In-situ ETEM Observation of Change in CaO Powder Composition by High-humidity Gas

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In previous studies, environmental transmission electron microscopy (ETEM) was used to observe the changes in microstructures of materials in gas atmosphere, and gases were introduced into the ETEM chamber. However introducing of gas having high humidity was not tried positively with more attention to mechanical adverse affect of ETEM. In the future, it is very important to study the material reaction when property improvement of fuel cell and to investigate the preparation of materials such as metal alloys. Therefore, in this study, the method of introducing a high-humidity gas into the ETEM chamber was investigated. Such a gas was introduced into the ETEM chamber, and whether the material, which easily aspirates humidity, changed its shape and structure was investigated. The method of introducing high humidity gas into ETEM was held as follow.

First, distillated water was heated in the reservoir, and a flexible tube connected to the gas injection TEM holder was inserted in the reservoir. The gas in the reservoir was introduced into the ETEM chamber through a flexible tube. The humidity of the gas in the reservoir was controlled by blowing air into the reservoir and or by heat. The reservoir remained open; therefore, the gas pressure was equal to the air pressure. The high-humidity gas in the reservoir was introduced into the ETEM chamber by aspiration. Calcium oxide powder was used as the observed sample. Because calcium oxide easily absorbs moisture, and calcium oxide powder transforms into calcium hydroxide during moisture absorption, the volume increases and the shape gradually becomes rounder. These phenomena indicate that a high-humidity gas is introduced. The sample was set in the gas injection holder [1], and the holder was inserted into the ETEM chamber. Then, the shape and electron diffraction from the sample were observed during the introduction of gas. For comparison, air was introduced into the ETEM chamber and the sample room pressure of the ETEM chamber was maintained at 1.0×10^{-3} Pa in all the experiments.

When a high-humidity gas was introduced, the sample surface gradually became rounder, and the volume of the sample became slightly large (Fig. 1). On the other hand, when air was introduced, the sample did not change its shape (Fig. 2). Additionally, the electron diffraction pattern from each sample shows that only the sample, which was bombard with a high-humidity gas, aspirated the moisture and transformed from calcium oxide to calcium hydroxide (Fig. 3). In the electron diffraction pattern obtained from the sample bombard with a high-humidity gas, diffraction spots of calcium oxide and calcium hydroxide were confirmed; this implies that the sample did not completely transform into calcium hydroxide, and it is possible

that only the surface of the sample transformed. The above results indicated that the introduction of a high-humidity gas into the ETEM was successful, and the aspiration process of calcium oxide powder was observed using ETEM.

References

[1] Kamino et. al, Japanese: Densikennbikyo, 35, 232 (2000)

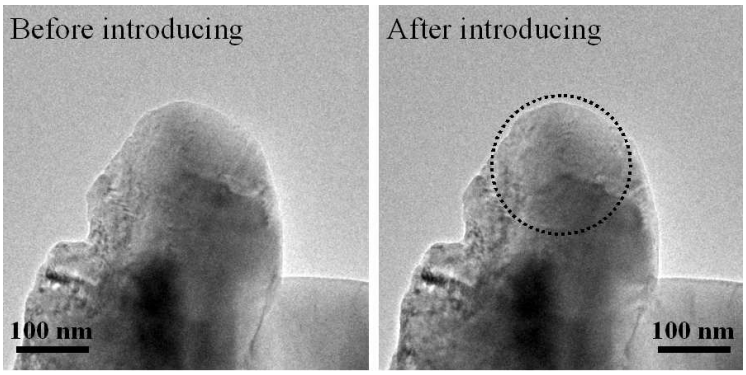

Fig. 1: TEM images of calcium oxide powder before and after Air introducing

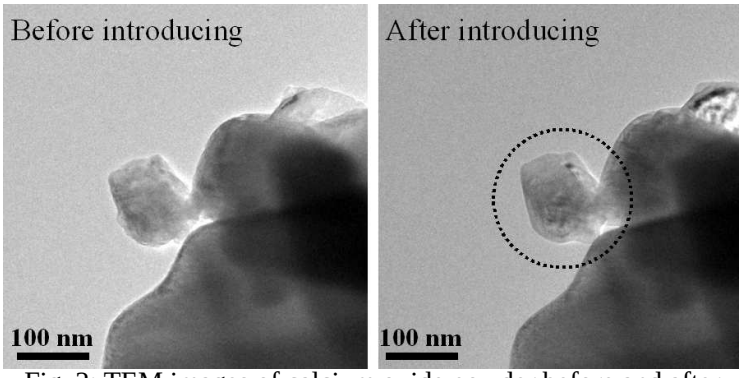

Fig. 2: TEM images of calcium oxide powder before and after high-humidity gas introducing

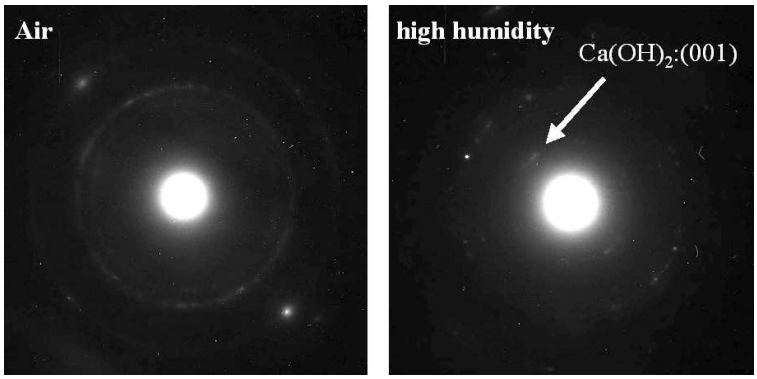

Fig. 1: Electron diffraction patters from samples after air and high-humidity gas introducing