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LaboTex

Version 2.1

The Texture Analysis Software for Windows

"Device-independent" pole figures for quantitative texture analysis. Texture Standards.

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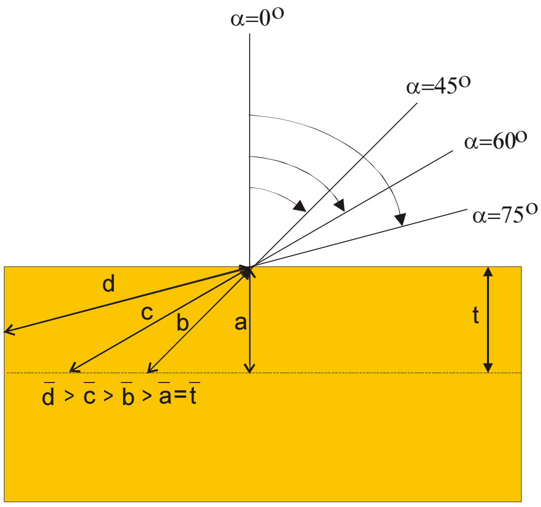
TEXTURE STANDARDS

"Device-independent" pole figures for quantitative texture analysis.

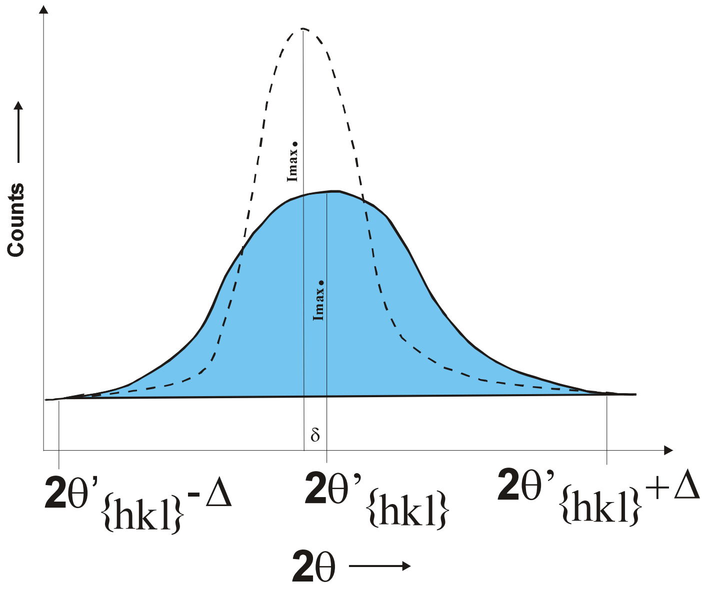
The **P**eak **I**ntensity **M**ethod (**PIM**) and the **I**ntegrating **M**ethod (**IM**) - basic information

When the radial angle (α) increases, the way of beam in sample for the same thickness is longer (see for Figure A below) hence different effects are observed: greater absorption, the peaks in the diffraction pattern are broadened, maximum of peaks can be shifted from 2θ (see for Figure B below). There are also other geometrical and physical apparatus problems connected with shift of radial angle (beam divergence, spectral width, source width, slit width). Instrumental aberrations are technique dependant. Microdistortions, microstrains, size effects of crystallites and other phases can be also present in sample. Details you can find in many monographs (for example H.P.Klug, L.E.Alexander :"X-Ray Diffraction Procedures" John Willey & Sons, 1974). These effects (defocusing, absorption) are generally called as the "defocusing effect".

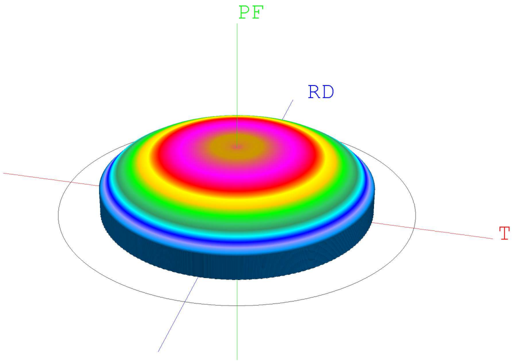
A)

