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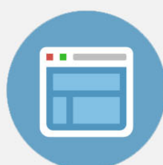
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Film structure and ferroelectric properties of vanadium-doped $Sr_{0.8}Bi_{2.3}Ta_2O_9$ thin films

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Vanadium-doped strontium bismuth tantalate $Sr_{0.8}Bi_{2.3}(Ta_{2-x}V_x)O_9$ (SBTV) ($x=0-0.8$) have been prepared on Pt/Ti/SiO_2/Si substrates using a metalorganic decomposition method. Well-crystallized and dense SBTV films can be obtained at an annealing temperature as lower as 650–700 °C that can be attributed to the enhanced densification due to the addition of vanadium. With partial substitution of tantalum by vanadium up to $x=0.15$, a larger remanent polarization ($2P_r$) of 30.5 μC/cm^2 can be obtained for the $Sr_{0.8}Bi_{2.3}Ta_{1.85}V_{0.15}O_9$ film at 800 °C that is attributed to the larger grain size and increased "rattling space." Furthermore, the vanadium substitution for tantalum ion can effectively reduce the leakage current density as lower as 5×10^{-8} A/cm^2 at an applied electric field of 100 kV/cm because of the reduced mobility of charge carriers. However, the incorporation of vanadium does not cause appreciable negative effect on the fatigue endurance of Sr-deficient $Sr_{0.8}Bi_{2.3}Ta_2O_9$ films until more than 20 mol % ($x=0.2$) vanadium was added. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1480110]

I. INTRODUCTION

$SrBi_2Ta_2O_9$ (SBT) is one of the most promising materials for application to nonvolatile random access memories due to its excellent ferroelectric properties, characterized by limited polarization fatigue and low coercive field.[1,2] SBT belongs to the family of Aurivillius compounds with a general formula of $(Bi_2O_2)^{+2}(A_{m-1}B_mO_{3m+1})^{2-}$, consisting of m-perovskite units sandwiched between bismuth oxide layers. (Here A and B are the two types of cations that enter the perovskite unit. A is Bi^{+3}, Ba^{+2}, Sr^{+2}, Pb^{+2}, or K^{+1}; B is Ti^{4+}, Ta^{+5}, Nb^{5+}, Mo^{6+}, or W^{6+}.)[3,4] The ferroelectric properties, crystal structure and microstructure are remarkably influenced by the composition fluctuation. Atsuki *et al.*[5] reported that the remanent polarization (P_r) of $Sr_xBi_{2z}Ta_2O_9$ ($0.7\leqslant x\leqslant1.0$, $2.0\leqslant2z\leqslant2.6$) increased with the decrease of the Sr/Ta mole ratio for films annealed at 800 °C. A maximum remanent polarization was also reported for the SBT films with 20 mol % Sr-deficient and 10 mol % Bi-excess composition.[6,7] The enhancement in P_r with decreasing Sr content from $x=1.0-0.8$ was attributed to the increase in grain growth. Watanabe *et al.* reported that the dependence of P_r on Sr content in $SrBi_2Nb_2O_9$ (SBN) films was entirely due to the sensitivity of grain orientation to Sr content.[8] The randomly oriented Sr-deficient SBN films have larger remanent polarization than c-axis oriented stoichiometric SBN films. However, the high postannealing temperatures and the low remanent polarization of SBT films are two major barriers for their application to random access memories.[9]

Many physical properties of materials may vary significantly with a subtle change of chemical composition through doping or substituting of desired impurities. The Nb substitution for Ta was also used to enhance the ferroelectric properties.[10,11] The partial substitution of Sr^{2+} by Ba^{2+} has resulted in the noticeable improvement of the dielectric constant and remanent polarization was also observed.[12] An enhanced remanent polarization was also observed in the SBT film with the addition of Pb^{2+} cation.[13]

Although the Sr-deficient SBT films have been reported to exhibit more excellent polarization compared to stoichiometric SBT films; little work has been done to investigate the role of B-site substitution in Sr-deficient SBT system. Several works demonstrate that the additive usually plays an important role in grain growth and has a remarkable effect on electrical properties of materials especially for electronic ceramics.[14] Vanadium oxide (Tm=~690 °C) or vanadium-based multiple oxide system are reported to have lower melting point and used as one effective sintering aid for low-firing ceramics.[15,16] Therefore, vanadium-doped SBT films were chosen to investigate the effect of vanadium incorporation on the structure, surface morphology, and electrical properties of Sr-deficient $Sr_{0.8}Bi_{2.3}Ta_2O_9$ thin films.

