SURFACE CLEANING TECHNIQUES
IN ULTRAHIGH VACUUM

By F. J. Brock

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SUMMARY

The objective of this program was to experimentally investigate methods of obtaining atomically clean metal surfaces, to experimentally investigate methods of measuring surface cleanliness, and to investigate the effect of surface cleanliness on adhesion between metal surfaces.

An ion gun having a controllable ion beam density and controllable ion energy was developed to clean surfaces by ion bombardment. Ion bombardment surface cleaning was experimentally evaluated and it was found that the technique produced clean surfaces. Clean tungsten surfaces were produced by an argon ion bombardment dose sufficient to remove approximately one atomic layer of tungsten.

Wire brushing was investigated as a method of cleaning metal surfaces. It was found that of the metals investigated only copper could be cleaned by brushing with a stainless steel wire brush.

The measurement of work function as a method of determining surface cleanliness was investigated. The retarding field diode technique was used to measure the surface work function relative to a reference cathode. It was found that surface coverages of a few percent (and perhaps lower) could be detected by this technique. The experimental results indicate that, if operated properly, the electron gun used in these measurements did not recontaminate the clean surface.

The experimental results indicate that, under proper system operation, a clean surface may be maintained clean for relatively long periods of time (at least of the order of hours).

1. INTRODUCTION

Adhesion between clean metal surfaces has frequently been suspected and sometimes known to be the cause of malfunction in satellites and spacecraft. As the scientific objectives of space research become more sophisticated, the spacecraft and its equipment become more complicated and expensive, requiring longer lead times and much more manpower. All of these place greater demands on the spacecraft designer to achieve unprecedented functional reliability in the spacecraft and its apparatus. The achievement of functional reliability, under space flight conditions, of operating mechanical components requires solutions to a set of formidable problems which were not ordinarily encountered in other fields. One of these problems is the tendency of clean metal surfaces to adhere. Since under space flight conditions most lubricants tend to evaporate and the particle flux in the solar wind tends to remove protective films by sputtering, operating mechanisms which have metal surfaces in contact or which periodically come in contact may have their function impaired by adhesion across the metal interface. Thus, considering that many space flight missions require a relatively long operational life, considerable attention must be given by the spacecraft designer and space research experimenter to the possibility of adhesion occurring between contacting metal surfaces in operating mechanisms.

Adhesion between metal surfaces is known to depend on very many variables such as nearly all material parameters and properties, surface smoothness and flatness, interface pressure, temperature, amount of deformation, and surface cleanliness. A great deal of work is under way to sort out the various parameters and conditions in order to determine those that actually have a substantial influence on adhesion.

This report presents the results obtained in an experimental program which was principally concerned with one of the adhesion conditions: surface cleanliness. It is generally not possible to determine, from the scientific literature, the precise effect that surface cleanliness has on adhesion since surface cleanliness data is usually not given in reports on basic adhesion studies and surface contaminants are usually unknown and uncontrolled.

That surface cleanliness is an important condition in the adhesion process may be clearly illustrated with two examples from common experience. In the conventional use of plain bearings it is well known that adhesion does not occur, even after

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moderate deformation, if the surface is well covered with a lubricant which is stable under high interface pressures. Opposed to this, in the process of roll-bonding metal plates by adhesion, it is found that ordinary cleaning techniques are adequate to achieve satisfactory adhesion. The substantial difference in adhesion in these two illustrations results from the fact that in the first, the surface is sufficiently contaminated such that metal to metal contact between the surfaces is effectively prevented; however, in the second, even though the surfaces may not have been atomically clean in the beginning, sufficient new (and clean) surface is formed in the rolling operation such that the original (contaminated) surface area is a small fraction of the final interface area, and adhesion bonding between the plates in regions of new surface forms a sandwich which is structurally integral. In fact, roll-bonded sandwiches of certain materials show no evidence of the original interface.

It may thus be concluded that surface cleanliness, at least on an atomic scale, is of secondary importance in those applications in which the interface pressure and/or temperature are sufficiently high that large interface deformation occurs. This same conclusion also applies to applications in which material diffusion across the interface is grossly accelerated at high temperatures such as occurs in sintering.

However, experimental evidence indicates that adhesion occurs in applications involving only moderate interface pressure and temperature if the surfaces are sufficiently clean that the metal atoms come in contact across the interface. It is principally with this class of applications that this program is concerned, the objective of which is to begin to define in a quantitative way the relation between surface cleanliness and adhesion.

The first principal step in evaluating the effect of surface cleanliness on adhesion is to establish a reliable method of cleaning the surface such that it is free of contaminants on an atomic scale. The second step (equally important) is to establish a reliable, sensitive, and quantitative method of measuring surface cleanliness. This work is therefore primarily concerned with developing solutions to these two problems which are directly applicable to adhesion experiments. This report presents the principal results obtained in developing at least partial solutions to these problems.

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2. SURFACE CLEANING TECHNIQUES

All of the surface cleaning methods considered here presuppose that the surface is clean in the ordinary sense, since cleaning methods which aim at attaining cleanliness on an atomic scale are more effective in application to surfaces from which the gross debris and contamination have been removed using proven chemical processing techniques.[1] The surface cleaning methods considered generally applicable to adhesion experiments are: thermal degassing, mechanical removal of the surface under vacuum conditions, and ion bombardment.

It is well known that cleaning a surface by vacuum degassing requires a relatively high temperature. Applying the required temperature to an adhesion specimen for long periods of time is frequently undesirable since it may alter the material structure by promoting grain growth, accelerating recrystallization, diffusing impurities into the crystallites, and so forth. A more severe restriction is that the desired degassing temperature may be above the melting temperature of many potential adhesion specimen materials. Thus, vacuum degassing at temperatures sufficiently high to assure that the surface is atomically clean is of limited usefulness. However, when applicable, it produces a clean surface which is smooth and well ordered on an atomic scale.

There exist several mechanical methods of surface removal in vacuum. However, considerable experience has been developed at National Research Corporation in the past with the technique of wire brushing under vacuum conditions, and since most of these methods are roughly equivalent, the wire brushing technique was chosen as a method of obtaining a clean surface. In this technique, the contaminated surface is mechanically removed by the abrading action of the bristles of a rotating wire brush. The surface obtained by wire brushing is extremely rough and disordered.

In ion bombardment surface cleaning, the surface is etched away atom-by-atom until the original surface and the contaminants are removed. In this technique relatively heavy, inert gas ions are formed, accelerated, and directed at the target surface. As the ion collides with the surface, a certain fraction of its kinetic energy is transferred to ions in the vicinity of the impact. If the incident ion energy is large compared with the binding energy of the surface atoms, the energy transferred from

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the incident ion to the surface atom may be sufficiently large
that the surface atom can not remain in the bound state. Thus,
one or more surface atoms receive sufficient energy to break their
bonds and escape from the surface. The atoms that are ejected
from the surface come mostly from the very first layer; however,
a non-negligible fraction of them come from the second layer and
a smaller fraction from the third layer, very few from the fourth
layer and apparently a negligible fraction from deeper layers.[2]
An incident ion may dislocate several other atoms in the vicinity
of the impact. Thus, the final surface may be quite disordered.[3]
After a number of atomic layers are removed, the final surface
can have no more foreign atoms in the surface than the mean im-
purity density in the bulk. Since it is possible to produce metals
with bulk impurities of the order of 1 PPM (or less), it is thus
possible to achieve surfaces which have a negligible contaminant
relative surface coverage by ion bombardment provided only that
the bombarding gas is sufficiently pure. However, impurity levels
of the same order are possible in gases useful for ion bombard-
ment. The gases found most useful are the inert gases since these
produce a minimum change in the electronic configuration of the
surface atoms. A certain fraction of the inert gas ions are
buried in the metal lattice resulting in local defects in the
lattice structure.[3] However, it is generally held that the in-
ert atoms do not disturb the electron distribution in the surface
lattice atoms except insofar as they strain the lattice locally
or dislocate atoms of the lattice. That is, existing evidence
(such as energy of desorption) indicates that the inert gas atoms
are not electronically bound in lattice sites but rather trapped;
thus they have a much smaller effect (if any) on the surface pro-
perties than bound impurity atoms. The surface disorder and de-
fects, as well as the buried inert gas atoms, may be removed by
elevating the surface temperature.

3. SURFACE COVERAGE MEASUREMENT TECHNIQUES

If cleanliness of the surface has a substantial effect on
adhesion, as expected, there must exist some relation between the
cleanliness of the surface expressed quantitatively and the magni-
tude of the adhesion bond. It has been found in other surface
studies that a useful quantitative measure of surface cleanliness
is the "relative coverage," which is defined as the ratio of the
number of surface sites/unit area occupied by foreign atoms to
the total number of surface sites available/unit area. Thus, to
establish a quantitative relation between the cleanliness of the

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surface and the magnitude of the adhesion bond across an inter-
face, some quantitative method of experimentally measuring the
surface relative coverage must be applied to the test surface
immediately prior to an adhesion experiment.

There exist several techniques for measuring surface rela-
tive coverage, (such as low energy electron defraction, energy
distribution of electrons ejected by Auger neutralization, varia-
tion in electron emission or reception with temperature or applied
field) all of which depend (in various ways) on the changes in
the electronic configuration of surface atoms resulting from the
adsorption of foreign atoms on the surface. A number of these
methods were studied with respect to their applicability to the
measurement of surface relative coverage in adhesion experiments.

From the results of this study it was concluded that the re-
quirements of the program were best satisfied by the retarding
field diode technique[4 through 9] which may be used either to
directly measure the specimen surface work function absolutely
or to measure the specimen work function relative to a reference
cathode. The chemical adsorption of a gas on a surface generally
changes the effective work function of the surface. The magnitude
of the change in work function depends on the kind of atom in
the surface, the kind of foreign atom adsorbed on the surface,
and the relative coverage. At low coverages ($\theta < 1/2$), theory[10]
predicts a linear relation between the change in work function
and the change in relative coverage, but at higher coverages,
theory[11] predicts a nonlinear relation. However, nearly all
theoretical relations between the change in work function and the
change in relative coverage are limited in applicability (usually to
low coverage and a specific bonding mechanism) and further are so
approximate that their usefulness is very limited.

In the retarding field diode technique an electron gun emits
an electron stream which is incident upon a collector which is
also the surface of the specimen under study. The electron
current density emitted by the cathode depends only on the cathode
work function and its temperature (apart from some fundamental
constants). The fraction of the emitted electrons which are re-
ceived by the collector depends only on the electron energy and
the work function of the specimen surface. Thus, for a fixed
cathode temperature the electron current received by the collector
depends only on the difference in work function between the cathode
and the specimen and the applied voltage between the two surfaces.
If the voltage applied between the cathode and the specimen

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surface ($V_{k,s}$) is such that the two surfaces have the same barrier height as shown in the energy diagram below, all electrons passing through the gun anode aperture are collected at the specimen surface.
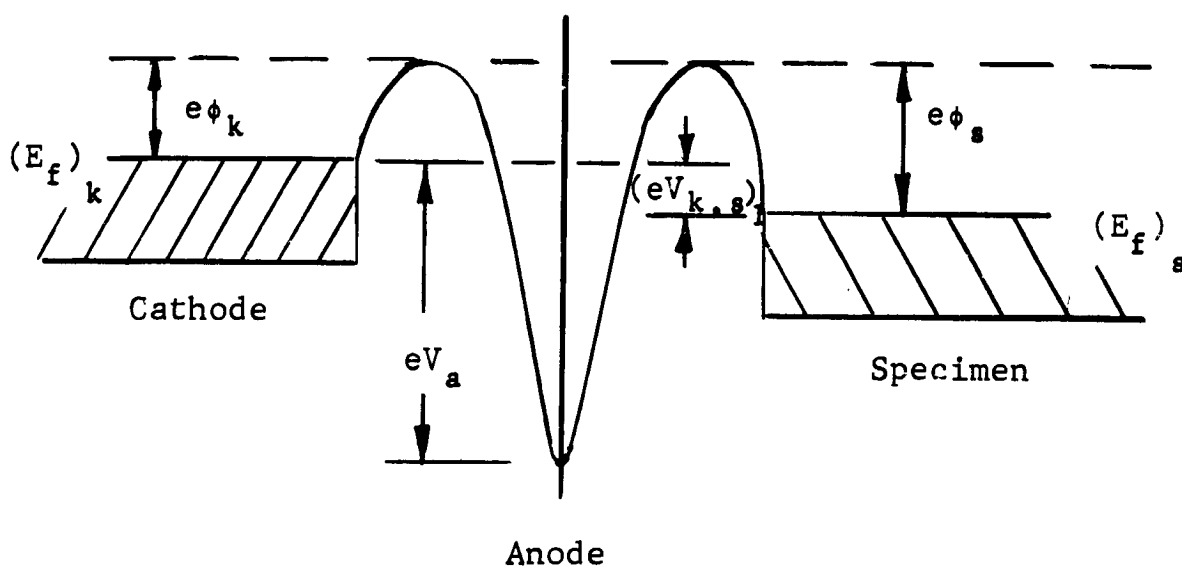
$e\phi_k$ $\equiv$ Cathode work function.

$(E_f)_k$ $\equiv$ Cathode fermi level.

V_a $\equiv$ Anode potential.
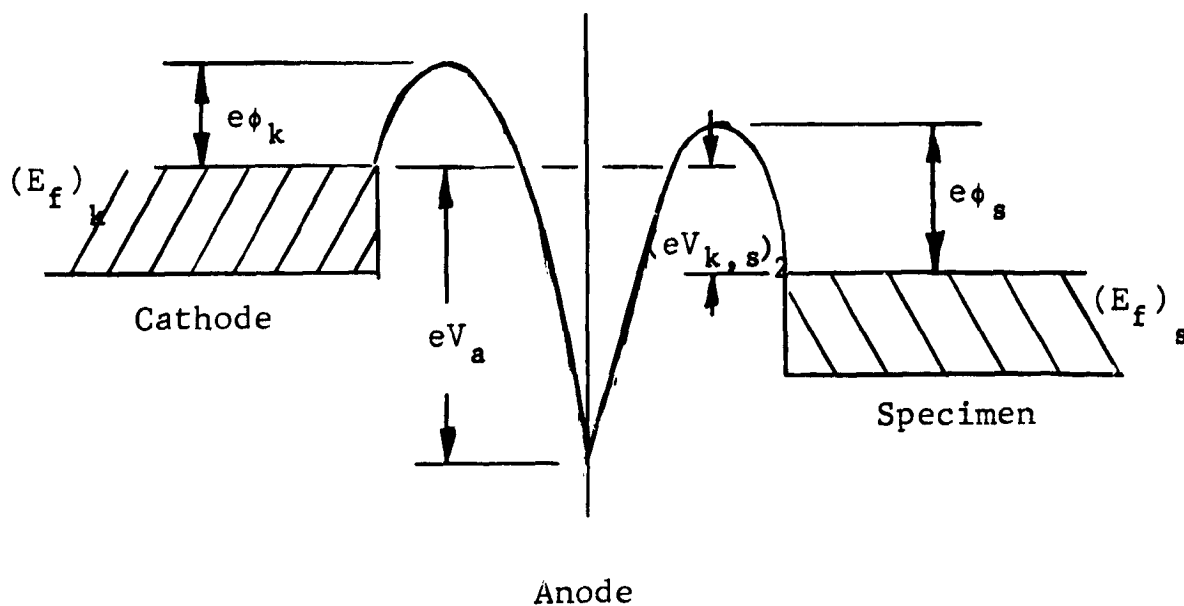
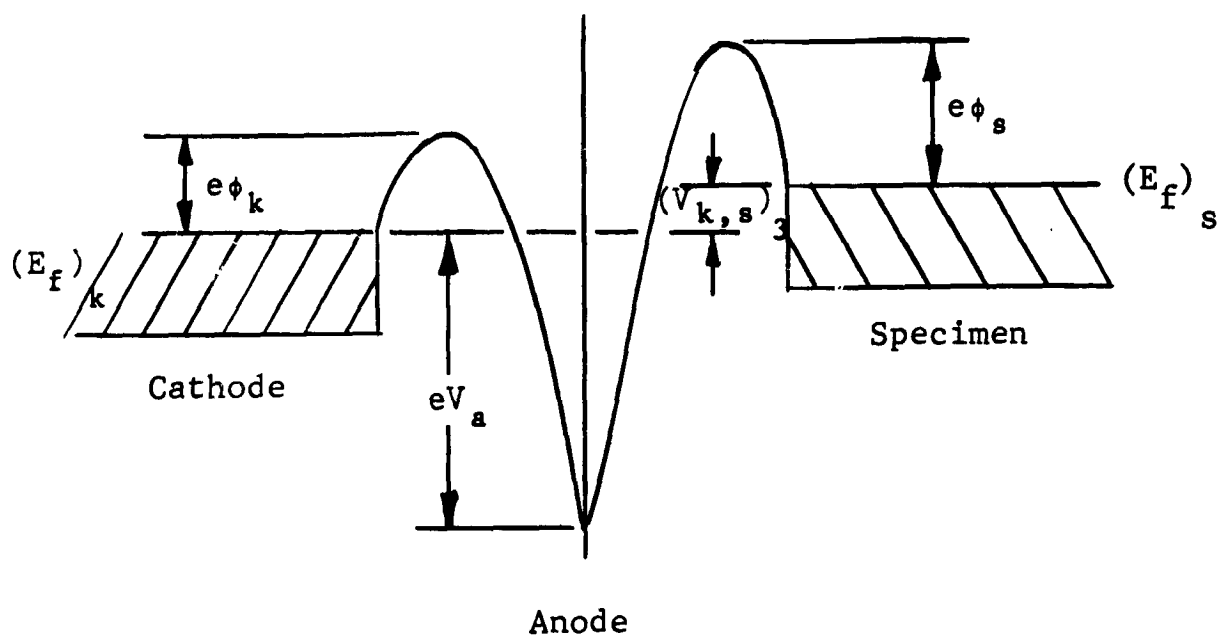$e\phi_s$ $\equiv$ Specimen work function.

