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Two-wave X-ray optical diagnostics of Ge_xSi_{1-x}/Si modulation-doped heterostructures

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Abstract

New X-ray optical methods—two-wave reflecto- and refractometry—have been used for the first time for the determination of Ge_xSi_{1-x}/Si composition and exact measurements of multilayer heterostructure thicknesses. Both techniques are based on simultaneous measurements of two intense characteristic X-ray lines separated from the polychromatic X-ray probe by semitransparent monochromators. X-ray reflectometry of multilayer structures completely eliminates intensity drift influence and geometrical errors at small grazing angles. The refractometry technique provides direct determination of refractive index decrement and the respective value of x in a solid solution with ~1% accuracy. Refractometry data are not influenced by mechanical strain and by presence of overlaying thin film structure.

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Keywords: X-rays; Reflectometry; Refractometry; Ge_xSi_{1-x}

1. Introduction

X-ray reflectivity measurements at small grazing angles are widely used for non-destructive diagnostics of surface layers and thin film structures [1,2]. However, for some important cases this technique does not provide reliable results. The first problem arises when the sample size is < 1–2 cm, which is typical for routine laboratory practice. In this case a correct calibration of specularly reflected intensity is difficult or impossible as the sample surface does not completely intercept the incident beam in the angle range of total external reflection and the adjacent angle interval. Another metrology problem is bound up with the number of variable parameters necessary for the mathematical processing of the reflectivity curve from multilayer structures. To start the fitting process in a computer simulation, the minimum number of parameters should be equal to $3N+2$, where N is the number of layers. In practice, additional parameters describing the interlayer diffusion and interface roughness also have to be taken into consideration. As a result, with increasing number of layers the reliability of fitting is quickly decreasing.

It has been demonstrated in Ref. [3,4] that two-wave reflecto- and refractometry methods provide much more reliable measurements allowing, in particular, complete exclusion of errors due to indefinite illumination at small grazing angles. In this study both methods have been used for the first time for the determination of Ge_xSi_{1-x}/Si composition and

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the exact measurement of multilayer heterostructure thicknesses.

2. Two-wave set-up and methods

The two-wave reflectometer X-ray scheme [3,5] is shown in Fig. 1. Two highly ordered pyrolytic graphite (HOPG) monochromators 7, 10 with corresponding scintillation detectors 9, 12 have been tuned to CuK_α and CuK_β characteristic lines. The first monochromator 7 is semitransparent and acts as an X-ray beam-splitter. Its coefficients of peak reflection for the CuK_α line and of transmission for the CuK_β line are equal to 22% and 87%, respectively. Monochromators 7, 10 and detectors 9, 12 are mounted on the common arm support of the goniometer. This arrangement makes it possible to measure simultaneously two angle diagrams.

The samples were grown by solid source MBE using a RIBER SIVA 45 system. A modulation-doped heterostructure consisting of a 100 nm undoped Si buffer, a 50 nm $Si_{0.7}Ge_{0.3}$ low temperature (T_{Growth} = 250°C) buffer, a 150 nm $Si_{0.7}Ge_{0.3}$ high-temperature (T_{Growth} = 500°C) buffer, a 10 nm $Si_{0.3}Ge_{0.7}$ channel, a 10 nm undoped $Si_{0.7}Ge_{0.3}$ spacer, a 2 nm B-doped supply layer (1.0×10^{19} cm^{-3}), 50 nm $Si_{0.7}Ge_{0.3}$ and 10 nm Si undoped capping layers were consecutively grown at 500°C.

At first the primary beam is calibrated by measuring the ratio of intensities for the chosen characteristic lines $K = I^0(\theta, \lambda_2)/I^0(\theta, \lambda_1)$ at $\theta = 0$ with relative statistical error 0.03–0.1%. It has been discovered earlier [3] that two-dimensional distributions of the X-ray flow density in normal cross-sections of incident beam are similar for both spectral lines at any operation parameters of the two-wave reflectometer. Thanks to this important property the ratio of the reflected intensities $RR(\theta, \lambda_1, \lambda_2)$ could be determined straightforwardly from the relation

$$RR(\theta, \lambda_1, \lambda_2) = KI^r(\theta, \lambda_1)/I^r(\theta, \lambda_2),$$

where $I^r(\theta, \lambda_1), I^r(\theta, \lambda_2)$ are reflected intensities measured during the θ–2θ scan.

It should be emphasized that $RR(\theta, \lambda_1, \lambda_2)$ is free from instrumental errors. For subsequent mathematical processing it also provides the following advantages: (1) the best fitting for $RR(\theta, \lambda_1, \lambda_2)$ can be achieved if both simulated reflectivity curves for λ_1, λ_2 are best fitted separately; (2) the peak-to-valley modulation of $RR(\theta, \lambda_1, \lambda_2)$ curve is much more pronounced allowing much more effective fitting.

