

Supporting information to:

Summary of ISO/TC 201 International Standard ISO 18516:2019 Surface chemical analysis -- Determination of lateral resolution and sharpness in beam-based methods with a range from nanometres to micrometres and its implementation for imaging laboratory X-ray photoelectron spectrometers (XPS)

by

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1. Setting up the Kratos AXIS Nova instrument for imaging and image acquisition conditions

Setting up the Kratos AXIS Nova instrument for imaging

- Successful imaging, as all other modes of the instrument, requires a successful calibration of the instrument.
- To have a stable X-ray beam for the intended measurement X-ray gun and monochromator need to run 30 minutes for “warming up”.
- Find the focus plane of the analyzer using a gold grid as a test sample with X-ray source at full power (300 W) and using a Field of View 2, 450 microns x 450 microns and pass energy $E_p=160\text{eV}$. Verify that center of the area seen by the analyzer is independent of the used Field of view.
- Find the focus plane for the optical camera of the instrument and determine the translation vector between the analyzer focus plane and that of the optical camera.
- Use a clean silver sample in the focus plane of the X-ray source (gun and monochromator) at full power (300 W) using field of View 1, 900 microns x 900 microns, pass energy $E_p=160\text{eV}$. Verify the position of the center of the X-ray beam and adjust if necessary the monochromator. Acquire a map from $BE = 375\text{ eV}$ to $BE = 360\text{ eV}$ at step width 0.5eV . If necessary, adjust the work function.
- Use a pure gold sheet sample at the focus plane of the X-ray source (gun and monochromator) at full power (300 W) using Field of View 2 and 3 at the BE of the maximum intensity of the Au 4f peak. Repeat the acquisition of Au 4f images. (For the precision measurements reported here we acquired six images each of 30 minutes to check for time stability. Stability was approved.) This test addresses the stabilities of (1) the DLD detector’s answer, (2) the locus of the X-ray spot on the gold sample and (3) the manipulator as well.

Measurement conditions (Kratos AXIS Nova) for imaging Ti features in Au on the ETHZ test sample [1]

- Define the field of view of interest by using the optical camera.
- Acquire an Au 4f image using FOV2 and 3 at $EP = 160\text{ eV}$, high resolution. Test the stability of the instrument by repeated acquisition of images (four repetitions are recommended when aiming on a validation of the instrument performance).

- To achieve an appropriate S/N ratio an acquisition time of 2 hours per image is required.
- It is recommended to divide the acquired Au 4f image (“raw image data”) taken from the ETHZ test sample by the image taken with the pure gold sheet sample (“reference image data”) to cancel the structures in the raw image data produced by the imaging system.
- Rotate image to have edge or lines (gratings, narrow line) oriented perpendicular to the X axis (using CasaXPS, see Paragraph 2.).
- Define a box around the edge or lines (gratings, narrow line) where the image is free of distortions. Sum up all lines of image data in the box to prepare a line scan for further evaluation. (using CasaXPS, see Paragraph 2.).

2. Evaluation of straight edge image data by using the CasaXPS software [2]: Determination of lateral resolution expressed as the ESF D_{12-88} parameter

How to use CasaXPS (for a [youtube video](#) use the link in [3])

CasaXPS software features here described are designed to support the use of ISO 18516:2019 methods for estimating lateral resolution within XPS image data. The objective is to provide quantitative results based on linescan data extracted from images.

An image is displayed in CasaXPS as a 2-dimensional rectangular array of pixels. A 1-dimensional line scan with spatial dimensions corresponding to the horizontal direction is formed by summing pixel intensities for pixels in the same column of pixels within an image. The first processing step for image data including edge, narrow line and grating features is to align an edge, a line or gratings perpendicular to the horizontal direction with respect to an image displayed in CasaXPS.

