Illuminating Electron Microscopy of Photocatalysts

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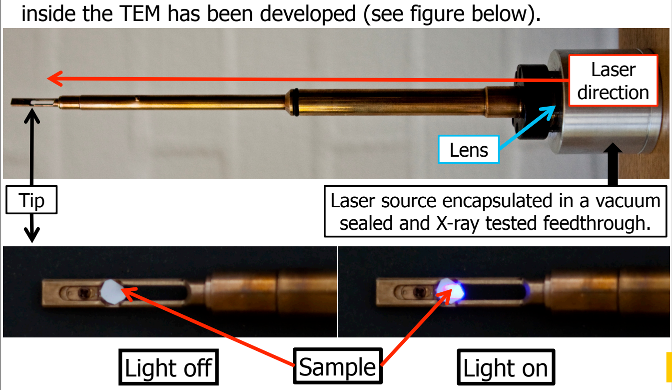
Background

Photocatalysts are of fundamental interest for sustainable energy research. By means of transmission electron microscopy (TEM) it is possible to obtain insight into their structure, composition and operation. In particular, the internal environment of a TEM can be modified in order to perform real time *in situ* experiments in the presence of light and gas or under other unconventional TEM conditions.

This project is part of the CAtalysis for Sustainable Energy (CASE) initiative and involves characterization of catalysts using methods available at DTU Center for electron nanoscopy (Cen).

Experimental setup

A new specimen holder capable of shining light onto samples inside the TEM has been developed (see figure below).



Working principle

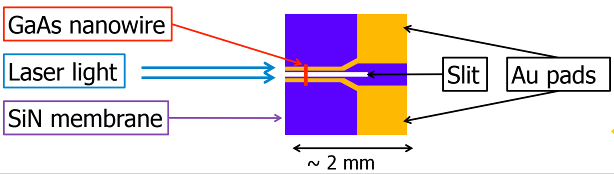
The holder is implemented with a laser diode and a lens system to guide and focus light onto the sample surface with maximum power transmission (no fiber optics). The source can be changed and tuned, in principle spanning the visible and UV spectrum.

It is possible to use the holder in DTU Cen's Environmental TEM (ETEM) to expose a specimen to a controlled gas atmosphere during illumination with light.

FEI Titan Environmental TEM

Future projects

- Characterization of a GaZnNO-based photocatalyst for water splitting application.
- Electron holographic characterization of GaAs junctions and nanowires under both applied bias and illumination with light.
- Implementation of electrical contacts in the present light holder.

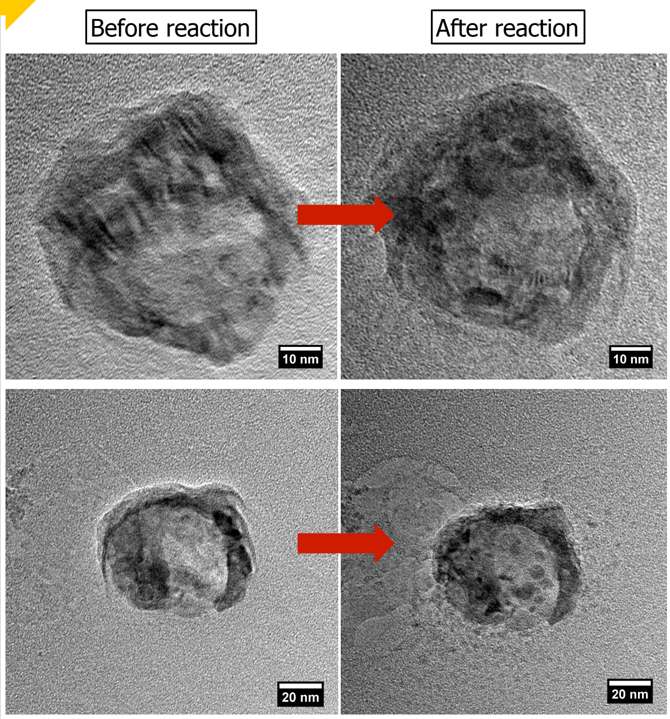


Specimen details

- Cu_2O is a photocatalyst for water splitting under visible light illumination [1].
- It undergoes photodegradation in an aqueous environment [2].
- Photodegradation has been investigated *in situ* in the ETEM.

First experimental results

- Cu_2O nanocubes are stable in the electron beam at 300 kV in high vacuum. In contrast, in H_2O vapor the particles degrade in a few seconds in the electron beam.
- The electron beam was therefore blanked before exposing the sample to H_2O and light and unblanked for investigation after high vacuum was restored.
- No significant heating by the laser is predicted for ~70 nm nanoparticles in a gas atmosphere due to the low laser power (~3.5 mW @ 405 nm) and because of efficient heat exchange between the nanoparticles and the surrounding gas.
- The photon energy density (~3.5 nJ/(μm^2 s)) is approximately six orders of magnitude weaker than that of the primary electron beam. It is therefore necessary to blank the electron beam in order to distinguish the effect of light and electrons.
- Time resolution is achievable by performing successive steps similar to that described before.



Degradation of Cu_2O nanocubes in H_2O (5 mbar) under visible light illumination (405 nm) for 3 hours.

References

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