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FLAME HEATED THERMIONIC CONVERTER RESEARCH

Report No. 4
Fourth Quarterly Report
(1 April 1962 to 30 June 1962)
Contract No. DA-36-039 SC-88982

Department of the Army Task No. 3A99-09-002-04
U. S. Army Signal Research & Development Laboratory,
Fort Monmouth, New Jersey

ATOMICS INTERNATIONAL
A DIVISION OF NORTH AMERICAN AVIATION, INC.

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FLAME HEATED THERMIONIC CONVERTER RESEARCH

Report No. 4
Fourth Quarterly Report
(1 April 1962 to 30 June 1962)

By

W. R. MARTINI
R. L. McKISSON
E. V. CLARK

Power Sources Division Technical Guidelines for PR & C
No. 61-ELP/D-4623 dated 23 December 1960

Department of the Army Task No. 3A99-09-002-04

Object: To develop the technology required for portable flame-heated thermionic power sources.

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.
P.O. BOX 309 CANOGA PARK, CALIFORNIA

CONTRACT: DA-36-039 SC 88982
ISSUED: SEP 28 1962

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PURPOSE

The purpose of the procurement is to investigate the various problems encountered in the design and construction of thermionic generators capable of producing from 5 to 200 w of power. These problems cover:

1) The selection of suitable materials for thermionic diode envelopes and heat ducts where high temperatures and corrosive gases are encountered.

2) The design of a fossil-fuel burner, capable of providing the required temperatures and heating rates.

3) The establishment of design parameters for thermionic generators of various power levels from 5 to 200 w.

4) The construction of a sample generator, rated at 100 w output, to demonstrate the feasibility of the design approach.

PROGRAM OUTLINE

TASK A - THERMIONIC CONVERTER DEVELOPMENT

1. Construct converters for testing. These will be flame heated converters and may have refractory metal or dispenser emitters.
2. Study the basic performance characteristics of diodes designed for use in the 100-w Thermionic Generator Program.
3. Using the same equipment as for Phase 2, operate diodes for up to 1000 hr to determine lifetime characteristics for the diode itself.

TASK B - HEAT SOURCE DEVELOPMENT

1. Develop aspirated regenerative burners for propane and for gasoline.
2. Develop a fan-powered regenerator burner assembly for gasoline fuel.
3. Develop temperature controls for emitter, collector, cesium reservoir, and fuel injector.
4. Construct and operate the 100-w thermionic generator prototypes. This will include the integration of the combustion chamber, the converters, and converter switching and control equipment.
5. Design converters for 5 to 200 w power levels at 6, 12, and 24 v dc, and analyze performance expectations.
6. Perform environmental tests on thermionic generator prototypes.

TASK C - MATERIALS DEVELOPMENT AND EVALUATION

1. Perform corrosion tests on molybdenum and molybdenum alloys, coated with molybdenum disilicide, in air and in flame component environments at thermionic temperatures. Also test coatings for other refractory metals. These coatings should be capable of over 500 hours of continuous operation in a flame environment at temperatures between 1200 and 2000°C and should also withstand temperature cycling.
2. Perform corrosion tests on silicon carbide base refractories and other related materials in oxidizing, neutral, and reducing flames at material temperatures up to 1700°C.

3. Determine permeability of gases through molybdenum or molybdenum alloy thimbles, coated with a superior anticorrosion coating, as determined by Phase 1. Identify the gases diffusing through at thermionic temperatures.

4. Investigate methods and materials for welding or brazing refractory metals.

TASK D - ELECTRICAL COMPONENT DEVELOPMENT

Analyze the problems of electrical power conversion and control for triggered thermionic converters. Develop the necessary circuitry for this type of electrical power conversion. Solve any other electrical problems that arise in the design and the construction of the power source.

TASK E - PROJECT COORDINATION AND REPORTS

Coordinate the various parts of this contract, and write seven quarterly technical reports and one final technical report.

OTHER PROGRAMS

The following programs at Atomics International are related to the present contract:

1) Company sponsored research on flame-heated thermionic converters.

2) Office of Naval Research sponsored research on the basic physics of thermionic converters.

3) Solar-heated thermionic converters for the Jet Propulsion Laboratory.

4) Research on uniform work function diodes for Aeronautical Systems Division, U. S. Air Force.

5) Company sponsored research on low-temperature thermionic converters, including low temperature emitters.

ABSTRACT

TASK A - THERMIONIC CONVERTER DEVELOPMENT

Two of the four flame-heated diodes have been fabricated and were tested. The first diode developed a leak in the Durak-B coated molybdenum thimble after 2 hr of operation. The second diode could not be made to operate satisfactorily, although it was heated for 3 hr, at an apparent temperature of greater than 1300°C, without causing failure of the molybdenum thimble. The inability to operate satisfactorily may have been due to:

1) Permeation of hydrogen through the heated portion of the molybdenum thimble
2) Over-filling of the cesium reservoir, causing the diode to be insensitive to the temperature of the cesium reservoir
3) A larger than expected temperature drop between the combustion chamber and the emitter surface.

