known morphotropic phase boundary (MPB), where two ferroelectric phases (rhombohedral and tetragonal) coexist. The stronger intensity of $\{002\}_R$ shows that the rhombohedral phase is dominant over the tetragonal phase. MPB is the main reason for the improved piezoelectric performances. There is no special change in XRD patterns with x increasing from 0.3 wt.% to 0.9 wt.%, thus excessive CuO addition has not obviously changed the MPB structure of $Pb_{0.92}Sr_{0.06}Ba_{0.02}(Mg_{1/3}Nb_{2/3})_{0.25}(Ti_{0.53}Zr_{0.47})_{0.75}O_3$ ceramics.

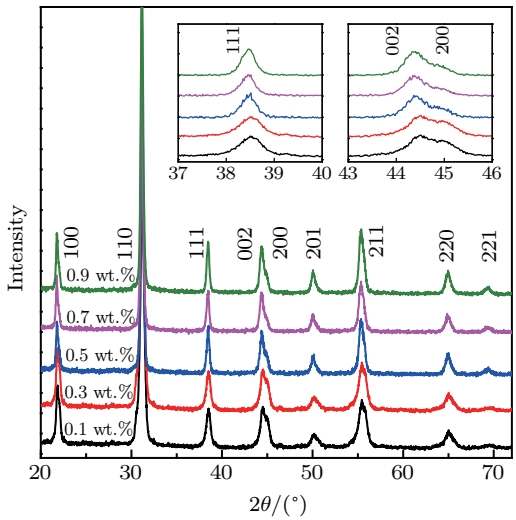


Fig. 1. (color online) XRD pattern of x CuO added ceramics ($x =$ 0.1 wt.%, 0.3 wt.%, 0.5 wt.%, 0.7 wt.%, and 0.9 wt.%) sintered at 850 °C.

The x CuO added specimens can be sintered at a lower temperature and the 0.7 wt.%–900 ceramic owns a density of 7.742 g/cm^3, which is 98% of the common specimen sintered at 1200 °C.[22] The density of 0.1 wt.%-850 ceramic is just 6.5 g/cm^3, however it increases linearly with the sinter temperature and reaches 7.1 g/cm^3 at the sinter temperature of 1050 °C (Fig. 2). The density of the x CuO added ceramics ($x = 0.3$–0.9 wt.%) increases initially and then decreases with the increase in sintering temperature and a maximum density of > 7.5 g/cm^3 is achieved in the range of 900–950 °C. The highest density of 7.742 g/cm^3 appears in the 0.7 wt.%–900

ceramic (Fig. 2). The excess CuO reacts with PbO and forms the liquid-phase of Cu_2O–PbO at about 800 °C,[21] which accelerates the reaction between PbO and other raw materials at relatively low temperature. However, CuO, PbO, and Cu_2O–PbO are easy to evaporate above 1000 °C, and the escaped gas can introduce air bubbles and cracks to lower the density, especially for 0.7 wt.% and 0.9 wt.% CuO added ceramics.

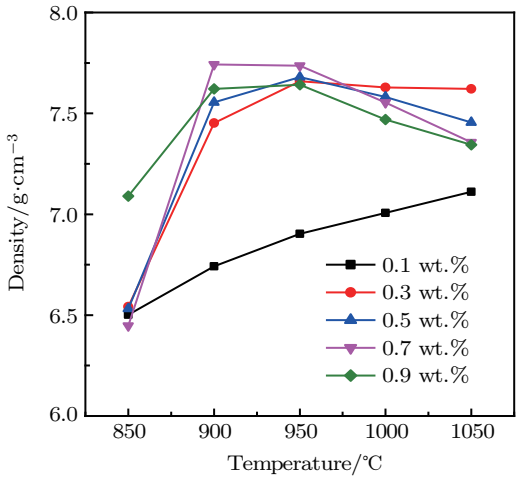


Fig. 2. (color online) Density of x CuO added ceramics ($x = 0.1$ wt.%, 0.3 wt.%, 0.5 wt.%, 0.7 wt.%, and 0.9 wt.%) sintered at 850 °C, 900 °C, 950 °C, 1000 °C, and 1050 °C, respectively.

