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SURFACE IONIZATION ON TUNGSTEN SINGLE-CRYSTAL FILAMENTS

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ABSTRACT

Surface ionization of atomic beams of strontium or calcium from the (110) and (111) planes of tungsten was studied mass spectrometrically. Results consistent with the Saha-Langmuir relation indicate that the method is applicable for the determination of first-ionization potentials or work functions. A value for the work function of the (110) plane of tungsten is 5.41 ± 0.04 eV. A value for the (111) plane of tungsten is 4.49 ± 0.04 eV.

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INTRODUCTION

The present study was prompted by an interest in (a) surface-ion sources for use in mass spectroscopy, and (b) surface ionization as a method of determining ionization potentials. The latter depends on the assumption that the Saha-Langmuir equation describes the correct relationship between the surface work function θ of a metal, and the ionization potential (I) of an atom evaporated from the metal surface.

The ratio of the number of ions to atoms leaving a hot metal surface is $\alpha = \frac{N+}{N_o}$, and is known as the degree of ionization. In a simplified case, with $N_o \gg N+$, α is determined by the relationship

$$\alpha = \frac{N+}{N_o} = \frac{g+}{g_o} \exp\left[\frac{e(\theta-I)}{KT}\right]. \qquad (1)$$

The ratio $\frac{g+}{g_o}$ gives the statistical weights of the ionic and atomic states, I is the ionization potential of the impinging element, and K, T, and e have their usual meanings. The determination of α experimentally depends not only on the measurement of the metal temperature, on θ, and on the ionization potential of the evaporating atom, but is also influenced by the physical and chemical condition of the metal surface, or by the degree of surface coverage by foreign atoms and the impinging atoms. The experimental results are also

influenced (a) by the electric field extracting the ions from the metal surface, and (b) under the usual conditions, by the potential drop along the emitting filament, since in most experimental cases the filament is not an equipotential surface.

To minimize a number of these experimental effects, a homogeneous single-crystal surface was used, ultrahigh vacuum conditions were maintained, low-intensity atomic beams were employed, and particular attention was given to field-extraction conditions.

Recent technological interest in ion-propulsion engines and in thermionic-energy converters has resulted in a series of survey papers on the subject of surface ionization.[1,2,3] A critical review of the subject of surface ionization by Zandberg and Ionov[4] appeared in 1959, and a timely review by Mueller[5] brings the literature coverage up to a recent date. Kaminsky[6] has written an excellent review of the subject in his article on "Atomic and Ionic Impact Phenomena on Metal Surfaces," which will appear shortly.

We have confined our studies to the surface ionization of metallic strontium or calcium on tungsten surfaces whose crystal surface orientation has been ascertained by x-ray techniques. Particular interest has centered on acquiring data from the (110) plane, from the (111) plane, and from the crystal planes obtained when polycrystalline ribbon filaments are heated for long periods at elevated temperatures. From these experiments new data have been acquired on the work function of these surfaces; also, an evaluation of the method as a means of determining ionization potentials or work functions has been made.

The field-emitting point technique,[7] and the bulb method[8,9] have been two well-known modes of obtaining information on the variation of work function with crystal structure. Values obtained by various workers using these

techniques seem to be in particularly poor agreement for the (110) surface of tungsten. Table I shows these data. The range of values is 1 eV or more.

In this study we employ an atomic-beam method, impinging the neutral beam on a hot tungsten surface of known crystal orientation. A given positive-ion species was detected with a mass spectrometer. The reproducibility in the value of a surface work function and the behavior of the Saha-Langmuir[8,10] plot were therefore taken as basic indexes that the method could be used to evaluate unknown ionization potentials.

A comparison of first-ionization potential values, determined by surface ionization techniques, with other methods is shown in Table II. Agreement is most cases is not too impressive.

Of particular interest to mass spectrometry is the value of θ assigned to the (110) tungsten plane. A stable high-temperature surface having a high value of θ would greatly improve small-sample efficiency in surface-ionization types of sources.

PREPARATION OF SINGLE-CRYSTAL FILAMENTS

Attempts were made to produce single-crystal tungsten filaments from multicrystalline tungsten ribbon stock by strain-annealing techniques. This method can produce single-crystal tungsten filaments but it has the disadvantage that there is little control over the orientation. The drawn or rolled direction of the tungsten ribbon is close to or is aligned in the [110] direction. This orientation remains with the large grown crystals after strain annealing, but the surface plane normal to the rolling axis may be (211), (311), (310), (411) or some other closely associated higher-index plane. At no time was a simple surface index such as (110) or (100) produced.

The strain-annealing method consists of supporting a tungsten ribbon ($\sim 0.030 \times 0.001 \times 5$ in.) at the top and attaching a 30 to 50-g weight at the other end. This ribbon is then bombarded with 1.2 to 1.5-keV electrons by slowly moving a single-loop filament along the ribbon. The rate of travel is approximately 1 cm/hr. The hot zone is kept between 2500 and 2900°K and emission controlled. The thermal gradient along the ribbon is approximately 2000°C/cm. The apparatus used is shown in Fig. 1. The start of large crystal growth is shown in Fig. 2.

Crystals several centimeters long could be produced by this method. When more than one crystal appeared across the ribbon each had the same orientation within one or two degrees. This structure results in a close-grouped double- or triple-spot Laue pattern. An example of this pattern is shown in Fig. 3. The orientation of all of the strain-annealed filaments seemed to fall into two rather distinct groups. In one case the [110] pole seemed to be precessing some 15° about the filament axis, and in the other it was parallel to the axis. Rieck has found that the purer the tungsten, the more likely the filament axis will be [110].[11] The surface normal of all the filaments processed seems to lie within an area as shown in Fig. 4. Not all these filaments were made from the same stock material; for example, two filaments processed at another laboratory had this two-pattern axis orientation and a surface normal near the (311) plane.[12]

The second method of producing single-crystal filaments, although tedious, resulted in filaments with a preferred orientation. We sliced filaments from bulk-tungsten single-crystal stock* by first cutting the crystal into 0.020 in. thick wafers. The wafers were ground to about 0.005 in.