II. EXPERIMENT

The starting materials for the metalorganic decomposition process were bismuth 2-ethylhexanoate $\{Bi[CH_3(CH_2)_3CH(C_2H_5)COO]_3\}$, strontium 2-ethylhexanoate $\{Sr[CH_3(CH_2)_3CH(C_2H_5)COO]_2\}$, vanadium isopropoxide $V(OC_3H_7)_5$ and tantalum ethoxide $[Ta(OC_2H_5)_5]$ with 2-ethylhexanoic acid as the solvent. The metalorganic precursors were mixed to form solutions with compositions of $Sr_{0.8}Bi_{2.3}(Ta_{2-x}V_x)O_9$. (SBTV). Prior to film deposition, the substrate (Pt/Ti/SiO_2/Si) was cleaned in acetone and alcohol ultrasonic baths, then blown dry with N_2 gas. The solutions were spin coated onto the substrate at a speed of 3000 rpm. After each coating, the as-deposited film was dried on a hot plate at a temperature about 350 °C to

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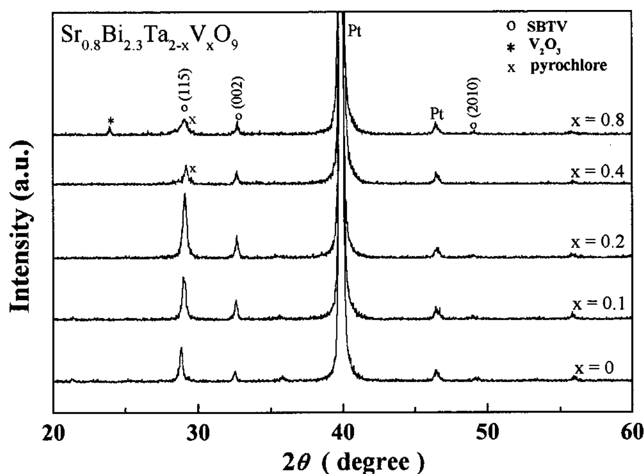

FIG. 1. XRD patterns of SBTV films annealed at 800 °C for 0.5 h.

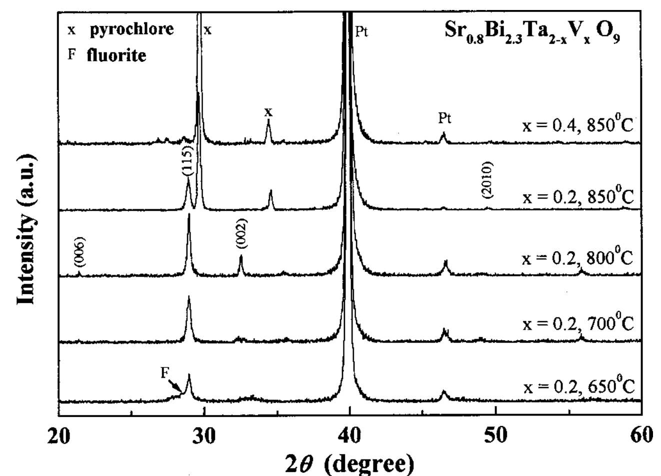

FIG. 2. XRD patterns of $Sr_{0.8}Bi_{2.3}(Ta_{1.85}V_{0.15})O_9$ films annealed at different temperature for 0.5 h.

remove the solvent before application of the next coating. After the process was repeated four times, the as-deposited films were sintered at 500–850 °C in air for 30 min by directly placing the coated substrate into a preheated tube furnace. Most of the data reported here were obtained for films with four layers ($t = 0.37-0.4\ \mu m$).

The crystal structures of the films were analyzed by using Siemens D5000 x-ray diffraction (XRD) with Cu $K\alpha$ radiation and a Ni filter. The chemical composition of the films was determined using inductively coupled plasma mass spectroscopy (Perkin Elmer, SCIEX ELAN 5000). The film thickness was measured by Dektak surface profilometer. Field-emission scanning electron microscopy (FESEM) (Hitachi S4000) was performed to investigate the surface morphology of the films. Patterned top Au electrodes were sputter deposited onto the SBT layers through a shadow mask on an area of 8.0×10^{-4} cm^2 area to define capacitors. A ferroelectric testing system (RT-66A, Radiant Technologies Inc.) operating in the virtual-ground mode was used to obtain the remanent polarization (P_r)-coercive field (E_c) hysteresis characteristics. Fatigue tests of the films were conducted using a bipolar square wave of 5 V at 1 MHz.

