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UNIFORM WORK FUNCTION CATHODE STUDIES
FOR THERMIONIC CONVERTERS

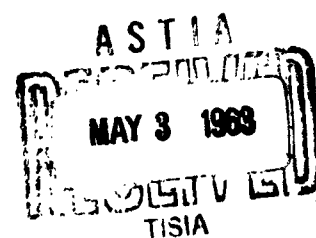
THIRD QUARTERLY REPORT

APRIL 1963

AERONAUTICAL SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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(PREPARED UNDER CONTRACT NO. AF 33(657)-8728 BY ATOMICS INTERNATIONAL,
A DIVISION OF NORTH AMERICAN AVIATION, INC., CANOGA PARK, CALIFORNIA;
M. N. HUBERMAN AND R. A. MOHR, AUTHORS.)

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FOREWORD

The work reported here was performed by Atomics International, A Division of North American Aviation, Inc., under the sponsorship of the Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

This report describes the progress made during the third quarter (12 January 1963 to 12 April 1963) of a program to study the improvement of uniform work functions of thermionic converter emitters under Contract No. AF33(657)-8726. The study program is under the technical supervision of M.N. Huberman of the Thermionics and Thermoelectrics Department of Atomics International.

ACKNOWLEDGMENTS

The development of techniques of chemical vapor deposition used in this program was performed at San Fernando Laboratories under the direction of R. Holzl. The x-ray measurements were made by W. Korst and K. Miller. C. C. Weeks, E. V. Clark, H. Hori, and R. Riccio contributed to the design and construction of experimental apparatus.

The guidance and interest of L. E. Porter of the United States Air Force Aeronautical Systems Division is gratefully acknowledged.

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ABSTRACT

X-ray analysis of vapor deposited molybdenum shows that either (111) or (100) preferred orientations can be obtained. Further work indicates that precise control of impurities is needed before reproducible results can be obtained. Cathodes having (111) orientations have been produced using vapor deposition techniques.

Methods of polishing cathode surfaces have been studied. X-ray studies show that heat treatment is adequate to restore the preferred orientation at a mechanically polished surface. The reproducibility of performance of test diodes has been established by comparison of current data with data from previous work.

Construction of an emission electron microscope for cathode work function distribution studies is in progress. Preliminary work has been done with an electron mirror microscope.

I. INTRODUCTION

The general purpose of this study program is to improve the efficiency of thermionic converters which have nonplanar geometries. Specifically, the investigation concerns the production of nonplanar cathodes having more uniform work functions and surface characteristics than are obtained with standard cathode fabrication techniques. This report describes progress of the study from January 12, 1963 to April 12, 1963.

Since the work function of a substance will depend on its crystallographic orientation, the most obvious way to obtain a uniform work function on a plane surface would be to use a single crystal. In the present study, uniform work functions must be obtained on nonplanar surfaces and single crystals are not suitable for this purpose. Instead, methods can be developed to obtain a small grained structure in which the grains have a preferred crystallographic orientation with respect to the cathode surface. The program then falls naturally into two phases: 1) the production of preferred orientations on cathodes of arbitrary nonplanar geometries, and 2) measurements of the uniformity of work function obtained with preferred grain orientations.

For the first phase, the potentialities of chemical vapor deposition of molybdenum were investigated. The previous quarterly report¹ described chemical vapor deposition techniques which produced (111) and (100) fiber orientations in thin deposited layers of molybdenum. In the present report the x-ray analysis of all samples and cathodes produced to date is summarized.

After preferred orientation depositions were obtained, alternate methods of polishing the cathode surfaces were studied. Results obtained this quarter indicate that mechanical polishing followed by annealing will be adequate.