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*Large single crystals of Tungsten were obtained from the Linde Co., Crystal Products Div., Los Angeles, California.

thickness and slit into 0.035 in. wide strips by cutting with a miniature sand-blast cutter. The strips were ground to 0.002 in. thickness and then electropolished† to approximately 0.001 in. thickness. The electropolished filament can be shaped at room temperature without breaking. Electropolishing also removes surface damage caused by the above mechanical treatment, and must always constitute the final filament treatment before heating or x-ray analysis. Back-reflected Laue pictures were taken of the filaments before and following experimental runs in the mass spectrometer.

MASS SPECTROMETER

The mass-spectrometer source, first employed by Werning,[13] is shown schematically in Fig. 5. The filament is mounted so its surface is aligned with both a small Knudsen-type effusion oven A and with the exit-source slit of the mass spectrometer. A magnetically actuated shutter H, operated remotely, can interrupt the atomic beam. Biased grids C and C' keep both electrons and positive ions, which originate at the oven, from striking the hot filament. The atomic beam passes down a narrow channel in the filament mounting plate E and is collimated at F before striking the hot filament G. All plates are separated by threaded Pyrex or fused alumina rod-and-spacer construction.[14] The atomic-beam oven and source are shown in Fig. 6.

The mass spectrometer is a 20-cm-radius 60°-sector all-metal instrument

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†Tungsten is successfully electropolished by electrolyzing the metal in 2% NaOH solution. The work is connected to a positive dc supply of about 5 to 10 V with a negative electrode of Nichrome V or stainless steel. As the voltage is increased, a current plateau is reached and electropolishing proceeds.

equipped with copper gaskets. It is pumped by mercury diffusion pumps. The detector is a 16-stage electron multiplier having silver-magnesium dynodes. The base pressure of the instrument is about 3×10^{-9} torr; with the source running hot the base pressure is about 8×10^{-9} torr. The output of the multiplier is fed to a vibrating-reed electrometer and the integrated signal is chart-recorded.

Source-filament temperatures were measured by an optical pyrometer. The pyrometer was standardized against a lamp obtained through the G. E. Lamp Development Laboratory at Nela Park. Necessary temperature corrections for tungsten emissivity and window transmission were made.[15]

LAUE PICTURES

The Laue back-reflected x-ray pictures of the filaments were made by using a specially constructed camera and sample holder fitted to standard Norelco equipment. White radiation from a molybdenum target was collimated to a 0.020 in. diam beam on the sample surface. The usual 3 cm spacing between the sample and film surface was employed. Exposures were from 4 to 7 hours. All exposures were plotted on an 18-cm-diam stereographic projection net, and repeated exposures of the same filament surface, where the sample was demounted and remounted, indicated shifts of less than $\pm 1^{\circ}$ resulting from mechanical misalignment.

EXPERIMENTS AND RESULTS

Three different filament surfaces were studied: multicrystalline stock tungsten filaments, and tungsten filaments whose surface planes were respectively (110) and (111). In the initial experiment we studied the surface-ionization behavior of strontium (whose first-ionization potential is well known) on the so-called stock tungsten ribbon-filament material. The atomic-beam oven was held at constant temperature and the strontium beam was allowed to impinge on the emitting filament. By measuring the Sr^{88} ion current as a function of filament temperature and by plotting these data as $\ln i_+$ against $\frac{1}{T}$, one should obtain a straight line. This is the familiar Saha-Langmuir plot where the slope has a value of $\frac{e(\theta-I)}{K}$ [see Equation (1)].

Deviations from a straight line occur at relatively low temperatures, but at filament temperatures above 2100°K the plot is linear. Werning[13] and Michel and Werning[16] have shown that some of the deviations from this straight-line relationship at low temperatures are due to chemical surface reactions of trace amount of oxygen or fluorine with the hot metal or with the ionizing atom. A plot of strontium on tungsten through both the high- and low-temperature range (Fig. 7) shows the deviation from a straight-line function at low temperatures. This is similar to Werning's results with barium on tungsten.[13] Fluorine could not be seen directly in this experiment, but the SrF^+ ion was detected. A plot of the SrF^+ current with the same temperature scale as the strontium-ion current indicates that both low-temperature peaks occur within nealy the same temperature range. Also, an additional experiment indicated that fluorine was coming from the filament. If the hot filament were flashed at 2500°K and then allowed to collect a quantity of strontium atoms at room temperature, a subsequent heating at 1750°K resulted in a very short pulse of

strontium ions with essentially no decay time. If the filament were kept at a slightly elevated temperature (1000 to 1200°K) for a few minutes, and then allowed to collect strontium on its surface at 300°K, a subsequent heating at 1750°K resulted in a sudden rise and a long decay period. No reasonable amount of exposure time at 300°K produced this result, indicating that residual-gas absorption was not contributing to this observation. This would indicate that a compound of strontium and fluorine could be formed, and that the residence time was different from that of strontium atoms. At a steady-state temperature at which both strontium and strontium fluoride could exist on the surface, the total emission could be enhanced by the presence of fluorine atoms. Simple calculations indicate that there is not sufficient fluorine present to raise the bulk work function. This surface reaction will receive further study.

More than 50 separate runs were made of strontium striking a stock polycrystalline filament. Most of these runs were used to study the various parameters of the equipment. These results gave an average slope of -1.08 ± 0.03 eV. If the first-ionization potential of strontium is taken as 5.69 eV,[7] the filament surface has a work function of 4.61 ± 0.03 eV. A typical plot of these data is shown in Fig. 10.

That the value of the surface work function did not come out to be 4.52 eV, the accepted value for tungsten, is not surprising. This 4.52-eV value[18,19] was obtained from circular wire filaments and represents an average distribution from a number of planes.[20] The 4.61-eV value agrees quite well with that of Werning,[13] who obtained 4.58 ± 0.01 eV for barium on tungsten ribbon filaments by similar methods. Working with copper and silver beams on tungsten surfaces, Weiershausen[21] obtained 4.66 and 4.65 eV as the work function. This investigation was done above 2500°K. In a paper by Allen, a value of 4.58 eV was obtained from the (311) face of single-crystal tungsten ribbon.

