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d2Dplot: 2D X-ray diffraction data processing and analysis for through-the-substrate microdiffraction

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The *d2Dplot* computer program provides a set of tools for the visualization, processing and analysis of 2D X-ray diffraction (2DXRD) data. Among the operations available there are the sum/subtraction of 2DXRD images, conversion to 1D data (powder pattern), azimuthal plotting, calibration of instrumental parameters, background subtraction and a command-line mode to run operations inside data processing pipelines. The graphical user interface allows easy use of the program. It also includes two main features: (i) the possibility of creating a user compound database to help in the fast phase identification of similar samples, and (ii) a detailed peak analysis routine for the application of the through-the-substrate microdiffraction methodology.

1. Introduction

The collection of diffraction data using 2D detectors is nowadays common practice. Depending on the nature of the sample and the purpose of the experiment, different data processing procedures are applied (He, 2009). For polycrystalline samples, data measured as 2D X-ray diffraction (2DXRD) images are often integrated to generate the corresponding 1D powder diffraction patterns. For single crystals, 2DXRD data are routinely collected with the rotation method using small steps (Arndt & Wonacott, 1977) and then processed to obtain the list of measured reflections with their respective intensities. Sometimes, however, it can be important to inspect the individual images for specific sample features like texture or graininess. There are several software solutions available for processing 2DXRD patterns, such as the well-established and complete *FIT2D* (Hammersley, 1997, 2016) and the general-purpose *GSAS-II* (Toby & Von Dreele, 2013). Also, software for specific data processing is available, such as *XDS* (Kabsch, 1988), *CrysAlis* (Agilent Technologies, 2014), *DIALS* (Winter *et al.*, 2018) and *MOSFLM* (Battye *et al.*, 2011) for single-crystal data reduction, *adxv* (Arvai, 1994) for the visualization of images, and libraries for azimuthal integration of 2DXRD images such as *pyFAI* (Kieffer & Karkoulis, 2013; Ashiotis *et al.*, 2015).

The computer program *d2Dplot* has been designed to prioritize the visual inspection of 2DXRD images while also implementing basic data processing operations such as the summation or subtraction of frames, conversion of 2DXRD data to 1D data, azimuthal plotting, calibration of instrumental parameters, background subtraction and a command-line mode to run operations inside data processing pipelines. It also includes two main features: (i) the possibility of creating a limited database with a reduced number of compounds (those most frequently occurring) to help in the fast phase identification of similar samples, and (ii) a detailed peak analysis

routine whose output can be used either independently or as input for the application of the through-the-substrate micro-diffraction methodology (tts- μ XRD) described by Rius *et al.* (2015). This methodology allows analysis of transmission diffraction data from polished thin sections mounted on glass substrates and has already been applied to the solution of petrological/mineralogical, cultural heritage and metallurgical problems (Vallcorba *et al.*, 2017; Maritan, Piovesan *et al.*, 2018; Schmalen *et al.*, 2018). Analysis of tts- μ XRD data is the main motivation for *d2Dplot*. It was created as the graphical tool for the development of the tts- μ XRD methodology and now it has become a key part of its application, providing the peak search and integration algorithms and helping during the selection of the correct crystal orientation(s). *d2Dplot* has also proved useful in sample mapping by tts- μ XRD, offering a quick identification of phases during the selection of the zones to be measured.

The most relevant aspects of *d2Dplot* will be described in the next sections.

2. Technical description, availability and requirements

d2Dplot has been written in Java and can be executed on any PC with the Java runtime environment installed (version 1.6.0_10 or higher). The following third-party libraries have been used: Apache Commons Math (version 3.6.1, Apache licence), *MigLayout* (version 4.3, written by Mikael Grev, BSD licence) and *imageJ* [version 1.50i (Schneider *et al.*, 2012), Public Domain] for reading and writing images in TIFF format. The software can be downloaded from the ALBA Synchrotron Light Source web site (<https://www.cells.es/en/beamlines/bl04-mspd/preparing-your-experiment>) and includes a user manual detailing the options and operations that can be performed as well as the file formats that can be used. The

software can be used free of charge for non-commercial and academic purposes.