Work on the second phase, the problem of determining the nature of the work function distributions obtained, has been continued through the third quarter. Two approaches have been considered. In the first approach performance characteristics of thermionic diodes having standard arc cast cathodes can be compared with the performance of diodes having vapor deposited cathodes. Performance characteristics of thermionic diodes with standard arc cast cathodes have been studied in this quarter. In the second approach the detailed work function distributions of preferred orientation surfaces can be studied by more direct means, such as a mirror microscope

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or an emission electron microscope. Ultimately the two approaches can be correlated by showing that: 1) existence of preferred orientation results in uniformity of work function and, 2) uniformity of work function produces changes in performance characteristics of thermionic diodes. In this quarter the reproducibility of diode performance has been established, designs have been completed and construction begun of an emission electron microscope, and preliminary experiments have been carried out with an electron mirror microscope.

II. SAMPLE ANALYSIS

TEST SAMPLES

Previous results showed that the pentachloride pyrolysis samples exhibited (100) fiber orientation while more recent data shows that (111) is also possible. The hexafluoride reduction samples had (111) orientations for the most part with some (100) orientations also occurring. For the hexafluoride samples, correlations between deposition conditions and resultant orientations have been sought. Table I lists the hexafluoride samples in order of decreasing (111) orientation. The hexafluoride samples having strong (100) orientations are listed in Table II in order of decreasing degree of orientation.

TABLE I
HEXAFLUORIDE REDUCTION SAMPLES
(in order of decreasing (111) orientation)

Sample No.	Deposition Rate (mils/min)	Deposition Temperature (°C)	Thickness (mils)
15A	1.6	725	10
13	1.7	850	10
16B	0.6	725	10
7	1.8	850	30
15B	1.6	725	10
5	1.8	900-925	10
8	1.1	750	30
6	0.6	700-750	10
10	1.5	800-850	10
2	0.4	700	7-8
3	0.2	800	3-1/2 - 4-1/2
9	1.5	800-850	10
1	0.5	750	11
11	0.9	700-750	10
16A	0.6	725	10
4	1.5	850	10
2	0.4	700	7-8
5	1.8	900-925	10
25	0.8	800-850	8

TABLE II

HEXAFLUORIDE REDUCTION SAMPLES
(in order of decreasing (100) orientation)

Sample No.	Deposition Rate (mils/min)	Deposition Temperature (°C)	Thickness (mils)
4	1.5	850	10
25	0.8	800-850	8
11	0.9	700-750	10
16A	0.6	725	10

Although there is considerable scatter to the data, there is a trend towards greater degrees of (111) orientation at higher deposition rates. There appears to be no correlating factor for the (100) orientations. Other work indicates that impurities can play a major role in determining the growth habits of vapor depositions.² Vacuums of the order of 10^{-10} torr would be needed to eliminate such effects. The conclusion at this time is that vapor deposition does produce preferred orientations of crystallographic planes parallel to the deposition surface. Both (111) and (100) orientations have been produced; however, close control of impurities present during deposition will have to be established before the mechanisms can be understood.

CATHODES

Six vapor deposited cylindrical cathodes have been produced. They were produced by first machining undersized arc cast molybdenum cathodes and then vapor depositing out to larger than normal cathode dimensions to allow for final polishing down to the close tolerances needed for thermionic diodes. In their present state their surfaces have deposited layers with a thickness of 10 mils, 5 mils of which will be machined and polished off before their use in diodes.

Cathodes 4C, 5C and 6C were produced by hexafluoride reduction while 7C, 8C and 9C were prepared by pentachloride pyrolysis. A smaller deposition chamber than previously used was employed to obtain a cleaner system.

