Microscopic four-point probe based on SU-8 cantilevers

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A microscopic four-point probe (μ4PP) for resistivity measurements on thin films was designed and fabricated using the negative photoresist SU-8 as base material. The device consists of four microscopic cantilevers, each of them supporting a probe tip at the extremity. The high flexibility of SU-8 ensures a stable electrical point contact between samples and probe tip with all four electrodes even on rough surfaces. With the presented surface micromachining process, μ4PPs with a probe-to-probe spacing of 10–20 μm were fabricated. Resistivity measurements on thin Au, Al, and Pt films were performed successfully. The measured sheet resistances differ by less than 5% from those obtained by a commercial macroscopic resistivity meter. Due to the low contact forces ($F_{cont} < 10^{-4}$ N), the μ4PP is suitable to be applied also to fragile materials such as conducting polymers. Here the authors demonstrate the possibility of performing resistivity measurements on 100-nm-thick pentacene ($C_{22}H_{14}$) films with a sheet resistance $R_s > 10^6$ $\Omega/\square$. © *2005 American Institute of Physics.* [DOI: 10.1063/1.2140443]

I. INTRODUCTION

The four-point probe (4PP) method is used for the measurement of the resistivity of thin metal or semiconductor films. Four linearly arranged and equally spaced electrodes contact the sample. Usually, a current is applied to the outer pins and the voltage difference between the inner pins is measured (see the inset to Fig. 1). The resulting voltage-to-current ratio allows the calculation of the sheet resistance R_s of the thin film. The advantage of this method is that the measurement errors due to the contact resistances between sample and probe tips are practically eliminated.

There is an interest in miniaturization of the 4PPs to obtain higher surface sensitivity, an increased spatial resolution, and less damage to the sample.[1,2] Several attempts have been made to reduce the probe-to-probe spacing. A four-tip scanning tunneling microscope (STM)[3] and a microscopic four-point probe (μ4PP) based on silicon cantilevers were reported.[4–6] The latter are fabricated by a bulk-micromachining process and have already been commercialized by Capres S/A.[7]

The negative photoresist SU-8 is used for the fabrication of structures with high aspect ratio and has mechanical properties that allow it to be introduced as base material for an increasing number of micromechanical devices.[8–12] In this article, we present the design, fabrication, and characterization of μ4PPs based on SU-8 cantilevers. The results obtained on thin metal films are compared to measurements performed with a commercialized macroscale resistivity meter (Omnimap RS75, KLA Tencor).

Since the Young's modulus of SU-8 is 4–5 GPa,[8] which is about 40 times lower than that of silicon, this photoplastic material allows the fabrication of cantilevers with very low spring constants. A particular advantage of the soft SU-8 cantilevers is the reduced contact force, which allows the characterization of fragile materials such as polymer semiconductors. Pentacene ($C_{22}H_{14}$) thin films, for instance, are used in organic thin-film transistors (OTFTs).[13] Until now, four-point resistivity measurements on pentacene were realized by direct evaporation of metal electrodes on the sample surface.[14,15] In this paper, we demonstrate that the fabricated μ4PPs represent a possible alternative method for localized characterization of fragile polymer semiconductors.

II. DESIGN AND FABRICATION

A 200-μm-thick SU-8 body chip with lateral dimensions of 3 mm$\times$5 mm allows the handling of the device and the electrical connections to the measurement setup (see Fig. 1). The body chip has a honeycomb structure to accelerate the final release-etch and to reduce the interior stress in the SU-8 (see Fig. 3). An SU-8 cantilever with 500 μm length and 10 μm thickness is split at its free-standing extremity into four equally spaced microcantilevers that have a length of 125 μm. This design ensures a stable electrical contact between samples and probe tips with all four electrodes even on rough or inclined surfaces. The width and the spacing of the microcantilevers are varied from 4 to 10 μm to determine the lithographic and mechanical limits of the chosen microfabrication process. Each microcantilever supports a pyramidal probe tip with a base length corresponding to the cantilever width. The metal electrodes are integrated in the polymer structure, covering the microcantilevers and the probe tips.

