Pyrometer calibration, sample mounting, and sample processing for Silicon (111)-7×7 reconstruction

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Sample preparation for UHV consists of a series of steps mandatory to be followed in order to get a clean surface. We present here a short recipe from pyrometer calibration to the final step: that of proving that the reconstruction really occurred, namely the 7x7 pattern of the Si(111) surface revealed by LEED and UPS.

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1. Introduction

Si and Ge surfaces are amongst the best characterized of all semiconductor surfaces. The 1×1, 2×1 (for cleaved surfaces), 5×5 (by annealing cleaved samples in the range 300-600 °C and 7×7 (for surfaces heated above 600 °C) symmetry are characteristic for Si(111)(1,2). The 7×7 reconstructed surface was one of the most intriguing problems of surface physics (3). It is the most stable (energetically favoured) Si (111) surface. All others are metastable.

In order to obtain such surface one should anneal the Si(111) sample above 950 °C. A similar result is obtained using the method of Ar sputtering followed by an annealing period (4, 5). Farnsworth was the first to report the 7×7 reconstructed LEED pattern of Si(111) surface (6). There are many recipes for preparing the 7×7 reconstructed Si(111) surface (7, 8). In comparison to Si(100)-2×1 surface the Si(111)-7×7 surface presents a complex geometry structure as described by DAS (dimer-adatom-stacking fault) model – Si dangling bonds with different reactivity towards incoming adsorbates due to their different geometric and electronic structure (9). The Si(111) -7×7 is of constant interest (9, 10, 11, 12, 13).

2. Experimental details

We performed our experiment in two UHV chambers operating at the same pressure. One has both the XPS/UPS facility, the other one has only the LEED and the STM facility. Two identical samples were mounted on identical pods and processed using the same recipe presented below. The 7×7 reconstruction is to be revealed by UPS and LEED.

The annealing temperature for the Si samples was read through an IR window. The first step was that of checking for a Si coating on the UHV window. In order to do this on a piece of Ta foil (0,2 × 17× 5 mm^3) - same size as that of our Si sample- was spot-welded the chromel-alumel thermocouple (CHAL - 003, OMEGA Engineering, Inc.).

For heating the sample an AC source was used. Comparing the plot of the thermocouple temperature *vs.* the Pulsar II pyrometer (Model No. 7000 GP – temperature range 400-3000F) temperature for the glass window (ie the view-port), and the plot of the thermocouple temperature *vs.* the Pulsar II pyrometer temperature for the IR window was found that the IR window is clean as retrieved from the two slopes of the two graphs – Fig. 1 and Fig. 2. Then the experiment was repeated with a Si sample and the pyrometer was calibrated for an emissivity of 0,57. The Si sample was heated by using a DC XKW 40-75 XANTREX power supply (0-40 Volts, 0-75 Amps).

A first sample of p-Si (boron as dopant – Virginia Semiconductor Inc.) double side polished, (5×21 mm^2), thickness 300 μm ± 25 μm, orientation <111> ± 0,5°, 0,7-1,2 Ohm-cm resistivity was cut from the Si wafer and wiped with methanol using a Q-tip then degreased by sonication in MeOH for 5 min. then in acetone for 3 min. and again in MeOH for 5 min. and finally rinsed with deionized water (ρ= 18 MΩcm). Then the oxide was grown ((H_2O-NH_4OH(30%)-H_2O_2(30%) 4:1:1 at 80 °C for 5 min.), then H_2O-H_2O_2(30%)-HCl(37%) 1:1:3 as long as reaction continued (~20 min.)) then rinsed repeatedly in deionized water and dried by using pre-purified N_2.

The sample was mounted on a pod by using two Ta shims (5×5×0,5 mm^3) in order to avoid hot spots which are always responsible for the sample melting at elevated temperatures. The sample was manipulated by using only teflon tweezers. Outgassing via resistive heating lasted for 12 hours at 700 °C until the pressure was in the low 10^{-10} Torr range. Oxide and carbide removal was done by repeatedly flashing in the 1147-1177 °C range for a total time of ~ 2 min. During flashing the pressure never increased above $1×10^{-9}$ Torr. The cooling process was rapid from the highest flashing temperature to 850 °C (this was done by decreasing the current in 0,2 amps increments until the temperature reached 850 °C) and then more slowly from 850 °C to room temperature (the current was constantly decreased in 0,1 amps increments). During the entire process a close inspection for hot spots was carried

out. The fact that during the heating process the colour of the Si sample showed an increasing intensity from sides to the middle suggested a correct way of mounting and hence of heating it. The pressure recovered from < 1×10^{-9} Torr to < 10^{-10} Torr within 25 seconds of cooling.

A second Si sample – p-Si (same characteristics) was processed using the above recipe in the UHV chamber that has both the XPS and UPS facility.

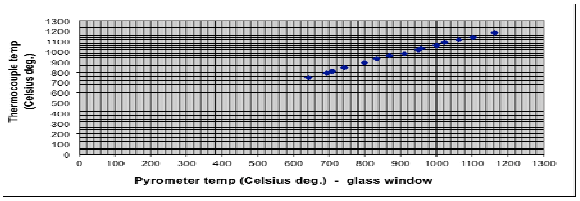

Fig. 1. The thermocouple temperature vs. Pulsar II pyrometer temperature (glass window).

