Developmental mechanism for the resistance change effect in perovskite oxide-based resistive random access memory consisting of $Bi_2Sr_2CaCu_2O_{8+\delta}$ bulk single crystal

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Resistive random access memory (ReRAM) structures of M/$Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi-2212) bulk single crystal/Pt (M = Al, Pt) were prepared and their memory characteristics and superconducting properties were evaluated. The resistance change effect developed only in the Al/Bi-2212/Pt structure and was enhanced with decreasing critical temperature by annealing in Ar atmosphere. Due to the large resistance anisotropy of bulk Bi-2212 single crystals, the resistance change effect was confirmed to occur at the interface between the Al electrode and the Bi-2212 single crystal. These results indicate that introduction of an oxygen-depleted layer to the Bi-2212 single crystal is required to develop the resistance change effect, which could be achieved by the deposition of electrodes with low Gibbs free energy and raising the temperature to exceed the activation energy for oxygen ions to move from Bi-2212 to the electrode. A model is proposed to explain the resistive switching of perovskite oxide-based ReRAM by generation/recovery of the oxygen-depleted layer. The resistance change effect developed also in the Pt/Bi-2212/Au structure annealed in hydrogen gas, in which an oxygen-depleted layer is formed with the assistance of catalytic effect of Pt on the surface of the Bi-2212 at the Pt/Bi-2212 interface, proving the validity of the model. © *2011 American Institute of Physics*. [doi:10.1063/1.3651465]

I. INTRODUCTION

Resistive random access memory (ReRAM) has a structure where a transition metal oxide (TMO) is sandwiched between top and bottom electrodes. This simple structure enables high integration and has attracted attention as a nonvolatile memory to replace flash memory. The resistance change effect of perovskite oxide-based ReRAM, in which perovskite oxides such as $Pr_{1-x}Ca_xMnO_3$ (PCMO)[1–5] and $SrTiO_3$ (Refs. 6 and 7) are used as the TMO layer, is thought to be related to the migration of oxygen ions. However, the switching mechanism is yet to be clarified.

One factor that hinders elucidation of the mechanism could be attributed to the use of thin films. The presence of grain boundaries may affect electrical conduction and the migration of oxygen ions when polycrystalline thin films are used.[3,4,6,8] Mismatching with a substrate introduces stress into the film and complicates elucidation of the mechanism, even if epitaxially grown TMO films are used.[1,5,7,9,10]

Therefore, introduction of a bulk single crystal as the TMO layer would be effective for elucidation of the switching mechanism. In addition, the superconducting critical temperature (T_c) for a single crystal of the high temperature $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi-2212) superconductor is strongly dependent on the oxygen content.[11,12] Therefore, the dependence of the resistance change effect on the oxygen content can be obtained by evaluating the relation between the resistance change effect and T_c. The large crystalline anisotropy of Bi-2212 single crystal enables cleavage of the crystal into thin plates with clean and flat surfaces.[13,14] Above all, for elucidation of the resistance change effect, it is most important to specify where the resistance change occurs.

In this paper, the resistance change effect of ReRAM was investigated using M/Bi-2212 single crystal/Pt (M = Al, Pt) structures. It was clarified that the resistance change effect was caused at the interface of Al and Bi-2212 in the Al/Bi-2212/Pt structure and was enhanced by annealing the structure. This suggested that the resistance change effect occurs in the oxygen-depleted layer of the Bi-2212 single crystal formed at the interface between an electrode with low Gibbs free energy and a Bi-2212 single crystal.

II. EXPERIMENTAL

Bi-2212 single crystals were grown using the vertical Bridgman method[15,16] and were cleaved under the ambient atmosphere to produce thin plates with typical dimensions of $2.0 \times 1.5 \times 0.02$ mm^3. All the cleaved crystals were annealed in flowing O_2 at 500 °C for 20 min to induce uniform oxygen content. Al and Pt electrodes were deposited, respectively, on both of the surfaces (*a-b* plane) of the Bi-2212 single crystals by sputtering to produce M-TE/Bi-2212/Pt-BE (M = Al, Pt) structures, where TE and BE represent top and bottom electrodes. The size and thickness of both the TE and BE were 1.0×1.0 mm^2 and 100 nm, respectively. Current-voltage (*I-V*) characteristics were measured using a semiconductor parameter analyzer (Agilent 4155 C).

