UNIVERSITY OF ILLINOIS

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..... Joe John Hanson ....

ENTITLED...The design and construction of a piezoelectric ....

..... driven Kelvin probe. ....

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF......... Bachelor of Science in Chemical Engineering ....

.... College of Liberal Arts and Sciences ....

Instructor in Charge

APPROVED:....._Richard Alkire_.........

HEAD OF DEPARTMENT OF...

The design and construction of a

piezoelectric driven Kelvin probe

by

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Thesis

for the

Degree of Bachelor of Science

in

Chemical Engineering

College of Liberal Arts and Sciences

University of Illinois

Urbana, Illinois

1989

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Abstract/Summary

A Kelvin probe was designed and constructed based on a piezoelectric crystal as a driving mechanism for oscillation. The Kelvin probe is a device which measures the work function (contact potential difference) of a solid relative to a reference substance. The Kelvin probe works on the principle of the vibrating capacitor, ie. if two conductors (a reference and a sample) are placed in close proximity to each other, a difference in work function will create a potential difference between the two conductors, and the set up approximates a capacitor. If the distance between the conductors is varied, the capacity is also varied, and a detectable current is generated. If an external compensating voltage is applied to one surface such that the contact potential difference (cpd) equals zero, then the generated current will also equal zero. The compensating voltage that is required to create this condition is equal in magnitude and opposite in sign to the value of the work function of the sample relative to the work function of the reference electrode.

A gold on tantalum electrode was attached to the piezoelectric crystal to form the working 'tip' of the probe. The piezoelectric crystal was made to oscillate with an approximate total amplitude of 100 microns by applying a 10 volt, 100 Hz alternating current driving voltage to the crystal. Variation of the power from a 1.5 volt battery was accomplished by the use of a potentiometer and was used to null the signal generated by the oscillating probe. The contact potential difference of four different samples was determined with a precision from 90 millivolts to 200

millivolts. Further refinement of the apparatus will continue in the future. More detailed information on the exact construction and operation of the probe is contained within the following report.

Introduction

This report describes the construction and method of operation of a Kelvin probe. This device is used to measure the work function (contact potential difference) of a solid, based on the vibrating capacitor method (Kelvin method). Work function measurements can provide information on the state of a solid surface and the surface uniformity. The purpose of the study was to develop and construct a workable design for a Kelvin probe based on the previous designs found in the literature. After construction of the probe a limited amount of tested was performed on a few different samples in order to ascertain whether or not the design was successful and to introduce refinements into the design.

Survey of Literature/Theory

Information of the theory of operation of a Kelvin probe as well as past kelvin probe designs has been drawn from a number of sources which are listed in the reference section of the report. Specifically [1], [2], and [4] were helpful, as was the design data from a kelvin probe constructed by Dr. J. Abelson.

The work function ($\emptyset$) is defined by the energy $e\emptyset$ that is required to transfer an electron from the Fermi energy to the vacuum level. Two different surfaces, A and B, in the same environment will each have a characteristic work function, $e\emptyset_A$ and $e\emptyset_B$, ie. each surface will have a Fermi energy level at a distance $e\emptyset i$ from the vacuum level where 'i' identifies the surface. When the surfaces are electrically connected, the equilibrium state is one of equal chemical potential which is also equal Fermi energies. In order to obtain this equality, electrons will flow from the surface with the higher initial Fermi level to the lower level. This results in an electron deficiency in one surface, and an electron surplus on the other. The inequality of electron distribution creates a potential difference which is equal to the difference in work function between the two surfaces.

If an external potential is applied to one surface such that the vacuum levels of the two surfaces again correspond, the applied potential is equal in magnitude to the difference in work function between the two surfaces. By making one surface a reference surface with a constant work function, changes in the work function of the other surface can be detected by changes in the amount of voltage that is necessary to compensate for the potential difference. The Kelvin method provides a means to determine when the applied compensating voltage is equal to the difference in work function. If two conducting surfaces, A and B, are arranged such that the separation between them is small (<1mm [1]) the resulting arrangement can be considered a capacitor. Any existing potential difference, V_{AB},

between the two conductors will lead to a charge on the capacitor arrangement according to $Q=C \cdot V_{AB}$. The Capacity, C, is dependent on the specific geometry of the arrangement including the separation distance between the two conductors. Therefore, if the separation between the conductors is varied at a periodic frequency, an alternating current will be generated that can be detected. If an external voltage V_{ex} is applied to one surface, then $Q=C(V_{AB}+V_{ex})$. When the capacity is changed, ΔC, then $\Delta Q=\Delta C(V_{AB}+V_{ex})$. When $\Delta Q=0$ then $V_{AB}=-V_{ex}$. The Kelvin method operates on this nulling principle. If an external compensating voltage is applied to the arrangement as stated before there will no longer be a charge on the capacitor, and consequently there will be no current in the external circuit. Thus when the compensating voltage is applied such that the current is nulled, the compensating voltage is equal and opposite to the work function difference.

