Testing of Lateral Resolution in the Nanometre Range Using the BAM-L002 - Certified Reference Material: Application to *ToF-SIMS IV* and *NanoESCA* Instruments

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The certified reference material BAM-L002 "Nanoscale strip pattern for length calibration and testing of lateral resolution" was used for the determination of the lateral resolution in surface analysis by ToF-SIMS IV and NanoESCA instruments. The BAM certified reference material BAM-L002 is an embedded cross section of epitaxially grown layers of $Al_xGa_{1-x}As$ and $In_xGa_{1-x}As$ on a GaAs substrate. The surface of the sample provides a flat pattern with strip widths ranging from 0.4 to 500 nm. The combination of gratings, isolated narrow strips and sharp edges of wide strips offers possibilities for the determination of the lateral resolution and the calibration of a length scale at selected instrument settings. The feasibility of the reference material for an analysis of the lateral resolution is demonstrated with an TOF-SIMS IV instrument and an Omicron NanoTechnology NanoESCA spectrometer using synchrotron radiation at the UE52 SGM beamline at BESSY II.

INTRODUCTION

ISO 18115: 2001 - Surface chemical analysis -Vocabulary defines the lateral resolution as a "distance measured either in the plane of the sample surface or in a plane at right angles to the axis of the image-forming optics over which changes in composition can be separately established with confidence" [1]. In Note 2 to this definition this standard vocabulary refers to two possible measures of the lateral resolution: "In practice, the lateral resolution may be realised as either (i) the full width at half maximum of the intensity distribution from a very small emitting point on the sample or (ii) the distance between the 12% and 88% intensity points in a line scan across a part of the sample containing a well-defined step function for the signal relating to the property being resolved. These two values are equivalent for a Gaussian intensity distribution. For other distributions, other parameters may be more appropriately chosen. Often, for a step function, the distance between the 20% and 80% intensity points

or the 16% and 84% intensity points in the line scan are used. The latter pair gives the *two sigma width* for a Gaussian resolution function." [1]. For known intensity distributions the different resolution parameters can be converted into each other [2].

In practical surface analysis test samples are needed to determine the lateral resolution. According to the measures of lateral resolution described in Note 2 (see above) these samples must have very narrow structures (method i) and/or structures with very sharp "chemical edges" (method ii). Furthermore it was found that periodic structures like gratings are useful for the direct estimation of the lateral resolution during the adjustment of an instrument.

Nowadays different kinds of samples, e.g. straight edges, mesh bars of copper grids or even gold islands, are used for the determination of the lateral resolution of an instrument. However, no samples with well defined structures below 100 nm are available. To close the gap between the dimensions of lithographic patterns (> 100 nm) and the crystal

Feature	Certified Value	Expanded (k=2)
		Uncertainty U _{CRM}
Calibration length L (centre to centre)	964 nm	35 nm
Strip width in grating 1	288 nm	16 nm
Strip width in grating 2	74 nm	6 nm
Strip width of S5	145 nm	9 nm
Strip width of S6	478 nm	25 nm

Table 1: Certified stripe distances and stripe widths of BAM-L002 (cf. scheme in Fig. 1).

lattice (< 1 nm) BAM has developed a test sample providing the required structures. The certified reference material BAM-L002 "Nanoscale strip pattern for length calibration and testing of lateral resolution" is an embedded cross section of epitaxially grown layers of $Al_xGa_{1-x}As$ and $In_xGa_{1-x}As$ on a GaAs substrate. The surface of the sample provides a flat pattern with strip widths ranging from 0.4 to 500 nm (cf. fig. 1). Details on the preparation of BAM-L002 are published elsewhere [3].

Information on the procedure applied to certify one strip distance and four strip widths are given at a website [4] and in ref. [3]. The certification is based on Transmission Electron Microscopy (TEM). The magnification of the TEM was calibrated by means of a certified magnification calibration sample, MAG*I*CAL[™] from Norrox Scientific, Canada. The length scale of this calibration sample was traced back to the (111) lattice spacing of silicon. The certified properties of BAM-L002 are summarized in Table 1.

