ESRF	**Title** Dewetting dynamics of strained-silicon on insulator (sSOI) by GISAXS and GIXD	**Experiment number**: **32-03-681**
Beamline: BM32	**Date of experiment**: From: 08/07/2009 to: 16/07/2009	**Date of report**: 28/07/2009 *Received at ESRF:*
Shifts: 21	**Local contact(s)**: Dr Tobias Schülli	

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The purpose of this experiment was to study *in situ* the dewetting kinetics of thin Si(001) films on SiO_2. Dedicated samples based on the SOI (silicon on insulator) technology have been studied. The key parameters of this dewetting transition are temperature and film thickness. More particularly it has been proposed that Si nanocrystals agglomeration during dewetting is a stress-driven morphological instability caused by the thermal expansion mismatch between Si and SiO_2. In order to put in evidence the potential role of interfacial stress we used samples fabricated on the basis of the recent strained silicon-on-insulator (sSOI) technology. This technology allows tuning the lattice parameter of the Si film from 0 to +1.2% on the same SiO_2 amorphous material. Grazing Incidence Small Angle X-ray Scattering and Grazing Incidence X-ray Diffraction (GIXD) measurements have been performed in real-time in order to characterize the structure and the morphology of the Si thin film before and during dewetting.

First the Si(100) surface of SOI and sSOI substrates have been cleaned *ex situ* by standard chemical etching ('shiraki' cleaning) followed by a final cleaning (mixture of $HCl:H_2O_2:H_2O$, 3:1:1) to protect the surface with a thin (1-2 nm) oxide layer before introducing the sample in UHV. Then the thin oxide layer has been removed in the main chamber increasing slowly the temperature up to 900°C. This desoxydization procedure has been checked in real-time by RHEED looking at the appearance of the Si(1x2) surface recontruction. This has been confirmed by GIXD showing a clear in-plane signal from the (1x2) reconstruction typically observed in the case of a flat Si surface.

After desoxydization we have characterized the Si film on the different SOI samples (thickness, strain state). Out-of-plane scans have been performed giving rise to Kiessing fringes nearby Bragg peaks providing an accurate measurement of the thickness of the Si films before dewetting (see Fig.1). The mean strain state of strained Si samples could also be obtained with great precision from the in-plane and out-of-plane position of the Bragg peaks. The Bragg peaks are not only shifted compared with the underlying Si wafer but also broader showing that the strain state of the film is not homogeneous.

The kinetics of dewetting has been studied alternating GISAXS and GIXD measurements on three different samples: unstrained Si (20 nm thick), 0.8%-strained Si (9 nm thick) and 1.2%-strained Si (10 nm thick). Fig.2 displays for instance the evolution of GISAXS and GIXD as function of time for the 1.2% strained Si film.The appearance of tilted scattering rods on GISAXS patterns is a clear indication of the agglomeration of the film into Si nanocrystals exhibiting extended (113) facets and smaller (111) facets. Changing the sample azimuth by 45° it has been shown that nanocrystals exhibit also (110) facets. Simultaneous measurements of radial scans by GIXD indicate that the Si film stress relaxes immediately and completely as nanocrystals are formed during

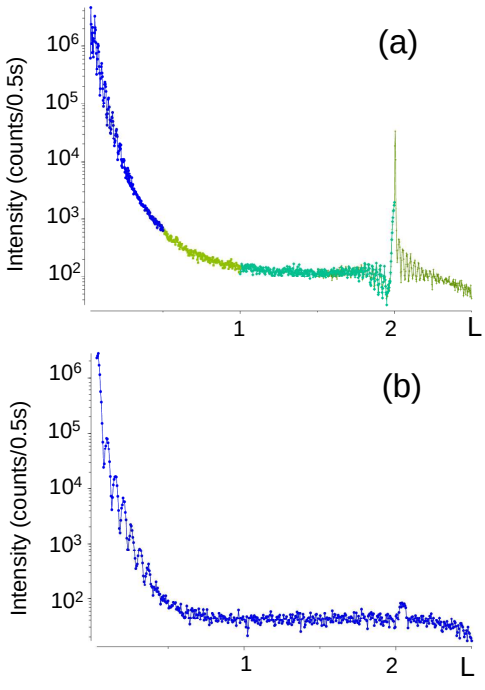

Fig.1 (22L) scans for (a) unstrained and (b) 0.8%-strained Si thin films. Kiessing fringes provide an accurate value of the Si thickness (resp. 20.6 and 9.2 nm.)

dewetting. At the end of dewetting we completed the GIXD and GISAXS results by mapping in-plane the reciprocal space around (220) Bragg peak and by measuring the full 3D reciprocal space around (000) by GISAXS rotating the sample by step of 1° over 90°. No other facets have been detected.