The absolute value of the work function for a given sample can not be determined by use of the Kelvin method. However, any change in the work function can be obtained to a reported accuracy of ~1 meV [1]. The Kelvin method is frequently used to determine the change in work function after a gas has been adsorbed onto the sample surface. Since the measurement is taken relative to the reference electrode, the work function of the reference electrode must not change, and must therefore be made of an inert material. It has been shown that gold, oxidized tantalum, and oxidized tungsten are suitable for this purpose [1,4].

Apparatus

Several different mechanisms exist that can provide the oscillating reference electrode necessary for the Kelvin method. The traditional method incorporates buzzers, or loud speaker mechanisms to induce the oscillation [3]. The specific driving mechanism chosen for this design is based on a piezoelectric crystal.

The properties of a piezoelectric crystal are such that when a voltage is applied to the crystal, the shape of the crystal is distorted. If the crystal is held stationary on one end, the applied voltage will result in a deflection of the free end. If an alternating current is used, the piezoelectric crystal will be deflected back and forth at the frequency of the applied alternating current. Simultaneously, a direct current can be applied to the crystal that has the effect of repositioning the zero point about which the crystal oscillates. This principle is used to allow the crystal to be positioned close to the sample surface with potentially greater accuracy than a micrometer alone.

The overall design of the Kelvin probe involves applying an alternating current to the piezoelectric crystal to induce a constant oscillation. A gold tipped probe is attached to the one end of the crystal, and the other is supported on a translational stage attached to a micrometer. The micrometer is used to position the probe close to the sample surface. A current is generated by the vibration of the probe and can be monitored on an oscilloscope. A battery and potentiometer setup is

then used to apply a voltage to the sample which will null the potential difference between the surfaces and therefore null the signal on the oscilloscope. A schematic drawing is provided in figure 1.

Specifically, a nickel plated piezoelectric crystal approximately 1.75 inches long by 0.22 inch wide by 20 mils thick was purchased from Piezo Electric Products, Inc. of New Jersey. With an applied alternating current at 10 Volts the total oscillation amplitude of the free end of the crystal is approximately 75-80 microns. The reference electrode is then attached to the free end of the crystal. The electrode is attached to the crystal to yield a total electrode oscillation amplitude of approximately 100 microns. The electrode itself consists of a 10 mil thick tantalum foil, 0.7 inch by 0.1 inch. A 0.125 inch diameter ball bearing was used to obtain a roughly spherical 'tip' on the electrode. The tip was made by placing the foil strip on an aluminum block, then placing the bearing on the foil and holding it all in place by another aluminum block. The top block was then struck with a hammer. Since the aluminum is a fairly soft metal, the bearing molded an indentation in the foil that was roughly half of a sphere. The convex side of this 'tip' was subsequently plated with gold (Au). Plating was accomplished using an acetylene torch and 0.18mm, 99.995% gold wire bought from Morton Thiokol, Inc. This process was first attempted on a piece of steel foil, but upon heating the foil a large amount of black powder material covered the entire heated surface. The composition of the material is not known, but it appeared to prevent the gold from forming a sufficient bond with the electrode. Tantalum was chosen as a readily available alternative, and when used with Eutector #127 flux, the gold

melted and flowed over the electrode tip surface very readily. The electrode was then glued with a non-conductive epoxy glue to the free end of the crystal where the nickel plating had been scratched off. This electrically isolated the electrode from the crystal surface itself.

The crystal/electrode is held in place by two pieces of copper that form a clamp. The copper pieces are approximately 0.125 inch thick by 0.5 inch wide. One piece is 1.5 inches long, and the other 2 inches long. The 'back' piece is electrically grounded while the 'front' piece is connected to the alternating current power supply. The two are kept electrically isolated from each other by use of Nylon spacers and mica disc washers around two small screws that are used to clamp the pieces together and hold the crystal in place. Copper is used because it is electrically conductive and is soft enough to mold slightly around the crystal and provide good electrical contact. The entire assembly is attached to a L-shaped aluminum bracket, oriented such that the crystal/electrode oscillates in the horizontal plane. The aluminum bracket is attached to the flat top of a single axis translational stage with a micrometer. Specifically a model 4002 from Daedal, Inc. with 0.5 inch of travel and resolution at 1 micron. The entire assembly is secured to a 6 inch by 4 inch by 0.75 inch aluminum base. Top and side view drawings of the constructed apparatus are provided in the appendix as figures 2a & b.

Sheet metal has been bent to form a cover for the apparatus. This is designed to reduce stray electrical noise in the system. The cover is not permanently attached and therefore is easily removed to provide access to

the apparatus. Permanent pieces of sheet metal are attached to the base in some places in order to provide places to attach the ac driving voltage power supply, the compensating voltage power supply, and the detector cable. An additional shield is provided between the crystal/supporting apparatus and the electrode/detector portions of the system.