3. Program functionality and features

The main window of the program (Fig. 1) contains (i) a top menu allowing access to all the options and operations it can perform, (ii) a left panel with information about the current pixel of the image pointed to by the mouse and the image contrast adjustment, (iii) a right panel with shortcuts to the most common operations, (iv) a bottom log panel with output messages from the program and (v) the central region displaying the current opened frame. The interaction with the opened frame is done by using the mouse (left, right, middle buttons and the wheel). Several 2DXRD data formats are supported by *d2Dplot*, namely EDF (ESRF data format, <http://www.esrf.eu/computing/scientific/SAXS/doc/SaxsKeywords/SaxsKeywords.pdf>), IMG (Arvai & Nielsen, 1983), GFRM (Bruker, 1999), SPR, CBF v1 (Bernstein & Hammersley, 2005) and TIFF. If present, the instrumental information within the image header is read. This information can be accessed and edited inside the 'Image Parameters' dialog. If consecutive frames with the same name are numbered, easy change of images is possible by simply clicking the next/previous buttons.

3.1. Basic 2DXRD processing

d2Dplot provides a set of processing tools to prepare the 2DXRD data for further analysis. The main operations available are (i) summation or subtraction of frames (pixel-by-pixel operations), (ii) conversion between data formats, (iii) calibration of instrumental parameters, (iv) definition of excluded zones, (v) background subtraction, (vi) conversion to 1D powder diffraction pattern, and (vii) generation of azimuthal plots. Details of the most relevant operations are listed below.

3.1.1. Calibration of instrumental parameters. *Preliminary estimates.* The experimental sample-to-detector distance (D), the beam centre position on the detector plane (given by the x_0 and y_0 pixels) and the orthogonality of the detector during the experiment are derived from the 2D powder diffraction data of a calibrant substance. The program includes LaB₆ and Si, but other substances can be added as calibrants by introducing a list of d spacings in the configuration file. The detector orthogonality deviation is defined by two angles: (i) the *tilt* angle measuring the deviation from the orthogonality of the beam with respect to the detector plane, and (ii) the *rot* angle describing the clockwise rotation from the vertical y axis to the minor axis of the ellipse (fitted to the experimental Debye ring) as shown in Fig. 2. The definition of the detector deviation from orthogonality using two orthogonal angles is the most extended method to correct this distortion in the calculation of the effective 2θ for each pixel (Hinrichsen *et al.*, 2008; Hart *et al.*, 2013), and is the method normally used in diffraction software, e.g. *FIT2D* (Hammersley *et al.*, 1994).

The calibration is carried out by taking the values of the detector distance, the wavelength and the beam centre from the frame header (image parameters) and by automatically

Figure 1
Main window of *d2Dplot* after opening a 2DXRD frame.

Figure 2
tilt and *rot* angles used to define the deviation from detector orthogonality with respect to the beam.

selecting or, alternatively, by clicking several points on the elliptically shaped inner Debye ring of the calibrant. After the least-squares ellipse fitting to the Debye ring, the estimates of the instrumental parameters, (x_o, y_o) , D , *tilt* and *rot*, are obtained from the expressions (Hart *et al.*, 2013)

$$\text{tilt} = -\sin^{-1}(e \cos 2\theta), \quad (1)$$

$$D = \frac{R_m^2}{R_M \tan 2\theta}, \quad (2)$$

$$c = R_M \tan 2\theta \tan \text{tilt}, \quad (3)$$

where R_M , R_m and e are the major axis, minor axis and eccentricity of the ellipse, respectively. The beam position on the detector is calculated from c , which is the distance from the ellipse centre to the beam position on the detector surface along the major axis. This process is automatically repeated from the inner to the outer Debye rings, thereby using the new estimates of the instrumental parameters. As preliminary estimates of the instrumental parameters, the mean values over all the rings are taken.

