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ABSTRACT

A low-temperature (≤450 °C) crystallization path for the solution-derived lead zirconium titanate (PZT) thin-film, the first choice for high-density ferroelectric/piezoelectric nanodevices, has been sought for long but with limited success. In this work, we report on a facile route enabling the crystallization of PZT thin-films at 300–450 °C by using a novel solution-combustion-synthetic (SCS) approach. The SCS precursor solution consists of metal nitrates and organic fuels (tricine and/or urea) as oxidizing and reducing agents, respectively. The essential aspect of this route is that the single and intense exothermic combustion reaction between the oxidizer and fuels occurring at 234 °C provides high self-localized thermal energy, which allows for the efficient conversion of the amorphous metal-oxide gel framework into final crystallized oxides even at very low externally applied temperature. Effects of annealing conditions such as temperature and time are systematically investigated. At optimized conditions, high-quality PZT thin-films are achieved with pure perovskite phase, large remanent polarization (~37 μC/cm²), and small leakage current (~0.1 μA/cm² at 600 kV/cm). Furthermore, an application of the developed low-temperature PZT film for the ferroelectric-gate memory transistor is demonstrated. The potential integration of low-temperature processed PZT layers with other active components may redefine the design concept of classical nano-microelectronic devices.

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I. INTRODUCTION

Ferroelectric materials find applications in a variety of electronic and electromechanical devices. Among them, lead zirconate titanate (PZT) is the first choice for high-density ferroelectric/piezoelectric nanodevices because of its excellent properties and relatively low processing temperatures compared to the other two major options, strontium bismuth tantalate (≥700 °C) and bismuth lanthanum titanate (≥650 °C). However, the current processing temperatures of PZT, usually 600–700 °C, are still too high, and the usual silicon-based CMOS circuits are severely damaged upon integrating with PZT elements. The thermal budget currently recommended for manufacturing sub-100 nm semiconductor devices sets a maximum limit of 450 °C. Therefore, a low-temperature fabrication process for high-quality ferroelectric films is highly desired. Compared to vacuum-based deposition methods (e.g., sputtering, pulse laser deposition, and metal-organic chemical vapor deposition), the chemical-solution-deposition (CSD) ones offer advantages, such as simplicity, low cost, large area deposition, and direct printability, making it more preferable for future practical applications.

So far, CSD-based methods for low-temperature PZT fabrication, including seeding the film, tailoring pyrolysis and heating processes, hydrothermal treatment, microwave heating, laser-assisted annealing, ultraviolet (UV)-assisted annealing, and activated photosensitive/seed-photosensitive precursors, have been intensively studied. For example, Bharadwaja et al. reported crystallization of the (111)-oriented PZT film at 375 °C on PbTiO₃-coated Pt/Ti/SiO₂/Si substrates by means of the KrF pulsed excimer laser-assisted annealing. The Morita group demonstrated an epitaxial growth of PbTiO₃ on single crystal SrTiO₃ substrates using a hydrothermal method at a temperature below 200 °C, where
metal ion sources are placed in a high pressure container with KOH solution. The microwave heating utilizing the magnetic field with a high frequency of 2.45 GHz is capable of crystallizing the solgel-derived PZT film at 450 °C with a random crystalline orientation.

In general, for CSD-based metal-oxide films, the decomposition, densification, dihydroxylation, and impurity removal steps of processing typically require annealing above 450 °C. In order to lower the processing temperature, one of the effective ways could be the use of an auto-combustion process. The combustion approach has been proven to achieve the energy-efficient synthesis of bulk materials such as chalcogenides, III-V semiconductors, carbides, binary oxides, and intermetallics. An application of the solution combustion synthesis (SCS) method was recently reported for the general multicomponent oxides such as semiconducting In–Ga–Zn–O and conductive In–Sn–O thin-films.

In this work, a redox-based SCS approach is applied to PZT thin-films, using metal nitrates as metal sources as well as oxidizers and urea or tricine as a fuel. Effects of fuel type, fuel/oxidizer ratio, and annealing conditions (time, temperature) were systematically investigated. It was found that the activity of the combustion reaction is strongly influenced by the type of fuel as well as the fuel/oxidizer ratio. By optimizing the above-mentioned parameters, we are able to grow device-quality PZT thin-films on platinized silicon substrates with various compositions at temperatures as low as 300–450 °C.

