ESRF	**In situ GIXD study of the displacement field induced by steps on vicinal Si surfaces**	**Experiment number**: Si-1586
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The elastic properties of Si play a major role in many mechanisms occurring at Si surfaces. For instance, surface morphology instabilities are supposed to be strongly dependent on the step-step elastic interaction. The reduced coordination of the atoms belonging to the step edges give rise to lattice distortions that elastically propagate into the bulk. Since the seminal work of Marchenko [1] it is known that this displacement field can be calculated by modelling the steps by a linear distribution of localised forces. Recently, some of us have shown that this force distribution can be extracted from careful Grazing Incidence X-ray Diffraction (GIXD) experiments [2].

Our goal was to use well-selected vicinal Si surfaces to (i) measure the lattice displacement field and analyse the results in terms of equivalent elastic force distribution, (ii) study the evolution of the force distribution as a function of the step density and zone axis orientation. Indeed, a good knowledge of the elastic properties of Si surfaces is the basis for a further control of the nanostructuration of these surfaces.

Following our preliminary experiments at the lab, the Si(7 7 10) surface appeared to be ideally suited to perform such experiments since (i) the clean surface exhibits a high degree of order with (ii) a faceting instability from triple step bunching to single monoatomic steps at the 1x1→7x7 surface phase transition temperature. This surface thus appeared to be promising to study the elastic dipole force model as a function of the step density since crossing the 1x1→7x7 temperature transition is enough to tune the surface morphology from a single step network to a triple step network.

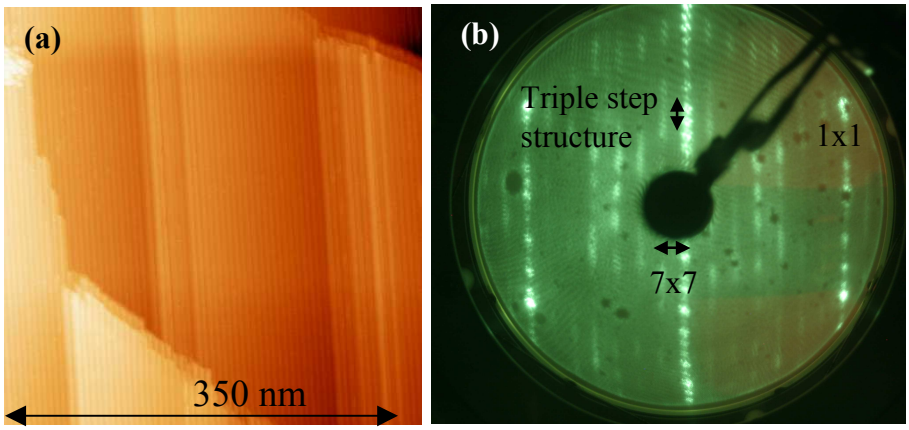

Fig. 1 (a) STM image of the Si(7 7 10) surface at RT. A periodicity of 5.7nm corresponding to the triple step structure is clearly measured on large terraces.(b) LEED pattern of the corresponding surface. The 7x7 surface reconstruction and the periodicity of the triple step structure are highlighted

At the ESRF the surface has been prepared using the same experimental protocol as in our lab. It means that Si single crystals (20x2x0.3 mm^3) were prepared

by *ex situ* chemical cleaning (ethanol and acetone) then outgassed *in situ* at 900 K followed by several cycles of flash annealing at 1500 K in UHV to remove the oxide layer. The flashes were performed by a DC direct current through the sample in the <110> direction, i.e. parallel to the step edges to prevent the surface from being unstable *via* step bunching [3]. A STM image and a LEED pattern of the sample surface prepared at our lab are given in Fig 1.

At the ESRF, the *in situ* cleaning procedure has been checked by following the surface structure quality measuring the intensity of a Crystal Truncation Rod (CTR) nearby a Bragg peak. During outgassing of the sample and sample holder at increasing temperature, the intensity of the CTR arising from the surface decreased and vanished (Fig. 2a) indicating a high roughening of the surface. After repeated flashes at 1500 K, the CTR reappeared and got thinner and more intense (Fig. 2b) in agreement with a reordering of the surface structure/morphology. However despite repeated efforts (5 samples have been tested) to improve the surface structure and morphology, we did not manage to get sufficiently extended CTR perpendicular to the surface plane to extract an accurate description of the elastic displacement field induced by the triple step structure (our calculation shows that the CTR must at least extend to a l value corresponding to the Bragg peak of the next neighbor CTR to extract the strain field induced by steps). On the best sample, the CTR intensity could be measured only up to 1/3 of the required extension. Fig. 2c shows a map of the reciprocal space. Wide scattering rods arising from nanometric size (111) and (113) facets are put in evidence (Fig. 2c). However no satellite from the superperiod of the triple step organization has been measured whereas from our LEED measurements well defined satellites are observed (Fig. 1b). Moreover, we did not measure any rod associated to the 7x7 reconstruction, in the k-direction. All these features clearly indicate that the surface remained very rough, which means that it was not correctly cleaned.