The μ4PP is fabricated by a surface micromachining process for hybrid polymer-metal microstructures.[12] The process flow is represented in Fig. 2. A 100 mm silicon wafer with 2 μm of thermally grown oxide on both sides is used as

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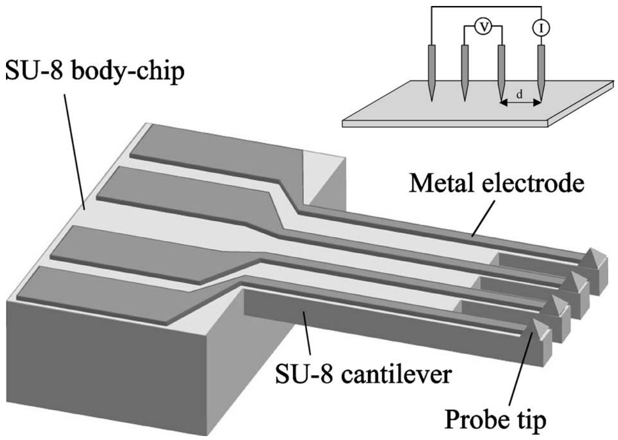

FIG. 1. Schematic representation of the microscopic four-point probe (μ4PP); upper right: four-point measurement principle; d=probe-to-probe spacing.

initial substrate. The oxide is patterned after the photolithography by wet etching in buffered hydrofluoric acid (BHF) to form the mask for the tip molds. Pyramidal holes are then etched in the silicon wafer using potassium hydroxide (KOH) as an anisotropic etchant [Fig. 2(a)]. A concentration of 40% KOH at a temperature of 60 °C is selected resulting in an etch rate of about 19 μm/h. The residual oxide layer is removed by BHF and the wafer is cleaned by a standard RCA procedure before the second oxidation. A 200-nm-thick wet oxide layer is grown and a 2-μm-thick polysilicon layer is deposited by low-pressure chemical vapor deposition (LPCVD). The polysilicon has the function of a sacrificial layer and the wet oxide serves as an etch stop layer during

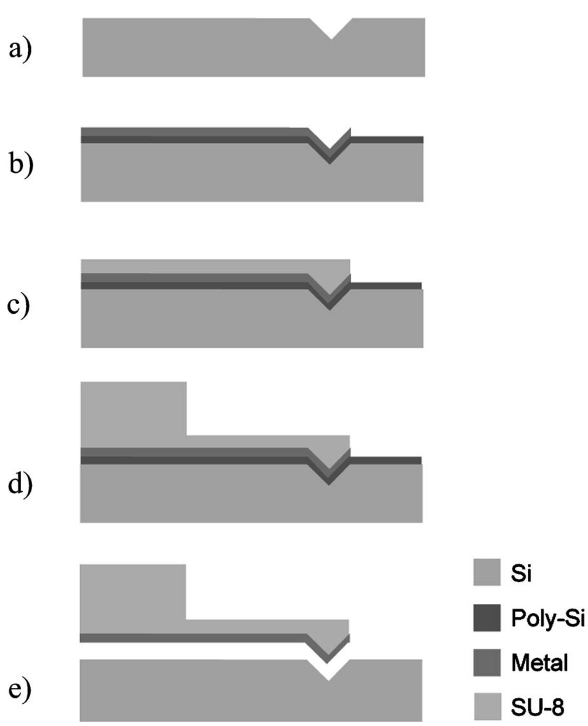

FIG. 2. Fabrication of the μ4PP: (a) KOH etch of tip molds; (b) wet oxidation and poly-Si deposition, metal layer (50 nm Pt/20 nm Ti/300 nm Al) patterned by Cl dry etch; (c) 10-μm-thick SU-8 layer for cantilevers; (d) 200-μm-thick SU-8 layer for body chip; (e) release etch with SF_6.

the final release-etch. As electrode material, a three-layered metal of 50 nm Pt, 20 nm Ti, and 300 nm Al is deposited by sputtering. The Pt layer forms the outer layer of the electrode and the other two metals are chosen to improve the adhesion to the SU-8. The metal layer is etched in one single step using a Cl-based inductively coupled plasma (ICP) dry etch [Fig. 2(b)]. A first layer of SU-8 is spin-coated, exposed, and developed to define the structure of the thin cantilevers [Fig. 2(c)] and of the probe tips. Then a second step of SU-8 photolithography is used to structure the body chip [Fig. 2(d)]. Finally, the entire device is released by dry etch of the sacrificial layer with SF_6 [Fig. 2(e)] and the four-point probes are removed mechanically from the silicon wafer.

Figure 3 shows a μ4PP with a probe-to-probe spacing d=20 μm fabricated with the presented microfabrication process. For devices with d down to 15 μm, the yield is close to 100%. The smallest probe-to-probe spacing achieved is 10 μm.

