Certified Reference Material

BAM-L200

Nanoscale stripe pattern for testing of lateral resolution and calibration of length scale of imaging SIMS, AES and XPS instruments

Certified values

Characteristic	Certified Value (nm)	Expanded (k=2) Uncertainty (nm)
W1	691	23
W2	691	23
W3	293	9
W4	294	9
W5	19.5	1.7
W6	195	6
W7	195	6
W8	38	2.6
W9	3.6	0.8
W10	14.2	1.5
W11	3.5	0.7
W12	96	2.6
P1	587	17
P2	389	10
P3	273	7
P4	193	5
P5	136	6
P6	97	3
P7	67.5	2.5
P8	48.5	2.6
P9	76.5	2.4
P10	57	2.2
P11	42	1.3
P12	31	1.1
P13	23	1.1
P14	17.5	1.0
P15	13.3	1.1
P16	9.4	1.4
P17	6.9	1.0
D1	4670*	48*
D2	986	22
D3	492	11.3
D4	1264	25
D5	237	8.3
D6	114	2.8

W – stripe width, P – period of a square-wave grating, D – centre to centre distance between stripes or between stripes and gratings, respectively.

Values are taken from TEM measurements.

* D1 is taken from SEM measurements.

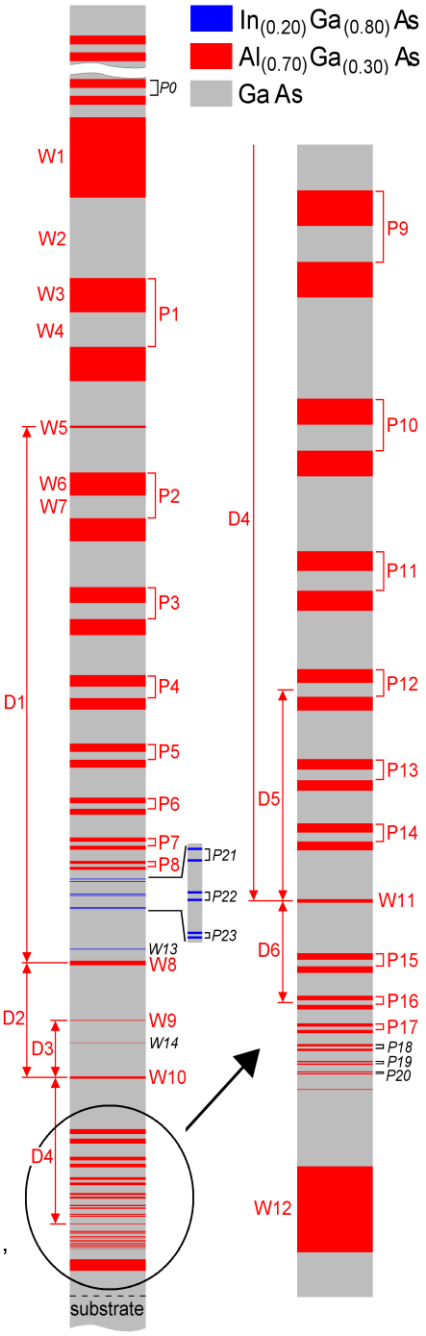

Fig. 1: Certified (red) and non-certified (black italic) features

The stripe pattern shows single stripes and series of square-wave gratings with finely graded periods P (see Fig. 1). Each of these gratings consist of three stripes of the same width: Two $Al_{0.7}Ga_{0.3}As$ stripes are separated by one GaAs stripe. The period is twice the stripe width.

Non-certified values are indicated by black italic lettering in Fig. 1. The values are given by the manufacturer. Certified values deviate between 2% (W or P > 300 nm) and 10 % (W or P between 4 and 10 nm) from the nominal values. Similar deviations are expected for non-certified values. The non-certified 5 nm thick $In_{0.2}Ga_{0.8}As$ layers W13 and P21 – P23 may show variations in layer thickness due to the formation of quantum dots.

