LEEM and XPEEM studies of C-AFM induced surface modifications of thermally grown SiO_2

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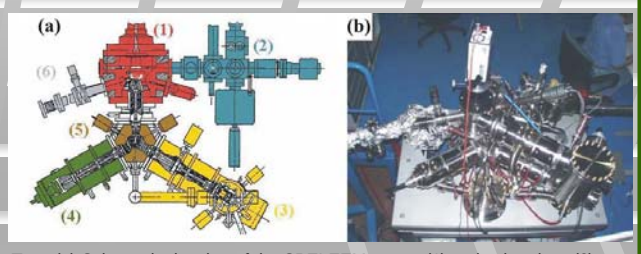
Abstract

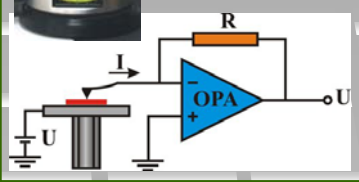
The process of local anodic oxidation, where a positive voltage is applied between a sample and a conductive probe, is well understood. Here, Conducting Atomic Force Microscopy (C-AFM) induced surface modifications of thermally grown Si oxides are investigated for *opposite* (i. e. negative) sample bias. Also at this polarity, surprisingly the appearance of protrusions is observed. To obtain information on the nature of these protrusions, low energy electron microscopy (LEEM) and x-ray photoemission electron microscopy (XPEEM) measurements were performed at the Nanospectroscopy Beamline at the synchrotron radiation source Elettra in Trieste, Italy. With LEEM we find that the protrusions are heavily charged after their formation, but this charge is annihilated after exposure of the sample to short pulses of synchrotron radiation. After extended x-ray exposure, a radiation induced desorption of the structures as well as a desorption of the thermal oxide is observed. The spectra obtained from XPEEM images at different photoelectron kinetic energies reveal that the structures formed by C-AFM are chemically homogeneous, and that they are caused by the growth of additional SiO_2 on the sample surface.

Experimental

Left: Digital Instruments Multimode AFM.



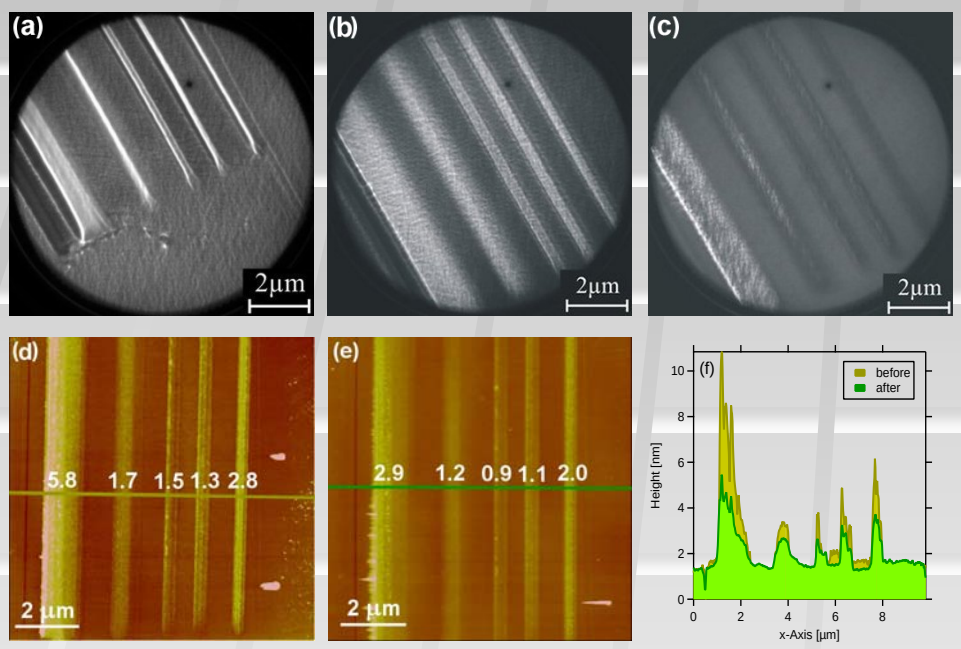
Top: (a) Schematic drawing of the SPELEEM setup**:** (1) main chamber, (2) sample preparation chamber with load lock, (3) image column, (4) electron gun, (5) beam separator, and (6) connection to the synchrotron beamline. (b) picture of the SPELEEM.



Left: Draft of the basic C-AFM setup with the sample (red) mounted to the sample holder and sitting on the moving piezoscanner. The voltage is applied to the sample holder. The current through the tip is amplified and converted to a voltage by the current-to-voltage converter (OPA). The feedback resistor (R) determines the amplification factor.

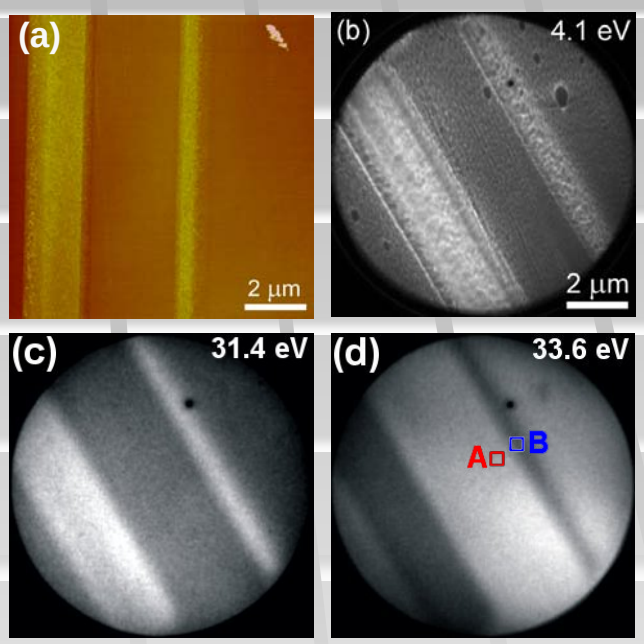
Exposure to X-rays

Short term exposure (few seconds):
Annihilation of negative charges deposited in the lines during creation.
Long term exposure (several 10 minutes):
Photodesorption of parts of the lines and of the thermal oxide.

