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Development of a versatile TEM specimen holder for the characterization of photocatalytic materials

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Photocatalysts are of fundamental interest for sustainable energy research [1]. By means of transmission electron microscopy (TEM), it is possible to obtain insight into their structure, composition and reactivity. Such insight can then be used for their further optimization [2]. Here, we combine conventional TEM analysis on photocatalysts with several *in situ* TEM techniques including environmental TEM (ETEM), *in situ* photo activation and localized surface plasmon resonance (LSPR) spectroscopy [3-4].

These experiments are facilitated by the construction of a specimen holder capable of illuminating samples inside the TEM with a laser diode and an optical system to guide light onto the sample with maximum power transmission. The source can be changed and tuned between the visible and the UV range. The specimen holder is equipped with five electrical contacts that can be used to perform *in situ* electrical, pressure and temperature measurements as well as to power custom MEMS-based *in situ* heaters. It can be used inside an ETEM allowing specimens to be analysed in a controlled gas atmosphere during biasing and illuminating experiments.

A second, more advanced version of the holder has also been developed allowing simultaneous illumination of a specimen by several light sources using a combination of up to three monochromatic wavelengths and/or broadband light. Light sources and combinations can be changed during operation without interfering with the electron microscope operation. An additional optical probe provides the ability to collect light emitted from the sample and to perform spectroscopic analysis in real time, in parallel with all other *in situ* techniques and analytical capabilities offered by the specimen holder and the microscope.

The holders are presently being used to study a variety of photoreactive materials and structures, including photocatalysts (Fig. 1), photonic devices and solar cells with a particular focus on LSPR spectroscopy performed by indirect nanoplasmonic sensing [3-4], a novel experimental platform for measurements of thermodynamics and kinetics in/on nanomaterials and thin films. We present results from combined ETEM-LSPR studies of thermally-induced catalytic phenomena and on metal nanoparticle sintering, an important process in catalysis, which we study *in situ* in both real and model systems by means of indirect nanoplasmonic sensing and ETEM.

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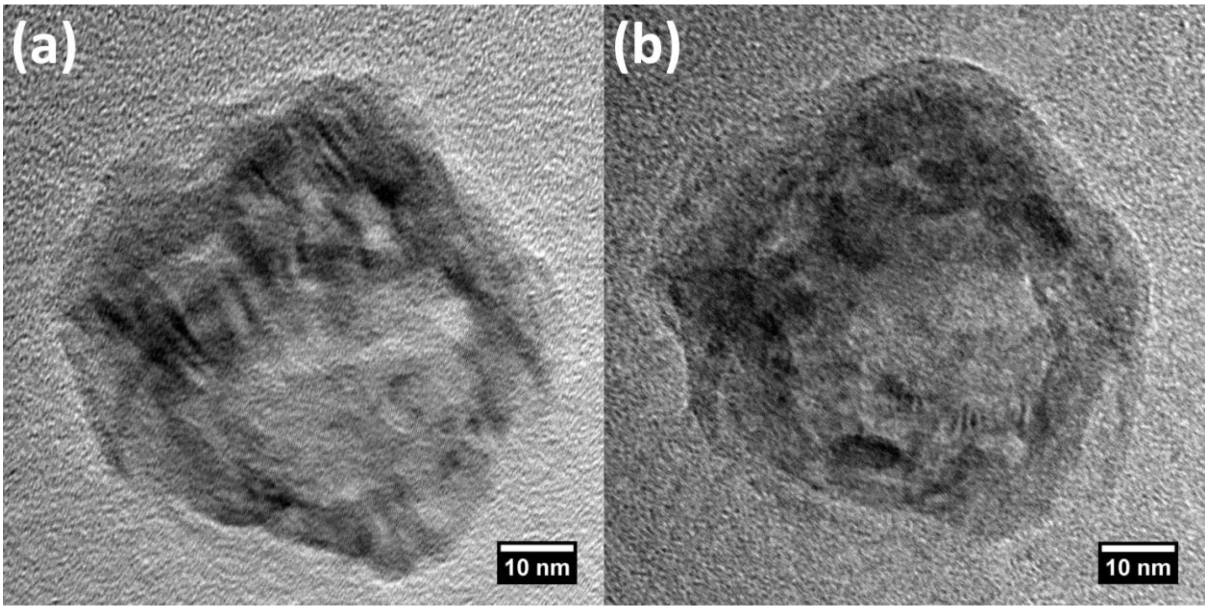

Figure 1. TEM micrographs of Cu_2O hollow nanocubes before (a) and after (b) photo-induced *in situ* reduction in H_2O vapor atmosphere (5 mbar) and visible light illumination (405 nm) for 3 hours.