This was determined by thermionic methods. It is indeed remarkable that such close agreement is obtained for tungsten surfaces whose metallographic history and experimental conditions are not likely to be identical.

Microscopic examination of this filament upon completion of the above runs showed that the tungsten had recrystallized into several large crystals. This filament appeared similar to the strain-annealed filaments. Back-reflected Laue exposures gave closely spaced multiple spots all oriented within 1 or 2° of each other. The surface normal was close to being in the [310] direction. Thus, this tungsten ribbon was no longer multicrystalline, but consisted of only 2 or 3 adjacent crystals having the same preferred orientation. It seems most probable from the above considerations that a number of closely related planes grouped as shown in Fig. 4 have very nearly the same work function.

A single-crystal filament, cut from a large Linde single crystal of tungsten by processes previously outlined, was then substituted for the multicrystalline in the mass spectrometer. This filament was oriented in the [110] direction along the axis, as was the surface plane. After many hours of outgassing the filaments, oven, and ion-source parts, this (110) surface was subjected to a constant beam of strontium atoms. The ionization of strontium was studied between 2300°K and 2800°K. The average value of 14 runs was 5.47 ± 0.10 eV for the work function of this surface. With time, the points between 2000 and 2200°K approached a straight-line Saha-Langmuir plot; this was apparently a function of reduced residual gases and reduction in the SrF^{+} ion peak. In this case, the surface work function was quite close to the strontium ionization potential, this fact no doubt contributing to the sensitivity of the data to what is believed to be residual chemical effects. Figure 8 is a stereographic plot of this filament material, which shows that the (110) plane is the surface plane.

The effusion oven was next loaded with metallic calcium and a new filament having a (110) surface was substituted in the mass spectrometer. This surface was studied by using both strontium and calcium atomic beams, there being sufficient strontium remaining from the first run to give almost equal ion currents of Sr^{88} and Ca^{40}. Since the first ionization potential of calcium is 6.111 eV,[15] a greater slope would be expected for this element on the (110) tungsten than that obtained from strontium.

An average of 10 strontium runs gave 5.44 ± 0.05 eV for the work function of the (110) tungsten surface. A similar number of runs with calcium gave 5.45 ± 0.05 eV.

Another filament having a (110) surface orientation was substituted for the previous emitter, and a series of four determinations with calcium gave 5.39 ± 0.04 eV, and nine runs with strontium gave 5.38 ± 0.03 eV for this surface.

A filament having a (111) surface orientation was mounted in the spectrometer ion source. For this surface, the published literature values agree within ±0.2 eV and average around the value 4.4 eV. A series of runs on this surface gave the value 4.49 ± 0.04 eV. A check on the surface orientation is shown in the Laue pattern of Fig. 9. The crystal filament gave a nearly perfect single-crystal pattern.

DISCUSSION OF EXPERIMENTS

The surface-ionization method can be used to determine first-ionization potentials within an accuracy of a few hundredths of a volt. To obtain this accuracy one must be convinced that the emitting surface remains stable, and that the true Saha-Langmuir process is not masked by side chemical reactions with the beam material or with the filament surface in the temperature range under study. It is indicated from these experiments that the data can be influenced by the following parameters:

(a) Low-temperature data do not always give a straight-line Saha-Langmuir plot. This seems to be related to a chemical reaction of some trace impurity taking place at the surface of the metal either with the beam material or with the surface material or both. At high temperatures, the residence time for all absorbents on the surface is relatively very short, and the above trace reactions do not occur.

(b) One must never allow the filament to be electrically biased so that it can emit electrons to the surrounding surfaces. A small suppressor voltage between the filament and adjacent mounting plate was found to be necessary to prevent such emission. In addition, the atomic beam must impinge on an area that is as near an equipotential surface as possible. The electrical center of the filament must coincide with the area seen by the atomic beam as well as with that sampled by the mass spectrometer.

(c) The atomic beam must not contain free electrons or ions. These have to be removed before the beam enters the hot-filament region.

(d) The potential drop between the filament and the first accelerating plate was around 200 V. This is about a 400 V/cm gradient. Changing this gradient had no apparent effect on the Saha-Langmuir slope, but did have considerable effect on beam optics through the ion source.

(e) No attempt was made to study the effect of flooding the filament with a large neutral beam of one element while taking data on a smaller sample beam. One observation of a nonquantitative nature indicated that a very large strontium beam caused spurious results on measurements of a Ca^{40} beam. The calcium beam was smaller than the strontium beam by about a factor of 10^4. This condition will require additional study. The intensity of the atomic beam was in all other cases very low, and, in most cases, the fraction of a monolayer adsorbed never became greater than 0.001.

CONCLUSIONS

A value for the work function of the (110) plane of tungsten is determined. It is also demonstrated that this surface is stable and constant at high temperatures over long periods of time. An average value for the (110) plane is 5.41 ± 0.04 eV.

Our value for the (111) plane of tungsten agrees quite well with values previously given in the literature. Our value is 4.49 ± 0.04 eV.

These results are determined by using two different elements whose first-ionization potentials are well known. Essentially, there is good agreement in the experimental work function of the surfaces studied using either of these elements.

The Saha-Langmuir equation can be used to give correct values for unknown ionization potentials provided detailed attention is given the parameters of possible chemical reaction, physical adsorption, clean-surface conditions, low residual-gas pressure, and electric-field conditions. The ionization potential should,of course,be greater than θ for this method.

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FOOTNOTES AND REFERENCES

*Based on work performed under the auspices of the U.S. Atomic Energy Commission.

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Table I. Variation of literature values for $\theta_{(110)}$ and $\theta_{(111)}$ planes of tungsten.

Worker	$\theta_{(110)}$ (eV)	$\theta_{(111)}$ (eV)	Reference No.
Nichols	4.68	4.39	22
Smith	4.72	4.38	23
Becker	4.90	4.40	24
Wilkinson	4.68	4.64	25
Dyke	5.00 to 5.5	----	26
Müeller	6.0	4.39	27
Shuppe	5.5	4.2	28
Anderyev	4.60	4.3	29
Hutson	5.1	4.38	30
Shuppe	5.14	----	31
Ahern	4.58	----	32
Hughes	5.25 [110] direction	----	33

Table II. Comparison of ionization potential values determined by surface ionization methods and by other methods.

