Automatic kelvin probe compatible with ultrahigh vacuum

I. D. Baikie,[a] K. O. van der Werf, H. Oerbekke, J. Broeze, and A. van Silfhout

Department of Applied Physics, University of Twente, P. O. Box 217, 7500 AE Enschede, The Netherlands

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This article describes a new type of *in situ* ultrahigh-vacuum compatible kelvin probe based on a voice-coil driving mechanism. This design exhibits several advantages over conventional mechanical feed-through and (*in situ*) piezoelectric devices in regard to the possibility of multiple probe geometry, flexibility of probe geometry, amplitude of oscillation, and pure parallel vibration. Automatic setup and constant spacing features are achieved using a digital-to-analog converter (DAC) steered offset potential. The combination of very low driver noise pick-up and data-acquisition system (DAS) signal processing techniques results in a work function (*wf*) resolution, under optimal conditions, of < 0.1 meV. Due to its high surface sensitivity and compatibility with standard sample cleaning and analysis techniques this design has numerous applications in surface studies, e.g., adsorption kinetics, sample topography and homogeneity, sputter profiles, etc. For semiconductor specimens the high *wf* resolution makes it eminently suitable for surface photovoltage (SPV) spectroscopy.

INTRODUCTION

The kelvin method[1] is an indirect null-field technique[2] utilized to measure the work function (*wf*) of both metals and semiconductors over a broad range of temperatures and pressures. It determines the arithmetic mean *wf* of a surface and not that biased towards low *wf*[3,4] patches even if they constitute only a small fraction of the surface area, as in (direct) electron emission methods, e.g., thermonic, photo, and field emission.

The above factors are of particular importance in both surface characterization and adsorption studies where high temperatures, strong magnetic fields or low *wf* patches could considerably prejudice the measurements. The vibrating kelvin probe technique[5] has been applied in a wide range of forms having diverse driving mechanisms: electromagnetic solenoid,[6] voice coil,[7] piezoelectric,[8] electrostatic,[9] mechanical,[10] etc., an excellent discussion of which is given in the reviews of Surplice[11] and Rivière.[12]

The extension of this technique to the study of clean surfaces under ultrahigh vacuum (UHV) conditions ($\leqslant 1 \times 10^{-9}$ Torr) has, however, been limited by the availability of suitable materials. To date only a few design variations exist which involve *in situ* mounting of the driving mechanism, e.g., piezoelectric: Besocke (1976),[8] Germanova (1986),[13] and electromagnetic solenoid: Simon (1959).[14]

All other designs rely on some form of mechanical feed-through, i.e., resonant bar, the chief disadvantage of which is the extremely small (often $\leqslant 0.1$ mm) amplitude of vibration, necessitating very small mean spacings. These probes types are consequently very prone to unwanted mechanical noise. Typically the probe design incorporates fixed mounting geometry, often directly above the specimen.[13] This prevents, for example, sputter cleaning of the specimen surface, annealing (due to depolarization of the piezoelectric foil or outgassing), etc., and are, thus, not directly compatible with the requirements with modern-day UHV measuring systems.

We discuss here a new design, based on the voice-coil principle, which features a totally isolated drive system that can produce large (1-cm ptp) amplitudes of oscillation. This is most advantageous in applications where the capacitance of the connecting cables is a dominant factor,[15] or where pickup noise from the driver limits the cpd resolution.[8,11,16,17] The probe suspension system consists of two very thin, circular, stainless-steel disks, vibrating in a perfect plane-parallel fashion having negligible off-axis displacement, and has, thus, general application possibilities as an *in situ* vibration mechanism.

I. VOICE-COIL PROBE

The apparatus is composed of the four basic units sketched in Fig. 1, namely driving element (voice-coil and magnet), optical transducer, suspension-static displacement system, and vibrating reference electrode. In addition, a rotating table, mounted directly below the probe, allows the sample holder to be securely supported during measurement. All the components can be mounted on a single 150-mm flange.

The coil driving element consists of two layers (35 turns/layer) of 0.5-mm oxidized Al wire (Highway International), tightly wound around a hollow stainless-steel cylindrical housing, to which a 300-mm stainless-steel tube has been welded. A permanent magnet (Philips AD 8068) fits snugly over the thin 0.2-mm walls of the coil housing. The small separation distance between coil and housing, necessary to maximize the magnetic field strength, places rather stringent requirements on the suspension system. Various systems were tested; however, the most satisfactory was that manufactured from a 0.2-mm stainless-steel disk by cutting a series of discontinuous radial grooves, see Fig. 2. The two disks used in the final construction coupled flexibility along the axis of vibration with high rigidity for off-axis displacements.