$(E_f)_s$ $\equiv$ Specimen fermi level.

If the cathode to specimen voltage is larger than this value (see energy diagram below) the electron current reaching the specimen is unaltered since the field between the cathode and anode is unchanged and the net field between the anode and specimen has increased (accelerating).

Anode

However, if the cathode to specimen voltage is smaller than $(V_{k,s})_1$ (shown in the first diagram) the specimen barrier is higher than the cathode barrier (the net field between the anode and specimen is retarding). Some of the electrons that pass through the anode aperture have insufficient energy to pass over the barrier and are reflected at the specimen surface. (See energy diagram below.)

Anode

In this retarding potential region the specimen current is given by

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$$i = CT^2 \, Exp_o \left[-\frac{e}{kT} (V_c - V_{k,s}) \right]$$

where V_c is the contact potential difference or the difference in the cathode and specimen work functions. Therefore, a plot of log i vs. $V_{k,s}$ (for constant cathode temperature and constant work functions) has the shape illustrated in the diagram below which also shows the three values of $(V_{k,s})$ indicated in the above energy diagrams.
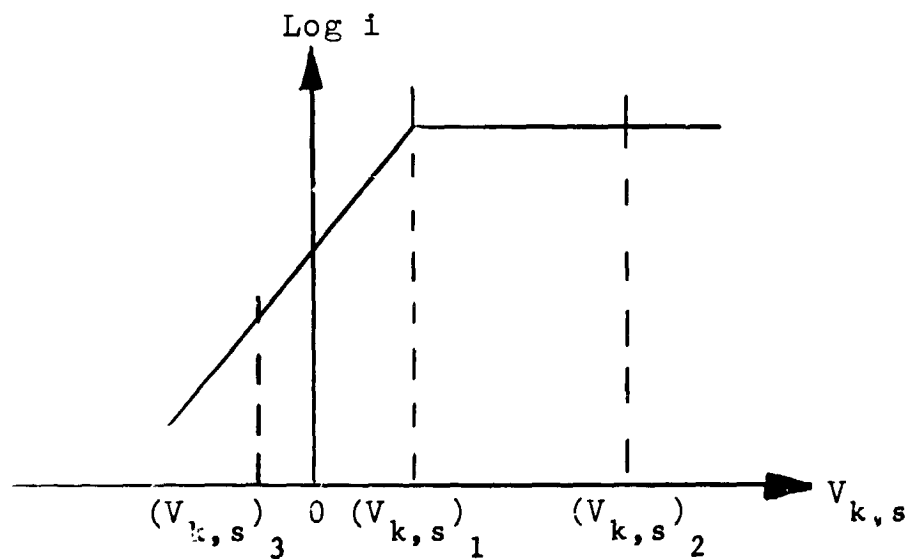
It may be seen from this sketch that the value of $(V_{k,s})_1$ is equal to the contact potential difference.

In an experiment in which the cathode temperature is constant and the cathode work function does not change, the change in specimen work function resulting from a change in the specimen surface relative coverage may be determined by a measurement of the cathode to specimen voltage at which saturation occurs, $(V_{k,s})_1$. For a number of reasons,[4, 5] the experimental curves obtained in such experiments do not generally have the ideal shape illustrated above. However, with respect to determinations of contact potential difference the deviations are usually unimportant. The contact potential difference is then determined by the location of the intersection of the extrapolation of the retarded potential curve with the extrapolation of the saturation curve.

4. APPARATUS

The apparatus developed for this program was installed in a diffusion pumped stainless steel vacuum system capable of achieving pressures of the order of 10^{-11} Torr.[12] (See Fig. 1.)

4.1 Vacuum System

The vacuum system was a vertical cylindrical stainless steel vessel, the upper and lower closure plates of which were o-ring sealed to the vessel flanges. The flanges were refrigerated to minimize the rubber desorption products. Heater strips were distributed over the entire system for degassing.

The main pumping system was a 10 in. diffusion pump above which was an antimigration LN_2 cold trap. This assembly was attached by refrigerated o-rings to a right-angle duct which was welded into the side of the vacuum vessel. The duct contained three internal LN_2 cold traps. The main diffusion pump was backed by a diffusion pump which was in turn backed by a mechanical pump.

On the lower closure plate was mounted an adjustable screw ram (see Fig. 2) which passed into the vacuum vessel through a metal bellows seal. The lower half (cone) of the adhesion specimen pair was mounted in a fixture on the upper end of the ram. A load cell was installed in series with the ram to measure the interface forces applied to the specimen and to measure the resulting adhesion forces. The lower specimen holding fixture was mounted on the ram through bearings to allow for rotating the specimen while under vacuum, for cleaning purposes, by a chain drive powered by an electric motor within the vacuum vessel. The lower specimen holding fixture was equipped with a locking mechanism which permitted vertical motion but prevented rotation. The locking mechanism was necessary to prevent rotation of the lower specimen during abrasion tests. (See Fig. 3.)

On the upper closure plate of the vacuum vessel was mounted a direct rotary drive. (See Fig. 4.) The upper half of the specimen set was mounted in a holding fixture attached to the lower end of the rotary drive. The rotary drive was tripple sealed with refrigerated rubber and teflon seals and each seal compartment was diffusion pumped. The rotary drive provided the
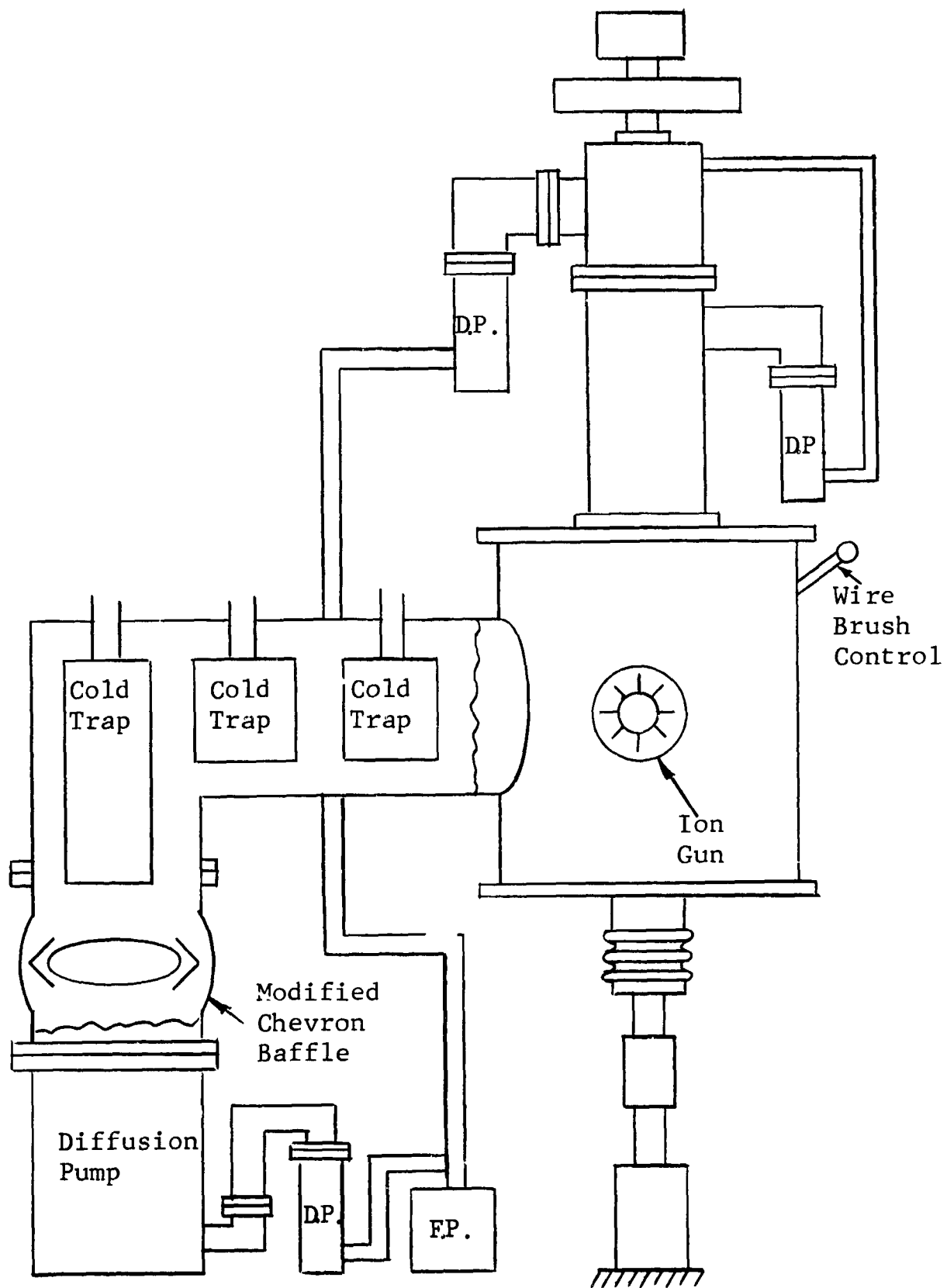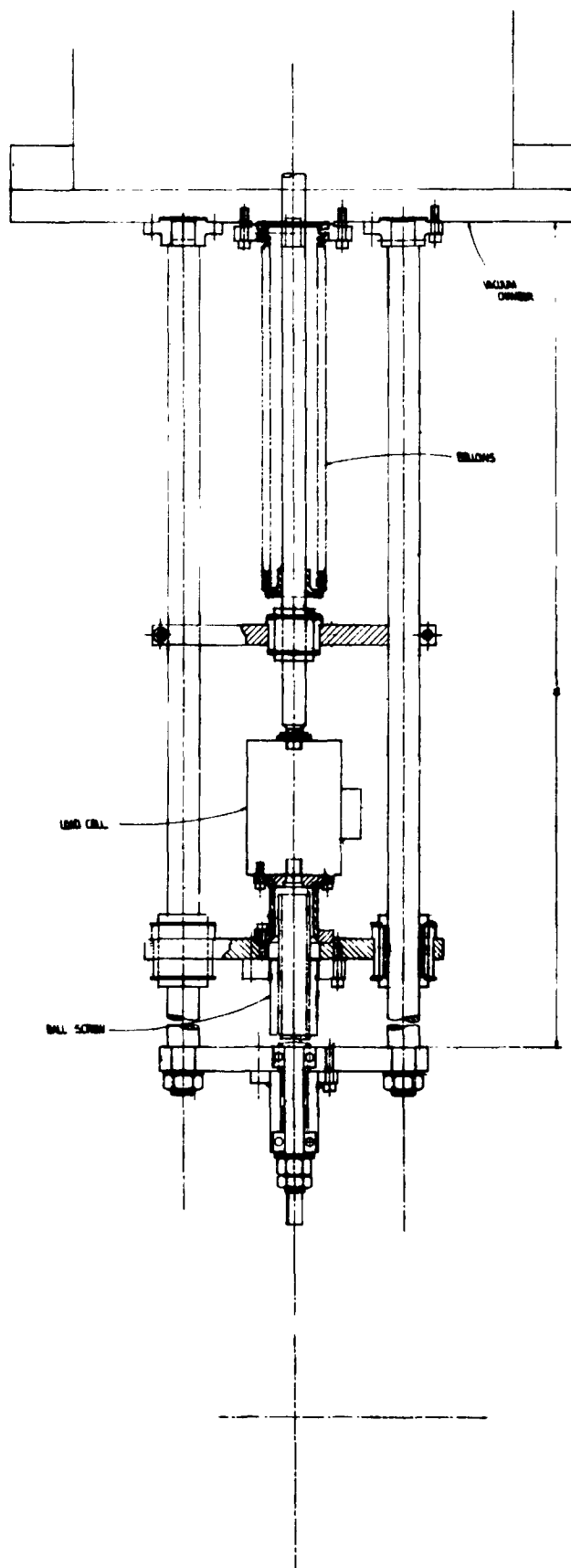
FIG. 1   VACUUM SYSTEM (SCHEMATIC)