The following example addresses the straight edge method in ISO 18516:2019 where an image of a straight edge has to be acquired first and analyzed after specific image data treatments. Image data made available in ISO 14976 Data Transfer Format (VAMAS format) when opened in CasaXPS appears as VAMAS blocks, one per image, ordered with respect to species/transition VAMAS block fields and arranged by experimental variable. Selected VAMAS blocks are displayed in the left-hand-pane display tiles. Display and processing operations are available via the Image Processing dialog window which is an option on the Options menu of CasaXPS. Alignment of an edge feature with a given orientation is performed by displaying an image in the left-hand-pane. Then using the mouse by dragging the cursor between two locations on the image, an orientation for a processed image is defined. The start and end points for image pixels, indicated by the mouse, represent the limits for a straight line which is displayed over the image once the drag action terminates. An image with orientation where the rows within the image align with the line so indicated is performed using the Image Processing dialog window via the VAMAS Info property page (Figure 1). The button labelled *Rotate* within the *Edge Resolution* section of CasaXPS makes use of a line drawn via the mouse to compute the transformation required to rotate intensities to form a new image. Intensities associated with pixels in the original image are moved within a new image to align the line previously drawn with rows of pixels in the new image. The rotated image appears in a newly created VAMAS file (Figure 2).

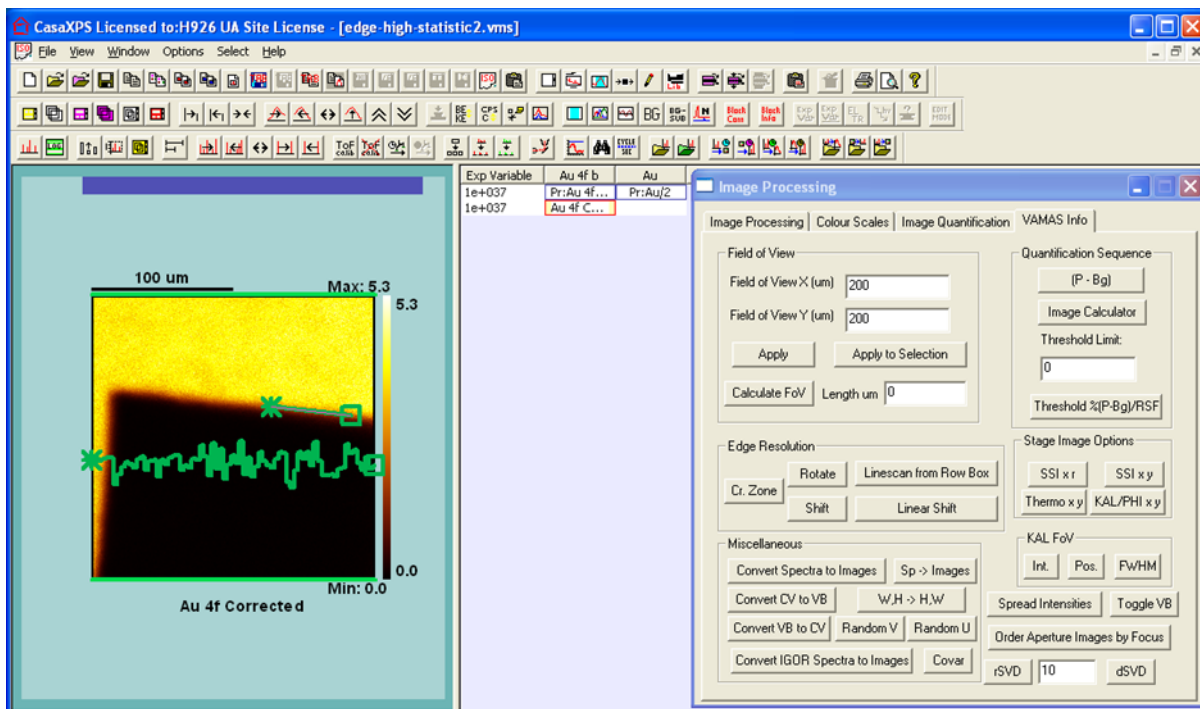


Figure 1: A CasaXPS window displaying image data and the *Image Processing* dialog window with the VAMAS Info property page top-most. A line scan is marked on the image to specify a transformation of pixel intensities that would align rows of pixels with the indicated line. The image in Figure 2 is constructed in two steps. The first step is to rotate the original image to align rows of pixels with the line shown here. After the initial rotation, a second rotation of the horizontally aligned image through 90° is performed to obtain the image displayed in Figure 2.

