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TRANSMITTAL OF PROGRESS REPORT

Date 19 June 1963

TO: Lewis Research Center
Space Electric Power Office
21000 Brookpark Road
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Attn: John W.R. Creagh

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Very truly yours,

ELECTRO-OPTICAL SYSTEMS, INC.

Richard N. Strong

Richard N. Strong, Manager
Contract Administration

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Monthly Progress Report
19 April 1963 to 19 May 1963

RESEARCH PROGRAM RELATED TO VAPOR THERMIONIC CONVERTERS FOR NUCLEAR APPLICATION

Prepared for:

Lewis Research Center
Space Electric Power Office
21000 Brookpark Road
Cleveland 35, Ohio

Contract NAS 3-2529

EOS Report 3410-ML-6 17 June 1963

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CONTENTS

ILLUSTRATIONS

1. INTRODUCTION

This is the sixth monthly report of progress on Contract NAS 3-2529, a research program related to vapor thermionic converters for nuclear applications.

2. PROGRAM STATUS

2.1 Grain Growth Investigations in Cesium Vapor

The vac-ion pumped test chamber has been completed and testing will begin as soon as some minor difficulties with the oven heater are overcome.

2.2 Cesiated Emission Investigations

Experimental results have been obtained on the electron emission from cesiated molybdenum bar stock. The temperature range of the emitter was from approximately 600°C to 1500°C and the cesium reservoir temperature is being varied over the range from 290°C to 350°C. The measured emission densities are in the range from 0.2 amperes/cm^2 to 10 amperes/cm^2. Generally speaking, the emission at the high arrival rates follows the Langmuir-Taylor type of "S" curve. The minimum work function achievable on cesiated bar stock surface appears to be approximately 1.7 ev. The more detailed results and the time at temperature effects on the emission will be presented in the next report.

2.3 Electron Emission Microscope (Results of Molybdenum Bar Stock)

A sample of molybdenum bar stock (R3) was placed in the microscope and operated at 1500°C for 121 hours. The sample had received no preliminary heat treatment. At the end of the 121 hour run, the sample was raised to 1700°C for 1/2 hour and then to 1800°C for 1/2 hour. Although it will be some time before a detailed analysis of the results can be completed, some immediate conclusions can be drawn from the preliminary results. A selection of photographs of the electron emission image at various times in the run is shown in Figure 1 and Figure 2 in support of the following tentative conclusions:

1. Grain Growth - In the first 1-1/2 hours of heating at 1500°C, the average grain size increased to about .06 mm. At the termination of the run (121 hours total) the average grain size was about .07 to .08 mm. The sequence of pictures, (see Figure 1) shows some grains growing preferentially at the expenses of others which entirely disappear on the average. Those grains which remain at a given time have increased in size as direct examination of the photograph indicates.

It was observed that for a given temperature, most of the grain growth occurred during the initial time that temperature was first attained. Also, the rate of grain growth increases with temperature shown in Figure 2. After 1/2 hour at 1700°C, the average grain size had increased to .12 mm. After 1/2 hour more with the temperature increased to 1800°C, the average grain size has increased to .15 mm.

2. Surface Work Function - One of the major results obtained to date is the outstanding ability of the electron emission microscope to identify the relative work function of the various surface crystals on an emitter surface. The darkest areas in the photographs of the emitter image are the highest work function faces, the lightest areas are those of lowest work function, and the gray areas are those having intermediate values of work function. There is no doubt about the relative work function of surfaces or grains which are undergoing preferential growth and which grains have the same work function.

