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Optimized FIB silicon samples suitable for lattice parameters measurements by convergent beam electron diffraction

L. Alexandre [a], K. Rousseau [a,1], C. Alfonso [a,*], W. Saikaly [b], L. Fares [c], C. Grosjean [c], A. Charaï [a]

[a] TECSEN-UMR 6122, Université Paul Cézanne, Aix-Marseille, Faculté des Sciences de Saint-Jérôme, 13397 Marseille cedex 20, France
[b] CP2M, Université Paul Cézanne, Aix-Marseille, Faculté des Sciences de Saint-Jérôme, 13397 Marseille cedex 20, France
[c] ST Microelectronics/Rousset, RCCAL, Zone Industrielle de Rousset, 13106 Rousset Cedex, France

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Abstract

The aim of this paper is to check the effect of artefacts introduced by focused ion beam (FIB) milling on the strain measurement by convergent beam electron diffraction (CBED). We show that on optimized silicon FIB samples, the strain measurement can be performed with a sensitivity of about 2.5×10^{-4} which is very close to the theoretical one and we conclude that FIB preparation can be suitable for such measurements in microelectronic devices.

To achieve this, we first used CBED and electron energy loss spectroscopy (EELS) which provide a procedure permitting an exact knowledge of the sample geometry, i.e. the thickness of both amorphous and crystalline layers. This procedure was used in order to measure the FIB-amorphized sidewall layer. It was found that if the FIB preparation is optimized one can reduce this amorphous layer down to around 7 nm on each side. Secondly different preparation techniques (cleavage, Tripod™ and FIB) permit to check if the surface damaged layer introduced by FIB influences the strain state of the sample. Finally, it was found that the damaged layer does not introduce measurable strain in pure silicon but reduces appreciably the quality of the CBED patterns.

Keywords: Convergent beam electron diffraction; Electron energy loss spectroscopy; Focused ion beam; Damaged layer; Amorphized layer thickness; Specimen thickness measurement; Strain measurement

1. Introduction

Size reduction of integrated circuits poses more and more technological problems for the microelectronic industry. One of them, related to geometries and stack layers of different materials during the fabrication process, is the development of important local stresses in the substrate which affect device performance and reliability. It becomes therefore necessary to quantitatively determine these stresses in order to control their effects. Because of components size (critical dimension $<0.13\ \mu m$), stress/strain analysis techniques with a high spatial resolution and a good sensitivity are required. Among these techniques, convergent beam electron diffraction (CBED) is really suitable for local strain measurements because it combines a sensitivity similar to X-ray diffraction ($\Delta a/a \sim 10^{-4}$) with a spatial resolution defined by the electron beam size, on the order of few nanometres.

This technique is so sensitive to local variations of lattice parameters that the obtained results may be affected not only by stress relaxation but also by sample preparation method (polishing, ionic milling) which may modify the original strain state in the thin lamella. Hence, some precautions must be taken whatever the sample preparation technique used. One of the most recent and efficient methods is focused ion beam (FIB) milling which uses a high energy gallium ion beam. However, it is known to introduce damaged layers on both surfaces of the thin lamella which might affect the crystalline thickness of the sample, modify its stress state or simply reduce the CBED pattern signal to noise ratio. This may lead to large uncertainties on strain measurement and hence potential misinterpretation with simulated data given by dynamical diffraction and Finite Element simulations. There are many studies (Ishitani et al.,

* Corresponding author. Fax: +33 4 91 28 87 93.
E-mail address: claude.alfonso@univ-cezanne.fr (C. Alfonso).
[1] Present address: Serma Technologies, Avenue de Rochepleine, BP 333, 38523 Saint-Égrève Cedex, France.

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1998; Kato et al., 1999; Delille et al., 1999; Langford and Petford-Long, 2001; Mc Caffrey et al., 2001; Rubanov and Munroe, 2004; Gao et al., 2004) dealing with FIB damage and many attempts to reduce it but, to the best of our knowledge, no extensive work has been published on its effect on CBED precision. Indeed, the presence of amorphized/implanted layers on the surface of the thin foil can impact measurements either by reducing image quality and/or by directly deforming the crystal. So, first, it is very important to minimize the thickness of the damaged layer and then to quantify its effect, if there is any, on local lattice parameter determination using CBED.