III. RESULTS AND DISCUSSION

A. Microstructure and phase evolution

XRD analysis was used to investigate the effect of vanadium content on the structural development of $Sr_{0.8}Bi_{2.3}(Ta_{2-x}V_x)O_9$ films ($0 \leq x \leq 0.8$) annealed at 800 °C for 0.5 h. As shown in Fig. 1, both intensity and sharpness of the (115) oriented peak were enhanced with increasing vanadium content in the range of $x = 0-0.2$ content, suggesting an increase in grain size compared to $Sr_{0.8}Bi_{2.3}Ta_2O_9$ without vanadium. The (115) reflection is slightly shifted to the lower diffraction angle, indicating that the V ions could be dissolved into TaO_6 octahedral structure to substitute for Ta ions. Nevertheless, with the addition of vanadium content above 0.2, the (115)-oriented peak intensity was rapidly decreased along with the accelerating formation of the pyrochlore phase (labeled as x phase in Fig. 1), indicating the layered perovskite structure became unstable. The pyrochlore phase was similar to Bi-deficient pyrochlore as reported by Rodriguez *et al.*[17] XRD studies of $Sr_{0.8}Bi_{2.3}(Ta_{1.8}V_{0.2})O_9$ films also indicate that the films annealed at 500 °C are amorphous. From 600 °C, a broad diffraction peak appears at around $2\theta = 28.5$, which indicates that a mixture phases of fluorite and perovskite are evolved from the amorphous SBTV films. At 650 °C, as shown in Fig. 2, a strong (115) diffraction peak along with a small broad matrix are formed, which indicates that the films are not fully crystallized. However, at 700 °C, well-crystallized perovskite phase has been completely developed in the $Sr_{0.8}Bi_{2.3}(Ta_{1.8}V_{0.2})O_9$ thin films, which is much lower than that of $Sr_{0.8}Bi_{2.3}Ta_2O_9$ films without doping vanadium. In other words, the addition of vanadium lowers the annealing temperature and promotes the crystallization of layered-structure ferroelectric SBTV films. At 850 °C, the diffraction peak around 29.5° labeled with x phase in Fig. 2 becomes stronger and the (115) peak of perovskite phase almost disappears. Even at 800 °C, as the vanadium content (x) was added more than 0.4 in the SBTV film, complete pyrochlore phase with no trace of perovskite has been developed. On the contrary, at 850 °C, although the pyrochlore phase can be detected for the undoped SBT films ($x = 0$), the film is primarily composed of perovskite phase. The findings imply that the addition of vanadium causes perovskite-SBTV more unstable.

Figure 3 illustrates typical (FESEM) surface images obtained from SBTV films having various vanadium contents and heated at 800 °C. The microstructure of SBT ($x = 0$) film presents a rod-like grain matrix. With the addition of vanadium, a dense microstructure consisting of larger and sheet grains was observed for the SBTV films with $x < 0.4$. However, an increase of vanadium content (i.e., $x = 0.4$) leads to the reduction of grain size and the formation of pyrochlore phase. Typical SEM microstructure of SBTV ($x = 0.2$) films annealed at 650–850 °C was shown in the Fig. 4. The film at 600 °C presents featureless microstructure. At 650 °C, a microstructure consisting of rod-like grain matrix with some bright micrograins intervened in between grain boundaries was observed, indicating SBTV perovskite phase has been

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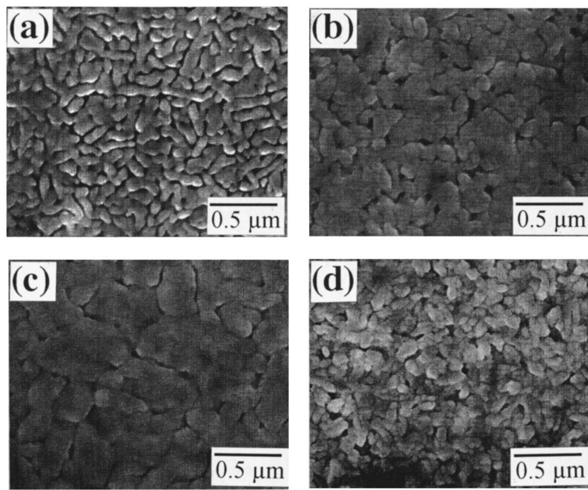

FIG. 3. SEM surface micrographs of SBTV with (a) $x=0$, (b) $x=0.2$, (c) $x=0.4$, and (d) $x=0.8$ films annealed at 800 °C for 0.5 h.