Excess CuO addition not only accelerates the chemical reaction of raw materials but also suppresses holes and cracks according to the SEM images. Figures 3(a)–3(c) show the SEM images of 0.3 wt.% CuO added ceramics sintered at 850 °C, 900 °C, and 950 °C, respectively. The grain size increases and the cracks/holes reduce with the increase of the sintering temperature. There are few cracks/holes in the ceramics sintered at 950 °C, being consistent with the high density of 7.66 g/cm^3. Figures 3(d)–3(f) show the SEM images of 0.5 wt.% CuO added ceramics sintered at 850 °C, 900 °C, and 950 °C, and the latter two ceramics show large crystal grains with few cracks/holes consistent with their high densities of 7.54 g/cm^3 and 7.68 g/cm^3. Figures 3(g)–3(i) show the SEM images of 0.7 wt.% CuO added ceramics sintered at 850 °C, 900 °C, and 950 °C. Few cracks/holes are observed in the latter two ceramics, especially the middle one with the highest density of 7.742 g/cm^3. Figures 3(j)–3(k) show the SEM images of the Ag electrode sintered at 900 °C, 925 °C, and 950 °C. The Ag electrode remains stable at 900 °C and 925 °C for 2 h, however it evaporates seriously at 950 °C. This suggests that it is possible to prepare multilayer piezoelectric actuators with pure Ag electrodes and 0.7 wt.% CuO added ceramic at 900 °C.

The 0.7 wt.%-900 ceramic shows the highest saturated and remnant polarizations (P_S, P_r) of 53.4 μC/cm^2 and 40 μC/cm^2, respectively. Figure 4 shows the P–E loops of the x CuO added ceramics ($x = 0.3$ wt.%, 0.5 wt.%, 0.7 wt.%, and 0.9 wt.%) sintered at 850 °C, 900 °C, 950 °C, 1000 °C, and 1050 °C. The P_S and P_r of 0.3 wt.% CuO ceramics enhance with sintering temperature until they reach 50.4 μC/cm^2 and 39.7 μC/cm^2. The P_S and P_r of 0.5 wt.%-950 ceramic show the highest values of 51.7 μC/cm^2 and 38.7 μC/cm^2, the P_S and P_r of 0.7 wt.%-900 ceramic show the highest values of 53.4 μC/cm^2 and 40 μC/cm^2, and finally the P_S and P_r of 0.9 wt.%-900 ceramic show the highest values of 48.5 μC/cm^2 and 36 μC/cm^2. Obviously, the highest P_S and P_r were achieved in the ceramics with the highest density and few cracks/holes.

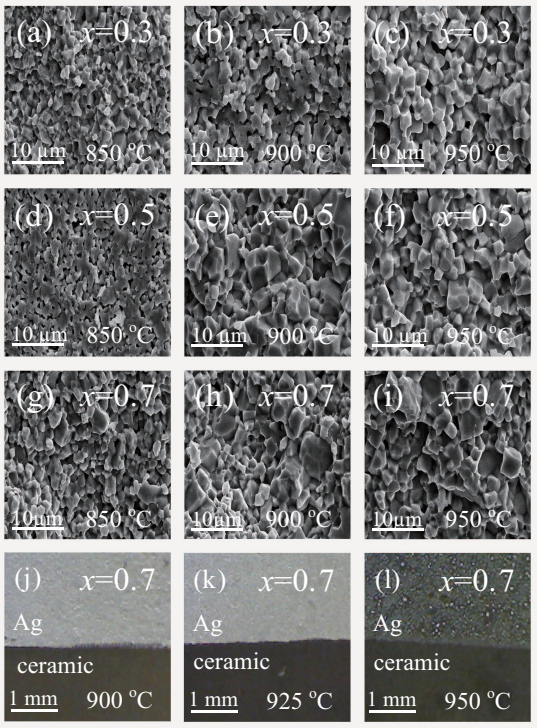


Fig. 3. (color online) SEM images of x CuO added ceramics with x=0.3 wt.% (a)–(c), 0.5 wt.% (d)–(f), 0.7 wt.% (g)–(i) sintered at 850 °C, 900 °C, and 950 °C, respectively. Optical images of Ag electrode on ceramics surface sintered at (j) 900 °C, (k) 925 °C, and (l) 950 °C.

