Brief Guide to using "TexturePlus.exe" (12/21/99)

A. DATA REQUIREMENTS FOR TEXTURE MEASUREMENT

To measure texture using a diffraction peak with Bragg angle θ_B, the program requires:

1. A θ-2θ scan of the Bragg peak.
2. A θ scan (a.k.a. rocking curve or ω scan) using this peak.
3. The diffractometer scattering angle used for the ω scan - $2\theta_B$.
4. The divergence angle of the incident beam for fixed slits, or the illuminated area for fixed area.
5. The radius of the diffractometer.
6. The length of the specimen in the diffraction plane (if <~ 20 mm).
7. IF specimen is a thin film, the thickness and x-ray absorption coefficient are needed.

(Below, input and output data are underlined; software interface items – buttons etc - are **bold**)

B. DATA COLLECTION

1. Ensure there are no anti-scatter slits in place on the diffractometer. If there are anti-scatter slits and they cannot be removed, open the slits as far as possible.

2. Specimen mounting: orientation and rotation
   There are two main considerations: 1) Will any part of the x-ray footprint miss the specimen at any point during either scan? 2) Are deviations from texture axisymmetry being investigated? For fixed slits, the footprint is smaller for positive ω (tilt towards source) and these data maybe more reliable. If the specimen is smaller than the largest footprint, mount it with a mirror plane (e.g. center line of rectangle) in the diffraction plane, measure the length of specimen in the diffraction plane and do not rotate (spin) the specimen. In general, rotating the specimen is unwise – you lose information on texture asymmetry which can be investigated by analyzing the specimen at different . Specimen length is only important for low angles of incidence where part of beam may miss specimen. Note that the specimen height should be accurately fixed in the $\theta = 0$ plane.

3. Collect θ-2θ scan over Bragg peak of interest, going about 2° either side of the peak. Criterion is to scan into the flat background region on both sides of the peak.

4. Measure mid point of peak ($2\theta_B$). Cursor on screen plot is accurate enough method.

5. Set diffractometer scattering angle to $2\theta_B$ and collect ω scan. The maximum useful ω range is about two degrees less than $2\theta_B°$. Highly textured specimens will frequently not require such a large range. Making $2\theta_B$ the center of the scan range has advantages. For example, if $2\theta_B$ = 29.8°, a good scan would be from 4.9° to 24.9°. The scan would then be over 20° with ω = 0° as the midpoint.

6. For both the ω scan and the θ-2θ scan, use the same beam divergence (fixed slits) or illuminated area (fixed area) and the same receiving slit.

7. Find out what diffractometer radius is.

8. If specimen is a thin film, determine film thickness and x-ray absorption length of material.

C. DATA FILES

1. Convert both θ-2θ scan and θ scan files to ascii format with each line containing 2θ or θ and intensity. The numbers must be separated by at least one space. Other non-numeric characters (e.g. commas) may be placed between the two numbers, but do not use tab delimited data; in fact it is best if your files don't contain tabs. If you use the default extension .xyp for the ascii files your life will be easier. You can ignore all the default file naming conventions in the software if you want to. All Win95 legal filenames are acceptable.

2. As a convenience, if the root part of your peak file name, 'root.ext', ends in a number, e.g. mdv371.xyp, then the default ω scan filename will have the same starting letters and the number will be 1 greater, i.e. mdv372.xyp.

3. Comment lines starting "c " can be included at the beginning of the .xyp file. These comment lines will print to the text output window during execution of TexturePlus.exe. They can include specimen information, and also scan information such as the incident beam divergence (I), receiving slit divergence (R) and the two theta value (2theta) for the theta scan. The format for this information is: I and then the next numeric characters will be interpreted as the value; e.g. I.5 and I: .5 would both give the incident divergence as 0.5°, and 2theta = 29.8 would give $2\theta_B$ as 29.8°. They can be(and usually are) strung together on one line together with other information, e.g. "Specimen name I.5 R.2 2theta=29.8 no rotation". (The quotation marks are not needed.).

4. The program picks the values of I, R, 2theta, etc, out of the data file, which saves you having to enter them and makes bookkeeping easier. In many XRD systems, the diffractometer software puts the specimen identification text that the user enters when setting up the x-ray scan into the ascii data file as a comment line, which makes the whole process easier to document. It does not matter if the I and/or 2theta values are not in the comments; they can be entered at program run time.

5. It helps to put your data files in a well-organized directory structure. The output files can be put in the same or a different directory from the input data files. There are three output files: for example if the theta scan data file is 'thetascan.xyp', the extension .xyp is removed leaving the 'root' of the file name, 'thetascan', and then 'thetascan.crc', 'thetascan.ovl' and 'thetascan.inf' (see below for file definitions) become the default output filenames; these of course can be changed (see OUTPUT part of window), but all output files go to the same directory.

