QUANTITATIVE ANALYSIS of HRTEM IMAGES from AMORPHOUS MATERIALS : ABOUT the ESTIMATION of C_s and δf from HRTEM DIFFRACTOGRAMS

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A quantitative High Resolution Transmission Electron Microscopy (HRTEM) study of amorphous materials has been undertaken in the study-case of amorphous germanium. The analysis consists in a modelling of amorphous germanium, suitable to run multislice calculations, and to simulate HRTEM images, the computed diffractograms of which are numerically compared to experimental ones obtained from a through-focus series taken on a dedicated High Resolution microscope equipped with a field-emission gun. The final aim of this work is to quantify the structural information that can be retrieved from HRTEM images of amorphous materials. As a preliminary step, the coefficient of spherical aberration (C_s) of the microscope, as well as the amount of defocus (δf) of each micrograph have to be known. The aim of this paper is to determine, as precisely as possible, these parameters.

Although the usual methods for such measurements are well-known [O.L. Krivanek, Optik 45 (1976) pp.97], it will be seen that they are not accurate enough to allow a reliable and quantitative match of HRTEM diffractograms from amorphous materials [J.M. Gibson, Ultramicroscopy 56 (1994) pp.26].

A new method has been presented here to determine the C_s and δf values from numerical diffractograms obtained from HRTEM images of an amorphous film. It consists in a numerical treatment of the diffractogram intensities, or rotationnally-averaged profiles RAP(u), on the basis of a calculation of theoretical profiles $RAP_{calc}(u, C_s, \delta f, t)$ from multislice iterations and image simulations, and a minimization of a mean profile error function PAF(Cs, δf, t) when varying C_s and δf within the calculations. The critical steps of this approach are : (i) the accuracy of the model used to describe the amorphous material, (ii) the estimation of the experimental thickness. Point (i) is in principle not very important, and rough models, such as the 'white noise' [G.Y. Fan and J.M. Cowley, Ultramicroscopy 21 (1987) pp.125], could probably give acceptable results, since the multislice iterations account proeprly for the propagation phase shifts ; however, the estimation of the experimental thickness is a rather difficult problem, and a 'correct' modelling of the amorphous material is in fact needed if the method used in this work is to be applied. This has been possible in the case of amorphous germanium, since atomic distribution functions are available in the literature for this material.

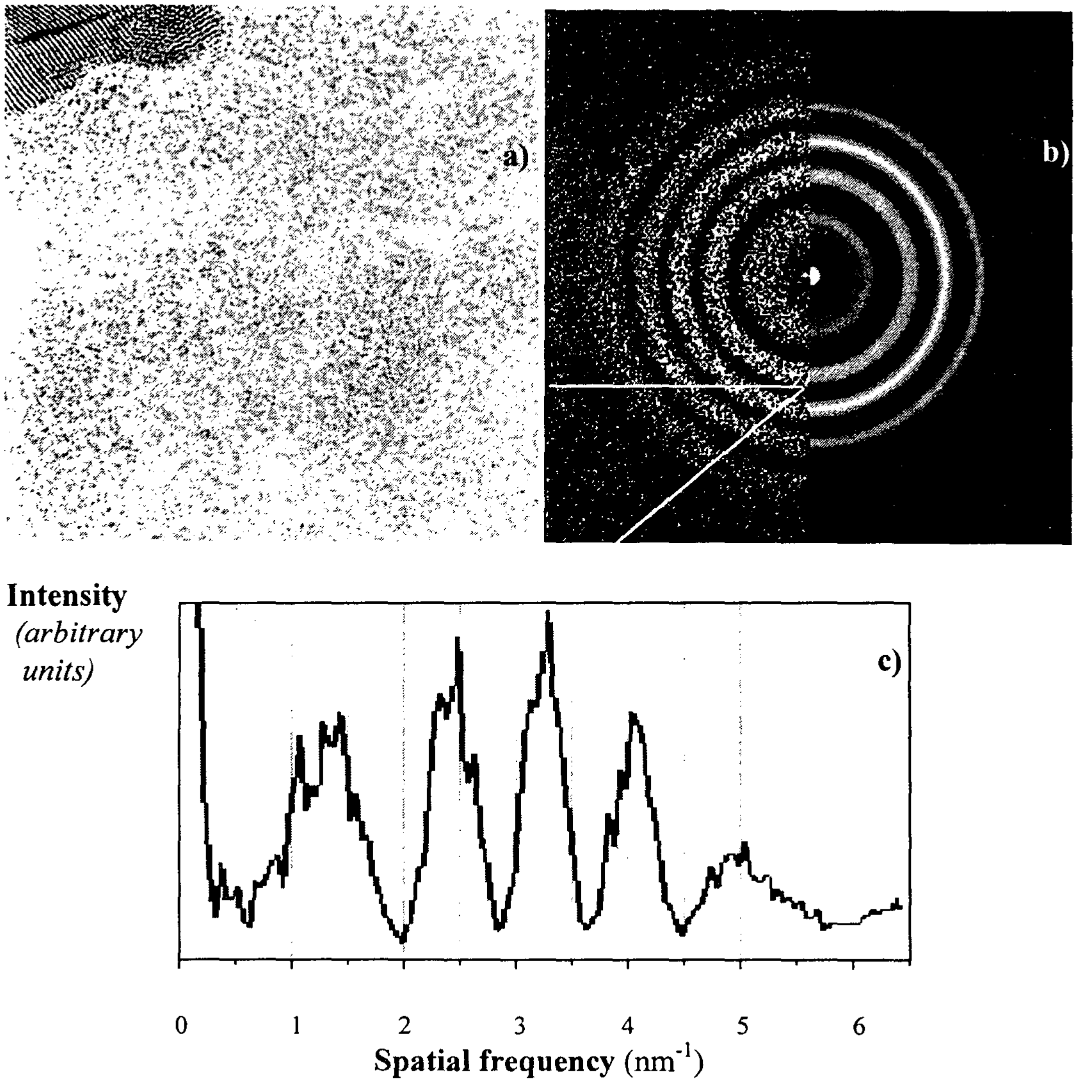

Figure 1: rotationnally-averaged profile $RAP_{exp}(u)$ of the numerical diffractogram of HRTEM image 87 (a), obtained over an angular sector of 40°, in order to minimize the effect of the possible residual astigmatism. The display on the (c)shows the profile after a background substraction (a linear approximation has been used to model the background, its level being taken as the average 'noise' at high spatial frequency). In (b), the experimental diffractogram is shown on the left-hand side of the image, and