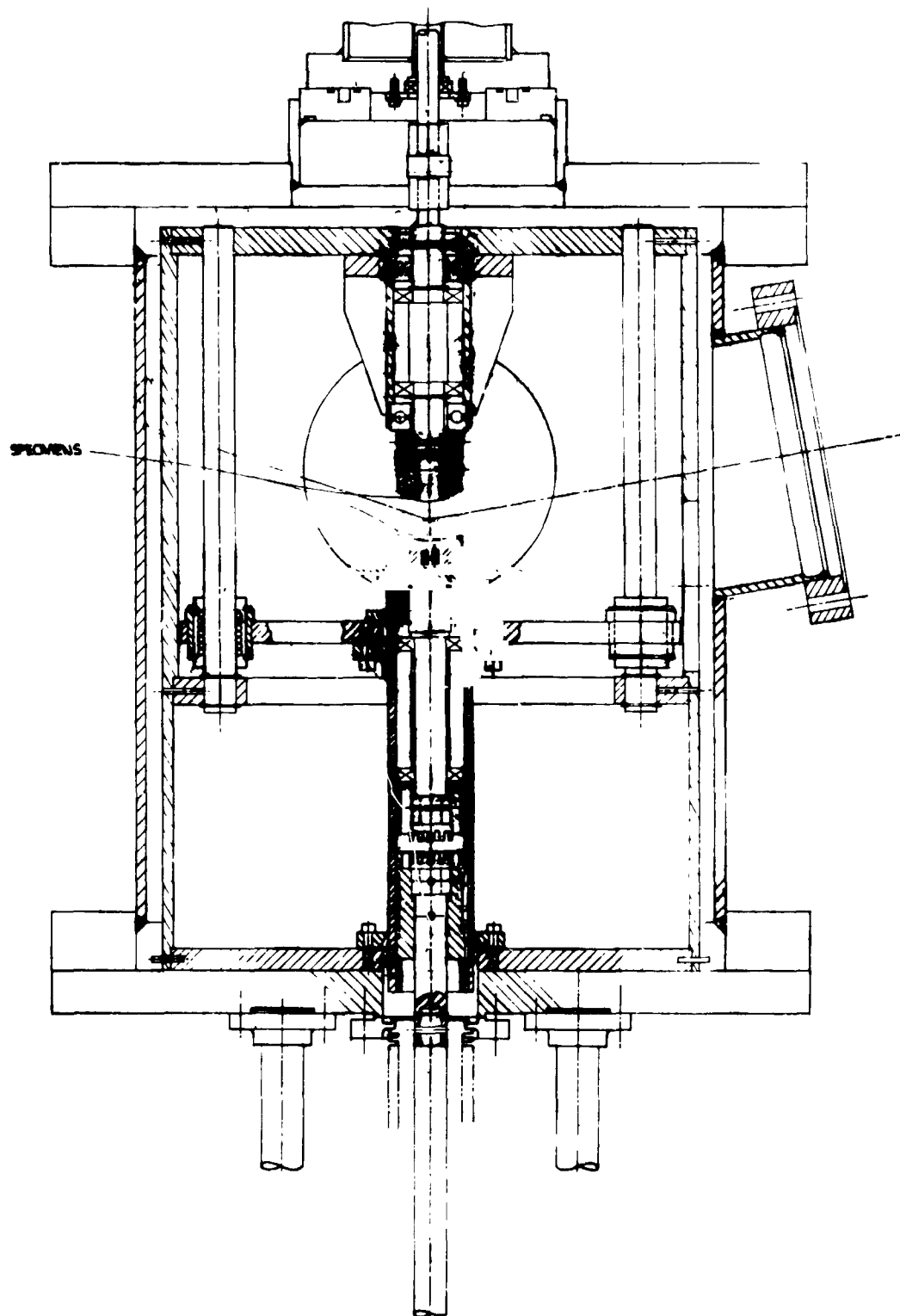FIG. 2   LOWER CLOSURE PLATE (SCHEMATIC)

SPECIMENS

FIG. 3 SPECIMEN MOUNTING FIXTURE

FLYWHEEL
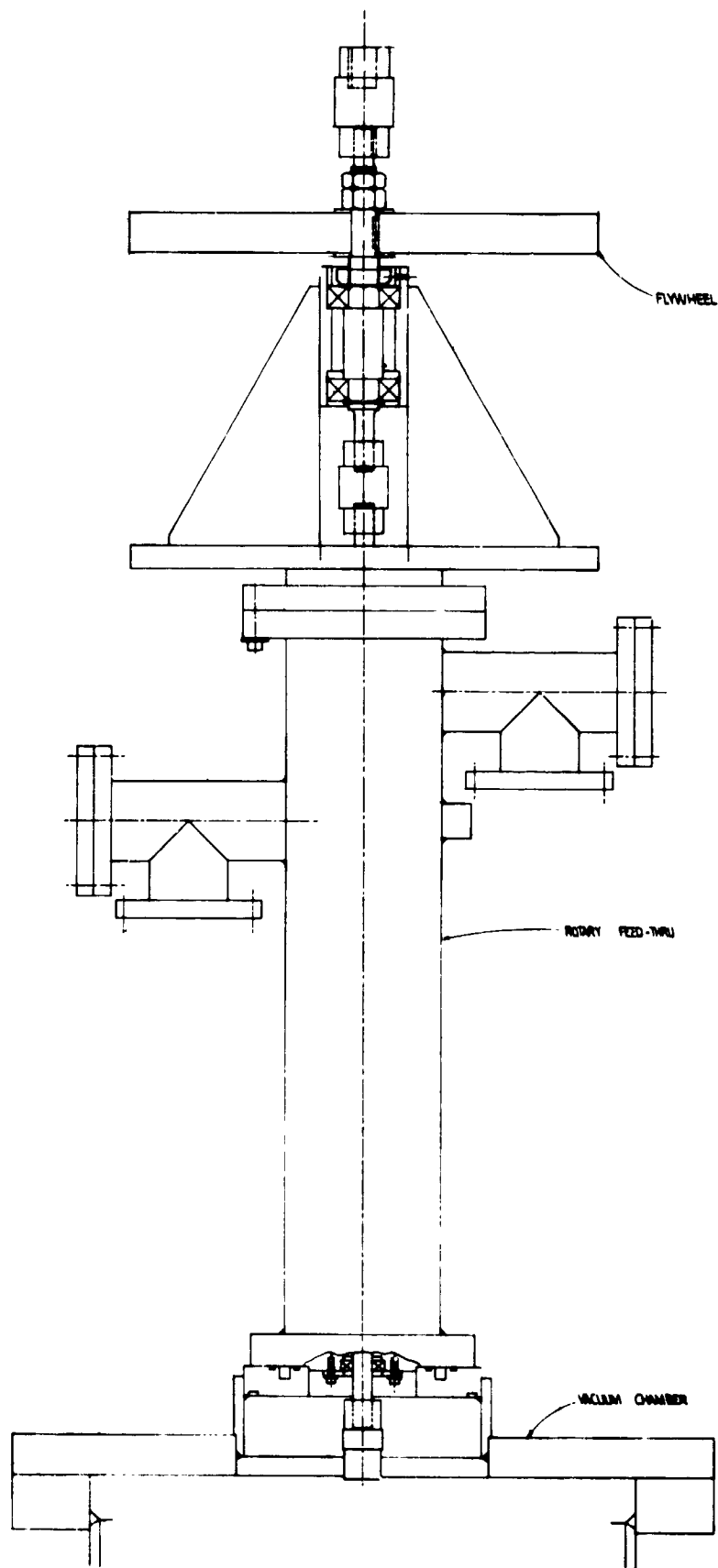
ROTARY FEED-THRU

VACUUM CHAMBER

FIG. 4   UPPER CLOSURE PLATE

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rotational degree of freedom required for cleaning operations, measurement of surface coverage, and abrasion tests. The shaft seal at the upper closure plate was a low conductance seal and the housing compartment above this seal contained an LN_2 cold trap between the seal and the pump port. The shaft seal at the upper end of this compartment was teflon.

4.2   Ion Gun

The ion gun developed for use in these experiments was designed to operate at moderate pressure levels with an extracted ion beam current density corresponding to sputtering rates up to 1/10 monolayer/sec. The ion gun was mounted on, and enclosed within, the main vacuum system. The ion source has some similarity to both a magnetron and a Penning tube. (See Fig. 5.) It is an axially symmetric structure in which the central cathode extends only part way into the anode. The magnetic field (600 to 800 gauss) is axial. The central cathode is attached to a cathode disc which closes the discharge space. The gas is injected radially into the discharge space through orifices at the end of the central cathode. Ions are extracted from the opposite end of the discharge space through a 95% transparent tungsten screen cathode disc. The extracted ions are focused into a beam as they pass through the cylindrical lens system. Electrostatic deflection plates at the lens exit provide for deflecting the beam up to 5° to correct for alignment errors between the ion gun axis and the target (specimen).

The performance characteristics of the ion gun were experimentally determined over a sufficiently wide range to include all normal operating conditions.

The ion beam current density is approximately a linear function of pressure over the range 10^{-5} to 10^{-8} Torr. The beam current density also is approximately a linear function of anode voltage over the range 1 to 8kV. The beam current density radial distribution is such that the average current density at 1/2 m over a 1 cm diameter Fariday target centered on the beam axis is approximately nine times the average current density reaching a 4 cm diameter target behind the Fariday collector. The beam ion energy distribution was measured using the conventional retarding potential method with a Faraday collector appropriately guarded by suppressor grids. It was found that the maximum ion
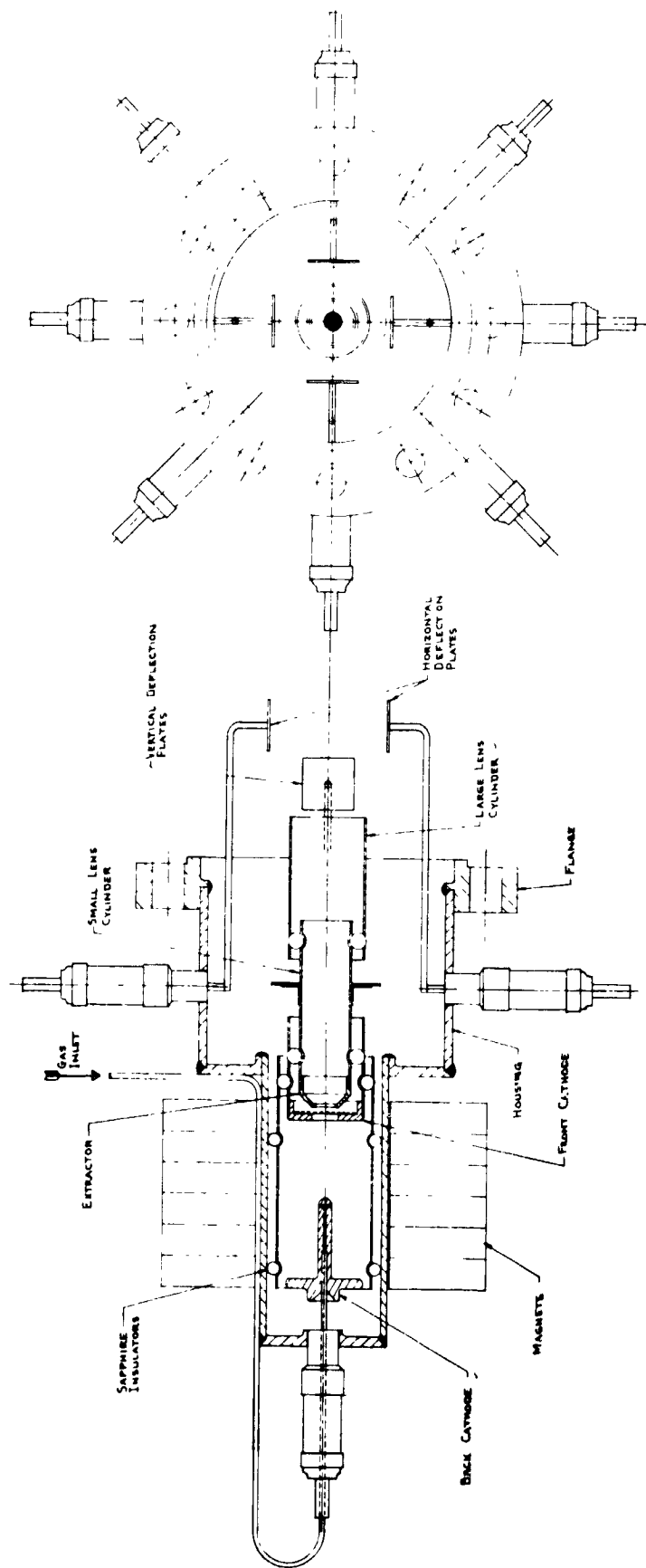
15

FIG. 5   ION GUN

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energy over the anticipated operating range was approximately $2/3\ eV_a$ where V_a is the anode voltage. Typical energy distribution curves are presented in Fig's 6 and 7.

4.3 Wire Brush

The apparatus used in these experiments to perform surface abrasion was a stainless steel (304) wire brush. The brush bristles (1/4 mm dia.) were wound into a twisted, four wire core which also serves as a drive shaft (5 mm dia.). The brush bristles extend radially such that the diameter of the brush was 1.3 cm. The useful brush length was approximately 2 cm. The brush shaft was secured to a motor shaft with a coupling. The motor mount was secured to gimbals inside the vacuum system and a tail shaft extension passes through a flexible bellows seal at the vacuum wall to the outside of the system. The brush position and the brush-specimen contact pressure were manually controlled by this shaft. The unloaded motor speed was 600 rps but during the actual brushing operation the speed was substantially lower since the motor was a low torque motor.

4.4 Electron Gun

The electron gun used for the contact potential difference measurements is shown schematically in Fig. 8. A rolled tungsten ribbon cathode was used to minimize the number of lattice planes exposed at the surface.[5][13] Several guns were made and used. Microscopic inspection of the cathodes after use revealed that the tungsten ribbon had recrystallized (probably during degassing) into a small number of relatively large crystals, some of which extended across the full width of the ribbon and along the length for several mm. It is thus probable that the extracted electron current was emitted from a single crystal face. The attachment rods and support posts were also made of tungsten to avoid any limitation on the cathode degassing temperature since during degassing the end of the support posts near the cathode reached temperatures in the neighborhood of $2000^{\circ}C$. It is essential that the cathode support posts be thoroughly degassed to prevent recontamination of the cathode by contaminants diffusing out of the supports during long periods of operation at elevated temperatures. The anode aperture was approximately 1/2 mm diameter. A wide angle cone of material was removed from the outside of the anode plate around the hole to minimize the effective thickness of the anode at the aperture.

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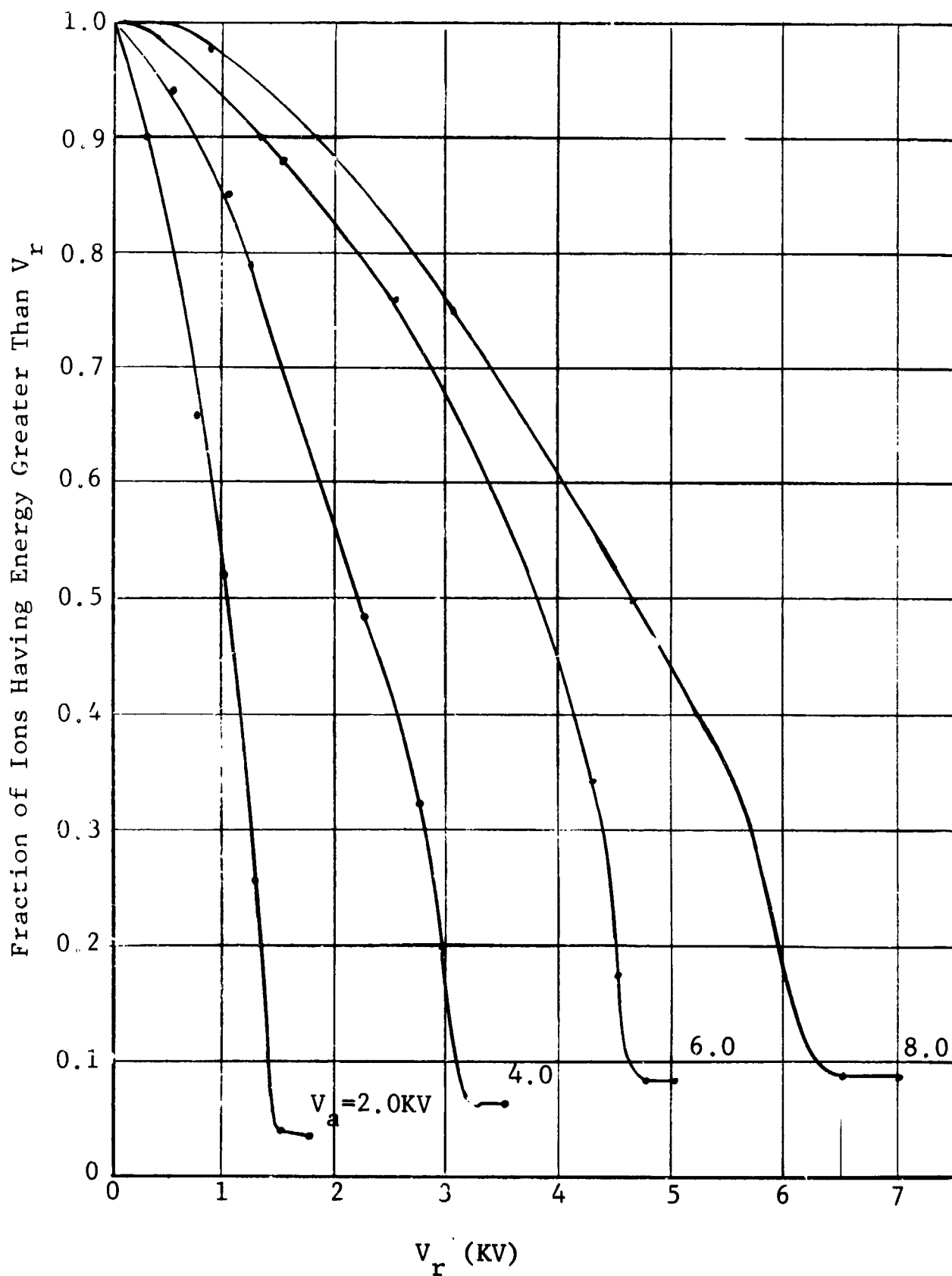
FIG. 6   ION ENERGY DISTRIBUTION (P $\simeq$ 10^{-7} TORR)

