Applied Surface Science 157 (2000) 256–262
applied surface science
www.elsevier.nl/locate/apsusc

Kelvin probe force microscopy using near-field optical tips

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Abstract

We report on the use of near-field optical force sensors for Kelvin probe force microscopy (KPFM) and surface potential measurements. It is shown that a very good potential sensitivity of less than 5 mV can be obtained using such tips. In addition, it is found that the contact potential difference measured using these tips is independent of the scanning height, as long as it is below 40 nm, and of the applied AC amplitude as long as it is in the range of 1–3 V. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Scanning probe microscopy has opened new opportunities to image semiconductor electronic properties with unprecedented spatial resolution. Both scanning tunneling microscopy (STM) [1], and atomic force microscopy (AFM) [2] have been modified to obtain high resolution maps of the electric surface potential distribution. However, the application of the STM is limited to conductive samples. With the development of the Kelvin probe force microscopy (KPFM), this major disadvantage was overcome, because forces are measured instead of tunneling currents. The KPFM has found many diverse applications in recent years. The technique has been applied to materials science applications such as: work function mapping [2], and ordering in III–V compound semiconductors [3]. Kikukawa et al. have conducted surface potential measurements of silicon pn junctions [4], and Vatel et al. have demonstrated potential measurements of resistors [5], and n-i-p-i heterostructures [6]. KPFM has also proved to be effective in electrical characterization of submicron devices like high electron mobility transistors (HEMTs) [7,8], and light emitting diodes [9,10].

An important application of the KPFM technique is surface photovoltage (SPV) measurements. Illumination of a semiconductor surface by monochromatic light results in charge exchange between the bands, and between the band edges and local electron states, if the latter are present within the semiconductor band gap. Illumination by photons with energy $h\nu = E_C - E_t$ may produce electron transitions from a gap state at an energy E_t into the conduction band, where E_C is the conduction band minimum. This gap state depopulation is accompanied by a change in the surface potential and therefore will change the CPD between the sample and the tip. Similarly, this illumination may also cause the population of a gap state situated at an energy E_t above the valence band maximum, which is accompanied by an opposite sign change in the CPD. Thus, by measuring the SPV under monochromatic illumination using the AFM tip, a two dimensional map of the recombination centers (in the case of super bandgap excitation) or surface states (in the case of sub-bandgap illumination) can be obtained.

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0169-4332/00/$ - see front matter
PII: S0169-4332(99)00536-X

In order to obtain high spatial resolution SPV data, the semiconductor has to be excited with nanometer-size light spot at exactly the same location where the CPD is measured by the AFM tip. We propose to do this using a sharp fiber tip like that used in the near-field scanning optical microscope (NSOM). In such a case, the semiconductor will be excited with a laser passing through the near-field fiber which raster scans above the crystal surface. A metal coating on the fiber helps to keep the light from leaking out in the tapered region, and will be used also as the conducting material for the CPD measurements. In this work, we demonstrate for the first time the measurement of electrostatic forces and CPD with near-field optical fibers. It is found that a very high potential sensitivity can be achieved depending on the tip and cantilevers properties.

2. KPFM experimental setup

Fig. 1 shows a schematics of the KPFM measurement setup using an L-shape optical fiber force sensor (Nanonics supertips Inc.). It is fabricated from a single mode optical fiber with special techniques for tapering such glass structures [11–13]. The optical fiber tips were coated with gold, which also served as the electrical contact to the fiber through the fiber holder in the same way as for conventional tips. The fiber is vibrated by a piezo mounted above the cantilever. The KPFM measurements were done using a standard KPFM setup [2] (see Fig. 2).

