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Electron Holography of a Hetero-Interface in a Solid Oxide Fuel Cell.

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Electrostatic potentials at the interface of an oxide ionic conductor and a metal electrode are observed with applying an external electric field.
Solid oxide fuel cells (SOFC) are expected as a new large scale energy source, which is operated at a high temperature. It has many advantages to other fuel cells such that many kinds of fuel gas can be utilized, Pt catalysts are not required, high electricity-generation efficiency is expected and so on. It is required, however, to solve the problem of overpotential effect, that is, a drawable voltage decreases from the expected value in a stable state as an operating current. One of the origins of this effect is to the stacking oxygen ions near an anode or the lack of reserved oxygen ions near a cathode.
If an external electric field is applied to the solid electrolytes in a vacuum, oxygen ions may be drawn to the anode. The shift of oxygen ions causes the shift of the potential, then the ion flow might be blocked unless new oxygen ions are reserved from a cathode. This is the same phenomenon as the overpotential effect.

Electron holography has been utilized to the observation of electrostatic inner potentials at the interface of an oxide-ionic-conductor, gadolinium-doped-ceria (GDC), and a platinum electrode.

A noble specimen holder for transmission electron microscopy has been developed, in order to applying an electric field to the specimen [1]. The holder shown in Fig.1 has four electrodes, two of them are for heating the specimen and the other two are for applying the voltage to the specimen up to 5V. It can heat the specimen up to 800 degrees in centigrade.
Specimen was mounted on the edge of the Ta heater, and connected with one of the electrodes on the specimen holder with an Au thin wire. The GDC sample was deposited by Pulse Laser Deposition (PLD) method on a Si wafer and a Pt electrode was deposited on the GDC layer. A specimen prepares by a micro-sampling with a focused ion beam (FIB) for the cross-sectional electron-holographic observation.

Figure 2 shows the secondary ion microscope image of the specimen mounted on the side edge of a Ta heater of 6mm long, 0.5mm wide and 0.03mm thick. The Au lead wire is 5μm in diameter.

The inner potentials are investigated with the voltage applied up to ±1.0V. Figure 3a shows the mean inner potential without the external field, where positive potential corresponds to the plus electron phase shift. The Pt has larger mean inner potential than the GDC, and the dip in the phase profile (Fig.3b) shows the interface. Figures 3c and 3d show phase distribution image and its profile with applying the external voltage of V= −1.0V to the Pt electrode. Subtraction of the phase distribution Fig. 3a from Fig. 3c is shown in Figs. 3e and 3f. This difference shows a pure effect of external voltage on the electrostatic potential. Figure 4a shows the same profile as in Fig.3f and Fig.4b shows that when V= +1.0V applied to the Pt electrode.

External voltage should increase the potential toward a positive electrode (right hand side in Fig.3d).

However, the profile of the phase difference, Figs.4a and 4b, shows that an electric double layer was formed at the interface and that anions, oxygen ions, in the GDC were drawn toward the positive side and stacked in the area not so far from the anode, and reduced the potential partially.
The thickness of the specimen has not been defined, nor is the exact value of the potential determined. It was confirmed for the first time, however, the localization of oxygen ions was caused by an external electric field in solid electrolytes, using the in-situ electron holography.

References

[1] H. Moritomo et al., Proc. 16th International Microscopy Congress, Sapporo, 2006, **2**, 1154.
[2] The present work was supported by the Grant-in-Aid for Scientific Research on Priority Area, “Nanoionics” (439) by the Ministry of Education, Culture, Sports, and Technology, Japan.

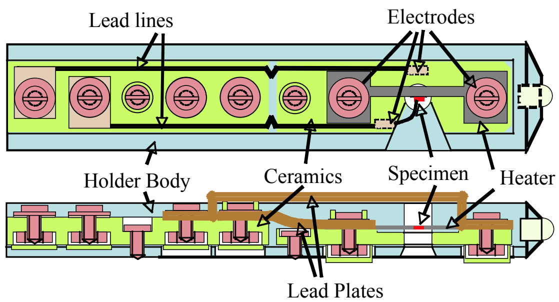

Fig.1. New specimen holder with 4 electrodes.

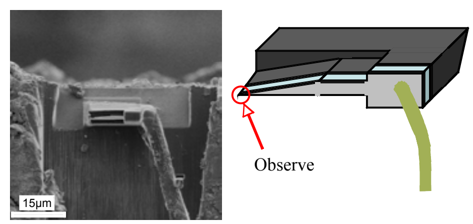

Fig.2. Specimen mounted on the side edge of the heater.

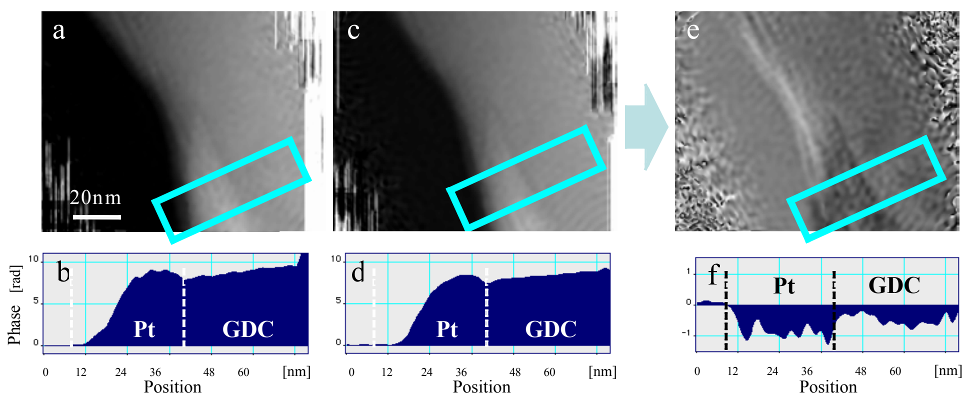

Fig.3. Reconstructed phase images and their line profiles, (a) and (b):without an external field, (c) and (d): with applying -1.0V to the Pr electrode, (e) and (f):subtracted (a) from (b). Frames are areas averaged for profiling.

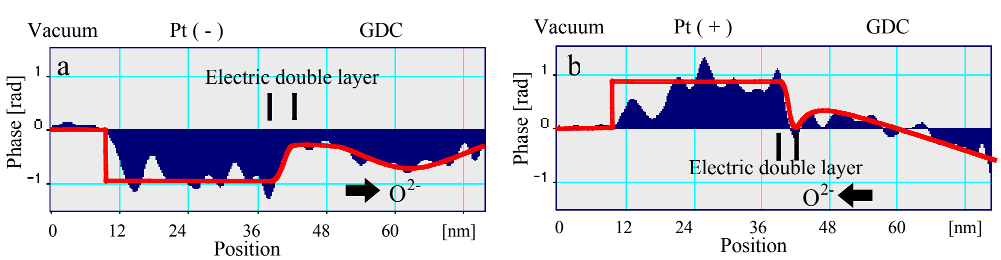

Fig.4. Phase differences between that of -1.0V and 0 (a), and that between +1.0V and 0 (b) show electric double layers and localization of oxygen anions. Solid red lines correspond to electrostatic potential.