Refinement of the instrumental parameters. By starting with the preliminary instrumental parameters, the refined ones are obtained by minimization of the sum of absolute differences between the theoretical and experimental 2θ values of the calibrant reflections,

$$\sum_i \sum_{j(i)} |2\theta_i - 2\theta_j|, \quad (4)$$

where the outer sum extends over the considered calibrant rings and the inner sum over the (x_j, y_j) points on each calculated i ellipse. Following the description by Hinrichsen *et al.* (2008), the calculated 2θ for a given measured pixel (x, y) , defined from the beam centre at (x_o, y_o) and considering the detector *tilt* and *rot* as defined in *d2Dplot*, is

$$2\theta = \tan^{-1} \left[\frac{x_{Ro}^2 \cos^2 \text{tilt} + y_{Ro}^2}{(D/\cos \text{tilt} + x_{Ro} \sin \text{tilt})^2} \right]^{1/2} \quad (5)$$

wherein (x_{Ro}, y_{Ro}) results from applying the $(-rot)$ rotation to (x, y) , namely $x_{Ro} = x \cos rot - y \sin rot$, $y_{Ro} = x \sin rot + y \cos rot$.

3.1.2. Excluded zones. The frame zones excluded from further calculations can be set by an intensity threshold, a

frame margin, a detector radius, using geometrical shapes (polygon, arc, beamstop) or painting the pixels with the mouse. The information on the excluded zones can be saved either as an independent text file or as a zero-valued mask image with excluded pixel intensities equal to -1 .

3.1.3. Background subtraction. Background subtraction is normally necessary when the intensity contribution from the holder (*e.g.* glass) is significant. A background image can be directly subtracted from the data or, alternatively, the background intensity at a given pixel can be estimated by averaging either square areas (*avsq*, with the side in pixels as the N parameter) or arc-shaped areas (*avarc*, with the radial width and azimuthal aperture as parameters) around the pixel in several iterations. This is equivalent to the smoothing procedure described by Brückner (2000) adapted to 2D data.

For non-polycrystalline data there are two additional faster but less accurate procedures available: (i) the circular averaging of the pixel intensities in the corresponding 2θ ellipse (*avarc*); (ii) the selection of the minimum value among I_0 , I_1 , I_2 and I_3 , where I_0 is the intensity of the pixel and I_1 , I_2 and I_3 are the intensities of the related pixels resulting from the application of a mirror reflection to the image on the x axis, y axis and both, respectively. Instead of single pixels, square zones (*minsq*) or arc-shaped zones (*minarc*) are used.

3.1.4. Conversion of a powder 2DXRD image to a 1D powder diffraction pattern. The 1D powder diffraction pattern is obtained from the 2DXRD image by populating a histogram at regular 2θ intervals with the pixel intensities and posterior division by the number of pixels contributing to each interval (the excluded pixels are not taken into account). The 2θ of each pixel is calculated with expression (5). The minimum interval size allowed is the smallest 2θ value variation between two adjacent pixels. Azimuthal bins (or ‘cakes’) can also be defined. For randomly oriented powders, the Lorentz and polarization factors affecting the intensities of the 1D pattern (Lipson *et al.*, 2004) are, respectively,

$$L = \frac{\sin^2 2\theta}{\cos \theta}, \quad (6)$$

$$P = \frac{1}{2}(1 + \cos^2 2\theta). \quad (7)$$

Since powder patterns taken on conventional diffractometers have the same Lorentz–polarization expression, direct comparison between them is possible. The conversion to a 1D powder pattern is useful not only for phase identification purposes, for example in combination with the PDF4 file of the International Centre for Diffraction Data (Faber & Fawcett, 2002), but also for Rietveld refinement or for quantitative analysis.

Even for tts - μ XRD experiments with significant substrate contribution (*e.g.* glass support), the conversion to a 1D pattern is helpful. Recent applications are the characterization of laser-welded Al–Cu joints (Schmalen *et al.*, 2018) and the identification of secondary phases in ancient ceramics (Maritan, Casas *et al.*, 2018).

