DAC_restrict
Users manual

A program to calculate accessible reflections from single-crystal diffraction DAC data.

RJ ANGEL

rossangelsoftware@gmail.com

http://www.rossangel.com

INTRODUCTION

The biggest reduction in the precision and accuracy of structure refinements of crystals held at high pressures within a diamond-anvil pressure cell, compared to a dataset collected from the same crystal in air, comes from the reduction in the number of accessible reflections. As a rough guide, a transmission diamond-anvil cell with an maximum half-opening angle of 40^o allows access to only 1/3 of reciprocal space out to $2\theta = 60^o$. The correct choice of orientation of the crystal within the DAC can therefore make a considerable difference to the precision of the resulting high-pressure structure refinements. DAC_restrict is a program to calculate the reflections accessible from a single-crystal held in a transmission geometry diamond-anvil pressure cell and can be used to design the optimal orientation for a crystal prior to loading into a DAC.

For most transmission DACs the body of the diamond cell restricts the maximum values of Ψ_I and Ψ_D to 40 degrees or less. These restrictions apply equally whatever type of detector, point or area detector, is being used. On an area detector the limits to the ψ angles appear on the data images as shadowed areas of the detector. For a point detector the diffractometer control software is set up so that reflections obscured by the pressure cell are not collected. Maximum access to reflections is achieved by operating the goniometer in "fixed-phi" mode (Finger and King 1978, Angel et al. 2000). Nonetheless, for a transmission cell with ψ restricted to 40^o or less, only one-third of the total number of reflections out to 60^o in 2θ are accessible. Furthermore, these accessible reflections are not distributed equally over reciprocal space, but are restricted to those reciprocal lattice vectors that are close to being perpendicular to the axis of the DAC. In order to achieve high precision in all three fractional coordinates of all of the atoms in the structure, one must therefore take care to ensure that the crystal is oriented in such a way so as to allow access to as large a portion as possible of an asymmetric unit of reciprocal space.

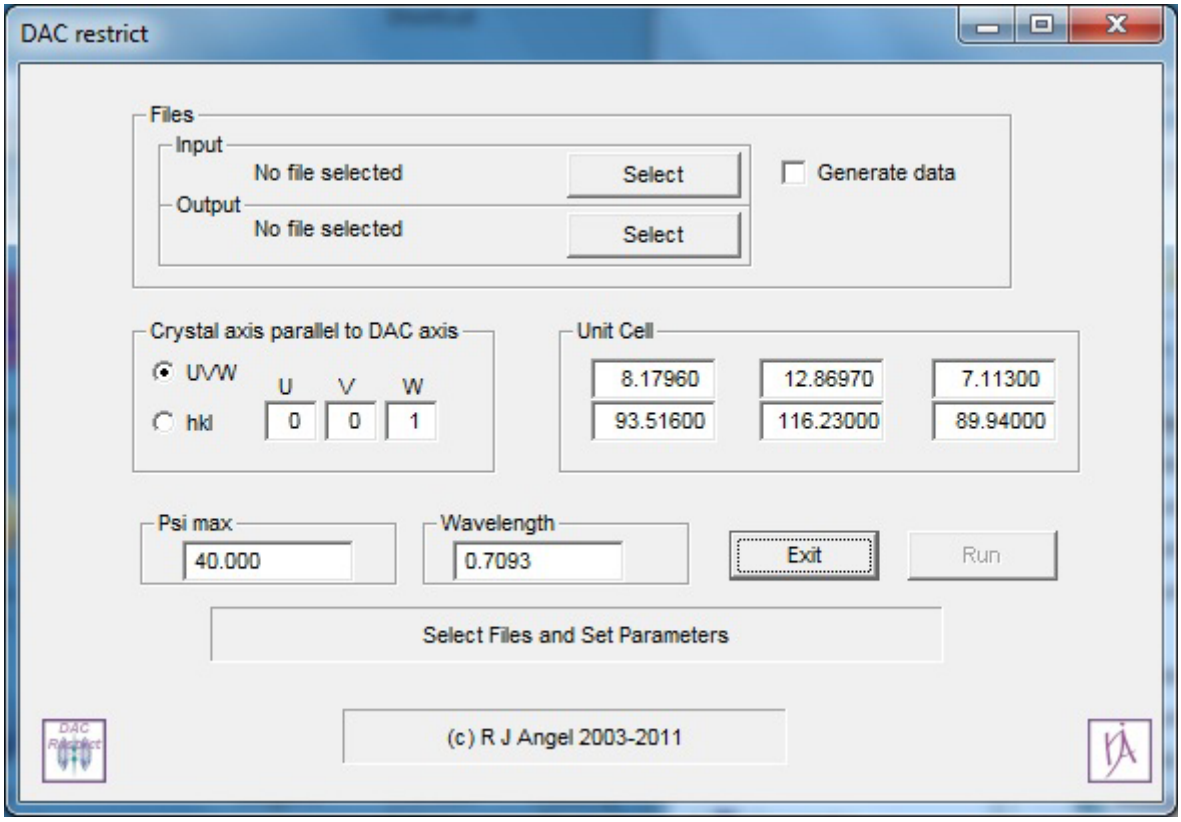
For crystals that are loaded at room pressure, as opposed to being grown *in-situ* in the DAC, the effect of the restricted access can be simulated prior to preparation of the crystal and loading the diamond anvil cell. This allows the optimal orientation of the crystal to be calculated in advance and the sample to be prepared appropriately for the high-pressure experiment. The general principle is that, for measurements in transmission-geometry DACs, crystals belonging to uniaxial crystal systems should whenever possible be prepared as *hk0* crystal plates, so that the c-axis lies parallel to the culet surface of the diamonds. This ensures that reflections with both high values of *l* and high values of *h* and/or *k* are accessible. For lower-symmetry crystal systems it is often possible to choose an orientation that still provides reasonable resolution along all three reciprocal lattice vectors (e.g. Downs et al. 1996). When one data collection is not sufficient to provide the necessary data coverage, it is then necessary to collect two separate datasets. This can be achieved by reloading the same crystal twice into a cell in different orientations and collecting data at the same pressures for each orientation (e.g. Angel 1988). An alternative is to load together two (or more) crystals of the same sample in different orientations in the diamond-anvil cell and to collect the data from each crystal.

