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Influence of Experimental Parameters on the Determination of Tetragonal Distortion in Heterostructures by LACBED

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Abstract. — The LACBED technique has been applied to the determination of the tetragonal distortion in $Si_{1-x}Ge_x$/Si heterostructures, which are of great interest in the device technology. The strain determination has been performed on plan sections in an analytical electron microscope. The agreement between this strain value and the tetragonal distortion is influenced mainly by the local sample flatness and the acceleration voltage.

1. Introduction

The problem of mechanical stress in microelectronic devices is of increasing interest, due to its effect on the device performance. To investigate stress fields, a technique with high spatial resolution such as convergent beam electron diffraction (CBED), which can be performed in an analytical electron microscope, has proved to be useful [1] A complementary CBED technique, called LACBED (Large Angle CBED) [2] is now becoming of widespread use, as it allows one to determine the tetragonal distortion ϵ^T in plan sections of superlattices [3] and heterostructures [4]. In this work, the LACBED technique has been applied to the study of silicon-germanium films, coherently grown on silicon substrates, which are the core of heterojunction bipolar transistors with transit frequencies up to 100 GHz [5].

2. Experimental

$Si_{1-x}Ge_x$ alloys were grown by either solid source MBE or by CVD (low and atmospheric pressure, LPCVD or APCVD). The details on the preparation procedures have been reported in [6]. Generally, a silicon buffer is first grown on (001) silicon substrates, then the Si- Ge alloy is deposited. Finally a protective cap (5 or 50 nm thick) is grown on top of some of the heterostructures. The features of the specimens investigated in this work are reported in Table I. The germanium

Table I. — *Layer thicknesses, germanium concentrations and misfit f of the analyzed samples.*

SAMPLE	Film Thickness (nm)			Ge conc. (at.%)	Misfit f ($x10^{-3}$)
	Cap	Alloy	Buffer		
SIGE1	5	100	100	7.7	2.90
SIGE2	—	500	100	13.4	5.05
SIGE3	—	130	25	5.1	1.92
SIGE4	50	150	700	10.0	3.77
SIGE5	5	100	100	16.4	6.17

concentrations have been determined by EDS and/or RBS. All the samples show coherent Si-Ge/Si interfaces, without dislocations; as such, the misfit f with respect to the substrate can be deduced from the formula given by Dismukes *et al.* [7] (see Sect. 4 for details).

TEM [001] plan sections were obtained either by lapping 3 mm discs down to 20 μm and then ion beam milling to perforation, or by lapping to 100 micron, dimpling to 5−10 μm and finally ion milling. LACBED patterns were taken at 300 kV in a Philips CM30 TEM, by using a Gatan liquid-nitrogen cooled double tilt holder.

3. The Influence of Thickness on the Strain Determination from LACBED Patterns

LACBED techniques enable us to simultaneously observe the rocking curve approximately along the beam direction of different diffracted beams [2]. In the case of pseudomorphic, strained heterostructures, strain can be observed in LACBED patterns taken on TEM plan-view samples as a split of Bragg contours relative to crystallographic planes inclined at an angle θ with respect to the plane of the interface of the heterostructure (Fig. 1). For undeformed isostructural crystals θ is the same for both the epilayer and the substrate (Fig. 1a). In Figure 1b, where the substrate is supposed to be infinite, only the planes of the thin overlayer undergo a rotation, whereas an intermediate situation occurs if both substrate and film have a small, comparable thickness (Fig. 1c). In the bottom part of the figure a schematic of the corresponding < 120 > LACBED patterns is shown, where the dashed lines mark the < 840 > line position in the undeformed crystal. Obviously, in order to measure a strain from a LACBED pattern, the geometrical splitting, $\Delta\theta_r$, must be larger than the angular width of the Bragg contours. The angular width is approximately $2/gt_{\text{eff}}$, where g is the operating diffracted beam and t_{eff} is the projected thickness of the appropriate layer or the extinction distance ξ_g, for the kinematical and the dynamical case, respectively.