After disassembly for inspection, the diode was found to be leaktight and to contain a considerable quantity of unreacted cesium. The interior of the diode was shiny. However, the sapphire spacer which separated the emitter from the collector was shattered.

TASK B - HEAT SOURCE DEVELOPMENT

During this quarter, 10 different designs were tried, and 45 separate runs were recorded, in order to develop for the demonstration converter a heater which would have both a reasonable heating efficiency and a reasonable pressure drop at an emitter temperature of ~1400°C and a heat flux of ~20 w/cm^2. The best performance reported in the last Quarterly Report[1] was duplicated. However, by the use of KT silicon carbide for all components of the demonstration diode heater, the lifetime of the heater was extended indefinitely. A lifetime of 80 hr was run, and the components showed no signs of corrosion or change in performance.

TASK C - MATERIALS DEVELOPMENT AND EVALUATION

Testing of Durak-B coated molybdenum continued, in order to round out the series of measurements on this coating. Some of our early observations

indicated that this coating might be sensitive to thermal cycling at temperatures greater than 1300°C. Consequently, a testing apparatus was built to automatically thermal cycle electrically heated wires. Some data are presented.

Data of several preliminary runs of an experiment, designed to measure the rate of permeation of gas through a thin molybdenum capsule coated with Durak-B, are given. These data indicate that hydrogen is essentially the only gas that diffuses through the wall. Hydrogen apparently diffuses through at the rate of 1 std.* cc/hr-cm^2 at 1400°C. The hydrogen is an intermediate product in propane-air flames.

*at 0°C and 1 atm pressure.

PUBLICATIONS, LECTURES, REPORTS, AND CONFERENCES

W. R. Martini attended the 16th Annual Power Sources Conference, and presented a paper entitled "Design Considerations for Gasoline Powered Thermionic Generators." The meeting was sponsored by the Power Sources Division of the U.S. Army Signal Research and Development Laboratory, and was held on May 22 to 24, 1962, in Atlantic City, New Jersey.

Three papers were prepared and presented at the Symposium on Thermionic Power Conversion, May 14 to 17, 1962, in Colorado Springs, Colorado. They were:

1) "Theoretical Calculation of the Thermal Conductivity of Cesium Vapor at Thermionic Temperatures," by W. R. Martini

2) "Progress in the Development of Flame-Heated Thermionic Power Sources," by W. R. Martini and R. L. McKisson

3) "Emitter Corrosion in Thermionic Converters," by R. L. McKisson

Mr. Joseph Angello and Mr. Frank Wrublewski visited Atomics International on April 17, 1962, for the purpose of discussing the progress of this contract. They met with R. C. Allen, D. E. McKenzie, and W. R. Martini.

W. R. Martini visited Dr. Gene Shultz, at the Institute of Gas Technology, Chicago, Illinois, on May 18, 1962, for the purpose of obtaining information on burner technology.

W. R. Martini visited the Hunter Manufacturing Company, Cleveland, Ohio, on May 21, 1962, to obtain information concerning gasoline burner technology. This firm makes two models of gasoline burners which could possibly be used as heaters.

TECHNICAL PROGRESS

TASK A - THERMIONIC CONVERTER DEVELOPMENT

Phase 1 - Diode Construction

Work during this report period has been devoted to the construction of internally flame-heated diodes. The reasons for undertaking the development of this type of diode are given in the previous quarterly report,[1] along with a description of some of the novel design features. Modifications of the diode design presented in that report (see Figure 4, Reference 1) are described in this report.

Emitter Thimbles

This part of the diode has caused the most difficulty. Our experience in the preparation of the first demonstration converter, in June, 1961, indicated that molybdenum thimbles, machined from bar stock, produced leaktight thimbles when coated with a protective coating of Durak-B, but welded thimbles did not. Accordingly, six thimbles were machined with 12-mil walls. Although these were helium leaktight before the coating was applied, only one was tight after the coating was applied; the others had developed longitudinal cracks, or cracks at the transition between the thin wall and the thick bottom. This thimble was built into the first diode.

In order to try other methods of fabricating molybdenum thimbles, and to get at least three more thimbles for preparation of the sample product, procurement was started on thimbles made by the following processes:

1) Machining, with intermediate heating to remove stresses

2) Welding of an end cap to a drawn tube

3) Deep drawing

To date, Processes 1 and 2 have been successful. Two machined thimbles were coated successfully. One of these was used in the second diode, and one awaits use in the vacuum-insulated heat transfer experiment. Five welded thimbles were coated successfully and are leaktight. One of these is being used in the third diode. The product of the other process has a longer delivery time, but it is expected to be superior.