The BE was grounded and a bias voltage was applied to the TE. The compliance current was set to 60 mA, where the compliance current is a limiting value to which a current

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flow through the M-TE/Bi-2212/Pt-BE structure is limited during the set process [resistive switching from a high resistance state (HRS) to a low resistance state (LRS)]. The samples were annealed in 100% Ar atmosphere at 300 and 400 °C for 60 min to control T_c, and the correlation between the resistance change effect and T_c was investigated. In addition, a Ag electrode, used for monitoring interface resistance between the M-TE and the Bi-2212 crystal and between the Pt-BE and the Bi-2212 crystal, was formed on the same side of the Bi-2212 single crystal as the TE. The Pt-TE/Bi-2212/Au-BE structure was annealed in Ar + H_2 (Ar:H_2 = 19:1) atmosphere at 400 °C for 10 min to introduce an oxygen-depleted layer into the surface of the Bi-2212 single crystal in the vicinity of the Pt-TE by reduction effect of H_2 gas with the assistance of the catalytic effect of Pt.[17–19]

III. RESULTS AND DISCUSSION

Figure 1(a) shows the *I–V* characteristics of the as-prepared Pt-TE/Bi-2212/Pt-BE structure and those annealed in Ar atmosphere at 300 °C for 60 min and at 400 °C for 60 min. The voltage was ramped up from 0 V to +1.0 V, and then down to 0 V in steps of 10 mV. The voltage was then ramped down from 0 V to −1.0 V, and then back to 0 V in steps of 10 mV. No resistance change effects and no significant differences were observed in the *I–V* characteristics, independent of annealing temperature.

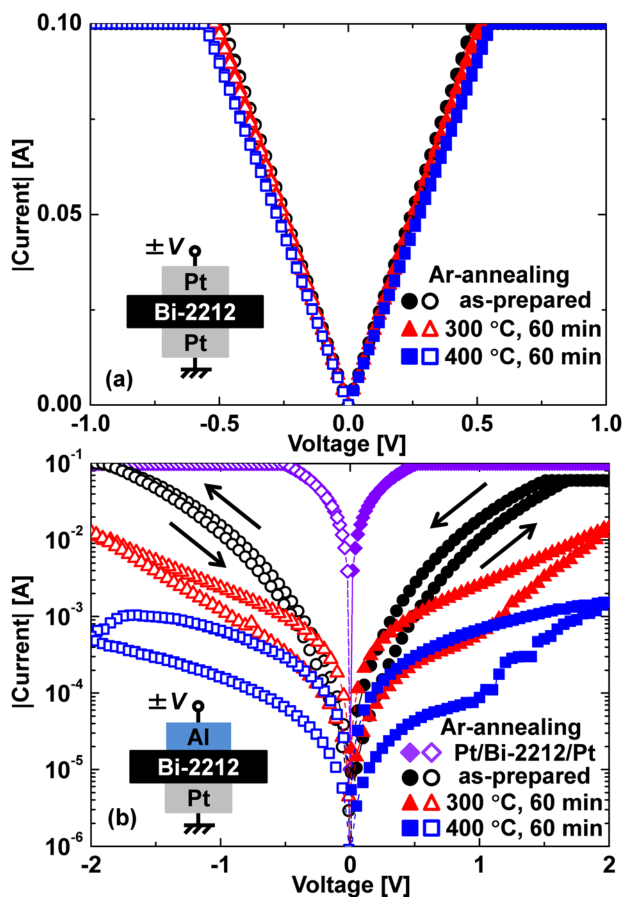

FIG. 1. (Color online) *I-V* characteristics of as-prepared and annealed (a) Pt-TE/Bi-2212/Pt-BE and (b) Al-TE/Bi-2212/Pt-BE structures. The device structures are shown in the insets.