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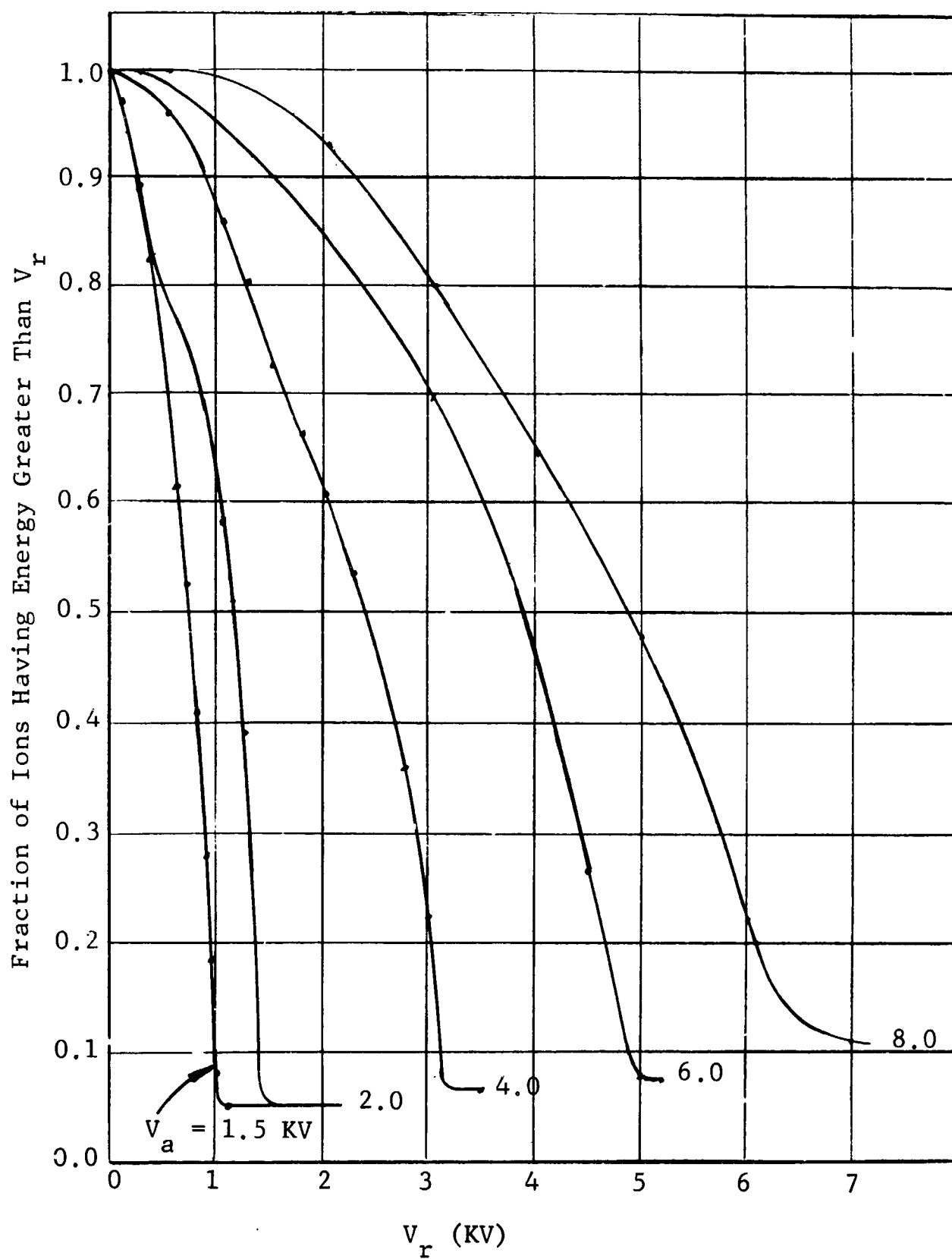
FIG. 7   ION ENERGY DISTRIBUTION (P ≃ 10^{-8} TORR)

Al$_2$O$_3$ Supports

Tungsten Support Posts

Anode (Nickle)

Nicrome V Mounting Posts and Current Leads

0.5 mm

Tungsten Attachment Rods

1.0 mm

Tungsten Ribbon Cathode
.025 mm Thick
2.0 mm Wide
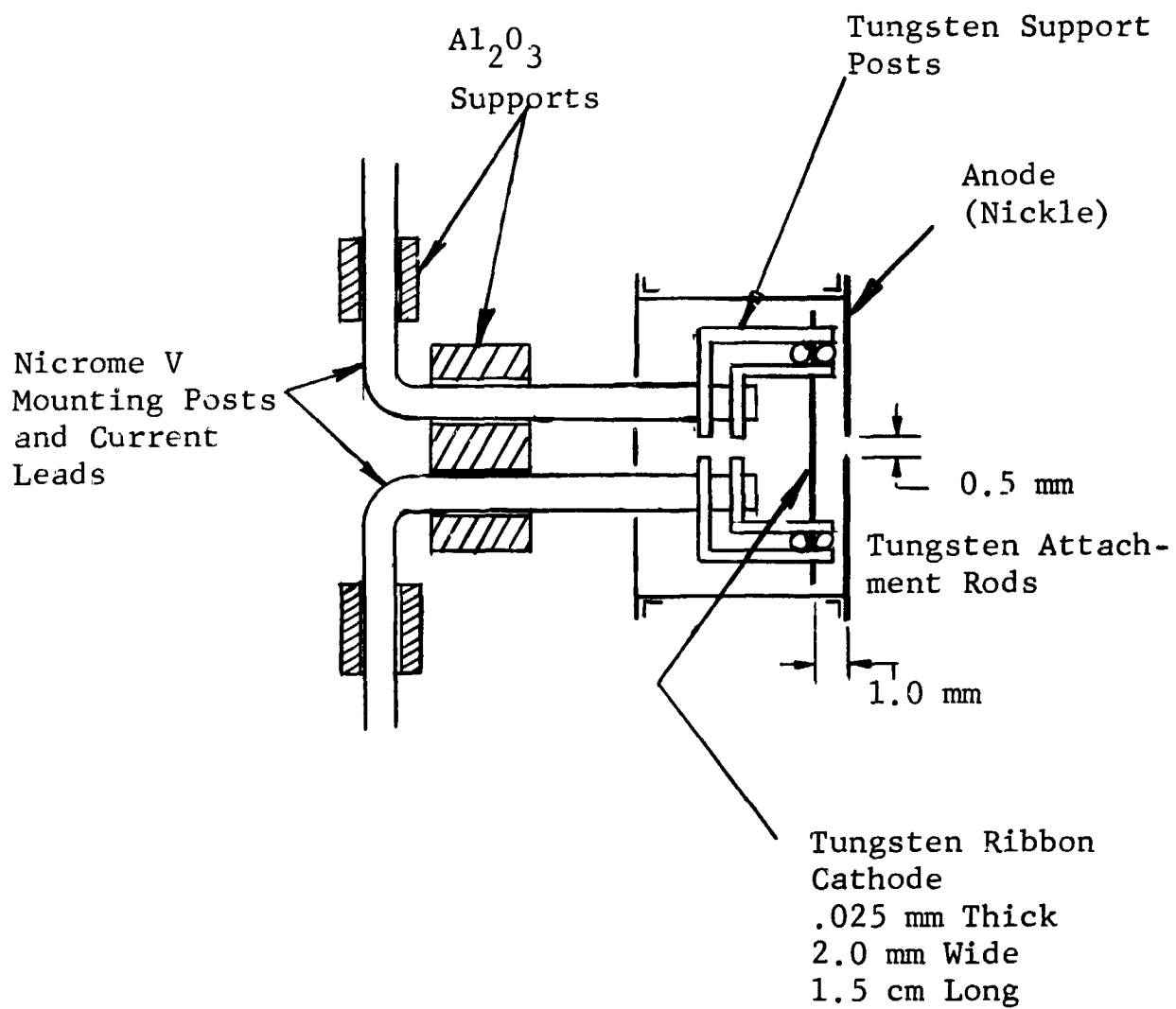1.5 cm Long

FIG. 8 ELECTRON GUN SCHEMATIC

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4.5   Instrumentation and Controls

The emission current control circuit used in the contact potential measurements is shown schematically in Fig. 9.   A voltage proportional to the electron gun anode current is derived by tapping off the desired fraction of the anode current IR drop through a variable potentiometer.   A bias is added to this voltage and applied to the input of an operational amplifier.   A large fraction of the operational amplifier output is feedback (out of phase) to the low end of the anode potentiometer winding.   The amplifier output is applied to the input of a programmable dc power supply, the output of which is the cathode heater current. Thus, a decrease in emission current applies a correcting voltage (through the amplifier) to the power supply which increases the heater current and thus the cathode temperature and returns the emission current to its set value.

It was observed that normal line variations produced emission current variations which were less than 0.1% of the set value. An anode accelerating voltage variation of 50% produced no observable change in emission current.   Target (specimen) voltage variations of + 8V produced no observable change in emission current.   Since many of the circuit components are floating, the target potential may not be changed rapidly.   It was found that to achieve best accuracy in plotting the target current vs. target voltage ("retarding potential curve"), 5 to 8 min. were required to sweep the target from -6V to +8V.

5.   PROCEDURE

5.1   Specimen Preparation

Tungsten, stainless steel (304) and copper (OFHC) specimens were manufactured according to the drawings in Fig's 10 and 11. After finish machining, the specimen test surfaces were polished using standard metallurgical specimen preparation techniques and equipment; the final polish was performed with a diamond paste having a mean grit size of $\sim$1/2 micron.   The specimens were then ultrasonically scrubbed in a detergent solution and rinsed in distilled water, then ultrasonically scrubbed successively in acetone and alcohol and distilled water.   Following this preliminary preparation, the specimens were etched in a formic acid - hydrogen peroxide solution[1] at 80°C and finally ultrasonically
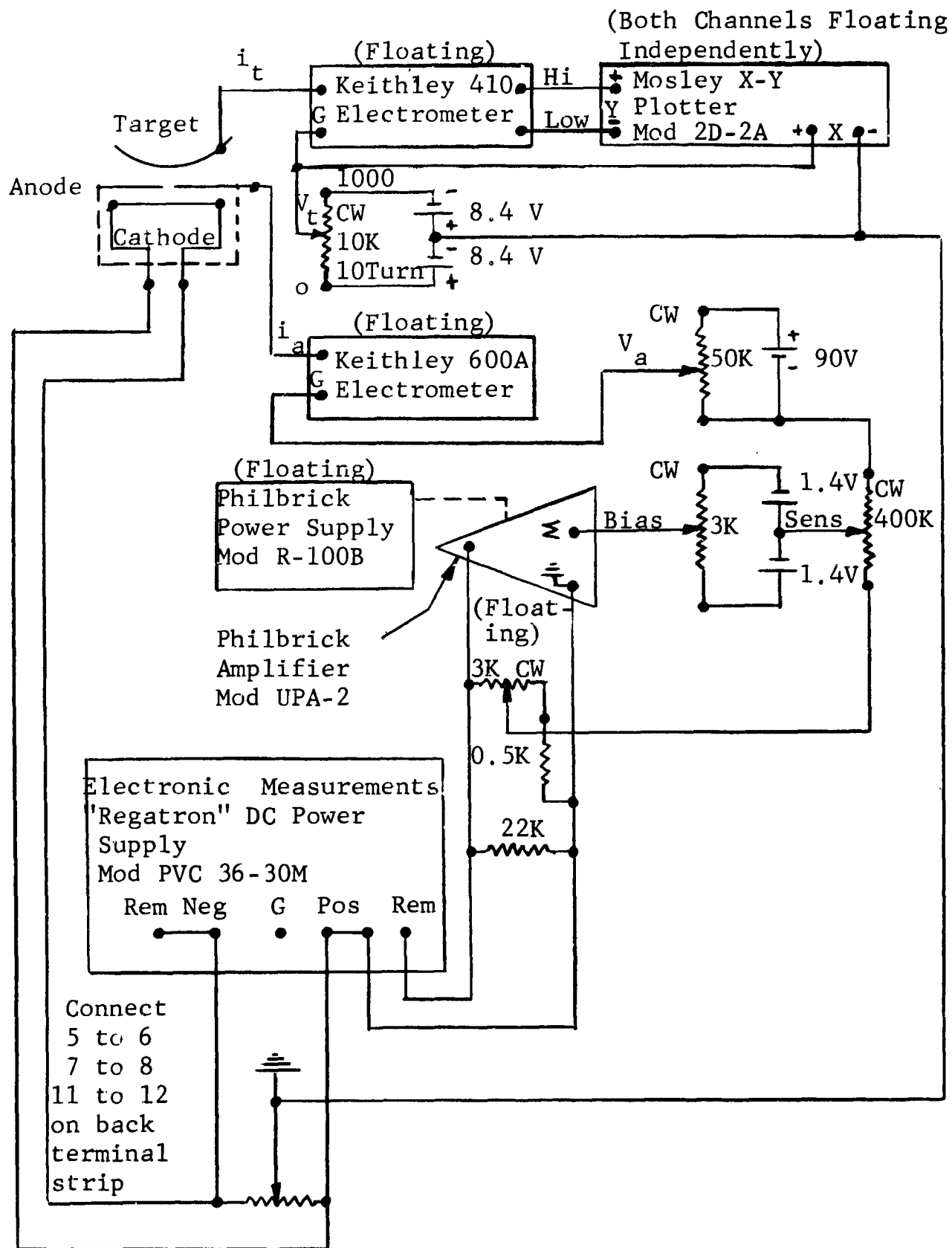
FIG. 9 INSTRUMENTATION AND CONTROLS (SCHEMATIC)