Element	Surface-ionization Method (eV)	Other methods (eV)	Reference No.
Nd	5.51	6.3	34, 17
	6.31		35
	5.10		13
Pr	5.48	5.8	34, 36
		5.7	36
Ce	5.60	6.54	34, 36
		6.91	36
Tb	5.98	6.7	34, 36
U	6.08	4.0	41, 38
	6.25		13
	4.5 to 5.0		37
Pu	5.1	---	39
Am	----	6.0	40

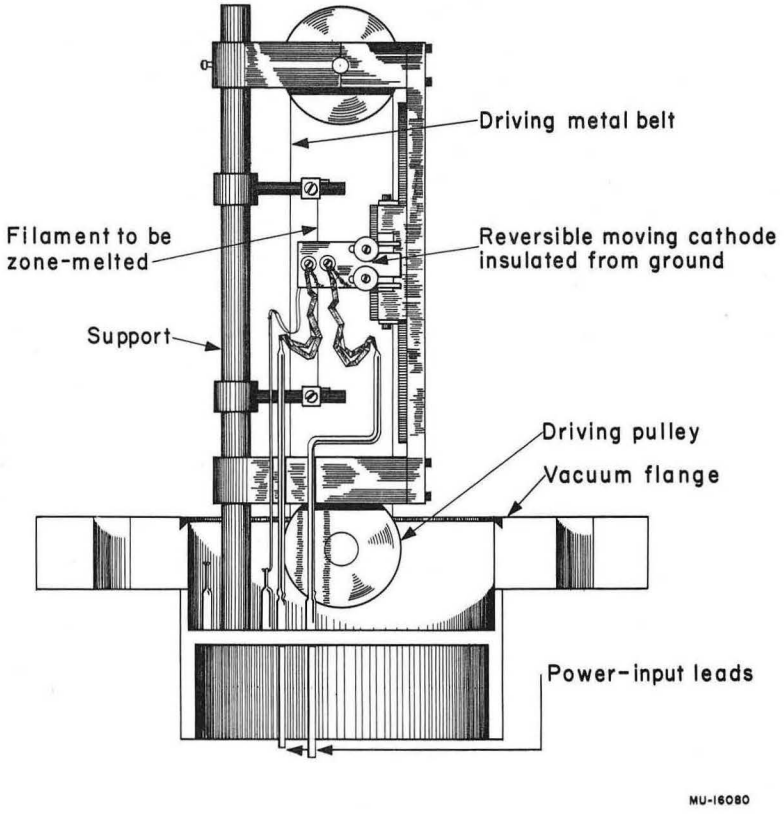

Fig. 1. Equipment used to strain-anneal tungsten filaments.

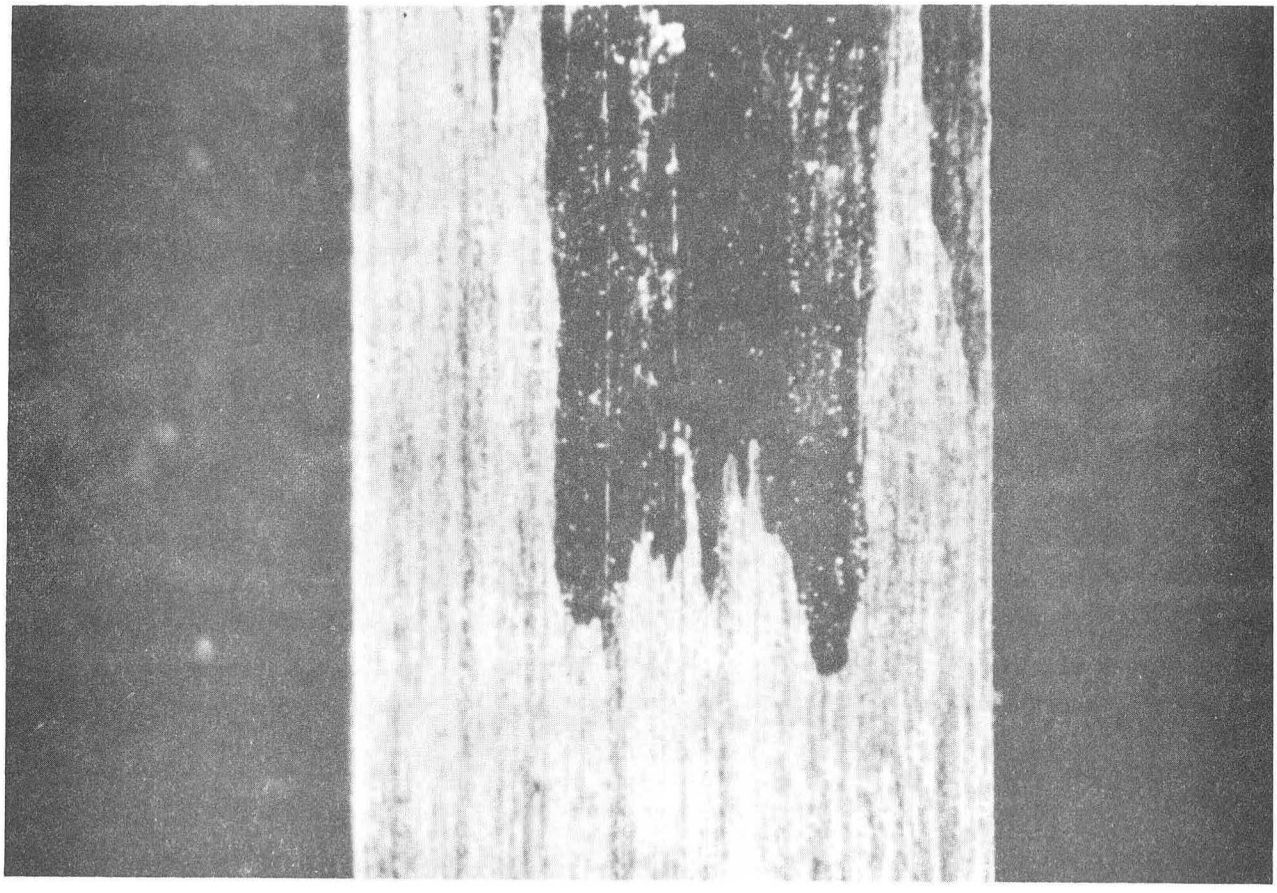

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Fig. 2. Dark section is the start of several large parallel tungsten crystals in a polycrystalline ribbon filament produced by strain-annealing techniques.