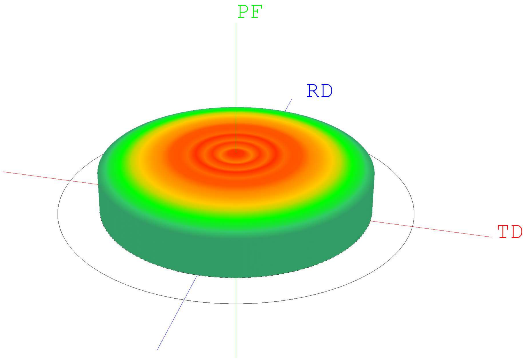


B)



You can directly observe the defocusing error on the 'random' samples (texture free samples, samples with no preferred orientation) for which all pole figures values should be the same. The pictures below show the examples of experimental pole figures {110}(left) and {211} (right) for 'random' sample made with powder of Ferritic Stainless Steel (reflection XRD technique, axial symmetrization):





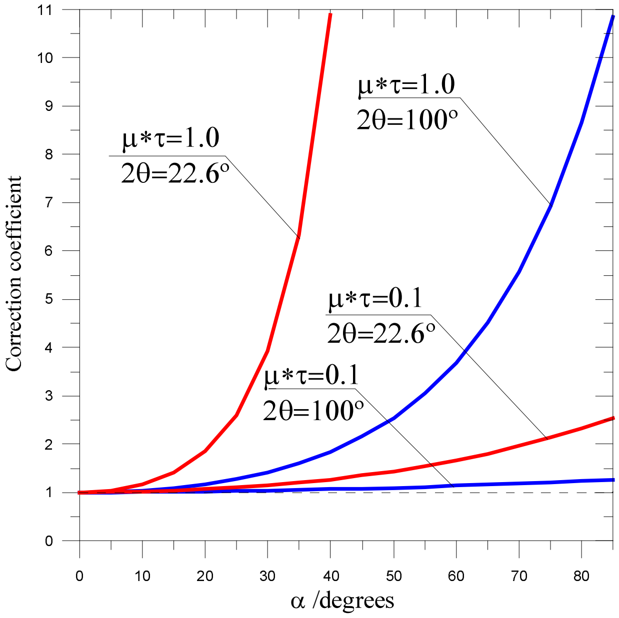
As you can see, the defocusing effect is greater for the {110} pole figure than for the {211} pole figure. Schulz formula (J.Appl. Phys., 20, 1033, 1949) for reflection technique may to some extent explain these effects:

$$\frac{I_{\alpha=0}}{I_\alpha}=\frac{1-\exp\left(-\frac{2\mu t}{\sin\theta}\right)}{1-\exp\left(-\frac{2\mu t}{\sin\theta\cdot\sin(90-\alpha)}\right)}$$

where,

μ - absorption coefficient [1/cm], t - penetration depth [cm]

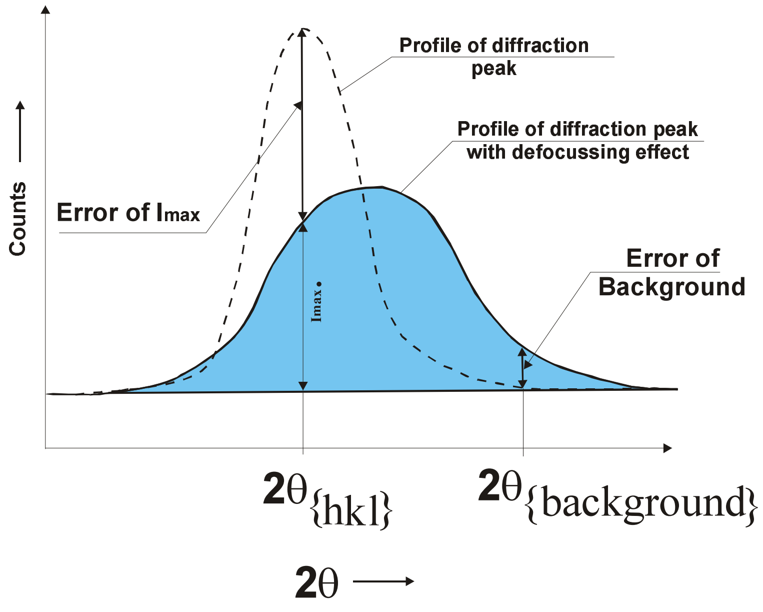
Figure below shows changes of correction coefficients for defocusing calculated from Schulz formula for different parameters 2θ and μ·t