II. EXPERIMENTAL PROCEDURE

A. Synthesis of PZT solution using SCS method (SCS-PZT sols)

Lead nitrate Pb(NO₃)₂, zirconium oxyxinitrate ZrO(NO₃)₂·2H₂O, and titanium isopropoxide Ti[OCH(CH₃)₂]₄ were used as starting metal precursors. For Pb-sol, Pb(NO₃)₂, tricine, and ammonium nitrate with a molar ratio of 1:1:1 were dissolved in 2-methoxyethanol (2-MOE). The mixed solution was stirred at 1000 rpm for 30 min on a hot-plate to form a transparent homogeneous solution and then filtered using a 0.2 μm PTFE syringe filter before use. Similarly, Zr-sol and Ti-sol were synthesized by mixing ZrO(NO₃)₂·2H₂O and Ti[OCH(CH₃)₂]₄, respectively, with urea and ammonium nitrate (molar ratio of 1:1:1) in 2-MOE using the same procedure. The concentration of Pb-, Zr-, and Ti-sols was adjusted to be 0.4 mol/kg. Finally, the synthesized Pb-, Zr-, and Ti-sols were mixed together with the Pb:Zr:Ti ratio of 120:52:48 to form SCS-PZT sol. The synthesized SCS-PZT sol is highly stable and can be stored at room temperature for months. A schematic diagram of the SCS-PZT sol preparation is described in Fig. 1.

In addition, various fuels such as acetylacetonate, urea, glycine, citric, and hydrazine were tested to reveal the importance of the fuel type.

B. Preparation of PZT thin-films

Throughout the research, the Pt(200 nm)/TiO₂(20 nm)/SiO₂(500 nm)/Si (500 μm) substrate was utilized as the bottom electrode for growing PZT films. Prior to SCS-PZT deposition, the substrate was cleaned by acetone, ethanol, de-ionized water, N₂ purging, and, finally, oxygen plasma treatment. The SCS-PZT sol was deposited on the substrate by spin-coating at 2000 rpm for 25 sec, followed by a pyrolysis at 250 °C for 5 min using a hot-plate. These processes were repeated to make a film with desired thickness. Finally, the
SCS-PZT gel films were annealed at different temperatures in the range between 300 °C and 450 °C for 10–360 min using the hot-plate in ambient air.

C. Fabrication of ferroelectric-gate memory transistor

A PZT-gate memory transistor with a bottom-gate and bottom-contact structure was fabricated. In the structure, the Pt thin-film (200 nm) grown on a SiO₂/Si substrate was used as the bottom-gate electrode. The SCS-PZT film as the ferroelectric-gate insulator with a thickness of approximately 220 nm was deposited on the Pt gate electrode and annealed at 400 °C for crystallization. After that, the Pt source and drain electrodes (100 nm) were formed by sputtering deposition and the photolithography technique. Finally, an indium-zinc-oxide (IZO) semiconducting channel layer (20 nm) was deposited by spin-coating and annealed at 400 °C in ambient oxygen, followed by channel patterning using photolithography. The defined channel width and length were 60 μm and 30 μm, respectively.

III. RESULTS AND DISCUSSION

A. Analysis of PZT precursor solutions

Finding an effective fuel/oxidizer pair is critically important for realizing the SCS reaction, and the pair actually depends on the individual metal-oxide system. In general, the fuels which have strong reducing property are favorable in order to efficiently activate the SCS reaction. Here, we investigated five different types of fuels including acetylacetone, urea, hydrazine, glycine, and tricine for each individual Pb, Zr, and Ti precursor solutions by thermal analyses (Fig. S1 and Fig. 2). It was found that the tricine/ammonium nitrate and urea/ammonium nitrate pairs were the most effective ones, which clearly showed single intense exothermic peaks at 175 °C and 260 °C for Pb and Zr precursor solutions, respectively. Moreover, as a key parameter determining the efficiency of SCS reaction, the impact of the metal to fuel to oxidizer ratio on the thermal behavior of SCS solution was also investigated by thermogravimetry (TG)-differential thermal analysis (DTA) (Fig. S3). It was found that the combustion reaction effectively occurred when the ratio was either 1:1:1 or 2:1:1. On the other hand, the reaction was suppressed when the ratio was 1:2:2. This could be attributed to the molar stoichiometry of the redox SCS system; however, detailed analysis is needed to confirm this hypothesis. The optimal SCS-PZT solution was then prepared by mixing these optimized individual solutions.

