CBED and FE Study of Thin Foil Relaxation in Cross-Section Samples of Si /$Si_{1-x}Ge_x$ and Si /$Si_{1-x}Ge_x$ /Si Heterostructures

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Summary: In order to determine residual stress/strain fields in CMOS devices and validate tools used to quantify the strain field, we first studied residual strains in Si/$Si_{1-x}Ge_x$ and Si/$Si_{1-x}Ge_x$/Si TEM samples. Because of sample thinning for TEM observations, elastic relaxation occurs and modifies the initial stress present in the bulk sample. Nevertheless, if the main parameters which play a role on the elastic relaxation process can be determined, we show that it is possible to reproduce from FE and diffraction simulations the complex profile of the HOLZ lines observed on experimental CBED patterns which makes possible the determination of the initial stress state.

1 Introduction

The determination of mechanical stresses is of major interest to the microelectronics industry. Whether they are undesirable because they generate defects in components, or voluntarily introduced to optimize certain electrical properties, they have to be perfectly controlled. Because of component size (critical size <180 nm), a strain measurement technique with a very high spatial resolution combined with a very good sensitivity has to be used.

CBED has been widely employed for many years in order to determine local strains in many systems such as microelectronic devices and epitaxially strained layers. Nevertheless, one of the major problems related to these measurements concerns the sample thinning down to electron transparency which induces a stress relaxation via the formation of free surfaces perpendicular to the milling direction. In such cases, lattice planes bend near interfaces giving rise to HOLZ line broadening [1,2] because of inhomogeneous variation of lattice parameters. Conventional strain analysis using quasi-kinematical measurement of HOLZ line shifts for such broadened lines becomes impossible and new models have to be developed [3-6].

Because elastic relaxation causes HOLZ lines broadening we tried to prevent plane bending by depositing a cap layer with mechanical properties similar to those of the substrate. This allowed us to perform conventional strain measurements in certain areas. We combined finite element (FE) simulations and CBED measurements in order to validate the elastic model used in our mechanical simulations and electron diffraction simulations. In this way, we evaluated the importance of initial stress, sample geometry and thickness on stress relaxation in Si/$Si_{1-x}Ge_x$ and Si/$Si_{1-x}Ge_x$ /Si strained heterostructures.

2 Samples and CBED Acquisition

The studied $Si_{1-x}Ge_x$ layers were deposited on (001) silicon substrates. The Ge composition was of 1, 3 or 6% in order to obtain a different residual strain level in each sample. Layer thickness was about 100 ± 5nm. Moreover, in order to prevent plane bending at least in some parts of the layer, a fourth sample was fabricated by depositing a 100 nm silicon cap layer on a $Si_{0,97}Ge_{0,03}$ layer. Table 1 summarizes expected strains and stresses in $Si_{1-x}Ge_x$ layers.

Ge composition (%)	$a_{Si1-xGex}$	*misfit (%)*	*biaxial* ε_{xx} *(%)*	ε_{zz} *(%)*	*biaxial stress* σ_0 *(MPa)*
1	0,54330	0,042	-0,042	0,030	-75
3	0,54375	0,125	-0,125	0,096	-225
6	0,54443	0,241	-0,241	0,173	-450

Table 1. Strains and stresses in bulk $Si_{1-x}Ge_x$ layer versus Ge concentration.

In order to check the influence of sample preparation technique on relaxation process, transmission electron microscope (TEM) specimens were prepared by two different methods: tripod polishing and ion milling (PIPS).

CBED experiments were mainly performed on a JEOL 2010F microscope operating at 200kV and equipped with an imaging filter (Gatan GIF 200). A FEI Tecnai microscope equipped with a STEM was also used to record CBED patterns profiles exhibiting sharp HOLZ lines. In a previous paper [7], we showed that it is possible to reach a good experimental sensitivity on strain measurement (~ $2,5.10^{-4}$) with this microscope even if no filtering system is used.

All patterns were acquired along the <230> Si zone axis. The samples were studied in a thickness range between 200 and 500 nm. To determine the sample geometry, sample thickness measurements at different positions in the lamella were performed. Sample thickness values were determined by fitting the experimental intensity profile of the diffracted disk 004 with the theoretical one given by dynamical diffraction theory [7, 8]. The precision of this method is about 1% on the crystalline thickness measurement.

