

## Measurements of pole figures with a low cost interface

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**ABSTRACT.** An old Philips PW 1078/24 was updated with two stepping motors and software to measure texture scans. A programmable interface based on an Intel 8052AH microcontroller was built for us, which enables the texture goniometer to measure up to 20 scans. This interface is suitable therefore to be customized for special needs of any laboratory; the device furthermore is autonomous since once running, the computer can be used for other tasks. The adaptation to the texture and powder goniometers is straightforward. The cost of this interface is very low compared with the expenses for adaptation of parts of modern diffractometers.

**RESUMEN.** Se hizo la adaptación de dos motores de pasos y el *software* correspondiente para un antiguo goniómetro de texturas Philips PW 1078/24, para hacer barridos de textura. Se mandó construir una interfase programable basada en el microcontrolador Intel 8052AH, el cual permite al goniómetro de texturas hacer hasta 20 barridos. Esta interfase, por lo tanto, es adecuada para las necesidades de cada laboratorio; el dispositivo es además autónomo, puesto que una vez en operación la computadora puede ser empleada para otras tareas. La adaptación a los goniómetros de texturas y polvos es directa. El costo de esta interfase es muy bajo comparado con los costos de adaptación usando partes de difractómetros modernos.

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### 1. INTRODUCTION

Pole figures (PF) are the graphical elements obtained by X-ray or neutron diffraction which deliver practical information about preferred crystallite orientations, or texture, of polycrystals. As such, this information is essential for studying physical properties of solids where anisotropic effects are important. Texture analysis through PF's is also complementary to metallographic investigations. From PF's, crystallite orientation distribution functions (CODF) can be determined, which are used for anisotropic properties calculations as well as further graphical information.

To measure a PF with an X-ray diffractometer, the detector should be fixed at an angle  $2\theta$  corresponding to a convenient Bragg peak and the sample is rotated around two axes: one ( $\beta$ , from  $0^\circ$  to  $360^\circ$ ) normal to the sample surface, and the other ( $\alpha$  from  $0^\circ$  to  $90^\circ$ ), fixed on the diffraction plane. The first axis moves in a plane perpendicular to the diffraction plane (Fig. 1). The diffracted intensity is proportional to the diffracting volume,

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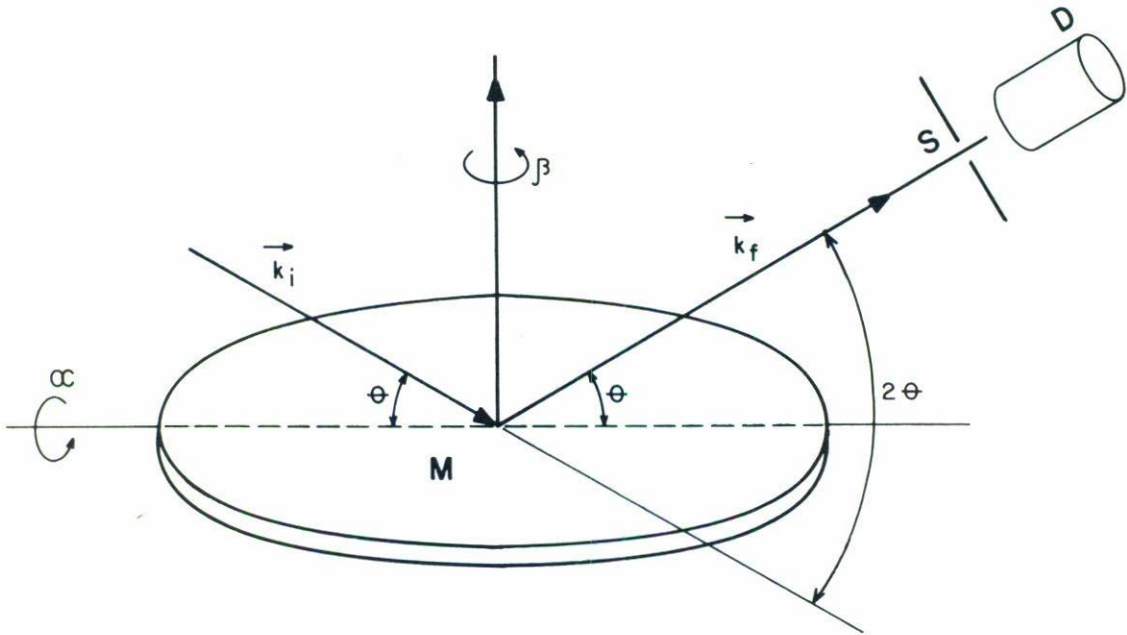