The driving and suspension systems are attached to a

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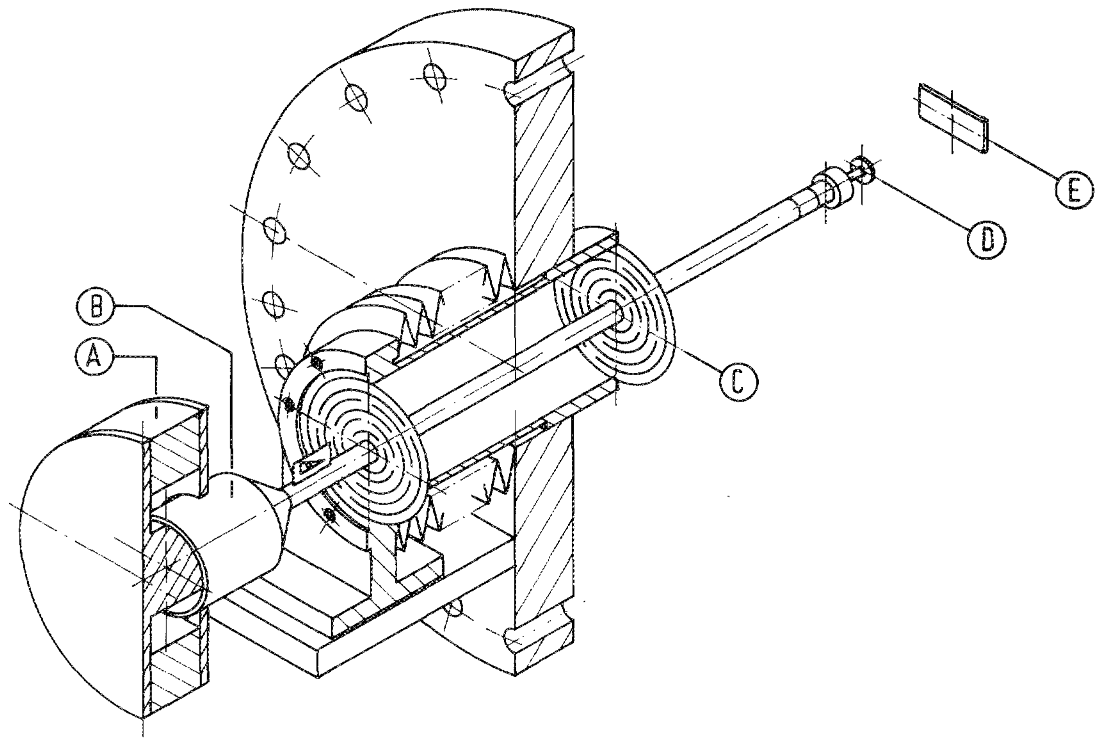

FIG. 1. Voice-coil probe: (a) magnet, (b) coil, (c) disk suspension system with static displacement manipulator, (d) probe head incorporating universal joint and insulator, (e) sample. The split for the optical transducer is shown mounted on the central shaft near the coil.

bellows and manipulator arrangement allowing a 0–30-mm variation in the mean spacing. However, the dc offset allowed by the coil within its housing eliminated the need to retract the probe during sample rotation.

The reference electrode displacement is measured using an optical transducer, comprised of a fixed angled plate mounted under the main shaft near the coil housing, which intersects a narrow light beam. This system is, thus, completely isolated from the Kelvin circuit and can detect displacements of $\pm 10\,\mu$m, further, the detector output can be coupled to the driving circuit to stabilize the oscillation.

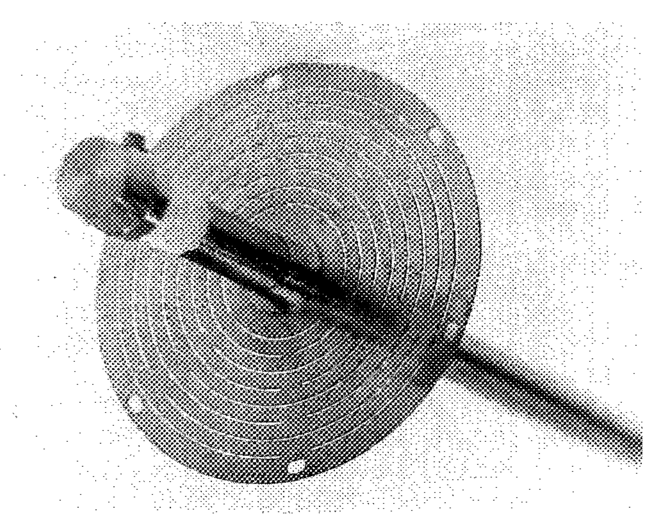

FIG. 2. Detailed view of the disk suspension system.