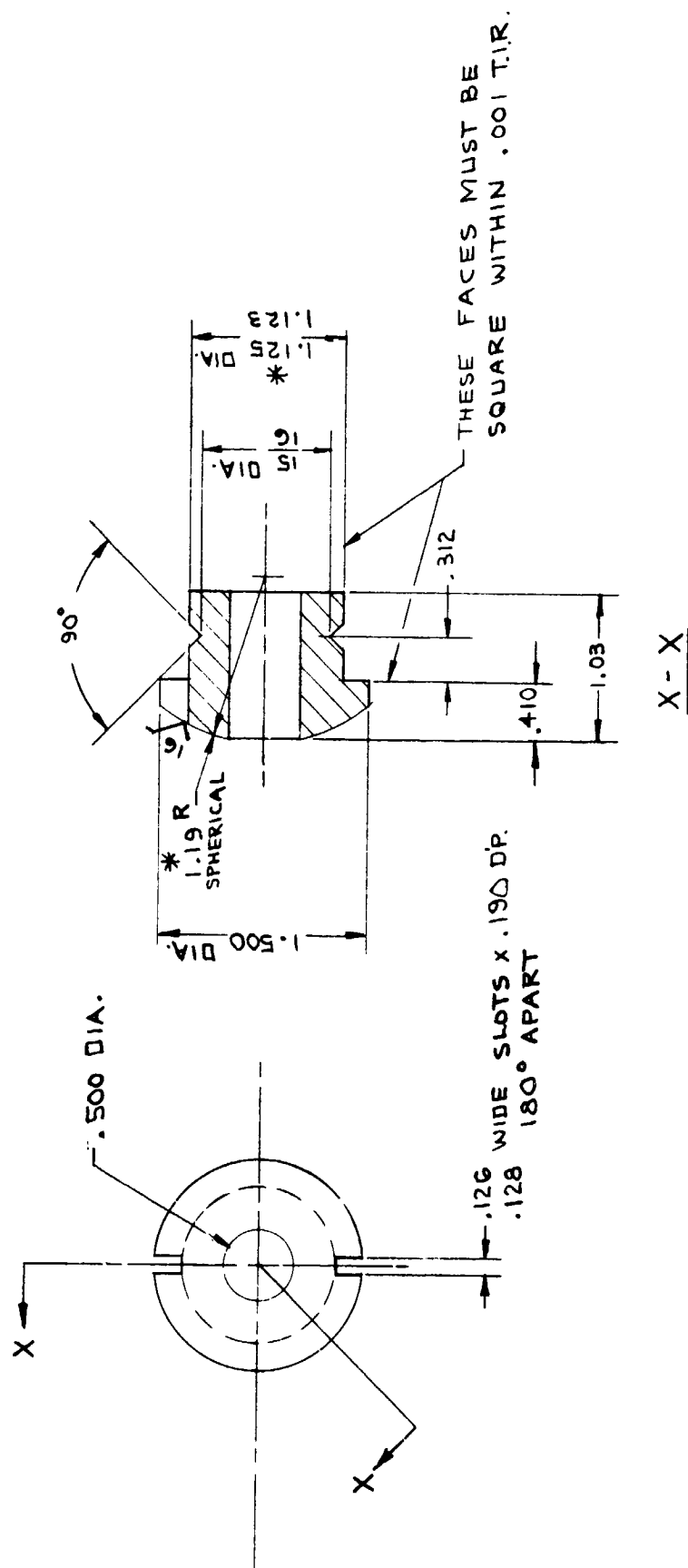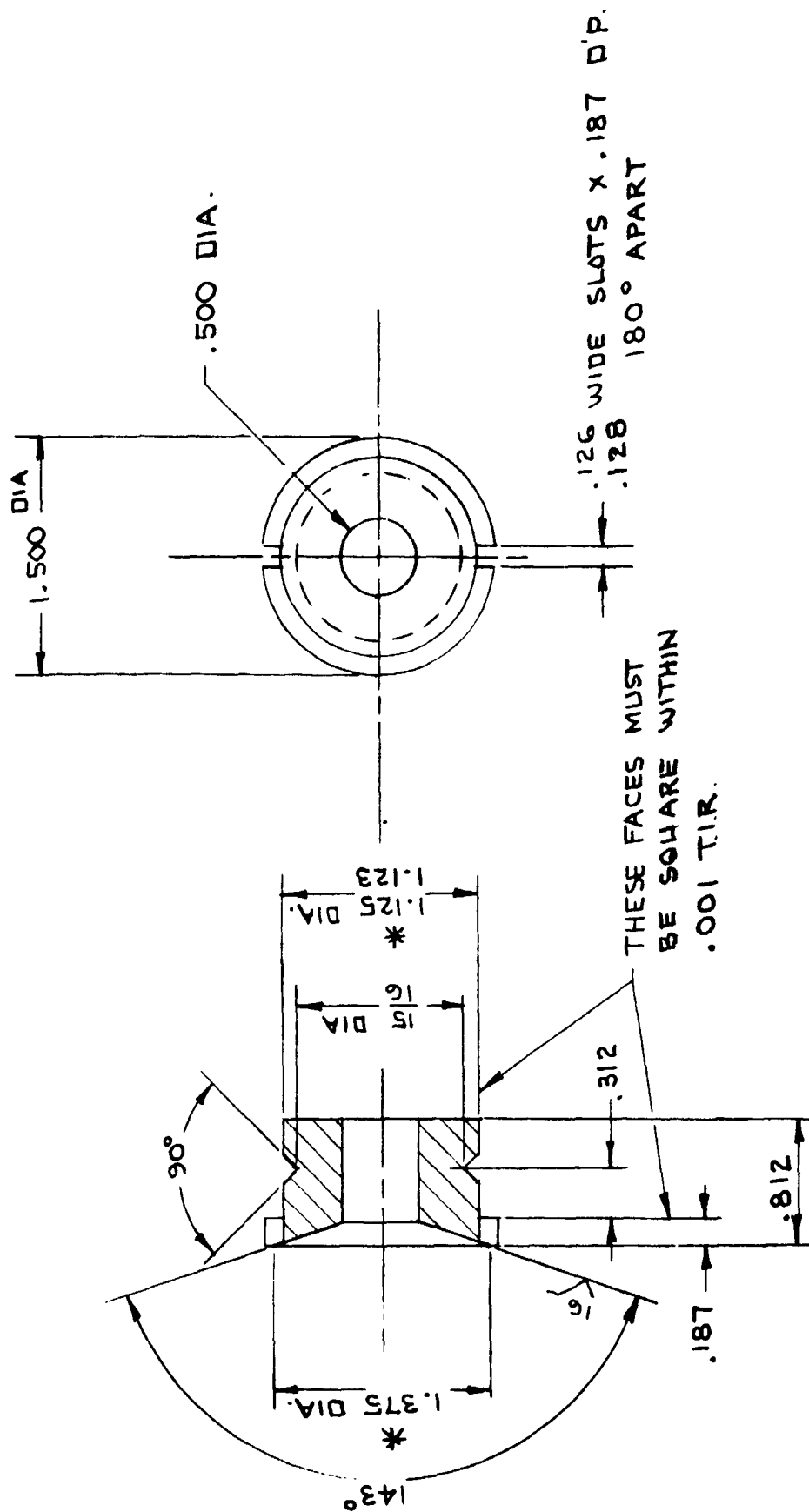FIG. 10 SPHERICAL SPECIMEN

X - X

THESE FACES MUST BE
SQUARE WITHIN .001 T.I.R.

90°

* 1.19 R
SPHERICAL

1.500 DIA.

1.123
1.125 DIA. *

.15 DIA.
.16

.312

.410

1.03

.500 DIA.

.126 WIDE SLOTS x .190 DP.
.128   180° APART

FIG. 11 CONICAL SPECIMEN

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scrubbed in distilled water and dried in argon. Immediately after cleaning, the specimens were stored in sealed desiccators.

5.2 Apparatus Preparation

The vacuum system and experimental apparatus was given a preliminary degassing prior to installation of the specimens. Following cooldown, the system pressure dropped below 3×10^{-10} Torr, the X-ray limit of the Nottingham type gage used in this system. The electron gun used to measure the specimen surface contact potential was degassed with the cathode at $\sim 2500^{\circ}$C and the anode at $\sim 650^{\circ}$C. The ion gun and gas supply plumbing were degassed with a thermal blanket at 350°C. The vacuum system was backfilled with argon. A specimen set was removed from its desiccator and installed in the specimen mounting fixture. The specimen was electrically insulated from the mount by a sheet of mica and secured in the mounting fixture by three sapphire spheres. The measured leakage resistance (in air) between the specimen and mounting fixture was in excess of 10^{14} ohms. The mounting fixture and specimen were installed in the apparatus which was immediately closed and pumped down. Conventional clean room techniques were used in all manual operations on the specimen, insulation, leads, and mounting fixture. Specimen installation and removal was performed through the ion gun mounting port which was equipped with a copper shear seal.

After pumpdown, the specimen, vacuum system, and auxiliary apparatus were degassed. During this degassing, the system shell was maintained between 350 and 550°C. The internal wire brush drive motor was maintained at $\sim 100^{\circ}$C and certain main assembly flanges which were sealed with neoprene "O"-rings were maintained at $\sim 90^{\circ}$C. During degassing, the main pumping system heaters were off and the pumps were cooled, the system was evacuated by the backing pumps which were also diffusion pumps. The cold trap isolating the main pumping system from the vacuum system (immediately above the diffusion pump) was maintained at 77°K and all other cold traps were degassed along with the system. Near the end of the degassing interval the main pumps were turned on. After the pumps were operating, the degassing heaters were turned off and all rubber O-rings were rapidly cooled to $\sim 230^{\circ}$K. This procedure assured that desorption of organics from the rubber was reduced to a negligible level and that the system and internal apparatus remained sufficiently hot to prevent adsorption of any

degassing products that may have desorbed from the rubber during
O-ring cooldown. At no time during subsequent experimental opera-
tions was the rubber temperature allowed to rise above $0^{\circ}C$.

The electron gun cathode temperature was maintained at
$\sim 2500^{\circ}C$ for an interval of time ranging between 1 and 5 hours
after turning the system degassing heater off, depending on its
previous history. The electron gun anode structure was simul-
taneously degassed by radiation heating and electron bombardment
at a temperature of $\sim 650^{\circ}C$. The criteria applied in determining
if the electron gun had been sufficiently degassed was that no
system pressure rise should be observable upon abruptly raising
the cathode temperature from room temperature to operating tempera-
ture ($\sim 1750^{\circ}C$).

Shortly after starting cooldown, but while the specimen re-
mained hot, the ion gun was operated on a low gas flow rate and
with zero extraction voltage to further clean the gun electrodes
by ion bombardment. Even after prolonged ion gun degassing at
$\sim 350^{\circ}C$ this procedure generally released substantial quantities
of gas as determined by the system pressure gage. The fact that
this gas load eventually disappeared even though the gas supply
pressure to the ion gun remained constant was taken as evidence
that the ion gun was sufficiently degassed for experimental opera-
tions. After this procedure the ion gun could be turned on with-
out the release of a gas burst.

5.3 Contact Potential Difference Measurement

The following preliminary experiments were performed with
the electron gun and a tungsten specimen to assist in deciding on
certain operating parameters, to determine repeatability of the
contact potential difference measurement and to determine if
operation of the electron gun produced any change in observed con-
tact potential difference.

During the initial measurements of collector current as a
function of retarding potential it was found that at relatively
large retarding voltages (low collector currents) the leakage in
the "floating" electrometer was sufficiently high to introduce
non-negligible errors. Therefore, specimen current vs. retarding
potential data were not used below currents of approximately
10^{-10} amps.

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Electron gun target (tungsten specimen) current was measured as a function of retarding potential. Curves were taken for various values of electron gun anode voltage and for various electron gun cathode temperatures to determine that temperature and accelerating voltage which gave the closest approach to the ideal curve. (See Ref. 5.) It was found that successive machine records of collector current vs. retarding potential could be plotted on top of each other (within line width) over a considerable range of voltage and cathode temperature. However, to achieve a more directly interpretable curve shape most of the data was taken with a cathode temperature of $\sim$ 1750°K and an accelerating potential of 90 volts.

A series of experiments were performed to electrically locate the emitting spot on the cathode at ground potential, that is the spot from which the electrons were emitted that passed through the anode aperture and finally reached the target. However, since a strong magnetic field was generated at the surface of the cathode ribbon by the cathode heating current (6 to 8 amp) and since the cathode power leads had unequal hot resistance, it was found impractical to operate the emitting spot of the cathode at ground potential. This precludes the possibility of reducing the contact potential difference measurements to absolute work function determinations of the specimen. Because of this and because the geometry chosen (to satisfy other requirements) was not well suited to make absolute measurements, all measurements made were relative to the tungsten cathode work function, and with an arbitrary electrical reference "zero." The electrical reference was fixed during each series of contact potential difference measurement for each specimen. However, the location of the electrical reference with respect to the tungsten work function was not determined.

With the electron gun cathode clean, a series of measurements were performed on the contaminated tungsten specimen surface to determine if operation of the electron gun produced any change in the measured contact potential difference between the contaminated specimen surface and the clean reference cathode. The specimen position in front of the electron gun was fixed and the collector current was recorded as a function of retarding potential at intervals of $\sim$ 500 sec. This procedure was continued for about 1.5 hr. during which time the system pressure was less than 3 x 10^{-10} Torr. Comparison of the machine records by direct overlay showed no consistent variation in collector current vs. retarding potential which was greater than the line

width of the plotter trace.

A series of measurements were made on the contaminated tungsten specimen surface at various system pressures to determine if the system pressure caused any change in the measured contact potential difference. The system pressure was raised gradually by lowering the liquid nitrogen level in one of the cold traps. Collector current vs. retarding potential curves were taken successively as the system pressure increased. It was found by direct comparison of the machine records that no change occurred in the curves of collector current vs. retarding potential for slight increases in pressure. The actual magnitude of the pressure increase was unknown since the ion gage continued to indicate its X-ray limit. Continuing this procedure for nearly an hour eventually produced small but systematic an' consistent differences in the plots of collector current vs. retarding potential. The actual magnitude of the pressure at which changes began to appear in the collector current curves was not determined since the system pressure had not yet increased above the X-ray limit of the ion gage. The collector current vs. retarding potential curve obtained immediately after refilling the liquid nitrogen trap was identical with the curve obtained prior to raising the pressure.

These measurements were repeated several times, during some of which the system pressure was allowed to rise above the gage X-ray limit. The results were substantially the same: the collector current vs. retarding potential curves obtained at low pressures (system limit) were identical but curves obtained at higher pressures (10^{-9} Torr) showed substantial deviations from the low pressure curves.

During one of these controlled pressure excursions the electron gun cathode was cold. The collector current curve obtained immediately following refilling the liquid nitrogen trap showed a small but unambiguous deviation from the previous low pressure curves. The electron gun cathode was flashed to degassing temperature for a few seconds and returned to normal operating temperature. The collector current vs. retarding potential curve obtained following this treatment coincided with those previously obtained at low pressure.

The following conclusions, applicable to subsequent measurements, were drawn from the results of the above preliminary experiments:

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1) Operation of the electron gun does not alter the work function of the contaminated specimen surface.

2) The work function of the contaminated specimen surface is not altered by adsorption or desorption of gases if the pressure is near the system limit (somewhat less than 3×10^{-10} Torr).

3) During any series of measurements it is preferable to maintain the electron gun cathode at or near normal operating temperature to minimize gas adsorption on the cathode surface. If other requirements make it necessary to turn the electron gun off while the pressure is elevated, it should be flashed to degassing temperature for a short period to remove adsorbed gasses prior to making contact potential difference measurements.

4) At room temperature and at sufficiently low pressure, the work function of the contaminated surface is independent of time, implying that the surface contamination relative coverage is stable.

It will be seen from the results of experiments performed subsequently that most of these conclusions also apply to clean surfaces.

5.4 Ion Bombardment

After selection of a contaminated spot on the specimen and measurement of its contact potential difference, the specimen was rotated such that the center of the spot coincided with the ion gun axis. A prescribed ion bombardment dose was then applied to the area of the specimen under study by controlling the ion gun anode and extraction voltages, the gas supply pressure, and the duration of bombardment. The average target (specimen) current and interval of bombardment were recorded.

Following each ion bombardment dose, the specimen was returned to its original position such that the center of the specimen surface selected for study was again coincident with the electron gun axis. The contact potential difference was then measured and the difference between this result and that obtained previously was interpreted as the change in specimen work function caused by the preceding bombardment.

6. RESULTS

6.1 Tungsten Specimens

A series of ion bombardment experiments were performed to determine the variation in surface work function (contact potential difference) as a function of ion bombardment dose. Dose is used here to denote the time-integral over the bombardment period of the ion current density onto the target. The test surface on the tungsten target was turned away from the ion gun and the gun was turned on at a low gas flow rate to degass the gun electrodes by self-bombardment.

