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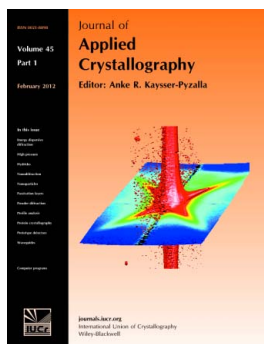
**Alejandro B. Rodriguez-Navarro, Krzysztof Kudlacz and Miguel Ortega-Huertas**

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# Automatic sample changer for the analysis of powder samples on an X-ray single-crystal diffractometer equipped with an area detector

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A low-budget automatic sample changer that allows the rapid analysis of powder or polycrystalline samples using an X-ray single-crystal diffractometer equipped with an area detector is here described. It uses control software that synchronizes the movement of a linear stage with the collection of frames by the diffractometer. Small pieces of thin solid samples are directly attached to a 30 cm ruler, which is used as a disposable magazine, while loose powder samples are loaded into holes on an acetate strip covered on both sides with Kapton tape and attached to the ruler. The use of this type of diffractometer with a sample changer is a simple, low cost and effective solution for studies requiring the analysis of a large number of samples by X-ray powder diffraction. With the current design up to 30 samples can be measured in a single experiment. The current sample changer was designed so that it can be fitted into a Bruker SMART APEX X-ray single-crystal diffractometer. However, it can be easily adapted to diffractometers from other makers.

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## 1. Introduction

In many industrial processes (*e.g.* polymorphism studies, raw materials control) and research studies (*e.g.* new drug or materials synthesis by combinatorial chemistry) it is necessary to characterize a large number of samples by X-ray diffraction in a short time (He, 2009). To do this efficiently, it is necessary to achieve some degree of automatization in the measuring process. While modern X-ray powder diffractometers have sample changers that fully automatize the measurements, they are typically designed to measure relatively large amounts of sample (a few grams) in order to achieve the good particle statistics necessary to obtain high-quality diffractograms (Klug & Alexander, 1974; Cullity, 1977; Olsen *et al.*, 2010). Data acquisition takes a few tens of minutes per sample. Alternatively, it is now possible to use X-ray single-crystal diffractometers equipped with an area detector for the analysis of powder samples (Rodriguez-Navarro, 2006; Reibenspies & Bhuvanesh, 2009). This type of diffractometer uses a small collimated beam ( $<0.5$  mm) that is especially suited for the analysis of microsamples (a few micrograms; Bhuvanesh & Reibenspies, 2003). On the other hand, the use of an area detector and special optics (monocapillary) can reduce enormously the data acquisition time to a few seconds per sample (Berthold *et al.*, 2009). The use of such detectors also compensates partially for the effects of poor particle statistics and/or preferred orientation intrinsic to small samples. The latter problems are resolved by calculating the equivalent powder diffractogram by radially integrating the whole two-dimensional pattern using specialized software (Hammersley *et al.*, 1996; Rodriguez-Navarro, 2006). Additionally, two-dimensional diffraction patterns contain more information regarding the microstructural characteristics of samples (*i.e.* grain size, preferential orientation, mosaicity, stress *etc.*) than the line profile in diffractograms registered with a conventional X-ray powder diffractometer (Rodriguez-Navarro, 2006).

In this technical note, we describe the construction, setting and use of an automatic sample changer that can be fitted into an X-ray single-crystal diffractometer. Although it has been designed for a Bruker AXS (Madison, Wisconsin, USA) SMART APEX diffractometer, the same device can be easily adapted for other X-ray single-crystal diffractometers. The device components and mode of operation are detailed hereafter.

## 2. Experimental

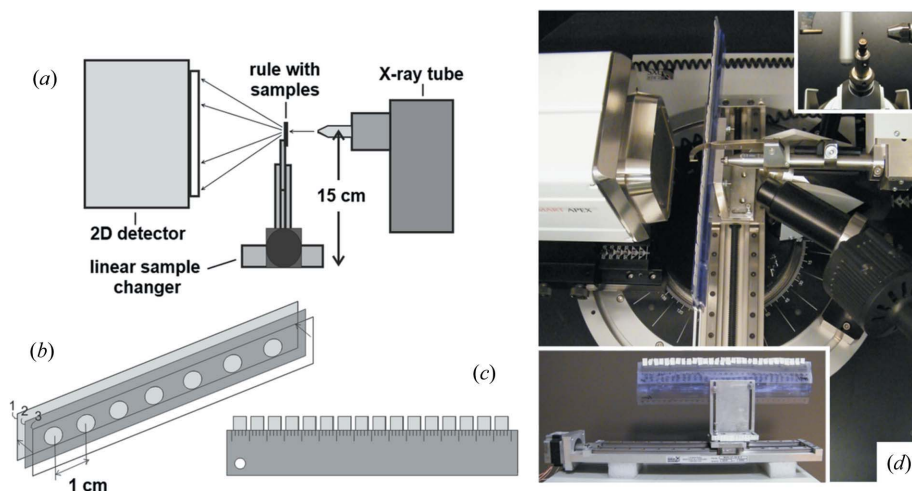
In Fig. 1, a schematic view of the experimental setting for the sample changer is shown. The sample changer is placed between the detector and the X-ray source so that samples are measured in transmission mode. The linear stage of the sample changer translates the ruler holding the samples so that each sample is consecutively moved into the measurement position (that is, the X-ray beam path).