6. The output files are:
   *.crc - the corrected ω scan curve - typically 2 to 3 times the size of the ω scan file.
   *.ovl - the overlap file - about the same size as the ω scan file – see below.
   *.inf - the information file, lots of useful (!?!) extra information - < 1.5 KB

7. To obtain the overlap file, the ω values at the half maximum intensity either side of the peak in the corrected ω scan are determined. Their separation is the full width at half maximum intensity (FWHM) of the corrected ω scan and their average is the center of the texture distribution, which should be close to $\alpha = 0$, but may deviate slightly. The crc is split in two halves, above and below the distribution center, and the below half is "overlapped" or folded onto the above and the mean of these two is calculated. The .ovl file contains the mean**,** above and below curves.

D. DATA ANALYSIS

1. The first time you run the program "TexturePlus.exe" you will be prompted to enter:
   1. Input Directory (you can browse by double clicking the box)
   2. Peak File (there is a browse button)
   3. ω scan File (there is a browse button)
   4. Diffractometer Radius (enter a commonly used value – it can always be changed)
   5. Specimen Length (see B.2; 20 mm is good default value)
   6. Beam Divergence (enter a commonly used value – it can always be changed)
   7. Output Directory (you can browse by double clicking the box)

2. Note the following color conventions:
   - data entry boxes have a white background;
   - command boxes/buttons are bright blue (and some go dim blue once executed);
   - option selection boxes are red;
   - box labels are shades of green or yellow.

3. Select the type of calculation you are planning to carry out: **Omega scan, Chi scan, Offset peak, or Simulation** (red border)**.** The commonest type is **Omega scan** and will be treated first.

4. The first time you run the program you will not be allowed to read any data files until you have hit the **Store Defaults** button (bottom left) to create the file c:\Windows\TexturePlus.ini. You may hit this button at any time, and you will also be prompted at exit to see if you want to store defaults.

5. When entering filenames, if you give no extension, input files will have the extension .xyp added and the extensions in C.5 (above) will be automatically appended to output files. To enter a filename without an extension, put a period at the end of the filename. Until there is a valid entry in any box, the box text will be red (not black). The **Bragg peak file name** box and the **Rocking curve file name** box remain red until the file is read.

6. Once the **Read Peak File** and **Read Omega File** buttons appear, hit **Read Peak File**. The data file will be read and the low intensity part of the peak profile will be displayed in the **Peak File** window so that a background line can be determined; a suggested background line is displayed in red. A text box with instructions on background selection will appear and also a box giving the 2θ and intensity coordinates of the cursor when it is on the plot window. The background line can be selected by clicking on 2 points. The 2θ range of the peak file to be used in the texture calculation will also be selected by these 2 points; this allows a single peak to be selected from a peak file which contains multiple peaks. Alternatively, the red line can be accepted as the background by right clicking anywhere on the plot window (and the whole peak will be used). Once the background is defined the selected peak range with background subtracted is displayed.

7. Hit **Read Rock File** to read the rocking curve data file. One or more background data files may be collected. This is important for thin films with wide texture profiles. (If no background data files are collected, profiles are calculated and they are accurate for bulk specimens[2].) The convention for the background file names is to add L or R to the root of the ω scan file name. Thus ω scan mdv372.xyp could have background files mdv372L.xyp and/or mdv372R.xyp. If files fitting this convention are found, you will be prompted to see if you want to use them. (Try comparing the results with and without the background files.) Examination of the θ-2θ scans shows where the background can be measured without interference. Typically the scattering angle is set at either 2° below (Left) or 2° above (Right) the ω scan value.

8. The following protocol is used to establish the **Rocking curve two theta** value; up to 3 values are used: 1) the "2theta" value in the data file (see C.2); 2) the middle of the ω scan range (see B.5); 3) the 2θ value at the peak maximum. If your data file does not contain value 1, you will be asked to enter the value (with the default set as value 2). If this value differs from value 3 by more than 0.05% you will be prompted again to make sure.

9. Select whether the specimen is **Bulk** or **Thin Film**. If it is thin film you must enter **Film Thickness** and the x-ray **Abs. Coeff.** of the film material.

10. Select whether the data were collected under **Fixed Slit** or **Fixed Area** conditions and enter the **Beam Divergence** or **Illuminated Area** as appropriate.

11. The **Correct Texture Scan** box will appear once data have been read in and all the necessary values have been set. You can change the number of points for **Smoothing** the output data. Then click on **Correct Texture Scan** and the raw (blue) and corrected rocking curve (red) curves (destined for the .crc file) will be calculated and displayed in the **Rocking Curve Plot** window [2, 3]. For comparison, the previous corrected rocking curve is displayed (green). If the

rocking curve data are particularly noisy, it may not be possible to calculate the FWHM of the corrected rocking curve as described in C.6. You will be informed if this is the case; one option is to increase the smoothing number – another is to inspect your input data and remove egregious spikes. Before the three curves destined for the .ovl file are calculated (see C.7), you will be prompted for the center α value (with the value calculate as in C.7 as default). If you are happy with the results and also with the target OUTPUT filenames, click on **Store Results** to save the .crc, .ovl and .inf files.