In Fig. 2, TG-DTA data are plotted for the conventional and optimal SCS-PZT powder precursors. As clearly seen, the SCS-PZT system exhibited a substantially lower complete conversion temperature (<250 °C) than the conventional ones (>450 °C). Unlike the conventional PZT system, which exhibited broad endotherms for oxide lattice formation and exotherms for organic impurity removal,22–24 the SCS-PZT system showed a single, intense exotherm at 234 °C in the DTA [Fig. 2(a)] that corresponded exactly to the abrupt mass loss in the thermogravimetric analysis (TGA) [Fig. 2(b)] and was possibly sufficient to drive the reaction rapidly to completion. For the conventional precursor route, adequate film quality is typically only possible for annealing temperatures greater than that for metal oxide lattice formation or organic impurity oxidation.15,17 On the other hand, note that, the principal driving force for oxide lattice formation in combustion synthesis derives mainly from internal chemical energy. Thus, the required temperature can be described as that for reaction initiation rather than a temperature that must be continuously maintained to drive the reaction.21–24 Accordingly, it would be expected that a significantly lower temperature is required for crystallization of the PZT thin-film via the SCS.

B. Structural properties: Effects of annealing temperature and time

1. Annealing temperature dependence

Figure 3 shows XRD patterns of SCS-PZT thin-films pyrolyzed at 300 °C and annealed at various temperatures (300–450 °C) for 60 min. A reference 700 °C-annealed PZT thin-film fabricated using a commercial precursor solution (Mitsubishi Chemical Corp.) was also prepared for comparison. By 300 °C annealing, a diffraction peak located at around 38.4° on the left shoulder of the Pt(111) peak, which corresponds to the crystallized PZT(111), was observed. Although this peak is overlapped by the Pt(111) peak due to weak intensity, its appearance indicates the nucleation of the perovskite PZT phase at a significantly lower temperature (300 °C) compared to the conventional ones. With the increase in the annealing temperature, the intensity of PZT(111) was subsequently enhanced, indicating the improvement in the film crystalline quality. The formation of the PZT pyrochlore (Py) phase, which is normally grown at
a low-temperature annealing (300–400 °C) by conventional methods, was not observed in the SCS-PZT samples. In addition, all the films exhibited (111)-preferentially crystalline orientation with minor formation of the other perovskite phases such as (100/001) or (101). The FT-IR analyses of SCS-PZT films for different drying temperatures revealed that carbon-related compounds still remain inside the films after the combustion step (Fig. S4). The residual carbon might have reduced the film, leading to the formation of PbPt$_x$ intermetallic phases at the interface between the bottom Pt electrode and the PZT layer during the annealing. As a result, the PbPt$_x$ phases, which have a lattice constant ($a = 4.05$ Å) similar to that of the perovskite PZT(111) phase ($a = 4.08$ Å), facilitate the growth of (111)-oriented PZT films. The SCS-PZT films annealed at 400–450 °C presented comparable (111) peak intensity with that of the conventional PZT film prepared at 700 °C. Such a low crystallization temperature can be attributed to the huge thermal energy internally generated by the combustion reaction, which is able to drive the reaction to completion. Note that, although the local temperature at the combustion front is very high (a significant fraction of the adiabatic temperature), if the effective volume is small like in the case of SCS-PZT films ($\sim 20$ nm for each layer), the surrounding material may remain at low temperature. Previously, Kim et al. reported combustion processing of n-type oxide semiconductor thin films on a polyimide substrate, although the estimated generated heat from their combustion reaction under ideal adiabatic conditions would reach 700 °C. The fabrication of the p-type Li:NiO$_x$ thin film transistor on a flexible polyethylene terephthalate (PET) substrate using a combustion process was also demonstrated. The related studies on combustion processing suggest that the substrate of a SCS film may not be affected by the
FIG. 5. XRD patterns of SCS-PZT thin-films annealed at 400 °C for different time courses (10–360 min).

high local temperature of the film. However, evidence over what volume the local temperature rises has not been clarified yet and needs further study.