In order to express $\Delta\theta_r$ in term of the strain, we assume that the thinned specimen is constrained to lie flat and to have a total thickness $t = t_e + t_s$, where t_e and t_s are the uniform thicknesses of the epilayer and of the substrate, respectively. In addition, we suppose that the two layers are coherently matched in the plane of the interface, so the in-plane lattice parameter a_i in general differs from the relaxed lattice constants a_s and a_e, which correspond to the bulk substrate and the epilayer, respectively. In this configuration, both the epilayer and the thinned substrate are strained. Using isotropic elasticity theory, the in-plane and perpendicular strains for the two layers can be defined as:

$$\epsilon_e = (a_i - a_e)/a_i, \qquad \epsilon_e^{\perp} = (a_i - a_e^{\perp})/a_i \tag{1}$$

with $\epsilon_e^{\perp} = \epsilon_e(1+\nu)/(1-\nu)$ for the epilayer and

$$\epsilon_s = (a_i - a_s)/a_i, \qquad \epsilon_s^{\perp} = (a_i - a_s^{\perp})/a_i \tag{2}$$

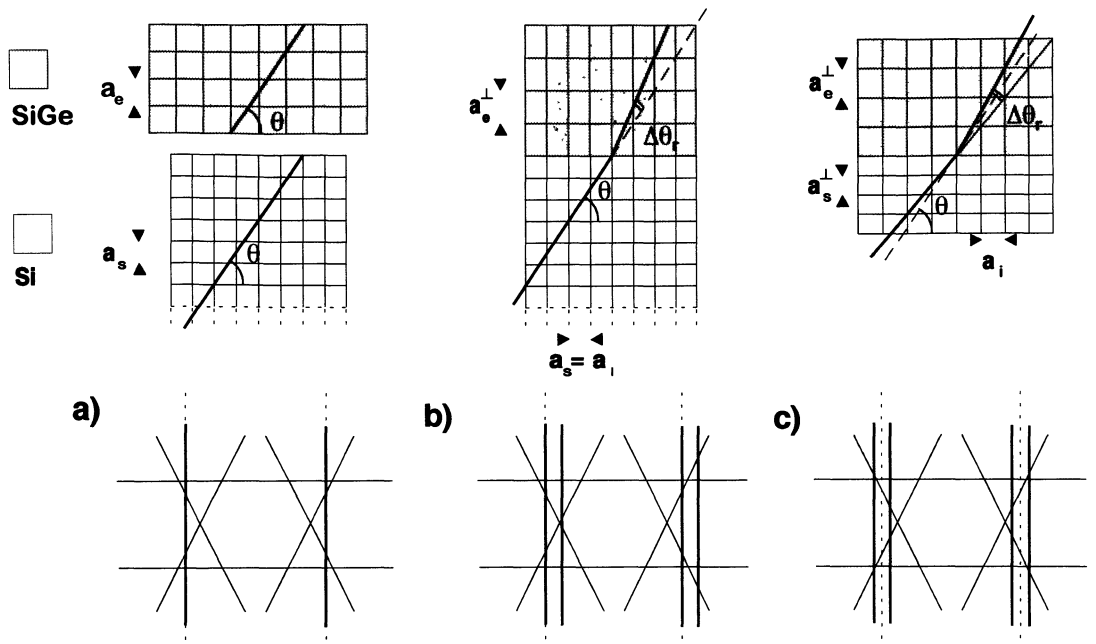

Fig. 1. — Schematic diagram showing the arrangement of the planes (top) and of the < 840 > Bragg contours in a < 120 > LACBED pattern (bottom) in relaxed crystals (a) and heterostructures with infinite (b) and thin (c) substrates, respectively. Note that in b) and c) the < 840 > contours are split by $\Delta\theta_r$. The dashed lines in the LACBED patterns mark the position of the undeformed crystal. For sake of clarity, the splitting of the other contours have not been drawn.