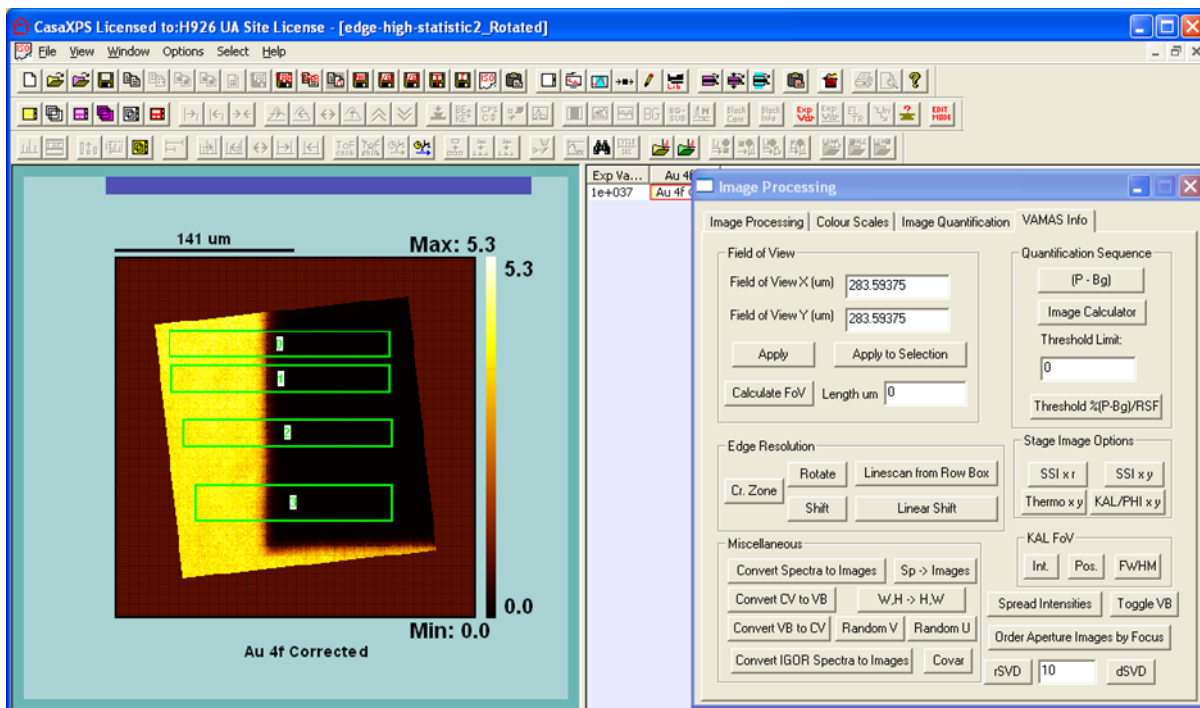


Figure 2: Following rotation of the original image, rectangular zones are created to specify sets of pixels that are destined for use in creating line scans. Four zones are defined as indicated by four green rectangles plotted over the image. From these four zones four line scans are created by summation of

pixel intensities in the vertical sense to provide intensity variation as a function of spatial information defined by the horizontal dimension for each rectangle.

It is possible the most convenient direction for drawing a line on the original image aligning the edge of interest in the rotated image with the horizontal rather than the required vertical. To allow for this contingency, an image can be rotated through 90° by giving focus to the window created by the previous action and then pressing the *Rotate* button without any cursor action to draw a line. In the absence of a line drawn over an image, the action of the *Rotate* button is to create a new VAMAS file where the image is at right angles to the target image for this processing option.