The x-ray data for five of these cathodes is shown in Table III. The method of x-ray analysis has been discussed in the previous report.¹ Table III also includes, for purposes of comparison, the relative intensities for a random orientation. A satisfactory diffraction pattern could not be obtained for 7C because of

TABLE III
X-RAY ANALYSIS OF VAPOR DEPOSITED CATHODES

Cathode No.	hkl	Position *			Cathode No.	hkl	$\frac{I_{hkl}}{\sum I_{hkl}}$ [†]	Position *			$\frac{I_{hkl}}{\sum I_{hkl}}$ [†]
		1	2	3				1	2	3	
4C	110	7	6	24	6C § (Cont.)	220	0.121	0	0	0	0.000
	200	3	0	1		310	0.013	0	0	0	0.000
	211	20	30	66		222	0.378	19	8	94	0.306
	220	0	0	5	8C	110	0.016	1	1	0	0.007
	310	0	0	1		200	0.003	0	0	0	0.000
	222	15	9	120		211	0.469	20	13	18	0.185
5C	110	2	8	4	9C	220	0.099	0	0	0	0.000
	200	18	0	24		310	0.296	0	0	0	0.000
	211	6	24	25		222	0.387	95	65	62	0.807
	220	0	9	2		110	0.077	2	4	33	0.241
	310	0	3	0		200	0.021	0	0	0	0.000
	222	6	1	10		211	0.120	10	42	40	0.568
6C §	110	0	2	2	3/8 in. diameter random	310	0.011	0	0	0	0.000
	200	0	0	0		222	0.000	15	10	5	0.185
	211	27	63	9		110	0.275				0.628
	220	0	0	0		200	0.000				0.116
	310	0	0	0		211	0.000				0.165
	222	62	10	185		220	0.715				0.025
6C §	110	3	0	0		310	0.008				0.058
	200	1	0	0		222	0.003				0.008
	211	200	29	42			0.684				

*For each sample the different positions correspond to approximately 120° rotations around the cathode axis.

†Averaged over all 3 positions.

§Data from independent measurements taken several weeks apart to check reproducibility of data.

its surface roughness. Another attempt to x-ray it will be made after machining and annealing.

All samples had strong (222) components in their diffraction patterns indicating (111) preferred orientations. For a random orientation the (222) component is only .008 of the total intensity. The (211) components were also stronger than would be obtained for a random orientation although the effect was not nearly as pronounced as for the (222) lines.

The high intensity of the (211) is probably due to the fact that the angle between the (111) and (211) planes is only $19^{\circ}28'$ in the cubic system. The x-ray reflections are detected from a finite arc on the cylindrical sample surface and the reflecting plane is truly tangent to the deposition surface at only one line parallel to the cylindrical axis. A second factor is that the oriented crystallites are not all aligned with their (111) planes perfectly parallel to the surface. These two factors combine to give an angular spread of (111) planes relative to the reflecting plane. It is shown in Table IV that, of the planes observed, the (211) planes are closest to the (111) plane and consequently would be the first to reflect.

TABLE IV
ANGLES BETWEEN (111) AND OTHER CRYSTALLOGRAPHIC
PLANES IN THE CUBIC SYSTEM

hkl	Values of angles between (111) and (hkl) planes
100	$54^{\circ} 44'$
110	$35^{\circ} 16'$, 90°
211	$19^{\circ} 28'$, $61^{\circ} 52'$, 90°
310	$43^{\circ} 6'$, $68^{\circ} 35'$

The two results listed in Table III for cathode 6C are for independent analyses taken several weeks apart to test the reproducibility of the data. The change in measured relative intensities for 6C indicates that the data should be interpreted as qualitative rather quantitative determinations of preferred orientation. It should be noted, however, that the conclusion that cathode 6C has a (111) orientation still holds for either set of data, since for a random orientation the ratio $I_{211}:I_{222}$ would be 165:8, which is much larger than the observed ratios.

Two factors that probably contribute to the lack of quantitative reproducibility are surface roughness and variations in degree of orientation over the sample surface. The data was taken with cathode surfaces in their "as deposited" condition, which, as mentioned in previous reports, is a coarse structure with protruding pyramidal grains. The detailed structure of the surface roughness can lead to angle-dependent x-ray absorption phenomena which in turn would affect the relative intensity of the Bragg reflected rays from different crystallographic planes. There is evidence however, as discussed in the section on surface preparation, which serves as a check on the adequacy of the rough surface analysis for qualitative determinations of preferred orientation.