12. Tools are provided for further analysis of the corrected omega scan to measure volume fractions of textured material [4]. The **integrate texture** button calculates $P(\omega) \sin \omega$ over an ω range selected by the user (0 to ω_o) and puts the results in a text box. The numbers are: the raw integral, the integral after removal of the random component and the intensity at ω_0 due to the random component. These values can be written to a log file (c:\Windows\TexturePlus.log) by clicking on **File Data**; they are in labeled columns in a convenient format for further analysis. To delete the log file, right click on **File Data**; you will be prompted for confirmation.

E. OTHER FEATURES

1. If you select **Simulation** as your calculation type, a box requiring **M-D r value** appears. This is the r parameter of the March-Dollase function which is a single parameter texture profile[1]. Untextured material has an r value of 1 and fiber textured material has $r < 1$. Once the Bragg peak has been read in (D.6) and the **Diffractometer Radius, Specimen Length**, **Rocking curve two theta** and **Beam Divergence** have been set, click on **Simulate Rocking Curve**. Once the simulated curve has been calculated and displayed, one possibility is to select option **Rocking Curve** and correct the curve as in D.9. Simulation can be very instructive.

2. If you select **Offset peak** as your calculation type, you read in a Bragg peak (D.6), enter **Theta Offset** and **Texture Factor**, and click **Calculate Offset Peak.** The program calculates what the Bragg peak would look like if the specimen had a θ offset and multiplicative texture factor as entered. To compare with an experimentally determined θ offset scan, read the data file using **Read Comp File** before clicking **Calculate Offset Peak**.

3. Once a Bragg peak file has been read in and the background has been determined, a text box with instructions will appear. Clicking on the plot at a particular intensity will zoom the plot so that intensity is full scale. Double clicking anywhere on the plot window will unzoom to the full intensity range of the peak. To change your background line, right click anywhere on the plot window and the low intensity part of the profile is displayed as before.

4. The symmetry of the Bragg peak can be investigate using the **peak symmetry** button. The peak is split and the two halves are plotted, together with the asymmetric component (the difference between the two halves). The user can manipulate this plot by left, right and shift-left clicking as described in the instruction text box. Right click allows entry of the user's choice of the peak center. Initially, the peak is split at the angle which makes the integral of the asymmetric peak equal zero.

5. A text box on each plot window displays the angle and intensity coordinates of the cursor while it is in the window.

6. A number of parameters describing the background-subtracted peak are calculated and displayed in a textbox in the upper right corner of the **Peak Plot** window (under the angle-intensity box). The values can be written to the log file (c:\Windows\TexturePlus.log) by clicking on **File Data**.

F. EXAMPLES

1. Data files were installed with the software package to demonstrate the use of the software. There are three examples, two from bulk alumina (one random - SRM676 - and one textured) and one from a textured PZT thin film.

2. Load "untextured peak.xyp" and define or accept the background. Load "untextured rocking curve", and note how the rocking curve two theta value is read in from the data file. Set the diffractometer radius to 200 mm and the beam divergence to 0.68°. Click **Correct Texture Scan**; the corrected scan will be of constant intensity, as expected. Increasing the **Smoothing:** number reduces the noise level.

3. Load "textured peak" and "textured rocking curve" as above. When **Correct Texture Scan** is clicked, the corrected scan will be very similar to the raw data. The user is prompted for the midpoint of the texture profile to make the **overlap curve** (see C.7). You can perform the integral (see D.12) by clicking **Integrate texture**; by setting the option next to **Integrate texture** to **Auto** the intergral will automatically be performed after the **overlap curve** is plotted.

4. Click **Thin Film** option and load "Thin film peak.xyp". When "Thin film rocking curve.xyp" is loaded, the software will detect a background file "Thin film rocking curveL.xyp" and ask you if you want to use it; click **OK** the first time. Set the film thickness (200 nm) and the absorption coefficient (0.135 μm^{-1} = 1350 cm^{-1}) and click **Correct Texture Scan**. To see the effect of using the background file, reload "Thin film rocking curve.xyp", reject the background file and click **Correct Texture Scan**. There are noticeable differences at low intensity; the measured background result does not have upturns at $|\omega| > 8°$ (more marked for positive omega). To see the effect of the thin film correction, click on the **Bulk** option and on **Correct Texture Scan**. There are small differences particularly at larger $|\omega|$ values.

G. FEEDBACK / COMMENTS / COMPLAINTS

1. This is a work in progress – if there is a feature you would like, contact me (I may already be working on it).

2. So, please address all of the above to Mark Vaudin

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References

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