To further evaluate the quality of the low-temperature SCS-PZT film, XPS analysis was performed on the sample annealed at 400 °C. The XPS depth-profiles of C 1s, O 1s, Pb 4f, Zr 3d, and Ti 2p are shown in Fig. 4. Typical XPS spectra of Pb 4f, Zr 3d, Ti 2p, and O 1s [Figs. 4(a)–4(d)] agree well with some prior reports. In particular, the O 1s peak can be deconvoluted into two components centered at 529.5 eV and 531.0 eV, which correspond to the lattice oxygen of PZT (O_{PZT}) and surface adsorbed oxygen (O_{ads}). The hydroxyl-related peak positioned at 532.9 eV was not observed in the present study, indicating nonexistence of hydroxide layers. The peak positions and intensities were almost unchanged as the etching time increased, indicating that the film was homogeneous. The C 1s spectrum of the surface PZT film (etching time = 0 sec) showed a characteristic C 1s peak at around 284.6 eV [Fig. 4(e)], which can be attributed to physisorbed carbon on the sample surface. More importantly, the peak related to C disappeared when the

FIG. 6. (a) Top-view and (b) cross-sectional SEM images of the SCS-PZT thin-films annealed at 400 °C for different times: 10 min, 30 min, 60 min, 120 min, and 360 min. P and Py are represented for perovskite and pyrochlore phases, respectively.
etching time increased, indicating that the amount of residual C in the bulk was negligible (below the detection limit of XPS). Besides, depth-profile elemental analysis of the PZT film annealed at 400 °C by the Nuclear Reaction Analysis (NRA), Rutherford Backscattering Spectroscopy (RBS), and Hydrogen Forward Scattering spectrometry (HFS) further confirmed the negligible amount of H and C remains in the film after the crystallization annealing (Fig. S5). It is widely known that the remaining C and/or H would degrade the film quality; thus, improved electrical properties of the obtained C- and H-free SCS-PZT films would be expected.

2. Annealing time dependence

The XRD patterns in Fig. 5 shows diffraction peaks of the SCS-PZT thin-films annealed at 400 °C for different annealing times after being pyrolyzed at 250 °C for 5 min. The broad peak centered at 29.5° is identified as the (222) crystal plane of the pyrochlore PZT phase. It was found that although diffraction peaks of the perovskite phase were observed in the films annealed for 10–30 min, the peak of the pyrochlore phase still remained. The non-ferroelectric pyrochlore phase almost disappeared in the films annealed for 60 min or longer time. Thus, the pyrochlore phase was first formed and then transformed to the perovskite phase while the annealing time prolonging, in which the phase transformation process has accomplished after thermal treatment for 60 min.

Figures 6(a) and 6(b) show the top-view and cross-sectional field-emission scanning electron microscope (FE-SEM) images of the SCS-PZT films, as a function of the annealing times, respectively. The films annealed for a shorter time (10–30 min) was mainly composed of the pyrochlore phase, as evidenced from top-view FE-SEM images [Fig. 6(a)], and exhibited an internal porous structure [Fig. 6(b)]. As the annealing time increased to 60–360 min, the ratio of the pyrochlore to perovskite phase decreased, which indicates the occurrence of the transformation process from the pyrochlore to perovskite phase. At the optimal annealing time (60–120 min), a typical surface structure of the PZT perovskite phase was observed together with a highly dense columnar structure. Excess annealing time (360 min) led to formation of defects such as hill-locks on the surface, as seen from the top-view SEM image.

Furthermore, the effect of excess Pb on reduction in the crystallization temperature was also investigated. The XRD and ferroelectric hysteresis loops of SCS-PZT films with 0 wt. %, 10 wt. %, and 20 wt. % excess Pb are demonstrated in Figs. S6 and S7, respectively. The SCS-PZT films prepared with lower excess Pb (10 wt. % and 0 wt. %) exhibit a well-crystallized structure with the minor pyrochlore phase and show typical hysteresis loops comparable to those of SCS-PZT films using 20 wt. % excess Pb. These results suggest the possibility to reduce the amount of Pb excess in the SCS method, owing to its lower annealing temperature.