FIGURE 1. Sketch of the array to measure Figure Poles.  $k_i$  and  $k_f$  are initial and final wave vectors respectively;  $\gamma$  is the Bragg angle for the particular reflection and remains constant; M is the sample, S the slit and D the detector.  $\alpha$  and  $\beta$  vary from 0 to 90° and from 0 to 360° respectively.

however, two main corrections should be made, namely absorption and defocusing. The first one is necessary because for different sample orientations different volumes are exposed to X-rays, and the second one, because by increasing  $\alpha$ , the diffracted beam broadens and the slit S in front of the detector lets less intensity to be recorded, so that intensity falls gradually to 0 at  $\alpha = 90^\circ$ . Traditionally both corrections are made by measuring on a textureless sample with otherwise the same characteristics as the sample under study. PF are then intensity level curves in a stereographic projection. (For details of the measuring method see Ref. [1]).

Present day goniometers for  $\alpha$  and  $\beta$  rotations, *i.e.* texture goniometers, are provided with two motors which move independently but are programmed so that for a fixed  $\alpha$  value a full rotation is made by  $\beta$ , stepwise or integrating intensities over 5 degrees intervals, then a new value of  $\alpha$  is fixed and the rotation in  $\beta$  is repeated, so that the measurement is made "in circles" in the stereographic projection;  $\alpha$  is also varied in 5 degrees steps.

Software to show constant intensity curves, or level curves, in the stereographic projection, *i.e.*, Pole Figures, and to calculate other related properties (as the CODF's) start from the assumption that measurements were made that way. Older texture goniometers, as the Philips PW 1078/24, nevertheless were provided with only one motor which moves the sample "in spiral", increasing simultaneously both angles  $\alpha$  and  $\beta$  with a selectable ratio. Furthermore, the motor is synchronous, which means that it is not suitable to be programmed.

To be able to program an old texture goniometer, and to apply modern software for both, rotations control and PF evaluation by fitting modern texture goniometer devices