III. EXPERIMENTAL RESULTS

The experimental setup for the characterization of the fabricated μ4PPs consists of a stage for atomic force microscopy (AFM) with a spring-loaded mechanical clamping system to fix the chip. A laser is integrated into the stage for the optical readout of the cantilever deflection. Furthermore, the mechanical clamping system ensures the electrical contact between the multimetal electrodes on the μ4PP and the Au-coated electrodes on a printed circuit board (PCB) that is placed in the AFM head. The sample is approached to the probe until a stable electrical contact is established between all four electrodes and the sample. A 2400 Sourcemeter from Keithley was used as a current source and the voltage was measured with a 34401A voltmeter from Hewlett-Packard.

To evaluate the performance of the fabricated μ4PPs, the sheet resistances of thin films of Au, Al, Pt, and Cu were measured. Comparative measurements on the samples were realized with the commercialized macroscale resistivity meter Omnimap RS75 (KLA Tencor), having a probe-to-probe spacing of 1.016 mm. Figure 4 represents the four-point voltages measured on a 140-nm-thick Au film evaporated on a silicon substrate with a 1-μm-thick silicon oxide layer. The current was varied from 0.1 to 10 mA, and the resulting mean value of sheet resistance is $R_{s(\mu 4\text{PP})}=112.4\ \text{m}\Omega/\square$. This is comparable to the value $R_{s(\text{omnimap})}=111.9\ \text{m}\Omega/\square$ measured with the macroscopic 4PP at a current of 100 mA. For the measurements on Au, Pt, and Al, the sheet resistances differ less than 5% from those that measured by the macroscopic resistivity meter. For a stable electrical contact on Au and Pt, cantilever deflections of 15–20 μm were required. This corresponds to a contact force of the order of 10^{-5} N, and the contact pressure is estimated to be around 10^7 Pa. For the measurements on the Al sample, four times higher cantilever deflections were necessary to observe a stable contact. The reason is that the metal surface oxidized and the probe tips have to penetrate this thin insulating film. In the case of the Cu film, the con-

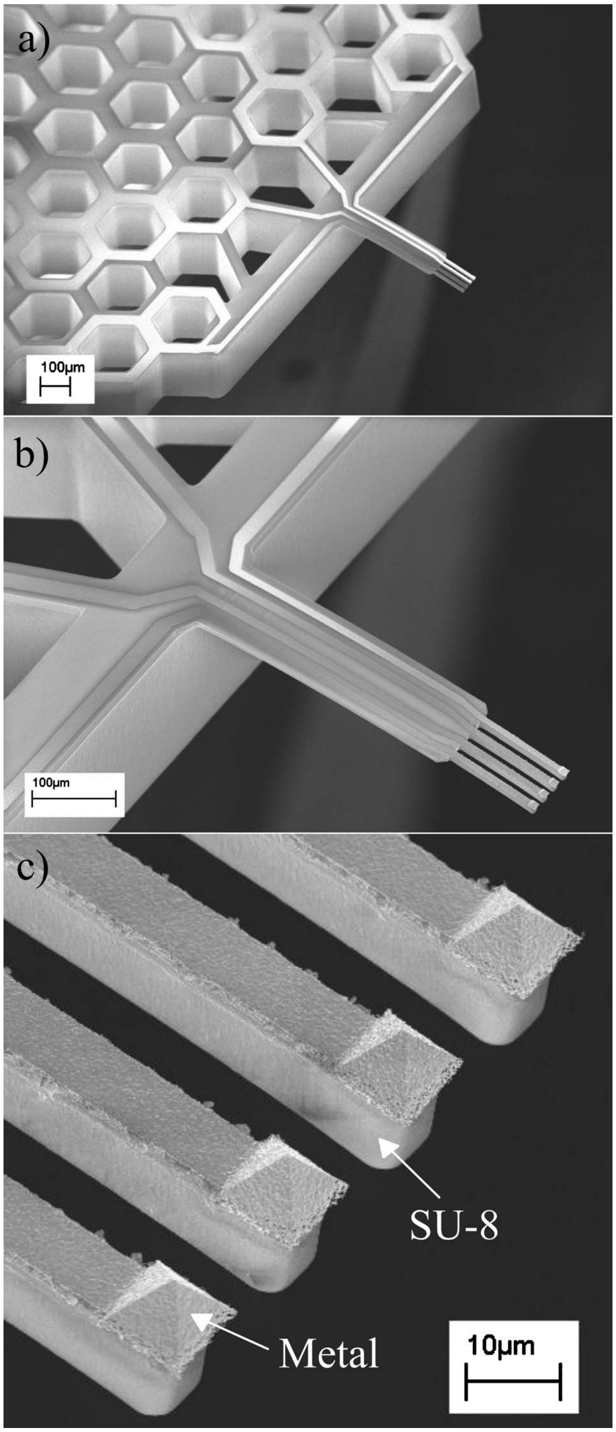

FIG. 3. SEM micrographies of μ4PP with probe-to-probe spacing $d = 20\ \mu$m. (a) Body chip with cantilever (all SU-8) and with four contact electrodes in metal; (b) SU-8 cantilever split up in four microcantilevers; (c) details of the pyramidal probe tips (SU-8 covered by metal layer) at the extremity of the cantilevers; tip height $h = 8.5\ \mu$m.

tact forces between probe tips and sample were too small to penetrate the oxide layer and four-point measurements were impossible.