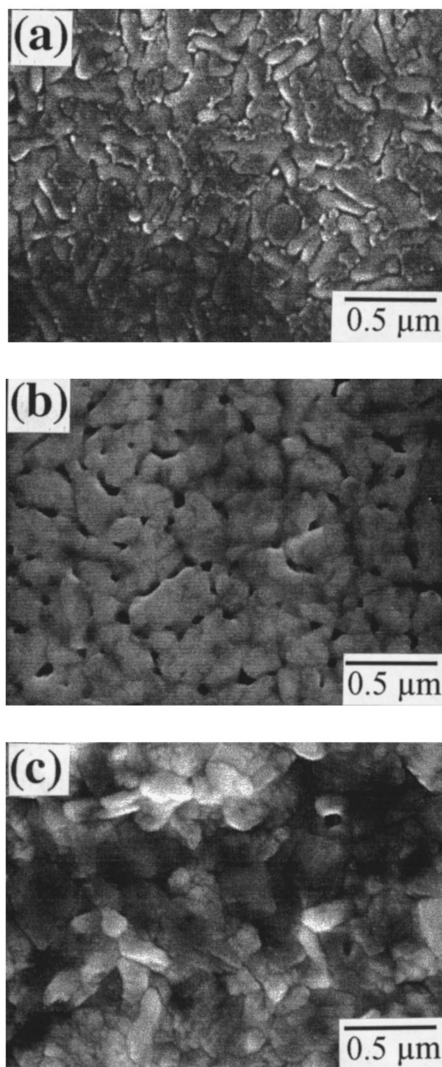

FIG. 4. Phase evolution of $Sr_{0.8}Bi_{2.3}(Ta_{1.85}V_{0.15})O_9$ films annealed at 850 °C for (a) 5, (b) 10, (c) 30, and (d) 60 min.

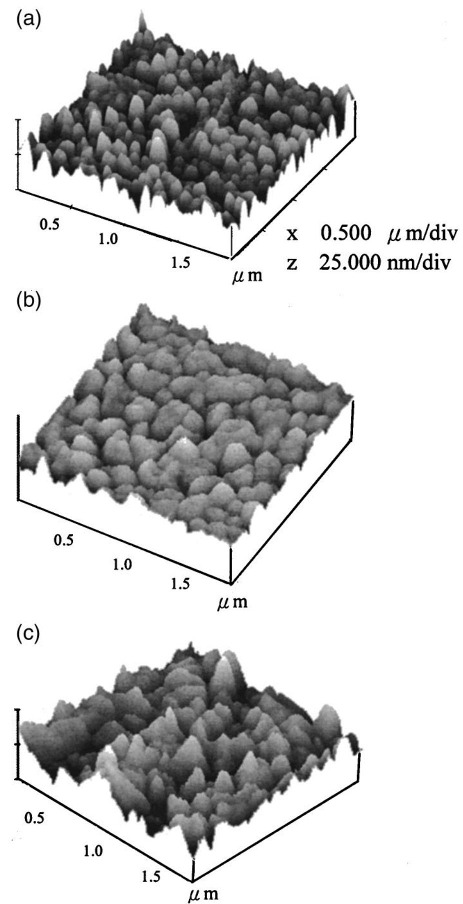

FIG. 5. Atomic force micrographs of SBTV with (a) $x=0$, (b) $x=0.2$, (c) $x=0.4$ films annealed at 800 °C for 0.5 h.

crystallized from the matrix. The micrograins are probable flourite phase as corresponding to the XRD pattern marked with arrow in Fig. 2. At 750 °C, the cluster grains are merged together to form larger sheet grains, which is probably related to the lower melting point of vanadium-based oxide. The average grain size were measured to be about 300–400 nm. At 850 °C, the microstructure exhibits different bright polygonal grains mixed with dark lump area that should be pyrochlore phase or other secondary phases in accordance to the XRD patterns of Fig. 2.

