Surface Properties of Etched Tungsten Single Crystals*

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An examination of the surface properties of chemically etched W single crystals has been made to determine the possibility of developing a macroscopic surface with adsorption properties equivalent to an ideal crystal plane.

Crystals up to three inches in length have been grown in 0.003-inch W wires by a thermal gradient technique. The crystals completely filled the wire cross section.

The etching appeared to have produced a surface which was homogeneous on a microscopic level. Thermionic emission was observed to be identical for the three crystallographic directions, [112], [11$\bar{2}$], and [110], with a value of $\phi_0{}^* = 5.25 \pm 0.05$ v. Surface ionization measurements gave the same value of the work function within experimental error.

INTRODUCTION

IN the past, a number of attempts have been made to produce a surface consisting of an ideal single-crystal plane. Cleaved or cut surfaces may have most of the properties of such a plane; however, there are always uncertainties as to the structure at the microscopic level. After the development of the field emission microscope,[1] it was possible to observe microscopically smooth single planes. With the field emission microscope it is possible to measure the properties of such planes and in particular, one may study the adsorption properties provided that the adsorbate changes the work function of the plane.[2] However, there are a number of useful techniques for studying surfaces which require a macroscopic surface area. Among these are the flash filament technique,[2] surface ionization,[3,4] and measurement of secondary emission properties.[5]

Recent work in germanium crystallography, suggested the possibility that an etched surface might expose only one type of crystallographic plane, and thus, have many of the properties of an ideal crystal plane. Ellis[6] found that it was possible to develop preferentially a number of the low-index planes by using various chemical etches on Ge spheres. Earlier, Taylor[7] stated that his "aging" technique for tungsten filaments produced a surface consisting of many small dodecahedrons made up of (110) planes. The "aging" may be termed "thermal etching" since, presumably, it results from preferential migration of tungsten atoms.

For this work, tungsten single-crystal surfaces have been produced by several etching techniques and examined for homogeneity. The thermionic work function was used as a primary measure of homogeneity. Since this method has the fault that any distribution of surfaces with different work functions will appear heavily weighted with respect to the lowest work function, the work function has also been determined from the surface ionization of sodium. In this method the highest work function in a distribution will have the heaviest weight.

EXPERIMENTAL

Crystal Growth

The tungsten single crystals were grown in the apparatus shown in Fig. 1. A 0.003-inch diameter wire[8] was slowly passed through a short heated zone produced by a small tungsten spiral heated to 2600°K. The wire was protected from oxidation by H_2 gas at atmospheric pressure. Owing to the background of light from the spiral, accurate pyrometer measurement of the wire temperature was impossible. However, it was estimated to be above 2400°K. At this temperature, a gradient of about 2000°K/cm existed at the ends of the heated zone.

A growth rate of 4 cm/hr was found satisfactory. Although no careful investigation was made, the tension of the wire during crystal growth appeared to affect the growth process.

Under these conditions, it was possible to grow crystals several inches in length which completely filled the cross section of the wire. X-ray diffraction[9] showed that the usual orientation existed with the [110] axis of the crystal parallel to the wire axis.[10]

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[1] E. W. Muller, Z. Physik **120**, 261 (1943).
[2] J. A. Becker and C. H. Hartman, J. Phys. Chem. **57**, 157 (1953).
[3] S. Datz and E. H. Taylor, J. Chem. Phys. **25**, 389 (1956).
[4] I. L. Kofsky and H. Levinstein, Phys. Rev. **74**, 500 (1948).
[5] H. D. Hagstrum, Phys. Rev. **96**, 325 (1954).
[6] R. C. Ellis, Jr., "Etching of single crystal germanium spheres," AJME Meeting, February 2, 1954 (unpublished).
[7] J. B. Taylor and I. Langmuir, Phys. Rev. **44**, 423 (1933). C. Herring and M. H. Nichols [Revs. Modern Phys. **21**, 185 (1949)] have given a very complete discussion of the requirements of thermodynamic stability of surfaces undergoing thermal etch.
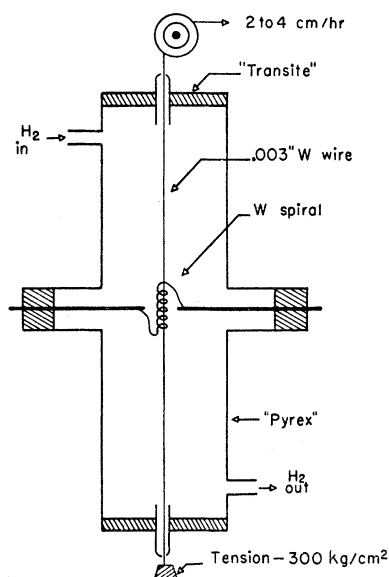
[8] Obtained from Kulite Tungsten Company, Union City, New Jersey. The list of impurities given by the manufacturer contains the usual manufacturing fluxes. We have found that single crystals made from this wire contain about 1/100th of the alkali impurity contained in crystals made from the "G.E. 218" wire and that there is no spectroscopically observable carbon content.
[9] The authors are indebted to Dr. L. Luedemann for the x-ray work discussed in this paper.
[10] R. P. Johnson and W. Shockley, Phys. Rev. **49**, 439 (1936).