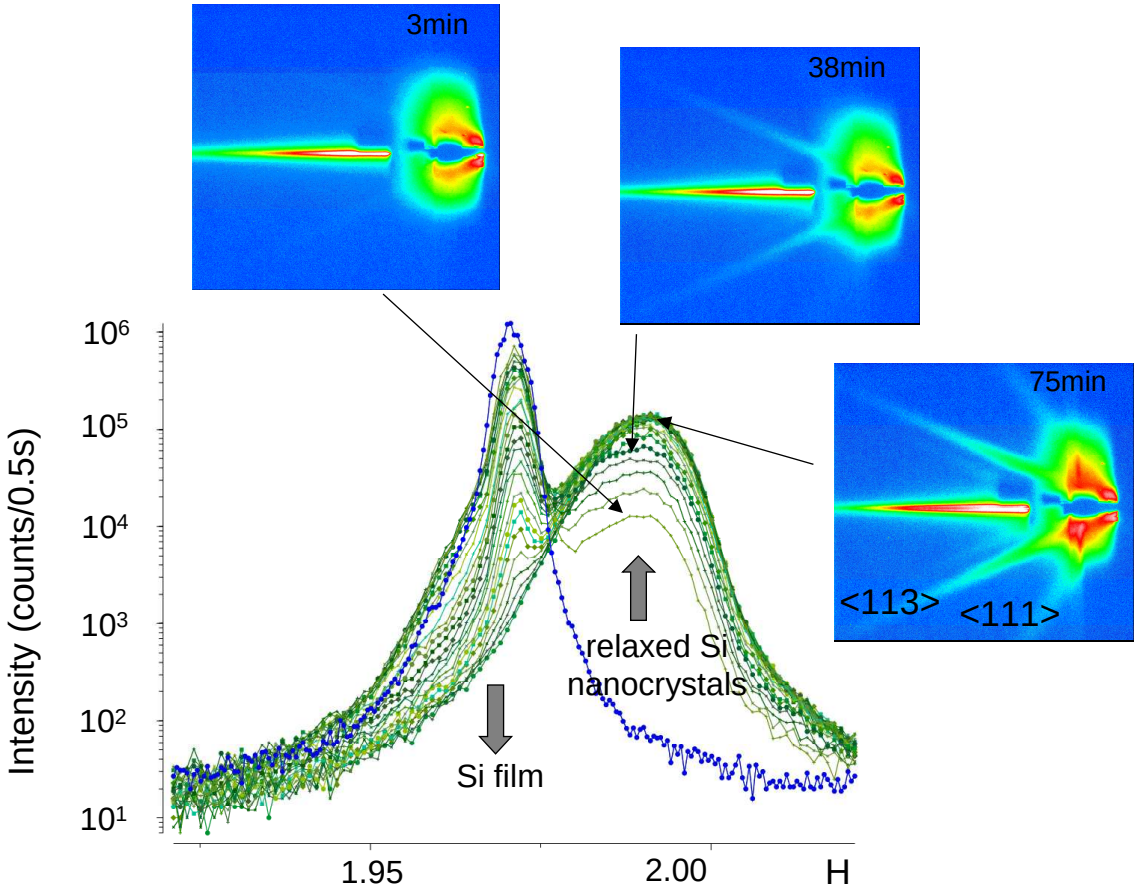

Fig. 2. Overview of the evolution of GISAXS (beam aligned along <110>) and GIXD (scan along (hh0)) as function of time during dewetting of a 1.2%-strained Si film at 950°C. The dewetting starts by the appearance of a bump at (220) corresponding to relaxed Si nanocrystals. Simultaneously, extended strikes arising from <113> and <110> facets appear on the GISAXS patterns. The intensity of this bump from Si nanocrystals increases while the Si film Bragg peak decreases. The time scale for complete dewetting is about 1h.

Interestingly out-of-plane scans reaveled that forbidden reflexions like (222), (442) and (420) are measurable (see for instance Fig.1a at L=2). This effect is usually asigned to an asymmetric charge distribution of Si due to covalent bonding of valence electrons or anharmonicity of the thermal vibrations. However in our case additional effects should be considered arising from the Si/ SiO_2 interface and from the strain state which can break the symmetry of the nearest neighbour boundings. Qualitative understanding of the Si thin film structure and interface can be gained from this data set. More quantitative measurements of the CTRs should be necessary to characterize precisley the Si thin film and interface structure.

In summary sample preparation and dewetting of SOI and sSOI have been well reproduced on the SUV apparatus. GISAXS and GIXD measurements have been alternatively performed *in situ* and in real time (steps of 3 min) providing a detailed characterization of the evolution of Si nanocrystal facets and internal relaxation. Dewetting of Si/SiO_2 gives also rise to very anisotropic organization of the nanocrystals as observed by *ex situ* AFM. However, this could not be observed by GISAXS due to the large length scale of the dewetted structures (100nm - 1µm). From GIXD we have also obtained a qualitative characterization of the interface structure for unstrained and strained Si samples. Detailed calculation of the interface structure must now be performed to compare with our results. This is part of our ANR-PNANO DEFIS project in collaboration with theoreticians of CINaM (P. Ganster, G. Tréglia). From the GISAXS and GIXD characterizations after dewetting we can fully determined the morphology (facets) and the internal relaxation of the nanocrystals. The broadness of the Bragg peaks of the nanocrystals is a clear indication that Si nanocrystals are completely relaxed on average but many different strain states still exist after complete dewetting.