One or more line scans may be created from the processed image by making use of rectangular zones marked on the image in the display tile (Figure 2). A rectangular zone is marked over an image using a drag action similar to the creation of a line used to mark an edge orientation. Once a drag action creates a line displayed over the image, pressing the Cr. Zone button also located in the *Edge Resolution* section as the *Rotate* button places a permanent box on an image. The box drawn over the image displays an index number used to identify line scans which are created from one or more boxes by pressing the *Linescan* from *Row Box* button. These buttons described here are all within the *Edge Resolution* section of the VAMAS Info property page of the *Image Processing* dialog window. Line scans corresponding to boxes drawn on the image displayed in the active tile are calculated and entered into a new VAMAS file.

Straight edge measurement statistics are computed making use of these newly created line scans.

Lateral resolution is calculated from a straight edge in a line scan by creating a quantification region via the *Quantification Parameters* dialog window (Fig. 3) and making use of the *Regions* property page. A quantification region defines the interval spanning the edge feature within a line scan. A set of parameters entered on the *Regions* property page determines the type of information calculated by the region. When characterizing an edge feature the *BG Type* field may be assigned the string D Parameter, which results in particular output values listed on the *Regions* property page. These output parameters include a FWHM value representing the spatial offset calculated based on the current set of region parameters. Region parameters that alter the output values for D Parameter include the Start and End values, the first parameter in the cross-section field and the Av. Width parameter.

If the *BG Type* field is assigned the string D Parameter within a region with limits spanning an edge-feature then the output value labelled FWHM is assigned the distance between two points calculated as follows. The spatial offset reported in the FWHM is determined from changes in intensity between the start and end of the region where the intensity drops to 100 - x and x as a percentage of the variation in intensity. The user-defined parameter with value corresponding to x/100 is entered via the cross-section field of the region. The first parameter in the cross-section field in the list of four cross-section values is used to change the value for x. The range of intensities used to compute the spatial offset with respect to x is determined by intensities associated with the two limits to the region specified by the Start and End parameters. These two intensities corresponding to the limits of the region are in turn computed using a range of intensities spanning a number of data channels, the width of which is specified using the Av. Width parameter. The function of the Av. Width parameter is to limit the influence of noise on the calculation by averaging intensities over a number of data channels. The value entered for the Av. Width parameter specifies indirectly the number of data channels used to compute an average intensity for the signal at each end of the region. The actual number of data channels used in this averaging process is 2n+1 where n is the number entered for Av. Width with the data channel corresponding to the Start and End parameters located at the centre of these 2n+1 data channels.