The specimen test surface was positioned in the ion beam and given successive doses of about 1 to 2 x 10^{14} Ar^+/cm^2 until a total dose of approximately 10^{15} Ar^+/cm^2 had been accumulated. Following each dose the test surface was positioned in the electron beam and the specimen current as a function of retarding potential was measured. The results of this series of experiments are shown in Fig. 12. It may be observed from these data that the contact potential difference between the tungsten specimen surface and the clean electron gun tungsten cathode decreased as the ion dose increased until the total dose reached about 5.5 x 10^{14} Ar^+/cm^2, and that increasing the total dose to approximately 10^{15} Ar^+/cm^2 produces no further change in the specimen contact potential. Since the contact potential between the two tungsten surfaces is a function only of their respective work functions and since the previous experiments imply that the cathode work function remains stable under operating conditions, the above results were interpreted to imply that the specimen work function decreased by 0.75eV as the result of an ion bombardment dose of 5.5 x 10^{14} Ar^+/cm^2. The fact that no further change in the specimen work function was observed, out to a total dose of about 10^{15} Ar^+/cm^2, was interpreted to imply that bombardment by 5.5 x 10^{14} Ar^+/cm^2 had removed all contaminants from the specimen surface. This is further supported by the relatively steep slope of the curve in Fig. 12 for small doses, indicating that removal of surface contamination is a rapid process under ion bombardment.

The magnitude of the observed change in the tungsten specimen work function agrees quantitatively with that observed by others[9] resulting from the removal of a monolayer of oxygen. If the specimen surface were covered with a monolayer of oxygen
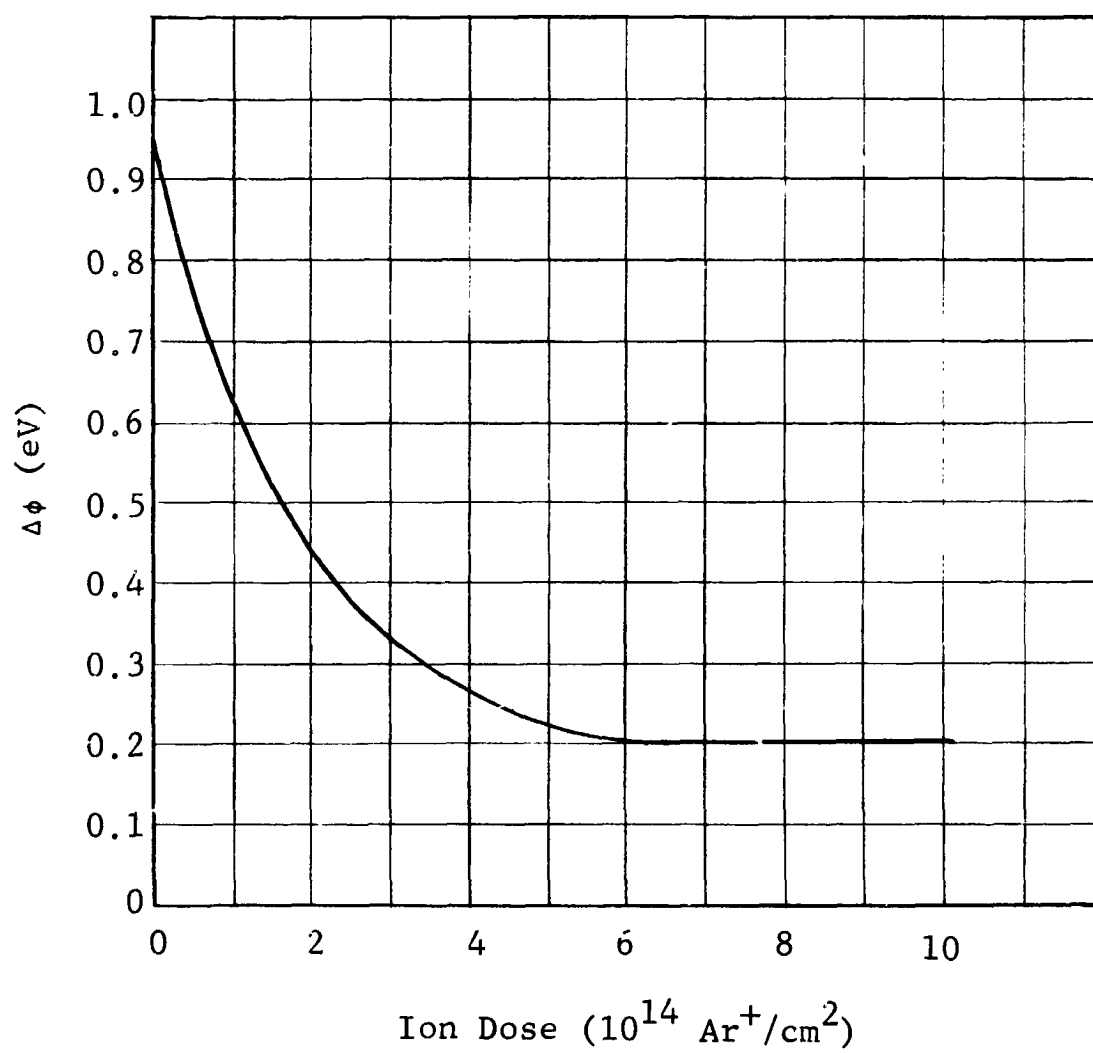
FIG. 12   CHANGE IN TUNGSTEN WORK FUNCTION (SPECIMEN 1)

(which is probable since the specimen degassing treatment was rather mild), the data of Fig. 12 imply that the average sputtering coefficient of Ar^+ with mean energy $\sim$ 750 eV on an oxygen covered tungsten surface is nearly 1. This is about the value of sputtering coefficient expected[14] for Ar^+ on bare tungsten.

The test surface of the tungsten specimen remained in the electron beam for about 1.5 hr. after which the contact potential difference was again measured. The value obtained coincided with the end point work function shown in Fig. 12. This clearly indicated that operating the electron gun (after proper processing, as described previously) did not recontaminate the test surface at a detectable rate. The agreement between the work function measured immediately after ion bombardment and that measured $\sim$ 1.5 hr. later demonstrated that the clean specimen surface could be maintained clean for relatively long periods of time if the background pressure were sufficiently low. The actual system background pressure during these experiments was unknown; however, it was at all times less than 3 x 10^{-10} Torr. Even during the intervals of ion bombardment, during which the argon partial pressure reached approximately 10^{-7} Torr, the partial pressure of the total impurities introduced with the argon corresponded to a pressure of $\sim 10^{-12}$ Torr.

In Fig. 13 are plotted the curves of target current (i_t) vs. retarding potential before ion bombardment and after ion bombardment with a total dose of 10.15 x 10^{14} Ar^+/cm^2. The shape of these curves are typical of the retarding potential curves obtained throughout these experiments. (The electron gun cathode temperature for the upper curve was slightly higher ($\sim 35^o$) than that for the lower curve. However, the work function of the cathode is sufficiently insensitive to temperature that the change in reference work function may be neglected.)

A new tungsten specimen and a new electron gun were installed in the apparatus. The entire procedure up to the point of measuring the contact potential difference between the specimen surface and the electron gun tungsten cathode was substantially the same as for the previous specimen with the exception that the electron gun was not degassed at high temperature. This change in procedure was necessary to permit the measurement of the contact potential difference between a contaminated reference surface (tungsten cathode) and the contaminated specimen surface and to experimentally determine the change in contact potential
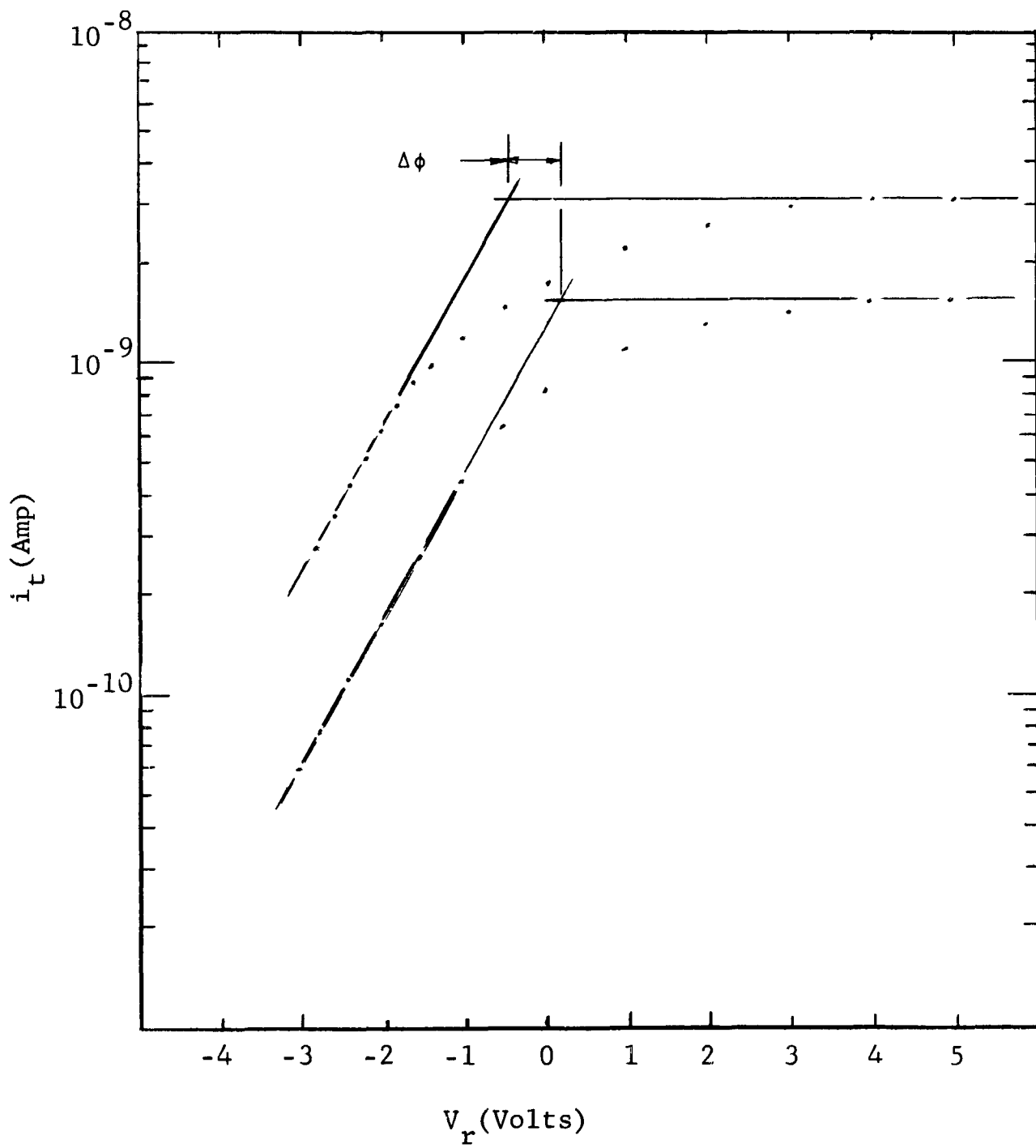
FIG. 13 SPECIMEN CURRENT VS. SPECIMEN POTENTIAL

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difference produced by removing the reference surface contamination by high temperature degassing.

The first collector current vs. retarding potential curve gave the contact potential difference between the two contaminated tungsten surfaces shown in Fig. 14. During this measurement the system pressure was approximately 5×10^{-8} Torr. This pressure rise resulted from operation of the undegassed electron gun. The contact potential difference was not a stable function of time since even at operating temperature the cathode cleanup rate was substantial.

The electron gun was then thoroughly degassed following the procedure previously described. After cooldown of the electron gun, the contact potential difference was again measured. As experienced with the previous specimen, this value was stable and repeatable. The 1.2 eV increase in contact potential difference (see Fig. 14) resulting from cleaning the reference surface, which was interpreted to imply a 1.2 eV decrease in the work function of the cathode, is not inconsistent with the maximum value expected for an oxygen contamination tungsten surface. [15]

The pressure rise which accompanied the operation of the undegassed electron gun of course contaminated the ion gage. Although the ion gage was degassed during and following the electron gun degassing, an auxiliary cathode (filament) in the ion gage was maintained cool. This was done to trap a small quantity of gas, known to be adsorbable on tungsten, which could be released into the system when desired.

The reversibility of the observed change in the work function of the reference surface was then examined by turning the electron gun cathode off and flashing the ion gage auxiliary cathode to approximately 2300°K such that the adsorbed gas was desorbed in a short pulse, a fraction of which was readsorbed on the electron gun cathode (reference surface). The contact potential difference was immediately remeasured. It was found that the contact potential difference had decreased by 0.1 eV. (See Fig. 14.)

The desorption pulse produced a system pressure pulse, the amplitude of which was approximately 10^{-6} Torr. The vacuum system time constant was approximately 0.5 sec. The contamination relative coverage on the reference surface after decay of
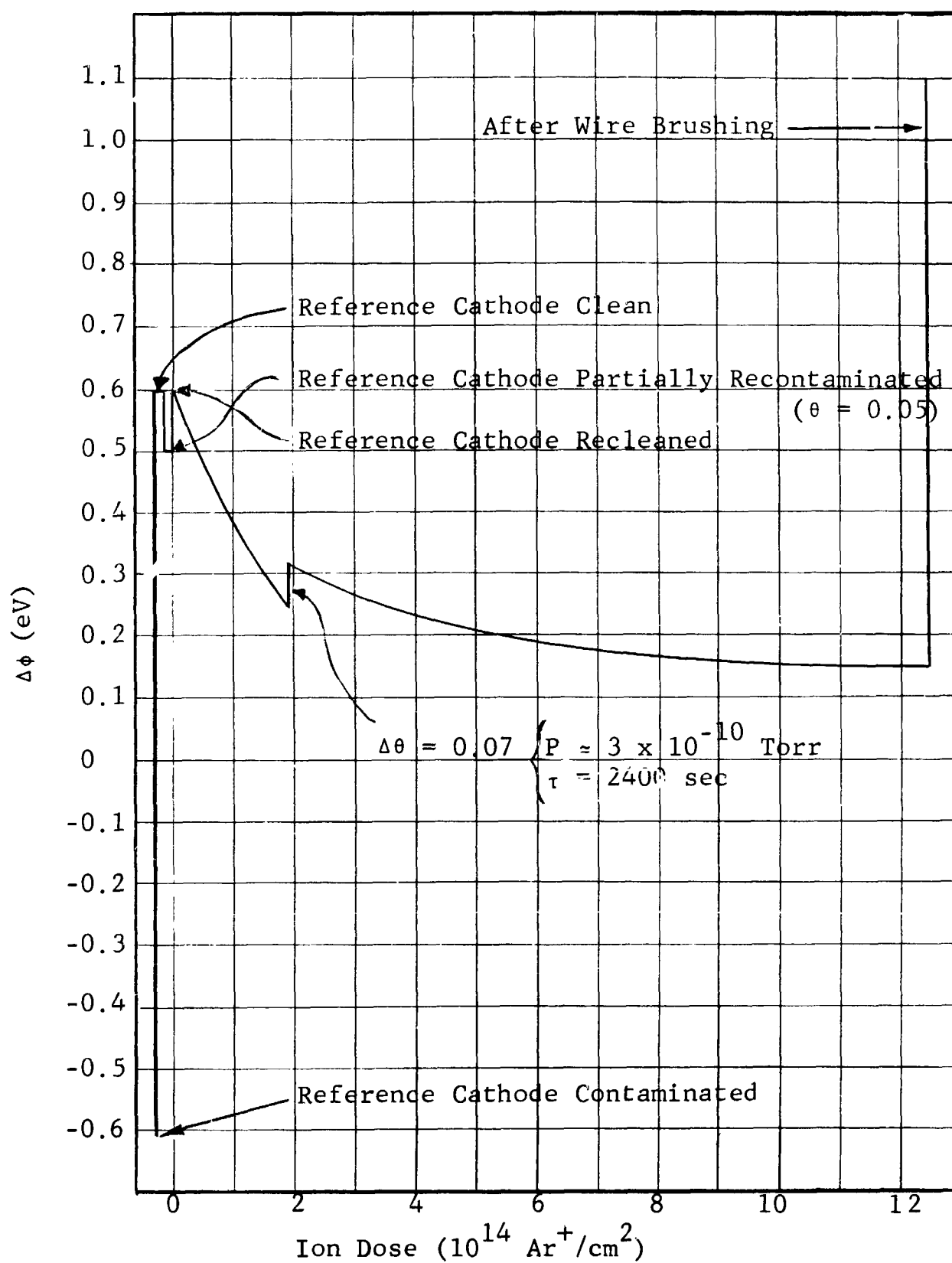
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FIG. 14   CHANGE IN TUNGSTEN WORK FUNCTION (SPECIMEN 2)

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the pressure pulse may be calculated (approximately) from

$$\theta = \frac{\alpha}{\sigma_o (2\pi mkT)^{1/2}} \int_o^\infty P(t)dt$$

$$= \frac{\alpha P_o \tau_s}{\sigma_o (2\pi mkT)^{1/2}} \; ,$$

where

α $\equiv$ capture probability
≈ 0.14 for O_2 on cl. . W (from Ref. 16),

σ_o $\equiv$ no. of adsorption sites/cm^2
$\approx 5 \times 10^{14}$ cm^{-2},

m $\equiv$ molecular mass
$m_{O_2} = 5.34 \times 10^{-23}$ gm,

T $\equiv$ gas temperature (in system)
$\approx 300^o K$,

k $\equiv$ Boltzmann constant
$= 1.38 \times 10^{-16}$ erg/$^o K$,

P_o $\equiv$ pressure pulse amplitude
$\approx 10^{-6}$ Torr $= 1.3 \times 10^{-3}$ μb,

τ_s $\equiv$ system time constant
≈ 0.5 sec.

