Strain characterisation at nanoscale using Transmission Electron Beam Based techniques

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INTRODUCTION

In the past decade, stimulated by the demand of the semiconductor industry, several new Transmission Electron Microscopy (TEM) based techniques have been proposed to measure strain. Presently, these TEM based techniques seem to be the only ones that can provide at the same time the high spatial resolution and the high precision required in nanoelectronics: resolution of about one nanometer and precision $\Delta a/a$ approaching $1e^{-4}$, where a is the lattice parameter. In a first part, a short overview of the TEM techniques for strain measurement is performed. In a second part, we present the solution that has our preference: Nanobeam Precession Electron Diffraction (**N-PED**).

OVERVIEW OF TEM TECHNIQUES FOR STRAIN MEASUREMENT

Since the built of the first TEM in Germany in 1931, TEM has greatly developed. TEMs are now quite complex instruments, but thanks to computers, their operations are becoming simpler when no software bugs happen. There are two main ways of working with a TEM: either a broad beam is made with the condenser lenses and a global image of the sample is performed (what we call "pure TEM" or shortly "TEM") or a tiny beam is made and scanned over the sample (this is Scanning TEM: STEM). Historically, STEM was harder to develop and lagged behind "TEM" for a long time. "TEM" provides nearly lived images and the image contrast can be easily enhanced by putting an aperture in the diffraction plane of the objective lens, method which is known as diffraction contrast imaging (**DCI**). In the last ten years, STEM has gained more and more interest thanks to important improvements and to our point of view seems more adapted for nanoelectronics applications[1].

As shown in table 1, there are many TEM techniques that can be used to measure strain. Here we cannot discuss on all these techniques and only some pros-and-cons are given in the last column of table 1. **The more effective way to measure strain in TEM is to measure directly the local lattice parameters** and there are 3 ways to do that. (i) Either the lattice planes are directly visualized and the variations of distances between the lattice planes i.e. strain, are directly measured. This happens in high resolution (HR) either **HR-TEM**[2-3] or **Z-contrast HR-STEM**[1-3]. Due to the necessity to have enough pixels between lattice planes and due to the limited number of pixels in the images, these techniques have generally a limited field of view and a limited precision. (ii) Or the lattice parameters are directly measured in the reciprocal space. This is what is achieved in all diffraction based techniques: **CBED**[3-4] (Convergent Beam Electron Diffraction), **NBED**[5] (Nanobeam Beam Electron Diffraction), **N-PED**[6-7], and **EBSD**[8] (Electron Back Scattering Diffraction). Although EBSD is a SEM (Scanning Electron Microscope) technique, we have put it in table 1 because it can can provide maps of the 9 components of the deformation matrix (6 strain components and 3 rotation terms) and because a transmission mode is under development and should improve its spatial resolution. The diffraction based techniques have generally a good precision but the realization of maps can be time consuming. (iii) Or a "parameter" which is linked to the lattice parameters is measured. In holography based techniques (**DFEH**[3-9] **:** Dark field Electron Holography or **DIH**[10] **:** Dark Field In-line Holography) this is achieved by measuring the phase maps of some diffracted beams. The sample have to be tilted out-of a zone axis in order to enhance a given diffracted beam **g** and it is the phase of this diffracted beam which is very sensitive to the

local lattice parameters. In **DCI**[11], the "parameter" is the amplitude of a diffracted beam. Unfortunately the amplitude is not very sensitive to strain and also depends on sample thickness and sample composition. **LACBED**[12] (Large angle Convergent Beam Electron diffraction) is a mixture of diffraction and image. LACBED and DCI seem more adapted for having a global view of the sample. In the next part, we present the solution we have developed and adopted: **N-PED**[6].

TABLE 1. Some characteristics of electron microscopy techniques (see text for meaning of abbreviations, except for SMF which is Scanning Moiré Fringes) that can visualize or measure strain field. Values in bold are a good point for the technique. Underline values means a weakness. 1D-strain (respectively 2D-strain and 3D-strain) means that only one component (respectively 4 and 9) of the deformation tensor are measured easily. FIB is Focus Ion Beam.