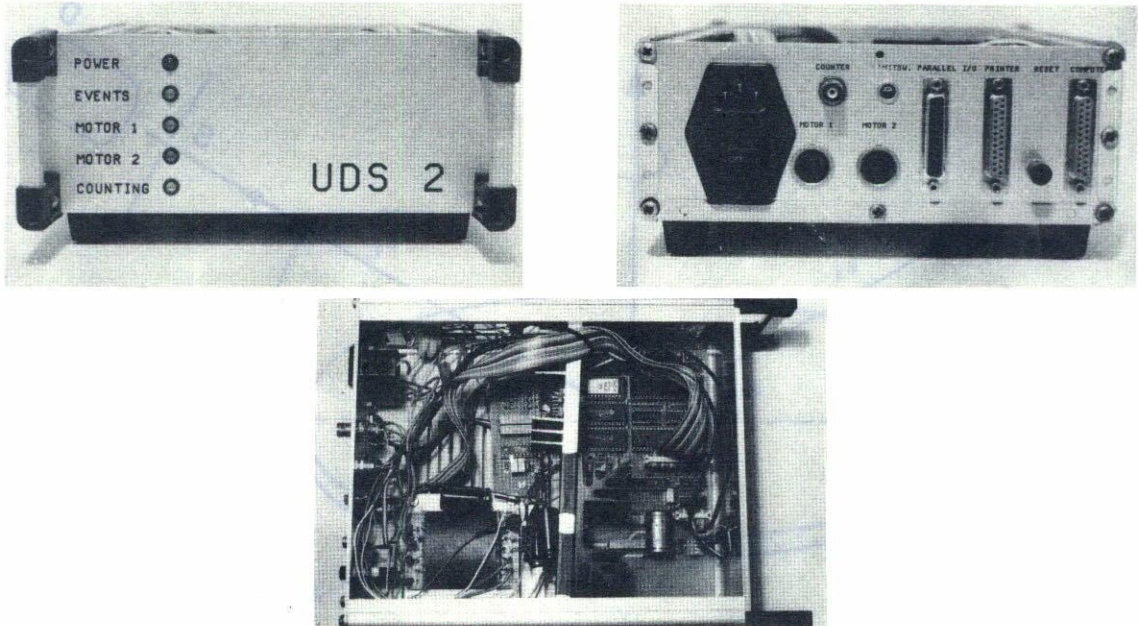


FIGURE 2. a) Front, b) rear and c) circuitry of UDS2 interfase.

has some disadvantages: it is not always reliable, leaves small room for customization to special necessities, and it is very expensive. Our laboratory has a low cost interface made by a small company for the Philips PW 1078/24 texture goniometer, which overcomes those inconveniences.

## 2. THE INTERFACE UDS2

Departing from an interface designed by Steuerungstechnik Skowronek in Jülich, FRG, for a powder diffractometer controlled by a synchronous motor, upon request from us and for the special requirements of our texture goniometer, this company expanded the design with a second stepping motor and its control software. Also, because the sample in the texture attachment has an additional oscillating motion to increase the scanned area, driven by the same single motor, measurements cannot be made stepwise every 5 degrees, but integrating. The interface, called UDS2, is a programmable controller which furthermore can be employed wherever a stepping motor is to be operated and events have to be counted.

The UDS2 was built around an Intel 8052AH microcontroller which has an on BASIC interpreter and can therefore be instantly adapted to carry out control tasks. It has the following resources: 216 bit counters, one timer with a time resolution of 5 ms, about 40 kBytes of user memory (RAM), one programmable peripheral interface chip (8255) with 3 parallel 8 bit ports (two of which are accessible at an internal socket), one 16 kByte Eprom with the necessary programming facility, and a bipolar stepping motor driver capable of driving motors with up to 2 A per phase.

