Among the advanced energy sources, the polymer electrolyte fuel cell, PEFC, is one of the most attractive options for automobile application. Two major issues to facilitate widespread dissemination of fuel cell vehicles are the cost and durability of the electrocatalyst. Many efforts are made to develop the electrocatalyst to meet such demands. However, it is necessary to clarify the details of degradation mechanism. In our previous study, in situ TEM observation was carried out to investigate the degradation mechanism of the Pt/C electrocatalyst, focusing on the effect of humidity in the air. We found that the oxidation of carbon support was accelerated at the interface of Pt particle and carbon support in the high humidity condition. In this study, the effect of reactant gases such as hydrogen, nitrogen, oxygen, on the structural change of the Pt/C was investigated. The catalyst sample was Pt supported on high surface area carbon, Pt/C, purchased from TKK. The in situ observation of the catalyst was carried out with a Hitachi H-9500 environmental TEM equipped with an AMT TV camera system. A specimen-heating holder with a gas-injection nozzle was employed for both heating of the specimen and introduction of the reaction gas. The reaction temperature, time, and the total pressure near the specimen was controlled to 220°C, 30 min, and approximately 0.6 Pa, respectively. Although the typical operation temperature of the PEFC ranges from 80°C to 100°C, the reaction temperature of 220°C was chosen to accelerate the in situ reaction with the gases. In the in situ observation, density of electron beam on the specimen was controlled to minimize the radiation damage of the specimen. Figure 1 shows a TEM image (a) and the corresponding selected area diffraction (SAD) pattern (b) of the Pt/C electrocatalyst before reaction. Figure 2 shows a TEM image (a) and the corresponding SAD pattern (b) obtained from the Pt/C electrocatalyst after in situ reaction under oxygen atmosphere. During the in situ reaction, the morphology of the carbon support was gradually changed due to the oxidation (Fig. 2(a)). An analysis of the SAD pattern revealed that the Pt nano particles were agglomerated and crystallized (Fig. 2(b)). We also carried out in situ observation under hydrogen and nitrogen atmosphere. Various interesting phenomena such as a slight shrink of the carbon support and a little increase in the crystallinity of Pt nano particles were observed and characterized.

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Fig. 1: Pt/C electrocatalyst before reaction. (a) TEM image and (b) selected area diffraction pattern.

Fig. 2: Pt/C electrocatalyst after reaction with O$_2$ (0.6 Pa) at 220°C for 30 min. (a) TEM image and (b) selected area diffraction pattern.