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ABSTRACT

The ionization of Sr and La was studied on the (110), (100) crystal surfaces of W and on polycrystalline W surfaces. It was shown that differences amounting to as much as 0.25 eV occur in the value of the surface work function when comparing Sr or La on the same surface. The work function of the $W_{(110)}$ plane was found to be 5.52 ± 0.05 eV. The value found for the $W_{(100)}$ plane is 4.56 ± 0.04 eV. Trace amounts of oxygen attack the $W_{(110)}$ crystal surface at elevated temperatures, exposing other planes and permanently reducing the work function of this surface.

INTRODUCTION

Surface ionization has been studied by various techniques for over 40 years. Most of the earlier work was done with alkali metals on tungsten. More recently, considerable studies have been made using other elements and employing single-crystal surfaces. Surface ionization has potential possibilities for studies of chemical reactions at the surface of heated metals and for determination of first ionization potentials, and offers a sensitive technique for the study of adsorption and desorption of gases on hot metal surfaces.

The mechanism involved in the process has received far less study. Only recently has there been research effort toward studies on adsorption lifetimes of atoms or ions on hot metal surfaces^{1,2} or studies of absolute efficiency in converting atoms to ions. Considerable experimental evidence seems to indicate that the theory, which worked well with the alkali metals, fails to account for results obtained with some other elements.³ Comparison with theory and with elements having high first-ionization potentials is often complicated or obstructed by existing experimental conditions that contribute to the difficulty of obtaining good experimental data. Among these, the following may be mentioned:

1. It is quite difficult to prepare a uniform single crystal surface of a refractory metal and to fabricate it into filament dimensions. Very often the surface can be damaged mechanically, thermally, or chemically and no longer represents a real crystal plane of known orientation.
2. The single-crystal surface can also be subject to change during the course of the experiment. The surface can be etched by introduced gases, adsorb other elements as impurities, be contaminated by impurities diffusing

to the surface from within the metal. The real sensitivity of these impurities to research on surface ionization has thus far not received adequate study. Most "pure" metals are notoriously "dirty". Certainly the refractory metals are no exception. For example, we have never been able to rid tungsten filaments, single or polycrystalline, of potassium. This is probably also true of carbon and traces of fluoride compounds. The sensitivity of the surface-ionization mechanism to impurities is evident from the fact that often plots of the logarithm of the ion current against $1/T$ do not result in a linear plot, even at elevated temperatures as predicted from theory.

3. Exterior to the experimental metal surface is the vacuum chamber, the necessary supporting structure, and, in our experiments, the residual atmosphere of the mass spectrometer at a working pressure of 10^{-9} Torr. All these sources can contribute impurities. To date we have looked at some effects of oxygen on the surface-ionization process, but there are distinct possibilities that the results are also sensitive to small amounts of F, CO, CO_2 , H_2O , and H_2 throughout some part of the experimental temperature range of 1000 to 3000° K. No ultrahigh vacuum system is completely free of these gases, yet few studies have been made relative to their effect on the surface-ionization process.

4. Two other conditions, in the form of corrections, have received little study with the surface-ionization process. These corrections are a result of thermally excited states, and the possibility of the partial reflection of atomic beams at the surface. Failure of the method to comply with theory is often thought to result from non-equilibrium conditions of atoms and ions on the metal surface.

In this work, Sr and La are impinged on single-crystal tungsten filaments oriented so that the filament surface is in the (110) or (100) plane. Similar beams are also employed on polycrystalline tungsten. The work-function changes of these surfaces were ascertained under various experimental conditions, or the same single-crystal surface was compared using the atomic beams of Sr and La.

EXPERIMENTAL

The mass spectrometer was described briefly in an earlier publication.⁴ No description has been published concerning recent changes to the source construction for surface-ionization work. The source housing is a 28-cm-diam stainless cylinder, equipped with a 10-cm-diam window normal to the axis of the beam tube and an additional 5-cm-diam window for pyrometer viewing. The entire exterior housing can be baked to 400° C using a specially formed Glas-Col heater. Gases may be introduced to the source region, controlled from a Granville-Phillips leak valve.