The power to drive the piezoelectric crystal is generated by a Stanford Research systems SR-50 lock-in amplifier. The signal from the lock-in is 1 Volt at approximately 100Hz. This signal is subsequently amplified to 10 Volts at 100Hz. The amplifier was designed and built by the SCS electronics shop. A drawing of the control panel is provided as figure 3. It also provides the capability to apply a direct current of up to 200 V to the crystal. This DC voltage is useful in positioning the electrode close to the sample surface. The maximum value of 200 V was chosen because the practical limit for the crystal was reported by the manufacturer to be 200 V. Dials are provided on the amplifier to allow variation of the AC signal from 1-10 V, and variation of the DC signal from 0-200 V. The current which is generated by the vibrating probe is picked up by a detector attached to the sample. The signal is amplified by a EG&G Parc model 181 current sensitive preamp. The preamp has scale settings from 10^{-4} to 10^{-9} Amps/Volt with a 6.5 Vrms maximum output voltage. The amplified signal out of the preamplifier is then directed to an oscilloscope or the lock-in amplifier. A compensating voltage of up to 10 V can be applied to the reference electrode itself. This connection is made using a 0.05 mm diameter bare Chromel wire spot welded to the reference electrode approximately halfway between the piezoelectric crystal and the

gold tip. The power is supplied by a battery with a potentiometer to provide for variation in the applied voltage.

Procedure

The piezoelectric crystal begins vibrating as soon as the ac driving voltage is applied. The amplitude of the oscillation is controlled by varying the degree that the 1 volt signal from the lock-in amplifier is amplified. When the driving voltage is first applied, the ac amplification factor is only about 1:1, and the dc voltage is nearly zero. The dc voltage is then increased to approximately 100 volts over a period of about one minute, which deflects the probe tip away from the sample by approximately 1 millimeter. This arrangement allows the probe tip to be placed closer to the sample by decreasing the dc voltage applied to the crystal, and thereby discourages the application of large voltages to the crystal. The ac amplification is then gradually increased. The signals are increased in this way so as to avoid undue stress on the crystal caused by large, rapid deflection. If the sample is small such as a silicon wafer 1cm x 1cm, then the sample is held in placed by a small clip attached directly to the BNC cable which leads to the preamplifier. The sample can be positioned by hand to within a few millimeters of the probe tip. The sheet metal shielding cover is then placed over the system to reduce the effects of electrical noise. At this point the micrometer, or variation of the dc voltage can be used to position the probe tip within a micron of the sample surface. As a standard procedure, it is important not to allow the probe tip

to contact the sample surface since repeated impact will damage the gold plating on the tip and may adversely affect the reproducibility of any measurements. However, when using the apparatus for the first time it may be difficult to determine when the probe tip is close enough to the surface without first touching the tip to the surface, and then pulling the tip slightly away from the surface. Once the probe tip has been positioned next to the surface, a detectable current will be apparent. The actual measurement is then accomplished by applying a dc voltage, V_{ex}, to the probe and varying that voltage until the current that was initially detected falls to zero. The voltage that was necessary to null the current signal is equal in magnitude and opposite in sign to the value of the work function difference between the reference electrode and the sample.

Results

After the physical construction of the probe was completed, the intent of the study was to determine whether or not the design of the apparatus would be able to provide accurate and useful information about surface work functions. Due to the additional complexity that would be caused by attempting to operate the Kelvin probe within a ultra-high vacuum system, the scope of this project was confined to testing that could be performed under atmospheric conditions. Since work functions are

dependent on the state of the sample surface, the inevitable adsorption of the gases and particles present in the atmosphere would be a factor in all measurements. According to Riviere [3], adsorption would in general be expected to increase the work function of a substrate. The amount of the increase depends on how much the particular substrate in use reacts with the adsorbate. For this design, it would be expected that the work function of the gold tip probe would not change appreciably, and that most of the samples tested would demonstrate a higher measured work function relative to the gold tip probe.

Initially, an oscilloscope was used in order to monitor the signals being received by the detector. The procedure involved using the micrometer and electrical positioning of the piezoelectric crystal to vary the distance between the probe tip and the sample. In the first tests the sample was a strip of tantalum foil that was attached directly to the BNC cable. The sample was attached in this way in order to reduce the possibility of the detector picking up stray signals. As soon as the detector was turned on, an amount of alternating current was detected and displayed on the oscilloscope. This signal was generally 80 millivolts on the oscilloscope at a detector setting of 10^{-9} amp/volt. This stray electrical noise in the lab environment could be diminished by placing the sheet metal cover over the apparatus. With the cover on, the noise produced a 10mV signal on the oscilloscope when the detector was on the 10^{-9} amp/volt setting. This value was obtained when the alternating current driving voltage was not connected to the piezoelectric crystal. If the power supply was connected to the crystal and a shield was not placed