The fact that not only the grain size but the surface work function area distribution has obviously changed during 121 hour operation at 1500°C points primarily to one of two situations; (1) the operation of the emitter under cesiated conditions would have changed during this time (electron emission change under fixed temperature and arrival rate) or

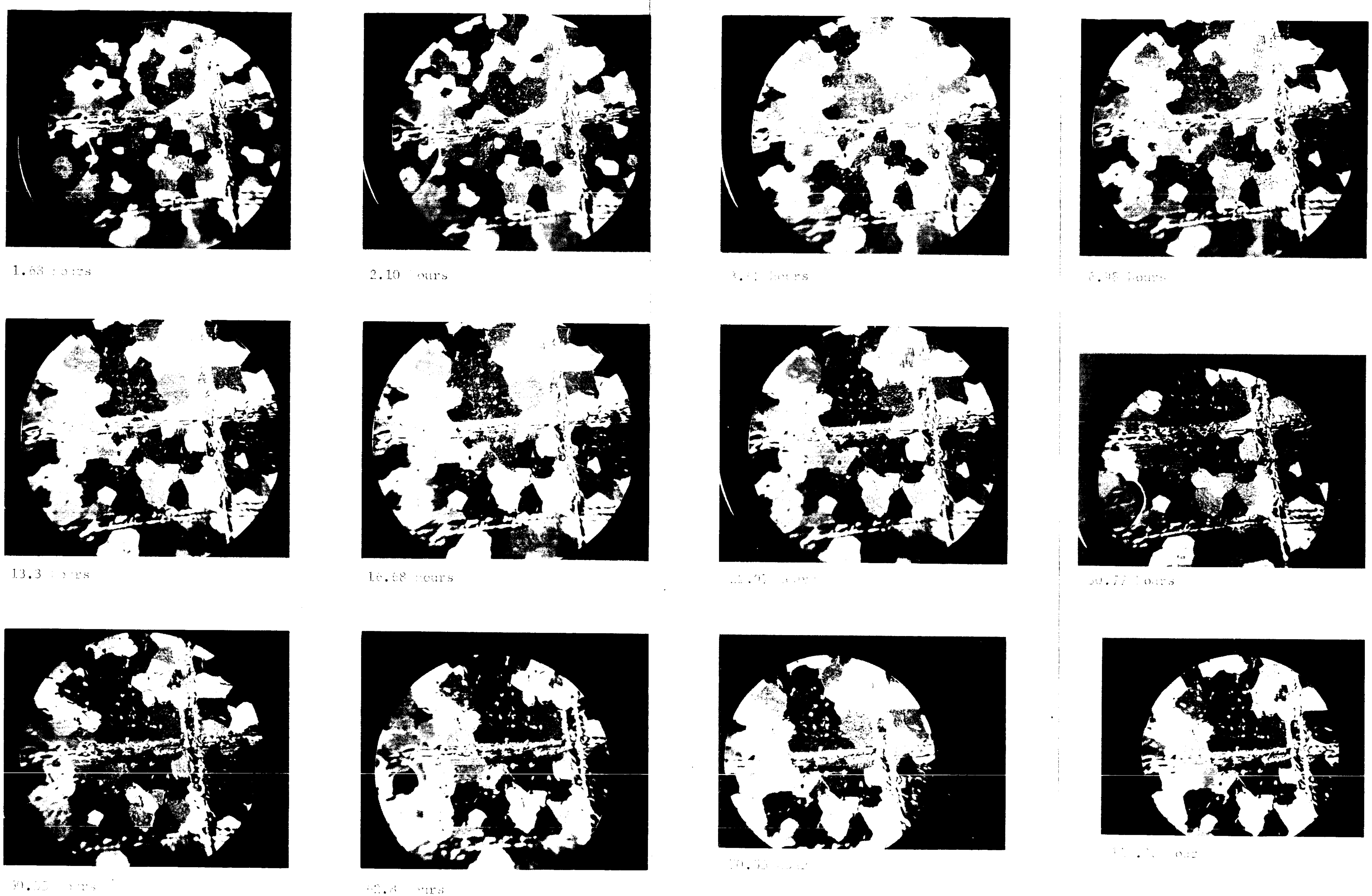
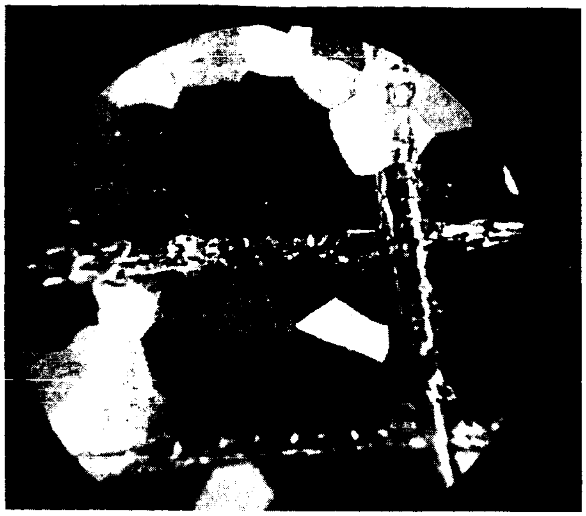


