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Systematic STEM characterization of supported size-selected Pt clusters

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Motivation

Clusters have evolved towards versatile and important tools in catalysis research [1]. In contrast to other catalyst preparation methods the use of cluster deposition enables the possibility of tuning particle size and coverage independently [2] with the control of the size down to a single mass.

Since cluster based preparation is limited to vacuum conditions, there is a lack of detailed studies of structure, stability and reactivity under ambient conditions [3]. This limitation has recently been overcome [4] and offers new insights under more realistic conditions.

In particular, TEM has proven to be a useful experimental technique for elucidating cluster properties on a local level [5]. Here, we present a systematic study by means of STEM.

Experimental

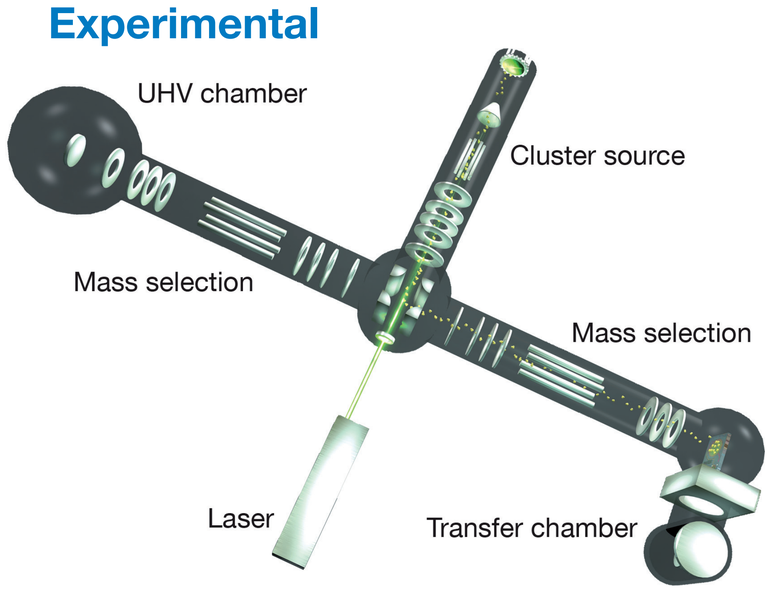

Figure 1 - Sketch of the experimental setup for deposition of size-selected cluster catalyst materials under UHV [6].

After synthesis by laser evaporation, the charged clusters are guided via a bender either towards a transfer chamber (right). Subsequently the clusters are mass selected by means of a QMS and then deposited onto different TEM grids supports and can be transferred to ambient conditions.

Mass selection

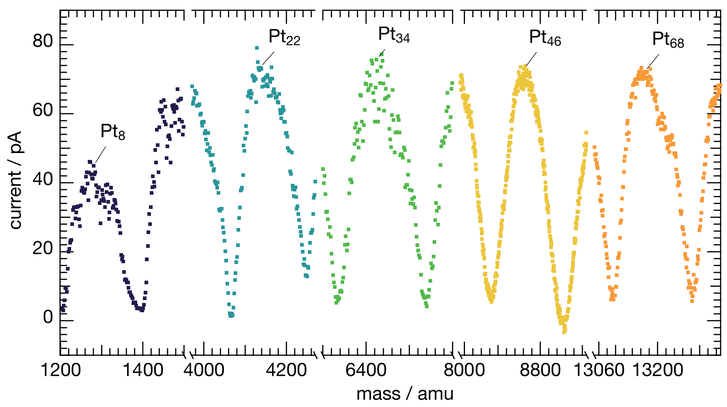

Figure 2 - Different ranges of mass scans, showing the size distributions of Pt^+ clusters selected by the QMS.

