Modulating electron beams in space and time to probe for the genuine structures and function at the atomic scale

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In high resolution electron microscopy objects are actively altered by the intense electron irradiation that is necessary to reach single atom sensitivity. In these circumstances a control of beam-sample interactions is no longer a commodity but a necessity [1]. Therefore, it is of outstanding interest to develop new tools and concepts that strive for a stricter control of the probing electron beam in space and time in order to optimize the detection of every scattering event [2].

This contribution summarizes research efforts that aim at exploiting the emerging ability to analyze and understand the pristine structure of catalysts or nano-materials in general, which often contain soft and hard matter components, surfaces and interfaces that are typically sensitive to the probing radiation because of lowered atom binding energies. We develop and apply low-dose rate electron in-line holography to determine atom arrangements in three dimensions [3] and to access dynamic properties at atomic resolution [1]. Our approach expands detection limits because it mimics best practices in biological research by capturing series of entirely noise dominated images with low dose rates (<20 e/Å^{2}s) and by allowing for an integration of time resolution that can reach towards an unexplored end. Image series are successively reconstructed to extract amplitude and phase information with unprecedented contrast and resolution.

In such images we observe a variety of unknown atom configurations that are otherwise hidden behind a barrier of beam-induced object alterations. It has even become possible to capture radiation sensitive structures at atomic resolution that are greatly affected by an exposure of the material to water vapor or other gases. For the specific case of Ziegler-Natta catalysts, we point out that in spite of its very successful deployment of in industrial processes, a detailed understanding of the catalyst structure is still lacking. The local morphology and structure of salts such as $MgCl_2$ is poorly explored and rarely reported because imaging the pristine state of these materials at atomic resolution is challenging for two reasons: First, their structural integrity is compromised by any exposure to oxygen or moisture (Figure 1). In addition, such materials are susceptible to damage by the probing electron radiation (Figure 1). Our investigations help elucidate the atomic-scale structure of $MgCl_2$ that improves our understanding of structure-property relationships of Ziegler-Natta catalysts.

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Figure 1:
Top row: Advancement of atomic resolution images of $MgCl_2$ by low dose-rate in-line holography. A resolution improvement from 5.9 Å to 1.7 Å is achieved.
Bottom row: Maintaining the pristine structure of $MgCl_2$ by protecting it from air or moisture exposure. The polycrystalline nature of the material is preserved.