

PASAD-tools, a software for quantitative analysis of SAD patterns

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When using non-homogeneous nanocrystalline materials it is necessary to get local quantitative information on the microstructural parameters (e.g.: grain size, domain size and order parameter). Up till now quantitative integral information can be gained by standard X-ray diffraction profile analysis (XPA) methods whereas quantitative local profile analysis of selected features was not readily accessible. To achieve this, selected area diffraction (SAD) patterns of areas of interest are recorded and a profile analysis of the SAD patterns (PASAD) is carried out [1,2,3].

PASAD is performed in three steps: (i) A SAD pattern is recorded. (ii) An intensity profile (intensity versus diffraction vector \mathbf{g}) can be achieved from the SAD pattern by azimuthal integration. (iii) The microstructural parameters are determined by profile analysis in an analogous way as done in XPA. To record the diffraction pattern, care has to be taken that the illumination is parallel as a convergent illumination would lead to additional peak broadening. It is also important that the recording device has a sufficient resolution. For a quantitative evaluation it is important that all intensities lie in the linear dynamic range of the recording device.

After having recorded a good diffraction pattern, the azimuthal integration is performed. For this step it is crucial to use the correct centre of integration, as any deviation has a huge impact on the resulting profile. When the centre of integration is the true centre of the SAD pattern, the resulting profile shows sharp peaks (cf. Fig. 1a), whereas a deviation from the correct centre of integration can cause artificial peak broadening and even double peaks as shown in Figure 1b. As the peak height reaches a maximum if the true centre of integration is used the software changes the centre of integration until a maximum peak height is reached (cf. Fig. 2). The software also corrects for elliptical distortions.

The resulting profile can now be used for a profile analysis. As unfiltered electron diffraction patterns show a significant background, the background has to be subtracted. After that the peak parameters of all the Bragg peaks can be obtained by fitting some peak function (such as a pseudo-Voigt function). These parameters can be used for a profile analysis in the same way as done in XPA. To ensure reproducibility and to make the routine automatic, it is important to determine the background and fit the peaks automatically using stable and precise routines (cf. Figure 3a). It is also possible to add and delete peaks or spline points manually using an interactive mode (cf. Figure 3b). It should be noted that the resulting peak-widths are a convolution of the instrumental broadening and the broadening due to the sample. Therefore it is important to deconvolute the peak-widths for the instrumental peak broadening.

The software was written as a DigitalMicrograph plugin and contains an intuitive graphical interface as shown in Figure 4. It is available for free from www.univie.ac.at/pasad.

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2. C. Gammer, C. Mangler, HP. Karnthaler, C. Rentenberger, Microsc. Micronal. in print.
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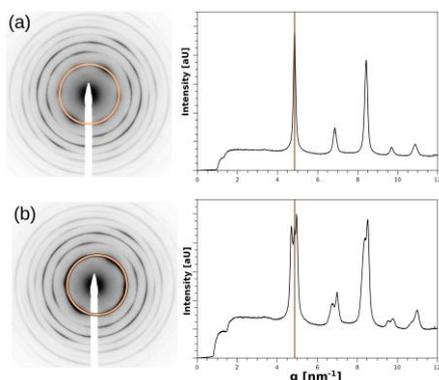


Figure 1. (a) When the centre of integration corresponds to the true centre, the azimuthal integral shows sharp peaks. One integration ring is indicated in the diffraction pattern. (b) If the centre of integration is slightly shifted, the resulting profile shows artificial peak broadening or even double peaks.

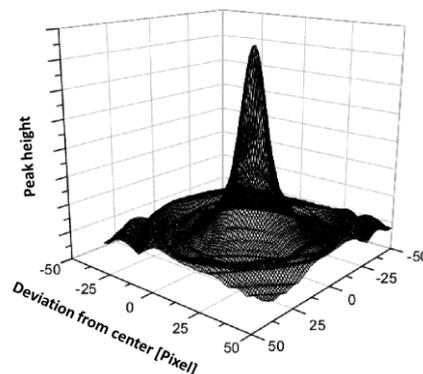


Figure 2. Plot showing the peak height observed in the azimuthal integral against the deviation of the centre of integration from the correct centre. As this curve has a sharp maximum it can be used to refine the centre of integration.

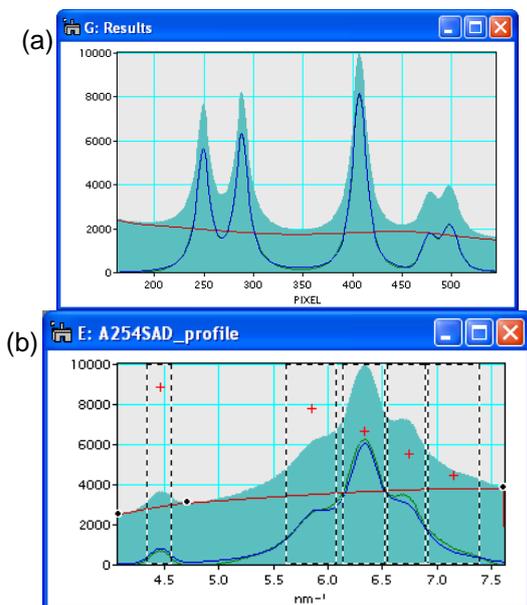


Figure 3. Peak fitting in PASAD-tools. (a) The software tries to find the peaks automatically. For the background a spline function is used. The minima in between the peaks are used as spline points while overlapping peaks are taken into consideration. (b) If the software does not manage to detect the peaks automatically, it is possible to add or remove peaks or spline points using the mouse.

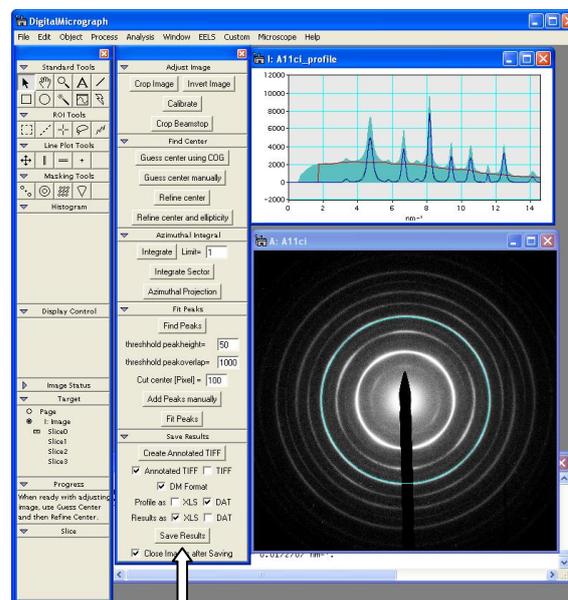


Figure 4. Screenshot of the graphical interface of PASAD-tools. The software is fully integrated into the Digital-Micrograph software. A window containing the available functions is displayed (cf. white arrow) when starting PASAD-tools. All functions can be used in an intuitive way using the mouse. Important help is displayed in the Progress or Results windows.