FIG. 1. Crystal-growing apparatus.

Surface Preparation

A number of different etch techniques were tried: (1) heating to 2600°K in O_2 at 5×10^{-5} mm Hg for several hours; (2) immersion in boiling H_2O_2 for 30 minutes; (3) immersion in a room temperature solution of 70% HNO_3:30% HF for 2 minutes. Under the optical microscope, the results of all three methods appeared similar, so the HF:HNO_3 etch was chosen for the rest of the work.

A photomicrograph of a crystal etched with HF:HNO_3 is shown in Fig. 2(A). The section for the photograph was made by drawing Nonex glass tubing over the wire, cutting with a diamond saw, and then sand-blasting the surface for photographic contrast. The outline around the photomicrograph shows the actual shape of the cross section.

The surface consists of four large fairly smooth surfaces, which are normal to the [112] directions and a series of smaller surfaces which are arranged in "steps" along a plane which is normal to the [110] directions. Although it was not possible to make goniometer measurements, it is believed that the faces of the "steps" are crystallographically the same as those of the four large surfaces.

Figures 2(B) and 2(C) show the wire as viewed from the [110] and [112] directions. The inked lines between 2(A) and 2(B) indicate the relationship between the "steps" (as viewed from the cross section) and the corresponding longitudinal faces of the steps (as viewed from a position perpendicular to the wire). Note the parallel "grooves" shown on the surface normal to the [112] direction in Fig. 2(C). They are at an angle of about 60° with respect to the wire axis and are about $1\,\mu$ apart and $1\,\mu$ deep. Electron micrographs[11] show

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[11] The authors wish to thank Dr. W. A. Cote for the electron microscopy.

that these "grooves" are also present on the faces of the "step" structure. High-temperature heating, in high vacuum for several hours, partially smoothes but does not remove the "grooves."

Thus it appears that, on a macroscopic level, the surface is made up of many crystallographically equivalent (112) planes. The presence of the "grooves," however, indicate that these are not "ideal" (112) planes on a microscopic level. In fact, the work to be discussed later indicates that the surface has a superstructure of (112) planes with a uniform microstructure consisting of (110) facets.

After etching, it was apparent that with this growth technique, there is an axial twist of about 5°/inch along the crystal. This checked with the observation that the x-ray Laue patterns were tilted slightly with respect to the wire axis.[9] The twist does not seem to be the result of the die drawing of the wire since two adjacent single crystals in a wire have been found to twist in opposite directions.

Thermionic Work Function Measurements

The experiment consisted in the determination of ϕ_0^* for various crystal directions from the equation:

$$i_v = \alpha A^* T^2 10^{-5050\phi_0^*/T} 10^{GV^{\frac{1}{2}}/T}, \qquad (1)$$

where α is the area, A^* is the experimental Richardson's constant, ϕ_0^* is the experimental field-free work function, and G is the geometrical constant relating the applied potential V to the electric field at the wire surface.