The ion source proper has gone through several development changes. The present source allows three filaments to be moved and aligned with either of two atomic beams coming from separate ovens. Each oven has a magnetic shutter for beam cutoff. Both ovens and shutters are small and of novel design. The ovens are made from 0.1-cm-o.d. Ta seamless tubing having a 0.0025-cm wall. A 0.004-cm-diam hole is drilled in the middle of a 2.5-cm length of tubing by spark-erosion methods using a tungsten tool pointed by electroetching. The tantalum tube is preheated and outgassed in a separate vacuum system and loaded with the sample in a dry nitrogen atmosphere. The

tube is then flattened at each end and spot-welded to suitable leads. The 0.004-cm hole is temporarily plugged with a small amount of collodion dissolved in alcohol-ether mixture to prevent air contact during transfer operations. This material burns off on first heating in vacuum and opens the oven hole.

The shutters are made as shown in Fig. 1. A sliding nickel tube is magnetically moved to provide the shutter action. Dimensions are kept small, and the shutter action is rapid. The shutter is operated from a condenser-discharge supply.

The entire source is shown schematically in Fig. 2. The source high-voltage plates are mounted on a machined boron nitride cylinder which rigidly holds the filament mounting plate. There is no position change in the slit plates when the various filaments are shifted into alignment.

Receiver detection employs a 16-stage electron multiplier, the output going to an electrometer and a recorder system.

The surface-ionization process was studied using atomic beams of Sr and La on single-crystal tungsten surfaces. The choice of these two elements was made for the following reasons: Both elements have nearly the same first-ionization potential. One has no low-lying energy states below 4000 wave numbers, while La has a number of energy states below this level. From previous observations, La had not given results predictable from theory or from comparison surface-ion experiments. The first-ionization potentials of both elements are assumed to be well-established from optical spectrographic work⁵: La = 5.61 eV and Sr = 5.69 eV.

An index of the experimental results on these surfaces is reflected in the calculated work function from the experimental data using the

Saha-Langmuir (SL) equation.⁶ As modified by Dobretsov,⁷ this equation is

$$\alpha = \frac{n_+}{n_0} = \frac{Q_+}{Q_0} \exp[e(\phi - I)/kT]$$

where

(5)

$$\frac{Q_+}{Q_0} = \frac{g_+}{g_0} + \frac{\sum_j g_+^j \exp(-E_+^j/kT)}{\sum_j g_0^j \exp(-E_0^j/kT)},$$

and α is the ratio of ions to neutrals. The ratio Q_+/Q_0 is the statistical weights g_+/g_0 and the sum of the j th excitation level of energies E^j , ϕ is the surface work function, I is the first atomic ionization potential, and e/k and T have their usual meanings.

In most experiments the experimental data are obtained by measuring the slope from a plot of the logarithm of the ion current, which is proportional to n_+ , against $1/T$ in $^{\circ}K$. From the slope, ϕ can easily be obtained. Deviations from linear plots are a sensitive experimental index for elements whose ionization potential is a volt or so greater than the surface work function. With elements close to or less than the surface work function, the results are far less sensitive to surface conditions. The term n_0 in Eq. (1) is assumed to be much greater than n_+ and is essentially constant in these experiments.

Emission from the (110) Plane

From a previously determined value of $\phi_{(110)}$ the ionization of Sr on this surface of W should result in a SL plot having essentially no slope. The value previously found was 5.41 ± 0.04 eV.³ In a series of further determinations on subsequent single crystals, the average of 22 runs with three

separate (110) tungsten surfaces gave $\phi = 5.52 \pm .05$ eV. In checking the back-reflected Laue pictures of the previous filaments, it was concluded that the original could have been 2 deg from a true (110) orientation. The present runs were made with better crystals.

In the absence of oxygen, and on a clean $W_{(110)}$ surface, Sr is converted to Sr ions at a temperature above 1650° K, with a very short residence time. The SL plot remains a straight line down to such temperatures that nonequilibrium conditions exist, and the Sr metal begins to coat the surface of the filament.

Most interesting is the result when trace amounts of oxygen are present. Visual and x-ray observations of the filament indicate that small amounts of oxygen on the (110) hot surface attacks the surface. With time, the high-temperature SL plot indicates a lowering of the work function from 5.52 to 5.30 eV. Apparently with this etching process other planes are being exposed. Also, with oxygen there is a rise in the Sr ion emission at temperatures around 1700° K. Here the reaction is different from the mechanism postulated by Weiershausen⁸ for the behavior of Sm on W. At no time was it possible to detect SrO^+ , but the lower-temperature portion of the curve has a maximum as found by Weiershausen for Sm and by Werning for Ba on polycrystalline W.⁹ The presence of oxygen apparently either increased the surface work function or changed the residence time caused by a new chemical species on the hot metal surface. Figure 3 shows the behavior of Sr in the presence of trace amounts of oxygen, plotting ⁸⁸Sr ion current against $1/T$. The extrapolated line indicates the behavior under clean metal, no oxygen, conditions. From the work of Hopkins¹⁰ it is postulated that the concentration of oxygen present in these experiments would not increase the work function. It seems more likely that another mechanism is present in the production of the low-temperature Sr

emission peak than can be explained by the increase in work function alone. Werning also came to this conclusion with studies of Ba on polycrystalline tungsten.⁸