Figure 1(b) shows the *I–V* characteristics of the as-prepared Al-TE/Bi-2212/Pt-BE structure and those annealed in Ar atmosphere at 300 °C for 60 min and at 400 °C for 60 min. Set and reset switching occurred by application of positive and negative voltages, respectively, where reset represents a switching from LRS to HRS. The ratio of R_{HRS} to R_{LRS} (R_{HRS}/R_{LRS}) increased with increasing annealing temperature, where R_{HRS} and R_{LRS} represent the resistances in the HRS and LRS, respectively. R_{HRS}/R_{LRS} of the as-prepared sample was 2, whereas those annealed at 300 and 400 °C were increased to 10 and 20, respectively. In addition, the initial resistances of the as-prepared Pt-TE/Bi-2212/Pt-BE and Al-TE/Bi-2212/Pt-BE structures were 5.1 Ω and 1.3 kΩ, respectively, and those of the Al-TE/Bi-2212/Pt-BE structures annealed at 300 and 400 °C were 2.5 and 12.7 kΩ, respectively. The resistance of the as-prepared Al-TE/Bi-2212/Pt-BE structure is higher than that of the as-prepared Pt-TE/Bi-2212/Pt-BE structure, and the resistance of the Al-TE/Bi-2212/Pt-BE structure increased with the annealing temperature. The Gibbs free energies of Pt and Al at 300 (600) K are 92.852 (9.356) kJ/mol and −1690.973 (−1717.192) kJ/mol,[20] respectively; therefore, the increase in the initial resistance was caused by reduction of Bi-2212 due to oxidation of the Al electrode. This is consistent with the large oxygen diffusion coefficient of Bi-2212 (1.6×10^{-17} cm^2/s), even at 300 K.[21] These results suggest that reduction of Bi-2212 is required for the development of the resistance change effect and that the effect was enhanced by the extent of reduction.

To specify where the resistance change effect occurs, two extreme cases shown in Figs. 2(a) and 2(b) are discussed for TE/TMO/BE structures which have a monitoring electrode (ME) on the same side of the TMO layer as TE. Figure 2(a) shows the TE/TMO/BE structure using the TMO layer with small resistance anisotropy. When bias voltage is applied between the TE and ME, current dominantly flows through the BE. This is due to the fact that a film thickness of a TMO layer is, in general, much smaller than a TE-ME distance. Therefore, we can regard Fig. 2(a) as the circuit given by connecting the ME/TMO/BE and BE/TMO/TE structures in series. In this case, resistances of the TE/TMO and TMO/BE interfaces cannot be measured independently because current flows through the both interfaces. On the other hand, Fig. 2(b) shows the TE/TMO/BE structure using the TMO layer with large resistance anisotropy such as a Bi-2212 bulk single crystal as the TMO layer. When bias voltage is applied between the TE and ME, a current

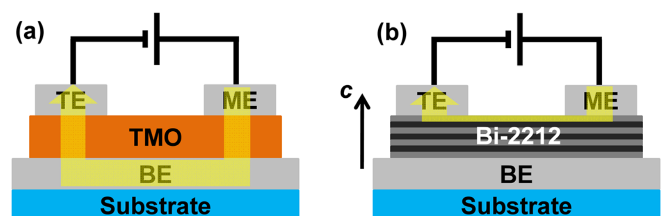

FIG. 2. (Color online) Schematics to explain current paths in TE/TMO/BE structures which have ME for the TMO layers with (a) small and (b) large resistance anisotropies.

dominantly flows along the surface of the Bi-2212 (*a-b* plane) between TE and ME due to its large resistance anisotropy ($\rho_c/\rho_{ab} > 10^3$).[13] Therefore, the resistances of the TE/TMO and TMO/BE interfaces can be obtained directly by the measurement of resistances between TE-ME and ME-BE, respectively.

To specify where the resistance change of the Al-TE/Bi-2212/Pt-BE structure takes place, a sample with a Ag-ME (D) on the same side of the Bi-2212 single crystal as the TE was prepared, as shown in Fig. 3(a). Set voltages ($+V$) and reset voltages ($-V$) were alternately applied between terminals A and C (A-C). At the same time, resistances between terminals A and D (A-D) and between terminals C and D (C-D) were measured. Terminal B was located at a different position than terminal A on the Al electrode, and the resistance between terminals A and B (A-B) was also measured. The results are shown in Fig. 3(b). The resistance between A-C was alternately switched between low and high resistance by application of $+V$ and $-V$, respectively. The resistance between A-D changed in accordance with the change of resistance between A-C. In contrast, the resistances between A-B and C-D were invariably independent of the resistance between A-C. The results indicate that the resistance change of the Al-TE/Bi-2212/Pt-BE structure occurs at the Al-TE/Bi-2212 interface.