It should be noted that the data are given in terms of crystal directions rather than crystal planes. That is, the emission from the [112] direction is not expected to be
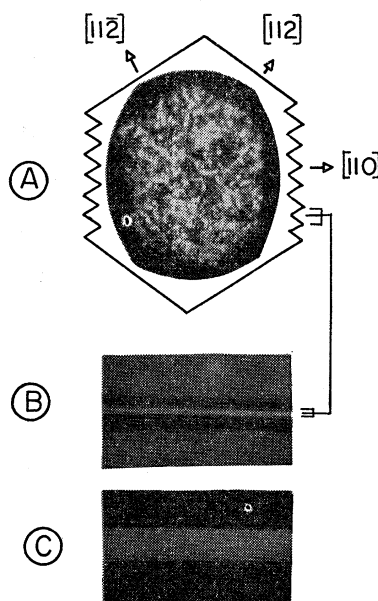

FIG. 2. (A) Wire cross section, (110) plane, 450×; (B) view normal to wire in the [110] direction, 100×; (C) view normal to wire in the [112] direction, 240×.
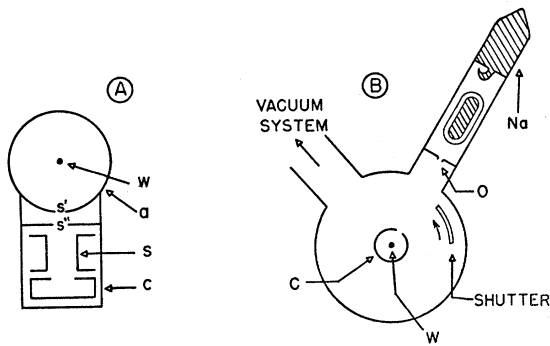
FIG. 3. (A) Thermionic emission electrode arrangement: a, 1.4 kv; S, 0.7 kv; C, 1.4 kv; (B) surface ionization tube.

characteristic of the (112) plane, due to the surface microstructure.

The apparatus, similar to that of Nichols,[12] is diagramed in Fig. 3(A). An etched single crystal, W, was mounted at the axis of a circular cylinder, a, with provision for rotating the wire about its own axis. Thus different portions of the wire circumference could be presented to the slit system S' and S''. Thermal electrons were drawn radially from the wire to the cylinder and some were passed through the slit system and measured at the collector, C. The geometry was constructed to give 3° resolution. With this geometry and slits 1 cm long, only those electrons were collected which originated on a strip of projected area of 2×10^{-4} cm$\times$1 cm.

The function of the electrode S was to suppress secondary electrons from the collector. Plots of collector current, i_V, versus suppressor voltage (at constant collector voltage) were made for several wire temperatures. The collector current rose with suppressor voltage, indicating inefficient suppression at low fields. At higher voltage it flattened out, and again dropped at voltages near those of the collector. This was presumably due to collection of part of the current by the suppressor. Thus the electrode seemed to be behaving as desired as long as the suppressor voltage was set within the plateau range.

The electrons were collected with high radial electric fields at the surface in order to remove space charge effects, and to overcome any tangential fields produced by work function variation on the surface. Nichols[12] and Smith[13] have given a very complete discussion of the latter requirement. Both investigators used a polished circular wire to produce a known geometry. The precise nature of the etched surface was, of course, not known. As will be shown later, the tube appeared to have good projection properties, even though it is probable that many surface microcrystals were not oriented normal to the field.

The electrode structure shown in Fig. 3(A) was sealed in a Pyrex envelope and conventional bakeout and degassing techniques were used. The wire was heat treated for several hours at 2600°K at a pressure of 5×10^{-10} mm Hg. The pressure was measured with an Alpert inverted ionization gauge. Since each data point was taken immediately after a 2300°K flash at a pressure of 5×10^{-10} mm Hg, no prolonged wire cleanliness measurements seemed necessary. However, flash filament checks indicated that after 30 hours at 1100°K the wire had adsorbed residual gas corresponding to about 10^{11} atoms/cm^2.

Wire brightness temperatures were measured with a micro-optical pyrometer with a precision of about ±2°K. Emissivity corrections were taken from Forsythe's tables.[14] Over the experimental range, no end-loss corrections were necessary. The current measurements were made with a Keithly Model 410 micro-microammeter. High-voltage measurements were made with a potentiometer and precision resistors.