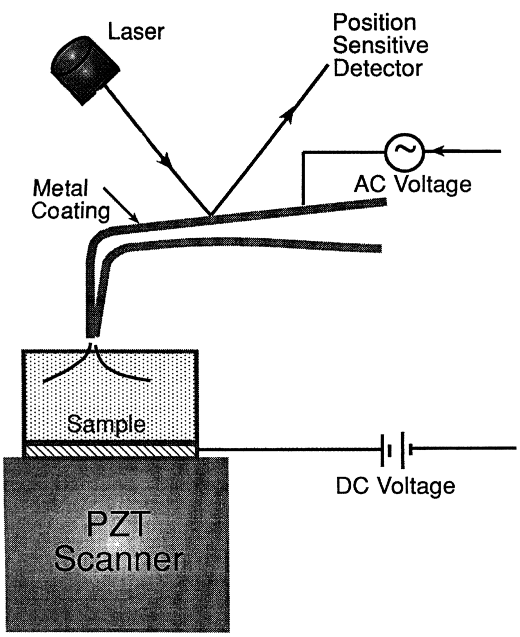

Fig. 1. A schematic diagram of the KPFM experimental setup using the near-field optical sensor. The AC voltage is applied to the fiber metal coating.

Fig. 2 shows the electronic setup used for the KPFM measurements. All measurements were conducted using Digital Instruments (Extended Multi-Mode Nanoscope III with the Extender Electronics Module) atomic force microscope. All measurements are taken in air at ambient pressure and humidity; the sample used in our measurements is a heavily doped silicon wafer.

3. Tip characterization

3.1. General

The main goal of this study is to check whether near-field optical fiber tips could be used as electrostatic force sensors and to characterize their electrical sensitivity. Therefore, we characterized the tips by setting the scanning size to zero in order to avoid noise resulting from the sample topography. As a consequence, most of the results we present below describe the CPD variations with time resulting from a square wave voltage applied to the sample. The characterization procedure consists of the following steps:

1. Measuring the electrical sensitivity of the tips by varying the amplitude of an AC voltage applied to the sample in the range of 10 mV–1 V.
2. Changing the height of the ''lift mode'' (from 5 to 40 nm) and monitoring the measured CPD.
3. Changing the amplitude of the AC voltage (at a frequency ω_e, see Fig. 2) that induces the AC electrostatic force between the fiber tip and the sample.

All the above steps were conducted with several optical force sensors having different lengths, L_c and two different shapes: cylindrical — Fig. 3(a), or triangular — Fig. 3(b). All the results were compared to measurements conducted using MESP tips (made from silicon coated with PtI_5 both on the tip

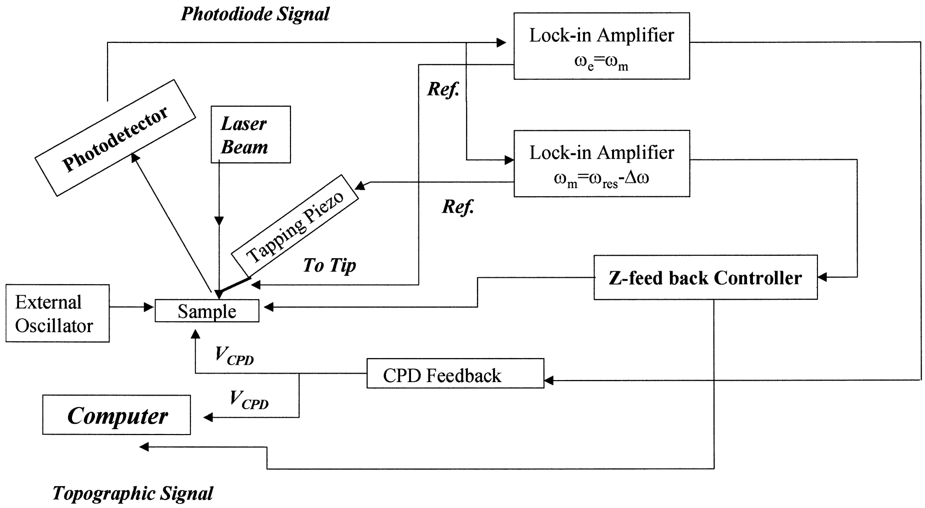

Fig. 2. A schematic diagram of the KPFM measurement system.

and the cantilever sides by Nanosensors Inc.); such tips are commonly used for KPFM measurements.