Non-certified values, for information only

characteristic	non-certified value (nm)
W13	*5.0*
W14	*1.0*
P0	*147 (80 AlGaAs + 67 GaAs)*
P18	*4.6*
P19	*3.0*
P20	*2.0*
P21	*23 (5 InGaAs + 18 GaAs)*
P22	*15 (5 InGaAs + 10 GaAs)*
P23	*10 (5 InGaAs + 5 GaAs)*

CERTIFICATION PROCEDURE AND HOMOGENEITY

The certified values were determined from the inner part (∅ 60 mm) of a 4-inch wafer and are valid for all samples taken from this area. The homogeneity of layer thickness within this area was controlled by High Resolution X-ray Diffraction, Optical Reflection Spectroscopy and Transmission Electron Microscopy (TEM).

A batch of 5 wafers was coated during one Metal Organic Vapor Phase Epitaxy (MOVPE) process. The wafer-to-wafer homogeneity was confirmed by Scanning Electron Microscopy (SEM) measurements with samples taken from different wafers.

Certification has been done by TEM and for the longest calibration distance D1 by SEM. The magnification of TEM was calibrated by means of a certified magnification calibration sample (MAG*I*KAL™ from Norrox Scientific, Canada) which itself was calibrated with respect to the (111) lattice spacing of silicon. The magnification of SEM was calibrated by means of a calibration standard with etched groups of lines in silicon (IMS HR 98 727-04 183) which was certified by the Physikalisch-Technische Bundesanstalt (PTB).

The determination of certified values is based on the evaluation of 7 TEM-lamellae. From these lamellae, more than 100 images were taken at different magnifications between 10 000 and 300 000. The uncertainties of the certified values were determined from the estimation of uncertainties and calculation of their propagation according to the Guide to the expression of Uncertainty in Measurement (GUM) and from repeated measurements.

STABILITY

$Al_xGa_{1-x}As$ - GaAs – multilayers are long-term stable systems. However, oxidation of the $Al_{0.7}Ga_{0.3}As$ leads to height differences of 2 – 4 nm between $Al_{0.7}Ga_{0.3}As$ and GaAs stripes in the surface. These small height differences do not influence the imaging. The formation of oxide layers during storage on air cannot be avoided. Sputtering can be an appropriate procedure to remove them. Ion beams used for sputter cleaning or as a probe may result in changes of surface topography. This effect has to be considered by the user.

A sample which is a cross section of a layer stack has the same lateral element distribution over the whole depth. Therefore, it is possible to renew the sample surface by careful grinding and polishing.

DESCRIPTION OF THE SAMPLE

The layer stack was grown by MOVPE on GaAs substrate. All layers (GaAs, AlGaAs and InGaAs) are n doped with Si ($5x10^{17}$/cm3 by ECV measurement). The whole wafer was sawed into platelets. A selected platelet is mounted in a clamp of nonmagnetic stainless steel (Fig. 2). A milled hole on the stainless steel area serves as a marker for the side of the platelet which is coated by the layer stack. The full metal packaging of the semiconductor platelet ensures the conductivity of the whole sample. The sample is suitable for ultra high vacuum applications.

Grinding and polishing of the sample surface produces a flat cross section with a roughness of about 1-2 nm. However, the sample surface may show a few scratches. Areas affected by scratches should not be used for imaging. The surface of the multilayer stack is displayed as a stripe pattern in Fig. 3.