The strain of 0.28% under 40 kV/cm has been observed in 0.7 wt.%-900 ceramic (Fig. 5(c)), which is consistent with the high-density ceramic with superior ferroelectric properties. For 0.3 wt.% CuO added ceramic, its strain increases with the increase of the sintering temperature from 850 °C to 1050 °C and finally reaches to 0.274%. For other ceramics ($x = 0.5$ wt.%, 0.7 wt.%, and 0.9 wt.%), the maximum strain occurs in the ceramic with the highest density and the largest P_S and P_r, i.e., 950 °C, 900 °C, and 900 °C for the ceramics with $x = 0.5$ wt.%, 0.7 wt.%, and 0.9 wt.%, respectively. For example, the 0.7 wt.%-900 ceramic shows the largest strain of 0.28% at 40 kV/cm. It is better than the previously reported PMN–PZT systems,[8,9] which makes it a promising material for actuator applications.

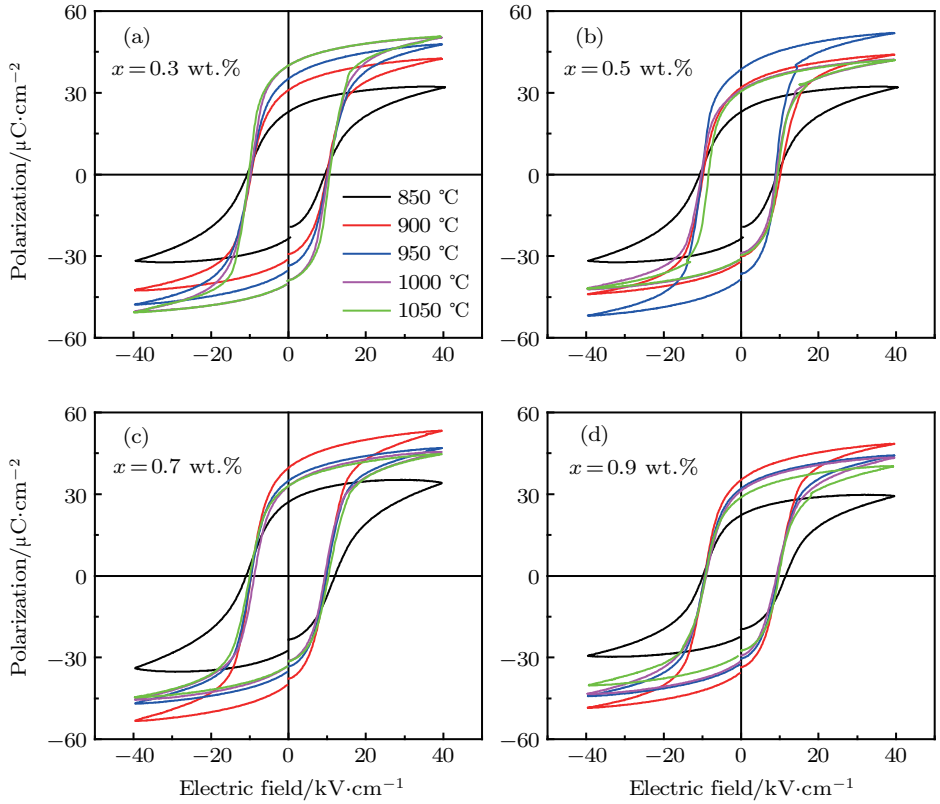


Fig. 4. (color online) *P*–*E* loops of *x* CuO added ceramics with $x = 03$ wt.% (a), 0.5 wt.% (b), 0.7 wt.% (c), 0.9 wt.% (d) sintered at 850 °C, 900 °C, 950 °C, 1000 °C, and 1050 °C.