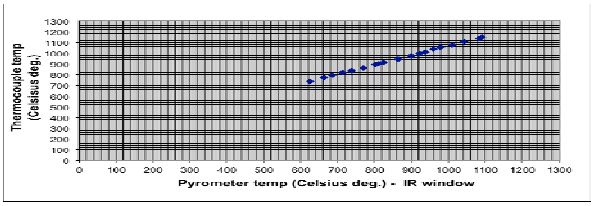

Fig. 2. The thermocouple temperature vs. Pulsar II pyrometer temperature (IR window).

3. Results and discussion

A second sample of p-Si (boron as dopant – Virginia Semiconductor Inc.) double side polished, (5×21 mm^2), thickness 300 μm ± 25 μm, orientation <111> ± 0,5°, 0,7-1,2 Ohm-cm resistivity mounted in different chamber - having the XPS and UPS facility and operating at the same pressure was processed using the same recipe described here. Then an XPS spectrum was performed showing a surface free of C and O as contaminants (Fig. 3).

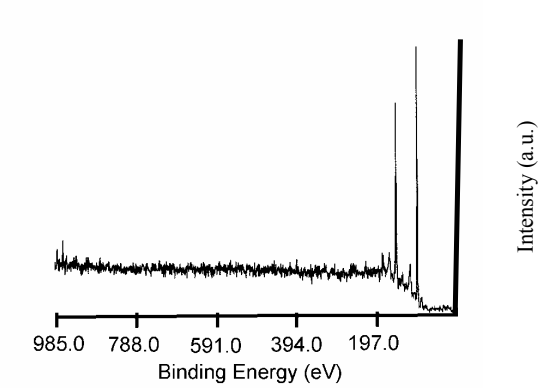

Fig. 3. XPS spectrum of clean Si(111)-7×7. It shows a surface free of C and O as contaminants. Peaks centered around 150.96 eV and 98.7 eV correspond to Si 2s and Si 2p respectively. UPS spectra proved that the clean surface was reconstructed 7×7.

The wide scan UPS spectrum of clean Si - same sample on which we performed the XPS spectrum - shows the Si valence band (Fig. 4). For binding energies between 0 and -2 eV the UPS spectra of clean Si(111)-7×7 consist of electron emission from surface states already discussed in Fig. 4. For energies higher than 2 eV the emission comes from bulk states (peaks at -4 eV and -8 eV). The data are consistent with those reported in the literature by Martenson et al. (14) and Uhrberg et al. (15).

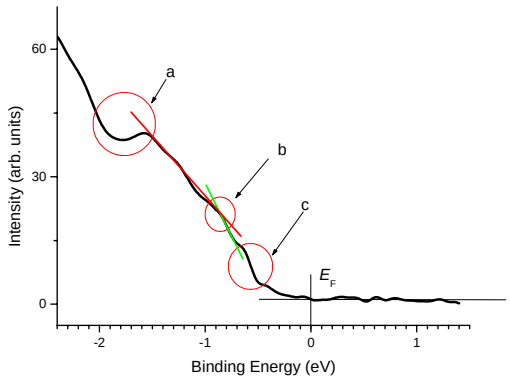

Fig. 4. The UPS spectrum for the bare Si(111) surface shows three surface states (15): 1. the surface state (a) at 1.8 - 2 eV related to the backbonds between the Si adatoms and the three Si atoms directly beneath them, 2. the surface state (b) at ~1 eV related to the filled dangling bond states situated on the rest atoms, and near the Fermi level E_F (0 eV), and 3. the surface state (c) at 0.4 - 0.6 eV related to a half-filled dangling bond state located at the adatom that forms part of the 7×7 reconstruction. The intensity of the state at 0.4 - 0.6 eV is a good indication of the quality of the 7 × 7 surface reconstruction.

After flashing, the extent of the 7×7 reconstruction for the first sample (the one that was processed in the UHV chamber having the LEED and STM facility) was studied by low energy electron diffraction (LEED) using Reverse View LEED-RVL 900 (Fisions Instruments) with a beam voltage of 84V. Sharp spots revealed an excellent 7×7 pattern (Fig. 5). The 7×7 reconstruction occurred on both samples as revealed by UPS and LEED.

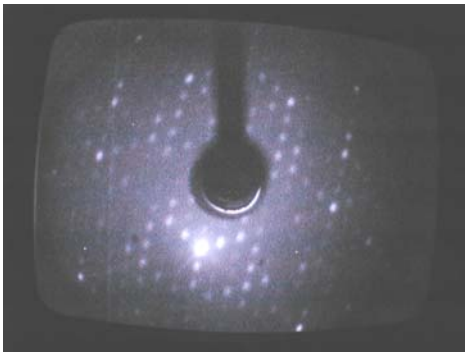

Fig. 5. The 7×7 pattern showing the correct reconstruction for the Si(111) surface.

4. Conclusion

A complete approach toward the 7×7 reconstruction of the Si(111) surface was described. Sample melting usually occurs when pyrometer reading is affected by the Si film deposited on the UHV chamber. A mandatory step should be (in the case of resistive heating) that of comparing the plot of the thermocouple temperature *vs.* the pyrometer temperature (glass window / IR window) in order to check for a correct reading through the IR window. We found that our method of pyrometer calibration, sample mounting, and sample processing was highly reproducible. The intensity of the surface state at 0.4 - 0.6 eV in the UPS spectrum (related to a half-filled dangling bond state located at the adatom that forms part of the 7×7 reconstruction) is a good indication of the quality of the surface reconstruction.

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