The atomic force microscopy (AFM) images in Fig. 5 indicate that the surface morphology of the 800 °C-annealed SBTV with $x=0.2$ film exhibits smooth. It could be elucidated with the fact that the addition of low melting-point vanadium-based content makes SBTV film relatively easy to merge small grains into large and flat grains during high-temperature annealing. However, excess vanadium (at $x \geqslant 0.4$) may induce the Ti outdiffused from the substrate to react with SBTV film to form pyrochlore phase and thus the film morphology becomes rather rough.

B. Ferroelectric properties

Ferroelectric hysteresis measurements were conducted on $Sr_{0.8}Bi_{2.3}(Ta_{2-x}V_x)O_9$ thin films in metal/ferroelectric/metal configuration at an applied voltage of 6 V using standardized RT 66A ferroelectric test system. The typical hys-

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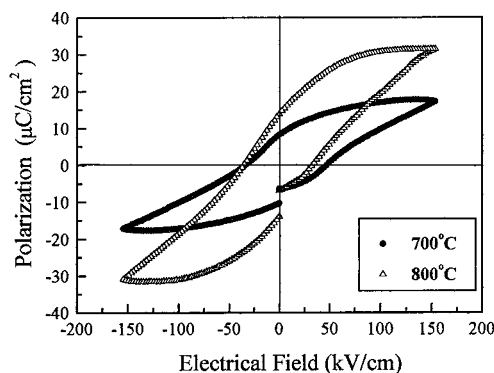

FIG. 6. Hysteresis loops of $Sr_{0.8}Bi_{2.3}(Ta_{1.85}V_{0.15})O_9$ films annealed at 700 and 800 °C for 0.5 h at an applied voltage of 6 V.

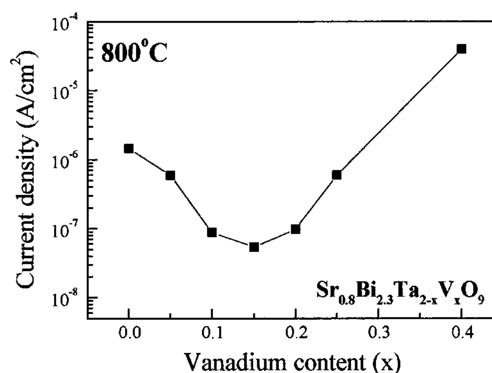

FIG. 8. Current density of vanadium-doped SBTV films at 100 kV/cm electric field.

teresis loops of $Sr_{0.8}Bi_{2.3}(Ta_{1.85}V_{0.15})O_9$ films annealed at 700–800 °C are shown in Fig. 6. It was observed that the saturated hysteresis loops have been developed from the films annealed at 700–800 °C that can be attributed to the enhanced densification due to the addition of vanadium. As illustrated from the SEM images in Fig. 3 for 700– and 800 °C-annealed SBTV films, it can be observed that the grain size of the film at 800 °C is larger but the density shows little difference compared to 700 °C-annealed film. However, the remanent polarization ($2P_r = 21.9\ \mu C/cm^2$) of the $Sr_{0.8}Bi_{2.3}(Ta_{1.85}V_{0.15})O_9$ films annealed at 700 °C is lower than that ($2P_r = 30.5\ \mu C/cm^2$) at 800 °C. The larger grain size is the cause of the larger P_r value for the SBTV films annealed at 800 °C.