1. INSTALLATION

1. The dacrestrict.zip file contains the executable and this manual as a pdf file.
2. Unzip the dacrestrict.exe and the pdf file containing the manual into one folder. It is recommended that this folder is not used for data files.
3. Create a shortcut to dacrestrict.exe either in the working directory, or on the desktop, by right-clicking on the file, dragging to the working directory or desktop and then selecting "create shortcut".
4. Run the program by double-clicking on the shortcut.

2. RUNNING THE PROGRAM

Start the program by double-clicking on the shortcut. The DAC_restrict dialogue box will appear:



Some guidance as to what to do at each step is provided in the message window (that says "Select Files and Set Parameters" in the example).

The program works in two modes:

1. By taking a dataset of reflections (in RFINE or SHELX HKL4 formats) and calculating which reflections would be accessible for a crystal with the specified orientation in a DAC. To use this mode, use the *Select* button to select the input data file.
2. By generating all possible *hkl* of reflections out to a 2θ equal to twice the psi_max angle, and then calculating which reflections would be accessible for a crystal with the specified orientation in a DAC. To use this mode, select the button labelled *Generate data*. Note that this calculation is based on a full sphere of data for a primitive unit cell without any systematic absences.

The reflections that would be accessible in the DAC are written out to the *output data* file which can be selected in the *Files* section of the GUI.

The orientation of the crystal within the DAC is specified by either the [UVW] of the zone axis of the crystal oriented parallel to the load axis of the cell, or the (hkl) of the plane normal of the crystal oriented parallel to the load axis of the cell. In either case, the correct unit-cell parameters must be entered on the GUI.

The DAC is assumed to be axially symmetric with respect to its load axis, with a half-angle of opening of *Psi_max*. Specify the wavelength of the radiation that you will use in the high-pressure experiment. This does not have to match the wavelength used to collect the data that you have put in the *input data* file.

Once you have specified all of these parameters and files, select the *Run* button to execute the calculation. The message window will report how many reflections were processed and how many were found to be accessible with the specified orientation in the DAC. The accessible reflections are written out to the *output data* file which, if you started the program with a real dataset of intensities, can be used to perform a test structure refinement.

REFERENCES

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