Quantitative analysis of Electron Diffraction Ring Patterns using the MAUD program

P. Boullay[1], L. Lutterotti[2] and D. Chateigner[1].

1. CRISMAT, CNRS UMR 6508, 6 Bd du Maréchal JUIN 14050 CAEN Cedex, France.
2. Department of Materials Engineering and Industrial Technologies, University of Trento, 38123 TRENTO, Italy.

philippe.boullay@ensicaen.fr
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The growing interest on nanosized polycrystalline samples raises problems such as phase identification, structure and microstructure characterization with quantitative and reliable approaches. Among the different techniques available in transmission electron microscopy, the quantitative analysis of electron diffraction ring patterns (ED-RP) appears as an interesting tool with computer program already existing (see [1-2] to cite a few). In the present work, the authors would like to present some new features of the MAUD program [3] targeted towards the quantitative analysis of ED-RP by exploiting methods already used in X-ray and neutron diffraction.

The data collection is performed using a parallel beam illumination from an area ranging from about 200µm^2 down to 1µm^2 provided the number of diffracting particules is sufficient to give a ED-RP. The diffraction patterns collected with a CCD are transformed in diffraction patterns using an ImageJ plugin that allows integrating azimuthally the intensity along the rings by sectors (caking in n patterns, each covering an angular range of 360/n°) as illustrated in Fig. 1a. In the case of ED-RP presenting no or weak texture effects, phase identification and volume fraction analysis can be performed using the Crystallographic Open Database [4]. Knowing the crystal structure of the phase present in the analyzed area, structural and microstructural information can be extracted using the Rietveld method (Fig.2). All these steps are implemented and accessible via the MAUD interface.

Once the camera length and instrumental profile function are known, quantitative information such as cell parameters, average size and shape of an assembly of nano-particules can be obtained from only one ED-RP. The results on few oxides will be presented and compared to similar X-ray diffraction experiments. Texture effects and a certain amount of graininess in the ring patterns can also be accounted for (Fig.3). In the case of texture analyses, several diffraction patterns collected for different orientation of the sample can be treated simultaneously (see [6]) in order to obtain an Orientation Distribution Function (ODF) taking advantage of the advanced texture analysis methods offered by MAUD and going beyond the March model.

The possibility to apply correction (Blackman [7]) in order to account for the dynamic nature of the ED-RP has been introduced in the MAUD program but mostly our results show that the use of the electron scattering factors in the kinematical approximation or even a simple pattern matching mode approach can give reliable results in the case of the analysis of an assembly of nanoparticules or textured nanosized polycrystalline thin film.

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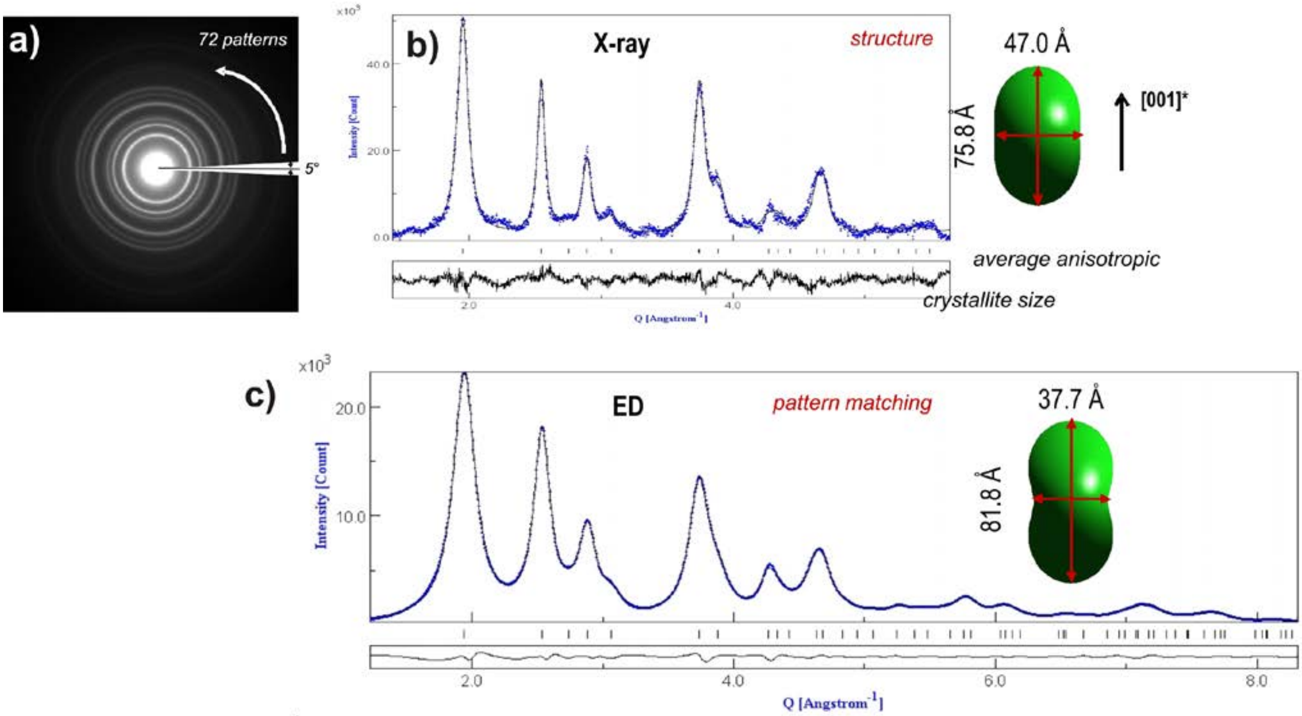