Figure 7 illustrates that the 800 °C-annealed SBTV films prepared from precursor solution with an addition of vanadium less than 0.3 exhibit better ferroelectric properties compared to $Sr_{0.8}Bi_{2.3}Ta_2O_9$ thin films. The maximum $2P_r$ value occurs at the SBTV films with $x = 0.15$. Above that ($x = 0.15$), the remanent polarization was found to decrease with increasing vanadium content. The measured remanent polarization ($2P_r$) values are 30.5 $\mu C/cm^2$ and 18.1 $\mu C/cm^2$, respectively, for $Sr_{0.8}Bi_{2.3}(Ta_{1.85}V_{0.15})O_9$ and $Sr_{0.8}Bi_{2.3}Ta_2O_9$ thin films. The improved polarization may be partially attributed to the enhanced grain growth as observed in the SEM micrographs (Fig. 3). Besides the effect of grain size, other effects to account for variation in remanent polarization have to be considered. According to the report of Subbarao for the layered perovskite structure,[18] the enhanced ferroelectric properties in the SBTV films could be well explained by increasing "rattling space" due to the substitution of vanadium for tantalum because pentavalence vanadium ions (V^{5+}, 0.58 Å) have a much smaller size compared with niobium ions (Ta^{5+}, 0.64 Å).[19] However, because V^{5+} can have six coordination of oxygen, it is too small to form a stable layered perovskite structure. As revealed by Wu and Cao, with niobium (Nb^{5+}, 0.69 Å) substituted by vanadium up to 10 mol % ($y = 0.1$) in the strontium bismuth vanadium niobate $[SrBi_2(Nb_{1-y}V_y)_2O_9]$ ceramics, the layered perovskite structure can be preserved.[16] Therefore, in the present work, the peroskite structure is still retained for $Sr_{0.8}Bi_{2.3}(Ta_{2-x}V_x)O_9$ of $x = 0.2$ films but for the SBTV films with the addition of vanadium more than 0.2, i.e., $x = 0.4$, the structure becomes so unstable and the pyrochlore phase is induced. Therefore, the remanent polarization increases significantly from $2P_r = 18.1\ \mu C/cm^2$ for SBT films without vanadium doping to $2P_r = 30.5\ \mu C/cm^2$ for SBTV films of $x = 0.15$ and then rapidly drops to $2P_r = 9.8\ \mu C/cm^2$ for SBTV films of $x = 0.4$.

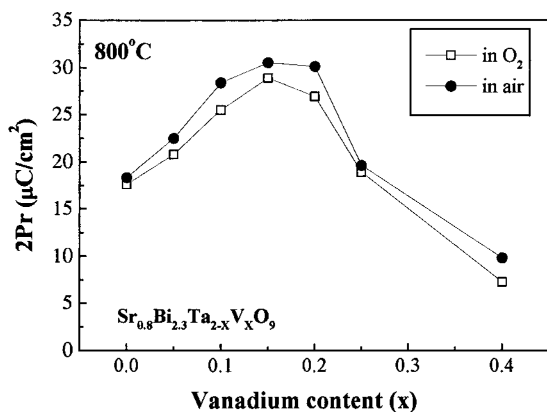

FIG. 7. Remanent polarization ($2P_r$) of vanadium-doped SBTV films annealed at 800 °C for 0.5 h at an applied voltage of 6 V.

C. Leakage current

In general, the occupation of Bi on Sr site often occurs in the Sr-deficient SBT compositions since both radii of Sr^{2+} and Bi^{3+} are very close, where some fraction of the positive charged $Bi_{Sr}^{\bullet}$ ions might be compensated by electrons.[20,21] Therefore, by considering only the most probable defects, Sr-deficient SBT films are characteristic of n type. In our work, a reduced leakage current shown in Fig. 8 was observed for the Sr-deficient SBT films with increasing vanadium up to $x = 0.15$. The phenomenon reveals that the concentration and mobility of intrinsic charge carrier in the Sr-deficient SBT films may decrease due to the incorporation of vanadium cation because a decreased leakage current suggests a reduced concentration of charge carriers. However, if it was assumed that only pentavalent vanadium cation exist in the SBTV films after air annealing, no appreciable oxygen vacancies are created for the samples with vanadium doping. Therefore, the decreased leakage current could be explained by two possible mechanisms. One is that the reduced mobil-

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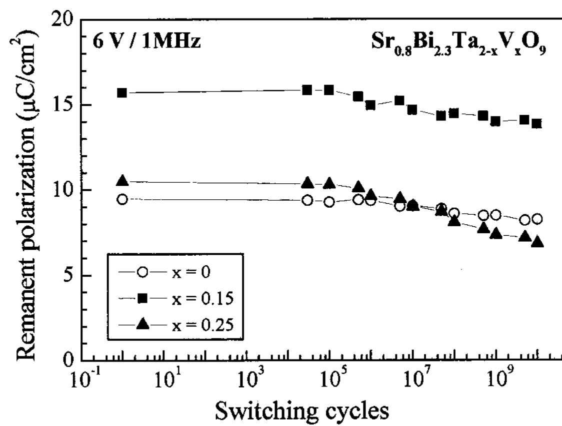

FIG. 9. Fatigue behavior of $Sr_{0.8}Bi_{2.3}(Ta_{2-x}V_x)O_9$ films with an applied voltage of 6 V at 1 MHz.

ity of charge carriers may result from the distortion and complexity in the crystal structure due to the substitution of tantalum by smaller vanadium ion. As the vanadium doping concentration exceeds a critical point, the perovskite structure becomes unstable and even destructive, resulting in pyrochlore phase formation as evidenced in Fig. 1. Consequently, an increased leakage current was observed in the SBTV films added with vanadium (x) exceeding 0.3.

