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Visible and UV Irradiation of ETEM Samples for In-Situ Studies of Photocatalysts

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Today, energy production and storage is a primary concern, for everyone from policy-makers to business professionals to fundamental researchers. Photocatalysis is one field of energy research where storage and production are integrated, an ideal situation. However, much fundamental research is still needed to quantitatively connect the structure and processing of catalysts with their properties and performance, both of which must be enhanced. Often the structural features involved in catalytic processes are at the nano-scale or below. Clearly, characterization techniques such as TEM are thus a crucial part of any such fundamental research. This research focuses on enhancing current environmental TEM studies of photocatalysts by providing high intensity visible and UV illumination of the sample within an environmental transmission electron microscope (ETEM). This will allow detailed analysis of the interaction between light and photocatalysts such as TiO_2 under reaction conditions.

This design uses an optical fiber to guide light produced by a high-brightness broadband source to a point just below the ETEM sample. The light is generated by an LDLS EQ-99, a laser driven xenon plasma source, developed by Energetiq. The ETEM for which the optical fiber system has been developed is an FEI Tecnai F20. As shown in Figure 1, the fiber enters the ETEM in a direction perpendicular to the sample rod, and has no physical contact with the sample holder. This configuration allows for movement, tilting, heating and cooling of the sample with little impact on the optical fiber. Alignment of the fiber with the sample and electron beam is crucial, so an XYZ manipulator is included in the design, also providing increased flexibility. The completed system should be capable of illuminating the sample at the beam location with a spectral irradiance of at least $2mW/cm^2/nm$ from 200 to 800nm, as shown in Figure 3, which is about 10 times that of the sun on the Earth.

Figure 2 shows the results of a theoretical calculation of the irradiance on the sample as a function of position. The hot stage used for the environmental studies occludes much of the TEM sample, so only the relevant section of the sample is shown. The calculation, performed using MATLAB, treats the fiber end as a large number of point sources, each emitting uniformly into a cone characteristic of the fiber end geometry. This allows precise determination of the ideal geometry for the fiber within the microscope. Knowledge and control of the spectral profile of the light is also imperative. Ideally, light of constant high intensity at any wavelength could be directed onto the sample. A reasonable approximation to this ideal can be achieved through the use of a high brightness, broadband light source coupled with filters, as shown in Figure 3. This spectrum is calculated from the spectra provided by the vendors of the light source and various optical components included in the design.

Once fabrication and integration of all components is complete, the theoretical spectra and irradiance profiles given here will be compared with their experimental counterparts, and the completed system's performance will be properly evaluated. Preliminary data will be presented showing the effect of light induced phase transformations on titania photocatalyst in the presence of hydrogen gas at variable temperatures.

References

[1] The support from US Department of Energy (DE-SC0004954) and the use of ETEM at John M. Cowley Center for High Resolution Microscopy at Arizona State University is gratefully acknowledged.

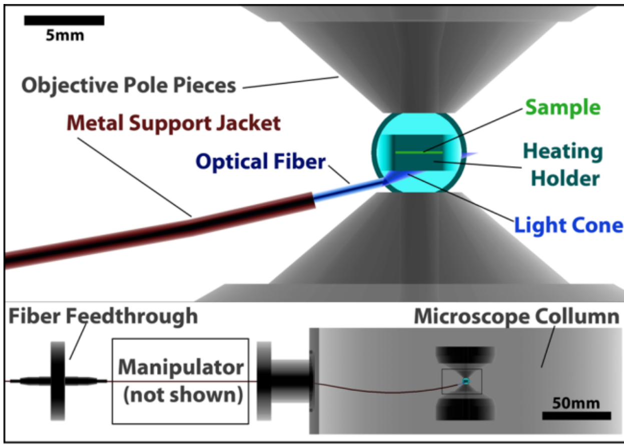

Figure 1: Microscope Modification. This figure shows, in 2 to-scale drawings, the basic design which allows an optical fiber to be fed into an FEI Tecnai Environmental TEM. The fiber enters the microscope perpendicular to the sample rod, and has no physical contact with the holder. Space between the pole-pieces is limited, but an angle-cut fiber directs the light onto the sample.