Correction coefficients for defocusing calculated from Schulz formula for different parameters 2θ and μ·t

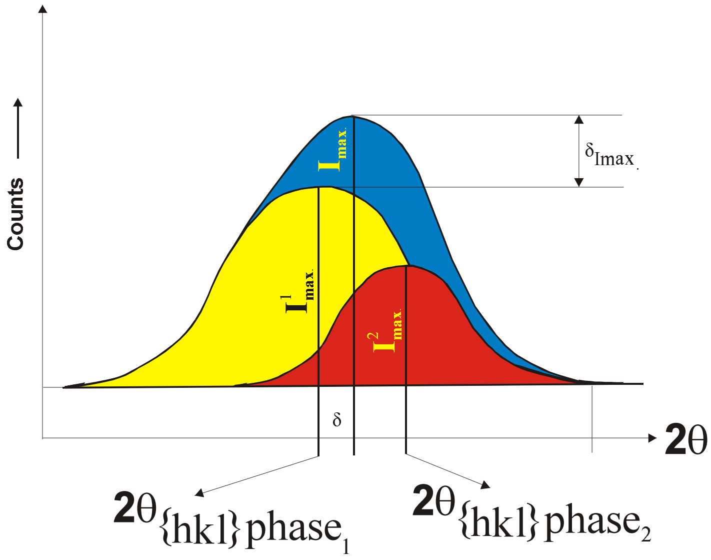
As you can see in the figure above, defocusing effect is greater for pole figures with lower 2θ, hence pole figure {110} ($2\theta=52.3^o$) is more deformed than pole figure {211} ($2\theta=99.5^o$)
Schulz formula is only approximation for real correction coefficients. For proper correction 'random' sample should be used. You can see in the figure above strong effect of absorption coefficient on the defocusing correction. Hence, it is very important for good correction use 'random' (powder) sample made with the same material (with the same absorption coefficient) as material of investigated sample.

Result of defocusing effect: shift and lowered of maximum, broadness of the diffraction peaks in 2θ when radial angle increase, can also conduct to bad correction of background (see figure below). In LaboSoft we often receive from our customers the pole figure data where background values are greater than values of diffraction peaks. LaboTex (from version 2.1.015E) informs user's when it finds data for which background data are greater than pole figure data. LaboTex also display percent of these data.



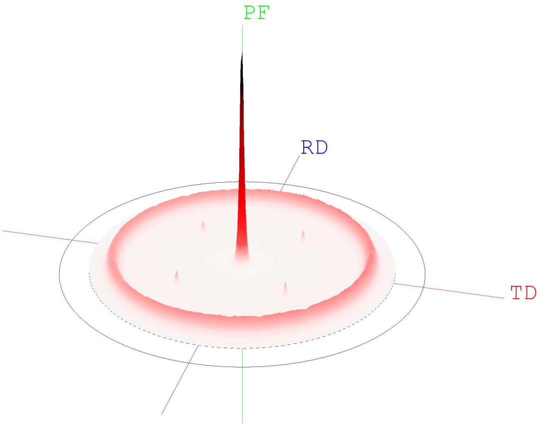
Defocusing effect: Errors in determination of parameters of diffraction peak.

There can be also errors introduced by presence of other phase(s) in massive samples or from substrate in case of the tin layers, as it is shown in figure below:



The observed (resultant) profile of diffraction peak (blue) in multiphase system where peaks are overlapped (profile of diffraction peak for phase 1 (yellow), profile of diffraction peak for phase 2 (red)).

In some case the presence of other phase can be easily observed on the pole figure as on figure below. Peak for plane (220) Si ($2\theta=47.37^o$) interference with peak for plane (111) Cu ($2\theta=43.34^o$). Pole figure shown below contains additionally four small peaks. These peaks come from the monocrystal substrate.