3.2. Results and discussion

Fig. 4 shows the CPD measured between five different types of tips and the sample without scanning. The CPD in Fig. 4(a) and (b) was measured with cylindrical shape fibers with $L_c = 400$ and 600 μm, respectively, and the voltage scans (c), (d), and

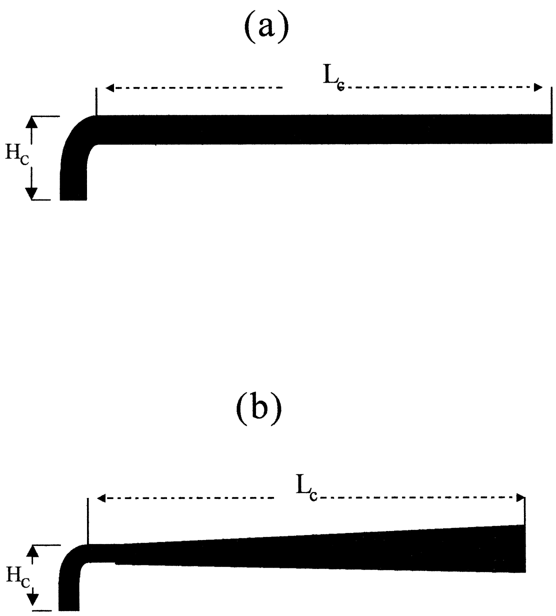

Fig. 3. A schematic diagram showing the shapes of the two fibers that were used in this study: (a) cylindrical shape and (b) triangular shape.

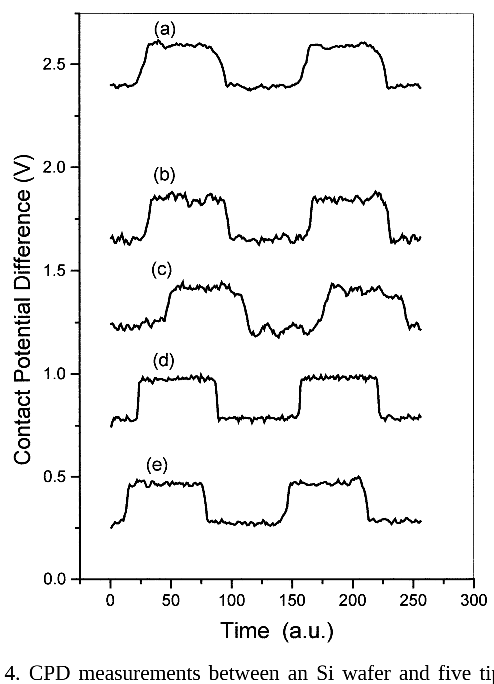

Fig. 4. CPD measurements between an Si wafer and five tips of different shapes and different lengths. (a) Cylindrical shape, L_c = 400 μm, (b) Cylindrical shape, $L_c = 600$ μm, (c) Triangular shape, $L_c = 500$ μm, (d) Triangular shape, $L_c = 600$ μm, (e) Triangular shape, $L_c = 400$ μm.

(e) were obtained using triangular shape fibers having $L_c = 500$, 600 and 400 μm, respectively. The signal that was applied to the silicon wafer was a 100 mV amplitude square wave, the scanning height in the ''lift mode'' was 10 nm, and the amplitude of the AC voltage applied to the tip was 3 V. Two observations can be made based on these results:

1. The longer the length of the optical tip, L_c, the better is the signal to noise ratio and the shape of the measured square wave (compare Fig. 4(d) and (e) and also Fig. 4(a) and (b)).
2. Tips with triangular shape give better results compared to tips of cylindrical shape (compare Fig. 3(b) and Fig. 3(d)).

We assume that the improved response of the longer tips results from their smaller spring constant. A small spring constant allows detection of small forces [14], thus improving the sensitivity of the tips. The spring constant decreases with increasing tip length; therefore, longer tips have a better sensitivity for electrostatic forces.

