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Kelvin probe force microscopy

M. Nonnenmacher, M. P. O'Boyle, and H. K. Wickramasinghe
T. J. Watson Research Center, IBM, Yorktown Heights, New York 10598

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Measurements of the contact potential difference between different materials have been performed for the first time using scanning force microscopy. The instrument has a high resolution for both the contact potential difference (better than 0.1 mV) and the lateral dimension (<50 nm) and allows the simultaneous imaging of topography and contact potential difference. Images of gold, platinum, and palladium surfaces, taken in air, show a large contrast in the contact potential difference and demonstrate the basic concept.

The scanning force microscope has been used previously to measure charges,[1] dielectric constants,[2] the film thickness of insulating layers,[3] photovoltage,[4] and electrical potential.[2,5,6] In this letter we demonstrate a new capability—the measurements of contact potential difference with high spatial resolution.

The contact potential difference (CPD) between two materials depends on a variety of parameters such as the work function, adsorption layers, oxide layers, dopant concentration in semiconductors, or temperature changes on the sample.[7,8] The measurement of the CPD can be used in principle to obtain information concerning these parameters. A common method to measure the contact potential difference is the vibrating capacitor method or Kelvin method.[8] In the Kelvin method two conductors are arranged as a parallel plate capacitor with a small spacing. In a simple model the contact potential between the two materials is $V_{\rm CPD} = 1/e(\Phi_2 - \Phi_1)$, where ϕ_1 and ϕ_2 are the work functions of the conductors including changes due to adsorption layers on the surface.[8,9] A periodic vibration of the distance between the two plates at ω results in a current $i(t)$ given by

$$i(t) = V_{\rm CPD}\omega\Delta C \cos \omega t,$$

where ΔC is the change in capacitance. For the actual measurement of $V_{\rm CPD}$, an additional bucking voltage is applied between the two plates until the space in between is field free and the current $i(t)$ goes to zero. The Kelvin method has a high sensitivity for potential measurement but integrates over the whole plate area and does not provide a lateral image of the variation of the CPD on the surface. Photo-electron emission spectroscopy (PEEM) has been used in the scanning electron microscopy to obtain high spatial resolution maps of work function.[10]

High lateral resolution CPD measurements can be achieved by using a modified version of the ac scanning force microscope.[2,3,5,6] The principle is similar to the Kelvin method except that forces are measured instead of currents. An additional voltage is applied between tip and sample until the electric field in between vanishes, as determined by measuring the force caused by the applied voltage.

The measurements were performed with an ac force microscope using a heterodyne interferometer to detect the motion of the force sensor.[11] In order to monitor topography and the CPD at the same time, we used a slightly modified scheme (see Fig. 1) to that proposed in Ref. 5. The ac voltage between tip and sample was applied at the resonance frequency while the piezo-induced vibration of the cantilever was driven slightly above resonance at $f_{\rm res} + \Delta f$, Δf being typically 2 kHz. The signal change in the piezo-induced vibration amplitude while approaching the tip was used in the conventional way to control the distance between tip and sample.[11] The second feedback loop was used to measure the CPD by minimizing the electric field between tip and sample.[5] There, the amplitude signal from the heterodyne interferometer was rectified, low pass filtered, and then measured at Δf with a lock-in amplifier. As described in Ref. 5, the integrator output was added to the ac voltage in order to minimize the electrostatic force between tip and sample.

Figure 2 shows a comparison between topographic and CPD images of gratings of gold, platinum and palladium, and gold, evaporated on a gold substrate. The layers were evaporated at a pressure of some 10^{-7} mbar and the thickness of the layers is 50 nm. Before the measurements the samples were cleaned in acetone (ultrasound) and isopropyl alcohol. The contrast in the CPD measurements are clearly visible and in the order of 90 mV for Pt on gold [Fig. 2(d)] and 65 mV for Pd on gold [Fig. 2(f)]. The CPD change on the gold/gold sample [Fig. 2(b)] is less than 15 mV. The CPD signal shows in addition to the grating, reproducible fine structure. At the interface between the substrate and deposited layers, additional local changes of the CPD signal occur; especially visible on the gold/gold sample. This change is induced by an increased

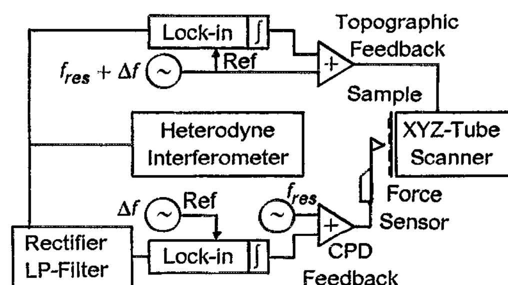

FIG. 1. Schematic diagram of the setup for simultaneous topographic and CPD measurements.