Figure 4(a) presents the resistivity-temperature (ρ-T) characteristics of an as-prepared Bi-2212 single crystal and those annealed in Ar atmosphere at 300 °C for 60 min and 400 °C for 60 min. The ρ-T measurement was performed using four-terminal method with four Pt electrodes. The inset shows four Pt electrodes formed side by side on the surface of the Bi-2212 single crystal, where the pairs of outer and inner electrodes were used as the current and voltage terminals, respectively. An enlarged view of the ρ-T characteristics around T_c is shown in the inset. No significant change in the ρ-T characteristics was observed, independent of the annealing temperature. The same measurements were performed for a sample on which an Al electrode was deposited between the voltage terminals and the results are shown in Fig. 4(b). The T_c of the as-prepared Bi-2212 single crystal with the Al electrode was 84 K, which was lower than the T_c of 88 K for the sample without the Al electrode. In addition, the T_c of the Bi-2212 single crystal with the Al electrode was

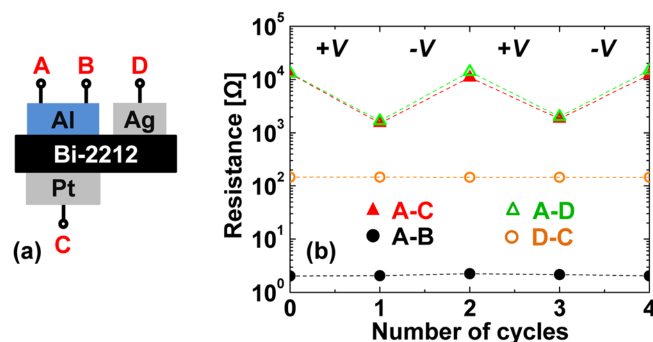

FIG. 3. (Color online) (a) Al-TE/Bi-2212/Pt-BE structure with a Ag-ME (terminal D) and (b) resistances measured between A-B, A-D, and D-C when the resistance between A-C was changed.

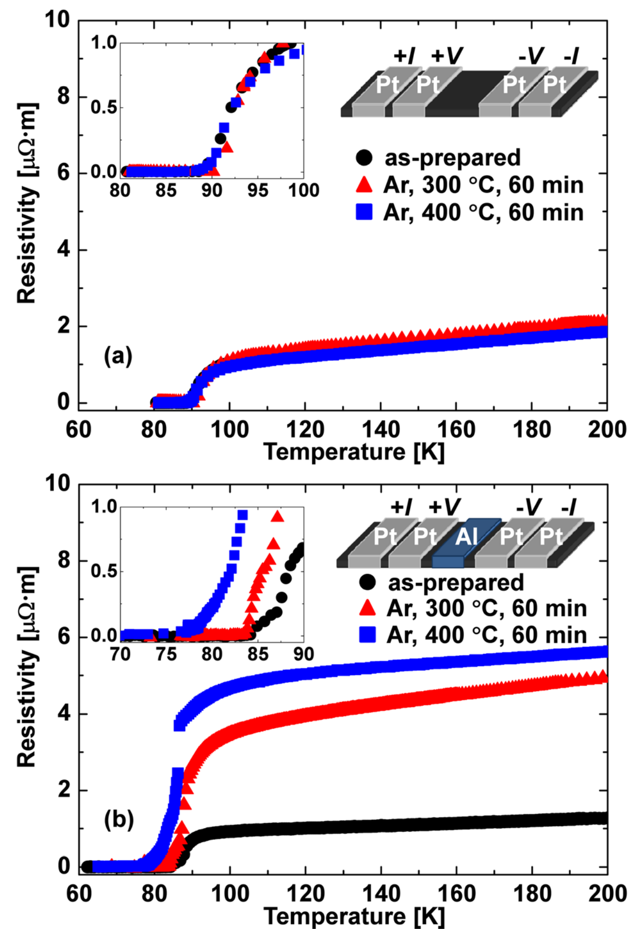

FIG. 4. (Color online) ρ-T characteristics of Bi-2212 single crystal measured using the four-terminal method with four Pt electrodes (a) before and (b) after Al deposition between the voltage terminals. Enlarged views of the ρ-T characteristics around T_c are shown in the insets.