The reference surface is formed from a stainless-steel disk attached to the main shaft via a universal joint. Alignment of the capacitor surfaces is, thus, easily accomplished by decreasing the spacing until just touching and gently tightening the coupling. For alignment purposes we utilized a portion of the surface above or below the normal measuring position, or a surface parallel with the sample, e.g., strip contacts.

Detailed information on the probe, its associated steering circuit and the transducer optical system can be found elsewhere.[18]

II. KELVIN CIRCUIT

The measuring circuit is composed of three levels as illustrated in Fig. 3. The first level is formed by the central computer where the main (HLL) program resides, the IEEE-488 bus and associated peripherals. The HLL program coordinates the measurement process, communicating with the external modules of level two, i.e., Data Acquisition System (DAS), digital to analogue convertors (DACs), digital oscillator, etc, via specific handling procedures (handlers), and performs data processing and storage. The third level, that of the Kelvin circuit itself, can best be described in terms of four sections: the automatic balance potential (ABP), filter section, impedance-transformer/preamp, and the kelvin probe driver unit. We ignore the parasitic capacitance terms in the latter level for clarity.

The ABP consists of an isolating amplifier (Analogue Devices 289) of unity gain, steered by an 12-bit DAC module (DAC1) and is situated on the high impedance rail to

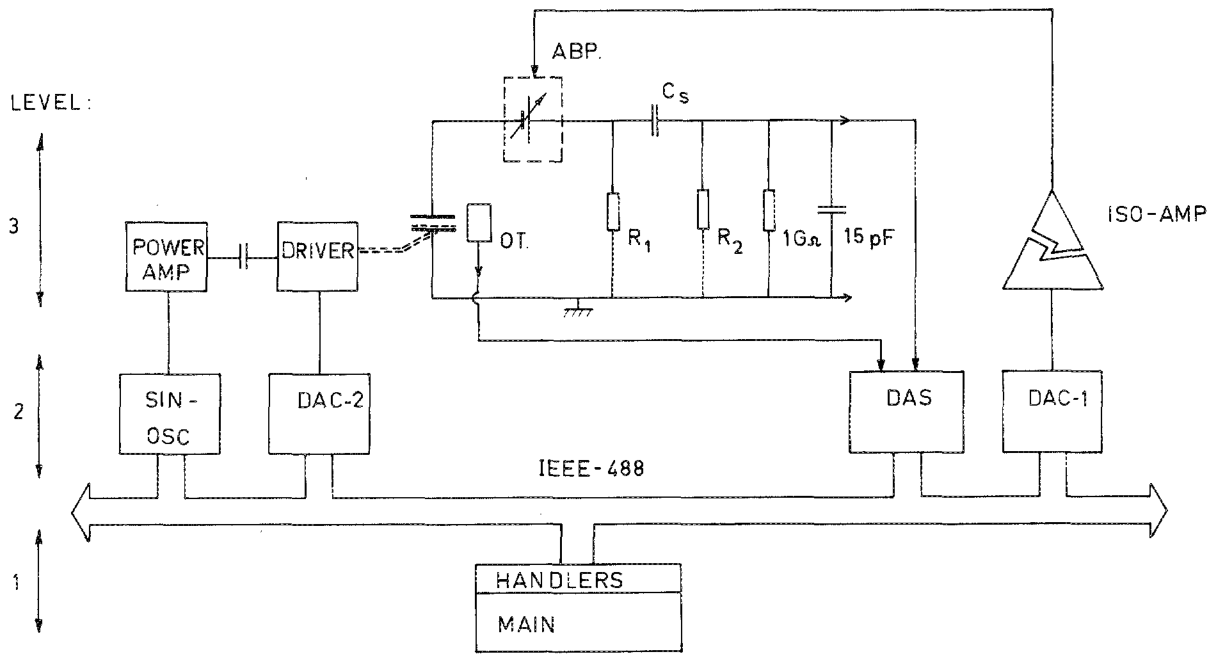

FIG. 3. Three levels of the automatic measuring system; (1) the main computer, handlers and IEEE-488 bus, (2) external modules, (3) the kelvin circuit and driving electronics. OT: optical transducer, ABP: automatic backing potential.