The sample changer consists of the following components:

(a) Linear stage: a linear stage model LPS-12-20 (Servo Systems Co., USA) with a 12.8 inch (32 cm) travelling distance driven by an NEMA 23 stepper motor was used as the main component of the sample changer. Cost: 850 USD.

(b) Step motor drive unit: a programmable step motor drive unit Si3540 (Applied Motion Products, USA) was used to control the linear stage. This unit uses a command language that allows the control of the stepper motor from a PC *via* a serial communication protocol. Cost: 1050 USD.

(c) Sample magazine: a 30 cm ruler is used as a disposable sample magazine. Thin solid samples can be directly attached to this ruler with double-sided tape. On the other hand, loose powder samples can be loaded into holes made in an acetate strip, covered on both sides with Kapton tape and attached to the ruler. Cost: 2 USD per unit.



**Figure 1**

Experimental setting. (a) A schematic of the X-ray single-crystal diffractometer with the sample changer. (b) The powder sample holder, consisting of an acetate strip covered with Kapton tape on both sides. (c) The ruler used to hold the solid samples for measurements. (d) Setup of the sample changer on the SMART APEX diffractometer. For measurements, the sample changer is positioned on top of the  $\omega$  circle plate. Inset at the bottom: side view of the sample changer loaded with a ruler with 30 hen eggshell samples. Inset at the top: powder sample in oil on a glass filament.

(d) Rule holder: a piece of aluminium, which is attached by screws to the platform of the linear stage, was used to hold the 30 cm ruler horizontally. The height of the ruler can be adjusted by these screws so that the samples come into the beam path during measurements. This component was designed and machined for this application. Cost: 300 USD.

To accommodate the sample changer components within the diffractometer chamber, the goniometer head is removed, the  $\omega$  angle is set at  $-90^\circ$  and the detector is set at a  $2\theta$  angle of  $0^\circ$ . Then the sample changer is placed on top of the  $\omega$  circle plate so that the magazine holder is parallel to the detector face and perpendicular to the collimator. We use white paint marks to place the sample changer in the same position. Then the ruler with samples is placed in the

magazine holder. The height of the magazine holder is adjusted so that the samples on the ruler are at the direct beam path height. Then, the linear stage is adjusted so that the first sample center is in the X-ray beam path (aligned with the collimator exit). After connecting the control driver unit to the linear stage motor and to the serial port of the PC, the measurement of samples is ready to start. If the sample changer is correctly set, after moving the sample changer from the first and the last samples, both should be aligned with the collimator exit and at the same distance from it. The position of the sample changer can be further adjusted so that all samples are in focus on the diffractometer video camera.

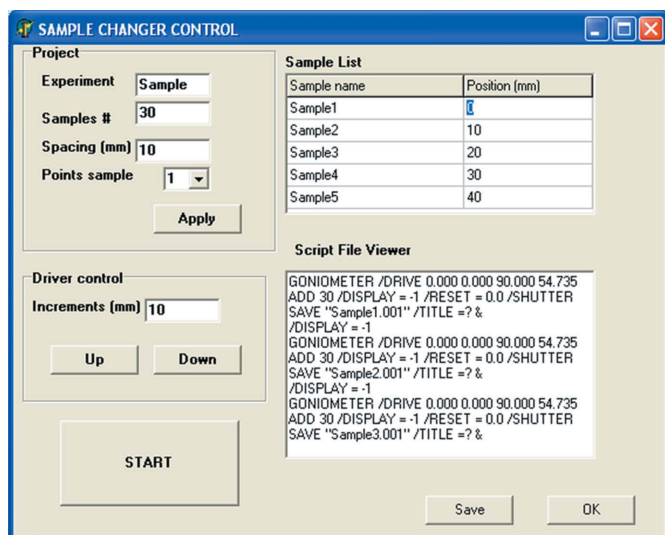
## 2.1. Sample changer control software

The control software is a Windows application, developed using Delphi (version 6; Borland, Austin, USA), that controls the stepper motor driver unit through a serial port and a communication protocol that uses the commands defined by the control driver unit. It allows control of the sample changer in coordination with the X-ray diffractometer measurements. Basically, the diffractometer is programmed to perform a sequence of repetitive measurements and the software detects when one measurement is completed and moves the sample changer to the next position to measure. The software also generates a script file with SLAM commands. This scripting language is defined by Bruker to control the diffractometer directly. An example of a script file used for collecting, saving and displaying a frame with an integration time of 30 s is shown here:

```
GONIOMETER /DRIVE 0.000 0.000 90.000 54.735
ADD 30 /DISPLAY = -1 /RESET = 0.0 /SHUTTER
SAVE 'Sample1.001' /TITLE =?&
/DISPLAY = -1
```