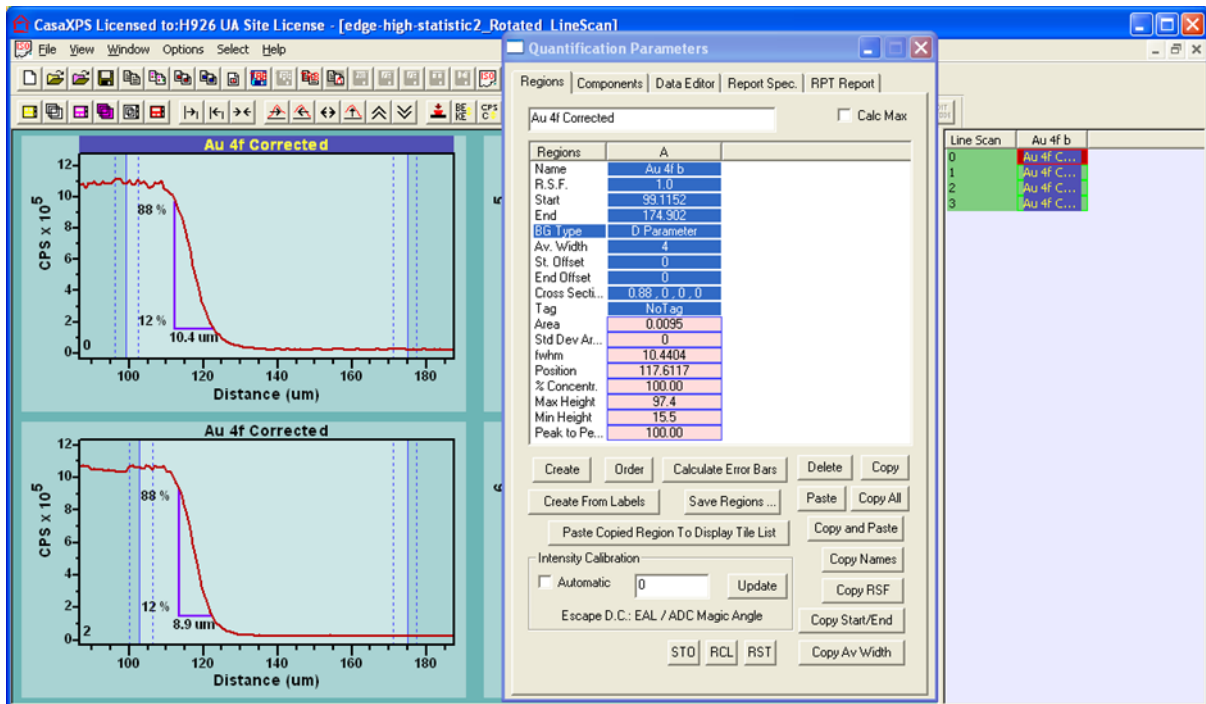


Figure 3: Quantification Parameters dialog window displaying parameters for a region defined for the line scan plotted in the active display tile. The lateral resolution expressed as the Edge Spread Function (ESF) parameter D_{12-88} (ISO 18516:2019, Clause 6) with a resulting value of 10.4 μm is computed using the D Parameter BG Type, which makes use of the first value in the cross-section field, namely 0.88, to define the value for x . The intensity change across the edge is calculated from intensities defined using the Start and End region parameters. These two parameters specify the locations with respect to the line scan where signal is averaged as a means of reducing the influence of noise on the computed rise or fall in intensity. The width for these intervals used to average intensities is specified via the Av. Width region parameter and is set to 4. The vertical dotted lines at the start and end of each region indicate the set of data channels corresponding to the Av. Width equal to 4.

Imaging the straight edge: Measurement conditions Raw data image, image after division by reference data, preparation of line scan profile (yielding the ESF) and analysis of straight edge profile

Measurement parameters for Kratos AXIS Nova:

MHSA Lens mode: F_MHSA_HIGH_MAGN

Aperture: IMAGA AP

Iris: Imaging high res.

X-ray source: Al mono (300W)