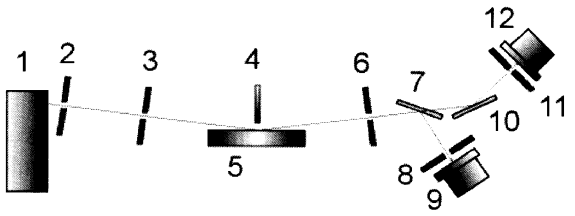

Fig. 1. X-ray optical scheme of the two-wave reflectometer: 1—X-ray tube, 2, 3—divergence slits, 4—beam knife, 5—sample, 6—receiving slit, 7, 10—semitransparent graphite monochromators, 8, 11—anti-scatter slits, 9—α-detector, 12—β-detector.

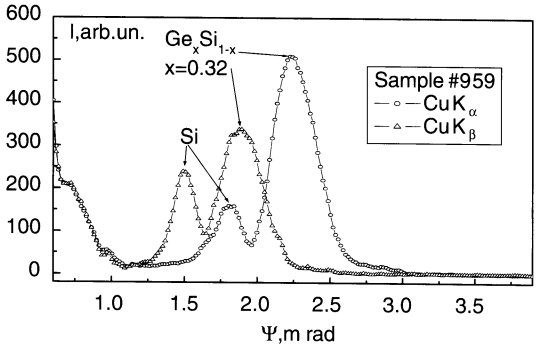

Fig. 2. Refractograms of Ge_xSi_{1-x}/Si heterostructure as intensity I vs. deviation angle Ψ for two spectral lines.

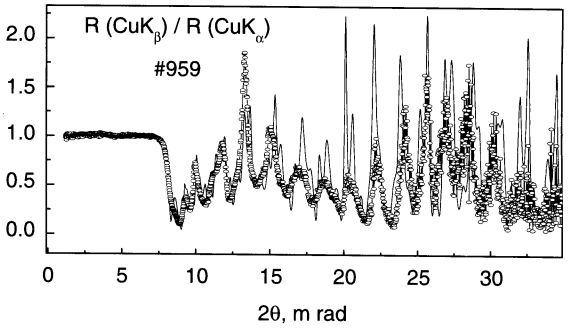

Fig. 3. Measured (dots) and calculated (solid line) ratio reflectivity curves $R(\theta, CuK_\beta)/R(\theta, CuK_\alpha)$ for Ge_xSi_{1-x}/Si heterostructure.

Table 1
Technological (d_t) and calculated (d_c) layer thickness (nm)

Sample		Si cap	$Si_{0.7}Ge_{0.3}$	$Si_{0.7}Ge_{0.3}$	$Si_{0.3}Ge_{0.7}$	$Si_{0.7}Ge_{0.3}$
#959	d_t	10	50	12	10	200
	d_c	9,5	49,3	9,9	11,6	200
#960	d_t	10	50	12	10	200
	d_c	9,9	46	12,6	11,9	203

In refraction mode the sample surface is fixed at a small negative grazing angle with respect to the incident beam. In this position the beam illuminates practically normally the cross-section of the heterostructure and passes in succession through the side face and sample surface. In this case, in accordance with Snell's law the angle positions of the refracted maxima are not influenced by the presence of oxide layers or deposited cap multilayers, thus allowing the direct determination of the refraction index of sufficiently thick layers and the respective value of x in solid solutions.

3. Results

Clearly, resolved refraction peaks from the Si substrate and the Ge_xSi_{1-x} layer under the Si cap-layer are shown in Fig. 2. Composition calculations of Ge_xSi_{1-x} from the refractive index for two characteristic lines gave $x = 0.32$ as compared with a nominal value of $x = 0.30$ and an XRD-value of $x = 0.29$. The experimental and calculated ratio reflectograms for the Ge_xSi_{1-x}/Si heterostructure are shown in Fig. 3. It is seen that the calculated curve can be fitted to the experimental one till zero grazing angles. It should be noted that to achieve this result no geometrical correction or any conjecture about the surface illumination conditions are required. Values of technological and calculated layer thickness are given in Table 1. The value of x measured by the refractometry method was used in a fitting procedure.

The presented results demonstrate that the proposed metrology based on simultaneous measurements with two spectral lines provide a much more reliable extraction of multilayer structure parameters. The ratio-reflectivity technique excludes the necessity of continuous reflectivity calibration, thus, making possible X-ray optical measurements of samples of any size and form, meeting the conditions of specular reflection.

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