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Abstract. — The Convergent Beam Electron Diffraction technique (CBED) has been applied to determine the lattice strain in $Si_{1-x}Ge_x/Si$ heterostructures and below patterned films on silicon substrates. The well known problem of the stress relaxation which occurs in thinned TEM samples has been overcome, in the case of the heterostructures, by applying the isotropic elasticity theory to the lattice constants measured along different crystallographic directions through the shift of the High Order Laue Zone (HOLZ) lines in the central disk of the CBED patterns. In this way bulk strain values have been obtained, in good agreement with values deduced from independent techniques In patterned structures, the high spatial resolution of the CBED technique has been applied to determine the distribution of the components of the strain tensor induced into oxidized silicon substrates by Si_3N_4 stripes A good agreement with the results obtained using numerical computations has been found.

1. Introduction

The need of characterization techniques suitable to investigate strain fields in semiconductors has strongly increased in the present decade, due to their impact on the device performance.

With the scaling down of integrated circuit dimensions, techniques with high spatial resolution, like Convergent Beam Electron Diffraction (CBED) in a Transmission Electron Microscope (TEM), are required to investigate lattice strain variation on a submicron scale. This technique has proved to be quite accurate in detecting relative variations of the lattice parameters ($\sim 10^{-4}$) on a spatial scale of few tens of nm [1, 2].

In this paper, the CBED technique will be applied to two types of structures which are of interest in semiconductor device technology. The first is the $Si_{1-x}Ge_x/Si$ heterostructure,

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which is the basis of the high frequency bipolar transistor; it will be demonstrated that the tetragonal distortion (*i.e.* the bulk strain) of these films can be simply deduced from a single CBED pattern, irrespective of the relaxation due to the specimen thinning.

The second example is a patterned Si_3N_4 film, commonly used as oxidation mask in IC processing and deposited onto an oxidized silicon wafer. After patterning non-homogeneous stress fields are present at the film edges; the values of the components of the strain tensor, have been deduced from CBED patterns taken in different points of the structure and are compared with the ones computed according to a two dimensional process simulator.

2. Experimental

The lattice mismatch analysis has been performed in two uniform $Si_{1-x}Ge_x/Si$ heterostructures. Layers 100 nm thick and with a nominal Ge content of 6.5 at.% (SIGEA) and 2 at.% (SIGEB), respectively, were obtained by conventional solid source MBE on [100] Si wafers.

CBED strain measurements have been also performed on specimen with patterned structures. After growing a 20 nm thick pad oxide by steam oxidation at 800 °C on a silicon wafer, a Si_3N_4 film, 200 nm thick, was deposited by low pressure chemical vapor deposition. Si_3N_4 stripes 0.9 μm wide with a separation of 3.5 μm were eventually obtained by conventional photolitography.

TEM cross-sections were prepared from these samples according to the standard procedure which involves glueing, sawing, mechanical lapping and ion beam milling to perforation. The normal to the specimen surface was the [010] and the [110] direction in the heterostructures and in the patterned structures, respectively.

A Philips CM30 TEM operating at 100 kV in the nanoprobe mode (spot size: 10 nm) was employed for the CBED analysis. A Gatan liquid-nitrogen cooled double tilt holder was used to reduce the thermal diffuse scattering, thus maximizing the High Order Laue Zone (HOLZ) lines visibility.

3. Determination of Strain Relaxation in the Heterostructures

The main drawback for CBED measurements is the strain relaxation occurring at the free surfaces of cross-sectioned samples for TEM/CBED analysis [3,4] which depends on the local specimen thickness. The solution can be easily found in either very thin or very thick regions of the specimen, but unfortunately the corresponding HOLZ lines patterns are either invisible or of too poor quality. Even in the simple case of mismatched heterostructures, several attempts have been made in order to describe this relaxation as a function of the sample thickness (see for example [5]). Efforts have been devoted to confirm the theoretical predictions, but only semiquantitative results were produced [6,7].

Thanks to the unique capability of CBED-HOLZ lines technique to measure with high accuracy the mismatches along different crystallographic directions from a single diffraction pattern, the specimen relaxation along the thinning direction can be quantified using the isotropic elasticity theory. The method has been applied to the heterostructures, where the obtained results can be compared with x-ray Double Crystal Diffraction (DCD) data.