with $\epsilon_s^{\perp} = \epsilon_s(1+\nu)/(1-\nu)$, for the substrate, ν being the Poisson ratio. The lattice parameter a_i can be expressed in terms of a_e, a_s, t_e, and t_s minimizing the total elastic energy of the bicrystal formed by the epilayer and the thinned substrate. Such an energy can be expressed as $E = B_e t_e \epsilon_e^2 + B_s t_s \epsilon_s^2$, where B_e and B_s are the elastic constants of the epilayer and the substrate, respectively. Assuming $B_e = B_s = B$ we find a minimum for the total elastic energy of the bicrystal if:

$$a_i = (k \cdot a_e^2 + a_s^2)/(k \cdot a_e + a_s), \tag{3}$$

with $k = t_e/t_s$. The relative rotation of the planes $\Delta\theta_r$ (see Fig. 1) is given by:

$$\Delta\theta_r = (\epsilon_e^{\perp} - \epsilon_s^{\perp})\sin(2\theta)/2 = [(1+\nu)/(1-\nu)]\eta\sin(2\theta)/2 = \epsilon^{\mathrm{LACBED}}\sin(2\theta)/2. \tag{4}$$

This expression is coincident with the one previously reported by Cherns *et al.* [8] if the natural mismatch η is defined as $\eta = (a_e - a_s)/a_i$. In order to compare the strain $\epsilon^{\mathrm{LACBED}}$ with the tetragonal distortion $\epsilon^{\mathrm{T}} = [(a_e - a_s)/a_s] \cdot (1+\nu)/(1-\nu) = f(1+\nu)/(1-\nu)$ we have expressed the ratio $f/\eta = (a_i/a_s)$ in terms of f, t_e, t_s. Neglecting second order terms in f we get:

$$f/\eta = 1 + (k \cdot f)/(k + k \cdot f + 1). \tag{5}$$

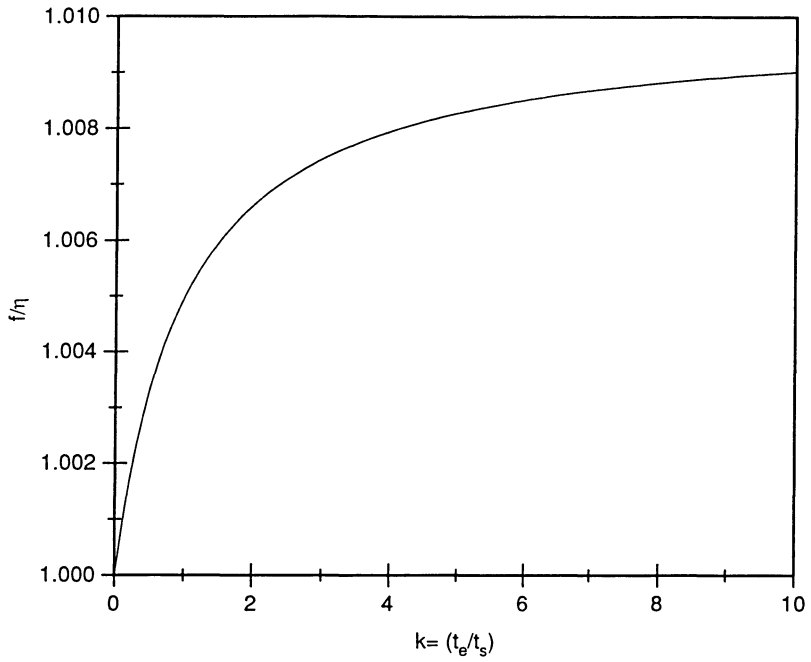

Fig. 2. — Plot of the relaxation factor f/η as a function of the ratio k between the film and substrate thicknesses. $f=0.01$.

In Figure 2 is plotted the parameter f/η *versus* k for $f=0.01$. It is evident that k has a negligible influence on f/η: for instance in the case of $t_e = t_s=100$ nm, f/η differs from unity by just 0.005.