decreased to 83 and 75 K when annealed in Ar atmosphere at 300 and 400 °C for 60 min, respectively. It is well known that both the resistivity in the normal conducting state and T_c are strongly dependent on the oxygen content of the Bi-2212 crystal;[11,12] the resistivity increases with decreasing oxygen content, whereas T_c decreases. An increase in the resistivity and decrease in T_c were observed only in the sample with the Al electrode, which suggests that the Al electrode removes oxygen from the Bi-2212 single crystal and an oxygen-depleted layer is formed in the Bi-2212 single crystal in the vicinity of the Al electrode.

Free energies for the states of $Al + Bi_2Sr_2CaCu_2O_{8+\delta 1}$ (α) and $AlO_x + Bi_2Sr_2CaCu_2O_{8+\delta 2}$ (β) are represented in Fig. 5. A shift of the energy state from α to β corresponds to a reduction of the Bi-2212 single crystal due to oxidation of the Al electrode by oxygen diffusion from the Bi-2212 single crystal to the Al electrode. The reaction rate, $v \propto \exp(-E_a/k_BT)$, is dependent on the annealing temperature T, and it is necessary to exceed the activation energy E_a, for the reaction from α to β to proceed. Accordingly, the resistance of the Al-TE/Bi-2212/Pt-BE structure is increased with increasing T. In contrast, oxygen ions will not move at the interface between the Pt electrode and the Bi-2212 crystal, because Pt is not easily oxidized. The reason for the higher resistance measured in the as-prepared Al-TE/Bi-2212/Pt-BE structure

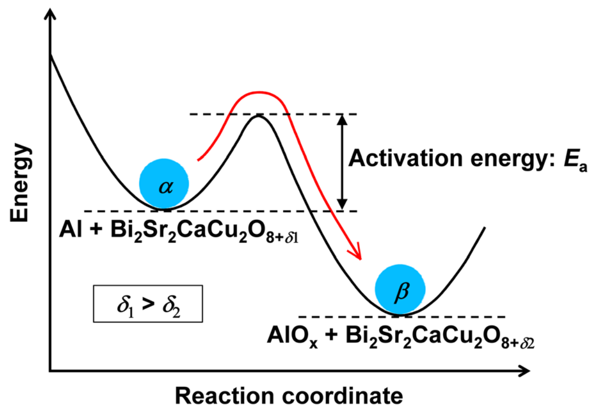

FIG. 5. (Color online) Free energies for the states of $Al + Bi_2Sr_2CaCu_2O_{8+\delta 1}$ α and $AlO_x + Bi_2Sr_2CaCu_2O_{8+\delta 2}$ β in the interface between Al-TE and Bi-2212 and the activation energy for oxygen ions to move from Bi-2212 to the electrode.

than that in the as-prepared Pt-TE/Bi-2212/Pt-BE structure is attributed to the sputtering energy during Al deposition. The radiant heat of the Ar plasma and kinetic energy of sputtering particles raised the temperature of the sample, which enabled partial reaction from α to β. Shono *et al.*[3] reported that a 10 nm thick TiO_x layer was naturally formed at the as-deposited interface between Ti and PCMO without annealing of the sample. Therefore, the reaction from α to β is caused by deposition of an electrode with low Gibbs free energy onto the Bi-2212 and heating it to a temperature corresponding to the activation energy. No resistance change occurred in Pt-TE/Bi-2212/Pt-BE structure, which suggests that the introduction of an oxygen-depleted layer into Bi-2212 is required for the development of the resistance change effect.