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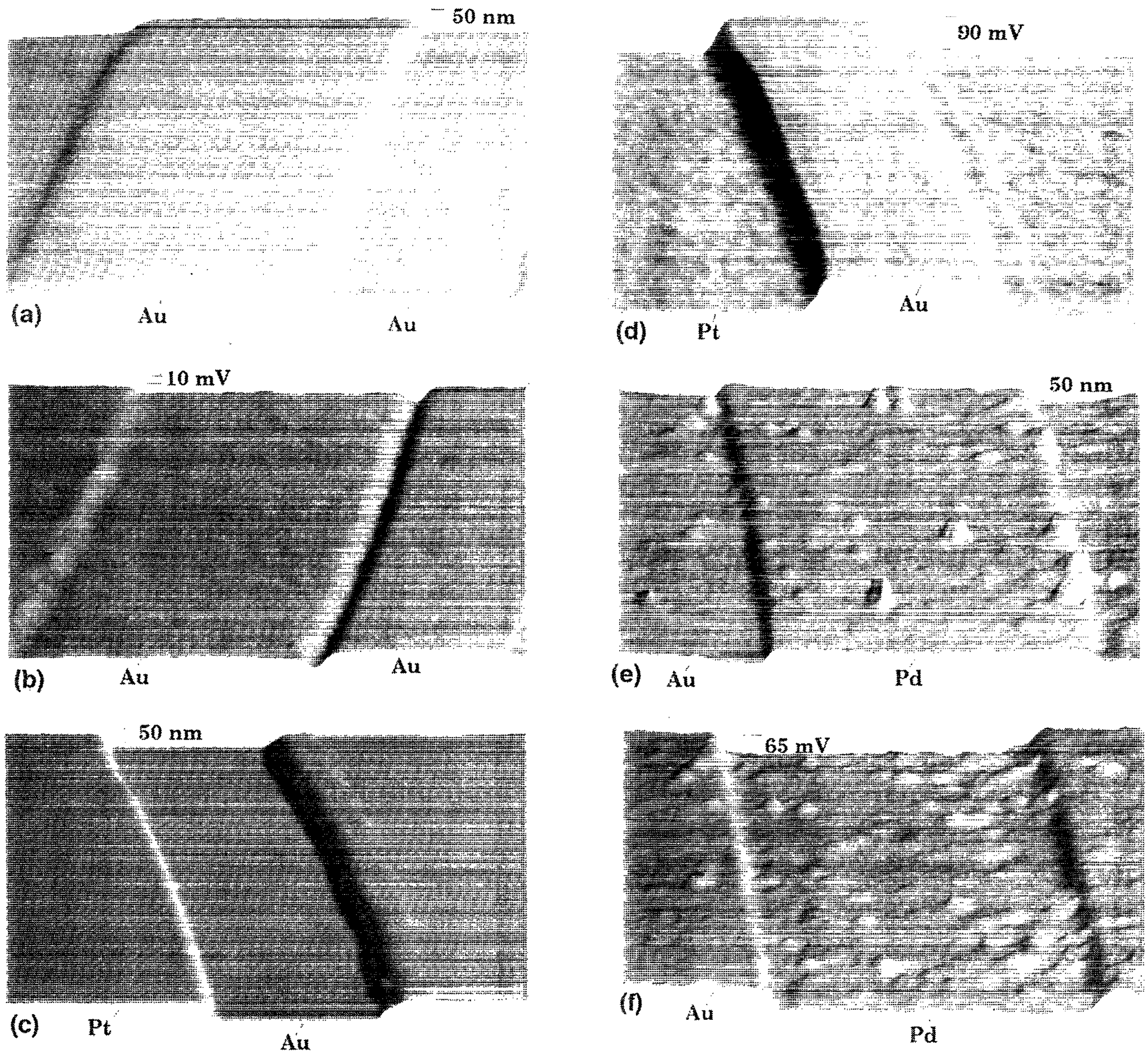