Figure 1. *a) ED-RP obtained from an assembly of TiO_2 nanoparticules [3]. The intensity is integrated by sectioning the rings in 72 arcs of 5° extension. b) RX vs. c) ED-RP microstructure analysis (MAUD). The "ED patterns" presented in c) corresponds to the summation of the 72 patterns integrated azimuthally along the rings.*

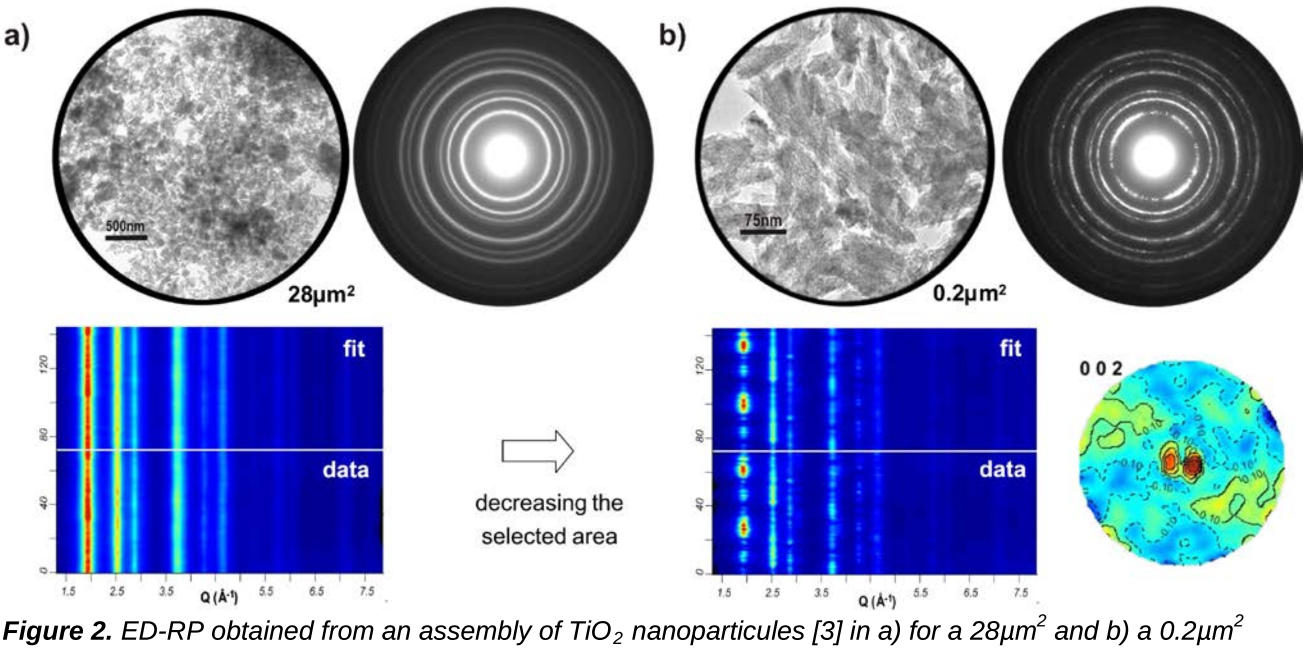

Figure 2. *ED-RP obtained from an assembly of TiO_2 nanoparticules [3] in a) for a $28µm^2$ and b) a $0.2µm^2$ area. In b), the graininess in the ring patterns and the texture effects more pronounced due to the limited number of diffracting particules can be accounted for. The 72 patterns extracted from the ED-RP patterns (data) and the fit obtained by Rietveld analysis are plotted in the form of a 2D map representing the azimuthal variation of the diffraction intensity versus the momentum transfer Q. Pole figure reconstructed from the ODF is given as an indication.*