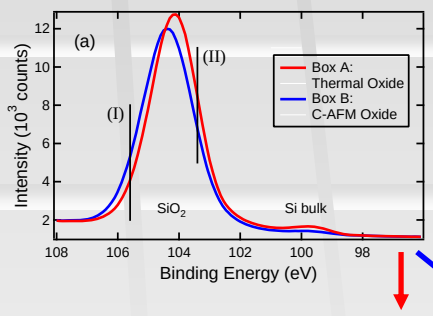


(a) MEM image obtained after writing lines by C-AFM. Electron energy: 3.0 eV. The image exhibits very strong contrast. This effect could be due to negative charges within the fabricated structures. (b) MEM image at 2.7 eV of the same structures after irradiation with 89.4 eV photons for 2 minutes. The contrast in the image is strongly reduced, and structures and background can be focused to at the same time. (c) Same as in (b) after extended x-ray irradiation at 89.4 eV for 30 minutes. A further contrast reduction is observed. (d) AFM image of the same structures, obtained after writing the lines by C-AFM. z-scale: 15 nm. (e) AFM image of the same sample area, after a total exposure time of 32 minutes to x-rays with a photon energy of 89.4 eV. z-scale: 15 nm. The numbers in (d) and (e) give the average height of individual lines in nanometers before and after exposure as measured with AFM. (f) The height profiles along the lines indicated in (d) and (e).

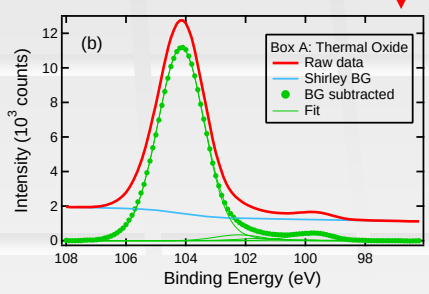
Spectroscopy

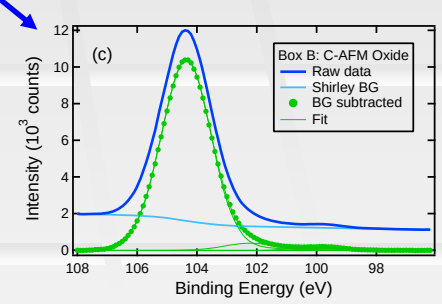


(a) 10 µm x 10 µm tapping mode AFM image of the patterned substrate, recorded prior to the XPEEM measurements. z-scale: 20 nm. (b) MEM image at 4.1 eV, measured before exposure to x-rays, showing two lines fabricated by C-AFM. (c,d) XPEEM images of the same area, taken with Si 2p photoelectrons at kinetic energies of 31.4 eV and 33.6 eV, respectively, which corresponds to binding energies of 105.6 eV and 103.4 eV. Photon energy for the XPEEM images was 141.5 eV. The boxes in (d) indicate the integration area for the spectra shown below. Field of view for all SPELEEM images: 10 µm.

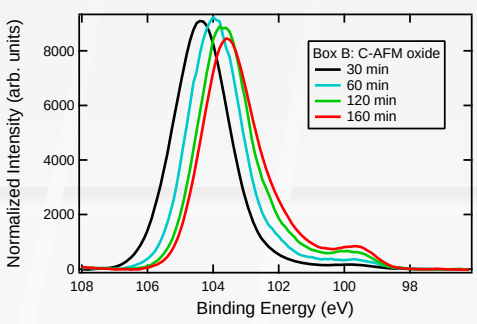


(a) Comparison of Si 2p spectra from a C-AFM line (blue) and from the thermal oxide (red). For the position of the boxes used for integration, see above. The SiO_2 component of the spectrum from the line written by C-AFM shows a shift to higher binding energies as compared to the spectrum from the thermal oxide. The bulk component is stronger in the spectrum recorded on the thermal oxide. The two vertical lines marked (I) and (II) indicate the energies at which the images in the pictures (c) and (d) above were taken. (b,c) Line profile analysis of the spectra from thermal oxide and C-AFM oxide, respectively. Photon energy for all spectra: 141.5 eV.





Time-resolved Spectroscopy



The oxide film thickness d was determined from the intensity ratio between the Si^{4+} component I_{SiO_2} and the substrate component I_{Si}. According to Himpsel et al. we have[1]

$$d = \lambda_{SiO_2} \times \ln\left(\frac{I_{SiO_2}}{I_{Si}}\frac{I_0}{I_\infty} + 1\right) \qquad (1)$$

with λ_{SiO_2} the photoelectron escape depth in SiO_2, I_0 the photoemission intensity for a bare Si substrate, and I_∞ the photoemission intensity for an infinitely thick SiO_2 layer.

[1] F. J. Himpsel, F. R. McFeely, A. Taleb-Ibrahimi, J. A. Yarmoff, and G. Hollinger, Phys. Rev. B **38**, 6084 (1988).