

## KPAN006: Application – Study of Thin Film Growth

*“In situ work function study of oxidation and thin film growth on clean surfaces” -*

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SURFACE SCIENCE, 1999

### Keywords

Work Function, WF, Kelvin Probe, Ultra High Vacuum, UHV, Metal Deposition, Oxidation, Ruthenium, Silicon

### Abstract

Using a novel ultra high vacuum compatible Kelvin probe a study is made of the work function ( $\phi$ ) changes on semiconductors and metals occurring during basic surface processing, for example, surface cleaning, sputtering, oxidation and thin film growth. It shows that damage of the  $7\times 7$  reconstruction due to Ar ion bombardment has a profound influence on the work function changes ( $\Delta\phi$ ) during oxidation on the Si(111) surface, tending to decrease or even reverse the surface dipole. Also following the variable temperature oxidation kinetics of Si(111) in the range of 100–600 K and show that magnitude of the  $\Delta\phi_{\text{peak}}$  during the initial adsorption curve decreases in a linear fashion with increasing substrate temperature. This is interpreted as being due to the rapid onset of oxygen permeation through the surface layer at higher temperatures producing a reverse or zero net dipole. Combining work function data with a localised technique such as scanning tunnelling microscopy permits monitoring of surface processes at both microscopic and macroscopic levels. In conjunction with Professor Behm’s group at Ulm University, Germany, this study monitored work function changes during evaporation of Al on Ru(0001) and show correlation between changes in  $\phi$  with topographic features such as island growth mechanism, monolayer formation, etc.



Figure 1. KP Technology UHV020 System.

## KPAN006: Application – Study of Thin Film Growth

### Research Area

The main objectives of this study were to; 1. Better understand the initial oxidation stages of Si(111); and highly sensitive to changes in surface and interface 2. To utilise a combination of in situ Kelvin probe (KP) and scanning tunnelling microscopy (STM) to elucidate changes occurring during initial growth processes. Both of these techniques are extremely sensitive to the top-most layers of the surface, and this unique combination allows one to correlate macroscopic work function changes with 'local' surface phenomena, for example, reconstruction and island growth.

The work function is a multi-parameter variable highly sensitive to changes in surface and interface chemical composition, adsorbate induced surface dipole and surface atomic geometry. It is an ideal tool in these experiments which exhibit significant adsorbate dipole interaction. For example in the oxidation of Si(111) we can resolve a 1 meV change in  $\phi$  corresponding to ca  $10^{-3}$  ML, this is at least two orders on magnitude better than typical Auger electron spectroscopy (AES) data. Further, STM data become difficult to interpret after ca 6 L due to oxide growth, however work function data are continuous in this regime and offer a previously untapped source of information.



The ultrahigh vacuum (UHV) compatible KP thus offers unparalleled sensitivity to changes in  $e$  surface dipole, the method produces the average work function of the sample under the tip and not that biased to low work function patches such as photo- and thermionic-emission. This system includes a sample-to-tip tracking algorithm without which work function measurements can be extremely difficult to interpret. This technique has previously been applied for research into semiconductor surface processing, surface charge imaging of oxides and operational electronic devices. The KP Has also been utilised in image contamination, sputtering induced surface roughness and the temperature/dose dependency of the Höfer precursor in the oxidation of Si(111)-(7×7).

## KPAN006: Application – Study of Thin Film Growth

### Use of Kelvin Probe

The KP used in this study has been developed from a high resolution ( $<50 \mu\text{eV}$ ) prototype,) featuring voice-coil actuator. This design is somewhat different from that previously published in that it utilises SmCo magnets located *inside* the vacuum envelope with the energising coil remaining outside. The suspension system readily produces amplitudes of oscillation of 1–2 mm permitting a stand-off distance, which can be regulated in the region 0.1–1.0 mm ensuring that tip-to-sample contact does not occur. The tip (2.5 mm in diameter) is fabricated from stainless steel which we have found to be resistant to most UHV processing methods. The ‘off-null’ work function measurement and tip-to-sample tracking procedures have been previously described. This system displays several important advantages over alternative systems involving piezoelectric actuators with lock-in amplifier detection. Firstly, driver talkover noise is eliminated due to the large physical separation ( $<200 \text{ mm}$ ) between actuator and probe tip. Second, the output signal level is substantially greater and there are no phase changes near balance or poor s/n levels due to nulling voltage feedback. Our work function resolution (1–2 meV) is achieved without recourse to any special pumping system or vibrational insulation requirements.