Technique	Precision	Spatial resolution	Field of view	Thickness	Comments
DCI [11]	not quantitative	2 nm	< 1µm	10-400 nm	Just for seeing defects, quantification is difficult
HR-TEM [2-3]	0.2 %	**0.2 nm**	100 nm	< 50 nm	Visualization of atomic columns, but need of very good TEM lamella (FIB is not good), poor precision
HR-STEM [1-3]	0.1 %	**0.2 nm**	400 nm	**5 - 400 nm**	**Good for maps of large strain (> 1%)**
DFEH [3-9]	**0.02 %**	2 nm	**1 µm (map)**	~ 100 nm	**Very good for 1D-strain** but off-axis, need of a near-by reference, complexity for 2D-strain
DIH [10]	**0.01 %**	**1 nm**	**1 µm (map)**	**1 - 400 nm**	No need of reference, **very good for maps of 1D-strain** but off-axis, image series
LACBED [12]	qualitative	~3 nm (?)	**1 µm (map)**	> 100 nm	**Good for an overview** of strained regions, poor resolution, quantification is difficult
CBED [3-4]	**0.02 %**	**0.5 nm**	**1 µm** (scan)	> 200 nm	Precise but only for thick crystal, off-axis, complex, however **3D information and 3D-strain is possible**
NBED [5]	0.06 %	3 nm	**1 µm**	< 200 nm	**On-axis, simple** but not robust. good for profiles
N-PED [6-7]	**0.01 %**	**2 nm**	**1 µm** (scan)	**1 - 400 nm**	**Simple, robust and versatile** (any thickness and orientation) for 2D-strain but maps require times
SMF [13]	0.1 %	2 nm (?)	**1 µm**	**5 - 400 nm**	Just for 1D-strain, works on traditional devices
EBSD [8]	**0.01 %**	20 nm	**microns**	surface	**9 components of deformation**, surface analysis, poor resolution, but a transmission mode is developed

NANOBEAM PRECESSION ELECTRON DIFFRACTION

N-PED is very similar to CBED and NBED: a small electron beam is made and diffraction patterns (DP) are acquired at different positions of this electron beam. One DP must be acquired in a region where no strain is assumed and this DP serves as a reference pattern, which can be acquired in a region far from the region of interest or in a different sample. The differences between N-PED, NBED and CBED are the diameter (d), the convergence angle (α) and the precession angle (α_p) of the beam. In fact there is a compromise between spot size and beam convergence: it is impossible to have a small parallel beam and the only way to reduce the beam size is to make it more convergent (see fig. 1). In classical CBED, diffraction spots are nearly tangent (i.e $\alpha \sim 3$ mrad) and the probe size is quite small (d ~ 0.5 nm), but the diffraction disks do not have a uniform intensity (Fig. 1b). In NBED, as the disks are smaller this non-uniform intensity is less visible (Fig. 1a), but it is still present making the exact location of the diffraction maxima less precise and making NBED not a very robust technique (variations of intensity in the disks changes with thickness, orientation and composition and give noise in the profile of Fig. 1e). In N-PED the incident beam is rotated by a small angle around the observation direction, i.e. it is precessed, and a descan is applied after the sample in order to bring back the diffracted beams to their unprecessed positions. The advantages of the precession for strain measurement appear in Fig. 1. When precession is on, (i) more diffracted spots are visible, (ii) the uniformity of the spot intensity is improved and (iii) a better precision is obtained by locating the edges of the disks and (iii) a more convergent beam can be used leading to a smaller probe and a better location of edges (Fig. 1d). We implemented precession on two microscopes: a JEOL 2010 FEF microscope equipped with the Astar system from Nanomegas and a TITAN ultimate microscope with precession made by FEI. On the JEOL 2010FEF, mapping is rather fast (15 minutes for a map of 100x50 points) but the quality of the DPs are not as good as on the TITAN: on the Astar system, the bigger images are 512x512, DPs have some distortions, we got some issues with the stability of the alignments and we could not get probe smaller than 3 nm in the precession setting[7]. On the TITAN, we had to use our own scripts to acquire maps larger than 2 Gb with a 2Kx2K Gatan Ultrascan CCD camera and the precession speed is limited to 0.1s. The advantage of the TITAN setting is to be very stable

and to be able to go back and forth between N-PED and HR-STEM. **Presently the drawbacks of N-PED are its speed and amount of data:** for instance in fig. 2, 100x50 diffraction patterns of 1Kx1K pixels were acquired in about 90 minutes and occupy about 12 Gb on hard disk. As can be seen in Fig. 2, very smooth maps of the four components of the strain tensor can be obtained with N-PED. A compromise has to be found between precision, spatial resolution and diffraction size. By increasing slightly the probe size to 3 nm, a precision of 9e-5 was obtained in the Si substrate with 2Kx2K patterns. However in most cases, patterns of 512x512 pixels should be sufficient. **N-PED is a technique that is simple to use and very robust**: thick or thin crystals can be analyzed; in the presentation, we will show how N-PED was successful for analyzing devices based on nanowires.