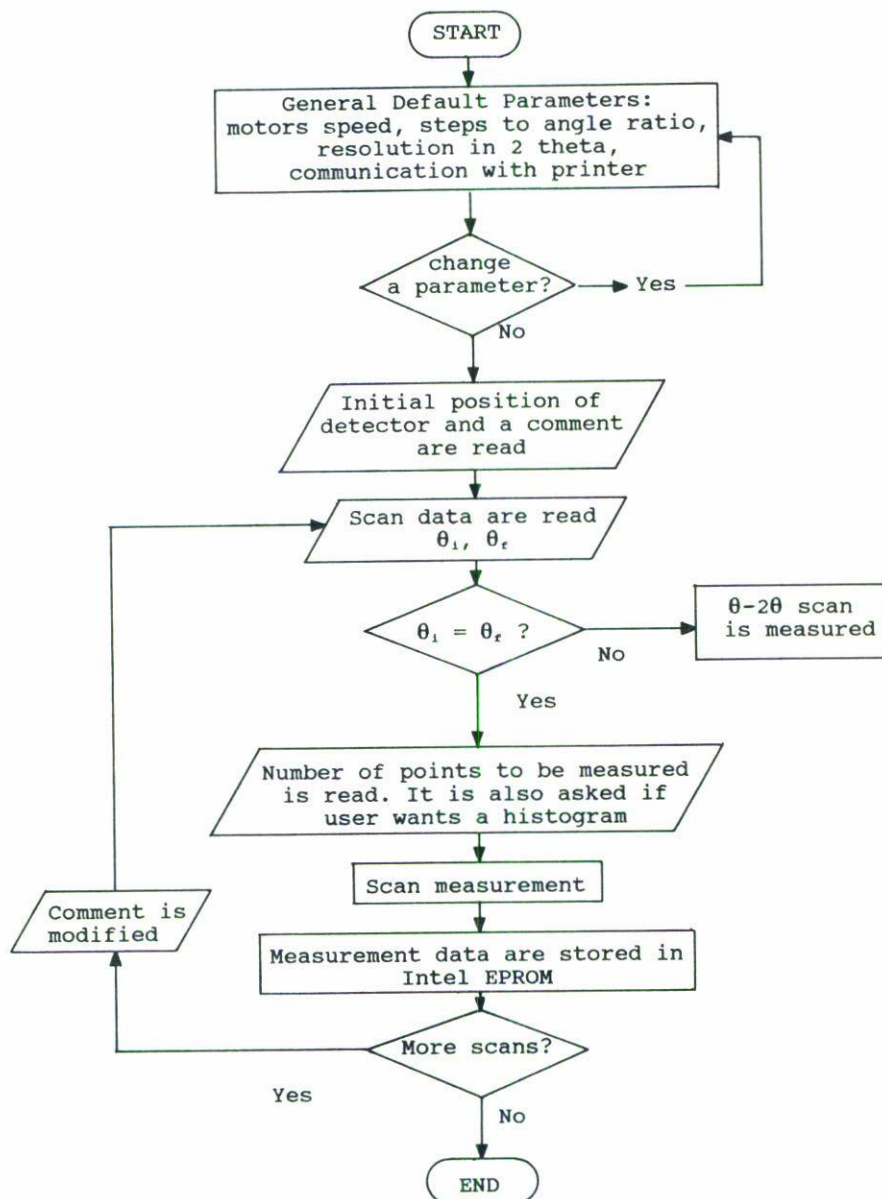


FIGURE 3. Control program flowchart.

The control program is stored in a 27128 Eprom, and includes instructions for the two stepping motors (one for  $\theta$ - $2\theta$  scans and the other for the texture goniometer which is supported by the  $\theta$ - $2\theta$  goniometer), and instructions to store counting rates, which can then be downloaded to a file for further evaluations. Up to 20 scans can be programmed at once; for each scan, the UDS2 asks for initial position in  $2\theta$ , final position in  $2\theta$ , interval between measuring points and measuring time in each position. When the operator gives the same value for initial and final positions, the interface drives the motor for texture