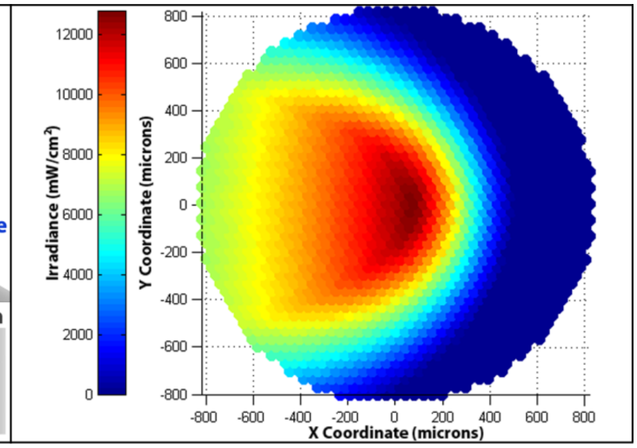

Figure 2: Sample Irradiance. This figure shows the calculated irradiance on the central 1.6mm diameter of the TEM sample. (The hot stage used in the microscope occludes the area of the sample outside this diameter.) The intensity is highest at the center, where the electron beam passes. Here it is 10 times that of the sun on the Earth's surface (see Figure 3)

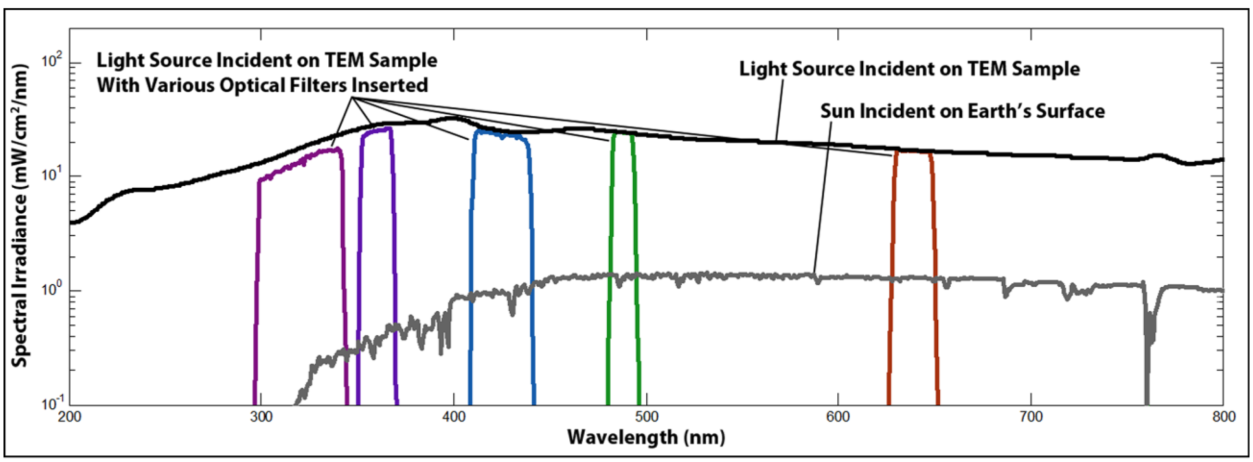

Figure 3: Spectrum Comparison. This figure shows the maximum spectral irradiance of the light which is incident on the TEM sample. This is the intensity at the position of the electron beam. The irradiance is quite high, and is compared, for reference, to the irradiance of sunlight on the Earth's surface. Also shown are several spectra which give the spectral irradiance at the sample after a filter has been inserted before the optical fiber. Each peak is a separate spectrum, for a different filter.All spectra (except solar) include spectral attenuation by all optical components including the optical fiber.

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