Previously, chloride pyrolysis produced only (100) orientations. The cathode results show it can also produce (111) orientations. This leads to the interesting conclusion that either fluoride reduction or chloride pyrolysis can be used to obtain (100) or (111) orientations. To fully exploit this capability for either process, close control of deposition conditions and impurities will be needed.

OTHER SAMPLES

Several vapor deposited samples from a second supplier* were obtained for analysis. The results, presented in Table V, show a (100) fiber orientation on a flat copper substrate, a mixture of (111) and (211) on a flat rolled molybdenum substrate, and almost random orientations on cylindrical arc cast molybdenum rods. The process used for these depositions is proprietary and no interpretation of the data can be made.

*Semicon of California, Inc., Watsonville, California

TABLE V
X-RAY ANALYSIS OF ADDITIONAL VAPOR DEPOSITED SAMPLES

Sample Description	hkl	Positions			$\frac{I_{hkl}}{\Sigma I_{hkl}}$
		1	2	3	
Mo deposited on flat copper plate	110	3			0.026
	200	76			0.667
	211	1			0.009
	220	0			0.000
	310	34			0.298
	222	0			0.000
Mo deposited on flat Mo disk	110	4	10		0.051
	200	0	0		0.000
	211	100	110		0.772
	220	1	1		0.007
	310	1	1		0.007
	222	28	16		0.162
Mo deposited on flat Mo disk	110	30	30		0.176
	200	14	7		0.062
	211	100	95		0.572
	220	5	3		0.023
	310	12	13		0.073
	222	12	20		0.094
Mo deposited on arc cast Mo rod	110	62	22		0.316
	200	19	20		0.147
	211	30	31		0.229
	220	7	13		0.075
	310	24	33		0.214
	222	2	3		0.019
Mo deposited on cylindrical Mo cathode	110	200	250	200	0.537
	200	110	71	90	0.224
	211	41	45	40	0.104
	220	13	20	14	0.039
	310	40	30	31	0.083
	222	5	6	5	0.013

III. SURFACE PREPARATION

GENERAL APPROACH

For thermionic conversion applications, the cathode surfaces will have to be machined and polished without disturbing the preferred orientation of the depositions. Initially, three different polishing techniques were considered: mechanical, chemical, and electrolytic polishing. It was decided, however, that chemical polishing would be unsuitable since it is known that the quantity of material removed by this process cannot be controlled accurately enough to obtain the close tolerances needed for thermionic converters.

Mechanical Polishing

Mechanical polishing has the advantage of producing extremely smooth surfaces with no danger of etching to produce other crystallographic planes. It is known, however, that the polishing operation severely distorts the crystalline structure at the surface creating the so-called Beilby layer⁴.

After mechanical polishing, metal can be heated to recrystallize or evaporate the disturbed layer. An experiment was performed to determine the effects of polishing and subsequent heating of a vapor deposited specimen. The flat end of sample number 5, which initially had a (111) preferred orientation was sanded down with No. 400 silicon carbide paper. After sanding the sample had an almost random orientation at the surface with some preponderance of (111) still showing. The fact that the (111) plane was still stronger than completely random was probably due to a combination of the surface layer not being completely randomized and the fact that x-ray reflections were also seen from the undisturbed volume just beneath the surface.

The sample was then inductively heated to 1800°C in vacuum for one hour. Subsequent x-ray analysis showed the surface to have an extremely strong (111) orientation with no other diffraction lines being detected. The conclusion is that heat treatment does return the surface layer to its original preferred orientation. The x-ray data for this experiment is tabulated in Table VI, which also lists for comparison the intensities obtained for a randomly oriented sample.