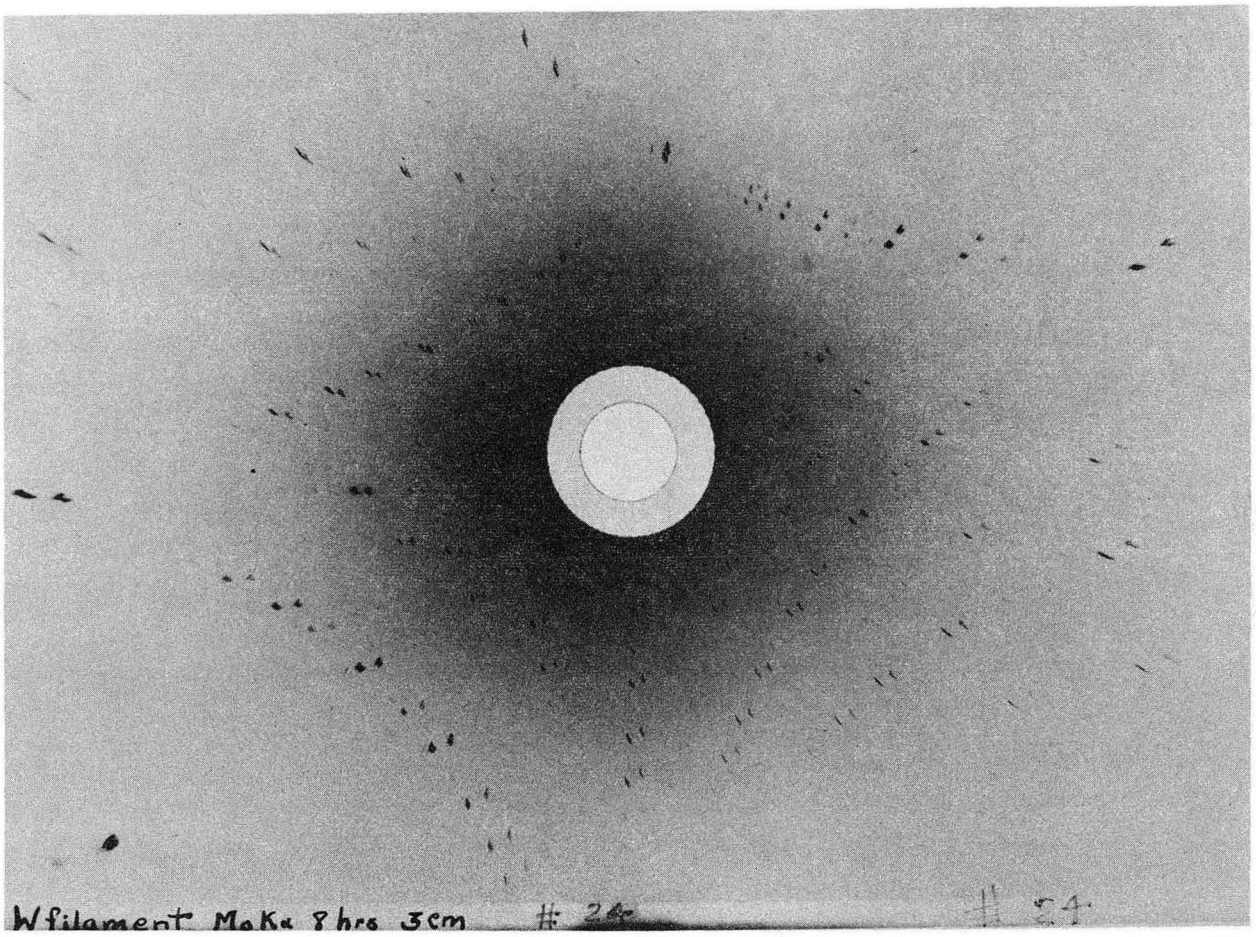

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Fig. 3. Laue pattern of a stock polycrystalline tungsten filament heated for many hours in the mass spectrometer. The filament is no longer polycrystalline, but consist of a few large crystals all of similar orientation. Most strain-annealed filaments gave similar patterns.

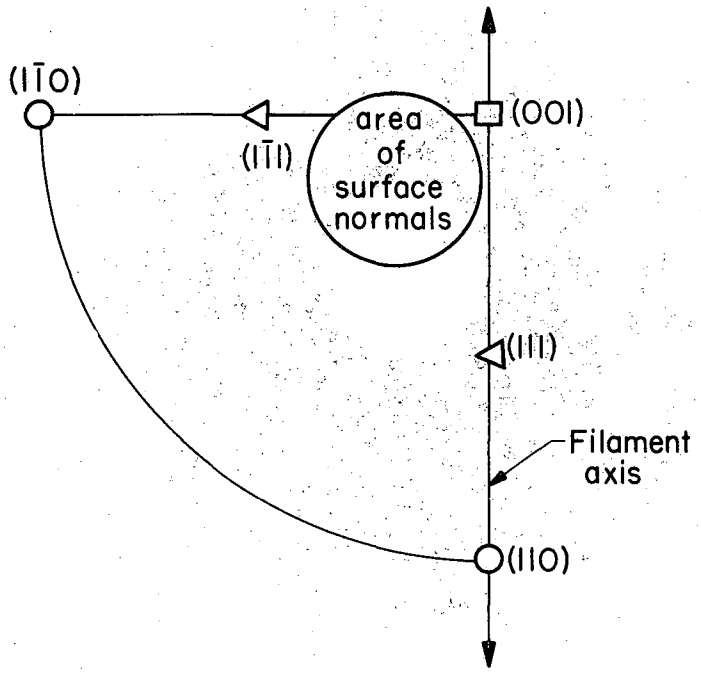

Fig. 4. Stereographic plot showing approximate area of surface normals in relation to the filament axis obtained from a number of strain-annealed crystal-growing experiments.