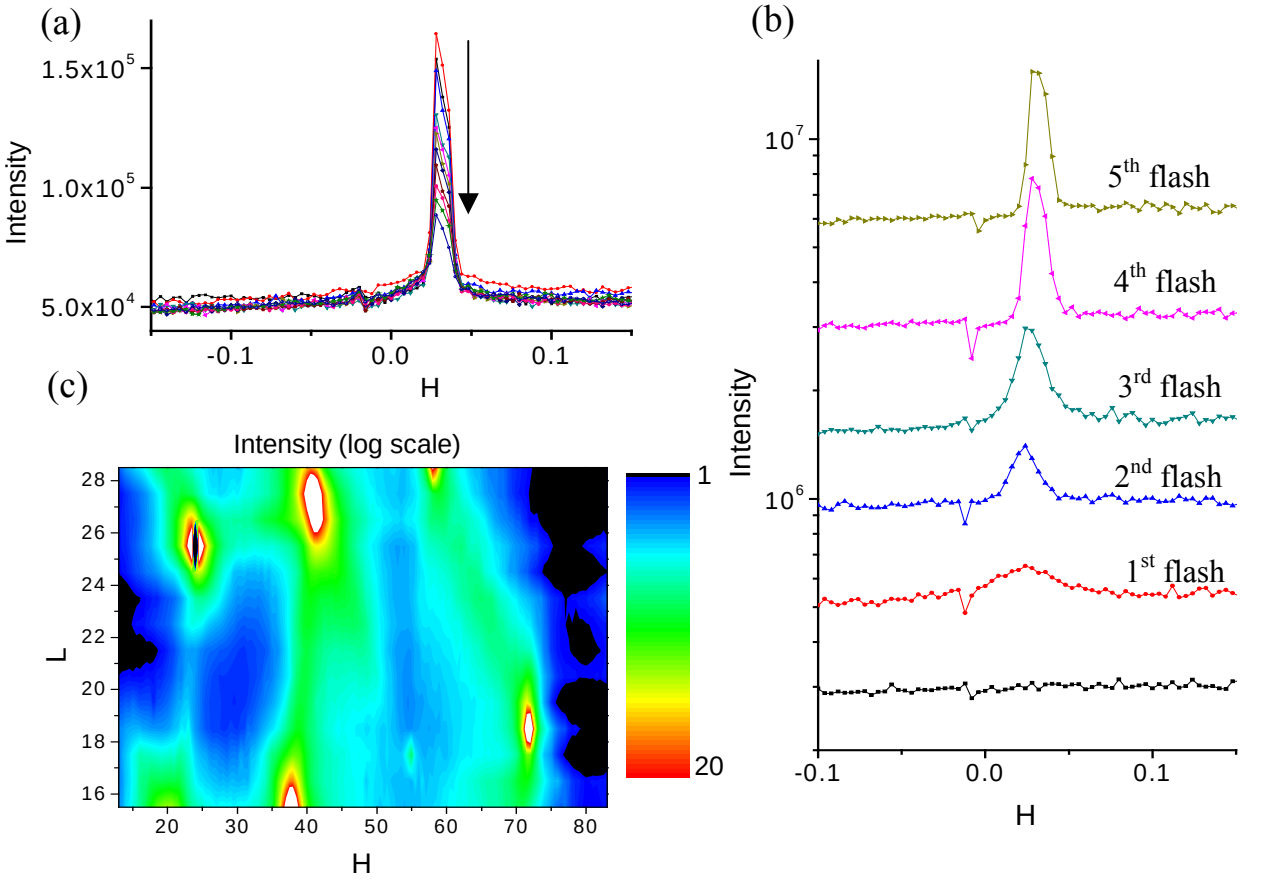

Fig. 2 (a) radial scan (H-scan) close to a Bragg peak. The intensity decreases as a function of temperature. (b)After a few flash at 1500 K (10 seconds) the intensity of the CTR increases and gets thinner. (c) (H -2 L) map of the reciprocal space. The Bragg peaks are connected by streaks originating from (111) and (113) facets. However no superstructure from the triple step organization is visible.

Thus, we have not been able to measure the strain field induced by steps on the Si(7 7 10) surface. The origin of the problem has been clearly identified: in spite of a rather good vacuum, 1.4 10^{-10} mbar at ambient temperature, the sample holder outgassed during the flashes at 1500 K due to its large size promoting the formation of silicon carbide at the Si surface. Indeed, the more encouraging

results have been obtained at the end of the run, when the outgassing of the sample holder has been long enough. However, this unavoidable surface contamination remained prohibitive to get a surface reconstruction. Thus the measurement of the strain field induced by steps on Si surfaces (e.g. Si(7 7 10) but also Si(1 1 8), Si(1 0 5), see proposal) with the ID3 sample holder needs to avoid the flash procedure currently used in our lab. The alternative approach consists in a *ex situ* 'shiraki' cleaning followed by a final 'Piranha' clean (mixture of H_2SO_4 and H_2O_2, 3:1 to get a surface with a thin (1-2 nm) oxide layer before the transfer into the UHV chamber. Then, *in situ*, the remaining oxide layer can be removed by slow deposition of Si (1 ML/min) at 700°C. This low temperature cleaning procedure is enough to prevent the sample holder from outgassing and contaminating the sample surface (contrary to high temperature flash). To check the efficiency of the new procedure, it has been proposed to clean the surface on a similar UHV chamber (with the same sample holder) located at the ESRF in which it is also possible to characterize the surface by LEED. Of course to succeed in cleaning Si surfaces, great care will be payed on the preliminary baking of the UHV chamber as well as on the outgassing of the sample and sample holder. Notice that such a cleaning procedure of Si surfaces has been successfully checked on ID3 by other groups [4].

At last, notice that due to the high crystalline quality of Si crystals and the high ordering of clean Si(7 7 10) surfaces as shown by LEED and STM measurements, there is no doubt that once clean surface will be reached, GIXD experiments will give us the strain field close to the steps and thus the corresponding force distribution.

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