Substituting these numerical values in the above equation gives

$$\theta \approx 0.05 \, .$$

This impl es that the adsorption of $\sim 1/20$ of a monolayer of contamination increased the reference surface work function by about 0.1 eV. The electron gun cathode was then flashed to $\sim 2500^{\circ}K$ and upon cooldown, the contact potential difference was again measured. The value obtained coincided with that previously obtained for a clean cathode. (See Fig. 14.)

A similar experiment was performed on the tungsten specimen surface but differed substantially in that argon ion bombardment was used to clean the specimen instead of high temperature degassing. The specimen was bombarded with an ion dose of approximately 2×10^{14} Ar^{+}/cm^{2} and the contact potential difference measured. It was found that the specimen work function had decreased by approximately 0.35 eV. (See Fig. 14.)

After 2400 seconds the contact potential difference was again measured. The result implied that the specimen work function had increased by approximately 0.07 eV in 2400 sec. During this interval the electron gun cathode was maintained at operating temperature and the system background pressure was somewhat less than 3×10^{-10} Torr. Thus, the partial pressure of all contam -nants in the system background spectrum could not have been greater than 3×10^{-10} Torr. Therefore, the maximum change in relative coverage that could have occurred is given by

$$\Delta \theta = \frac{\alpha \; P_{c} \; \tau}{\sigma_{o} (2\pi mkT)^{1/2}}$$

where

$P_{c} \equiv$ contaminant partial pressure
$= 3 \times 10^{-10}$ Torr $= 3.9 \times 10^{-7} \mu b$,

$\tau \equiv$ time between measurements of ϕ
$= 2400$ sec,

and the other parameters are as defined previously. Substitution of these numerical values gives, for the change in contaminant relative coverage,

$$\Delta \theta = 0.07.$$

That is, increasing the surface contamination relative coverage by 0.07 produced an increase in the specimen work function of 0.07 eV. This is in fair agreement with the results obtained on the reference surface if consideration is given to the fact that the data for the reference surface is clean surface data ($\theta = 0$), whereas the data for the specimen surface is for a moderately contaminated surface.

The specimen surface was recleaned by ion bombardment with an additional dose of approximately 10^{15} Ar^+/cm^2 and the contact potential difference again measured with the result as shown in Fig. 14. From the data on the previous specimen it is concluded that this bombardment dose was more than sufficient to produce a clean surface.

It may be expected that the measured contact potential difference should eventually return to its original value since both the reference surface and the specimen surface were contaminated in the beginning and both were clean in the end. However, since the history of the ribbon material was quite different from the specimen material there is no assurance that the contaminating species on the two surfaces were initially the same. It is improbable that the crystal planes exposed on the two surfaces were the same.[13] Further, there is evidence[17] that the ion bombardment produces considerable damage in the surface in the form of vacancies and dislocations. Also, there is evidence[18][19] that a certain fraction of the bombarding ions are buried as interstitial atoms. These facts would tend to allow for a different end point work function of two clean surfaces, one cleaned thermally and the other by ion bombardment. This view appears to conflict with some data obtained using the Auger electron ejection technique[20] where the ejected electron energy distribution is not substantially changed and the deduced work function is unchanged after ion bombardment. This view is, however, supported by some results of a low energy electron diffraction experiment[17] where ion bombardment did sufficient damage to extinguish the surface lattice pattern.

Immediately following the specimen final cleaning by ion bombardment and contact potential difference measurement, the specimen surface was wire brushed for 200 sec. The contact potential difference was again measured. The result indicated that the specimen work function had increased by 0.95 eV (since the reference surface remained hot and clean). Further wire

brushing produced no further change in the measured contact potential difference. The specimen was subsequently examined under X-ray florescence. It was found that the surface layer contained large quantities of iron, nickel, and chromium which were deposited on the surface by the stainless steel wire brush. However, these elements would not account for the large increase in specimen work function. It is therefore presumed that the specimen surface was contaminated with oxygen transferred from the stainless steel wire bristles.

A third tungsten specimen was installed in the apparatus and the procedure followed was the same as that for the first specimen except that the gas used in the ion gun was xenon. This was done to take advantage of the slightly higher sputtering rate expected[14] for 750 eV Xe^+ as compared with 750 eV Ar^+ and to reduce the ion burying probability in the target.[19]

After measuring the initial contact potential difference and cleaning the ion gun by self-bombardment, the specimen was bombarded with a dose of 15 x 10^{14} Xe^+/cm^2. It is estimated that this dose is sufficient to remove about 7 layers of tungsten atoms from the target. The surface was subsequently bombarded a number of times such that each dose was approximately twice the preceding dose until the total integrated dose had reached about 560 x 10^{14} Xe^+/cm^2. It is estimated that approximately 250 atom layers were removed by this total dose. The contact potential difference was measured after each dose and the results are shown in Fig. 15. Since the endpoint work function was achieved sometime during the first dose, these results indicate that ion bombardment by a dose sufficient to remove a few atom layers is also sufficient to remove the surface contamination remaining on a surface after the application of conventional cleaning techniques.

After $\sim 10^3$ sec, the contact potential difference was re-measured and it was found that the specimen work function had decreased by about 0.2 eV. This did not occur with Ar^+ bombardment of the first specimen. This result may indicate that considerably greater surface damage results from Xe^+ bombardment than for Ar^+ bombardment and that the change in work function is a result of room temperature annealing of the heavy damage. No further measurements were made since shortly after the above measurement, the vacuum system developed a leak which was sufficiently large to recontaminate the surface.
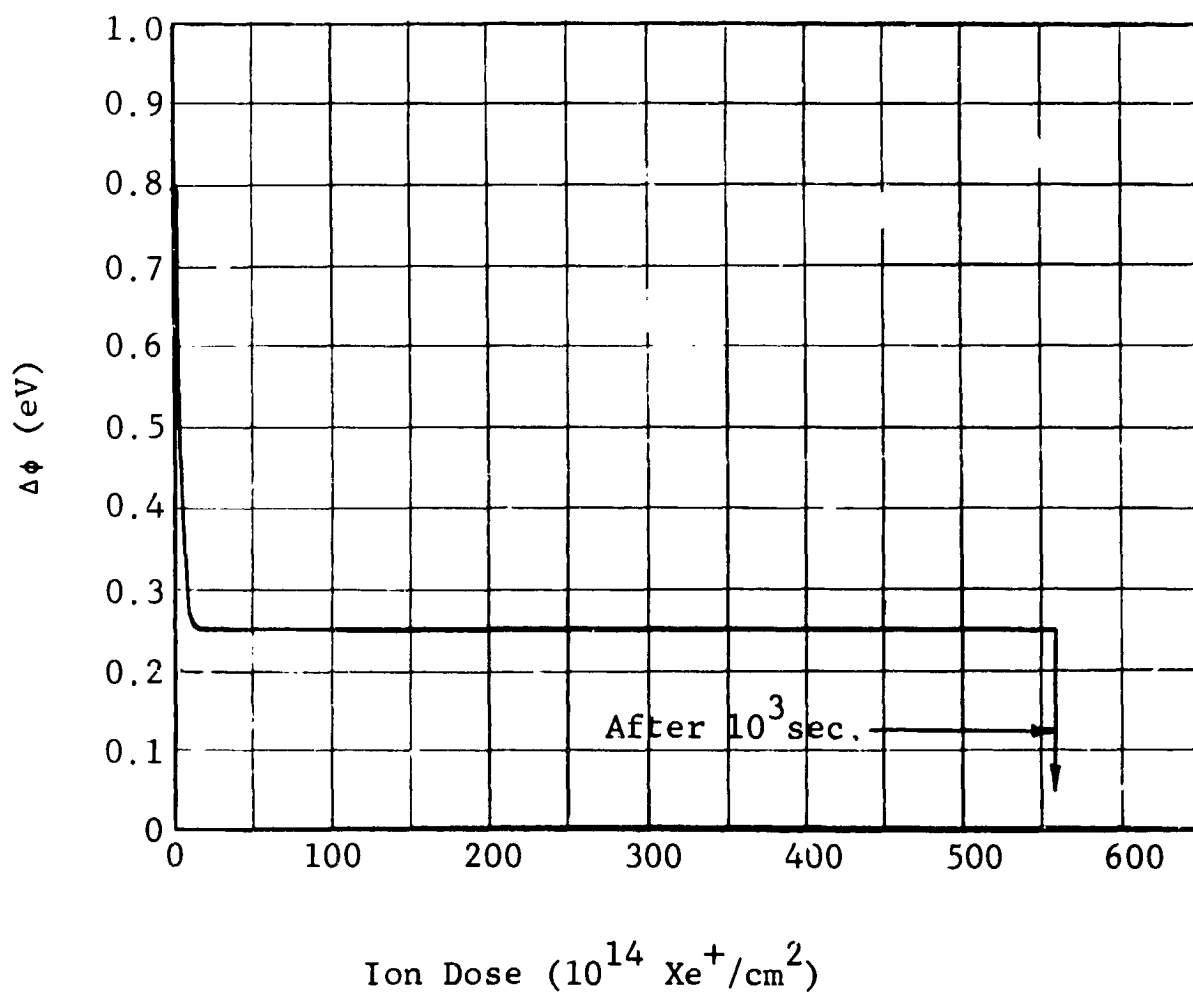
FIG. 15   CHANGE IN TUNGSTEN WORK FUNCTION (SPECIMEN 3)

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6.2 Stainless Steel Specimens

A stainless steel specimen was installed in the apparatus and the procedure followed was the same as that used for the 3rd tungsten specimen except that the Xe^+ bombardment doses were smaller. The stainless steel specimen work function reached an asymptotic value at a total bombardment dose of approximately 25×10^{14} Xe^+/cm^2 as shown in Fig. 16.

After the above ion bombardment the specimen remained in the system for 15 hours during which the system background pressure was somewhat less than 3×10^{-10} Torr. At the end of this interval the contact potential difference was again measured. It was found that the specimen work function had increased by 0.85 eV as shown in Fig. 16. It was estimated that the partial pressure of contaminants was not greater than 10^{-10} Torr. The increase in surface relative coverage during this interval (estimated by the formula given previously) was approximately $\theta = 0.53$. Thus, the stainless steel work function increased by about 0.85 eV as a result of adsorbing about 1/2 monolayer of contamination on the surface.

The specimen surface was recleaned by ion bombardment with a dose of approximately 21×10^{14} Xe^+/cm^2. The specimen work function returned to within 0.05 eV of its previous clean value as shown in Fig. 16. This slight difference between successive clean surface work functions may result from making the measurements on adjacent but slightly displaced spots on the surface of the stainless steel specimen.

Following the above ion bombardment the surface of the specimen was wire brushed for 200 sec. The contact potential difference measured immediately after wire brushing implied that the specimen work function had increased by 1.3 eV. This value is within 0.1 Ev of the contaminated surface work function. As with the tungsten specimen, it is surmised that this contamination was transferred from the brush to the specimen surface. Wire brushing for an additional 200 sec produced no further change in the specimen work function.

Comparing the above results for Xe^+ bombardment of tungsten and stainless steel, it appears that a somewhat larger ion bombardment dose is required to clean a stainless steel surface than is required to clean a tungsten surface. (See Fig's 15 and 16.) It also appears that a tungsten surface reaches its

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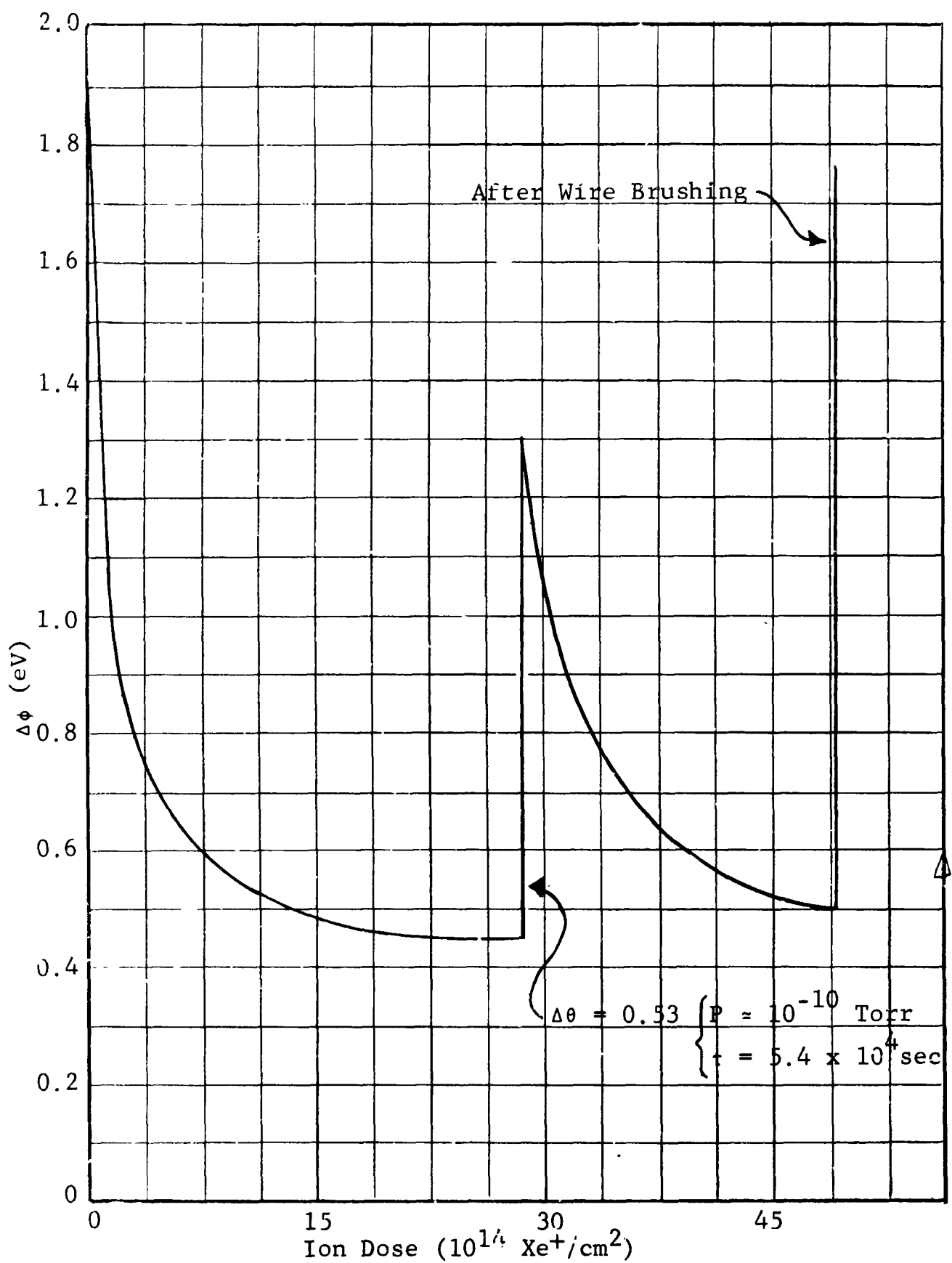
After Wire Brushing

$\Delta\theta = 0.53$ $\begin{cases} P \approx 10^{-10} \text{ Torr} \\ t = 5.4 \times 10^4 \text{sec} \end{cases}$

$\Delta\phi$ (eV)

Ion Dose (10^{14} Xe$^+$/cm^2)

FIG. 16   CHANGE IN STAINLESS STEEL WORK FUNCTION

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end point work function at a much lower argon bombardment dose than the xenon bombardment dose required to produce the end point work function of a stainless steel surface. (See Fig's 12 and 16.)