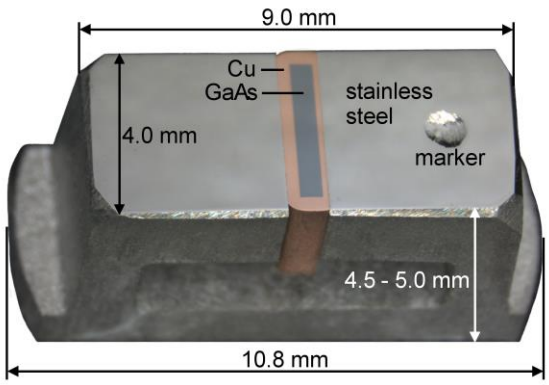

Fig. 2: Sample

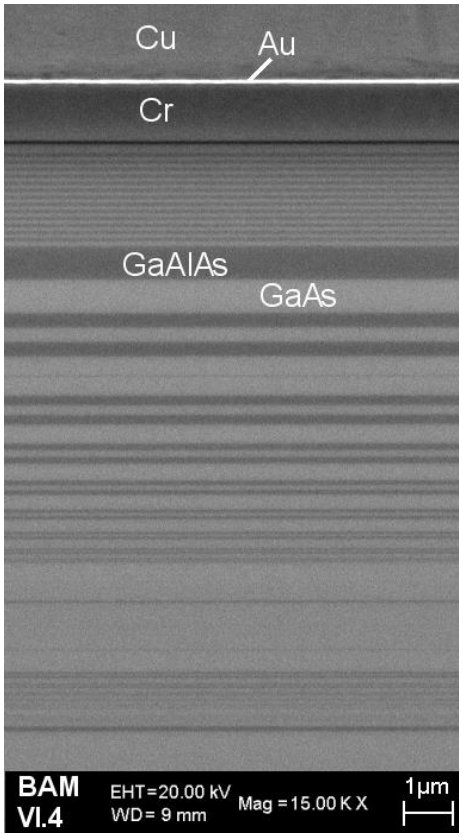

Fig. 3: SEM image of the sample surface

APPLICATION OF THE SAMPLE

BAM-L200 can be used by all methods of surface analysis which are sensitive to a material contrast between $Al_{0.7}Ga_{0.3}As$ and GaAs. Successful tests have been accomplished with Secondary Ion Mass Spectrometry, Auger Electron Spectroscopy and Electron Spectroscopy for Chemical Analysis (NanoESCA endstation at a synchrotron radiation source). BAM-L200 might be useful for other methods, too, but this is beyond its specification.

The delivered sample was flushed with distilled water and cleaned with isopropanol to remove particles from polishing. Despite this procedure, there may be, however, some particles remaining on its surface. Areas affected by those particles should not be used for imaging. Contaminations may be removed from the surface by wet cleaning or sputtering, but sputtering may change the surface topography.

Imaging of the sample allows the real time estimation of lateral resolution: The period of the smallest resolved square-wave grating corresponds to the lateral resolution of the image. Numerical analysis of the image of square-wave gratings of different periods enables the determination of the modulation transfer function (MTF). Furthermore narrow stripes between 1 nm and 40 nm enable the determination of the line spread function (LSF) and step transitions enable the determination of the edge spread function (ESF). All these functions describe the lateral resolution of the image. Certified distances D may be used for the calibration of length scales. The certified values are center-to-center distances between narrow stripes and/or fine 3-stripe gratings, respectively. This definition of distance does not depend on lateral resolution, because the center of a stripe or a 3-stripe grating (resolved or not resolved) does not vary with resolution.

CERTIFICATION REPORT

BAM-L200, Berlin, 2007, https://rrr.bam.de/RRR/Navigation/EN/Reference-Materials/RM-Certificates-reports/Layered-and-surface-materials/layered-and-surface-materials.html

Bundesanstalt für Materialforschung und -prüfung (BAM)
D-12200 Berlin, Germany, 2007-09-25

Department VI
Materials Protection and Surface Technology

i.V. G. Reiners

Dr. G. Reiners
p. p. Head of Department

Working Group VI.43
Surface and Thin Film Analysis

Dr. W. Unger
Head of Working Group

Supply of Reference Material: Bundesanstalt für Materialforschung und -prüfung

Richard-Willstätter-Straße 11, D-12489 Berlin, Germany

Phone : +49 30 8104 2061
Fax: +49 30 8104 1117

e-mail : sales.crm@bam.de
internet: www.bam.de