avoid biasing the vibrating electrode.[11] DAC1 can be set, via the IEEE-488 bus and associated handlers of level one, to any discrete value in the $\pm$ 10-V range with a bit resolution of 2.5 mV. Drift in the output voltage of DAC1 is smaller than 10-μV/hr. The contact potential difference (cpd) appearing across the kelvin capacitor upon (external) contact V_{kp} is proportional to the difference in *wf* of each electrode, i.e.,

$$eV_{kp} = e(W_R - W_S) \qquad (1)$$

where *e* is the electronic change and W_R, W_S refer to reference and specimen electrodes, respectively. The peak-to-peak output signal V_{ptp} is proportional to the difference between V_{kp}, and V_{ABP}, i.e.,

$$V_{\text{ptp}} = k(V_{kp} - V_{ABP}), \qquad (2)$$

the constant of proportionality *k* being determined by the circuit parameters.[17] The cpd balance point is determined by measuring V_{ptp} under a stepwise changing backing potential and extrapolating to find the intersection with the V_{ABP} axis. Changes in cpd are, thus, directly related to changes in W_S provided W_R is well stabilized.

The filter section, formed by $R_1C_sR_2$, serves two functions: it allows voltage transients caused by *ABP* switching actions to quickly decay to earth ($\sim$1 s) and prevents static charging of either the specimen or the preamp input (which would otherwise influence the cpd signal).

The preamp (PAR 113, represented by the 1-GΩ, 15-pF combination in Fig. 3) incorporates both roll-on, roll-off filters and variable gain, and its chassis is used as the earth point for all cable shielding. Its role here is mainly that of impedance transformer since direct input to the DAS, although possible, would involve long connecting cables which would diminish the output signal and act as an antenna for noise. The DAS contains dual microprocessor systems (Motorola 6809) in a master/slave configuration and has already been described.[19–21]

Finally, the probe-driver unit consists of a digital oscillator (Keithley), function generator (Waveteck 134) and power amplifier arrangement. The oscillator provides sync and trigger signals for the DAS and function generator. The dc component of the amplifier output is decoupled before the driver and a second 16-bit DAC (DAC2), controlled via the IEEE-488 bus, provides extremely high (0.1 μm) setting resolution.

We have developed software to monitor the probe displacement during operation so that, via DAC2, the Kelvin capacitor mean spacing can automatically be held constant. This active suspension system (ASS) is particularly useful in low temperature applications where, during cooling, spacing changes of 1 mm are involved. Further, the ASS can also be utilized in an automatic set-up mode to move the vibrating plate to a specific spacing or modulation index much more accurately than could be manually accomplished.

III. DISCUSSION

The apparatus has been tested to pressures $< 4\times10^{-11}$ Torr, including several bakeouts at 180 °C. No unusual peaks were found in the residual gas spectrum.

The above probe design is capable of producing large ($>$ 10-mm ptp) plate displacements at resonance, which occurs at 47 Hz. The amplitude of oscillation at the normal operating frequency of 140 Hz is about 2 mm, and due to the nature of construction, is largely insensitive to, say, 5-Hz frequency shifts. No system resonances were found above

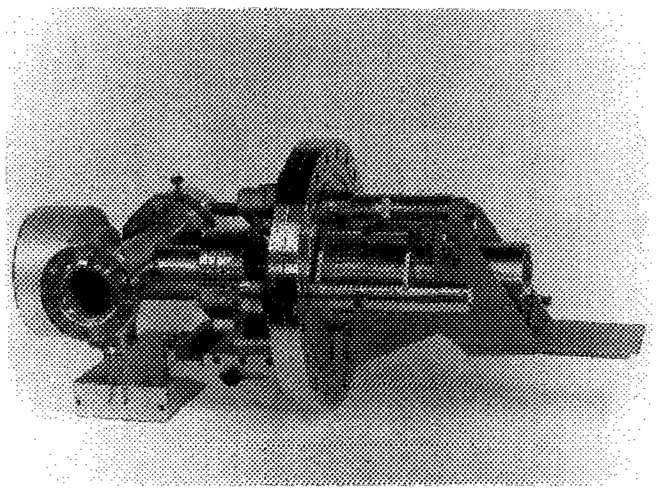

FIG. 4. Complete voice-coil kelvin probe including supporting table.

