



Experiment Brief

K3 IS Camera

Title

In-situ observation of the annealing of Pt islands into branched Ru nanostructures to make single-atom catalysts.

Gatan Instrument Used

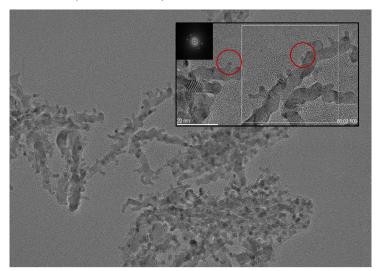
The K3® IS camera delivers simultaneous **low-dose imaging via real-time electron counting**, fast continuous data capture, and **a large field of view**.

Background

Platinum (Pt) is one of the most active metals for the methanol oxidation reaction (MOR), but it suffers from catalyst poisoning due to strongly adsorbed CO as an intermediate. Designing platinum-ruthenium (PtRu) electrocatalysts with structural durability and high activity will significantly impact the performance of direct methanol fuel cells (DMFC), aas Ru atoms decrease CO poisoning of the active Pt. To combine the exceptional activity of single-atom Pt catalysts with the benefits of an active metal support, we must overcome the synthetic challenge of forming single Pt atoms on noble metal surfaces. Here, we have developed a process that grows and spreads Pt islands on faceted Ru branched nanoparticles to make a single Pt atom on Ru catalysts.

Materials and Methods

The transformation of RuPt nanoparticles was observed using a Gatan K3 IS camera in a Titan ETEM with image correction operating at 300 keV [A.R. Poerwoprajitno, et al., Nat Catal. 5 (2022) 231–237. https://doi.org/10.1038/s41929-022-00756-9.]. The partial pressure of 10 mbar H_2 was introduced to the ETEM, and the sample was heated to 700 °C using a Protochips Aduro heating holder. The video was taken at 10 fps, and the sample was delivered a low electron dose of just 106 e'/Ų/s. Drift correction was performed exclusively in



DigitalMicrograph® via cross-correlation using the IS player. A bandpass pre-filter was simultaneously applied to improve the cross-correlation. The experiment was carried out at relatively low magnification of 69kx, enabling a wide field of view to be captured with the K3 IS without losing lattice resolution. The low magnification allows capturing a statistically significant number of Pt island annealing events in a single region of interest. Frames displayed here and in the linked video are each a sum of 25 original frames.

Summary

These *in-situ* observations will inform optimal conditions for the batch syntheses of highly active and durable RuPt nanostructures for MOR catalysis. Uniquely, the large field of view afforded by the K3 IS camera allows for analyzing a statistically significant amount of Pt island annealing events, which would not have been possible with a conventional camera.

Figure 1. An image frame from the high-speed *in-situ* observation of the annealing of Pt islands into branched Ru nanostructures at 700 °C, and under 10 mbar H_2 in the Titan ETEM, showing the entire field of view captured in the video. The inset contains a 'snapshot' of a smaller area, showing the annealing of Pt islands into the larger Ru surface (red circles), and the FFT of the area within the white square. The emergence and disappearance of Moiré fringes in the video indicates some reorientation of the crystalline Pt islands just prior to full incorporation to the Ru. A full *in-situ* video of the inset region can be found <u>here</u>.

Credit(s)

A special thanks to the Center for Integrated Nanotechnologies (CINT) and the University of New South Wales, including John Watt, Agus Poerwoprajitno, Dale L. Huber, and Richard D. Tilley.

Gatan, Inc. is the world's leading manufacturer of instrumentation and software used to enhance and extend electron microscopes—from specimen preparation and manipulation to imaging and analysis.

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