The procedure consisted of the following: a measurement of i_V versus T, the determination of $(-5050\phi_0^*+GV^{\frac{1}{2}})$ from a Richardson plot, and then the calculation of G from the slope of a Schottky plot. Figure 4 shows the Richardson plots for each of three crystallographic directions. The field-free value of $\phi_0^* = 5.25\pm0.05$ v was
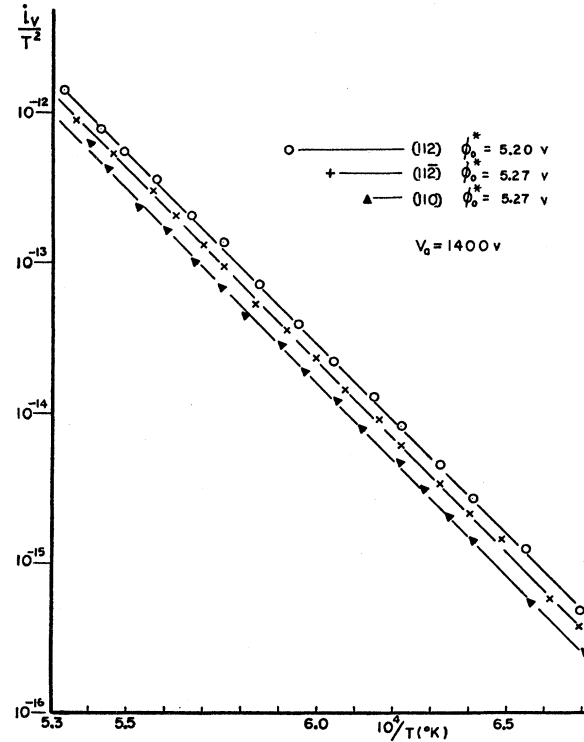

FIG. 4. Experiment plot of Eq. (1); work function data is "field-free."

[12] M. H. Nichols, Phys. Rev. 57, 297 (1940).
[13] G. F. Smith, Phys. Rev. 94, 295 (1954).
[14] W. E. Forsythe and A. G. Worthing, Astrophys. J. 61, 146 (1925).

obtained. The work function was the same within 0.02 v for angles ±10° about the indicated crystallographic directions.

For a polished circular wire, Nichols[12] and Smith[13] obtained values ranging from 4.32 v in the [111] direction to an estimated 5.26 v in the [110] direction. Even assuming a variation in work function of 0.05 v, it is obvious that etching produces a great increase in surface homogeneity.

It should be noted that this increase in homogeneity is not so pronounced for imperfect crystals. Cross sections of the emission crystal made after completion of the measurements disclosed an imperfection near one end, occupying the corner of the crystal opposite the (110)–(112) corner. The Richardson plot in this direction was nonlinear with a slope of $\phi_0^* \approx 5$ v.

With the etched surface, no area measurements were possible. Values of αA^* taken from the plots of Fig. 3 fall in the region, 39 ± 7 (amp/deg^2). Nichols[12] and Smith,[13] with much more precise apparatus, found deviations of 5–10% in A^* for equivalent crystallographic directions. Thus, the ±15% deviation in αA^* for different directions is probably not significant from the standpoint of homogeneity. The values of A^* would be useful as indicators of surface inhomogeneity. Since they were not available, it seemed desirable to obtain information with another technique such as the surface ionization process.

The Schottky plots also give information concerning the surface structure. At an accelerator voltage, $V_a = 1400$ v, the computed surface fields are about 320 000 v/cm. The drop in current from the Schottky line occurred near the relatively high value of 100 000 v/cm. There seemed to be no significant differences between the data for [112] and [110] directions. The values of G in Eq. (1) were 27 ± 2 (deg/volt$^{\frac{1}{2}}$) for the three crystallographic directions.

Surface Ionization Measurement

If an atomic beam of alkali metal impinges on a hot tungsten wire, the atoms are adsorbed on the surface. After steady state conditions are reached, a surface population will exist and ions and atoms will evaporate from the surface.[15] For surface concentrations below about 10^{12} atoms/cm^2, the alkali atoms do not interact with one another[16] and the Langmuir-Saha equation[17,18] gives the ratio of the number of positive ions desorbed per cm^2 per second, n_i, to that of neutral atoms desorbed per cm^2 per second, n_a.

$$\frac{n_i}{n_a} = \frac{\omega_i}{\omega_a} \exp\left(+\frac{\epsilon(\phi-I)}{kT}\right). \qquad (2)$$

The ratio of weighting factors, ω_1/ω_2, is $\frac{1}{2}$ for alkali metals; I is the alkali ionization potential and ϕ is the work function for the surface. Since the appearance of ϕ results from the use of Richardson's equation in the theoretical deviation of Eq. (2), $\phi \equiv \phi_0^*$.

Figure 3(B) shows the experimental tube for the surface ionization measurements. The alkali beam was produced through the aperture, O, and struck the etched crystal, W. Positive ions were drawn from W to the collector, C, with a potential of 45 v. The crystal was arranged so that the beam struck the surface at an angle about midway between the [112] and [110] directions; thus ions were drawn simultaneously from the surfaces normal to these directions.