near the point where the probe was connected to the crystal a considerable amount of noise was detected on the oscilloscope. This noise was as high as 50 mV shown on the oscilloscope at a detector setting of 10^{-8} amp/volt regardless of the distance from the crystal to the detector. However, when the shield was placed near the point where the probe was connected to the crystal the signal was reduced to a mere 30-40 mV at the 10^{-9} amp/volt setting. This noise level was obtained by using a piezoelectric crystal without the probe attached to the end since the probe should introduce a larger signal as it approached the sample/detector. A 40-50 mV value at the 10^{-9} amp/volt setting was obtained using the actual probe/crystal apparatus when the probe was positioned as far from the detector as possible (approximately 0.5 inch). It should also be noted that the shield design involved passing the probe through a hole in the shield. The original shield had a hole that was only large enough for the tantalum foil electrode, and not big enough for the piezoelectric crystal. It was found that this introduced additional signals that were as high as if the shield were not there at all. The hole was subsequently made large enough to pass the crystal through, and was place around the crystal near the electrode but not so close as to introduce the large erroneous signals.

As the probe is brought closer to the surface, the sine wave signal that is generated begins to increase. The signal will becomes 4X to 6X as large. This signal is dependent on the contact potential difference between the electrode and the sample, therefore the signal can be enhanced by applying an external voltage to the electrode. This is useful when positioning the probe close to the surface as it yields a signal which

is much larger than any noise in the system. The normal work function (contact potential difference) is less than 1V, so that the contact potential difference can be increase at least 10X by applying 8-10 volts to the probe. When the probe gets to within approximately 1 micron of the surface the sine wave begins to become distorted. This distortion is illustrated in figures 4a and 4b in the appendix. This distortion is a good indication that the probe position is close enough to the surface to obtain adequate work function readings. Using the sine wave distortion as an indication of position reduces the need to impact the probe tip against the sample, and therefore prolongs the life of the probe tip. The signal is maximized at the shortest distance between the probe and the sample, and therefore is easiest to detect at this distance. It is also advantageous to have the distance very small as it minimizes the effect of contact potential differences between the electrode and any shielding which may be close to the electrode. The effect is minimized because the distance from the probe to the shielding is much larger than the distance from the probe to the sample, and therefore the sample signal should be larger than any unwanted signal. Once the probe is positioned in this way, a lock-in amplifier is used to obtain actual readings, as the lock-in can filter out many of the unwanted signals whereas the oscilloscope cannot.

Due to the possibility of damage to the probe caused by impacting against the sample surface, and because the generated signal is dependent on the distance from probe to sample, a test for drift in the ac and dc power supplies was conducted. The power supply was connected to a voltmeter, and set to the voltage range that would be used in the

experiments. The test was run for approximately 30 minutes. The drift in the dc voltage was 15 ± 2 millivolts and for the ac voltage 10 ± 3 millivolts over the entire 30 minute period. This corresponds to a drift of approximately 50 millivolts per hour which changes the position of the crystal by 0.2 microns every hour. The typical measurement is on the order of 15 minutes maximum so that the change in position during a measurement is approximately 0.05 microns. I do not feel that under normal circumstances this will make a significant difference in the value of the work function obtained.

Measurements were attempted on a few different samples. The lock-in amplifier was used to read the signal from the preamplifier after the probe had been positioned close to the sample surface using the oscilloscope to detect the distortion of the signal curve when the probe was close to the surface. The actual value of the signal read by the lock-in was not necessary for the measurement, only the point at which the signal became zero was required. Since some variation in the signal was anticipated, it was decided that 'zero' would be defined as a ± 2 millivolt signal to the lock-in. A simple 1.5 volt battery supplied the compensating voltage to the probe. A variable potentiometer was used to vary the value of the compensating voltage from +1.5 volts to -1.5 volts. The positive and negative values were obtained simply by reversing the position of the battery terminals in the system. A Fluke meter was installed in the circuit in order to read the value of the compensating voltage at any given time. The meter had an accuracy of ± 1 millivolt, however the potentiometer only allowed an accuracy of ± 5-10 millivolts. A total of four different samples

have been tested to date. The testing was by no means exhaustive, and will need to be continued along with several refinements of the apparatus, before the Kelvin probe will come close to the accuracy of 1 millivolt reported in the literature [1]. The data from the tests so far are presented in table 1 of the appendix. Different runs were simulated by moving the probe tip away from the sample, then repositioning the probe tip next to the sample, and nulling out the current signal again. Measured values are reported in millivolts. The actual work function can be calculated by adding the millivolt reading to the work function value of the reference electrode/probe. I do not know the actual work function value of the reference electrode/probe, however Riviere [3] reports several values for the work function of gold (Au) which should provide a reasonable approximation. The accepted value for the work function of gold is 5.32 ±.10 eV however there is some deviation from this value when the gold is plated onto some other substrate. Riviere reports data which shows a 0.29 eV increase from gold on glass (4.68) to gold on tantalum (4.97). He also states that these particular measurements were made in a mercury contaminated environment which is why they are much smaller than the accepted work function value of gold. Since the precision of the apparatus and not the absolute accuracy is the main concern of the project at this point, I have simply used the accepted value of 5.32 eV for comparison of the measured sample work functions to accepted values. The four samples that I used were: tantalum, silicon, copper, and aluminum. Each was chosen because of its availability in easily used form. All of the samples were soaked in dichloromethane for ten minutes before readings were taken as an attempt to clean the surface as much as possible. The samples