The first step was to measure the thickness of the damaged layer. Several authors have proposed direct methods to visualize and measure it (Kato et al., 1999; Langford and Petford-Long, 2001; Mc Caffrey et al., 2001; Rubanov and Munroe, 2004; Gao et al., 2004). For our purposes, we chose the indirect approach coupling CBED in two-beam conditions with electron energy loss spectroscopy (EELS). These two methods are widely used to determine the thickness of TEM samples (Egerton, 1996; Mac Gillavry, 1940) and have been often associated either for calibrating the inelastic mean free path of electrons (Mayer et al., 1997; Plitzko and Mayer, 1999) or for thickness measurement itself (Delille et al., 1999). Even though this procedure is indirect, it has the big advantage to be applicable at any stage of strain characterization on the same area of any sample for future analyses. The second step of this work was to qualify FIB-prepared samples for high accuracy CBED measurements. More particularly, we checked if the surface damaged layer introduced by FIB could influence the strain state in the sample.

2. Materials and methods

2.1. Thinning methods

All samples were prepared from standard (0 0 1) industrial silicon wafers. The different preparation techniques used were: FIB (Overwijk et al., 1993), Tripod™ polishing (Benedict et al., 1990) and Small Angle Cleavage Technique (SACT) (Walck and Mc Caffrey, 1997). The two latter methods were chosen because not only they allow to have an almost negligible surface damaged layer but also because they offer a wide range of thicknesses on the same sample. The SAC wedge was first cleaved along {1 2 0} plane and then along {1 1 0} plane giving rise to a tip whose angle is about 18°. For FIB samples, we used the so-called liftout technique. The first steps of our milling procedure followed the standard ones which consist of creating two trenches (~15 μm long, ~5–10 μm wide and 5 μm deep) around the zone of interest and then of alternatively milling each side of the lamella with a beam voltage of 30 kV, an incidence angle of ±1.2° and a successively lower beam current density (between 5000 and 100 pA). When the desired specimen thickness was reached (between 200 and 300 nm), we used "a cleaning step" which consists of tilting the sample to an angle of ±7° and using a very low beam current (~50 pA at 10 or 30 kV) for a short period of time on the whole surface. This step is employed in order to remove redeposition and to reduce the damaged layer thickness generated during the previous steps. The sample was then cut with a beam voltage of 30 kV–300 pA normal to the surface to avoid sidewall-ion implantation. Finally, the lamella was placed onto a Formvar-coated Cu TEM grid using a micromanipulator under an optical microscope.

2.2. Acquisition conditions of CBED patterns and EELS spectra for thickness measurements

Sample thickness measurement can be either done by EELS or CBED. CBED patterns were acquired in 0 0 0/0 0 4 two-beam conditions at about 10° from the ⟨2 3 0⟩ zone axis direction. We used a convergence semi-angle of 9 mrad in order to avoid disc overlapping and a camera length allowing to observe both discs in the 1 K × 1 K CCD camera field of view. The probed region was about 7 nm. Distances in reciprocal space have been calibrated by measuring the distance between the centers of 0 0 0 and 0 0 4 discs. Afterwards, we used the method which consists of fitting the experimental intensity profile of the diffracted disk 0 0 4 with the theoretical intensity given by dynamical diffraction theory based on papers by Kossel and Möllensted (1939), Mac Gillavry (1940), and several other authors (Kelly et al., 1975; Allen, 1981) and, more recently, described in details by Delille et al. (2001). This profile depends on two parameters which are the extinction distance ζ_g and the crystalline thickness of the sample t_c.

The information we get from EELS is expressed by the ratio t_{tot}/Λ, where t_{tot} is the total thickness of the lamella and Λ the inelastic electron mean free path (IMFP) (Egerton, 1996). This ratio is very sensitive to experimental conditions particularly the collection angle, crystal orientation, channelling effects, etc. (Botton et al., 1995; Bardal and Lie, 2000). We did not study the influence of these parameters but we made sure that all spectra were acquired in the same conditions. Moreover, directly after the CBED pattern acquisition, an EELS spectrum from the same zone is recorded with the same two-beam orientation, the microscope still in diffraction mode and without any objective aperture, the transmitted disk of the CBED pattern centred on the spectrometer entrance aperture. Spectra were recorded with an energy dispersion set at 0.5 eV per channel over 500 eV energy range. The camera length was of 700 mm and the spectrometer entrance aperture of 3 mm which implies an acceptance angle of 1.1 mrad. This configuration leads to an acceptance angle largely lower than the incident one which means than the angular distribution of the intensity can be very different from the one predicted by commonly used relations giving the angular dependence of inelastic cross-section. The effect of incident-beam convergence could be expressed in terms of an effective collection angle depending on the incident and acceptance angles and on the energy loss (Egerton, 1996). We did not try to take into account such a correction. As already explained, our aim was to develop a procedure applicable at any stage of strain characterization for future analyses making sure to analyse the same region. Hence, we choose to determine a value of