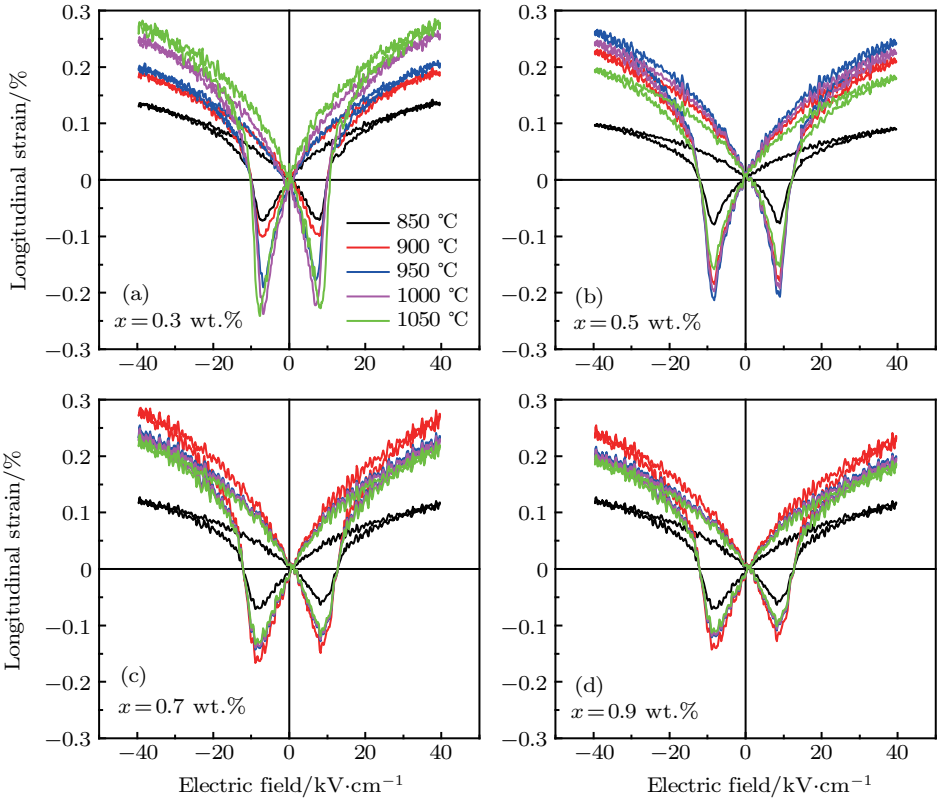


Fig. 5. (color online) *S*–*E* loops of *x* CuO added ceramics with $x = 0.3$ wt.% (a), 0.5 wt.% (b), 0.7 wt.% (c), 0.9 wt.% (d) sintered at 850 °C, 900 °C, 950 °C, 1000 °C, 1050 °C.

The dependencies of piezoelectric d_{33}, k_P, and Q_m of x CuO added ceramics on the sintering temperature are presented in Figs. 6(a) and 6(b). The maximum d_{33} and k_P occur in the ceramics with the highest density, fewest cracks/holes, largest Ps and strain. For example, the d_{33} of 0.7 wt.%-900 ceramic is as high as 630 pC/N, which is higher than that of 0.3 wt.%-1050 ceramic. Furthermore, the k_P of 0.7 wt.%-900 ceramic shows a significantly higher value of 61%, which is 90% to the previously reported value of ceramic sintered at much higher temperature of 1200 °C.[8] The Q_m of 0.7 wt.% CuO added ceramic increases from ~ 80 to 150 with the increase of the sintering temperature to 1050 °C. In short, these ceramics show typical characteristics of soft PZT-5.[23] Meanwhile, figure 6(c) shows the correlation between the density and the Q_m values of x CuO added ceramics sintered at 900 °C. The maximum Q_m and density are achieved in 0.7 wt.%-900 ceramic. A dense structure not only improves the piezoelectric response but also increases the Q_m value by reducing the dissipated energy. The d_{33} and SEM analysis of 0.7 wt.%-900 ceramic also confirm the same trend.[24,25]