3.1.5. Azimuthal (circular) plot. By selecting this operation the pixel intensity evolution along an ellipse specified by the

2θ angle and a given tolerance (2θ window in degrees) is plotted. This plot allows extraction of valuable information on the grain coarseness and/or sample texture.

3.2. Grain analysis

d2Dplot includes a set of tools to process 2DXRD images containing contributions from a single or several grains. This is one of its main features which also complements the *tts-μXRD* methodology for the analysis of transmission diffraction data from thin sections mounted on glass substrates (Rius *et al.*, 2015). *tts-μXRD* consists of a series of measurements at selected sample points by using the rotation method with wide angular overlapping increments (typically between 5 and 15°) to cope with eventual diffracting volume variations along the section depth during the measurements (Rius, Vallcorba, Crespi & Colombo, 2017). The orientation of the crystal(s) is then found, and the intensities are extracted and scaled by using all the measurements. The integrated intensities can be used later for structure solution and/or refinement. *d2Dplot* assists *tts-μXRD* by finding the spots on the image, extracting the intensities, preparing the input files, launching the *tts_software* (Rius, Vallcorba & Frontera, 2017) and, finally, checking the orientation results at the various stages (Fig. 3). The program modules and the main options involved in these steps are detailed below.

3.2.1. Peak identification module. To find the peaks on the image, the search algorithm first applies several tests to each image pixel. These check the following:

(1) That the pixel intensity is greater than a threshold. The threshold is set as $I_{\text{mean}} + n\sigma_I$, where I_{mean} is the mean intensity of the image and σ_I is the corresponding standard deviation. A default value of n can be set by the program. If the back-

ground calculation is used (available as an option) then I_{mean} and σ_I refer to the background image estimated by the *avsq* method defined above (with $N = 3$).

(2) That the eight neighbouring pixels have lower or equal intensities.

(3) That in a surrounding 5×5 grid pixel zone the number of pixels with intensity greater than I_{mean} is higher than the minimum required number of pixels to consider it a peak (*minpix*).

Pixels surviving these tests provide a list of candidate peaks which are further processed as follows:

(4) Saturated peaks are labelled and the centroid is found when the plateau is spanned by more than one pixel.

(5) Close peaks are merged. The option *peak merge zone* defines the radius (in pixels) of the circular zone around each candidate peak. Peaks inside are merged and the final peak position is found by intensity weighting [Fig. 4(b)].

(6) Additional peak merging is performed along the azimuthal direction of each peak (double of the *peak merge zone* value) to minimize mosaicity effects.

The resulting diffraction peaks are integrated (Y_{sum}) by adding the intensity of all the pixels inside an arc-shaped zone containing the peak and subtracting the background intensity of the zone. The zone dimension can be defined manually or automatically by checking the intensity variation in the radial and azimuthal directions from the peak centroid. At the end of the process, the peak search table contains the following information:

- (i) Peak position ($x_{\text{pix}}, y_{\text{pix}}$).
- (ii) Distance in pixels from the beam centre to the peak (*radius*).
- (iii) Maximum and mean intensity of the peak ($Y_{\text{max}}, Y_{\text{mean}}$).

(iv) Intensity of the peak corrected for Lorentz and polarization effects according to

$$|F_H|^2 = Y_{\text{sum}} L^{-1} P^{-1}, \quad (8)$$

where

$$L^{-1} = 2 \sin \theta (\sin^2 \varphi - \sin^2 \theta)^{-1/2}, \quad (9)$$

$$P^{-1} = (\cos^2 \psi + \sin^2 \psi \cos^2 2\theta)^{-1}, \quad (10)$$

with φ being the angle between the axis of rotation and the reflecting plane, and with ψ being the angle between the normal to the polarization plane and the vector going from the centre of the 2D detector to the ($x_{\text{pix}}, y_{\text{pix}}$) pixel (Lipson *et al.*, 2004). The associated standard deviation (s_{FH2}) is also given.

(v) Intensity of the background (Y_{bkg}) and the associated standard deviation ($s_{Y_{\text{bkg}}}$).