Pass Energy: 160 eV

Total acquisition time: 620 mins

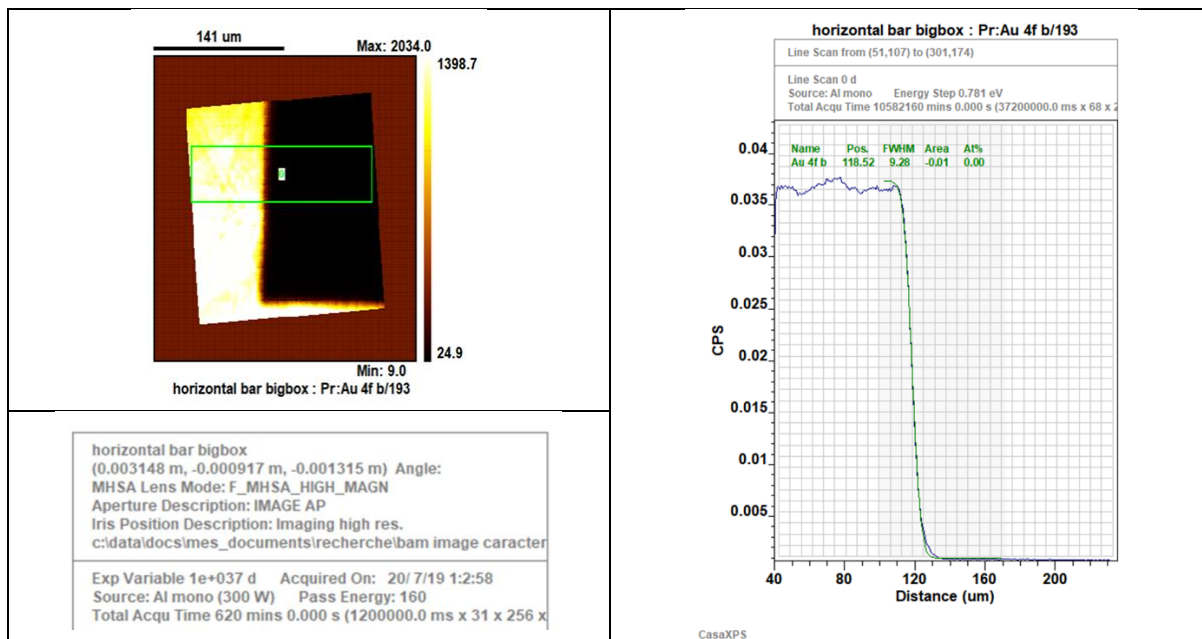


Figure 4: Image and derived line scan profile used for the determination of D_{12-88} as presented in Fig. 2 the main paper.

3. Narrow line and grating images: Used AXIS Nova image acquisition parameters and data evaluation by CasaXPS

MHSA Lens mode: F_MHSA_HIGH_MAGN

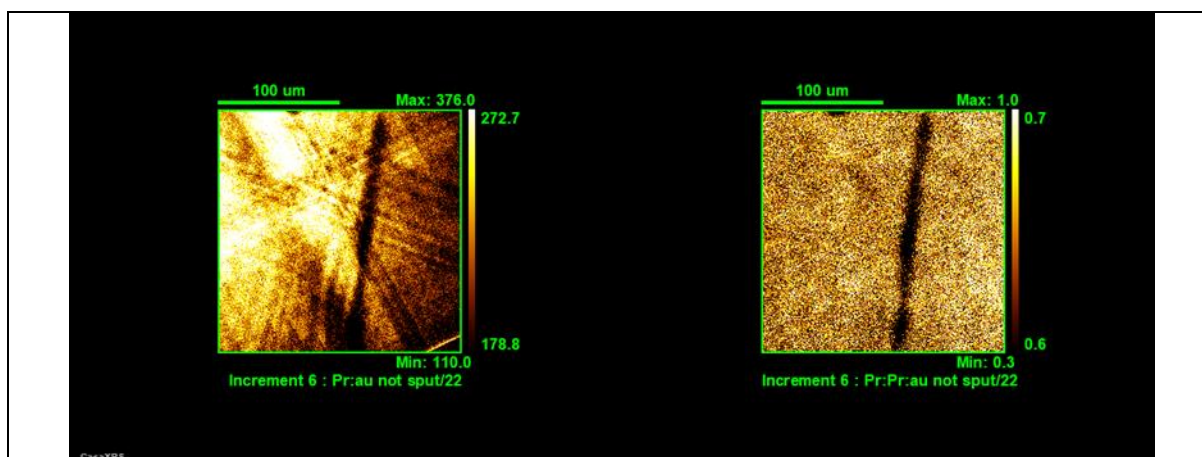
Aperture: IMAGA AP

Iris: Imaging high res.