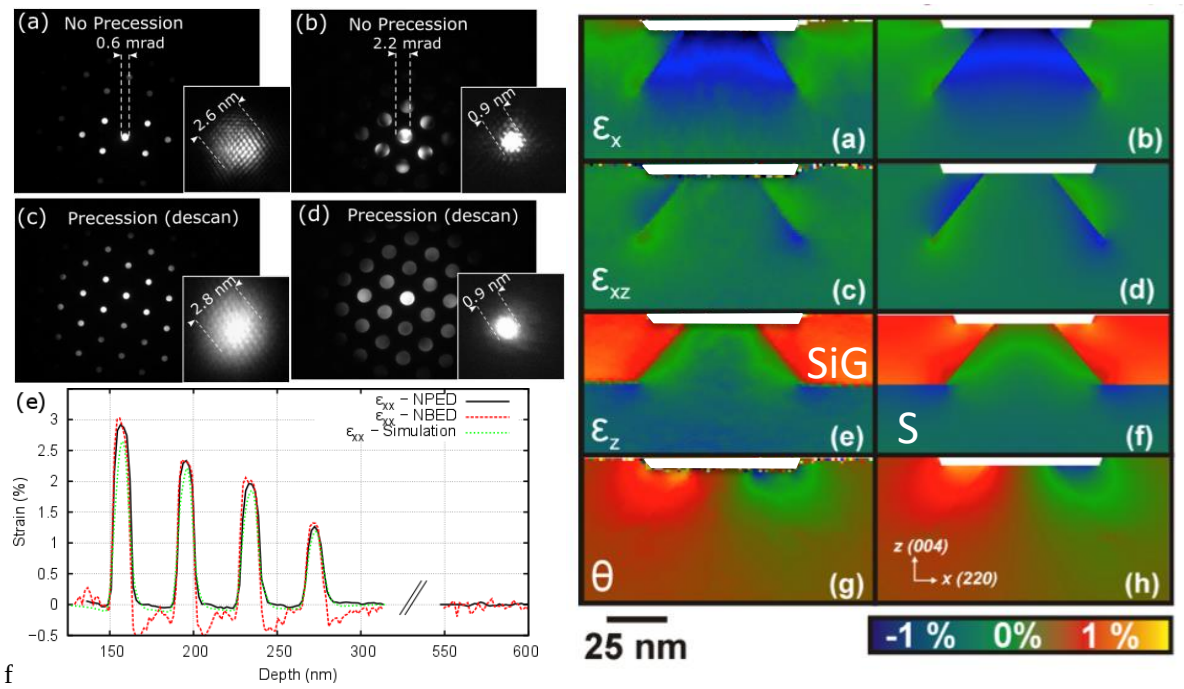

FIGURE 1. (left) (a-b-c-d) Diffraction patterns and the associated images of the electron being going through a perfect Si crystal observed along a <110> direction. (e) Three strain profiles (1 simulation and 2 experimental ones) taken along the growth [001] direction of 4 Si_xGe_{1-x} /Si layers (x=0.2, 0.31, 0.38 and 0.45) (see [6] for more details)

FIGURE 2. (right) Strain maps obtained in a device containing SiGe stressors and a Si_3N_4 layer above the SiO_2 layer (the whited boxes) above the channel. (a-c-e-g) Experimental maps obtained from N-PED. (b-d-f-h) Maps obtained by Finite Element simulations: to reproduce experimental data, a -1.9 GPa stress was put in the amorphous Si_3N_4 layer.

REFERENCES

1. J. L. Rouviere, E. Prestat et al, *Journal of Physics: Conference Series* **471** 012001 (2013).
2. F. Hue, M. Hytch, H. Bender et al, *Phys. Rev. Lett*. **100 (15)** 156602-4.3.(2008).
3. A. Béché, J.L. Rouviere , J.P. Barnes, D. Cooper *Ultramicroscopy* **131** 10-23 (2013).
4. A. Spessot, S. Frabboni, R. Balboni, A. Armigliato, *Journal Of Microscopy* **226 (2)** 140–155 (2007).
5. A. Béché, J.L. Rouvière, L. Clement, J.M. Hartmann, *Appl. Phys. Letters* **95(12)** 123114 (2009).
6. J. L. Rouviere, Béché A et al., *Appl. Phys. Lett.* **103** 241913 (2013).
7. M. Vigouroux et al. *Appl.Phys. Lett.* **105** 191906 (2014).
8. S. Villert, C. Maurice et al., *Journal of Microscopy* **233** 290-301 (2009).
9. M. Hÿtch et al. *Transmission Electron Microscopy in Micro-nanoelectronic* John Wiley & Sons (2012).
10. C. T. Koch, V.B.Özdöl and P. A. Van Aken *Appl. Phys. Lett.* **96** 091901 (2010).
11. D. D. Perovic, G. C. Weatherly and D. C. Houghton *Phil. Mag. A*, **64:1**, 1-28 (1991).
12. H. H. Liu, X.F. Duan, Q. X. Xu and B.-G. Liu, *Ultramicroscopy* **108** 816-820(2008).
13. S. Kim, S Lee, Y. Oshima et al., *Appl. Phys. Lett.* **102** 161604 (2013).

KEYWORDS

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