TABLE VI
X-RAY DATA FOR MECHANICAL POLISHING STUDY

hkl	Initial Intensities	After Sanding	After Heating	Intensities for Random Orientation
110	5	85	0	76
200	0	9	0	14
211	8	23	0	20
220	1	5	0	3
310	1	7	0	7
222	19	14	70	1

The re-establishment of the preferred orientation at the surface is due to a combination of the effects of evaporation and recrystallization. The Beilby layer is of the order of $50\text{\AA}$ thick. Between the Beilby layer and the undistorted metal there is a transition zone of deformed metal of the order of 10 to 100 microns thick³.

At 1800°C approximately $1000\text{\AA}$ of molybdenum would have evaporated in one hour, which was more than adequate to remove the $50\text{\AA}$ Beilby layer. At the same time 1800°C was well above the temperature needed to recrystallize the remaining deformed metal.

A second conclusion can be drawn concerning the reliability of the rough surface x-ray data. The final flat surface data from the polishing experiment can be interpreted as confirmation of the (111) orientation originally obtained from the rough surface data. If the original finding of a (111) orientation had been incorrect it would have been highly improbable that a (111) orientation would have been found again after polishing and heating.

Electrolytic Polishing

Several experiments with electrolytic polishing were performed. Two different electropolishing solutions were tried. The polishing samples were molybdenum disks $3/4$ inch in diameter by $1/16$ inch thick. A similarly dimensioned Type 304 stainless steel disk was used as the cathode.

The first solution consisted of 5% sulphuric acid, 1.25% hydrofluoric acid, and 93.75% methyl alcohol. Cathode-anode spacings of 0.125 and 0.975 inch were tried. The results in each case were unsuccessful due to lack of an

adequate power supply. The highest input possible with the power supply used, 40 volts and 22 amperes, was still not sufficient to reach the polishing plateau of the volt-ampere curve.

A second solution, 12.5% sulphuric acid and 87.5% methyl alcohol was then used. After initial unsuccessful attempts with this solution, it was found that better results would be obtained if the stainless steel cathode was sanded clean to remove possible residues from previous polishing operations. With a cathode-anode spacing of 0.975 in. the polishing plateau was located at 22 volts input. Bright mirror finishes were then obtained with polishing times of the order of 20 seconds and solution temperatures of 60°F.

Microscopic examination, however, showed a rough microstructure that apparently was due to the failure of the electropolishing to remove the effects of the initial rough mechanical polishing. At this point the next logical step to take would have been to use finer mechanical polishing procedures prior to electropolishing. In view of the success of the mechanical polishing experiment at this time, however, further work along these lines was discontinued.

Electropolishing can be used if preceded by careful mechanical polishing. Its disadvantages are that extreme care must be used to avoid etching and that large currents of the order of 100 amperes will be needed to polish normal cathode surface areas. Its advantages are that precise control of the amount of material removed can be maintained and that it works equally well on nonplanar surfaces.

IV. DIODE TESTING

Diode characteristics were measured for a converter having a standard machined molybdenum cathode to serve as a standard for comparison with data obtained with vapor-deposited cathodes. The purpose of the measurements reported here was to check the reproducibility of diode characteristics, thus insuring that changes in diode characteristics for vapor-deposited cathodes are indeed due to work function changes rather than statistical fluctuations. The diode model used was a cylindrical diode, described previously⁵, that showed good reproducibility in a previous engineering study⁶ in which three identical diodes constructed from the same lot of raw materials was tested.

The data obtained is shown in Figures 1 through 3. These graphs also show for comparison data from reference 6 for an emitter temperature of 1800°C, a collector temperature of 750°C, and an optimized cesium reservoir temperature. The only serious discrepancy between the two sets of data arises from the fact that in the present study the measured optimum reservoir temperature is 360°C compared to 375°C for the previous study. This discrepancy probably is due to the fact that the upper surface of the cesium reservoir is in a region of high temperature gradient. This location of the reservoir makes it difficult to obtain an accurate determination of the temperature at the reservoir surface with the thermocouple externally positioned as in this experiment.