PRINCIPAL POSSIBILITIES OF APPLICATION OF THE REFERENCE MATERIAL BAM-L002

The certified reference material BAM-L002 can be used for the calibration of a length scale, the determination of instrument parameters (lateral resolution, characteristics of the probe beam) and the optimisation of instrument settings. It can be used for all surface analytical methods which are able to differentiate between the materials forming the semiconductor strip pattern.

Length calibration

The certified calibration length L = 964 nm is the distance between both 50 nm wide strips 1 and 4 (cf. scheme in fig. 1). The certified value is a "centre to centre distance" and, therefore, the allowed probe beam full width at half maximum (FWHM) covers a broad range up to 400 nm.



Fig. 1: Scheme of the strip patterns at the surface of BAM-L002. The underlined numbers are nominal values and the respective certified values are given in Tab. 1.

Determination of Beam Shape and Width

Beam profiles are two-dimensional distributions of intensity. Their movement across a narrow strip yields an one-dimensional distribution of intensity, the line spread function. In the case of the twodimensional Gaussian distribution, which is often used to describe the intensity distribution in light and particle beams, the line spread function is identical to the cross section through the two-dimensional distribution of intensity of the probe beam [2]. It can be determined by imaging of a narrow strip or imaging of a step transition.

Imaging of a strip, which is narrow compared to the beam width, directly yields the line spread function. BAM-L002 provides narrow strips of 0.5 nm, 5nm, or 50 nm width. Alternatively the line spread function is gained from imaging of a step transition. The profile measured at the step transition corresponds to the integral over the line spread function. The first derivative of this profile yields the line spread function. Furthermore, in accordance with Note 2 in the standard vocabulary [1], the profile of a step transition yields information on the lateral resolution of the instrument used also without differentiating when the distance between the 12% and 88% intensity points or the distance between the 16% and 84% intensity points are used as measures of lateral resolution. BAM-L002 provides a 478 nm wide strip whose edges can be used as the required step transition.

Direct estimation of lateral resolution by imaging of gratings

Imaging of gratings provides information on the lateral resolution and, therefore, this procedure is of particular interest for the adjustment and parameter optimisation of imaging instruments. Samples with gratings of different periods enable a direct estimation of lateral resolution. It is equal or smaller than the period of the smallest grating, when the strips of the grating "can be separately established with confidence" [1]. An objective criterion gives the Rayleigh criterion $m \ge 0.152$, where $m = (I_{max} - I_{min}) / (I_{max} + I_{min})$ is the modulation of the image, I_{max} is the maximum intensity in the image of a grating and I_{min} is the minimum intensity between two strips. BAM-

L002 provides two gratings of 288 nm and 74 nm strip width, respectively.

Determination of the smallest detectable strip

Narrow strips of $Al_{0.65}Ga_{0.35}$ As (0.5 nm, 5 nm, 50 nm), $In_{0.33}$ Ga_{0.67} As (5 nm) and InAs (0.4 nm) at the surface of BAM-L002 enable the estimation of the smallest detectable structure.

EXAMPLES OF APPLICATION

SIMS by a ToF-SIMS IV instrument

Fig. 2 shows the strip pattern of BAM-L002 imaged by a ToF-SIMS IV instrument (ION-TOF GmbH, Münster, Germany) using Al⁺ and In⁺ secondary ions. The Al image reveals that Grating 2 (74 nm strips) is clearly resolved and the Rayleigh criterion is satisfied (modulation m = 0.195). Simulations of imaging were done by convolution of the strip pattern with different functions describing the intensity distribution within the primary ion beam. The best agreement with the measured profile was found with a Pseudo-Voigt Function (a weighted sum



Fig. 2: Tof-SIMS imaging of BAM-L002 using the extreme cross over mode at 25 keV Ga⁺. The Al⁺-image ($3.9 * 3.9 \mu m$) and the In⁺-image ($2.0 * 2.0 \mu m$) were taken with 100 scans over (128 * 128) points. The SIMS intensity profiles in the middle of the figure are accumulated from 120 horizontal line scans taken from the Al⁺ and In⁺ images, respectively. The simulated profile was calculated by convolution of the strip pattern with a weighted sum of a Gaussian (70%) and a Lorentzian (30%) of both 105 nm FWHM.



