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Preparation of a Model Platinum/Gamma-Alumina Catalyst for *in situ* Environmental TEM Experiments

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Platinum/gamma-alumina (Pt/γ-Al_2O_3) is an important catalyst system used in a variety of industrial applications, such as fuel cells, petroleum reformers, and catalytic converters [1]. Correlating theoretical and experimental studies of Pt/γ-Al_2O_3 has been hindered by the non-uniform crystallinity of commercially available γ-Al_2O_3 [2]. Synthesizing a well-defined, model system of Pt/γ-Al_2O_3 is crucial for bridging the gap between experiment and theory by enabling direct comparison of experimental results and theoretical simulations [3]. In this work, we present our synthesis of a model catalyst consisting of a single-crystal, defect free γ-Al_2O_3 thin film decorated with well-defined Pt nanoparticles evenly dispersed on the γ-Al_2O_3. Cross-sectional TEM samples made from the model system will be employed for *in situ* environmental transmission electron microscopy (ETEM) experiments, since observing Pt/γ-Al_2O_3 during reaction under relevant gas and thermal conditions is crucial to understanding the important structural features impacting the catalytic activity of the Pt nanoparticles.

Defect-free, single-crystal γ-Al_2O_3 (111) was synthesized through controlled oxidation of single-crystal NiAl (110) [2], the orientation of which was verified by X-ray diffraction (XRD). The NiAl surface was then polished through mechanical grinding techniques to promote homogenous growth of defect-free γ-Al_2O_3 (111) with root mean squared (RMS) surface roughness of 5.21 nm, as measured using atomic force microscopy (AFM). The NiAl was then oxidized in a tube furnace at 750°C for two hours in dry air. The resulting single-crystal γ-Al_2O_3 was determined to be of the (111) orientation via XRD, and the surface roughness was measured by AFM to be less than 6 nm RMS (Figure 1a).

A dual beam, focused ion beam-scanning electron microscope (FIB-SEM) was used to prepare a cross-section from the sample, exposing the NiAl/γ-Al_2O_3 interface (Figure 1a). From the SEM image of the cross-section, the γ-Al_2O_3 layer was determined to be about 60nm thick. 1-1.5 nm diameter Pt nanoparticles were then deposited onto the γ-Al_2O_3 surface by electron beam evaporation. A cross-sectional TEM sample was made from the Pt/γ-Al_2O_3/NiAl (Figure 2). Deposition of monodisperse, shape-controlled, 1 nm Pt nanoparticles prepared by the inverse micelle technique [4] onto the model γ-Al_2O_3 surface is underway.

In addition to the model catalyst, Pt/γ-Al_2O_3 sample were prepared using commercially available polycrystalline γ-Al_2O_3 for comparison. Polycrystalline γ-Al_2O_3 powder was solution drop-cast onto a copper TEM grid, upon which 1-1.5 nm Pt nanoparticles were subsequently deposited via electron beam evaporation. *In situ* environmental TEM experiments are being performed on both the model and non-uniform Pt/γ-Al_2O_3 catalysts to understand the role of the γ-Al_2O_3 support on Pt nanoparticle behavior under relevant reaction gas and heating conditions. [5]

References:

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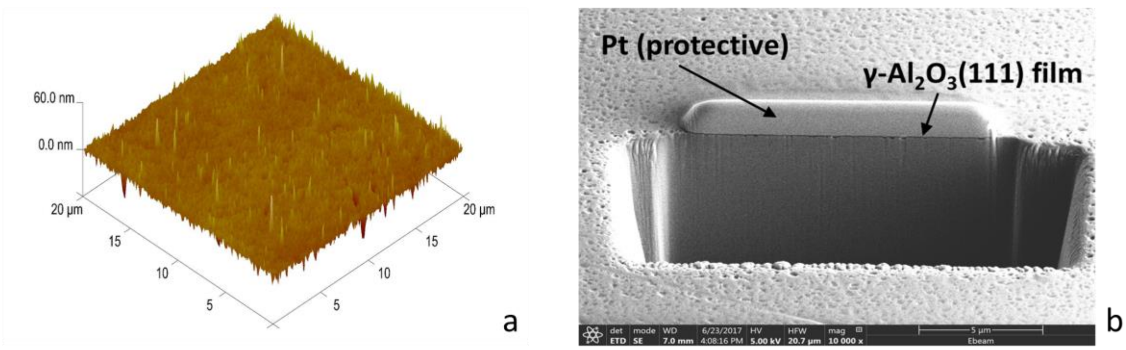

Figure 1: (a) AFM surface roughness measurement of one of the γ-Al_2O_3 films. (a) Cross-sectional SEM image of the NiAl/γ-Al_2O_3 interface, lifted from the sample in (b).

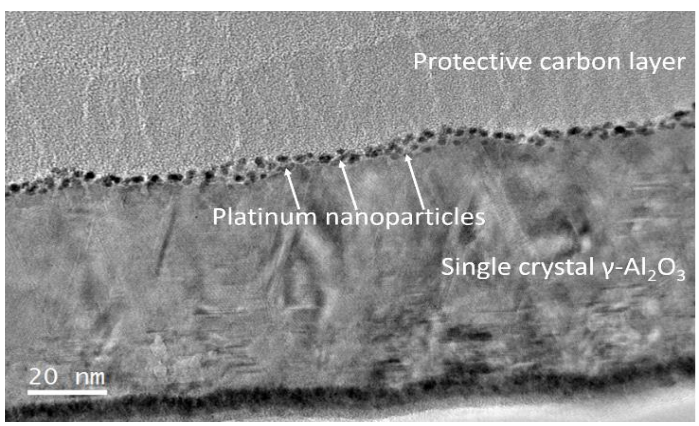

Figure 2: TEM micrograph the Pt/γ-Al_2O_3 interface the resulting lift-out, showing the dispersion of nanoparticles across the surface.