MS44-P06 | Automated Serial Rotation Electron Diffraction combined with Cluster Analysis as a Tool for Structure Determination

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Electron diffraction techniques have reached a level where crystal structures can be determined quickly and routinely, but data collection is still very time-consuming and laborious. Therefore, we have developed a strategy to automatically screen for crystals and collect electron diffraction data. In a Serial Rotation Electron Diffraction (SerialRED) experiment, submicron-sized crystals are detected at a low magnification using image processing algorithms, and continuous rotation electron diffraction (cRED) data are collected on each crystal while dynamically tracking the crystal movement during rotation using defocused diffraction patterns. On our JEOL 2100-LaB_6 microscope with a TimePix direct electron detector, data acquisition has been automated entirely, and can run unsupervised for hours after initial setup. In a typical experiment, data are collected on 50-100 crystals per hour. To deal with the large number of data collected, we employed hierarchical cluster analyses to select the optimal data sets for merging. Our tests on several zeolite and metal-organic framework samples show that the structures are determined and refined effectively against these data using standard crystallographic software and that the results are indistinguishable from data collected manually. The large number of crystals also enables phase analysis of materials with known and unknown phases. Data collected on two multi-phase samples show that hierarchical cluster analysis enables the individual phases to be grouped and their structures determined. SerialRED has lower requirements of expertise in TEM and is less labor intensive, and, in combination with cluster analysis, this makes it a promising high-throughput crystal screening and structure analysis tool.