AUXILIARY PROGRAMS FOR DIFFRACTION EXPERIMENTS

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Abstract

The target of this paper was to write and describe auxiliary programs for visualizing results of diffraction experiments or important information such as penetration depth. Three programs were created: *debye* program for a visualization of the so called back reflection experiment, *absorption coefficient* program for necessity to know the values of linear absorption/attenuation coefficients and *X-ray penetration depth* program for being informed about the irradiated volume of the material. Results of these programs are often included in papers and, moreover, are available in free version on website.

1. Introduction

Because of recurring pleas of colleagues who are frequent users of X-ray diffraction results and who wanted to know the relevant volume of material which was irradiated, programs for calculation of penetration depth and linear absorption coefficients of crystalline phases were written in MatLab environment. In addition to it, we were often faced with oddly positioned Debye rings obtained in back reflection experiment. Debye rings' convenient location on area, 2D, detector was, thus, achieved by repeatedly performing the experiments. That had led us to create a program which would visualize the outcome for selected experimental parameters.

2. Theory/experiment

Debye program was created as a visual aid for users doing the so called the back reflection experiment (or backscattering Debye-Scherrer experiment) [1]. It is a basic method for qualitative determination of real structure of materials. In materials only planes, which are oriented in the manner satisfying the Braggs' law, can diffract. The diffracted radiation has a character of diffraction cones with the apex angle of 4 . If this diffracted radiation impacts on a 2D detector, the so called Debye rings are detected. The user can make a model of the experiment upon changing the experimental parameters and also can check an expected result for untextured polycrystalline fine-grained materials. This is especially helpful for setting the right distance between the irradiated sample and 2D detector when the dimension of the detector is known. There is a main equation (1) representing calculation of a Debye rings radii l:

$$\tan(180 \quad 2 \ ) \quad \frac{l}{2D}, \tag{1}$$

where 2 is the diffraction angle of the given crystallographic plane and D is distance between the irradiated sample and 2D detector.

Absorption coefficient program was created because it is de rigueur to know the values of linear absorption/attenuation coefficients when the penetration depth is to be calculated since it describes the reduction of an energy beam upon passing through a specific material [2]. For calculation of a linear absorption coefficient it is necessary to know the amount of each phase in material, densities and chemical formulas of the phases, see equation (2)

$$— \quad \underset{i}{\quad} w_i — \underset{i}{\quad}, \tag{2}$$

where w_i is proportional by molar mass of each element in the given phase, is linear absorption coefficient, is density, or is mass absorption coefficient.

X-ray penetration depth program was created because the users of XRD results must be informed about the volume of the material to which the results are relevant. Irradiated volume is given by irradiated surface, defined by experimental set-up and the inserted slits, multiplied by penetration depth. Most commonly, the penetration depth is represented by values of the so called effective penetration depth T^{ef} which defines the thickness of surface layer that gives rise to 63 % of diffracted intensity [1]. Hence, this program provides the opportunity to furnish users with T^{ef} values, or courses, for the given radiation penetrating the irradiated material. Final values generally depend on an incidence, a rebound angle and on an absorption coefficient. The user can choose between four types of experimental set-up. Except for standard Bragg-Brentano geometry (BB), see equation (3a), and grazing incidence diffraction (GID), see equation (3b), courses of T^{ef} during residual-stress-aimed diffraction experiment for diffractometer (RS diffraction experiment) can be visualized for all measured tilts and T^{ef} for -scans, see equation (3c), can be plotted as well [3]. User interface of this program is in Fig. 3 on the left.

$$T^{ef} \quad \frac{\sin}{2}, \tag{3a}$$

$$T^{ef} \quad \frac{\sin \quad \sin(2 \quad)}{[\sin \quad \sin(2 \quad)]}, \tag{3b}$$

$$T^{ef} \quad \frac{\sin^2 \quad \sin^2}{2 \ \sin \ \sqrt{1 \quad \sin^2}}, \tag{3c}$$

where is diffraction angle, represents incidence angle in GID and is the inclination angle of the material surface normal with respect to the diffraction vector.

3. Results/discussion

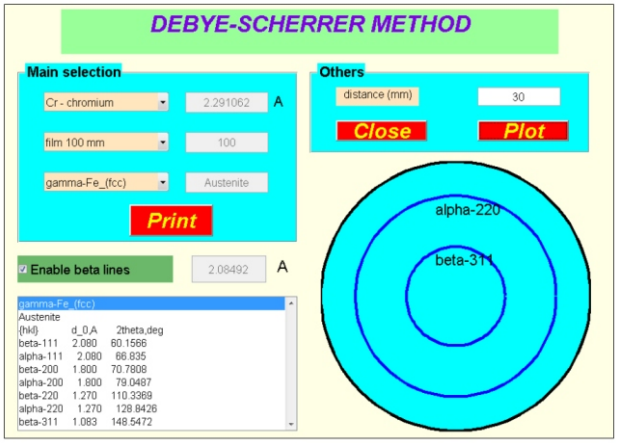

Figure 1. User interface of the *debye* program.