X-ray source: Al mono (300W)

Pass Energy: 160 eV

Total acquisition time: 620 mins



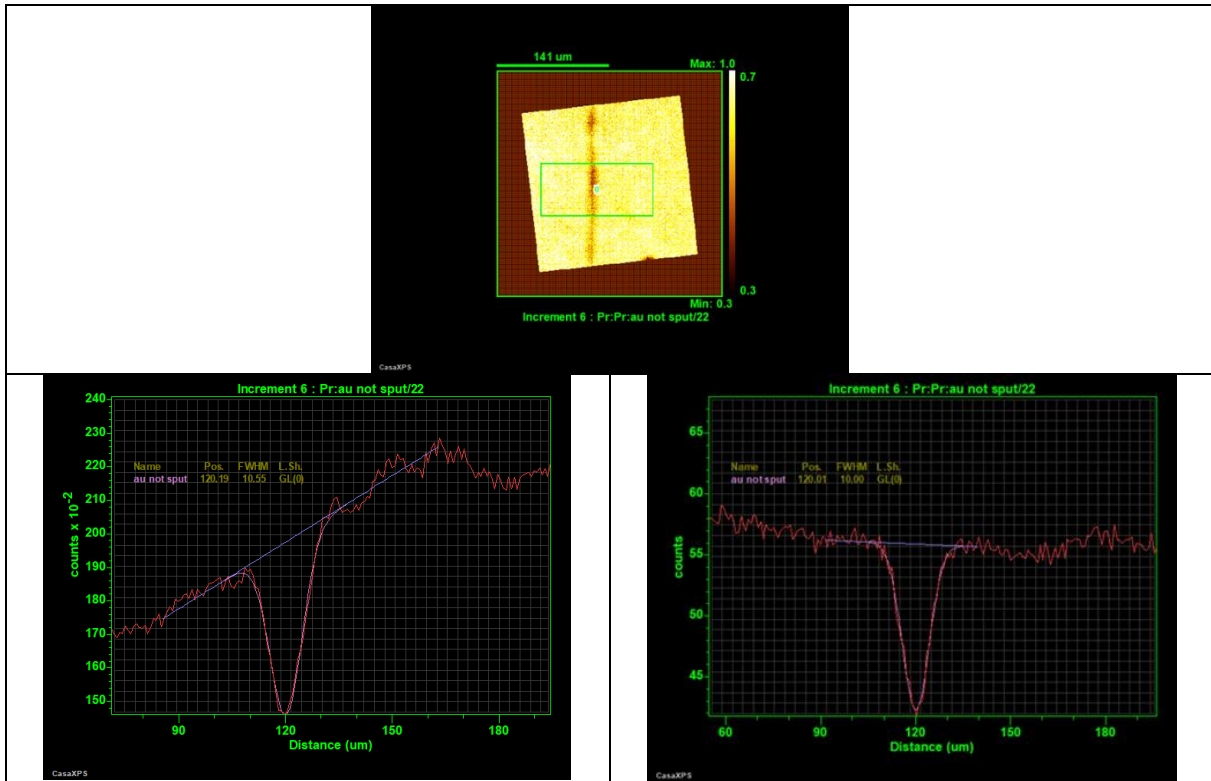


Figure 5: Top left: narrow line image as acquired (“raw image data”), Top right: image after division by “reference image data”, Middle: Image with box for summing up data lines after rotation, Bottom left: Line scan profile across narrow line after summing up data, Bottom right: Line scan profile after subtraction of background with peak shape analysis revealing w_{LSF} and shape of the Line Spread Function (LSF). The same procedure was used for the determination of the effective lateral resolution r_e by using the grating with the smallest period P on the ETHZ test sample.