the calculated diffractogram, based on the averaged profile $RAP_{exp}(u)$, is shown on the right-hand side.

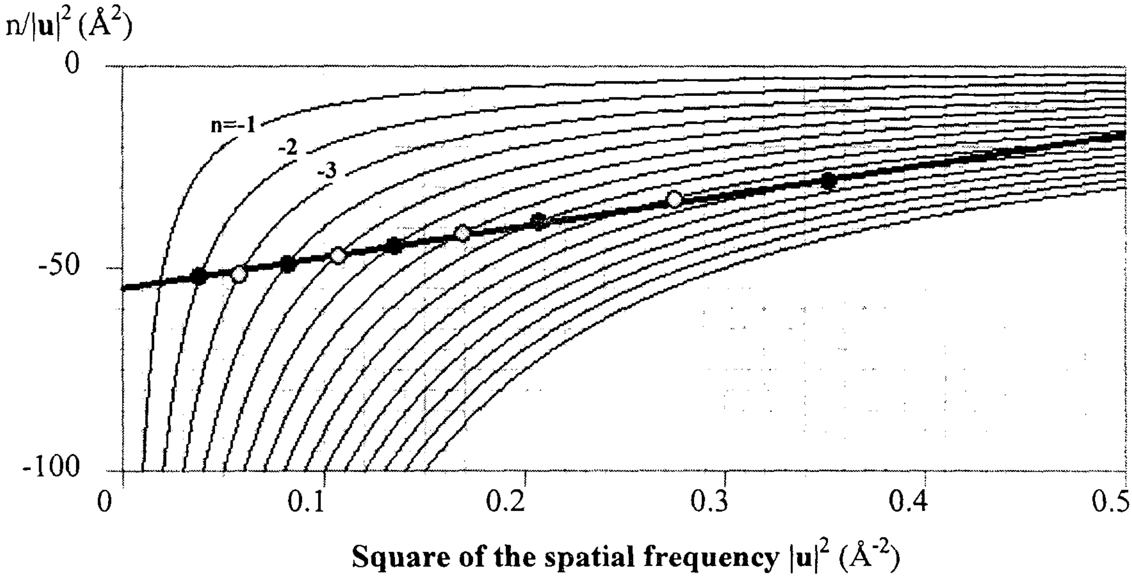

Figure 2 : kinematical treatment of image 87 based on Krivanek's method. for Germanium ; the 'zeros' are shown - grey areas - on the diffractogram) : usual plot $n/|\mathbf{u}|^2$ versus $|\mathbf{u}|^2$ of the zeros and the maxima - respectively, filled and empty circles - of the diffractogram ; the straight line corresponds to the fit shown above, with C_s = 0.496 mm and $\delta f \approx$ -110 nm (-1102 Å) .

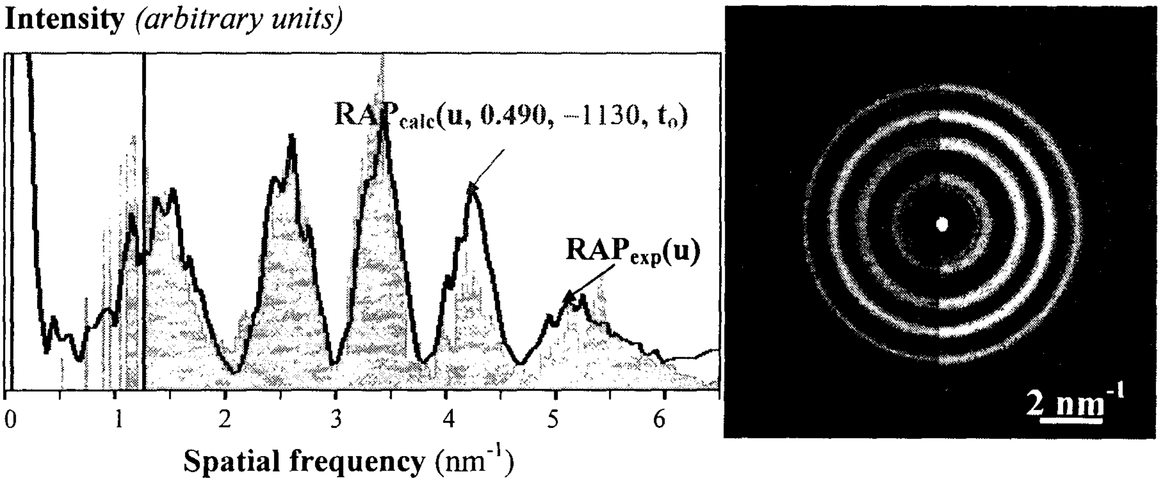

Figure 3 : dynamical match of the diffractogram profile $RAP_{exp}(u)$ for the image 87 with the calculated profile $RAP_{calc}(u, C_s, \delta f, t_k)$ for amorphous Ge at a thickness t_o = 10 nm, with C_s = 0.490 mm and δf = -113 nm