It is important to know the phase composition of the investigated material and it is necessary to have the determined phases in the database provided to the user as a part of *debye* program, see Fig. 1. The next step is to choose the used radiation (or input the wavelength), the size of the 2D detector and the sample-detector distance. Upon pressing "Plot" it is possible to check the expected diffraction pattern on the detector. By changing the above mentioned parameters, it is possible to adjust the experimental parameters in order to obtain the diffraction pattern in agreement with the intentions of the user without the need of performing any experiment. In the default setting, there is only the function to display alpha-lines (i.e. -{hkl}), but it is also possible to allow displaying beta-lines (i.e. -{hkl}) too. Rings are described accordingly (alpha-hkl or beta-hkl). If the user presses the button "Print", a list with information about the material will be displayed, i.e. name, chemical formula and all {hkl} planes, which diffract for the used radiation with 2 diffraction angles between 0° and 180°, with interplanar distances and diffraction angles. In Fig. 1, there is the result for austenite steel in the distance of 30mm from the detector with detector 100mm in diameter when radiation from X-ray tube with chromium anode is used.

Figure 2. User interface of the *absorption coefficient* program.

Table 1. Linear absorption coefficients for steel and aluminium oxide.

, cm-1	CrK	CrK	CoK	CuK	MoK
Fe	890	682	442	2393	297
Al_2O_3	407	310	198	128	13

Absorption coefficient program, see Fig. 2, offers the choice between X-ray radiations with different wavelengths. Next, the program requires entering phases and the amount and density of each phase in material. Pressing "Calculate" the program starts to calculate the particular linear absorption coefficient of each phase according to equation (2). At the end, the final result of linear absorption coefficient is calculated using a weighted average where the weight is the amount of each phase. In Fig. 2, there is the user interface of the program with the result – steel with oxidation layer (again for CrK radiation). This constant is subsequently applied in the calculation of corresponding penetration depth as can be seen in Fig. 3. There are few selected linear absorption coefficients in Tab. 1.

After starting the *X-ray penetration depth* program, see Fig. 3, the user chooses type of the result, e.g. for which geometry is necessary to know the value of the penetration depth, concrete in BB, GID, RS diffraction experiment or for -scan. This choice, windows stay visible which conclude required parameters for the displaying graph: e.g. the range of measuring but mainly the value of the absorption coefficient. It is possible to choose between the linear absorption coefficient or the mass absorption coefficient and the density of the material. Pressing "Execute" the corresponding graph is displayed or the program notifies of inconsistencies. User interface of this program is in Fig. 3 on the left while on the right; there are the results of T^{ef} courses for linear absorption coefficient calculated above and for each option of type of result.

There is approximate penetration depth for selected radiations into steel in Tab. 2. The values of penetration depth for GID are with using incidence angle 2°. The value of inclination is 0.2 in this case. Generally, it is obvious from Fig. 3d that measuring with different inclinations results different penetration depths.

Table 2. Approximate effective penetration depth selected radiation into steel**.**

T^{ef}, m	CrK	CrK	CoK	CuK	MoK
BB	4	6	9	2	13
GID	0.4	0.5	0.8	0.1	1
RS and -scan	5	6	10	2	15