The alkali chosen for the work was Na with $I = 5.12$ v. It was originally intended to bracket the ϕ_0^* value by using both Na and Li ($I = 5.35$ v). However, Li attacked Pyrex so rapidly as to be useless.

The Na was triply vacuum distilled from C.P. grade starting material. The tube was baked and degassed until pressures of 5×10^{-10} mm Hg were obtained. After bakeout, the break-off seal was broken with the magnetic slug and the side-arm was baked for several hours at 450°C. The magnetically operated shutter, S, was closed during this time. After this treatment, the pressure was 1×10^{-8} mm Hg when the Na side-arm was heated sufficiently to deliver a usable beam. At this pressure, the possibility of residual gas contamination arose.

In an auxiliary apparatus similar to Becker's,[2] it was found that, for pressures below 10^{-7} mm Hg, and etched W surface temperatures above 1100°K, the residual gas sticking probability is of the order of 10^{-3} or less. Thus, data taken within a few seconds after a 2300°K flash, in a pressure of 10^{-8} mm Hg, will be essentially clean data. This is supported by the fact that the Na ionization fraction remained unchanged after 30 minutes of residual gas adsorption.

Ion currents were measured with the Keithly micromicroammeter. During the bakeout of the Na side arm, Na was deposited on the walls of the tube. This prevented accurate pyrometer temperature measurements; therefore, the ion current was initially determined as a function of wire heating current. After all measurements were complete, the Na side arm was removed and replaced by a side-arm with an end window. The tube was then cleaned of Na residue, evacuated, and wire temperatures versus wire heating current data were taken. Ion current versus temperature was then determined. With this method, the temperature was reproducible to ±5°K.

The ion current is proportional to the ion current density, n_i, of Eq. (2). With this method, n_a may not be determined directly. However, with the technique of Johnson and Phipps,[19] one may measure the total beam

15 I. Langmuir and K. H. Kingdon, Proc. Roy. Soc. (London) A107, 61 (1925).
16 J. A. Becker, Ann. N. Y. Acad. Sci. 58, 723 (1954).
17 I. Langmuir, Phys. Rev. 21, 380 (1923).
18 M. N. Saha, Phil. Mag. 46, 534 (1923).

19 A. A. Johnson and T. E. Phipps, J. Chem. Phys. 7, 1039 (1939).

current, $N=n_i+n_a$, and thus determine the ionization fraction. The method requires that the work function be raised to the point where total ionization occurs. This is accomplished by adsorbing oxygen on the surface. In the presently described experiment, the wire was cooled to 300°K with the shutter closed and the Alpert gauge grid was heated. The pressure rose to 5×10^{-5} mm Hg. The tube was then pumped back down to 1×10^{-8} mm Hg, the shutter opened, and the wire temperature slowly raised. The maximum ion current obtained was taken to be the total beam, N. The steadiness of the beam intensity was monitored by periodic observations of the ionization current at a standard reference temperature.

Since, as the wire temperature rises, oxygen is desorbed from the surface and the work function approaches its clean value, it was difficult to keep enough oxygen adsorbed to measure the total Na beam. In the auxiliary apparatus mentioned above, crude duplication was made on the oxygen desorption experiments described by Hagstrum[20] and Ehrlich.[21] Desorption of oxygen and residual gas containing oxygen was found to take place in three stages. That is, if one adsorbs the gas at 300°K and slowly raises the wire temperature, a burst of gas is desorbed at $\approx$ 500°K, 1150°K, and 1750°K. Becker's desorption curves[22] for O_2 on various planes of a field emission point are roughly the same. They indicate that the work function will be of the order of 6 v or greater until the second desorption stage is completed, at which time the work function drops to approximately 5.5 v. The difficulty in keeping oxygen adsorbed was due to the fact that the second desorption stage began only slightly above the temperature of the surface ionization threshold. By heating the wire extremely slowly from the 300°K adsorption temperature, it was possible to retain the second adsorbed stage until the surface ionization threshold was reached. An error in total beam measurement will only move the ionization fraction to lower ϕ, by a constant amount.

The data were plotted in the form:

$$\frac{n_i}{n_i+n_a}=\left[1+\frac{\omega_a}{\omega_i}\exp\left(-\frac{\epsilon(\phi-I)}{kT}\right)\right]^{-1}. \qquad (3)$$

Figure 5 shows the results. With the precision obtained, it is estimated that no more than 10% of the surface could be of work function greater than 5.25 v.