Figure 3
Typical *d2Dplot* and *tts_software* workflow, showing their complementarity.

(vi) Total number of pixels in the integration zone (N_{pix}) and the number of saturated pixels (*satur*).

(vii) Radial width in pixels (*RadWth*) and azimuthal aperture in degrees (*AzimDeg*) of the integration zone.

(viii) d spacing of the peak in Å (*dsp*).

(ix) $p = \pi(Y_{\text{max}}/Y_{\text{sum}})^{2/3}$.

(x) Number of overlapping spots in the peak (*swarm*) and a flag to indicate if the peak integration zone is touching an excluded zone (*nearMsk*).

Fig. 4(a) shows the peak search result on a diffraction image of a polished thin section of a garnet (grossular) specimen mounted on a glass substrate. Diffraction data were collected at the microdiffraction/high-pressure endstation of the MSPD beamline at the ALBA Synchrotron (Fauth *et al.*, 2013) with a Rayonix SX165 CCD detector (round active area of 165 mm diameter, frame size 2048×2048 pixels, $79 \mu\text{m}$ pixel size, dynamic range 16 bit) at 184 mm of the sample using an energy of 29.2 keV ($0.4246 \text{ \AA}$) and a beam size of $15 \times 15 \mu\text{m}$. Five frames were collected with $\Delta\varphi = 7.5^\circ$ and an offset range -15 to 15° .

The results of the peak integration can be exported as a PCS file for the *tts_software* or as a text file. Batch integration of a set of images is also implemented.

3.2.2. *tts_software* module. This module is a graphical interface for the *tts_software* to assist in the *tts*- μXRD data

Figure 6
d2Dplot and *tts_software* applications. (a) Orientation results for two different domains of the mineral dickinsonite contained in the same microvolume. (b) Evolution of the γ -trimesic acid cell parameters as a function of pressure. Cell parameters are refined to match the new reflection positions when the pressure is increased. The green dots show the expected reflection positions before and after refining the unit cell.

information is stored in a plain-text format file to allow easy reading and modification. It consists of the list of d -spacing positions either entered as a file or generated from the corresponding CIF (Hall *et al.*, 1991). In the latter case, intensities are also calculated and stored. Selection of the displayed compound and the operations involving the UDB are performed from the Compound DB window (Fig. 7). Phase identification from the peak positions is carried out considering the current selected reflections (left mouse button on the frame to select a reflection) and all the compounds in the database, showing those which better match the entered d -spacing values.

In the course of a *tts*- μ XRD experiment, it often occurs that the number of possible phases in a thin section is rather small. In such cases it is convenient to have direct access from the main window (via a Quicklist) to this small subset of phases in the UDB. Selection of a compound through the Quicklist option quickens the visual control of the compound matching, since opening of the UDB window is not necessary.

3.4. Command-line mode

The program can be launched from the command line giving the image file path as an argument to open it directly. In addition, instrumental parameters calibration and conversion of 2DXRD data to a 1D pattern can be launched from the command line without displaying the graphical interface. This is accomplished by entering `-macro` as the first argument followed by additional arguments depending on the operations performed. This mode is intended to be used for pipelines of operations on images, for example to automatically

Figure 7
Database window showing (top) the list of compounds in the database with calculated rings displayed on the 2DXRD frame for the selected compound, and (bottom) the ranked list of matching compounds after the peak-based search.

generate the 1D pattern from 2DXRD data after collection with the 2D detector.

4. Conclusions

d2Dplot acts as the graphical interface of the *tts_software*. In the analysis of crystalline microvolumes with the *tts*- μ XRD methodology, it simplifies its application by guiding and helping users through the full workflow. *d2Dplot* provides a complete set of tools for the visualization and processing of 2DXRD data, especially for applications where the detailed inspection of diffraction frames is important. It also serves as a tool during the data collection stage, offering quick phase identification or batch processing capabilities. The combined use of *d2Dplot* and *tts_software* has been successfully applied to the *in situ* characterization of polished thin sections in complex mineralogical and cultural heritage problems.

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