FIG. 1 ELECTRON EMISSION MICROSCOPE IMAGES. Molybdenum bar stock sample (R3) at 1500°C as a function of time. No prior outgassing. Distance between centers of parallel scratched lines is 0.0115 inches. (Magnification is therefore about 90.) Same position on sample throughout sequence.



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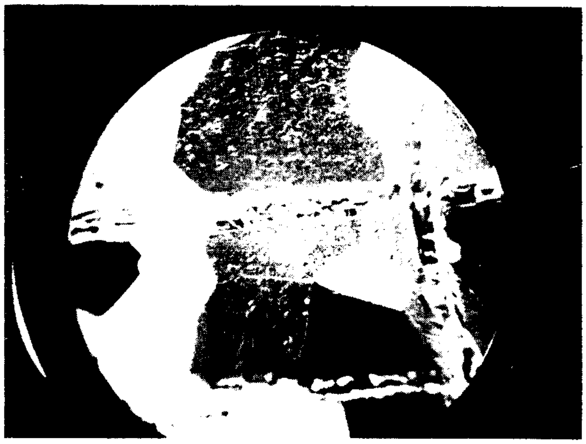

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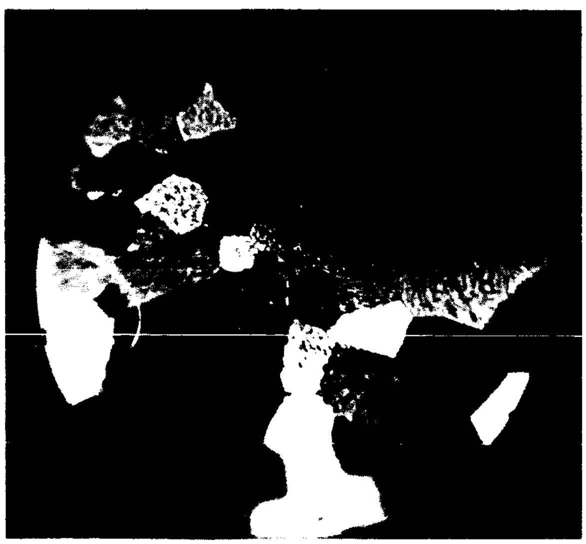

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FIG. [illegible] ADDITIONAL ELECTRON EMISSION MICROSCOPE IMAGES

(2) the cesiated work function of a substrate has only a <u>weak</u> dependence on the bare work function of the substrate. The second of these two situations is not in keeping with experimental evidence from numerous studies of the electron emission from cesiated substrates and, therefore, must be temporarily ruled out as a possibility. The first premise can only be ruled out in the unlikely circumstance that the final distribution of areas of identical work function is the same as the initial distribution of these areas. The manner in which the grains are changing with time and the total disappearance of some of the grains seem to preclude this latter line of reasoning. Therefore, it appears reasonable to assume that the cesiated emission results on a similar substrate under identical conditions will change with time at temperature.

3. <u>Impurities</u> - During the run at 1500^{o}C, low work function impurities in the form of small spots began to form on the molybdenum sample surface. These impurities seem to disappear at a temperature of approximately 1650^{o}C. After returning to 1500^{o}C from 1700^{o}C, the low work function spots were gone as shown in Figure 2. The nature of these impurities has yet to be determined.