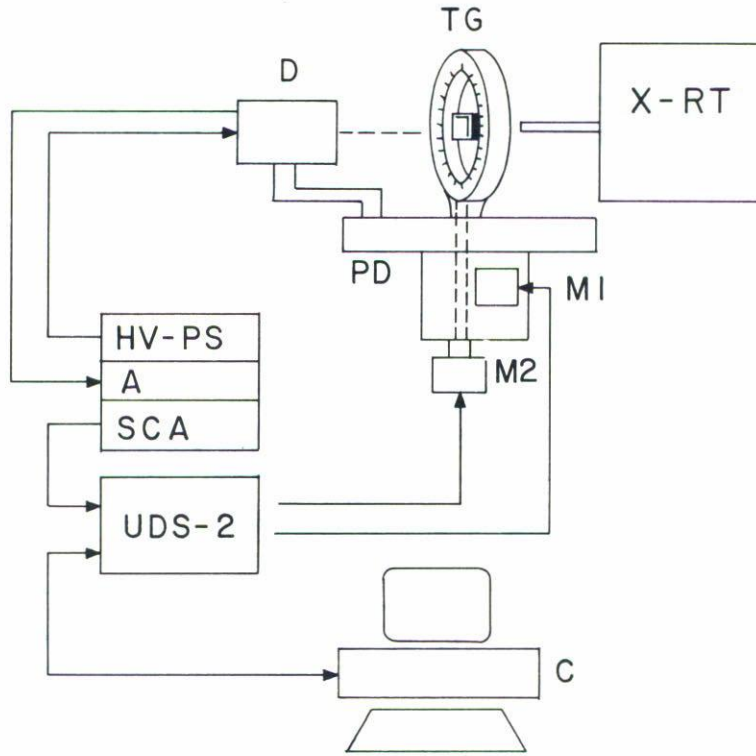


FIGURE 4. Pole figure measurements block diagram. X-RT: X-ray tube; TG: texture goniometer; D: detector with preamplifier; PD: powder diffractometer; M1: stepping motor for powder diffractometer; M2: stepping motor for texture goniometer; HV-PS: high-voltage power supply; A: amplifier; SCA: single channel analyser; UDS-2: UDS-2 controller; C: computer.

and instead of calling out the interval in  $2\theta$ , it calls out the interval in  $\beta$ , and number of cycles. The controller can be programmed using Basic for the 8052. Programs stored in the Eprom can be transferred to the host computer RAM to be modified.

Communication with the computer is performed through a RS 232 serial port and TTL level signals are required for countrates coming from the single channel analyzer. Once a scan is started, the UDS2 is autonomous, so that the computer can be used for other tasks (for example to plot scans previously measured), and at the end of the process the data can be downloaded to a file. Figs. 2a), b) and c) show the interface UDS2, and Fig. 3 shows the control program flowchart. Figure 4 shows a block diagram for the whole system.

### 3. DATA CORRECTIONS

A program in FORTRAN was written to correct absorption and defocusing effects. The program also contains a linear interpolation to obtain data in circles from the measured data in spiral; a three point interpolation was not successful because in the neighborhood



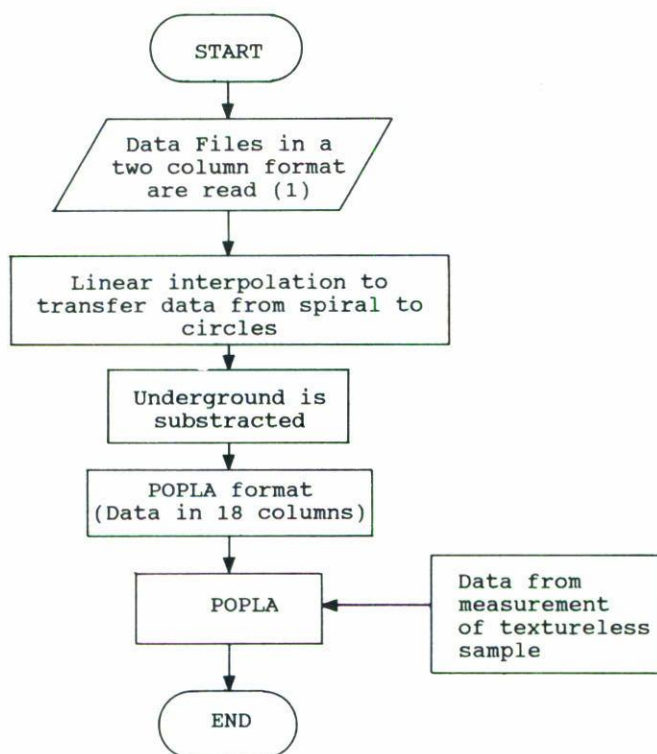


FIGURE 5. Corrections program flowchart. (1) Other formats can also be used, for example from Siemens.

of sharp peaks, the parabolic interpolation fit gives lower —sometimes negative— intensity values.

In order to apply corrections, a textureless sample has to be previously measured; then the program averages for every constant  $\alpha$  circle and stores data in a file to be used by the PF evaluation program. Because aside from the center of the PF, each constant  $\alpha$  circle contains 72 data for the  $\beta$  angles, it is necessary to repeat 71 times the value of the intensity of the PF center. To get a better statistics, this value is determined by the program as the average of the first 15 measured points. Diagram 3 shows the correction program flowchart.