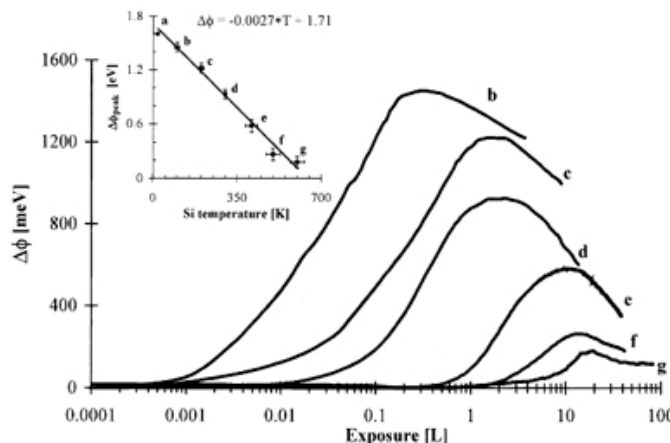


Figure 2. Work function change during Si oxidation 100–600 K, (b)–(g) correspond to substrate temperatures of 100, 200, 300, 400, 500 and 600 K. Insert,  $\Delta\phi_{\text{peak}}$  values versus substrate temperature.

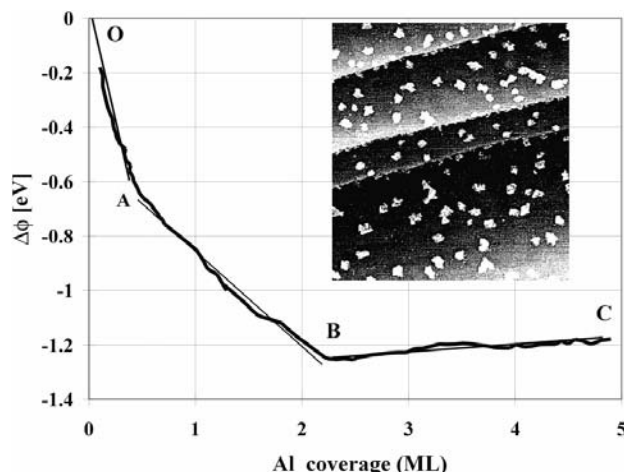


Figure 3.  $\Delta\phi$  data during the adsorption of Al on Ru(0001) at 300 K. The straight line segments OA, AB and BC are described in the text. (a) Shows a STM image recorded at a coverage of 0.12 ML.

The Kelvin method has a high *relative* accuracy – in the meV range – however, it does not provide *absolute* work function determination, which permits easy comparison with the literature data on clean surfaces. We have therefore installed in situ Al and Au reference electrodes on a linear translation arm allowing tip calibration before and after sample surface processing



## KPAN006: Application – Study of Thin Film Growth

### Conclusion

It is found that  $\phi_{\text{Si}(111)} = 4.85 \pm 0.030$  eV at 300 K, oxidation of the clean surface results in a peak work function change of some 950 meV at 2.5 L. The DAS reconstruction is dramatically affected by even a small amount of sputtering induced damage, which enhances oxygen permeation through the top layer. We find  $\Delta\phi_{\text{peak}}$  to be linear with decreasing substrate temperature in the region of 100-600 K. Finally, changes in gradient of  $\Delta\phi$  versus coverage data correlate well with changes in film growth modes measured by STM.

### Reference

1. Original publication: "In situ work function study of oxidation and thin film growth on clean surfaces" - I.D. Baikie, U. Petermann, B. Lagel

Full text available from:

Surface Science 433–435 (1999) 770–774 [PII: S0039-6028 (99) 00140-5]

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