Fig. 5(a) shows three measurements conducted at three different applied amplitudes of: (i) 10 mV, (ii) 20 mV, and (iii) 50 mV using the best optical force sensor (triangular shape with $L_c = 600$ μm) compared to identical measurements conducted using MESP tip (Fig. 5(b)). In both cases, a signal with an amplitude of 10 mV (line scan (i) in both figures) could be detected. The potential root mean square (rms) values were 5, 10, and 3 mV for the three applied voltages in the case of the optical fiber and ≃ 1.2, ; ≃ 1.5, and 3 mV for the MESP tip, respectively. The difference noise level measured for the optical probes and the MESP tips originated from the higher amplification of the feedback circuit in the former case. This results in reduced signal to noise ratio, an effect that can be observed in Fig. 7

The effect of the scanning height during the ''lift mode'' is demonstrated in Fig. 6. The figure shows the measured data at three different heights of (i) 0 nm, (ii) 10 nm, and (iii) 40 nm for both the optical fiber (Fig. 6(a)) and the MESP tip (Fig. 6(b)); the applied AC voltage amplitude was 100 mV. In both cases, the amplitude of the measured peak was inde-

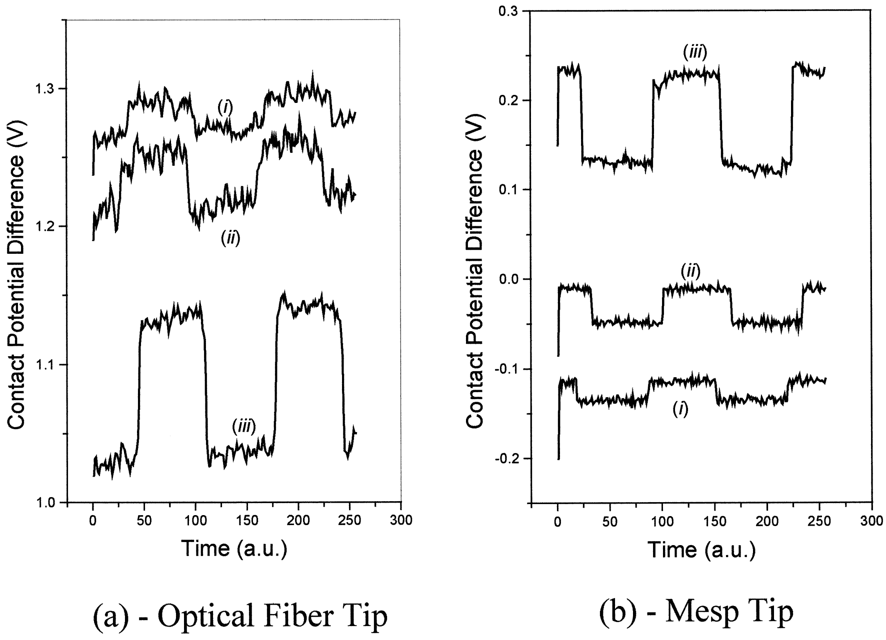

Fig. 5. Measurements of CPD by the optical fiber (a), and MESP tips (b), at three different AC biases of (i) 10 mV, (ii) 20 mV, and (iii) 50 mV, applied on an Si wafer.