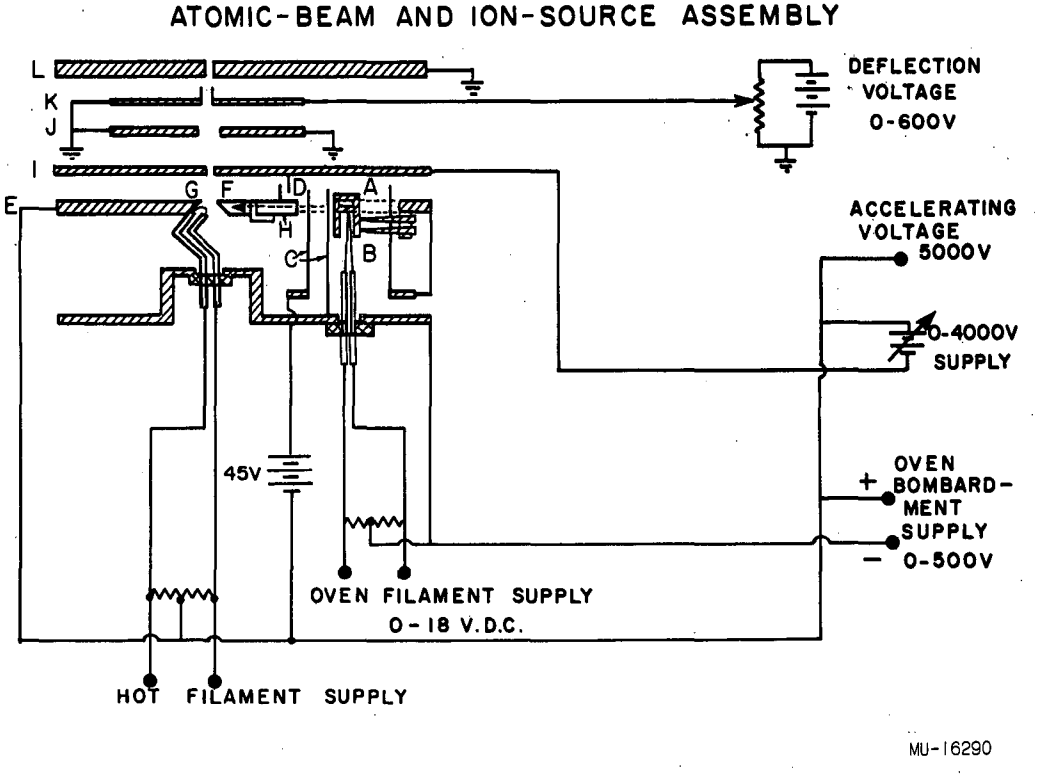

Fig. 5. Atomic-beam and ion-source assembly.

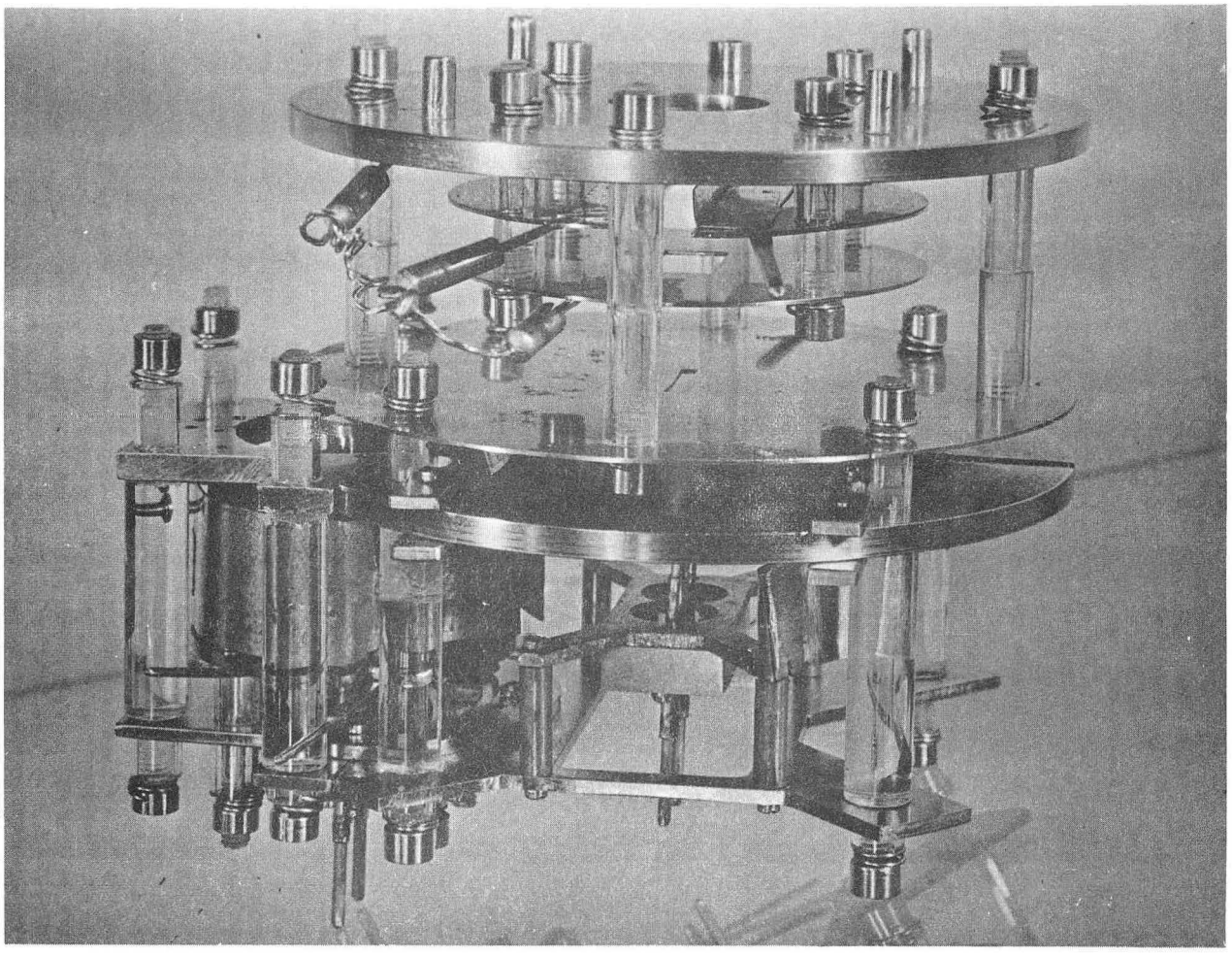

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Fig. 6. Completely assembled ion source.