For the above reasons determination of profile of diffraction peak is important condition if we want to receive a "device-independent" pole figures for quantitative texture analysis.

The **P**eak **I**ntensity **M**ethod (**PIM**)

The following procedure is used for elimination of errors in the peak intensity method (**PIM**):

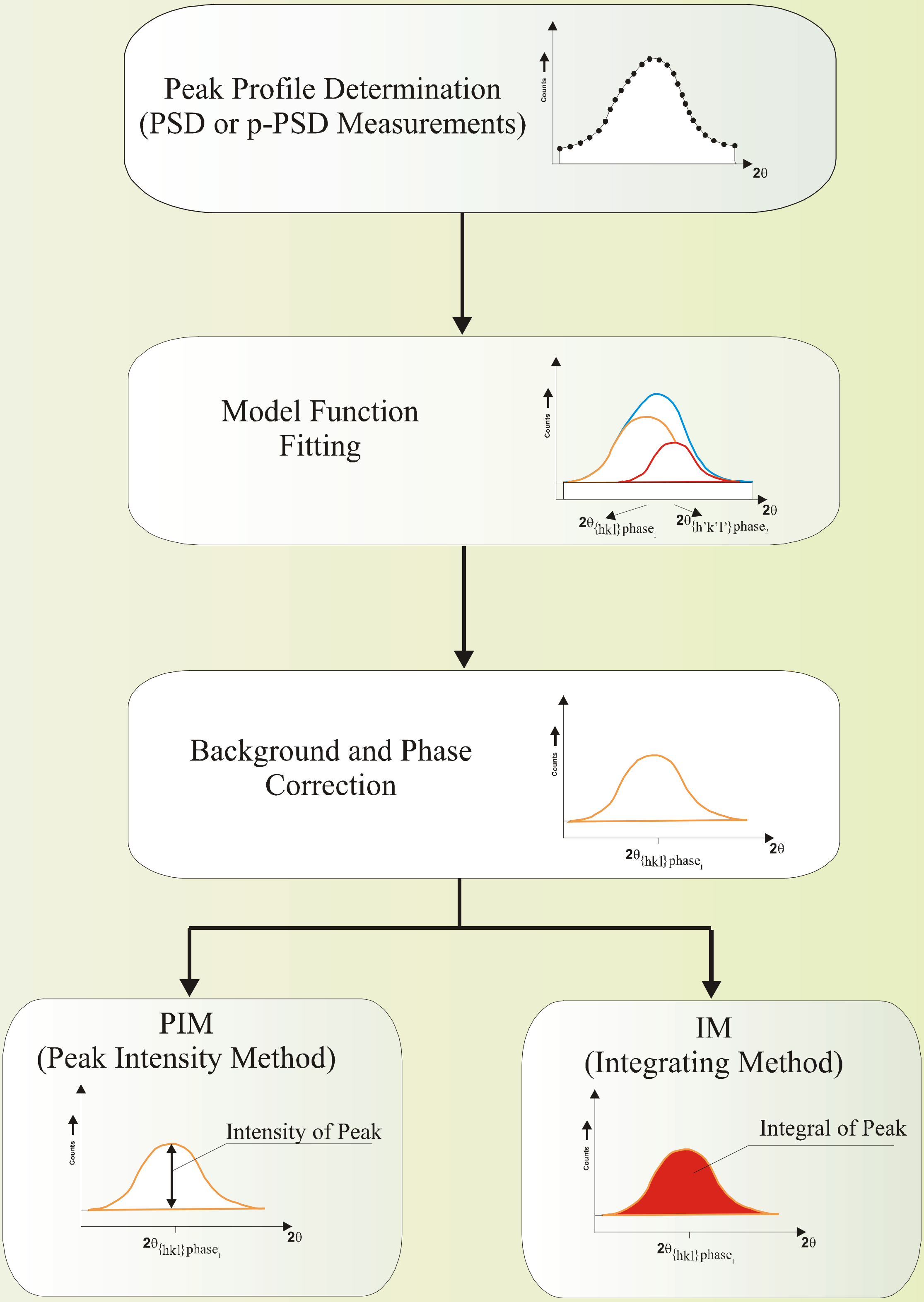
1. The profile of diffraction peak are determined using a pseudo Position-Sensitive Detection (pPSD) mode (of course, you can use full PSD(CPS) detector). The pPSD consists in registering the intensity of the diffracted beam in the chosen angular range, using a scanning θ-2θ or ω-2θ mode. As a result of such process a diffraction profile is obtained.
2. The optimal curve of a theoretical distribution according to: Gauss, Lorentz (Couchy), Modified Lorentz or Marlos & Thomas is fitted to each of the profile and next, if it is needs the deconvolution of diffraction profile on the component diffraction peaks is made. Background correction is made during fitting of profile.
3. Maximal value of peak (Peak Intensity) is determinated.