Additional evidence of surface changes was noted in the presence of small amounts of oxygen. The time to reach an equilibrium ion current was greatly increased, especially between 1600 and 2200° K with oxygen present. When the oxygen was removed, the response time was very rapid with Sr at any temperature above 1650° K. Also, when small amounts of oxygen were introduced to a (110) filament at 2500° K, there was an immediate slight reduction in beam intensity rather than a rise, as would be expected from an increase in surface work function. Back-reflected Laue x-ray spots of this filament were not sharp, and microscopic examination showed the surface to be etched. No change in surface structure of the $W_{(110)}$ filaments had previously been noted after long heating under good vacuum conditions in the mass spectrometer. A $W_{(110)}$ filament was heated in a separate vacuum system at 2300° K for 125 hours. The system pressure was only 6×10^{-7} Torr, and the heating current was dc. A Laue pattern of this filament showed no change from its original single-crystal sharpness. Here the amount of oxygen was at a minimum, the residual gases being mostly H_2 , CO, CO_2 , and H_2O .

The apparent chemical attack of oxygen on $W_{(110)}$ with subsequent lowering of ϕ was experimentally disturbing. From a theoretical viewpoint, if both ϕ and I are about equal in magnitude, the introduction of oxygen should not change the Sr ion intensity, since $W_{(110)}$ is already close to peak efficiency for converting Sr atoms to ions. However, with the partial destroying of the (110) surface and the exposure of new planes having a

lower ϕ , this effect of oxygen was impossible to study. With this filament, lower temperature data showed a rise in the Sr emission. It is difficult to explain this rise as a result of a reversal in surface work function.

Lanthanum studies on the $W_{(110)}$ plane were not carried out at low temperatures, but the high temperature results seem to be in fair agreement with the Sr data indicating a work function of 5.44 ± 0.05 eV. Below 2300° K, La shows considerable residence time on this surface, making data-taking tedious. At 2100° K, the time for one-half peak intensity is about 18 sec, and a burst of La ions resulted upon suddenly flashing filament to the higher temperatures.

Emission from the (100) Plane

The (100) plane of tungsten is very sensitive to emission anomalies. Long after the (110) and polycrystalline surfaces gave a straight line SL plot with Sr, the (100) surface retained a plateau in the emission curve as shown in Fig. 4. It would be logical to assume that traces of oxygen produced this result, yet no such characteristic plateau appeared with the two other filament orientations. This plateau continued for many days of data taking. After about 60 days the data resolved into two straight-line plots, with a slight plateau separating the two curves. The high-temperature slope gave $\phi = 4.52$ eV, the other lower-temperature plot about 5.07 eV. Both the (110) and the (100) filaments were cut from the same crystal stock. At present we have no reasonable explanation for the shape of these curves.

Initial runs with La on $W_{(100)}$ surface gave a work function of 4.69 eV. These were all high-temperature plots between 2900 and 2500° K. At 2500° K the points were beginning to rise above the normal SL slope. In most runs,

the lower temperature data for La was below the expected SL slope. With subsequent experiments involving the $W_{(100)}$ plane and La, there were conflicting results. These results were for a new $W_{(100)}$ filament. Initial work functions on this surface gave the same answer as above, $\phi = 4.60 \pm 0.03$ eV, but some 25 determinations later, this surface seemed to have stabilized on a surface work function of 4.80 ± 0.03 eV. Although comparisons between Sr and La on the same surface will be discussed later, it is of interest to note here that with this same filament surface, Sr gave a work function of 4.56 ± 0.04 eV. All data were taken at high filament temperatures--2850 to 2500° K. The La oven had to be heated very hot--an estimated 1800° K--in order to obtain sufficient beam. Indications that some oxygen was present were obtained from the shape of the La plots after several hours of oven heating, but neither the pressure measurements or the Sr runs on the same filament were sensitive to this oxygen contamination. This high ϕ value was obtained over a period of days. In looking for a reason for this difference in ϕ , let us assume for the moment that both the answer for La and that for Sr are correct, and that the difference of 0.24 eV has meaning. The low-lying energy states of both the La ion and the La atom, could perhaps account for this difference. After we calculated all the possible energy states below 4121 wave numbers and summed the data over the experimental temperature range, this approach could only account for a correction of 0.01 eV. This correction is obtained by writing the SL equation for two different experimental temperatures and subtracting to get