Softlanding on support

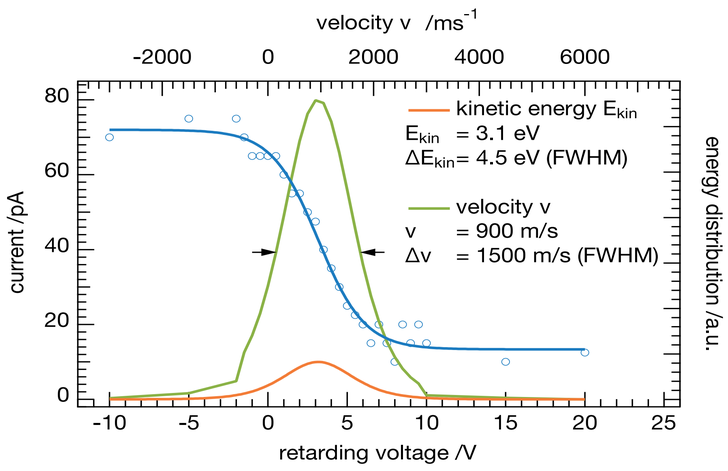

Figure 3 - Kinetic energy distribution of a beam of Pt_8^+ ions.

Scanning TEM (HAADF-STEM)

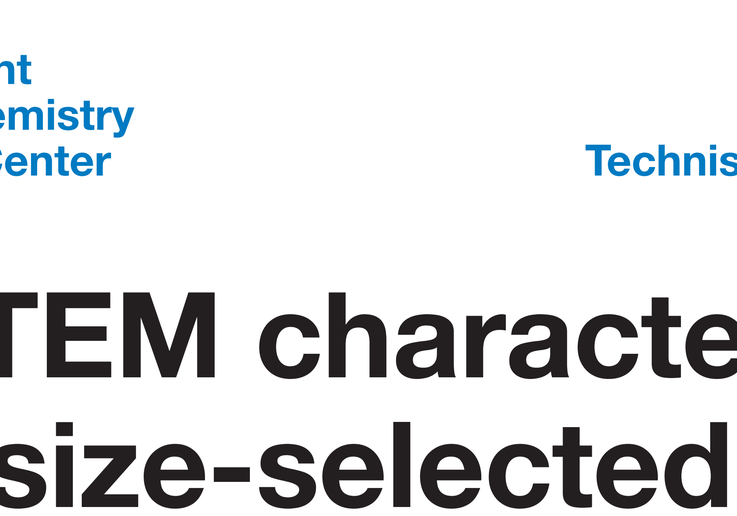

Figure 4 - STEM micrographs (30x30 nm^2) for Pt_n (n=8, 22, 34, 46, 68) clusters deposited under softlanding conditions onto carbon support (FEI Titan 80-300, 300 kV, 196 mm).

Control of coverage

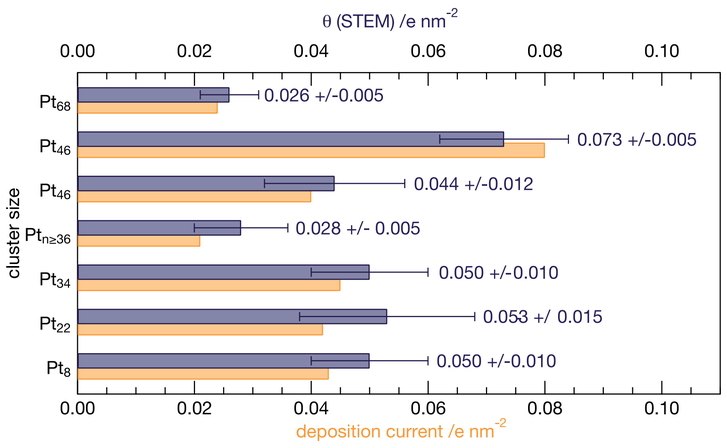

Figure 5 - Statistical analysis of the coverage control of a minimum of five micrographs per size.