As mentioned above, Eqs. (2) and (3) should be applicable only if the Na surface concentration, θ, is low enough to prevent Na-Na interactions. The data are not available to relate n_i or N to θ. However, $n_i=\theta/\tau$ where τ is the mean life in seconds of adsorbed Na$^+$ ions. The lifetime is temperature dependent and varies with the type of surface. Values of τ for Rb on the etched surface
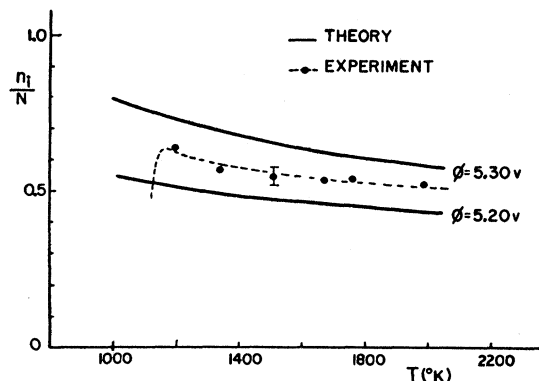

FIG. 5. Surface ionization ratio for Na on etched W; theory plot is Eq. (3) for different values of ϕ.

are available.[23] Evans[24] has measured τ for Rb and Na on another type of tungsten surface. By assuming that the ratios between the τ values for Na on the two different surfaces are the same as the ratios of the τ values for Rb on the two surfaces, an order of magnitude estimate may be obtained for τ_{Na} on the etched surface. At $T=1100°$K and ion currents of the order of 10^{-8} amp, $\theta\approx5\times10^9$ atoms/cm². At higher temperatures, θ will be much less. Thus, the ionization fraction will be independent of the Na surface concentration.

DISCUSSION

As mentioned before, the primary objective of this work has been to investigate the possibility of developing a macroscopic surface which would exhibit the adsorption and electrical properties of a nearly perfect crystallographic plane. From this standpoint, homogeneity of the surface becomes the primary consideration, and the various surface properties are to be regarded only as parameters which confirm or deny the hypothesis. At present, there is too little supplementary information available for such surfaces to permit one to consider the parameters as physical constants. In this connection, it may be mentioned that the spectral emissivity for such surfaces is not known and may be slightly different from that of mechanically prepared surfaces.[25] It is within this restricted frame of reference that the results will be discussed.

There are several points to be considered: (a) the high value of electric field required to produce a linear Schottky plot; (b) the unusually high value of αA^*, and (c) the experimental significance of the relationship $\phi\equiv\phi_0^*$.

The phenomena of (a) and (b) appeared to be related and to be the result of microcrystal development on the surface. The Schottky plots resembled those obtained by Smith[13] for the [110] directions. He attributed the

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[21] G. Ehrlich, J. Chem. Phys. 23, 1543 (1955).
[22] J. A. Becker and R. G. Brandes, J. Chem. Phys. 23, 1323 (1955).

[23] F. L. Hughes and H. Levinstein, Phys. Rev. 113, 1029 (1959), following paper.
[24] R. C. Evans, Proc. Roy. Soc. (London) A139, 604 (1933).
[25] A. G. Worthing, Astrophys. J. 36, 345 (1912).

effect to spurious secondary electrons from the accelerator, $(a,$ of Fig. 3), produced by electrons emitted from low-work-function areas of the W surface. This possibility seems unlikely in the present case since it requires a suppressor characteristic of a form much different from the one which was observed.

Another consideration was the possibility of variation in work function within a distance which is small compared to the resolution of the slits. Becker[26] has developed a successful model for polycrystalline surfaces which predicts a Schottky plot resembling that obtained for the etched surface. In this theory, he assumes a varying work function with periodic boundary conditions fixed at the edges of a "checker board" pattern on the surface. The Schottky emission is found to depend on the "checker" dimension and the variation in work function. For the etched surface, the "checker" dimension should be of the order of the optically observed microstructure, that is, about 10^{-4} cm. Reasonable values for the work function variation should be of the order of a few tenths of a volt. With these values the model predicts a Schottky plot which is linear to much lower fields than are actually observed and thus does not seem applicable to the etched surface.