Figure 6 shows schematics that indicate how the resistance change effect develops and how the resistive switching between the LRS and HRS occurs. First, an electrode with low Gibbs free energy (Al) receives oxygen from the Bi-2212 single crystal, and an oxygen-depleted layer is formed in Bi-2212 in the vicinity of the Al-TE, which results in the HRS [Fig. 6(a)]. Second, by applying positive voltage to the TE, oxygen ions in the bulk of the single crystal are drawn to the TE side by coulombic forces with the assistance of Joule heat. Therefore, the oxygen-depleted layer is partly recovered and the LRS is attained [Fig. 6(b)]. Application of negative voltage to the TE results in the movement of oxygen ions into the partly recovered oxygen-depleted layer of the single crystal bulk and the oxygen-depleted layer is formed again to give the HRS [Fig. 6(c)]. If the resistance change occurred due to redox reaction of the Al-TE at the Al-TE/Bi-2212 interface, the relationship between the resistance change and the bias polarity opposite to that observed in this study should be observed.[3,4] Therefore, the resistance change effect is caused by generation/recovery (reduction/oxidation) of the oxygen-depleted Bi-2212 layer formed at the interface between Al and Bi-2212. The most direct way to prove the validity of the proposed resistance change model is to show the development of the resistance change effect simply by inserting the oxygen-depleted Bi-2212 layer at the interface between a high Gibbs free energy electrode such as Pt and Bi-2212 layer. Utilizing the catalytic effect of Pt that drastically enhances reduction reaction of hydrogen,[17] we can introduce the oxygen-depleted Bi-2212 layer into the Pt/Bi-2212 interface by annealing a Pt-TE/Bi-2212/Au-BE structure in H_2 atmosphere. Since the Au is resistant to hydrogen and has high Gibbs free energy of −42.447 kJ/mol at 300 K,[20] the Bi-2212 layer is not reduced at the Bi-2212/Au interface during the H_2 annealing. Figure 7 shows the *I-V* characteristics of the as-prepared Pt-TE/Bi-2212/Au-BE structure and those annealed in Ar and Ar+H_2 atmospheres at 400 °C for 60 and 10 min, respectively. Resistance change did not occur in the as-prepared sample and that annealed in Ar atmosphere. On the other hand, the resistance change effect was observed in the sample annealed in Ar+H_2 atmosphere. Here, set and reset switching occurred by application of positive and negative voltages, respectively.

To specify where the resistance change of the Pt-TE/Bi-2212/Au-BE structure takes place, a sample with a Ag-ME (D) was prepared, as shown in Fig. 8(a). Set voltages ($+V$) and reset voltages ($-V$) were alternately applied between A-C. The results are shown in Fig. 8(b). The resistance between A-C was alternately switched between low and high resistance by application of of $+V$ and $-V$ respectively. The resistance between A-D changed in accordance with the change of resistance between A-C. In contrast, the resistances between A-B and C-D were invariably independent of the resistance between A-C. The results indicate that the

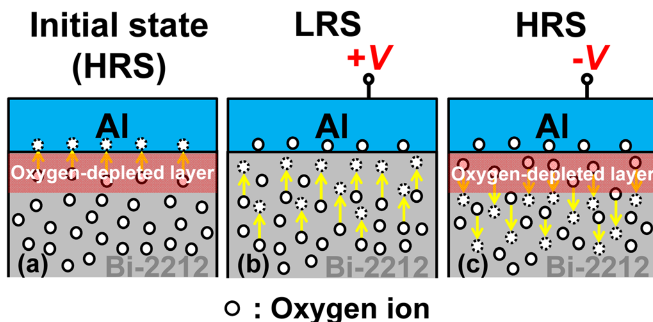

FIG. 6. (Color online) Schematics to indicate the oxygen movement until stabilization of the initial state (a) and during the resistive switching to LRS (b) and to HRS (c).

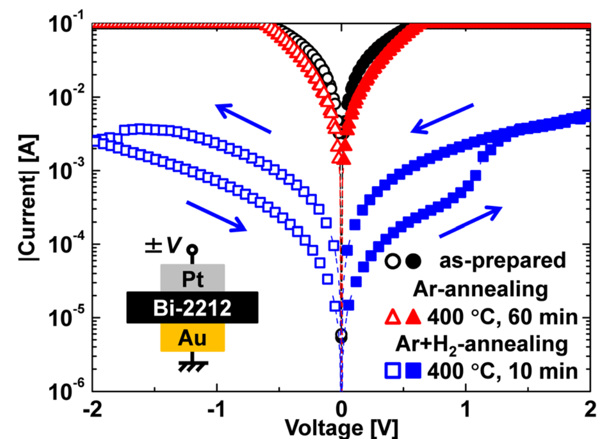

FIG. 7. (Color online) *I-V* characteristics of the as-prepared Pt-TE/Bi-2212/Au-BE structure and those annealed in Ar and Ar+H_2 atmospheres at 400 °C for 60 and 10 min, respectively. The device structure of Pt/Bi-2212/Au is shown in the inset.