FIG. 2. Comparison of topographic and CPD images ($8\ \mu m \times 6\ \mu m$) of different samples: (a) and (b) gold on gold, (c) and (d) platinum on gold, (e) and (f) palladium on gold.

thickness of the contamination layer at the interface. The effect on poorly cleaned samples was much higher compared to carefully cleaned samples. The images in Fig. 2 were taken within 1 h after cleaning the samples. We observed a very slow decrease of the CPD contrast over time, when the sample was exposed to air. For example the CPD contrast between Pd and Au was about 65 mV, 20 min after cleaning, and decreased slowly to about 35 mV after four days. We believe that the high local electric fields on the sample surface (due to changes in the contact potential) induced a local reorientation of dipoles (mainly water molecules) or increased the contamination layer, both reducing the CPD contrast. Changes in the humidity could also contribute to the change in the CPD signals.

It is well known that the work function of a film varies with the deposition conditions and that adsorption layers can alter significantly the contact potential of a material. Experimental measurements of the work function based on CPD measurement found in literature therefore show a wide variation. The values of gold, for example, vary between 4.68 and 6.24 eV.[12] Published data of the work functions are somewhere between 4.82 and 5.62 for Pt and between 4.64 and 5.4 for Pd.[12,13] Quantitative interpretations of the measured data are therefore difficult, and require better defined surface conditions [ultrahigh vacuum (UHV)], and a better understanding of the basic theory of the CPD on conductors with adsorbed layers.

For the actual measurement of $V_{\rm CPD}$ with the force microscope, an ac voltage ($V_{\rm ac} \sin \omega t$) is applied between tip and sample. Assuming a tip radius R and a spacing d between tip and sample, the resulting force F for $d \ll R$ is given by[14]

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$$F = \pi\epsilon_0 R/d[V_{ac}^2 + V_{ac}V_{CPD}\sin\omega_{res}t + 1/2V_{ac}^2(1-\cos 2\omega t)].$$

If the applied frequency of the ac voltage is the resonance frequency f_{res} of the force sensor (quality factor Q and a spring constant k) the electrostatically induced amplitude A at f_{res} will be

$$A = \pi\epsilon_0 V_{ac}V_{CPD}(QR/kd).$$

A comparison with the thermally induced noise N[11]

$$N = \sqrt{2k_BTQB/\pi k f_{res}}$$

finally gives the sensitivity of the smallest measurable contact potential difference $V_{CPD,min}$:

$$V_{CPD,min} = \sqrt{2k_BTkB/\pi^3 Q f_{res}}(1/\epsilon_0 V_{AC})(d/R),$$

where k_B is the Boltzmann constant, T the temperature, and B the bandwidth in which the experiment is performed.

We used two different force sensors for our experiments. A gold-coated silicons sensor[15] with $f_{res} = 532$ kHz, $k = 20$ N/m, and $Q = 500$ and a nickel chrome tip with $f_{res} = 83$ kHz, $k = 10$ N/m and $Q = 300$. The tip radius of both sensors was about 50 nm. Therefore, with $V_{ac} = 2$ V, $B = 1$ Hz, and $d = 10$ nm, the sensitivity for the CPD measurement is about 50 μV for the silicon sensor and 120 μV for the Ni–Cr tip.

It is interesting to note that even with a very small tip radius (5 nm) giving a high lateral resolution (<10 nm[16])
and at d = 2.5 nm ($B = 1$ Hz) the sensitivity for the CPD measurements is in the order of 100 μV for the silicon sensor. If the measurements are performed in UHV a further improvement of sensitivity of about ten times can be achieved, due to an increase of the quality factor by a factor of 100.[3]

The basic concept of the scanning contact potential microscope is introduced, allowing the simultaneous measurement of topography and contact potential difference with high resolution. In these first experiments, several images of different metals were taken in order to demonstrate the performance. We believe that further investigations under cleaner surface conditions as well as measurements concerning changes of the CPD due to changes in the temperature or dopant concentration of semiconductors, electrochemical surface reactions, adsorption or oxide layers will provide new and additional information about sample properties.

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