In view of the close agreement between the values of ϕ and ϕ_0^*, it is felt that while there is a periodic geometrical variation on the surface, the "facets" of the "hill and valley" structure have a fairly uniform structure and work function. If one assumes that the Schottky emission arises from a smooth surface of effective radius, $r_i,$ then the observed values of G give, $r_i \approx 1 \times 10^{-4}$ cm. With the emission tube geometry, structure of this kind will inevitably result in fields tangental to some or all of the "facets." Thus, one should not expect[27] the measured αA^* to be that equal to the "theoretical" value of 120α. It is interesting to note that,[28] where thermionic data exists for a number of surface preparations of the same metal, the high values of A^* are associated with high values of ϕ_0^*.

As was mentioned previously, one would expect, on theoretical grounds, that $\phi \equiv \phi_0^*$. Surface ionization for a surface with areas of different work functions, would be represented theoretically by a sum over terms of the type in Eq. (2).[29] The experimental value of ϕ for such a surface would be weighted to the high-work-function areas since those areas would produce more ions per unit alkali concentration. However, since the alkali is mobile

at the operating temperatures,[30] one must consider the possibility that migration may be concentrating most of the alkali on the lowest-work-function areas of the surface. It seems to be true that the binding energy of Na on W is highest on low-work-function surfaces.[30] However, the ratio: probability of migration/probability of evaporation $\approx e^{-\Delta Q/kT}$, where ΔQ is the difference between the migration activation energy and the desorption energy.[31] For Na on W in the temperature region of interest, this ratio is approximately 10^4, that is one would expect a Na atom to "hop" about 10^4 times before evaporating as an ion. At these low alkali surface concentrations, the situation is very different from the usual one where migration takes place due to a concentration gradient. With the values of θ which were used, there will be about one alkali atom per 10^5 lattice sites and, therefore, the problem of migration of an atom should be governed by "random walk" statistics. This implies that the most probable migration distance between the deposition and the evaporation sites will be of the order of 10^2 lattice constants. Thus, the migration is not expected to affect appreciably the initial alkali surface concentration on microcrystals of linear dimensions of 10 000 lattice constants.

CONCLUSIONS

Thermionic work function measurements have been made in each of three crystallographic directions on an etched W single crystal. The values were the same within experimental error and fall within the limits $\phi_0^* = 5.25 \pm 0.05$ v. The value agrees with that obtained by surface ionization measurements.

The data indicates that at least 90% of the surface is of the same structure. The surface appears to consist of microcrystal "facets" with dimensions of the order of 1×10^{-4} cm. The measured value of ϕ_0^* suggests that these are (110) planes.[13,32]

It would be desirable to make retarding potential measurements on etched surfaces with a more favorable geometry such as that attained by Shelton.[33]

[26] J. A. Becker, Revs. Modern Phys. 7, 95 (1935).
[27] W. B. Nottingham, *Handbuch der Physik* (Springer-Verlag, Berlin, 1956), Vol. 21, p. 28.
[28] S. Dushman, Revs. Modern Phys. 2, 381 (1930).
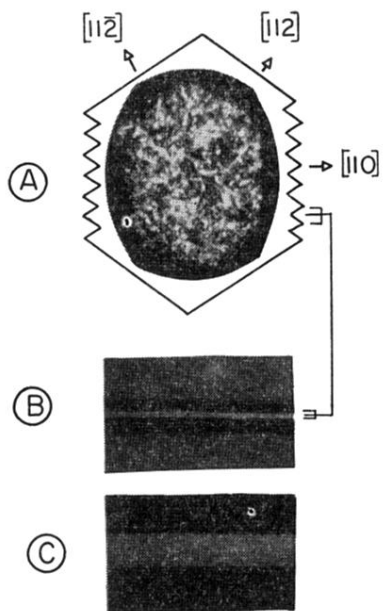[29] J. Zemel, J. Chem. Phys. 28, 410 (1958).

[30] M. Benjamin and R. O. Jenkens, Proc. Roy. Soc. (London) A180, 225 (1942).
[31] B. M. W. Trapnell, *Chemisorption* (Butterworths Scientific Publications, London, 1955), p. 205.
[32] Dyke, Trolan, Dolan, and Grundhauser, J. Appl. Phys. 25, 106 (1954).
[33] H. Shelton, Phys. Rev. 107, 1553 (1957).

FIG. 2. (A) Wire cross section, (110) plane, 450×; (B) view normal to wire in the [110] direction, 100×; (C) view normal to wire in the [112] direction, 240×.