#### 4. POLE FIGURE EVALUATION

To evaluate PF's, CODF's and related functions, as well as to correct with the textureless sample data file and to plot PF's and CODF's, the Preferred Orientation Package Los Alamos (POPLA) [2] was used. This program was developed at Los Alamos National Laboratory and evaluates the CODF's by the direct method and alternatively by the harmonic method. Figs. 6a) and b) show the raw data from a 95% cold rolled pure copper sample, and a recrystallized sample respectively, for the (111) reflection measured with

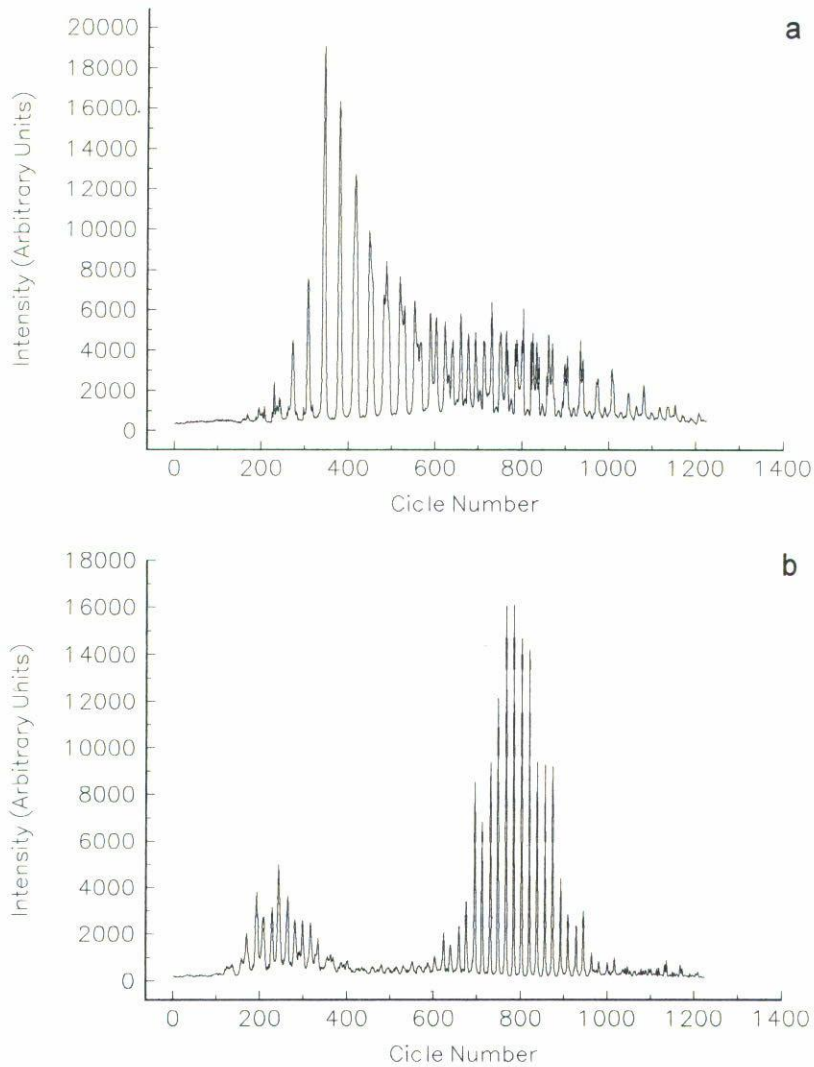


FIGURE 6. Raw data from the measurement of intensity vs cycle number. Each cycle corresponds to  $5^\circ$  in  $\beta$  and every 72 cycles correspond to  $5^\circ$  in  $\alpha$ . a) Cold rolled pure copper, b) Recrystallized sample. We call this type of plot the “Dimetrodon”.

this system, and Figs. 7a) and b) show the corresponding PF's. These PF's compare well with the PF's known from the literature [3].