Recently Hull [9] reported on the relaxation of the strain in plan-thinned, wedge shaped TEM samples of $Si_{1-x}Ge_x$ /Si heterostructures by means of finite element calculations. In uncapped structures he found a relaxation of the stress of the order of 2–20%, depending on the thickness of the sample. In particular, this relaxation affects the component of the stress normal to the edge of the hole in the plane of the specimen; however, it becomes almost negligible when the heterostructure is protected by a Si cap thicker than the epilayer. Probably in this latter situation the bending of the crystallographic planes produced by the relaxation of the stress normal to the hole in the thinnest area of the TEM plan section does not occur, as in the cases described in the present work.

4. Results and Discussion

In Figures 3a and 3b are reported two < 120 > LACBED patterns taken in sample SIGE1. The splitting of the Bragg contours is evident, as due to the different lattice parameters of the Si-Ge alloy and of the underlying silicon substrate. The disposition of the contours is, however, markedly different from the pattern in Figure 3c, which refers to unstrained silicon sample. The angles between the different contours as well as the distance between their intersections do not correspond to the ones in the distorted spectra. Clearly, a local sample relaxation occurs, which prevents the heterostructure from being flat. In fact, from equation (4) it turns out that $\eta = 2.1 \times 10^{-3}$ and 4.5×10^{-3} for Figures 3a and b, respectively, which are markedly different from the misfit f (Tab. I).

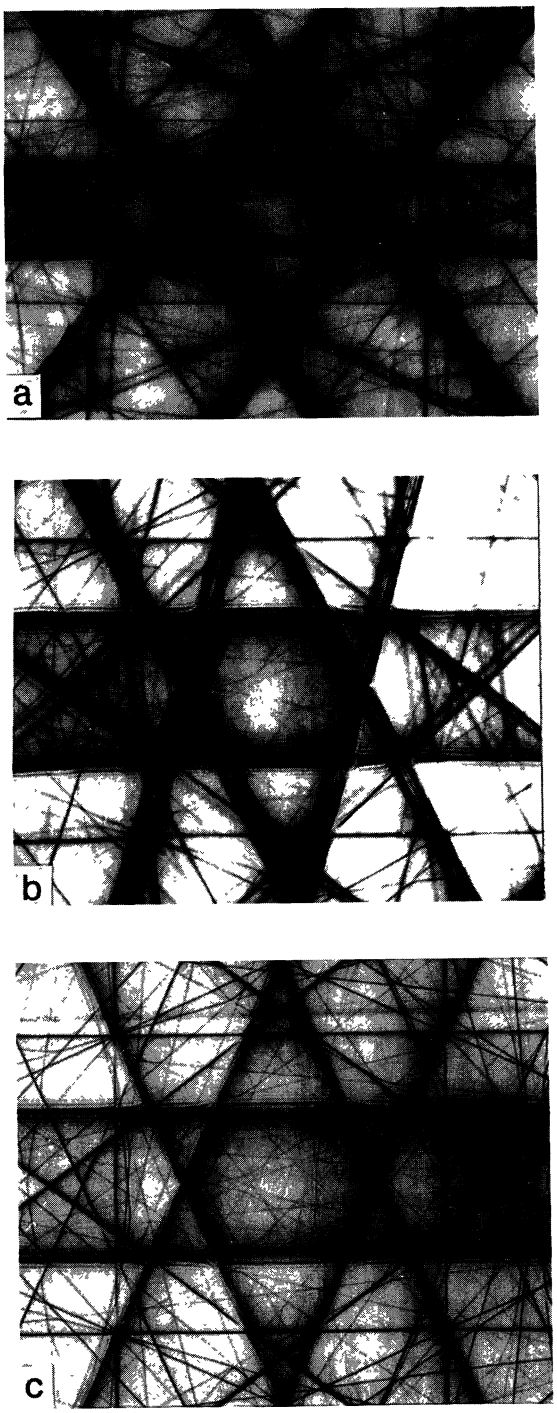

Fig. 3. — < 120 > LACBED patterns taken on plan sections of SIGE1 (a, b) and unstrained silicon (c). The patterns in a) and b) refer to regions of the sample where local bending occurs.

