MS19-05 **Tiny diamond anvil cell for high-pressure crystallography at low temperature.** Konstantin Kamenev,[a] Gaetan Giriat,[a] Christopher Cameron,[b] Stephen Moggach,[b] Simon Parsons,[b] *[a]School of Engineering, University of Edinburgh, Edinburgh, UK, [b]School of Chemistry, University of Edinburgh, Edinburgh, UK*
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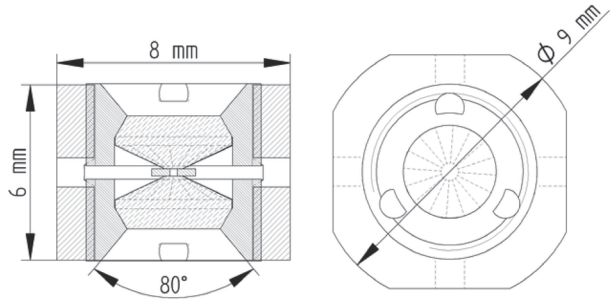
The ability to perform crystallographic measurements at cryogenic temperatures offers a number of advantages such as improved resolution and access to low-temperature phases. Combining low temperatures with high pressures poses an additional challenge and such measurements require use of bulky cryostats which fully enclose the pressure cell. We present a miniature diamond anvil cell for X-ray diffraction at low temperatures, which can be used with cryogenic gas-flow cryocoolers. The design of the pressure cell is shown in the figure below. It is based on the turnbuckle principle [1] in which force can be created and maintained by rotating the body of the device while restricting the counter-threaded end-nuts to translational movement. The load on the sample is created by rotating the body of the cell with respect to the end-nuts held in a clamp. The cell is designed around a pair of opposed diamond anvils of Boehler-Almax design [2] with 80° solid angle opening for scattered X-ray beam. The dimensions of the pressure cell have been optimized by use of finite element analysis. The cell is approximately a cylinder 6 mm long and 9 mm in diameter, and weighs only 2 g. The cell has been shown to reach the pressure of 15 GPa with 600 ěm culet diamond anvils. The pressure cell has been used with *Cryostream* and *N-Helix* gas-flow coolers from *Oxford Cryosystems*. Due to its small size the cell thermalizes rapidly. We discuss the use of the cell at low temperature as well as some preliminary results.



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MS20-01 **Crystal structure determination using automated electron diffraction tomography.** Peter Oleynikov,[a] Yanhang Ma,[a] Kyung Byung Yoon, [b] Osamu Terasaki,[ac] *[a]Materials and Envronmental Chemistry, Stockholm University, Stockholm, Sweden, [b] Department of Chemistry, Sogang University, Seoul, Republic of Korea, [c]Korea Advanced Institute of Science and Technology, Daejeon, Republic of Korea*
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A new method of quantitative three-dimensional reciprocal space scanning using automated 3D electron diffraction tomography (EDT) is developed [1]. Sweeping reciprocal space is implemented by using the electron beam tilt within a given angular range and a small step. The beam tilt is combined with the mechanical crystal tilt in order to cover the full range of tilt angles available for the accessible transmission electron microscope (TEM) goniometer. The method allows the collection of 3D data by sweeping reciprocal space in the range from -43° to +43° (double tilt holder) or from -72° to +72° using the single ultra-high tilt holder and produces ~2000 frames/hour including crystal tracking. The collected data of high quality can be used for further determination of a 3D crystal structure. Several known structures has been successively solved using the 3D EDT method (for example, $K_2Nb_{14}O_{36}$: s.g. *P*4/*mbm*, a = b = 27.5 Å, c = 3.94 Å; $BaSO_4$: s.g. Pnma, a = 8.856 Å, b = 5.44 Å, c = 7.137 Å; CoP_3: s.g. *Im*-3, a = b = c = 7.707 Å). The important outcome from the structure solution is that the lightest atoms (e.g. oxygen) can be easily identified in the calculated potential maps obtained during the structure solution using the 3D intensities extracted from the EDT datasets. Scanning reciprocal space using EDT allows registering not only the reflections, but also the 3D diffuse scattering intensity around diffraction spots which can be related to defects and stacking faults. Many zeolites and microporous compounds have a lot of defects due to the nature of their crystal structure. One of the representatives in the microporous titanosilicates family is ETS-10 with a framework that contains -Ti-O-Ti-O- chains and has three-dimensional 12-ring pore system with straight pores and pores that are bent due to the faulting [2]. Strong diffuse scattering was observed in the ED pattern frames due to extensive faults present in this material. From the reconstructed 3D reciprocal space we were able to conclude that the diffuse streaks appear only in the direction of the ***c**** axis. The proposed model of random stacking along [1–10] was used to build a super-cell with 40 units along the [001] direction which allowed us to get a good match between the experimental and the simulated diffraction patterns. Another important area of application of the 3D EDT technique is the combination of the electron diffraction data together with high resolution images which was successfully applied in the solution of the most complex zeolite ITQ-39 [3].

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