The **PIM** method eliminates errors connected with decrease of maximal intensity of diffraction peak in consequence of shift of the maximum of diffraction peak in 2θ when radial angle (α) is changed and measurements are made for the strictly the same value of 2θ. The **PIM** method makes also proper correction of background. The peak intensity method (**PIM**) represent the registration mode by means of a "point" counter for well justified goniometer.

The **I**ntegrating **M**ethod (**IM**)

Similar procedure is used for elimination of errors in the integrating method (**IM**). Difference between both methods are in point 3:

1. The profile of diffraction peak are determined using a pseudo Position-Sensitive Detection (pPSD) mode. The pPSD consists in registering the intensity of the diffracted beam in the chosen angular range, using a scanning θ-2θ or ω-2θ mode. As a result of such process a diffraction profile is obtained.
2. The optimal curve of a theoretical distribution according to: Gauss, Lorentz (Couchy), Modified Lorentz or Marlos & Thomas is fitted to each of the profile and then if it is needs the deconvolution of diffraction peak profile on the component diffraction peaks is made. This procedure eliminates also the effect of presence of other phases. Background correction is made during fitting of profile.
3. The integrated intensity of peak is determinate by integration of peak profile obtain in point 2. The integrated intensity of peak is a value of the experimental pole figure in a determined point: α,β.



Peak Profile Determination
(PSD or p-PSD Measurements)

Model Function
Fitting

Background and Phase
Correction

PIM
(Peak Intensity Method)

IM
(Integrating Method)

Determination of point (α,β) of pole figure for $phase_1$ by PIM and IM (Scheme)

The IM method eliminates errors connected with:

- decrease of maximal intensity of diffraction peak in consequence of shift of the maximum of diffraction peak in 2θ when radial angle (α) is changed and measurements are made for the strictly the same value of 2θ;
- decrease of maximal intensity of diffraction peak as results of broadening of peak profile when the radial angle increase.

The IM method makes also proper correction of background. It should be noticed that the **IM** is more precise texture measurement technique then the **PIM**. The DAMfit (Bonarski, 1999) computer program has been used to initial processing experimental data by both methods. Finally, the DAMfit program prepares input data for the LaboTex computer package in EPF and POW formats.

The final correction of pole figure data is made in LaboTex using pole figure for isotropic sample (texture-free powder sample) made from the material with the same composition as main sample.

Warning: CD attached to standard samples contains 2 different files with powder data for correction:

1. Powder data for correction evaluated by **PIM**;
2. Powder data for correction evaluated by **IM**.

You should use the same type of data when you input data to LaboTex . For example: for pole figures of standards sample evaluated by **PIM** (in EPF format) you should use powder data files (in POW format) evaluated by **PIM**.

If you would like examine quality of powder sample you should make correction of powder sample in EPF format by powder sample in POW LaboTex format (you input both samples simultaneously to LaboTex: first form list for pole figure data, second from list for defocussing data).

The delivered reference samples and the attached "device-independent" results allow evaluating the correctness of the procedure of texture analysis applied in your laboratory.

Basic information about transformation of the integrated intensities from diffractometer coordinate system {2θ, ω,γ,χ,φ) into the pole figure angles (α,β) and about measurements with Area Detectors and with Position Sensitive Detectors you can find in:

- H.J.Bunge, K.Klein, Z.Metallkunde, 6, 465 (1996);
- L.Wcislak, H.J.Bunge, "Texture Analysis with Position Sensitive Detector", Cuvillier Verlag, Gottingen (1996).