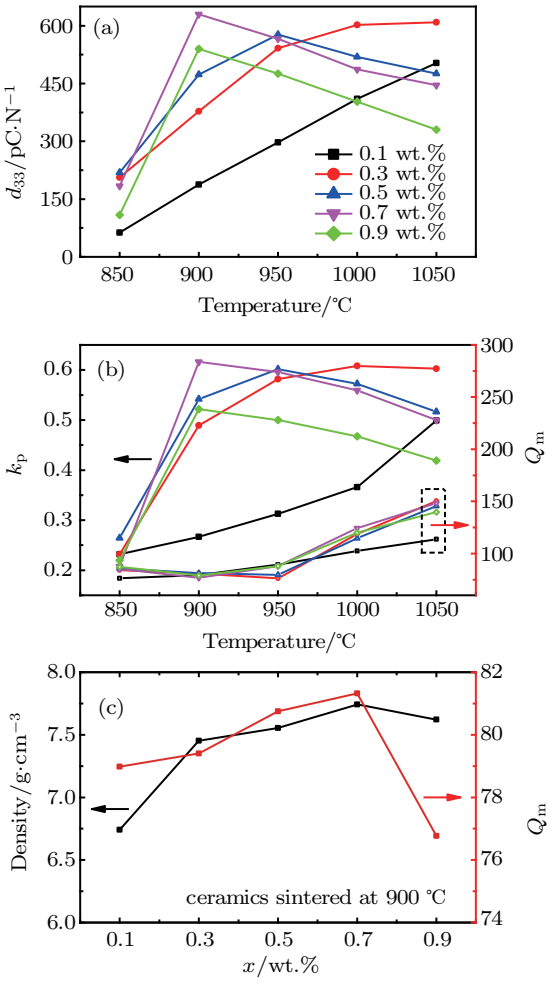


Fig. 6. (color online) (a) Piezoelectric d_{33}, (b) electromechanical coupling factor k_p and quality factor Q_m of x CuO added ceramics, (c) co-relation between density and quality factor Q_m of x CuO added ceramics sintered at 900 °C.

The dependence of relative dielectric constant ε_r on temperature suggests the dielectric relaxor characteristic and a Curie temperature (T_C) of ~ 200 °C. Here, the maximum ε_r (ε_m) occurs at a temperature (T_m) closer to T_C in the ε_r versus temperature curves (Fig. 7). The T_m of 0.7 wt.%-900 ceramic is 198 °C, 200 °C, 202 °C, 203 °C, and 204 °C at 100 Hz, 1 kHz, 10 kHz, 100 kHz, and 1 MHz, respectively. The strong frequency dependence suggests a typical diffuse phase transition of relaxor ferroelectrics. The relaxor characteristic is also confirmed by the large diffusion coefficient (γ) of 1.92, which is derived from the relationship of $\ln(1/\varepsilon_r - 1/\varepsilon_m) = \gamma \ln(T - T_m) - \ln C$ (inset), where C is a constant.[26]

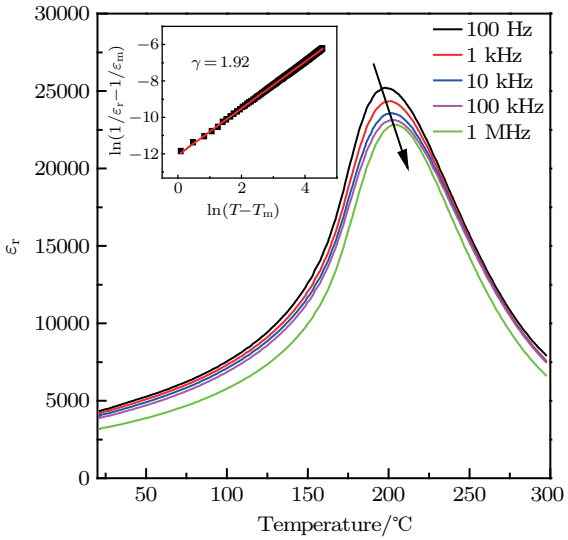


Fig. 7. (color online) The dependence of relative dielectric permittivity ε_r on temperature for 0.7 wt.%-900 ceramic, where the inset shows the modified Curie–Weiss law fitting.