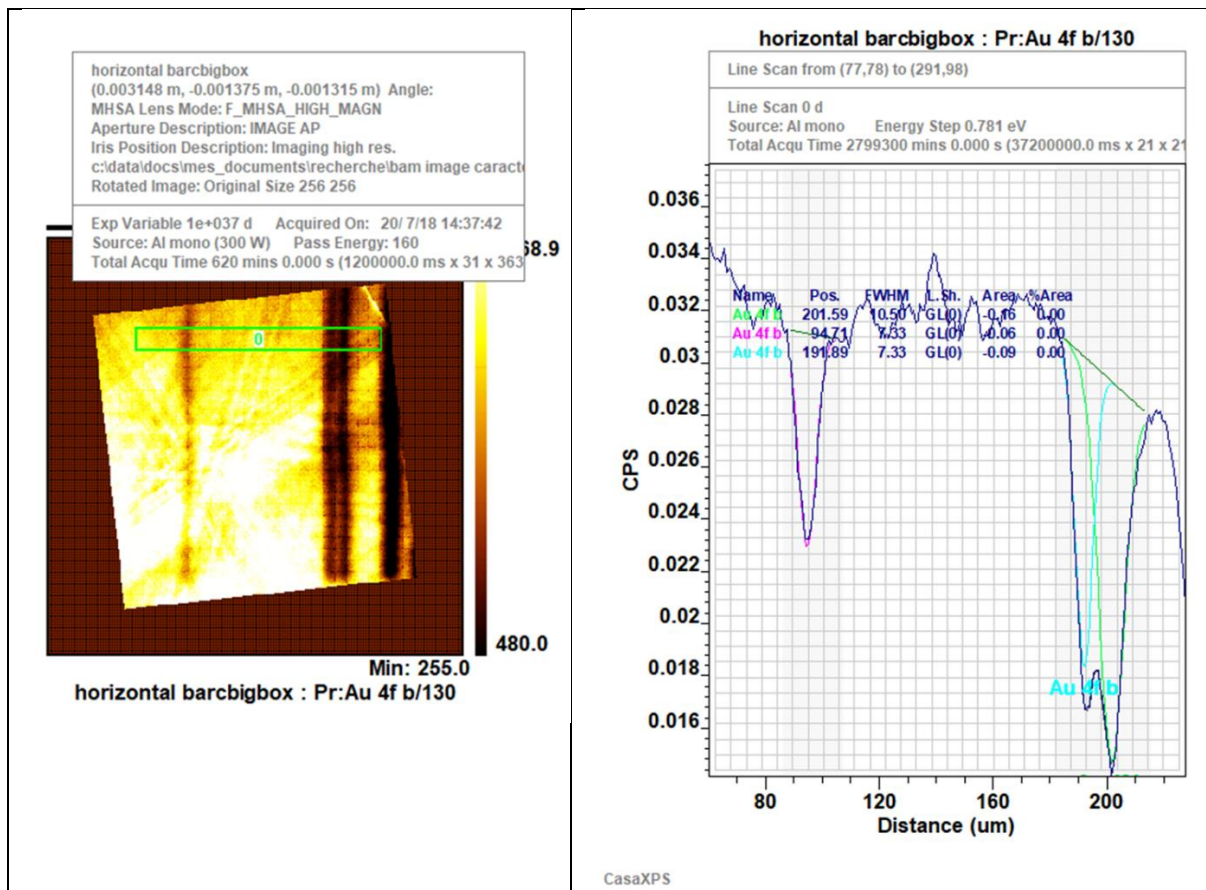


Figure 6: Example for the determination of the lateral resolution parameters w_{LSF} and r_e without division of the “raw image data” by the “reference image data”. Inspection of the “as acquired” image data (left), already aligned with a CasaXPS tool for taking the line scans to be summed up (resulting Au 4f intensity profile on the right side), reveals that the finest A-B-A grating (A,B bar widths $5 \mu\text{m}$, period $P = 10 \mu\text{m}$) on the ETHZ sample is resolved.

References:

[1] C. Passiu, A. Rossi, L. Bernard, D. Paul, J. Hammond, W. E. S. Unger, N. V. Venkataraman, and N. D. Spencer, Langmuir 2017, 33, 5657.

[2] casaxps.com

[3] <https://www.youtube.com/watch?v=F0qnM55kbco&t=0s>