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In situ Probing of Nanostructure Surfaces

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Nanostructures represent an interesting class of materials. Especially their surfaces which to a large extent govern the functionality of these materials are of fundamental interest. For example, the activity of heterogeneous catalysts strongly rely on the structure of these surfaces. The nature of these surfaces is a strong function of their size and the conditions to which the nanoparticle is exposed, i.e. surrounding gases and the temperature. Detailed quantitative information of the surfaces under operating conditions is inherently difficult to access due to limitations of characterization equipment.

The last couple of decades have seen significant advances in electron microscopy. These include aberration correction, high speed CMOS and direct detection cameras on the microscope side as well as MEMS in situ capabilities such is heaters, reaction cells and electrical biasing on the sample holder side. Put together, these advances allow us to probe the intrinsic properties of nanoscale materials with unprecedented temporal and spatial resolution (*1*).

These new capabilities mean that datasets become increasingly large and complex. In order to extract every ounce of information, automated analysis approaches are needed. In recent years, deep learning approaches have gained significant traction. This approach allows unattended exhaustive analysis of large datasets without human bias. Current research is trying to reveal whether this technique can extract more information from an image than a trained operator. Especially at low signal-to-noise ratios.

In recent studies, we have investigated fundamental properties of supported gold nanoparticles, especially in relation to applications in catalysis. Using environmental high-resolution transmission electron microscopy, gold nanoparticles of various sizes supported on cerium dioxide or soft-landed on a silicon nitride membrane have been imaged under varying conditions. The metal-gas and metal-support interface as well as the melting properties was investigated (*2*). The interfaces are important for the performance and stability of the sample and the melting properties are important in relation to the atomic configuration at the surface. The data is analyzed using automated real-space peak finding algorithms and convolutional neural networks developed in-house. The results reveal how the interfaces behave as a function of the surround

Figure 1 shows the observed melting point of gold nanoparticles as a function of particle size along with molecular dynamics simulations using two different interatomic potentials. As expected, the meting decreases with particle size. Figure 2 shows frames from an image sequence acquired at 612°C. In frame a), the crystalline lattice is clearly resolved with the five-fold symmetry, which is typically observed for gold nanoparticles. The crystalline particle is surrounded by a halo, which we interpret as mobile atoms (i.e. molten). Frame b) still shows fringes but not as clear. In frame c) which is acquired 0.2 seconds after frame b), no fringes are observed and the particle is molten.

These examples along with others show how these experimental techniques and advances is used to advance the performance of materials in chemical and energy production.

References:

[1] TW Hansen, JB Wagner, Eds., "Controlled Atmosphere Transmission Electron Microscopy", (Springer) p. 329.
[2] P Schlexer et al., Part. Part. Syst. Charact. (2019).

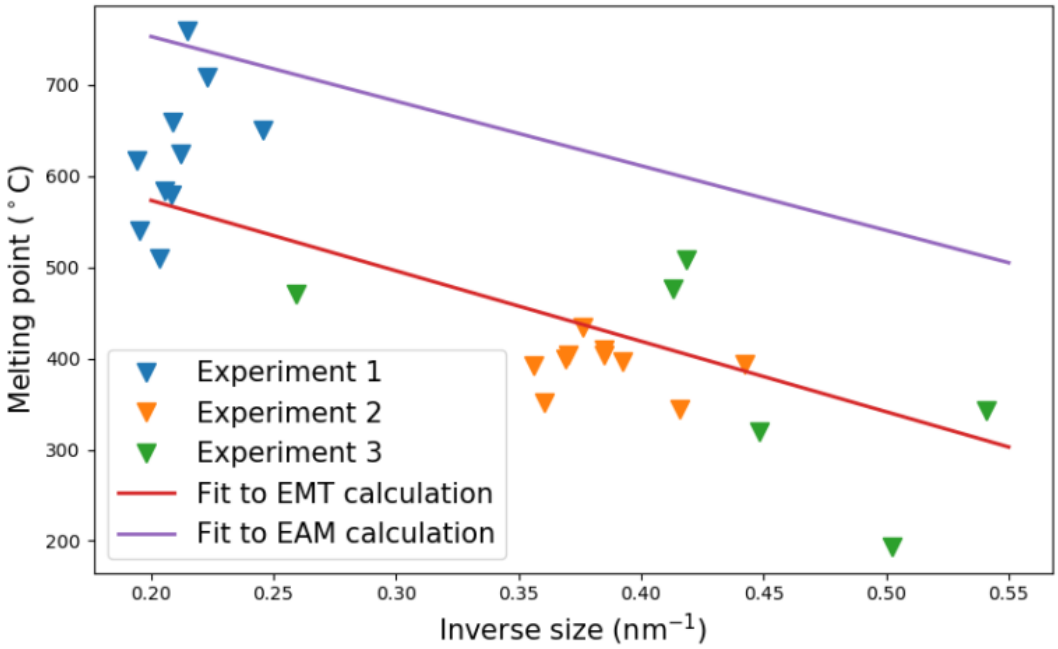

Figure 1. Observed and calculated melting point as a function of inverse particle diameter.

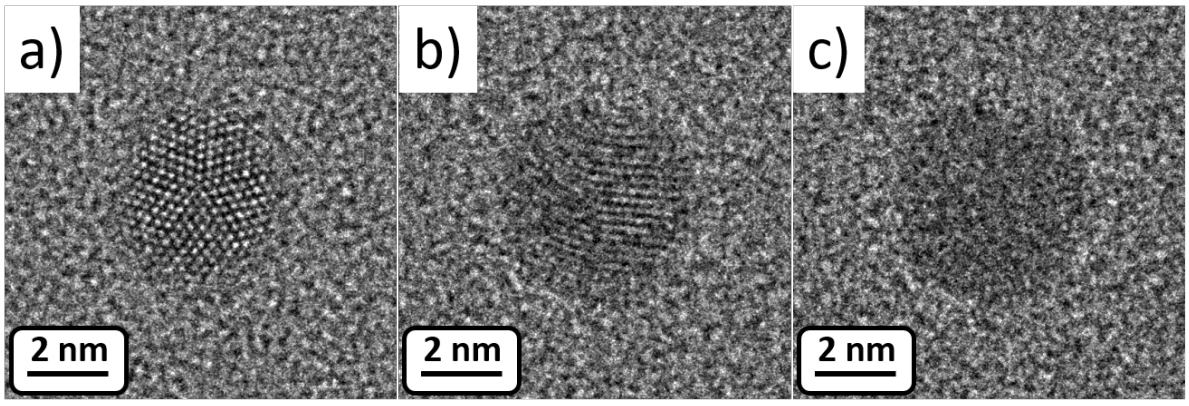

Figure 2. Snapshots from an image sequence acquired at 612°C showing the melting process of a 5 nm gold nanoparticle.

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