Finally, we fabricated the multilayer piezoelectric actuator with 0.7 wt.% CuO added ceramic sheets and pure Ag electrodes at 900 °C. The piezoelectric sheets were obtained by tape-casting ceramic slurry containing a mixture of piezoelectric powders, organic binders, a plasticizer, and solvents. The piezoelectric ceramic sheets were cut into 6 cm×6 cm square pieces by a knife cutting machine. The Ag paste was screen-printed on the ceramic sheet, and then laminated with 20 layers. After soaking at 500 °C for 6 h in air to remove the organic additives within the ceramic sheets, the laminated composites were co-fired at 900 °C for 10 h in air. The thicknesses of the ceramic layer and the Ag layer are 60–65 μm and 6–8 μm, respectively. Figures 8(a) and 8(b) are the optical micrographs of 11.5-mm-thickness multilayer ceramic. The SEM images in Figs. 8(c) and 8(d) clearly show the piezoelectric layer and the Ag electrodes. Each piezoelectric layer shows a d_{33} of over 450 pC/N, which is smaller than that of the bulk piezoelectric ceramics prepared by the conventional solid state sintering method. This d_{33} decrease is due to two factors. First, the ceramic sintered at 900 °C by solid state sintering

method has a higher density and fewer cracks than the piezoelectric pieces prepared by the tape-casting method and sintered at 900 °C. Second, the ceramic bulk shows a large ratio of height and diameter required by IEEE standard on piezoelectricity. Our 0.7 wt.% CuO added layer/Ag multilayer is feasible to operate at $\leq$ 10 Hz frequency, where the possibility of heat release from the piezoelectric actuator due to low Q_m and T_C will be minimized. To overcome this problem, it is important to enhance the T_C and/or Q_m value of Pb-based ceramics by doping or addition of the elements possessing high heat stability behavior.

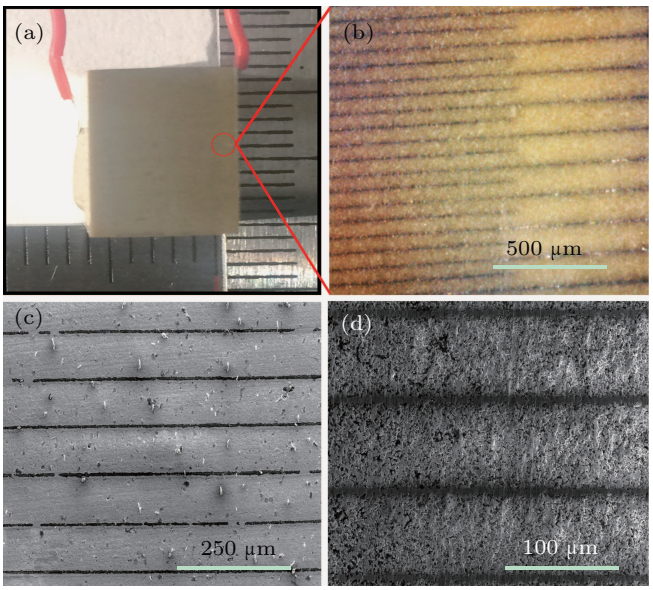


Fig. 8. (color online) (a) Optical micrograph of 0.7 wt.% CuO added layer/Ag multilayer prepared by tape casting method. (b) Optical micrograph of polished cross-sectional 0.7 wt.% CuO added layer/Ag multilayer. (c), (d) Magnified cross-sectional SEM images of 0.7 wt.% CuO added layer/Ag multilayer.

4. Conclusion

The dependencies of the ceramic sintering temperature, density, ferroelectric and piezoelectric properties on excess CuO addition were systematically studied in x CuO added $Pb_{0.92}Sr_{0.06}Ba_{0.02}(Mg_{1/3}Nb_{2/3})_{0.25}(Ti_{0.53}Zr_{0.47})_{0.75}O_3$ ceramics (x = 0.1–0.9 wt.%). 0.7 wt.% CuO addition has effectively reduced the sintering temperature from 1260 °C to 900 °C and the corresponding ceramic has a density of 7.742 g/cm^3. Furthermore, it shows P_S of 53.4 μC/cm^2, P_r of 40 μC/cm^2, 0.28% strain at 40 kV/cm, d_{33} of 630 pC/N, k_P of 0.61, Q_m of $>$ 80, and T_C of 200 °C. Finally, we prepared a multilayer piezoelectric actuator with 0.7 wt.% CuO ceramic sheets and pure Ag at 900 °C, where each 60–65 μm piezoelectric layer shows d_{33} of over 450 pC/N.

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