Another possibility is that the vanadium doping does not only enhance densification but also make film surface smooth. As shown in Fig. 5, the AFM analysis of the SBTV films shows that the SBTV films of $x<0.3$ exhibit flat and dense microstructure. Above that concentration ($x=0.3$), the film surface becomes rather rough, implying that the films have higher leakage current. Therefore, the lowest leakage current occurs at the SBTV film with $x=0.15$ composition. The leakage current of $Sr_{0.8}Bi_{2.3}(Ta_{1.85}V_{0.15})O_9$ films was measured around 5×10^{-8} A/cm^2 at an electric field of 100 kV/cm, much lower than that (1.5×10^{-6} A/cm^2) of undoped SBT film by proximately one and half orders of magnitude.

D. Fatigue behavior

Ferroelectric thin films of bismuth-containing layered perovskite such as $SrBi_2Ta_2O_9$ and $SrBi_2Nb_2O_9$ have been reported to have excellent good fatigue resistance. Fatigue endurance was tested with 1 MHz bipolar pulses at 6 V. Figure 9 shows the fatigue behavior of SBTV ($x=0$, 0.15, and 0.25) films prepared at 800 °C for 0.5 h. Although Sr-deficient $Sr_{0.8}Bi_{2.3}Ta_2O_9$ film shows an optimal remanent polarization of $P_r=9.7$ μC/cm^2, a loss of P_r of 12% was measured after 10^{10} cycles. On the other hand, for the SBT films with vanadium added ($x=0.15$), no rapid fall off in the P_r value was observed up to about 10^8 cycles, but the decay in P_r was about 10% of the initial value after 10^{10} cycles. This indicates that the incorporation of limited vanadium into the layered perovskite structure does not cause appreciable fatigue. However, the decay in remanent polarization becomes apparent with increasing vanadium content above 20 mol % ($x=0.2$) such as $Sr_{0.8}Bi_{2.3}(Ta_{1.75}V_{0.25})O_9$ film, indicating that the addition of vanadium seems detrimental to fatigue resistance of SBT films. There are several proposals that attempt to explain the fatigue behavior of ferroelectric thin films especially for ferroelectric lead zirconate titanate (PZT).[22–24] Oxygen vacancy and electron/hole injection have been suggested as possible mechanisms for the fatigue of PZT films. In our case, no oxygen vacancy we produced because both Ta and V have the identical valence state. Therefore, the oxygen vacancy mechanism is not applicable in explaining the fatigue behavior of SBTV films. However, as more than 20 mol % ($x=0.2$) vanadium was added, the increased fatigue behavior may be correlated with the increase of lattice distortion and the suppression of domain switching because of the structural complex. Further work is underway to understand the effect of vanadium doping on the fatigue properties of the SBT-based layered perovskite structures.

IV. CONCLUSIONS

(1) Enhanced ferroelectric properties and lower processing temperature of $Sr_{0.8}Bi_{2.3}Ta_2O_9$ films deposited on Pt/Ti/SiO_2/Si substrates can be obtained by doping vanadium.

(2) The measured remanent polarization ($2P_r$) values increases from 18.1 μC/cm^2 for $Sr_{0.8}Bi_{2.3}Ta_2O_9$ film to 30.5 μC/cm^2 for $Sr_{0.8}Bi_{2.3}Ta_{1.85}V_{0.15}O_9$ film at an applied voltage of 6 V, respectively.

(3) The incorporation of vanadium doping can lead to an increase in leakage resistance of $Sr_{0.8}Bi_{2.3}Ta_2O_9$ film. The leakage current as lower as 5×10^{-8} A/cm^2 can be obtained at an applied electric field of 100 kV/cm.

(4) The substitution of limited vanadium for tantalum does not cause appreciable fatigue. However, the addition of more than 20 mol % vanadium seems to adversely affect the fatigue resistance of $Sr_{0.8}Bi_{2.3}Ta_2O_9$ films due to large lattice distortion.

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