In order to establish a link between the bulk situation and the relaxed one, we shall start with the Hooke's equation in the isotropic elasticity theory. As shear stresses are zero in bulk samples one can write

$$u_{ij} = \delta_{ij}\frac{1}{E}[\sigma_{ij} - \nu\sigma_{ij}] \quad i,j = x,y,z; \tag{1}$$

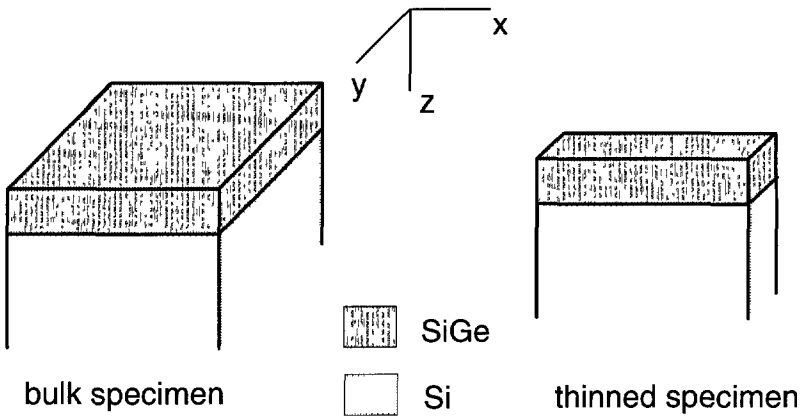

Fig. 1. — Coordinate system chosen in the model for strain relaxation in a TEM specimen.

where u_{ij} and σ_{ij} are the strain and stress tensors' components, respectively, E the Young modulus, ν the Poisson coefficient and $\delta_{ij} = 1$ if $i = j$, or $\delta_{ij} = 0$ otherwise In writing equation (1) we made use of the Einstein notation, *i.e.* summation must be done over the repeated indexes. In this case z is the [001] growth direction while y denotes the [010] thinning direction. The boundary conditions for the bulk case, where relaxation is allowed only along the z direction, are $\sigma_{xx} = \sigma_{yy} \neq 0$, $\sigma_{zz} = 0$ and $\sigma_{ij} = 0$ if $i \neq j$, and equation (1) reduce to

$$u^{\mathrm{b}}_{xx} = u^{\mathrm{b}}_{yy} = \frac{(1-\nu)}{E}\sigma^{b}_{xx}$$

$$u^{\mathrm{b}}_{zz} = -\frac{2\nu}{E}\sigma^{\mathrm{b}}_{xx} \tag{2}$$

where the apex$^{\mathrm{b}}$ indicates that the quantity refers to the bulk situation. In (2)

$$u_{ii} = \frac{a_i^{\mathrm{SiGe/Si}} - a^{\mathrm{SiGe}}}{a^{\mathrm{SiGe}}} \qquad i = x, y, z \tag{3}$$

is the strain in the SiGe film along the i direction, with the constraint of a perfect lattice match at the Si/SiGe interface in the xy plane; $a_i^{\mathrm{SiGe/Si}}$ and a^{SiGe} are the lattice parameters of the film and the corresponding bulk alloy, respectively.

The relaxed case, occurring after the specimen thinning (Fig. 1), is much more complicated because, in general, the off-diagonal elements of the stress tensor are not zero, and $\sigma_{xx} \neq \sigma_{yy}$. Nevertheless, if the shear stresses are still negligible (this assumption is particularly valid in the middle of the analysed film), the strain tensor of equation (1) can be written in a very simple form, *i.e.*:

$$u_{xx} = \frac{1}{E}(\sigma_{xx} - \nu\sigma_{yy}). \tag{4}$$

In addition, it must be observed that if the pseudomorphicity along the film/substrate interface is maintained, $u^{\mathrm{b}}_{xx} = u_x$ and equations (2) and (3) can be combined to give

$$u^{\mathrm{b}}_{zz} = 2u_{zz} + \frac{2\nu}{1-\nu}u_{yy} \tag{5}$$

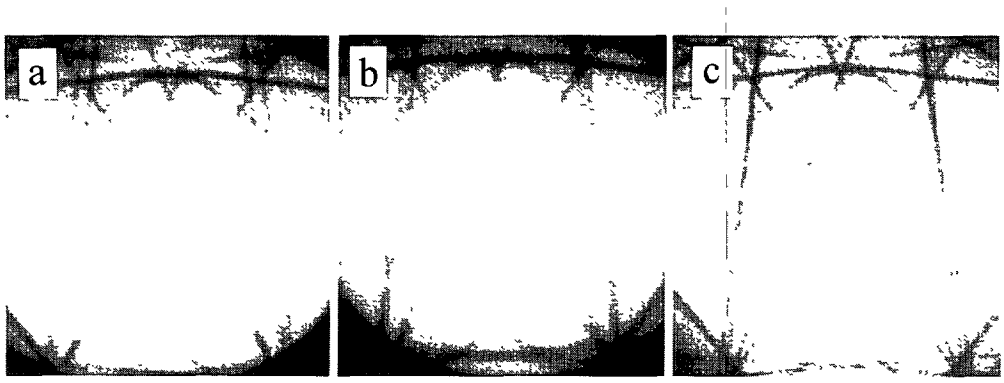

Fig 2. — < 130 > HOLZ line patterns taken in SIGEA (a) and SIGEB (b), as well as in the undeformed silicon substrate (c).

which states that the tetragonal distortion of the film in the bulk specimen can be obtained if the film strains along the growth and thinning directions are known.

