

**CAN HRTEM AND ELECTRON DIFFRACTION BECOME THE BASIS FOR ROUTINE STRUCTURE SOLUTION AND REFINEMENT IN INDUSTRIAL R&D?**

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The industrial chemist's notion of a successful application of high resolution transmission electron microscopy (HRTEM) would be to introduce the specimen, press a button and obtain the structure solution. As a microscopist, it both humbling and useful to consider this. Although microscopy has far to go, even incremental progress is of profound significance because of the central role which knowledge of structure has in the development of new materials in chemistry and elsewhere, and the degree to which lack of structure knowledge can be the limiting factor.

This talk will describe the recent structure solution of an industrially relevant catalytic material (pending patent approval) using HREM supported by other techniques. The example will serve to illustrate the principal difficulties in routine structure solution and refinement from electron microscope data.

The talk will also discuss several developing techniques, which both remove obstacles and improve the accuracy of the information obtainable from microscopy. Two developing areas which will be covered are the use of quantitative measurement of electron diffraction intensities to perform crystallographic direct methods and crystal structure refinement, and the combination of HREM images and diffraction data. It will be shown how both of these approaches can potentially remove a major obstacle due to microscope aberrations, and improve data obtained from microscopy in both quantity and quality.

**Keywords: ELECTRON MICROSCOPY ZEOLITES STRUCTURE DETERMINATION**

**QUANTITATIVE REFINEMENT OF NANOSTRUCTURES WITH X-RAY PRECISION BY HREM : A DREAM?**

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The improvement in the resolution of electron microscopes to below 1 Å has not to be regarded as a gradual improvement in the performance but more as a quantum leap. Indeed it now becomes possible to visualize individual atoms, the alphabet of nature. In X-ray terminology, electron microscopy is now able to resolve atomic structures. Since also the whole image formation process including the interaction of the electron with the object is now theoretically well understood it will also become possible to quantitatively refine the atomic structures with a precision that is comparable to that of X-ray diffraction. Moreover as compared to X-rays and neutrons, electrons are the most convenient imaging particles because they have the strongest interaction with atoms, they provide the most information for a given amount of radiation damage(1) and they can be deflected by lenses so as to yield information both in real space and Fourier space. These assets enable to study aperiodic structures such as crystal defects, nanostructures and amorphous materials and will give electron microscopy a unique position in the nanoscience of the future where precise quantitative atomic structure characterization of nanostructured materials will be imperative. However thus far these potentialities are not yet met.

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**ABERRATION-CORRECTED STEM: TOWARDS THE ULTIMATE RESOLUTION FOR IMAGING AND ANALYSIS**

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With probe sizes of 0.05 nm predicted after correction of aberrations, microscopy of zone axis crystals is poised to reach its quantum mechanical limit. For thickness greater than the kinematical limit, interpretable resolution will be limited by the size of the smallest Bloch states, the 1s states, which are typically 0.05 – 0.08 nm in diameter. In phase contrast microscopy, imaging with 1s Bloch states can be achieved by selecting a sample thickness in which the contribution of the 1s state to the exit face wave function is maximized. However, this thickness is dependent on composition and at greater thickness the contrast reverses. In Z-contrast imaging the detector selects 1s states. As the probe size is reduced, eventually, the Z-contrast image will become a direct image of the 1s Bloch states. Similarly with core loss EELS, provided the acceptance angle is large, delocalization is negligible. For single atoms the ultimate resolution becomes the geometric size of the core electron orbital, but in a zone axis crystal the ultimate resolution is again the 1s Bloch state. The aberration corrector installed on the 100 kv STEM at Oak Ridge has improved its resolution to 0.136 nm, giving images comparable to those obtained with the uncorrected 300 kv STEM.

**Keywords: Z-CONTRAST, STEM, ABERRATION CORRECTION**

**PRINCIPLES AND MOLECULAR MECHANISM OF MEMBRANE PROTEIN CRYSTALLIZATION IN LIPIDIC CUBIC PHASES**

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We have developed a novel concept for the crystallization of membrane proteins using highly viscous, optically transparent and by non-birefringent lipidic cubic phases (1)- the in cubo crystallization. The crystal structure of the light-induced proton pump bacteriorhodopsin (bR), grown from a monoolein lipidic cubic phase, was solved to 1.9 Å resolution, revealing the first high resolution structure of a biological membrane (2). In parallel, we have demonstrated that bR molecules packed in 3-D crystals undergo a light-induced photocycle that is very similar to that of bR in the native purple membrane (3). This finding was followed by the determination of the high-resolution structures of the first two intermediates (4,5) of bR's photocycle, illustrating the early rearrangements that occur upon photoexcitation. Recent crystallization of other membrane proteins from lipidic cubic phases (6,7,8), as well as elucidating the structure of the first intermediate in the photocycle of a sensory receptor (9) illustrates the generality of the in cubo crystallization methodology, for which we have recently proposed a molecular mechanism (10).

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