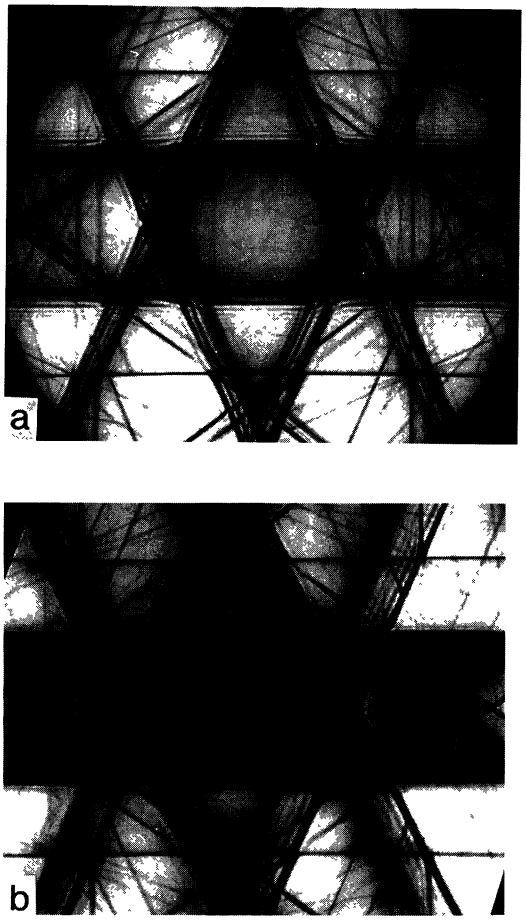

Fig. 4. — < 120 > LACBED patterns taken on plan sections of SIGE1 (a) and SIGE2 (b). Note that both the patterns are undistorted, hence suitable for the determination of the tetragonal distortion ϵ^T.

The correct situation is the one depicted in the two LACBED patterns reported in Figures 4a and 4b, which have been taken in the heterostructures SIGE1 (t_e= 100 nm) and SIGE2 (t_e = 500 nm), respectively. Here the local sample thicknesses, as measured from the rocking curve of the 400 Bragg contour, turn out to be of 430 nm and 720 nm, respectively: this yields $k = t_e/t_s$=0.3 and 2.27, respectively. As $f = 2.9 \times 10^{-3}$ for SIGE1 and $f = 5.05 \times 10^{-3}$ for SIGE2 we get, from equation (5), that f/η differs from 1 (hence ϵ^{LACBED} from ϵ^T) by 6.7 $\times 10^{-3}$ and 3.5 $\times$ 10^{-3} for SIGE1 and SIGE2, respectively. This means that ϵ^{LACBED} should practically coincide with ϵ^T.

On the basis of patterns like the ones in Figure 4 it is now possible to determine ϵ^{LACBED}. The first step is to choose kinematical contours for the strain measurements. This occurs when i) the splitting $\Delta\theta_r$ is larger than the sum of widths of the rocking curves of the substrate and the epilayer, and ii) the appropriate thickness is smaller than ξ_g for each layer. The latter condition is the more valid the higher is the acceleration voltage. The values of the various parameters, relative to the measured contours in the sample SIGE1, are reported in Table II.

Given a kinematical contour, ϵ^{LACBED} can be obtained from equation (4). These values are plotted in Figure 5 as a function of the germanium concentration in the films, which has been

Table II. — *Extinction distance, width of the rocking curve and geometrical splitting of the Bragg contours for different reflections in sample SIGE1. Acceleration voltage:* 300 kV. *Beam direction:* [120]. *The value in parenthesis (not measurable) has been assumed as equal to the one of* $g = \bar{8}44$.