Fig. 3: Line profiles through different parts of the Tof-SIMS AI^{+} image of Fig. 2.

- (a) Line profile across the edge of the 500 nm strip 6 of BAM-L002. For Gaussian beams the distance between the 12% and 88% intensity points corresponds to the FWHM of the beam profile.
- (b) Line profile across the 5 nm strip 3 of BAM-L002. The experimental values (•) were fitted by three types of functions.

of Gauss Function and Lorentz Function) with FWHM of 105 nm.

Actually, a FWHM of the probe beam profile of about 95 nm was measured by imaging a 5 nm wide strip (cf. Fig 3b). The profile through this image gives the line spread function of the instrument. It has slightly increased tails compared to the Gauss Function and can be described by a Voigt Function. The non-Gaussian character of the intensity distribution within the primary ion beam is confirmed by the analysis of the step transition. The step transition profile in Fig. 3a is characterised by a distance of 121 nm between the 12% and 88% intensity levels. For an ideal Gaussian beam this distance would be equal to the FWHM of the beam profile (95 nm in Fig. 3b). The In image in Fig. 2 shows that 5 nm and 0.4 nm wide strips of $In_{0.33}Ga_{0.67}As$ and InAs, respectively, can be detected by the ToF-SIMS IV instrument at the used settings.

ESCA by the NanoESCA instrument

Imaging XPS instruments with laboratory X-ray sources are currently limited to about 3 μ m lateral resolution but the NanoESCA instrument by Omicron consists of a newly designed band pass energy filter with intrinsically negligible image aberrations combined with a suitably modified PEEM that is used as an entrance electron optics

Characterisation measurements to determine the spatial and energy resolution of the current prototype instrument were carried out at the UE52 SGM beamline at BESSY II utilising its high brightness and tuneable photon energies [5]. Lateral resolution was tested also with BAM-L002. Fig. 4 reveals that the 300 nm strip grating is clearly resolved. Obviously structures with widths of 50 nm are distinguishable





Fig. 4: a) ESCA AI 2p image of BAM-L002 taken at BESSY's UE52 SGM beamline at 150 eV excitation energy and 100 eV spectrometer analyser pass energy providing a Ag $3d_{5/2}$ FWHM of 0.6 eV. b) The AI 2p line scan is constructed from the image data in the box inserted into Fig. 4a. The ideal AI profile to be expected from the BAM-L002 layout is inserted into Fig. 4b.



Fig. 5: Comparison of the measured ESCA AI 2p line scan of BAM-L002 with a simulation (convolution of the stripe patterns of BAM-L002 with a Gaussian Point Spread Function of = 105 nm)

from the background, but the 80 nm strip grating is not resolved.

The profile derived from the image of the strip pattern was simulated by a convolution of the Al profile characteristic of BAM-L002 with Gaussian profiles. The comparison of the measured Al 2p ESCA line scan with simulated profiles provides best results when a Gaussian profile with = 105 nm is used (cf. Fig.5). Correspondingly the line spread function of the NanoESCA imaging system has a FWHM of 250 nm.

CONCLUSIONS

The certified reference material BAM-L002 is well suited to be used as a test sample for the determination of lateral resolution in surface analysis. Its flat semiconductor strip patterns provide a chemical contrast without unwanted three dimensional topography. For the first time well defined structures with dimensions between 0.4 and 100 nm are available for the determination of the lateral resolution of surface analytical instrumentation. The combination of gratings, isolated narrow strips and sharp edges of wide strips offers improved possibilities for the calibration of a length scale, the determination of instrument parameters and the optimisation of instrument settings.

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