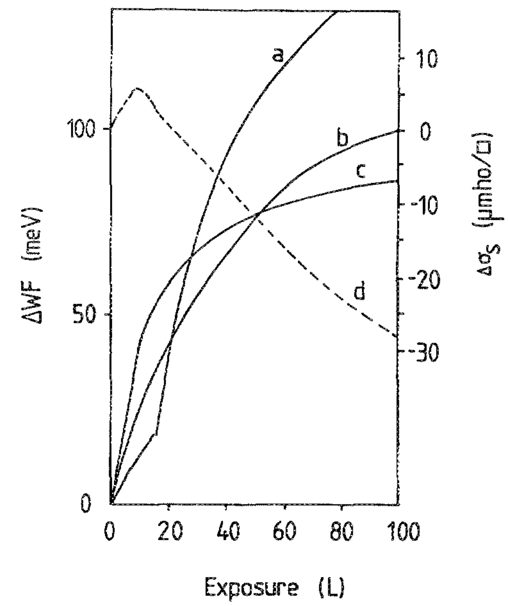

FIG. 6. Initial oxidation stage of Ge (111): *a*, *b*, *c* relate to the change in work function at 300, 200, and 100 K, respectively. Curve *d* refers to the change in surface conductivity at 300 K.

100 Hz. The driver is powerful enough to allow large plate areas or multitipped heads which is useful in that it allows a broader range of application (see below). The supporting table, see Fig. 4, substantially increased the stability of the output signal by damping out high frequency mechanical noise. The output signal was virtually free of pickup noise, that which remained could be removed using software processing techniques.

The voice-coil probe has multiple applications in surface studies due to its compatability with standard sample cleaning procedures and analysis techniques. It is particularly useful in adsorption studies as it can provide, via surface *wf* topography,[17,22,23] a "fingerprint" of contamination or damage of the clean surface.

We have utilized probe heads incorporating two tips: one of large area (78 mm^2) for high resolution (<0.1 meV) measurements, e.g., capacitive coupling effects,[24] adsorption and SPV spectroscopy,[25] and a pointed tip (0.5 mm diam) of moderate resolution (1–2 meV) for surface characterization, i.e., work-function (w.f.) topographies of clean, contaminated, and damaged surfaces.

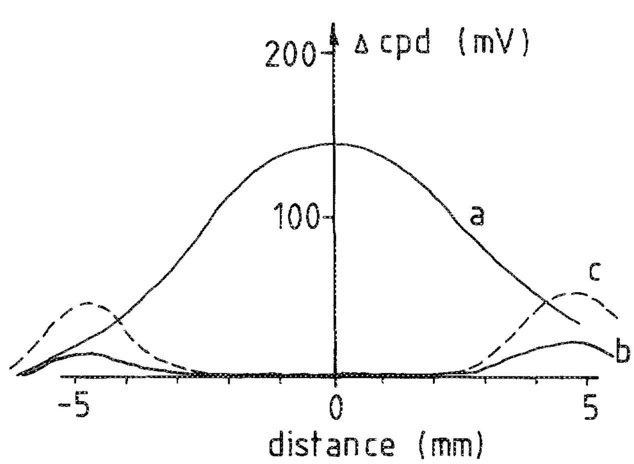

FIG. 5. Work function change Δ *wf* as a function vertical displacement, (a) after sputtering (Ar^+, 500 eV, 45°, $i = 2$ μA cm^{-2}), (b) after annealing, (c) after 30 sputter-anneal cycles.

We consider here two applications of the voice-coil probe which demonstrate use of each tip. Figure 5 shows the variation in *wf* along the length of a Si(111) sample, using the pointed tip probe, after a) 20 min Ar^+ ion bombardment, b) annealing at 800 °C for 1 h and c) 30 sputter-anneal cycles. The lower Δ w.f. values towards the sample ends in curve *a* indicates decreased damage which is in excellent agreement with a smaller ion flux at this region as measured with a Faraday cup. We observe in curve *b* that the sputter damage is not completely annealed out at the extremities, indeed after numerous sputter-anneal cycles (see curve *c*) things "wings" become increasingly prominent.

An example of the large diameter probe is given in Fig. 6, which shows combined w.f. and surface conductance data for the initial oxidation stage of Ge (111) as a function of oxygen exposure in Langmuirs[26] (1 L = 10^{-6} Torr s) at (a) 300, (b) 200, and (c) 100 K. The trend towards higher initial Δ w.f. gradients with decreasing substrate temperature is in agreement with measurements performed at higher temperatures.[27] The conductivity and *wf* measurements at 300 K, curves *d* and *a*, respectively, indicate that the small shoulders in Δ w.f. at 6 and 17 L can be attributed to small decreases in the surface potential, i.e., an increase in conductivity, superimposed on the predominant dipole interaction.

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