Control of size - atomic precision

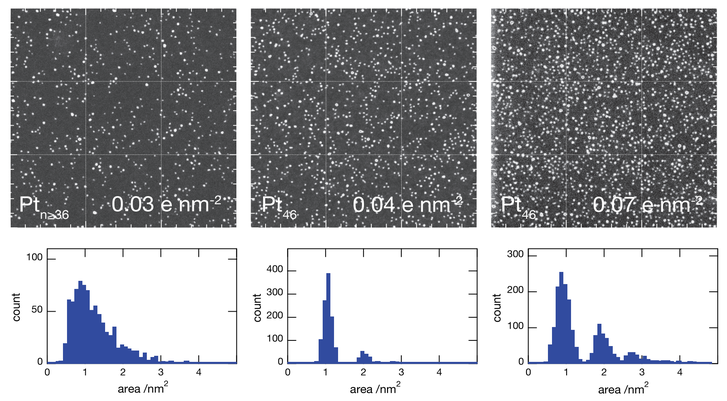

Figure 6 - STEM micrographs for different coverages, as well as selected and unselected clusters with area statistics.

Cluster heigth and volume

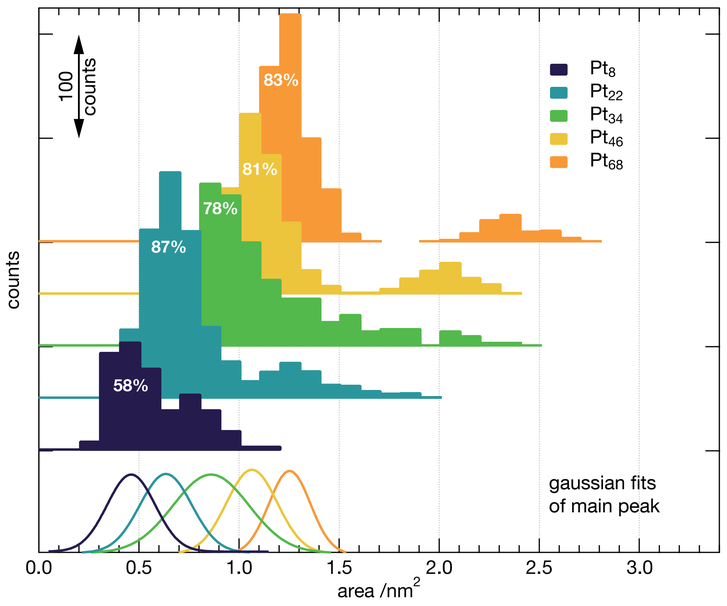

Figure 7 - Statistical analysis of the measured area of different sizes along with gaussian fits.

Cluster shape

Figure 8 - STEM micrographs (30x30 nm^2) and statistical analysis of a mixed sample with Pt_{22} and Pt_{68}.

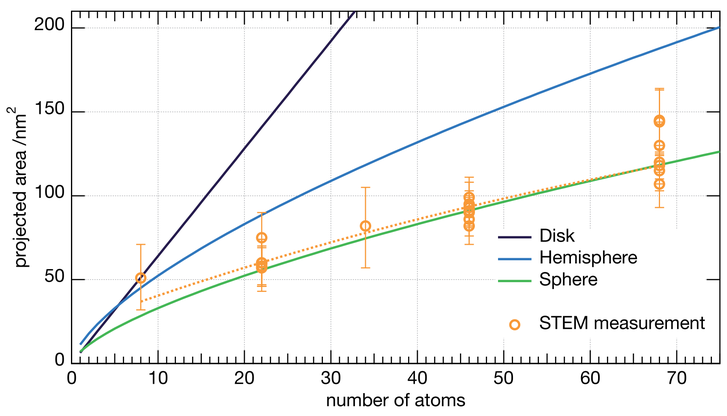

Figure 9 - Mean areas of different cluster sizes measured by STEM compared to a disk, half-sphere and sphere model.

Summary and outlook

- Coverage corresponds within 14 % (average error) to integrated current over time during deposition.
- Areas show cluster ensembles with distinct size, reflecting production of the source, ion optics and QMS.
- Height and volume of clusters' area accessible by measuring two sizes on the same sample.
- Comparison of the measured area with basic geometric models imply a sphere like cluster shape.
- These results provide a foundation for future ambient condition experiments of size selected clusters.

References

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