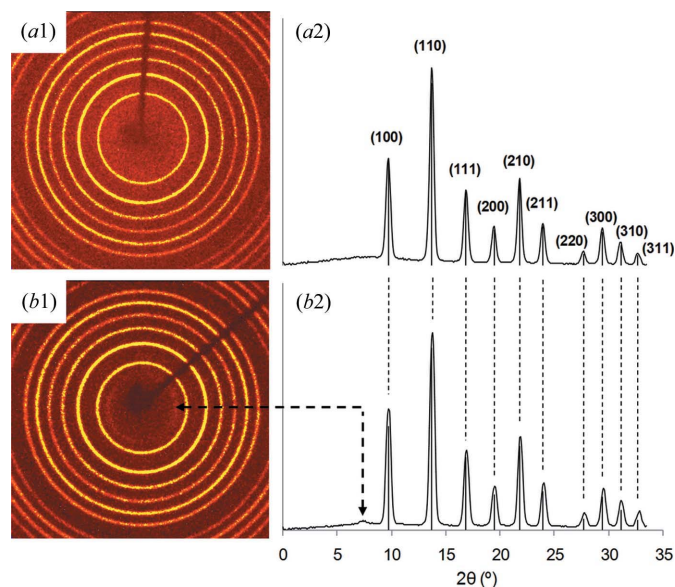
The script file needs to contain these command lines for each sample and/or measurement position.

To start a measurement, first we have to define the name of the experiment (e.g. Sample), the spacing between samples (1 cm by default) and also the number of measurements per sample (1 or 3 points) (Fig. 2). If we define three points, three adjacent points separated by 1 mm will be measured for each sample. Once the experiment parameters are defined, it is necessary to press the Apply and Save buttons to generate and save the diffractometer script file (Sample.slm) associated with the experiment. Next we need to click on the Start button to activate the linear stage and run the script file, Sample.slm, previously generated by the control software. To run the script file, it is necessary to go to the Bruker SMART software main menu and change the user level to the command-line mode. On entering the command @Sample.slm at the command prompt, the commands in the script will be executed sequentially. The shutter is opened and the frame is added and saved. On completion of the measurement, the linear stage moves 1 cm so that the next sample of the magazine comes into the X-ray beam path to be measured. This process is sequentially repeated until the last sample has been measured. Then, the user can load the next ruler loaded with new samples for measuring and start the sequence again.



**Figure 2**

Interface of the sample changer control software, in which the experimental parameters are defined.



**Figure 3**  
Two-dimensional patterns of  $\text{LaB}_6$  powder in oil (a1) and on acetate film (b1). Calculated  $2\theta$  scans of  $\text{LaB}_6$  powder in oil (a2) and on an acetate sheet (b2). The acetate sheet contributes with a weak band at about  $7.5^\circ$ .

### 3. Experimental results

In Fig. 3 are shown the two-dimensional diffraction patterns of a powder sample of  $\text{LaB}_6$  in oil and another sample loaded on an acetate sheet attached to a 30 cm ruler used as a sample magazine (Fig. 1). Samples were measured using the following working conditions:  $\text{Mo K}\alpha$  ( $\lambda = 0.7093 \text{ \AA}$ ), 50 kV and 30 mA, a 0.5 mm pin-hole collimator and an exposure time of 30 s per frame. The two-dimensional frames consist of continuous concentric rings characteristic of a fine powder sample. At the right of each two-dimensional pattern, the calculated  $2\theta$  scans are shown. They were calculated from the original two-dimensional frames using the *XRD2Dscan* software (Rodríguez-Navarro, 2006). In both cases, there is a very good agreement of peak intensities and positions with the  $\text{LaB}_6$  standard lines. The acetate sheet contributes with a minor band, which can be removed with conventional background subtraction methods (Figs. 3b1 and 3b2).

### 4. Conclusions

The use of an X-ray single-crystal diffractometer equipped with an area detector together with a sample changer like the one described

here offers the possibility of rapid screening of a large number of samples for mineral phase and microstructure characterization. The device described here is easy to implement and use, and the parts cost just about 2200 USD. The current sample changer was designed to fit into a Bruker SMART APEX diffractometer. This diffractometer has a three-circle fixed- $\chi$  geometry, which offers more free space at the center and makes it easier to place the sample changer. Diffractometers with  $\kappa$  geometry have less clearance and their adaptation would require a redesign of the sample holder to fit it into the smaller free space. Another important requirement for the use of this sample changer and control software is that the diffractometer has to have the capability of being programmed with a sequence of measurements. This capability is met by Bruker and other makers of single-crystal diffractometers (e.g. Oxford Diffraction). On the other hand, diffractometers that use Mo radiation offer several advantages for the use of the sample changer: (1) they are well suited to analyze mineral and/or inorganic compounds, (2) the highly penetrative radiation is ideal for measuring samples in transmission mode, and (3) the whole pattern is registered in one frame (down to  $0.93 \text{ \AA}$  using the current settings).

### 5. Availability of control software

The control software and further information on the sample changer and how to adapt it to other diffractometers can be obtained on request from the authors.

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