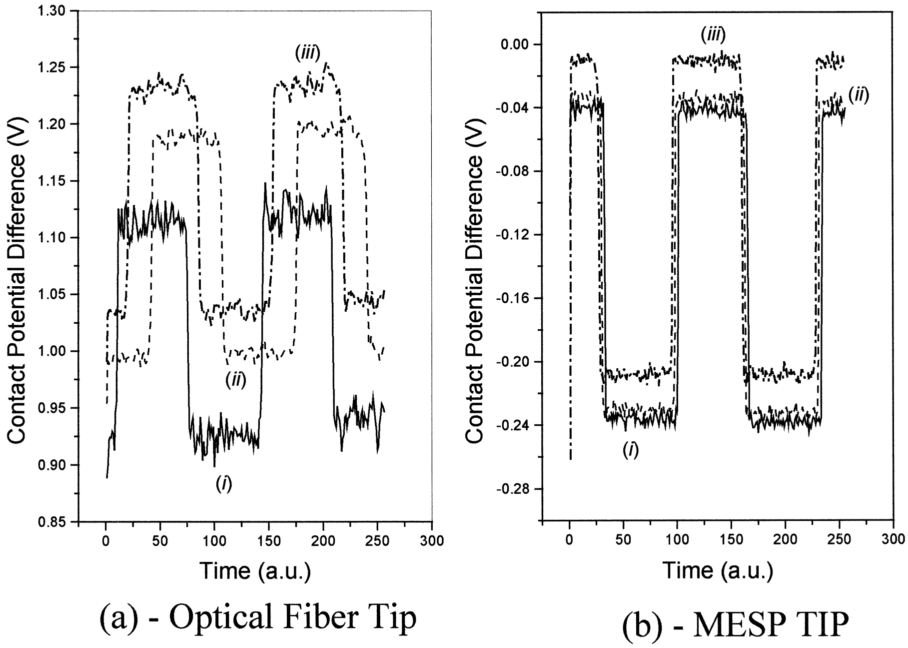

Fig. 6. Measurements of CPD by the optical fiber (a), and MESP tips (b), at three different scanning heights: of (i) 0 nm, (ii) 10 nm, and (iii) 40 nm.

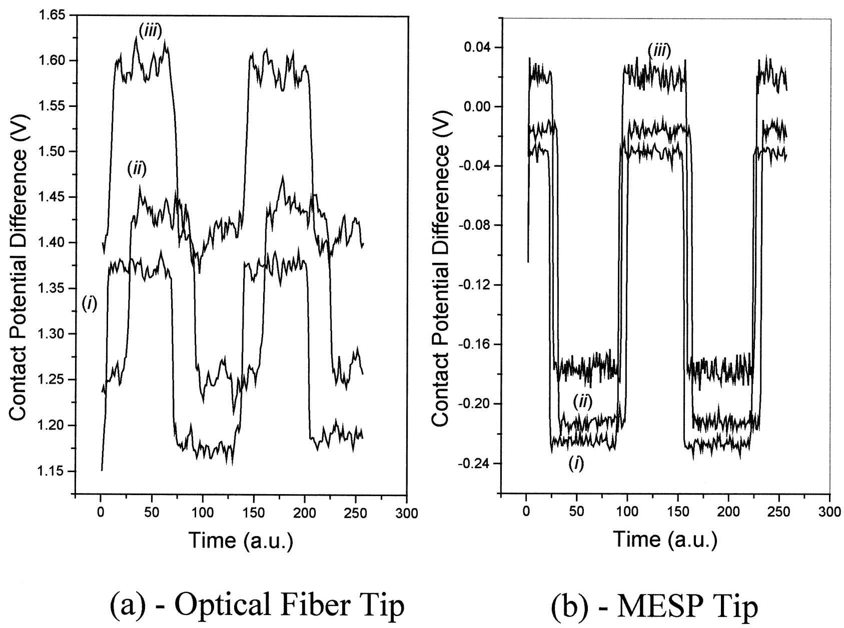

Fig. 7. Measurements of CPD by the optical fiber (a), and MESP tips (b), at three different AC applied voltages: (i) 1 V, (ii) 2 V, and (iii) 3 V, applied on the tip.

pendent of the scanning height. The only observed change is that the average DC voltage changes with the tip height. These effects may be due to capacitance and image force effects [6,14,15]. The change is somewhat more pronounced for the optical fiber than for the MESP tips.

When the scanning height is changed the capacitance between the tip and the sample changes, and the capacitive interaction with the cantilever increases [16]. This means that instead of measuring the CPD between the tip and the sample, the CPD between the whole cantilever and the sample will be measured; such is the case when the length of the tip is very small. In the case of the optical force sensor, this length H_c is on the order of 50 μm; hence, changing the scanning height should have a very small affect on the CPD due to capacitance changes. This means that the scanning height can be set to an arbitrary value without affecting the measured CPD. Based on this reasoning, the changes in the DC component of the voltage scans are attributed to image forces rather than capacitance changes.