$$\ln n_2^+ - \ln n_1^+ = \ln\left(\frac{Q_+}{Q_0}\right)_2 - \ln\left(\frac{Q_+}{Q_0}\right)_1 + \frac{e(\phi-I)}{k} \cdot \left(\frac{1}{T_2} - \frac{1}{T_1}\right)$$

$$\phi-I = \frac{k \ln n_2^+/n_1^+}{e\left(\frac{1}{T_2} - \frac{1}{T_1}\right)} + \frac{k \ln[(Q_+/Q_0)_1/(Q_+/Q_0)_2]}{e\left(\frac{1}{T_2} - \frac{1}{T_1}\right)}$$

The second term on the right gives the correction in eV.

Efficiency Measurements on Single Crystal Surfaces

A comparison experiment was done using two different crystal planes with Sr as the same beam element and then compared to the theoretical efficiencies calculated from the SL relationship. Both filament crystal surfaces were at 2500° K, and the (110) and (100) tungsten planes were used. A series of relative intensity measurements is shown in Table I.

Table I. Relative intensity measurements in the (110) and (100) planes.

<u>(110)</u>	<u>(100)</u>	<u>Ratio</u>
15300	325	47
14200	230	61
15300	360	42
15000	282	53
13640	212	64

These two planes gave work function values $\phi_{(110)} = 5.53 \pm .03$ and $\phi_{(100)} = 4.62 \pm .04$ eV. Based on these values, and the emission spectra value for the first ionization potential of Sr, the calculated efficiencies for the (110) surface at 2500° K should be 32.27%, and the (100) surface 0.69%. Thus, their ratio equals 46.76. The experimental ratios from the five runs in

Table I show considerable spread, but the average ratio is 53. This demonstrates the gain in ion emission that can be obtained from a tungsten filament having an increase in ϕ of 0.9 eV, and that experimental results at least approximate the value obtained from calculations using the SL equation.

Polycrystalline Surfaces

The term "polycrystalline surface" must be qualified, because most tungsten-ribbon filaments upon heating recrystallize, forming an exposed ribbon surface near the (113) plane. Work-function data from many laboratories show such filaments to be remarkably uniform considering the diversity of the filament sources. Most workers have found values near 4.60 eV for the polycrystalline W filaments considered.¹¹ The multitude of experimental runs made during these investigations with Sr gave results very close to the above value.

The polycrystalline filaments were compared either with the same element, or where experimental conditions permitted, with both Sr and La atomic beams. Early data on these surfaces with Sr often showed a hysteresis effect, with about 0.08 eV difference in slope between increasing filament temperatures and decreasing temperatures. With time this disappeared. Most data was taken by flashing the filament before each data point, however, after long heating periods the same results were obtained whether flashing was or was not done. After many days, a stabilized result could be obtained with both filaments agreeing within the range of 4.60 ± 0.02 eV.

Up to this time the oven containing La had not been fired to very high temperatures. Early runs using the La beam resulted in SL plots as indicated by Fig. 5. Under the same experimental conditions, Sr produced

a straight-line SL plot having the above work function. It was also noted that when both La and Sr beams occur on the hot filament simultaneously, the La curve gives a more linear SL plot. This seemed to indicate that Sr acted as a getter for some impurity. These results suggest that the hot La oven was a source of oxygen or other unknown impurities. Also the slope of the La plot indicated a work function near 4.86 eV, which is high for this surface. After one of these La runs, the same filament was studied using a Sr beam. Initially, the work-function data with Sr showed the surface to be 4.78 eV. Within a 30-min period, the slope gradually increased, lowering the work function to 4.58 eV, which is representative of values for this surface.

Work-functions for alternate La and Sr runs on a polycrystalline filament are shown in Table II. All data are between 2800 and 2400° K.