C. Electrical properties

Figure 7(a) represents P–V hysteresis loops of the SCS-PZT thin-films annealed at 400 °C for different times. Very small hysteresis loops ($P_r = 1.2 \mu C$ and $3.0 \mu C/cm^2$) were observed in PZT films annealed for 10–30 min due to the fact that they were mainly composed of the pyrochlore phase, which were clarified by XRD results. The remanent polarization increased significantly when the annealing time increased to 60–120 min and then decreased slightly for the film annealed for 360 min. The improvement of the ferroelectric properties can be explained by the increase in the proportion of the perovskite phase in the films. On the other hand, the decrease in $P_r$ at a longer annealing time might be related to generation of defects such as pin-holes or Pb evaporation, as anticipated from the AFM
analysis. The largest \( P_r \) value of 37 \( \mu \text{C/cm}^2 \) was achieved with the film annealed for 120 min.

The leakage current property of the films with different annealing times was measured using a delayed time of >60 sec to avoid any charging or poling currents [Fig. 7(b)]. The leakage current indicates a large variation with respect to the annealing time, which can be explained as following. Due to the existence of the pyrochlore phase, the films annealed for 10–30 min exhibited the largest leakage current (>10\(^{-7}\) A/cm\(^2\) at 600 kV/cm). In addition, the film annealed for 360 min showed a low breakdown field (<600 kV/cm) because of the defects, as observed from the FE-SEM image (Fig. 6). The lowest leakage current (0.1–1.0 \( \mu \text{A/cm}^2 \) at 600 kV/cm) was achieved when the films were annealed for 60–120 min.

Figure 8 illustrates the dielectric constant and the loss tangent of the SCS-PZT thin-films for different annealing times (30 min, 60 min, and 120 min) in the frequency range of 0.1 kHz to 0.1 MHz. The results indicate that the dielectric constant increases from 110 to 520 with an increase in the annealing time without a significant change in the dielectric loss. The observed loss tangents at 10 kHz were less than 0.2. The abrupt change in the dielectric constant when the annealing time varied from 30 min to 60 min might relate to the improved crystallinity at longer annealing time, as revealed by XRD and SEM.

D. Application of SCS-PZT for a non-volatile ferroelectric memory device

To demonstrate possible usages of the low-temperature SCS-PZT film for device applications, a ferroelectric-gate thin-film transistor (FGT) using the SCS-PZT film was fabricated and its characteristics are shown in Fig. 9. The transfer curve [Fig. 9(a)] exhibited a counter-clockwise characteristic originated from the nature of ferroelectricity of the PZT gate insulator.\(^{21}\) The obtained “on/off” current ratio and memory window were approximately 10\(^9\)–10\(^7\) and 2.5–3.0 V, respectively, which are comparable to previous reports using the conventional ferroelectric thin-films.\(^{21,31-33}\) The change in gate scanning voltage (5–14 V) resulted in the increase in saturation current but slightly affected the memory window. A clear “pinch-off” characteristic observed in the output curve [Fig. 9(b)] also assures a typical operation of the FGT. These results confirm that the SCS-PZT film processed at low-temperature is qualified for electronic device applications.

IV. CONCLUSION

In this work, a facile route enabling the crystallization of PZT thin-films at 300–450 °C was established by using a novel SCS approach. The SCS precursor solution consists of metal nitrates and urea or tricine fuels as oxidizing and reducing agents, respectively. We found that the single and intense exothermic combustion reaction occurred at 234 °C, which provides high self-localized thermal energy, thus allowing the efficient conversion of the PZT amorphous framework into the crystalline perovskite structure even at a very low externally applied temperature. A systematic investigation on the microstructure and electrical properties of SCS-PZT thin-films was done with respect to annealing conditions. As a result, device-quality PZT thin-films could be achieved with large remanent polarization (37 \( \mu \text{C/cm}^2 \)) and small leakage current density (0.1 \( \mu \text{A/cm}^2 \) at 600 kV/cm), which was successfully demonstrated for a ferroelectric-gate memory transistor application as a typical example. This simple low-temperature path may open a new era in the long history of ferroelectric films, allowing low-cost, large-scale production, a wider selection of device materials, and novel opportunities for interface engineering and device miniaturization.

SUPPLEMENTARY MATERIAL

Information including materials and device characterization and TG-DTA, FT-IR, and elemental profile analyses are provided in the supplementary material.

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