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## *Single Crystal 3D Rotation Electron Diffraction from Nano-sized Crystals*

X. Zou[1]

[1]*Stockholm University, Inorganic and Structural Chemistry and Berzelii Center EXSELENT on Porous Materials, Department of Materials and Environmental Chemistry, Stockholm, Sweden*

Electron crystallography is an important technique for structure analysis of nano-sized materials. Crystals too small or too complicated to be studied by X-ray diffraction can be investigated by electron crystallography. However, conventional TEM methods requires high TEM skills and strong crystallographic knowledge, which many synthetic materials scientists and chemists do not have. We recently developed the software-based Rotation Electron Diffraction (RED) method for automated collection and processing of 3D electron diffraction data. Complete single crystal 3D electron diffraction data can be collected from nano- and micron-sized crystals in less than one hour by combining electron beam tilt and goniometer tilt, which are controlled by the RED – data collection software.3 The unit cell, possible space groups and electron diffraction intensities can be obtained from the RED data using the RED data processing software. The figure below illustrates the data collection and data processing of a zeolite silicalite-1 by RED. 1427 ED frames were collected in less than 1 hour from a crystal of 800 x 400 x 200 nm in size. A 3D reciprocal lattice of silicalite-1 was reconstructed from the ED frames, from which the unit cell parameters and space group were determined (P21/n, a=20.02Å, b=20.25Å, c=13.35Å, alfa=90.13°, beta=90.74°, gamma=90.03°. It was possible to cut the 3D reciprocal lattice perpendicular to any directions and study the reflection conditions. The reflection intensities could be extracted. The structure of the calcined silicalite-1 could be solved from the RED data by routine direct methods using SHELX-97. All 78 unique Si and O atoms could be located and refined to an accuracy better than 0.08 Å. The RED method has been applied for structure solution of a wide range of crystals and shown to be very powerful and efficient. Now a structure determination can be achieved within a few hours, from the data collection to structure solution. We will present several examples including unknown inorganic compounds, metal-organic frameworks and organic structures solved from the RED data. Different parameters that affect the RED data quality and thus the structure determination will be discussed. The methods are general and can be applied to any crystalline materials.

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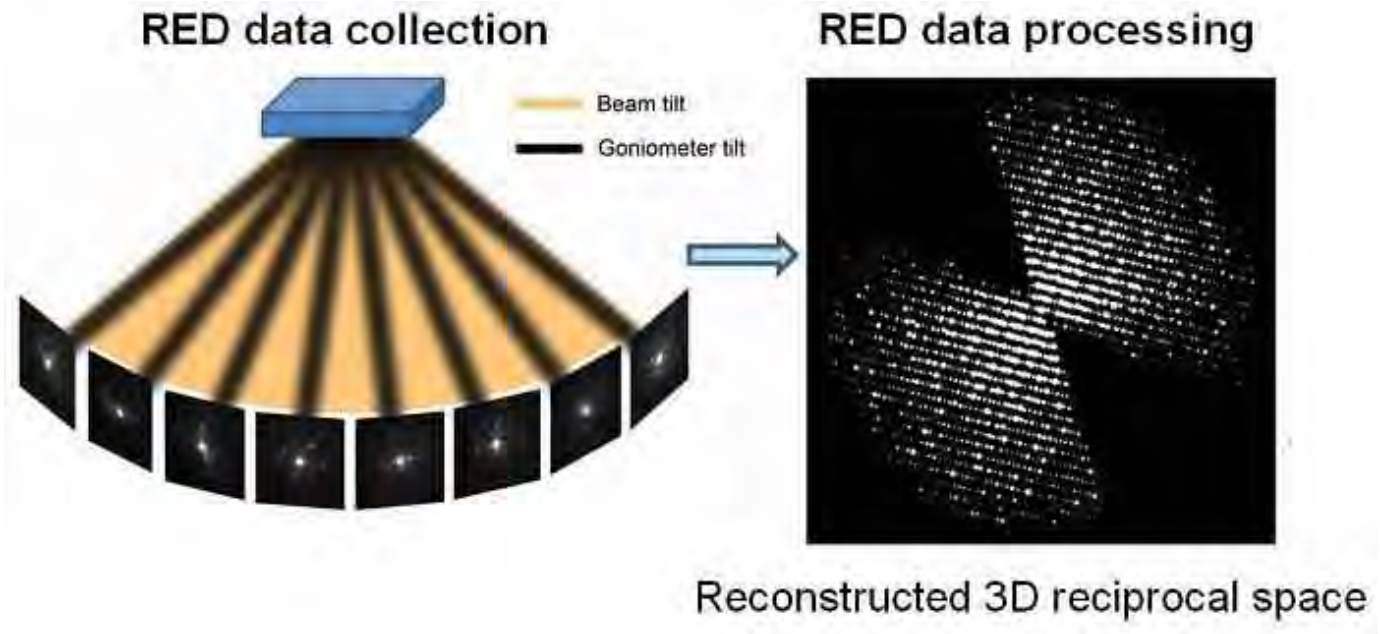


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