It must be observed that the quantities appearing in equation (5) are strains of the film when it is forced to match the substrate lattice, which are not directly measurable by means of the CBED technique. What we actually measure, in a CBED experiment using HOLZ-lines, are the mismatch

$$m_i = \frac{a_i^{\mathrm{SiGe/Si}} - a^{\mathrm{Si}}}{a^{\mathrm{Si}}}$$

between the film and the substrate lattice parameters, which are related to the film strains through the misfit parameter

$$f = \frac{a^{\mathrm{SiGe}} - a^{\mathrm{Si}}}{a^{\mathrm{Si}}}. \tag{6}$$

Combination of equation (5) and (6) with the set of equations (2) gives the final result

$$m_z^{\mathrm{b}} = m_z + \frac{\nu}{1-\nu} m_y \tag{7}$$

which is equivalent to equation (5), now written in terms of directly measurable quantities.

It is worth noting that this equation holds strictly only in the case of < 100 > cross-sections. For different thinning directions (*e.g.* < 110 >) the formula must be modified including the proper elastic constants.

4. Results

4.1. Bulk Mismatch Measurements. — In order to verify equation (7) in the isotropic case, we performed CBED experiments with a spatial resolution of about 10 nm, on different points of TEM cross-sectioned uniform $Si_{1-x}Ge_x/Si$ heterostructures.

The samples were oriented along the < 013 > zone axis and HOLZ patterns were recorded in the silicon substrate and in the SIGEA and SIGEB layers at a distance of some tens of nm from the interface in order to minimize the relaxation induced shear strain effects The three corresponding patterns are reported in Figure 2. The width of the HOLZ lines in the shown patterns gives an indication of the minimum detectable strain in this case, *i.e.* 1×10^{-4} for Si and 2×10^{-4} for SiGe, respectively In order to deduce the SiGe lattice parameters, HOLZ pattern simulations have been performed. Even though the origin of these lines is truly

Table I. — *Lattice parameters and bulk mismatches* m_z^b *(Eq. 7) in the two investigated samples. The bulk values* $m_\perp$ *obtained from DCD are reported for comparison. SIGEM is the sample analysed by Maher et al.* [3].

Sample	a_x(nm)	a_y(nm)	a_z(nm)	$m_z^b(10^{-3})$	$m_\perp(10^{-3})$
SIGEA	0.5429	0.5444	0.5447	4.3 ± 0.4	4.25
SIGEB	0.5429	0.5432	0.5436	$1\,5 \pm 0.4$	1.33
SIGEM	0.5429	0.5443	0.5441	$3\,2 \pm 0.4$	3.10

dynamical [8], it has been shown that a kinematical approach can be adopted in the calculations with an appropriate choice of the zone axis, provided an effective voltage, which depends on the zone axis and the mean atomic number, is used instead of the actual microscope voltage. This approach allows a very fast calculation to be performed by the EMS sofware package [9]. A χ^2 test similar to that proposed by Zuo [10] has been applied to obtain the best match between the experimental and the simulated diffraction patterns.

No shear strains, which would affect the symmetry of the patterns, have been detected. The results obtained are summarized in Table I. Using relation (7), the mismatch values reported in the fifth column were obtained; these were in good agreement with the bulk mismatch value. Our method has been applied to the data obtained, in similar SiGe heterostructures, by Maher *et al.* [3], and the results are reported in Table I also (SIGEM).

From the data in this table it is also evident that the difference between the (uncorrected) CBED values and the bulk ones is about 20%. The corresponding variation in the band gap of the strained-layer heterostructures, although increasing with the Ge concentration, is generally negligible. In fact it attains a value of few meV in the case of a $Si_{0\,5}Ge_{0\,5}/Si$ heterostructure.

4.2. NITRIDE STRIPES. — The possibility of analysing small volumes of the sample suggests that patterned structures are the most important field of application of the strain determination by the CBED technique [2, 4].