were however exposed to the atmosphere during testing so that the effect of the cleaning is at best very limited.

Tantalum provided the most precise results with a range of only 90 millivolts for the three measured values. The tantalum sample consisted of the tantalum foil that was fashioned into a 'clip' and soldered to the BNC cable connection. When the average of the three values is used with the accepted value of the gold probe the determined work function for the sample is 4.61 eV. The reported value [3] is 4.75 eV for a difference of 0.14 eV. While the calculated value is relatively close to the accepted value, it should be noted that the reproducibility of this number is still very limited. A range between three readings of 90 millivolts (0.09 eV) is not acceptable when obtaining accurate measurements.

When testing silicon, silicon chip was held in place by the tantalum clip. The range of the three readings was 110 millivolts with an average reading of -517 millivolts relative to the probe. This corresponds to a work function value of 4.80 eV. The reported value is 4.91 for a difference of 0.11 eV. Some difficulty was experienced when attempting to position the probe next to the silicon sample. The sine wave trace on the oscilloscope was extremely sensitive to position of the probe. The sine wave trace would remain perfectly smooth with no visible distortion almost to the very limit of the positioning ability of the apparatus. According to the sine wave trace on the oscilloscope, and the sometimes erratic behavior of the lock-in, I suspected that the probe may have been coming in contact with the sample during the initial testing. Therefore the

reported results for silicon were taken at a probe to sample distance that did not produce a distortion of the sine wave trace. This may have effected the readings that were obtained.

The range of readings for copper and aluminum respectively were 200 and 140 millivolts. The large range of values may have been due to variations in the kind or amount of material that was covering the surface. The calculated value for copper was 4.65 eV, the accepted value 4.29 eV for a difference of 0.36 eV. For aluminum the calculated value was 4.40 eV, the accepted value 4.19 eV for a difference of 0.21 eV. Overall the work functions for each of the four samples were reasonably close to the values reported by Riviere [3]. However, these values are based on a probe work function value which can not be supported by the data available so far, and they are based on measured values with much lower than acceptable precision.

Conclusions

1) The design developed for this report is a workable design that can provide potentially meaningful and accurate data.

2) Operation of the specific probe that was constructed in this study does generate a current signal that is proportional to the contact potential difference between the probe and the sample.

3) The current which is generated by oscillation of the probe can be nulled by the application of an external dc voltage to the probe.

4) The precision of the apparatus to this point has been demonstrated to be in the range of $\pm$ 0.1 volts.

Recommendations

1) Additional work must be done to eliminate the remaining sources of electrical noise. This may be accomplished by additional shielding within the apparatus itself, removal of noise generating devices in the apparatus environment, etc.

2) The precision of the apparatus must be improved. The precision may be improved by eliminating noise as in recommendation 1). It may also be necessary to re-plate the gold onto the tantalum electrode to obtain more complete coverage. This would reduce any interference caused by a signal generated by the cpd between the sample and the tantalum rather than the gold tip.

3) A variable potentiometer with a greater accuracy (~1 millivolt) must be obtained. This is not necessary until the noise is reduced to a lower level, but it will eventually be necessary to obtain accurate work function measurements.

4) Once the apparatus has been shown to give precise reproducible results, it will be necessary to adapt the apparatus for operation within an ultra-high vacuum system. This is only feasible after the precision is down to a few millivolts.

Acknowledgements

I would like to thank Professor John Abelson for providing a first approximation for the design of this probe, and especially for patiently accepting and answering my many questions throughout the course of the project.

Mike Westjohn for his advice with machining and assembling the various supports, clamps, etc.

The SCS electronics shop for their diligence in designing, building, and repairing the ac amplifier and dc power supply.

Pat at PEPI for rushing the piezoelectric crystal to me after discovering that the original order had been lost.