Agreement within 5% was found for the volt-ampere curve, the volt-power density curve, and the efficiency vs. voltage curves at 1800°C. It is then a safe conclusion to say that anything greater than a 10% change in performance for vapor deposited cathodes can be attributed to differences in the cathodes.

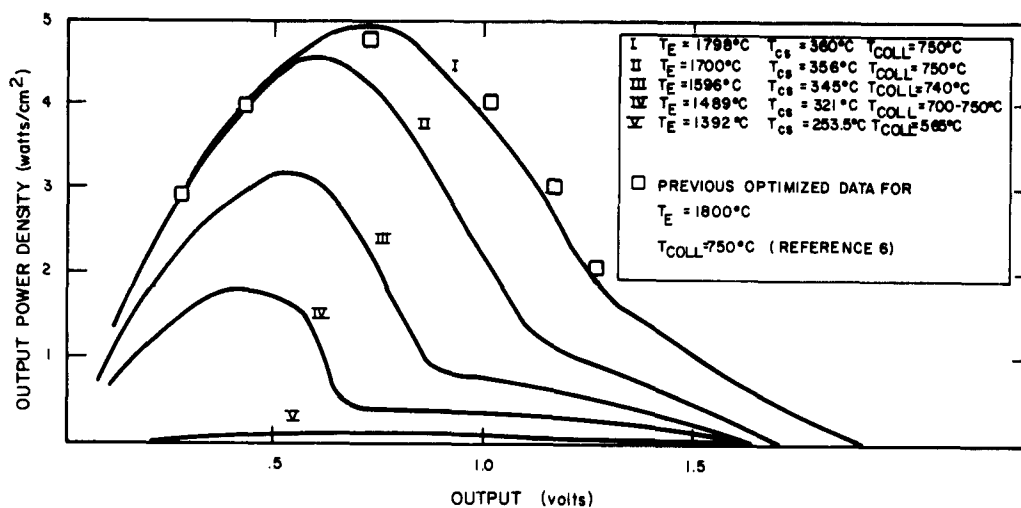


Figure 1. Output Power Density vs Voltage at Optimum Cs Reservoir Temperatures

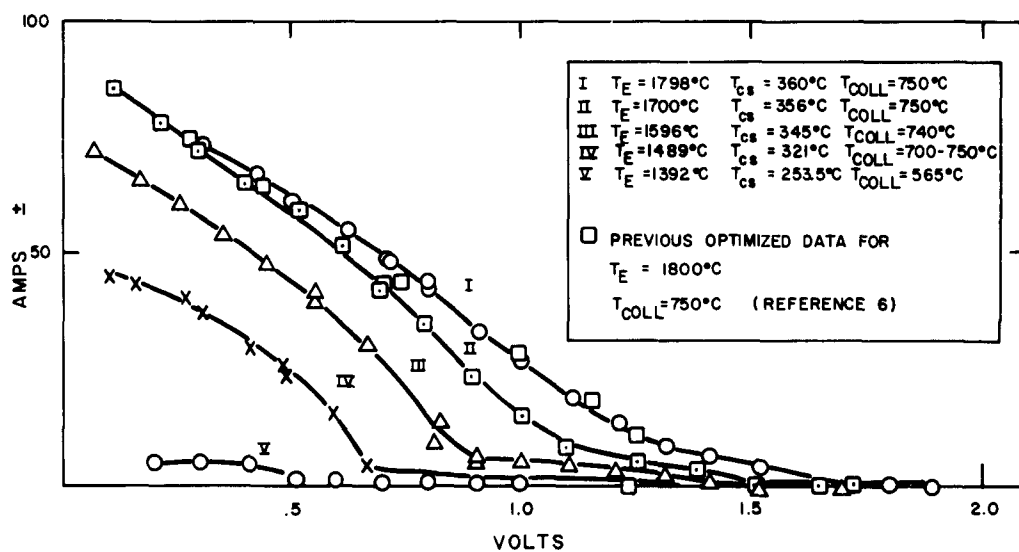


Figure 2. Volt-Ampere Curves at Optimum Cs Pressures

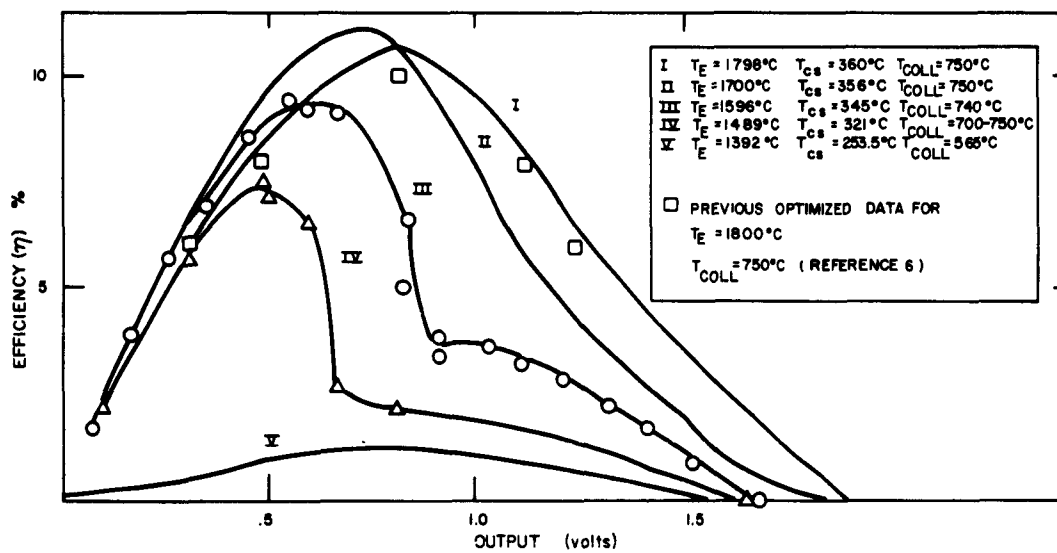


Figure 3. Efficiency vs Output Voltage at Optimum Cs Reservoir Temperatures

V. WORK FUNCTION STUDIES

EMISSION MICROSCOPE

An emission electron microscope is being constructed for use in studying the thermionic emission patterns and hence the work function distributions of cathode surfaces. The microscope incorporates a three-electrode cathode immersion lens of a type used in past studies of thermionic emission phenomena.⁷⁻⁹ Magnifications will be in the 100-200 range.

Flat samples will be used at first to determine if uniform work functions are attainable with vapor deposition, since it is a relatively simple matter to obtain a smooth finish on a flat surface. If curved surfaces were used first and the results unsuccessful the question would still exist as to whether the failure was due to surface preparation problems or to the fact that vapor deposition is not the answer to the problem. Once it is shown that uniform work functions are obtained on flat surfaces, later curved surface work can serve as a check on the adequacy of the method of surface polishing.

The present status of the microscope is as follows:

1. An electron bombardment cathode sample heater assembly has been built and tested.
2. Construction of the lens system is near completion.
3. A three-way movable mount for positioning the cathode inside the microscope has been designed and the machining of parts will begin within a week.
4. A glass tube and fluorescent screen is being assembled.
5. Single-crystal and vapor-deposited samples have been ordered.
6. The present schedule calls for assembly of the microscope by May 1. This will allow a month for troubleshooting and a month for taking data.

MIRROR MICROSCOPE

Preliminary pictures have been taken with a General Mills Electron Mirror Microscope.* In the mirror microscope the specimen is used as a negatively biased mirror to reflect a beam of high energy electrons. The reflected beam is

*This work was performed at Metal Control Laboratories, Huntington Park, California.

then focussed on a fluorescent screen to obtain a magnified image which can provide information about magnetic and electric fields at the surface of the specimen. In particular, variations in contact potential due to work function patches can lead to variations in the reflected intensity.