This is illustrated in the present paper by measurements of the strain induced in the silicon substrate by the deposition and definition of Si_3N_4 lines (Fig. 3). In such samples a more complicated situation arises with respect to the heterostructures. In fact the film edges induce a non-homogeneous stress field (which is at least biaxial) in the substrate, in a plane perpendicular to the stripes, *i.e.* in the cross-section plane. The stress field in the substrate caused by the discontinuity of a film has been studied by Hu [11], who also derived analytical expressions for the field itself. The action of the film edge can be modeled in a very simple way by assuming that a force, lying in the film/substrate interface plane, is applied to the substrate at the film edge. CBED measurements can also be compared with numerical computations using process simulators like SUPREM IV, which allows to do *ab initio* calculations of the stripe geometry and the associated strain distribution [12].

In Figure 4 the SUPREM IV results are reported for the stripes analysed in the present study. The curves of the ε_{xx}, ε_{zz} and ε_{xz} components of the strain tensor, as a function of the position along the x direction, are reported together with a few corresponding experimental values, deduced from the CBED analysis. The overall agreement is quite satisfactory.

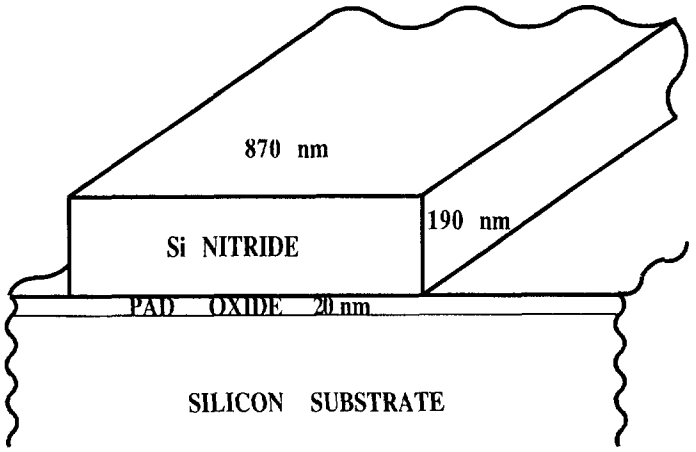

Fig. 3 — Sketch of the geometry of the Si_3N_4 line realised by photolitography onto an oxidized silicon substrate.

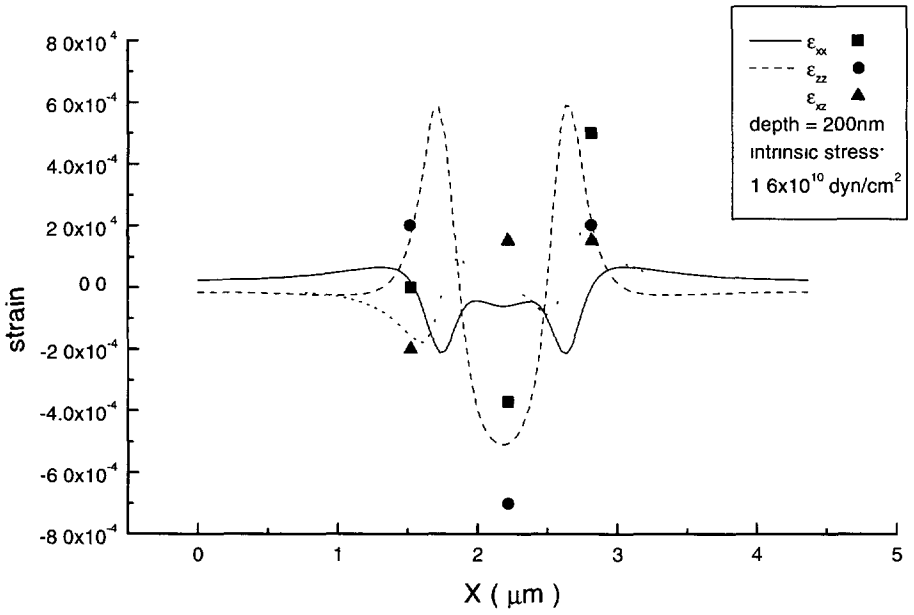

Fig 4. — Plot of the ε_{xx}, ε_{zz} and ε_{xz} components of the strain tensor computed by SUPREM IV for the structure shown in Figure 3 The superimposed symbols are the corresponding experimental results deduced from CBED analysis The abscissa x is the direction perpendicular to the stripe in the plane of the nitride/oxide interface; z is perpendicular to the wafer surface.

5. Conclusions

The CBED technique has proved to be a powerful tool to determine the local lattice strain in semiconductors with high spatial resolution. The relaxation effects in cross-sectioned heterostructures can be accounted for, thus obtaining the tetragonal distortion values for the bulk

case. In patterned structures, the components of the strain tensor can be deduced from CBED patterns taken in different points of the cross sections; the agreement found up to now with the corresponding simulated strain distribution is encouraging and suggests that this technique could be used to validate theoretical models proposed to describe the stress distribution in microelectronic devices.

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