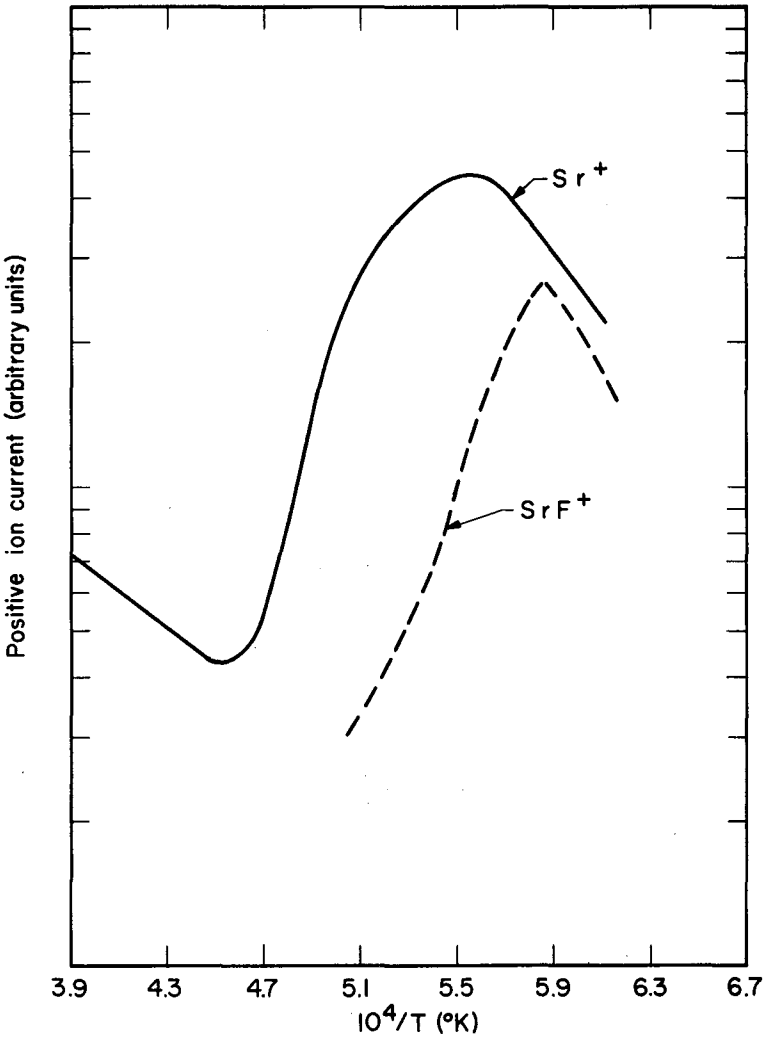

Fig. 7. The deviation from a straight-line plot for strontium on tungsten at low temperatures. Plotted on the same temperature scale is the strontium-fluoride ion peak; the intensity of this peak has been multiplied by an arbitrary factor.

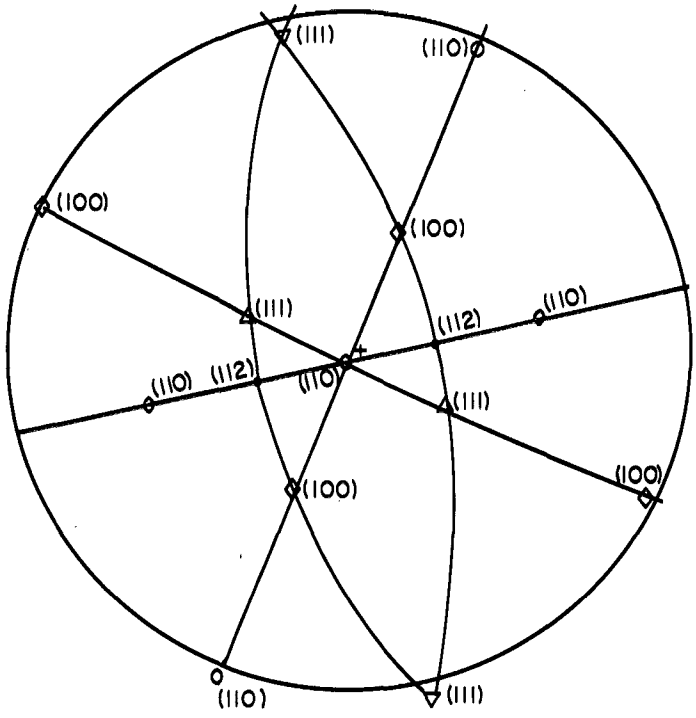

Fig. 8. A stereographic projection of a cut single-crystal filament oriented with a (110) plane the surface normal to the long axis.