Fig. 7 shows the effect of the amplitude of the AC voltage applied to the tip on the measured CPD. The objective is to keep this voltage as low as possible in order not to affect the measured CPD [3]. Fig. 7 shows measurements taken at three different amplitudes of: (i) 1 V, (ii) 2 V and (iii) 3 V; the amplitude of the square wave signal that is applied to the sample is 100 mV. The figure clearly demonstrates that the AC voltage does not influence the measurement as long as it is below 3 V.

4. Conclusions

We have demonstrated for the first time the use of a near-field optical fiber for KPFM measurements. We have shown that a very good sensitivity of less than 5 mV can be achieved using such tips; such sensitivity is of the same order of magnitude obtained with commercial metal coated silicon tips. Furthermore, our measurements show that the CPD measured using the optical fibers is insensitive to the scanning height as long as it is below 40 nm and to the AC amplitude as long as it is larger than 1 V and smaller than 3 V.

The use of optical force sensors for electrical measurements has the following advantages.

(1) The feedback laser spotlight can be positioned at around 200 μm away from the tip edge. This ensures that there will be very small influence of the feedback laser light on the semiconductor underneath. This is an important advantage for SPV measurements where the sample should be excited only by the light passing through the fiber.

(2) The height of the optical tip, H_C, is on the order of 50 μm. This ensures that the measured CPD will be governed by sample–tip interaction and not from electrostatic interaction with the cantilever [16,17].

(3) The special geometry of the optical tip may enable to conduct electrical measurements in regions with very high aspect ratios commonly found in VLSI circuits.

Acknowledgements

This research is supported by the Israel Science foundation administered by the Israel Academy of Science and Humanities-Recanati and IDB group foundation, and by grant 9701 of the Israel Ministry of Science. R.S. is supported be Eshkol special scholarship of Israel Ministry of Science.

References

[1] J.R. Kirtley, S. Washburn, M.J. Brady, Phys. Rev. Lett. 60 (1988) 1546.
[2] M. Nonenmacher, M.P. O'Boyle, H.K. Wickramasing, Appl. Phys. Lett. 66 (1991) 2091.
[3] Y. Leng, C.C. Williams, L.C. Su, G.B. Stringfellow, Appl. Phys. Lett. 66 (1995) 1264.
[4] A. Kikukawa, S. Hosaka, R. Imura, Appl. Phys. Lett. 66 (1995) 3510.
[5] O. Vatel, M. Tanimoto, Phys. Rev. Lett. 77 (1995) 2358.
[6] A. Chavez-Prson, O. Vatel, M. Tanimoto, H. Ando, H. Iwamura, H. Kanbe, Appl. Phys. Lett. 67 (1995) 3069.
[7] T. Mizutani, M. Arakawa, S. Kisimoto, IEEE Electron Device Lett. 18 (1997) 423.
[8] M. Arakawa, S. Kisimoto, Jpn. J. Appl. Phys. 36 (1997) 1826.
[9] R. Shikler, T. Meoded, N. Fried, Y. Rosenwaks, Appl. Phys. Lett. 74 (1999) 2972.
[10] R. Shikler, T. Meoded, N. Fried, B. Mishori, Y. Rosenwaks, J. Appl. Phys. 86 (1999) 107.
[11] A. Harootunian, E. Betzig, M. Isaacson, A. Lewis, Appl. Phys. Lett. 49 (1986) 674.

[12] S. Shalom, K. Lieberman, A. Lewis, S.R. Cohen, Rev. Sci. Instrum. 63 (1992) 4061.
[13] K. Liebrman, A. Lewis, G. Fish, S. Shalom, T.M. Jovin, A. Schaper, S.R. Cohen, Appl. Phys. Lett. 65 (1994) 648.
[14] R. Wiesendanger, Scanning Probe Microscopy and Spectroscopy, Cambridge, 1994.
[15] S. Hudlet, M.S. Jean, B. Roulet, J. Berger, C. Guthmann, J. Appl. Phys. 77 (1995) 3308.
[16] T. Hochowitz, A.K. Henning, C. Levey, C. Daghlian, J. Slinkman, J. Vac. Sci. Technol. B 14 (1996) 457.
[17] F. Rossi, Rev. Sci. Instrum. 63 (1992) 4174.