4. <u>Microstructures</u> - During the 1500^{o}C time at temperature run, microstructure of the type evident in Figures 1 and 2 was observed to be present in several areas of the sample.

2.4 Metallurgical Studies

The studies on vapor deposited tungsten have begun. Two samples of vapor deposited tungsten were prepared to determine if any preferred orientation were present. One sample was deposited on the end of a molybdenum rod and the other on a plate of copper. Two substrates were chosen to determine the influence of epitaxial growth on orientation. These samples were then scanned by an X-ray diffractometer. The preferential orientation of any plane within several degrees of the specimen surface would cause the diffracted peak from that plane to have a relatively higher intensity.

The intensity of the various peaks are given in Table I. Specimen 1 is the tungsten vaporized on copper; Specimen 2 is the tungsten vaporized on molybdenum, and Specimen 3 is the same sample as Specimen 2, except the sample was polished to produce a smoother surface. The relative intensities of the various reflections as given in the ASTM X-ray powder data file is shown in the last column in Table I. These intensities are approximately what would be observed for a random sample. The relative peak intensities of various diffraction lines of the specimen are as follows:

TABLE I

Plane	Specimen 1	Specimen 2	Specimen 3	ASTM Data
(110)	100	3	14	100
(200)	6	100	100	15
(211)	9	3	19	23
(220)	5	0.7	1	8
(310)	30	4	10	11
(222)	0.8	-	-	4
(321)	6	-	-	18

As can be seen from the table, there is a preferential orientation of (110) and (310) in Specimen 1 and a very strong preferred orientation of the (200) plane (the cube face) in Specimens 2 and 3.

The strongly preferred orientation in Specimens 2 and 3 may be the result of epitaxial growth of the deposit upon crystals of molybdenum in the substrate which have a preferred orientation as a result of mechanical working of the rod during its manufacture. The epitaxial growth of vapor deposited coatings upon a substrate is a well-known phenomenon. Considerable effort has been devoted to the growth of such deposits in the semiconductor industry. As far as is known, no research has been devoted to the growth of refractory metal deposits of preferred orientation by vapor phase deposition techniques.

3. PROGRAM FOR THE NEXT INTERVAL

3.1 Grain Growth Investigations

The vac-ion pumped system necessary for operation of the long term tests pertaining to grain growth in cesium vapor has been completed. The grain growth vehicles will be put on test in the next period.

3.2 Cesiated Emission Investigations

The measurements of electron emission from a cesiated molybdenum bar stock emitter sample will continue. Sets of data are being taken at 25 hour intervals in order to follow the change (if any) in cesiated emission from the sample as a function of time at temperature. The measurements will extend to a minimum of 125 hours duration. The emitter temperature will be held at 1800°K between measurements, and will not be allowed to exceed 1800°K during the measurements.

A second emission test vehicle employing a plate stock molybdenum emitter sample is being readied for test. The testing procedure used on the bar stock emitter sample will be duplicated for the cross rolled plate stock specimen.

3.3 Electron Emission Microscope Investigation

During the next reporting period, the electron emission microscope will be used to investigate the surface changes occurring in over a 100 hour period with the sample held at 1500°C.

3.4 Metallurgical Studies

Metallurgical examination and determination of grain growth for the molybdenum samples which have been run in cesium vapor will be accomplished as the samples become available.

4. FINANCIAL STATUS

Man-hours, dollar expenditures, and purchase committments from 19 April 1963 to 19 May 1963 are submitted as a separate enclosure to this report.

5. PROGRESS DESCRIPTION

We estimate that approximately 75 percent of the program has been accomplished.

6. PRINCIPAL CONTRIBUTORS

The following personnel have been principal contributors to the program over the past period.

A. O. Jensen, A. E. Campbell, D. G. Worden, W. Dong, H. Todd

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