The adhesion of the metal layer to the SU-8 cantilever is satisfying. Even at cantilever deflections higher than 50 μm, the electrodes remained stable. Repetitive measurements with the same devices indicate an elastic behavior of the microcantilevers. On the other hand, SEM observations of the μ4PPs after the experiments show that the quality of the probe tips is affected due to the physical contact with the hard metal samples.

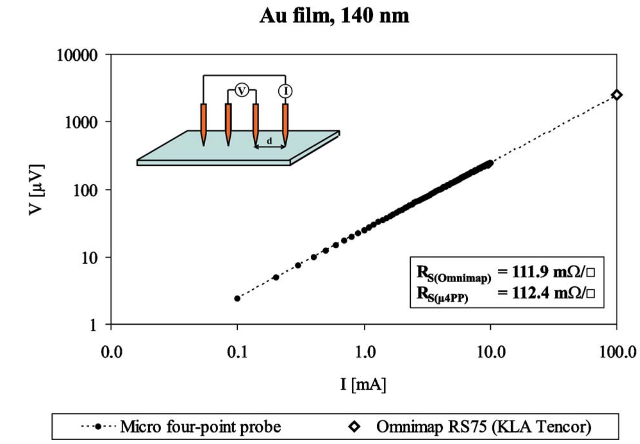

FIG. 4. 4PP resistivity measurement on 140-nm-thick Au film; currents of 0.1–10 mA were applied and the four-point voltages were measured; the resulting mean sheet resistance $R_{s(\mu 4PP)} = 112.4\ m\Omega/\square$ is comparable to the value $R_{s(omnimap)} = 111.9\ m\Omega/\square$ measured with the macroscopic resistivity meter Omnimap RS75 at a current of 100 mA.

To demonstrate the potential of the μ4PPs, we extended the experiments to thin organic semiconducting films. In particular, we performed resistivity measurements on 100-nm-thick pentacene films on silicon dioxide. Small currents of a few hundred pA were applied to avoid resistive heating at the electrical contacts. Figure 5 presents the result of a successful measurement with a resulting mean sheet resistance of 3.26 $M\Omega/\square$. The order of magnitude of this value corresponds to those reported for similar samples.[14,15] The contact forces required are in the order of 10^{-5} N. Further optimizations of the electrical readout of the experimental setup will increase the signal-to-noise ratio and allow measurements on thin organic semiconductor films with even higher sheet resistances.

In conclusion, we have developed a reliable fabrication process for microscopic four-point probes that allows characterization of samples with sheet resistances in the range of 10^{-1}–$10^{6}\ \Omega/\square$. The reduced contact force due to the use of SU-8 as cantilever material is a disadvantage if oxide layers

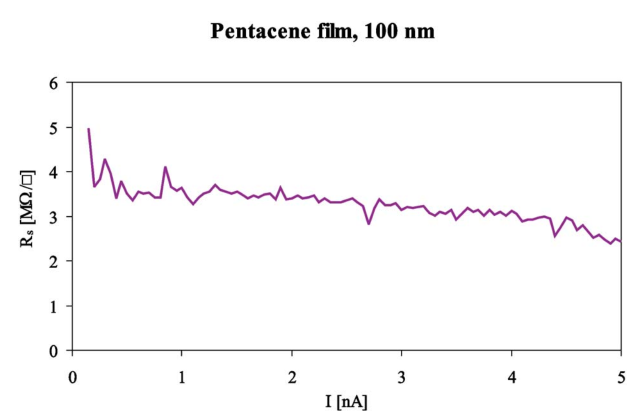

FIG. 5. Four-point measurement on 100-nm-thick pentacene film; a current sweep up to 5 nA with a step of 50 pA was performed; the resulting mean value of sheet resistance is $R_{s(\mu 4PP)} = 3.26\ M\Omega/\square$.

cover the sample surface. However, the low contact forces introduce the presented μ4PP as a possible alternative metrology tool to be used for measurements on fragile materials.

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