

In situ surface analysis of palladium-platinum alloys in methane oxidation  
conditions

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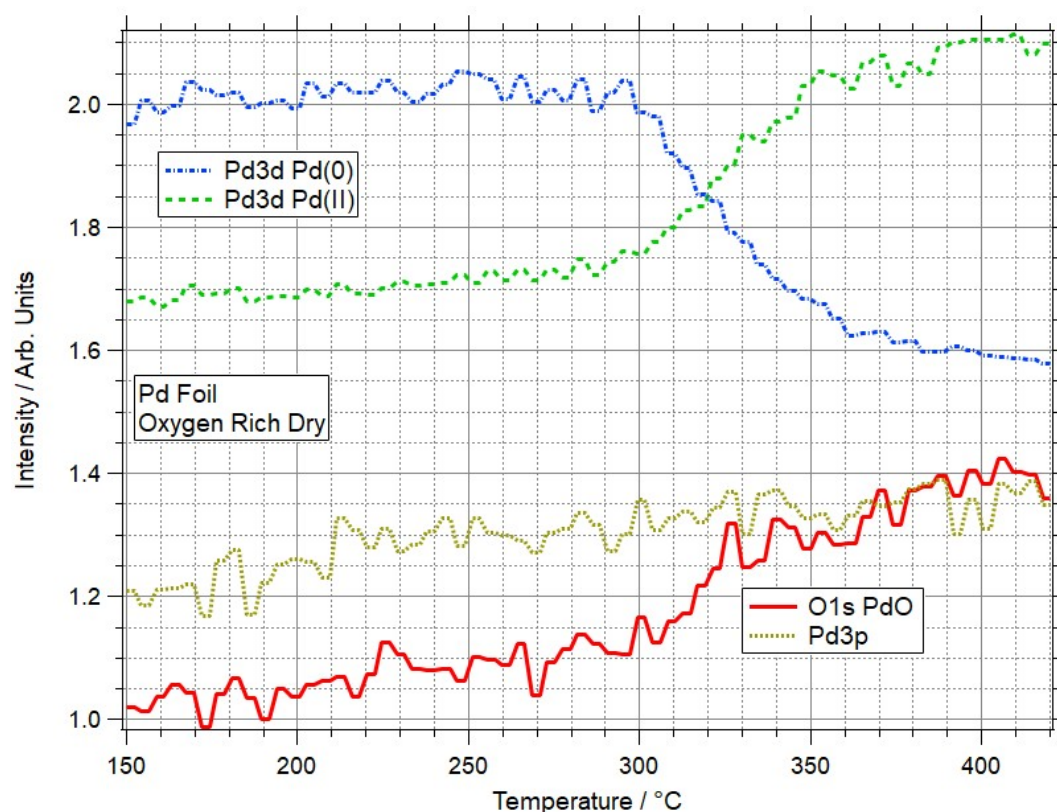
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Supporting Information

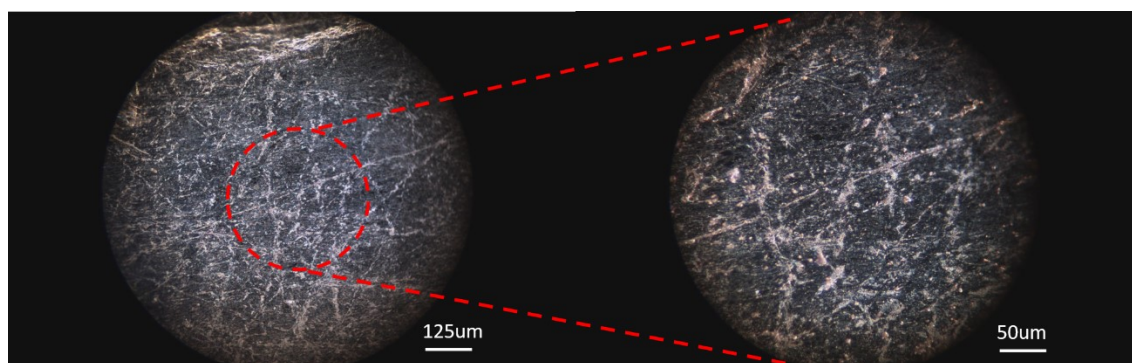
1. Peak intensities in O1s-Pd3p region during oxygen rich dry TP-XPS experiment



*Fig S1: Intensity plots for various peak positions in the Pd 3d and O 1s XPS regions, as determined during TP-XPS measurements on Pd foil in oxygen rich dry conditions. O 1s and Pd 3p intensities divided by oxygen gas phase to normalise. Pd 3d data normalised back on the background before the peak, then rescaled in intensity for clarity of presentation. Intensity summed over 0.5 eV windows around approximate peak positions.*

The formation of PdO is also visible in the O 1s and Pd 3p region. An example of this, taken in oxygen rich dry conditions, is shown in Fig. S1. In this plot, peak intensities at various positions are presented, normalised with respect to the low-binding-energy background (in the case of Pd 3d) or the oxygen gas phase peak (O 1s, Pd 3p), as these should be constant throughout, and can be used to remove any artefacts from variations due to sample movement and/or manipulator retraction. The increase of Pd(II) seen in the Pd 3d region is consistent with the increase in PdO in the O 1s region. The increase in intensity at the Pd 3p peak position can be attributed to the tail of the PdO O 1s peak, due to the small separation between these peaks.

## 2. Optical Microscope images of Pd foil (post NAP-XPS experiments)



*Fig S2: Optical microscope images of a Pd foil, taken after NAP-XPS experiments were performed in various methane oxidation reaction conditions. Images taken with 20x (left) and 50x (right) magnification. Scale bars shown for each image. Red dashed circle shows approximate area on left image in which higher magnification right image was taken.*

The images of a Pd foil, in Fig S2, show the rough surface of the sample. Within the beam spot of the B07-C beamline at Diamond Light Source (around 100x100  $\mu\text{m}$ ), domains with various surface orientations would be collectively measured.