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In situ Liquid TEM Study for Degradation Mechanisms of Fuel Cell Catalysts during Potential Cycling Test

Shinya Nagashima[1,5], Kenta Yoshida[2,3], Tomoki Hiroyama[2], Kun Liu[2], Yipu Kang[2], Toshihiro Ikai[4], Hisao Kato[4], Tetsuo Nagami[4] and Keisuke Kishita[5]

[1.] Materials Research and Development Laboratory, Japan Fine Ceramics Center, Atsuta-ku, Nagoya, 456-8587, Japan
[2.] Nanostructures Research Laboratory, Japan Fine Ceramics Center, Atsuta-ku, Nagoya, 456-8587, Japan
[3.] Institute for Advanced Research, Nagoya University, Chikusa-ku, Nagoya, 464-8603, Japan
[4.] Catalyst Design Department, Material Engineering Division, Toyota Motor Corporation, Toyota-cho, Toyota, Aichi, 471-8572, Japan
[5.] Material Analysis Department, Material Development Division, Toyota Motor Corporation, Toyota-cho, Toyota, Aichi, 471-8572, Japan

The polymer electrolyte fuel cell (PEFC) is a promising energy source for fuel cell vehicles. The typical electrocatalyst used in PEFCs consists of platinum nanoparticles on carbon black (Pt/C). The development of advanced electrocatalysts for PEFCs requires reduction of Pt usage and enhancement of durability [1-3]. To achieve further design improvements for Pt/C electrocatalysts, it is essential to understand the degradation mechanisms in real space for Pt/C electrocatalysts.

We applied *in situ* liquid TEM observation technique using a liquid flow cell TEM holder with electrical biasing capabilities (Poseidon, Protochips Inc.) into the differential pumping environmental TEM (Titan ETEM, FEI Company) for direct observation of structural changes of Pt/C electrocatalysts. The electrochemical cell simulating environment of activated PEMFC was comprised of deposited Pt/C electrocatalyst onto an electrode on a MEMS chip and flowing electrolytic solution of 0.1 M and 0.2 M $HClO_4$. We obtained cyclic voltammetry (CV) curves by electrochemical measurement and dynamic TEM movies simultaneously.

Figure 1-3 are selected area captured (SAC) images of movies obtained from Pt/C electrocatalysts in $HClO_4$ solution during potential cycling tests. We succeeded in direct observation of degradation behaviour of Pt/C electrocatalysts such as dissolution of Pt nanoparticles (Figure 1), detachment of a Pt nanoparticle from carbon support (Figure 2) and aggregation via Pt nanoparticles migration (Figure 3) which had been suggested [1-3].

In conclusion, *in situ* liquid TEM observation during potential cycling tests is a powerful tool for understanding of electrochemical behaviour of electrocatalysts of PEMFCs in nanometer scale.

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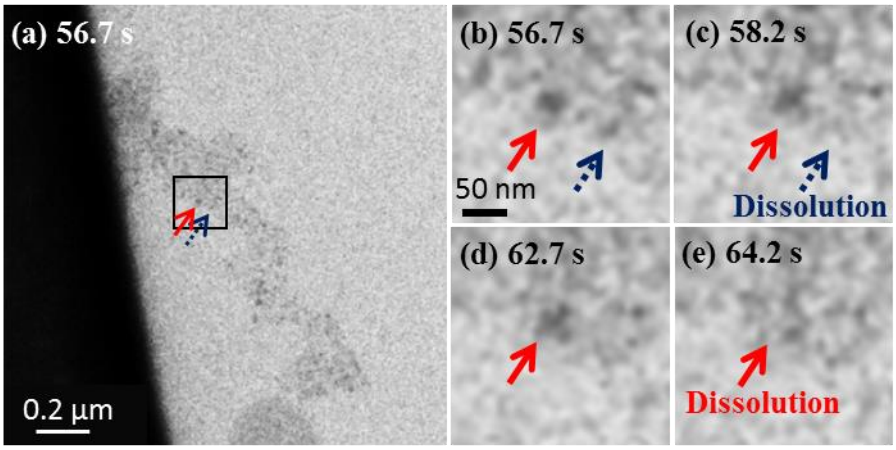

Figure 1. SAC images obtained from the Pt/C electrocatalysts over the potential range from -0.75 V to 0.30 V (vs. Pt) at a scan rate of 50mV/s in 0.1 M $HClO_4$ at room temperature.

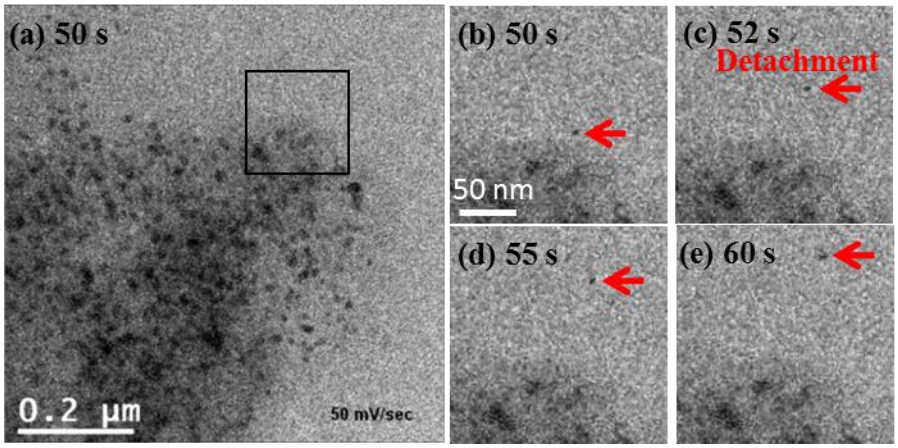

Figure 2. SAC images obtained from the Pt/C electrocatalysts over the potential range from -0.70 V to 0.45 V (vs. Pt) at a scan rate of 50mV/s in 0.2 M $HClO_4$ at room temperature.

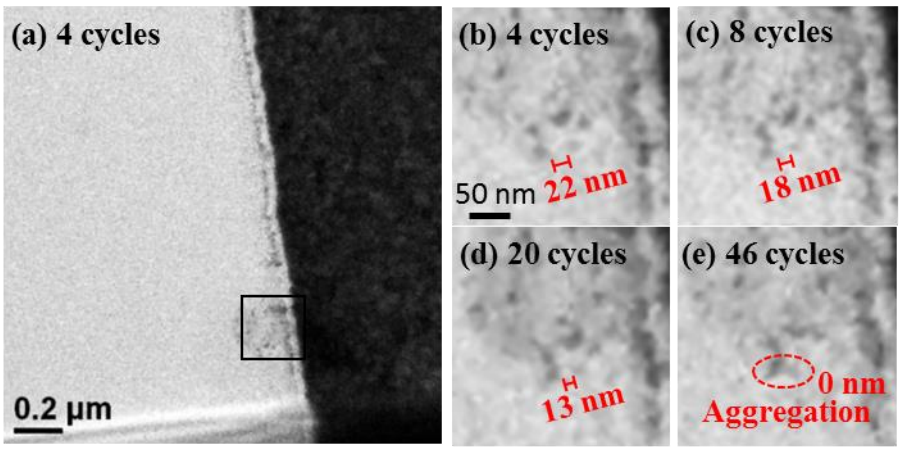

Figure 3. SAC images obtained from the Pt/C electrocatalysts over the potential range from -0.70 V to 0.45 V (vs. Pt) at a scan rate of 500mV/s in 0.2 M $HClO_4$ at room temperature.

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