To investigate the possibility that the end point work function may depend on the energy of the bombarding ions, a 2nd stainless steel surface was bombarded with a single xenon dose of approximately 55×10^{14} Xe^+/cm^2. The average ion energy was approximately 1500 eV or twice the ion energy normally used. The resulting work function measurement is shown as a single point plotted on Fig. 16. There is a difference of 0.15 eV between this value and the lowest value measured for the work function on the previous stainless steel specimen. It is not known how much of this difference is due to ion energy difference and how much is due to real differences in the polycrystalline stainless steel alloy surface.

Following the above bombardment, a series of measurements were made to determine the effect of the ion angle of incidence on cleaning. This was done by placing a portion of the specimen known to be contaminated in front of the electron gun and increasing the retarding potential until the current reaching the specimen disappeared into the background noise. The retarding potential was maintained constant and the previously cleaned portion of the specimen was brought under the electron gun. The current reaching the specimen was then measured. The specimen was then moved through a series of small increments until the contaminated portion of the specimen was again under the electron gun. The current reaching the target was measured at each position.

The results are shown in Fig. 17. The minimum angle of incidence (angle between the ion trajectory and the surface normal) was 45° and the maximum angle of incidence was 90° (grazing incidence). The resulting curve (ordinate) is proportional to the cleanliness of the surface. (There exists, however, a real but unknown variation in the work function due to the polycrystalline nature of the specimen surface.) Nonetheless, it may be observed from Fig. 17 that there is a general decrease in target current implying an increasing work function and increasing level of contamination as the ion angle of incidence increases (approaching grazing incidence).
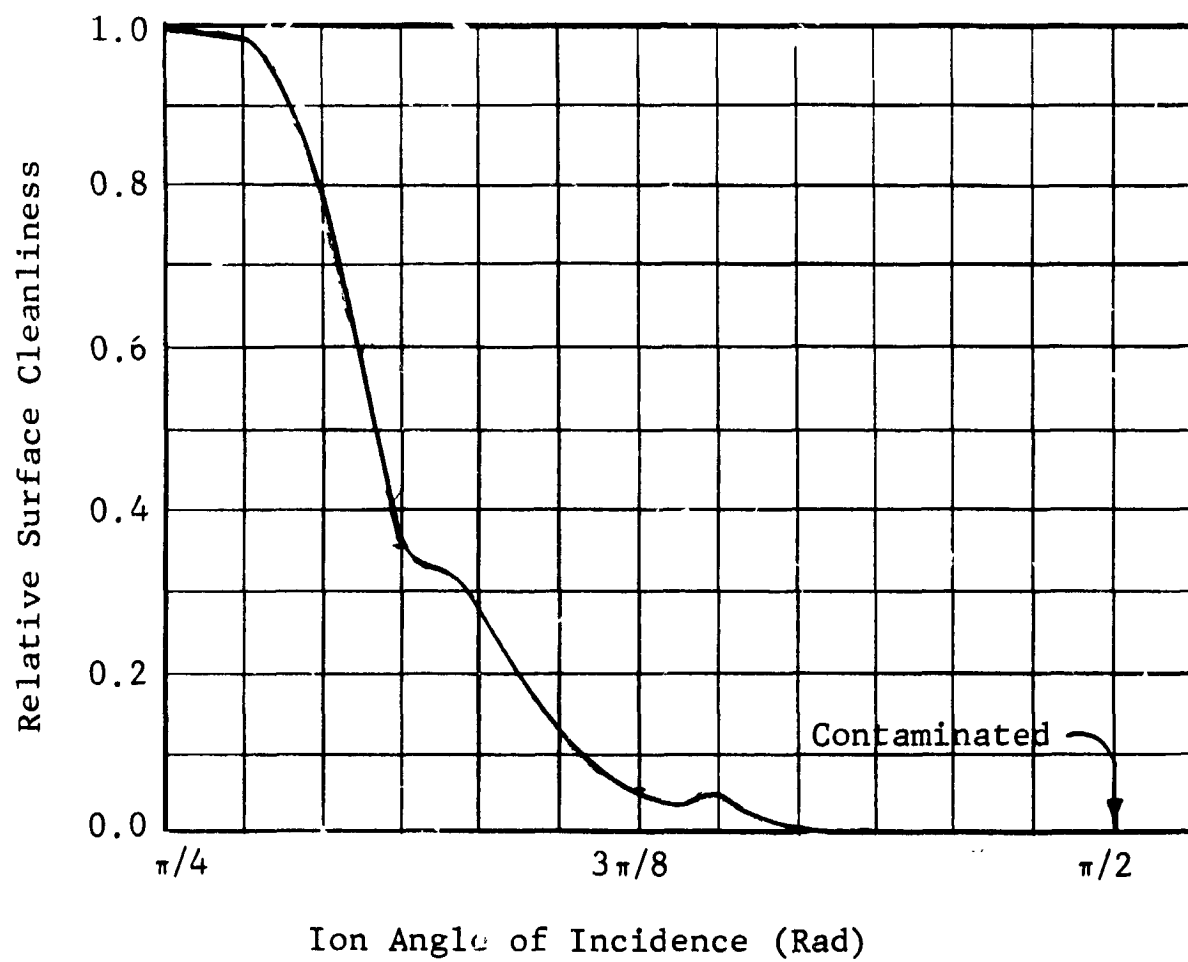
FIG. 17   SURFACE CLEANLINESS DEPENDANCE
ON ION INCIDENCE ANGLE

6.3 Copper Specimens

Experiments were conducted on several copper specimens using both argon ion bombardment and wire brushing. The procedures used were generally the same as those described previously for specimens of other materials. However, during most of these experiments the system background pressure was erratic, making it impossible to maintain a constant surface coverage for relatively long periods of time. Thus, only those contact potential difference measurements taken immediately after ion bombardment or wire brushing have directly interpretable meaning. Consequently, it was not possible to obtain a complete definition of the dependence of work function on ion bombardment dose. However, it was possible to obtain some meaningful and useful isolated data points for copper.

The contact potential difference measured on one copper specimen before and after an ion bombardment dose of 12.5×10^{14} Ar^+/cm^2 indicated a reduction in work function of 2.0 eV.

Another copper specimen was bombarded with a dose 118×10^{14} Ar^+/cm^2 and the observed reduction in work function was 1.65 eV. The same surface was subsequently bombarded with an additional dose of 175×10^{14} Ar^+/cm^2 but the measured contact potential difference indicated no further change in work function.

A third copper specimen was wire brushed for 200 seconds. The measured contact potential difference before and after brushing indicated that the brushing had reduced the work function by 0.45 eV. The specimen was wire brushed for an additional 100 seconds but no further change in work function was observed. The same specimen was subsequently ion bombarded but again no further change in work function was observed.

It is considered significant that of the three metals tested, copper is the only one that showed a decrease in work function following wire brushing. (The other two showed an increase.) This is interpreted to imply that only the copper was sufficiently soft (relative to the stainless steel brush) that the contaminated surface was abraded off the specimen leaving behind a new, clean surface. This interpretation is supported by the result that subsequent ion bombardment produced no additional work function reduction. It is also supported by subsequent microscopic studies of the specimen in which it was observed that the surface was highly abraded and extremely rough and that it was partly covered

with small copper particles, only lightly adherent to the parent metal. (See Fig. 18)

The fact that the change in work function was not the same for all copper specimens does not necessarily imply that some of the surfaces remained partly contaminated after ion bombardment or wire brushing. It is considered highly probable that each was clean. The difference in the change in work function observed on different specimens may be due to different levels of contamination at the beginning of the experiments on each specimen. This is especially probable for copper since its oxidation rate is so temperature sensitive. That is, even though each specimen received approximately the same preliminary preparation, it is quite possible that the surface contamination level could be different at the time of making the reference contact potential difference measurements, especially when it is recalled that the system background pressure was unstable during this series of experiments.


7.   CONCLUSIONS

From the above results the following conclusions may be drawn:

1.   It is possible, in a practical vacuum system and using existing UHV techniques, to maintain surface relative coverage fixed for intervals of time well in excess of that required to measure the surface properties. This conclusion applies even to an atomically clean surface.

2.   Wire brushing does not necessarily produce a clean surface and this technique of cleaning should be applied only after giving attention to the brush cleanliness, surface material, and brush material.

3.   Atomically clean surfaces may be produced at normal temperatures with relative ease by ion bombarding surfaces from which the gross debris has been removed by conventional techniques. They may also be produced by thermal degassing (melting or sublimation temperature permitting).

4.   Surface properties may be studied using electron beam techniques without altering the surface properties during measurement, provided only that the electron gun is given an adequate
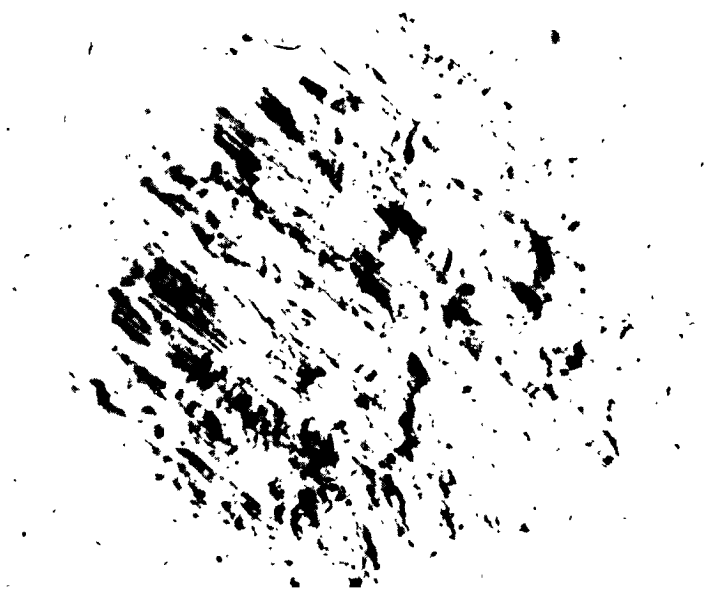
(30x)
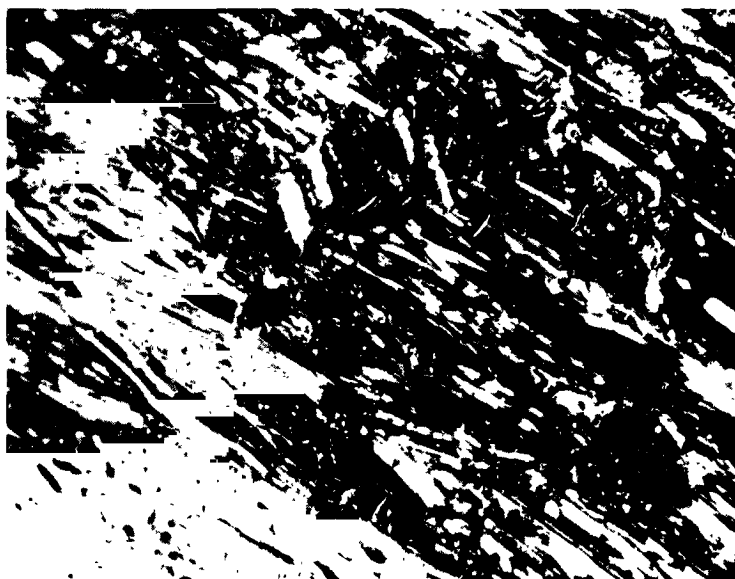
(65x)

FIG. 18   ABRADED COPPER SURFACE

preliminary thermal processing. This of course greatly restricts
the materials from which the gun may be fabricated.

5. Surface cleanliness is related to the work function of
the surface. The work function of a surface depends nonlinearly
on the relative coverage and on the contaminating species. The
relative coverage may be measured quantitatively by measuring
the surface work function.

6. The change in work function, resulting from a change in
relative coverage from one to nearly zero, may be somewhat
larger than one electron volt for certain contaminating species.

7. Surface contamination relative coverages as low as a
few percent may be measured with ease using the retarding field
diode technique and it appears possible to measure relative
coverages of less than a percent with this technique.

8. RECOMMENDATIONS

The following recommendations are made with respect to the
apparatus:

1. Further measurements of work function should be made on
an absolute basis. This necessarily introduces geometrical
restrictions (for example plane, parallel geometry) in the
specimen configuration. It is considered that these restrictions
are not serious constraints with respect to adhesion experiments
but they will substantially enhance the value of surface study
results.

2. It is recommended that a study be made of the possibility
of using another work function measurement technique to be used
in conjunction with the retarding field diode technique. The
purpose of this is to provide a secondary independent measurement
to minimize the possibility of systematic errors in the work
function determination. The vibrating reed technique is con-
sidered a good secondary method. One advantage of this method of
measuring work function is that it permits continuous measure-
ments. A disadvantage is that, as ordinarily used, the vibrating
reed technique provides only relative measurements, however,
when used in conjunction with an absolute method (retarding fielde
diode) this is not a substantial disadvantage.

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3. It is further recommended that the measuring circuit frequency response be increased to reduce the time required to make a work function measurement using the retarding field diode method so that more rapid changes in work function may be followed.

4. It is recommended that continuous spectrum analysis be performed on the system background gas.

The following recommended surface studies and experiments are considered important and valuable in the development of a reliable, accurate method of measuring surface cleanliness:

1. It is recommended that the work function of a number of metals be measured for a number of contaminating species (including the common gases) as a fu tion of relative coverage. From experience with various methods of controlling the specimen surface relative coverage, it is recommended that the experimental procedure include a combination of the gas pulse technique for introducing a known quantity of gas and flash desorption technique for removing the absorbed gas in a way that the accomodation coefficient can be measured. The results of these experiments would be a series of plots for various metals of work function vs. relative coverage for various gases. This data is necessary if surface cleanliness is to be deduced from work function measurements.

2. It is recommended that a number of metals having relative surface coverages ranging from one down to a few percent be ion bombarded with various inert gases for a range of ion energy. These experiments will yield data on the cleaning rate of various gases as a function of surface material, contaminating species, surface coverage, and ion energy. All these data are necessary if ion bombardment surface cleaning is to be reduced to a quantitative technique.

3. It is recommended that for certain materials an experimental investigation be conducted on the influence of ion bombardment induced surface damage on work function. Experiments should also be conducted to determine the effect of time and temperature on the annealing of surface damage. These experiments should be performed on materials that have sufficiently high melting points that ordered surfaces may be produced by thermal annealing for comparison and reference.

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