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Appendix

Table 1: Work function measurement data

sample	Vex (mV)			ave mV	range	ØAu[3] (eV)	Øcalc. (eV)
	a	b	c				
tantalum	-660	-710	-750	-707	90	5.32	4.61
silicon	-470	-500	-580	-517	110	5.32	4.80
copper	-550	-700	-750	-667	200	5.32	4.65
aluminum	-860	-900	-1000	-920	140	5.32	4.40

Figure 1: Kelvin probe schematic
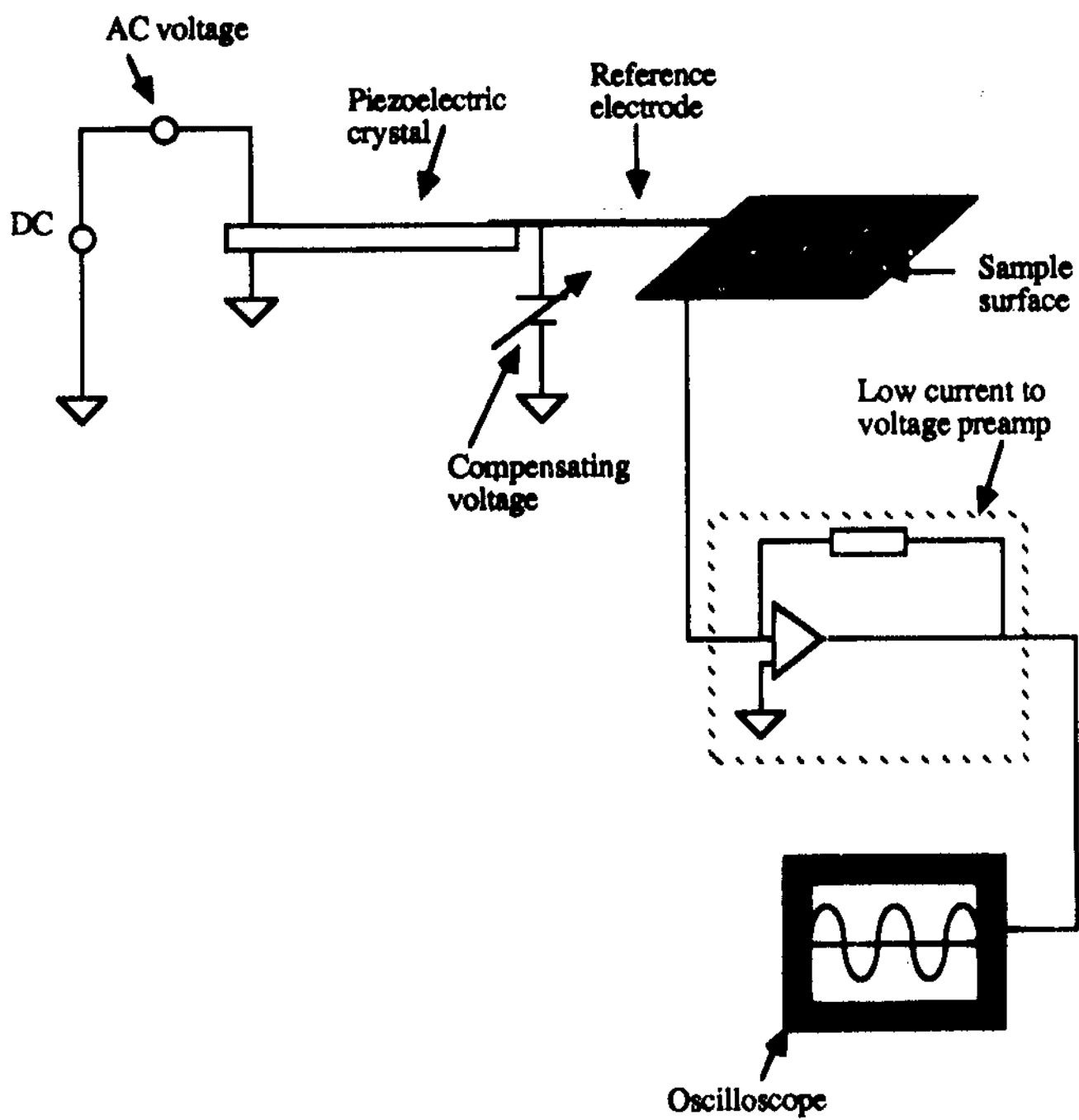
AC voltage