g	ξ_g (nm)	$1/gt_e^{\text{eff}} + 1/gt_s^{\text{eff}}$ (x10^{-4})	$\Delta\theta_r$ (x10^{-4})	nature of the Bragg contour
$\bar{4}26$	669.2	9.46	13	kinematical
$8\bar{4}\,0$	867.7	7.92	20	kinematical
$\bar{8}\,4\,4$	1001.7	7.22	19	kinematical
$4\bar{2}\,10$	1198.1	6.46	8.3	kinematical
$\bar{8}\,4\,8$	1392.6	5.88	16	kinematical
$\bar{4}\,2\,2$	364.3	14.44	(19)	dynamical

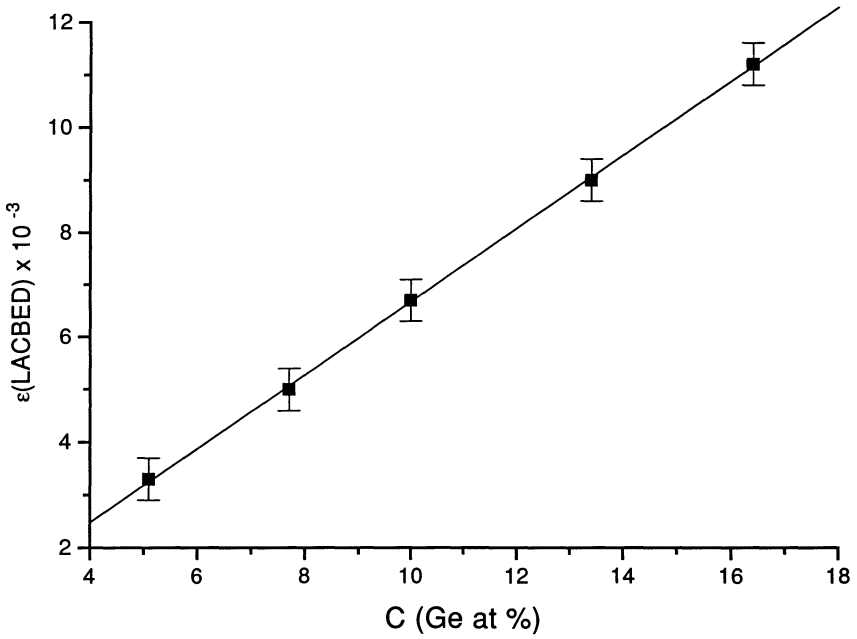

Fig. 5. — Plot of ϵ^{LACBED} *vs.* germanium concentration for the investigated heterostructures. From the slope of the straight line, a value of the parameter β, in agreement with the Dimuskes' one [7], is deduced.

deduced from Rutherford backscattering and X-ray microanalysis. The slope of the straight line in this figure gives the expansion coefficient β, as $\epsilon^{\text{LACBED}} = \beta \cdot N \cdot x(1+\nu)/(1-\nu)$, N being the atomic density and x is the Ge atomic fraction. It comes out that $\beta = (7.0 \pm 0.7) \times 10^{-25}$ cm^{3}at^{-1}, which is lower than that foreseen by the Vegard's law ($\beta = 8.34 \times 10^{-25}$ cm^{3}at^{-1}) and agrees with the one reported by Dismukes *et al.* [7]. This confirms that ϵ^{LACBED} is practically coincident with ϵ^{T}.

5. Conclusions

The LACBED technique can yield accurate values of the tetragonal distortion in $Si_{1-x}Ge_x/Si$ heterostructures provided the following aspects are taken into account:
i) The patterns must be taken in regions where the bending of the Bragg contours does not occur. In this case, the difference between the strain values determined from this method (ϵ^{LACBED} and ϵ^{T}) is quite negligible.
ii) The strain measurements should be performed on the basis of the splitting of kinematical Bragg contours, since the angular width of the rocking curve must be smaller than the splitting of the contours in the LACBED pattern. The largest splitting occurs for planes inclined with respect to the surface by angles as close to 45° as possible. In order to fulfill the kinematical requirement, high acceleration voltages are to be preferred.

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