A vapor-deposited sample and a randomly oriented sample were examined with the microscope. Figure 4, the pattern obtained with the randomly oriented sample, shows that different reflected intensities are indeed obtained from adjacent grains. No information could be obtained however about the vapor deposited sample because of improper polishing of its surface. Figure 5 shows the effect observed. More carefully prepared samples will be examined in the coming quarter.

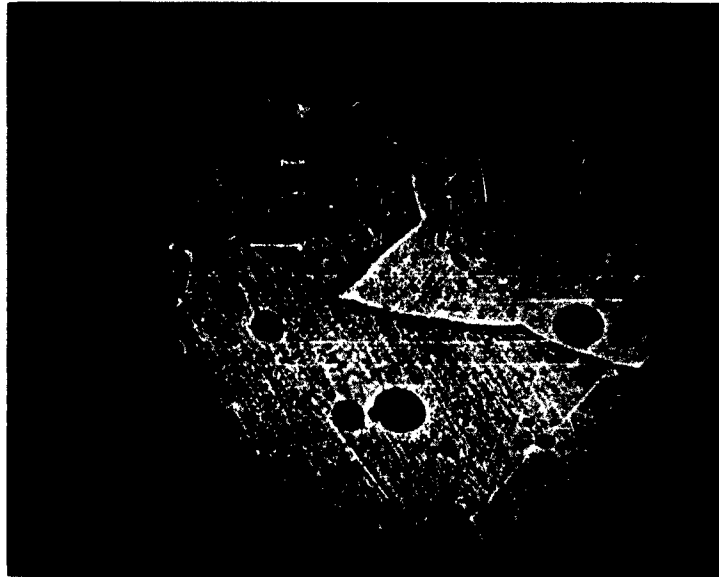


Figure 4. Electron Mirror Microscope Image
at Grain Boundary (Approx. 100x)



Figure 5. Effect Due to Faceting Caused by
Improper Polishing (Approx. 100x)

VI. SUMMARY OF PROGRESS

Progress made during this reporting period is summarized below.

- 1. A search was made for correlations in the sample x-ray data.**
- 2. Vapor deposited cathodes have been fabricated and x-rayed prior to final machining and polishing.**
- 3. Additional vapor deposited samples have been x-rayed.**
- 4. Surface polishing techniques have been studied.**
- 5. Diode data has been taken with a standard machined arc cast cathode.**
- 6. An electron bombardment cathode sample heater assembly for an emission microscope has been built and tested.**
- 7. The emission microscope lens system has been designed and construction is nearly complete.**
- 8. A three-way movable mount for positioning the cathode inside the microscope has been designed and the machining of parts will begin within a week.**
- 9. A glass tube and fluorescent screen is being assembled.**
- 10. Single crystal and vapor deposited samples for emission microscope studies have been ordered.**
- 11. Preliminary pictures of samples have been taken with an electron mirror microscope.**

VII. CONCLUSIONS

1. Both (111) and (100) orientations can be obtained with either chloride pyrolysis or fluoride reduction. Strict control of impurities and deposition conditions is needed for this to be fully exploited.
2. Mechanical polishing followed by heat treatment should be an adequate means of surface preparation. Heat treatment removes deformations in the metal structure and returns the surface layer to its original orientation.
3. Testing of a cylindrical thermionic diode with an arc cast cathode shows this type of diode to be reproducible to within 5% at an emitter temperature of 1800°C.
4. The mirror microscope shows some promise as a way of taking a quick qualitative look at patch distributions, although it will not provide the strong contrast expected with the emission microscope.

VIII. PROGRAM FOR THE FOURTH QUARTER

1. Complete fabrication and assembly of emission microscope by May 1. This will leave a month for trouble shooting and 1 month for sample analysis.
2. Produce single crystal, randomly oriented, and vapor deposited cathodes for emission microscope analysis.
3. Examine suitably prepared samples with the electron mirror microscope.

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