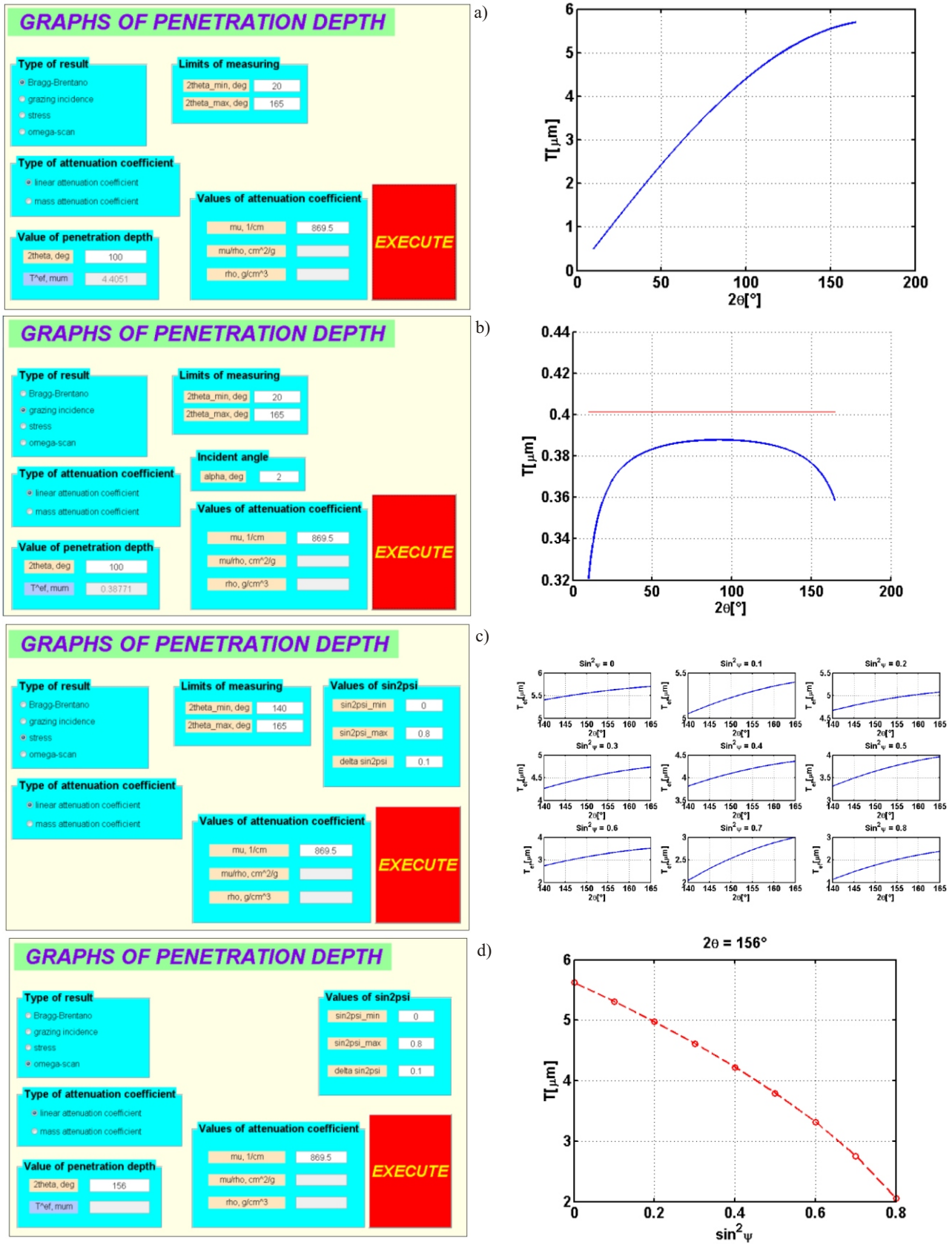

Figure 3. **a.** Options of the *X-ray penetration depth* program and result for BB. **b.** Options of the *X-ray penetration depth* program and result for GID. **c.** Options of the *X-ray penetration depth* program and result for RS diffraction experiment. **d.** Options of the *X-ray penetration depth* program and result for -scan.

4. Conclusions

The mentioned programs were created as auxiliary programs for diffraction experiments, in more detail, for visualizing results of diffraction experiments.

Debye program is used mainly for obtaining suitable experimental parameters in back reflection experiment. The outcome of program is a picture with Debye rings for untextured polycrystalline fine-grained materials. There is some basic information about this program:

- The program was used for identification diffraction plane in both untextured and textured, and in both small-grained and big-grained materials.
- It is the auxiliary program for checking diffraction angles too.
- In Fig. 1, there is result for austenite steel. This is one of options to check qualitatively grain-size and texture of planes {311} in austenite steel when radiation from X-ray tube with chromium anode is used.

The programs, *X-ray penetration depth* and *absorption coefficient*, are often used in practice. Resulting graphs or values of penetration depth are usually included in research papers for getting information of the relevant volume of material which was irradiated.

- In Fig. 2, there is obvious that linear absorption coefficient of steel is different compared with steel with oxidation layer.
- The well-known conclusion follows from Tab. 1 and 2; the values of linear absorption coefficient and penetration depth, respectively, depend on material and used wavelength of radiation.
- It is obvious that GID is suitable for X-ray diffraction analysis of thin films as is shown in Tab. 2. These values are smaller when smaller incidence angle is used.
- The value of penetration depth is important to known if residual stresses are determined. If there is stress gradient in the material, it is a huge difference in using CrK and MoK radiation. The exemplary example should be a honing (metalworking) material. There is presented stress gradient in 4 m surface layer [1].
- It is possible to detect 1 m thin films in Bragg-Brentano geometry using CuK radiation due to small penetration depth into steel in comparison with MoK radiation.

The programs are available for users who have at their disposal MatLab from http://electron.fjfi.cvut.cz/drupal/program-laborator-strukturni-rentgenografie.

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