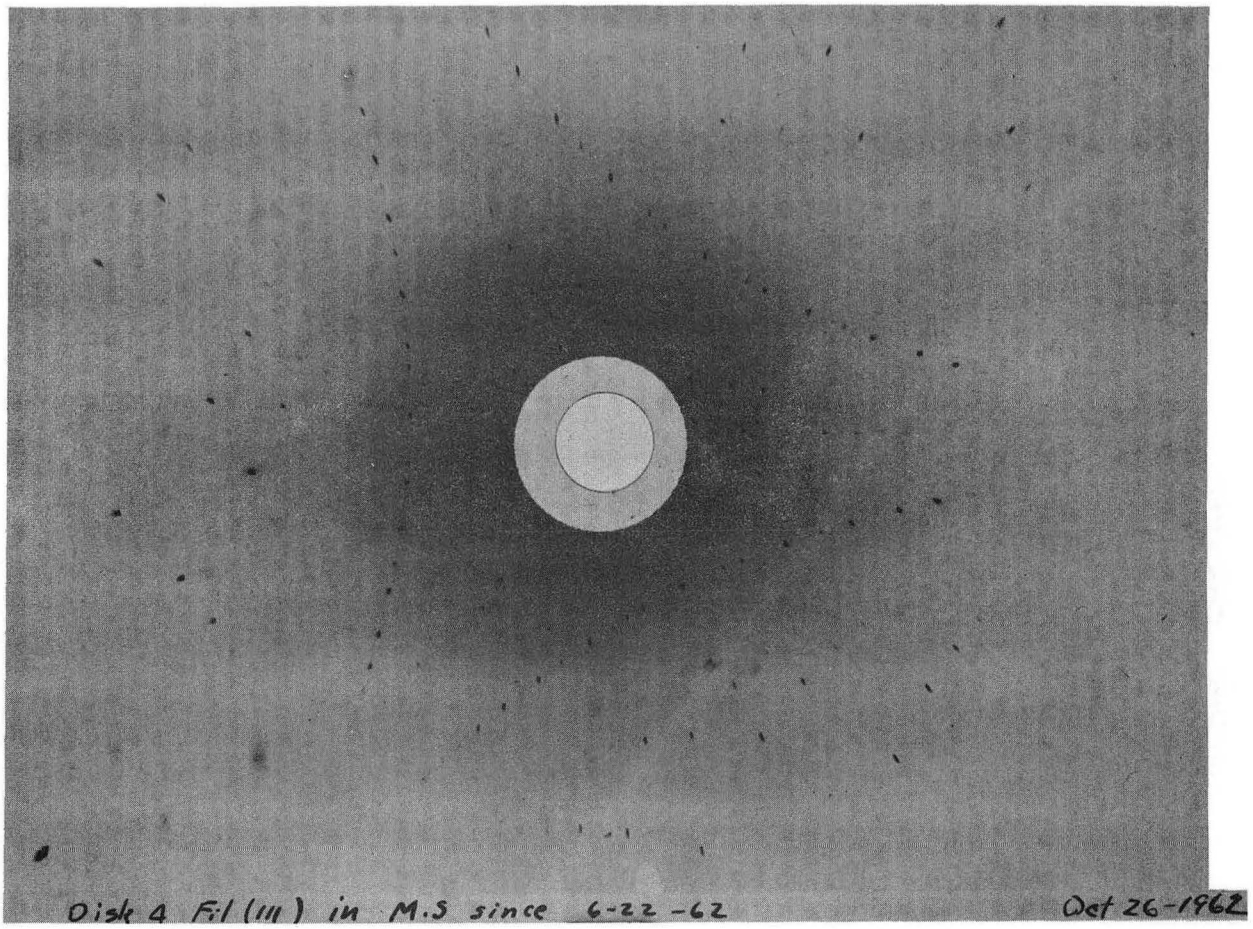

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Fig. 9. Laue pattern of the (111) plane filament after a series of mass-spectrometer work-function determinations.

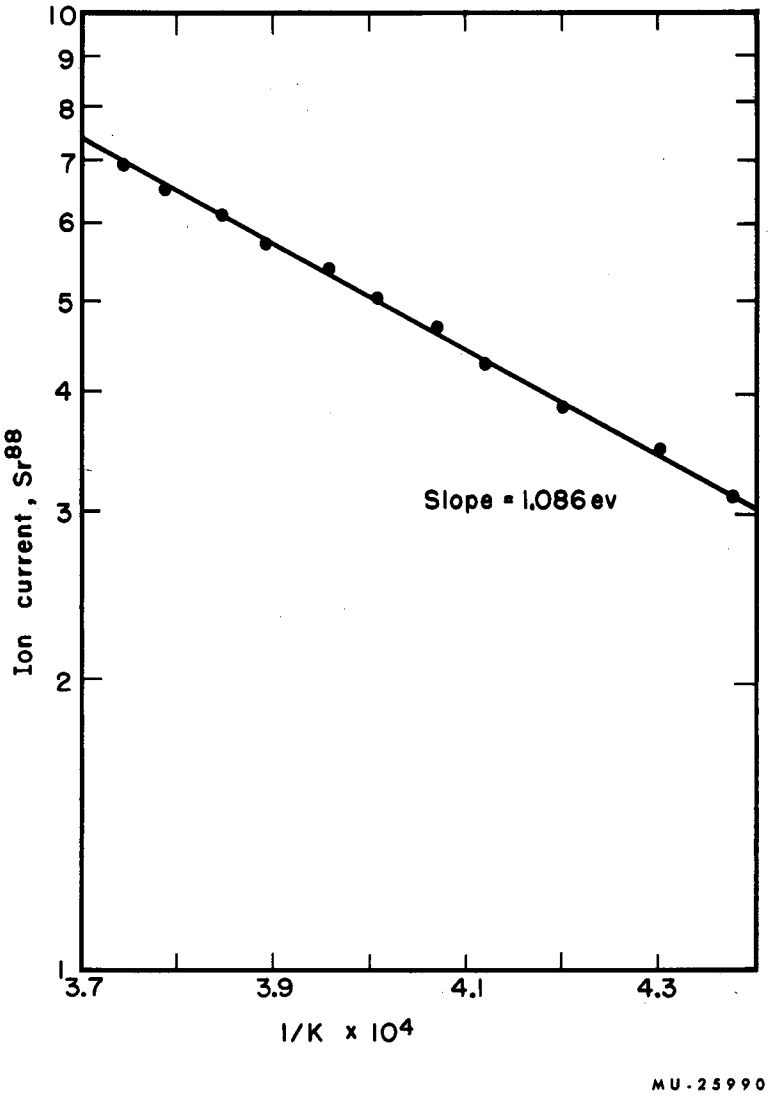

Fig. 10. Typical Saha-Langmuir plot of the ion current against reciprocal filament temperatures for strontium on "bulk" tungsten.