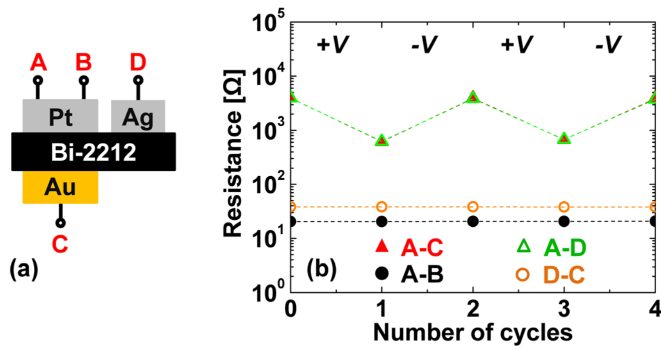

FIG. 8. (Color online) (a) Pt-TE/Bi-2212/Au-BE structure with a Ag-ME (terminal D) and (b) resistances measured between A-B, A-D, and D-C when the resistance between A-C was changed.

resistance change of the Pt-TE/Bi-2212/Au-BE structure occurs at the Pt-TE/Bi-2212 interface. In addition, resistive switching did not be observed in a Pt-TE/Bi-2212/Au-BE structure in which Pt-TE was deposited after $Ar + H_2$ annealing. Therefore, it was shown that an oxygen-depleted Bi-2212 layer was formed in the Bi-2212 single crystal in the vicinity of the Pt-TE due to the reduction effect of the $Ar + H_2$ annealing[18,19] and the catalytic effect of the Pt-TE.[17] These results as well as the bias polarity dependence of the resistance change indicate that the resistance change is caused by generation/recovery of the oxygen-depleted Bi-2212 layer as shown in Fig. 6. A similar resistance change effect was also reported in Ag/PCMO structures.[2] The resistance change of Ag/PCMO structures was reported to be caused by destruction/repair of the conductive Mn-O chain caused by a change in the concentration of oxygen ions near the Ag/PCMO interface, and which was enhanced in a PCMO film grown under oxygen deficient conditions compared with that grown under oxygen rich condition. Accordingly, a reduction of oxide ions near the structure interface plays a key role in the development of the resistance change effect by providing space for oxygen ions to migrate. A similar scenario may also be applicable to other perovskite oxides, independent of whether the TMO is a single crystal or poly crystalline. Considering the Gibbs free energies of the oxides and electrodes, the resistance change effect of perovskite oxide-based ReRAM can be controlled by application of an appropriate annealing temperature that exceeds the activation energy for oxygen diffusion from the TMO to the electrode.

IV. CONCLUSION

Perovskite oxide-based ReRAM was prepared using $Bi_2Sr_2CaCu_2O_{8+\delta}$ bulk single crystal for the TMO layer. The resistance change effect was observed in the Al-TE/Bi-2212/Pt-BE structure. An introduction of the Bi-2212 bulk single crystal enabled clarification that the resistance change effect occurs at the Al/Bi-2212 interface. An increase in the resistivity and decrease in the T_c with increased annealing temperature were confirmed, and the resistance change effect (R_{HRS}/R_{LRS}) was enhanced by increased annealing temperature. These results indicate that the introduction of an oxygen-depleted layer into the Bi-2212 single crystal is required for the development of the resistance change effect, which can be achieved by deposition of a low Gibbs free energy electrode in order to form an oxygen-depleted layer at the TMO surface. The resistance change effect developed even in the Pt-TE/Bi-2212/Au-BE structure, which has high Gibbs free energy electrodes, by inserting the oxygen-depleted layer into the surface of the Bi-2212 single crystal at the vicinity of the Pt-TE. This excludes the possibility that resistance change occurs due to the oxidation/reduction of the Al-TE. The resistance change of ReRAM is caused by the migration of oxygen ions under application of an intense electric field, and it is thought that the set/reset processes are caused by recovery/generation of the oxygen-depleted layer. This resistance change model provides a guideline for the selection of oxide and electrode materials for perovskite oxide-based ReRAM.

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