3 FE Modelling and Electron Diffraction Simulation

To quantitatively measure strains in samples and/or regions giving sharp HOLZ lines, quasi-kinematical simulations (ASAC™ software) have been used to measure the shift of these lines. In the case of broadened HOLZ lines a combination of FEM and diffraction calculations was employed. For this latter point, the methodology proposed by Clément et al. [3] was applied. It consists of a) thin lamella preparation with a given geometry, b) acquisition of CBED patterns with broadened lines (Fig. 1a) from which we extract experimental rocking curves, c) use of elastic relaxation model (FEM) to determine displacements field for thin sample, d) rocking curves calculations on the basis of FE results and e) comparison between simulated and experimental rocking curves.

FE simulations were performed in Finite Element Analysis Program (FEAP) [9] using a custom model based on anisotropic hyperelasticity [10]. Residual strains were calculated by introducing a lattice mismatch due to Ge composition and assuming an elastic accommodation of the layer. Elastic stiffness coefficients and lattice parameters in $Si_{1-x}Ge_x$ were calculated using Vegard's law. In our approach, we used 3D FE calculations to simulate plane strain state present in TEM sample. Size of finite elements was optimized to get the best compromise between accuracy and computation time. It was found that a point resolution of 2.5 nm is the most suitable.

Rocking curves calculations were done with home-made routines written in SciLab© (INRIA-ENPC) software using dynamical electron diffraction approach (two-beam approximation). To compare experimental and simulated rocking curves, a dedicated procedure has been developed to automatically estimate the error and determine the best simulated profile among a set of displacements extracted at different distances *d* from the interface. The best match between the simulated and experimental profiles corresponds to the lowest χ^2 value (Fig. 1b).

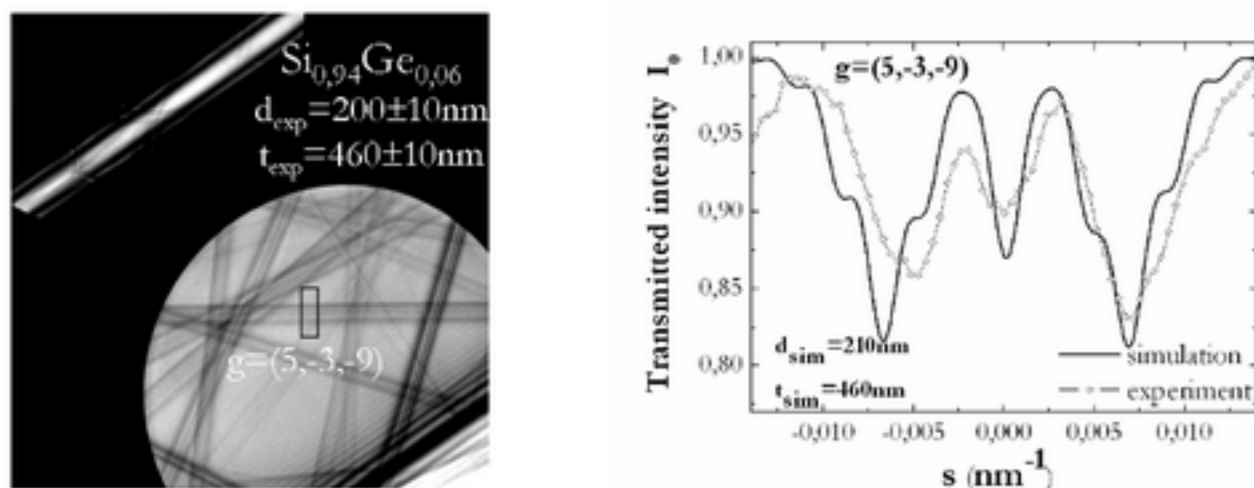

Fig. 1. a) CBED pattern with broadened diffraction lines. **b)** Best match between simulated rocking curve and experimental one for the (5 -3 -9) reflection (χ^2=0,073).