## 5. DISCUSSION AND CONCLUSION

The adaptation of the new interface and motors to our old Philips texture and powder goniometers did not involve large problems; there was no loss of adjustment in any of the goniometers, and the task lasted about two hours. Some other minor problems, as an initial strong vibration induced by the stepping motor attached to the texture device,

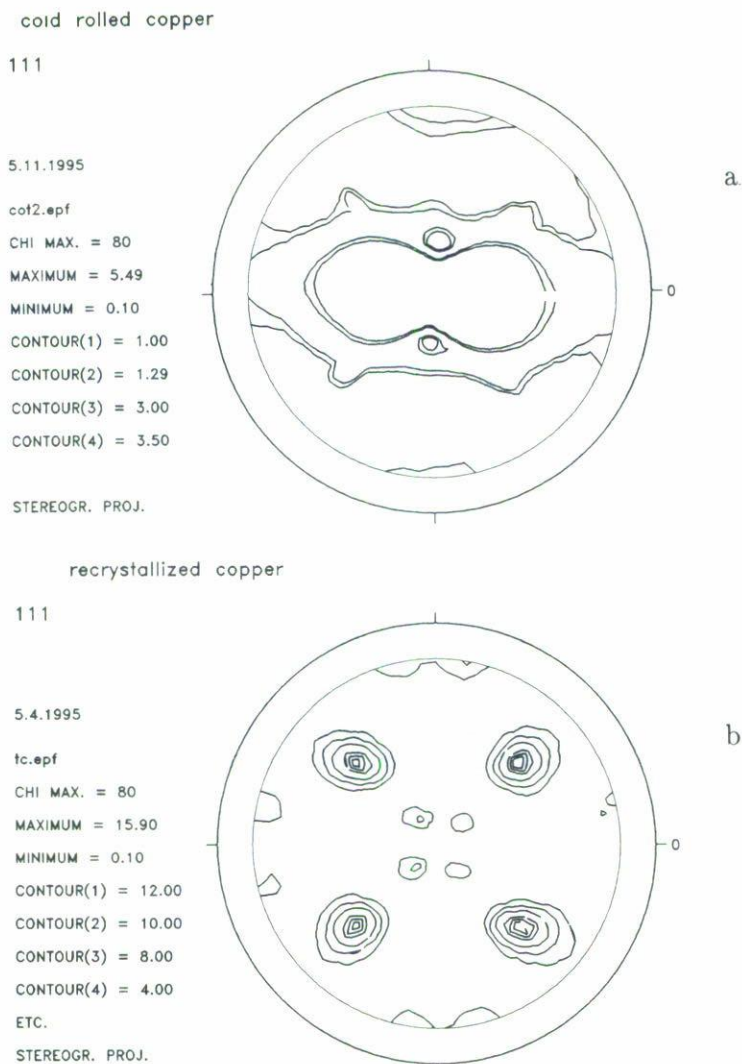


FIGURE 7. Pole figures corresponding to the raw data of Fig. 4. a) Cold rolled sample, b) recrystallized sample.

were solved. The interface's price was DM 7,800, *i.e.*, less than a third of the upgrading using parts of conventional new diffractometers.

The interface UDS2 lets the operator measure PF's easily, and if further customization is required, it can be reprogrammed; so for example, from a diffractogram it could look for the best peak positions; certain parts of the pole figure could also be measured at different speeds, etc.

Two more applications could also be mentioned. Because a single crystal, except for very special cases, is actually a highly textured polycrystal, this system could also be very useful for single crystal applications, such as orientation, quality control, measurements of integrated intensities, and so on. Also, because all the freedom degrees can be mechanically



decoupled in the texture goniometer, using only the  $\alpha$  tilt, residual stress measurements could also be performed.

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