Table II. Work functions for La and Sr

<u>La</u>	<u>Sr</u>
4.75	4.51
4.77	4.51
4.75	4.50
4.85	4.52
4.76	4.49
4.88	4.57

Values are the surface work function in eV. It is interesting to note that about the same difference appears between La and Sr as with the work on single crystal surfaces. During these runs, no evidence of LaO^+ could be found until severe outgassing raised the source pressure to 10^{-7} Torr.

In another series of experiments involving Sr on polycrystalline W, in the low-temperature region the filament was cleaned by flashing at 2700° K at a pressure of about 10^{-8} Torr. After flashing, the temperature of this filament was reduced to 1940° K and the Sr shutter opened. A delay period of minutes was observed for the emission to reach maximum. If reflashd at 2700° K and reduced to 1940° K to soak for the same delay time, the Sr would reach maximum emission instantly upon opening the shutter. At this point one might explain this result by assuming adsorption of a residual gas component, with a subsequent increase in the work function of the metal surface. However, if the filament was flashed at 2700° K and then turned completely off for an extended period, the same delay remained upon again heating at 1940° K and subjecting it to the Sr sample. This seemed to indicate that the residual gas was not the contributing adsorption factor. The residence time of Sr atoms on this surface was not involved, since closing the shutter at any stage would cut the beam to zero very rapidly. If the filament was kept at 1940° K and was not flashed at any time, the maximum emission was reached with no time delay. This seems to point to the contamination as coming from the metal filament itself. At high temperatures the diffusion rate is high, but the adsorbed material leaves the surface instantly. At the 1940° K temperature, the diffusion rate is slow and enhancement of the Sr emission builds up as coverage increases. Certainly the full explanation of such behavior could occupy a great amount of experimental time; at the moment it is presented only as an observation of the complexities of the surface-ionization process.

SUMMARY

When the surface work function ϕ is used as an index parameter, the surface ionization of Sr and La on $W_{(110)}$ or on $W_{(100)}$ surfaces does not always give the same value for ϕ . It was shown that these differences amount to as much as 0.25 eV. Full reason for this divergence is not known, but some attempts are made to point out various experimental conditions that influence the data.

Agreement between Sr and La on $W_{(110)}$ is fair, $5.52 \pm .05$ to $5.44 \pm .05$ eV respectively. The $W_{(100)}$ experiments gave mixed results, which could be caused by experimental conditions rather than any fundamental reason. With the polycrystalline surface, there is a distinct nonagreement between the Sr (4.60 eV) and La (4.86 eV). This variation cannot be accounted for from the low-lying excited states of the La ion or atom.

The work function of the $W_{(110)}$ plane was redetermined and found to be $5.52 \pm .05$ eV. The value found for the $W_{(100)}$ plane is $4.56 \pm .04$ eV.

Small amounts of oxygen attack the $W_{(110)}$ crystal surface at elevated temperatures, apparently exposing other planes of lower work function and reducing the work function permanently from 5.52 eV to 5.30 eV in these experiments.

While not completely proven, it seems from these data that the low-temperature rise in emission is not entirely because of an increase in ϕ . It is thought that a compound is formed whose residence on the surface differs from that of the metal atom.

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FOOTNOTE AND REFERENCES

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FIGURE LEGENDS

Fig. 1. Sketch of the shutter design. 1. Beam path, 2. copper wire, 3. nickel tube, 4. fired alumina tube, and 5. electrical lead.

Fig. 2. Schematic drawing of the ion source showing 1. filament mounting plate, 2. drawing-out plate, 3. split-focus plate, 4. shutter mounting plate, 5. shutter position, 6. oven position, 7. final slit plate, 8. boron nitride cylinder, and 9. three-filament positioner.

Fig. 3. Strontium on $W_{(110)}$. Circle plot shows the slope of the emission before introducing oxygen and final adsorption of Sr at low temperatures. Triangle plot is the permanent final slope after oxygen treatment and the effect of oxygen on the emission at low temperatures. If the oxygen is removed, the permanent slope will continue, as indicated by the broken line.

Fig. 4. Strontium on $W_{(100)}$, showing the plateau effect.

Fig. 5. Typical first-run Saha-Langmuir (SL) plot for La, showing no linear SL relationship at any temperature.

XBL 685 - 27 23

Fig. 1

XBL685 - 2724

Fig. 2

XBL685-2725

Fig. 3

XBL 685 - 2726

Fig. 4

XBL685-2727

Fig. 5

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