Piezoelectric crystal

Reference electrode

DC

Sample surface

Compensating voltage

Low current to voltage preamp

Oscilloscope

Figure 2a & b: Kelvin probe top and side views

Figure 3: Power Supply Control Panel

BNC input from
frequency generator

Output

AC
0-10X

DC
0-200V

Input
1 Vrms

+

−

Variable dials
range = 0-10

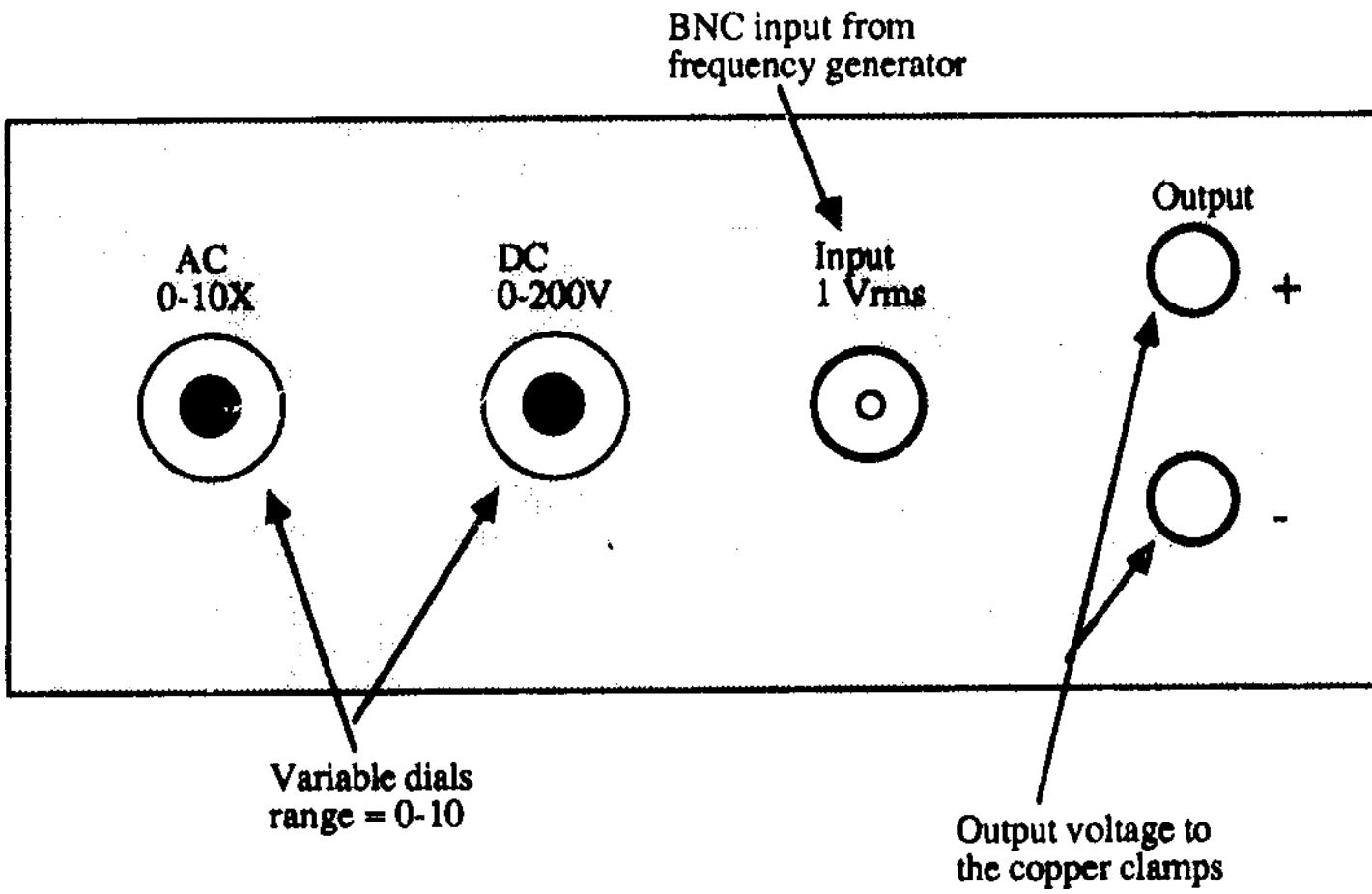Output voltage to
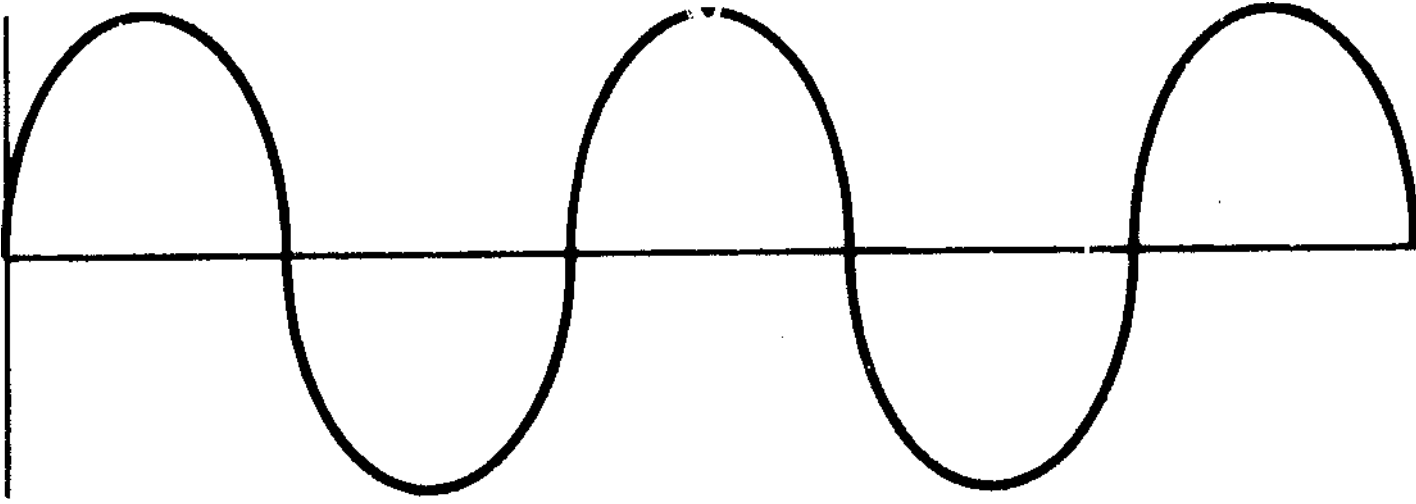the copper clamps
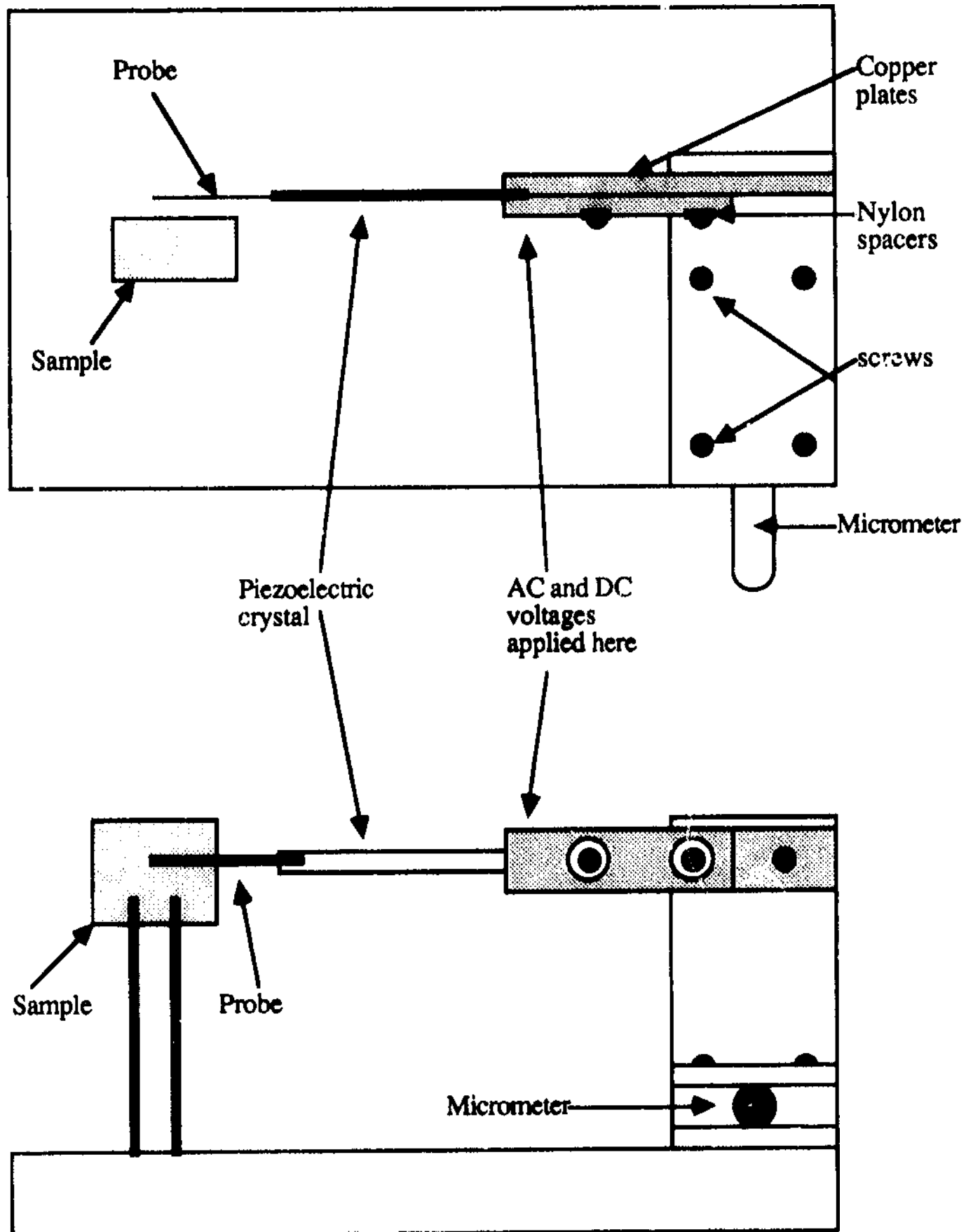
Figure 4a: Sine wave trace with probe away from sample

Figure 4b: Sine wave trace with probe very near sample