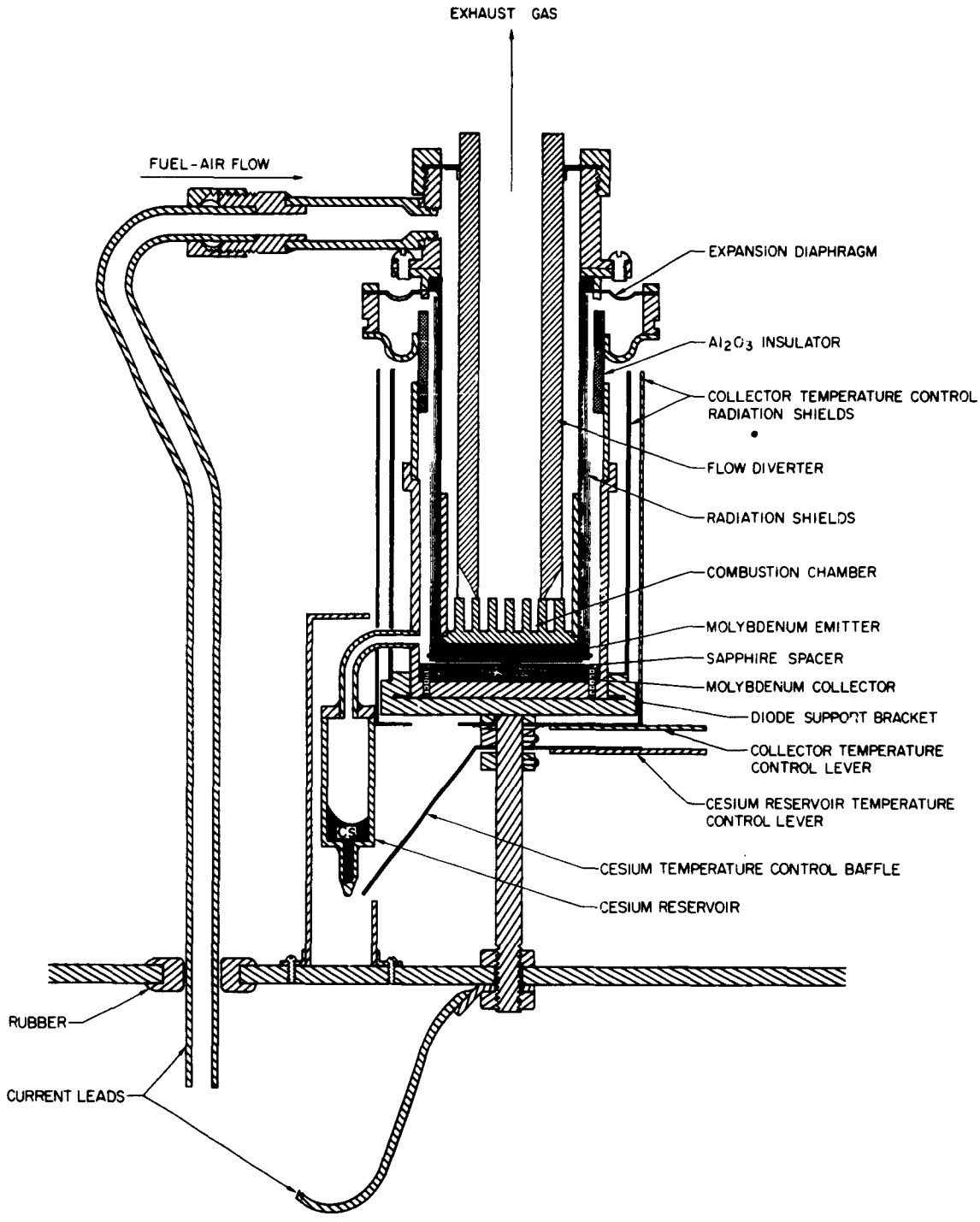

Figure 1. First Sample Product Diode Test Apparatus

Diode No. 1

A section of Diode No. 1, along with its combustion chamber, heat exchanger, and radiation shields, is shown in Figure 1. No particular difficulty was experienced in assembling the welded joints in the diode. However, the copper braze which joined the molybdenum thimble to the top flange proved to be difficult, because sufficient heat could not be applied to the joint.

The fate of Diode No. 1 is covered under Task B, Phase 4.

Diode No. 2

Upon receipt of the second batch of thimbles, Diode No. 2 was constructed, using a machined thimble. A section of this diode is included in Figure 2. As is explained under Task B, Phase 4, the auxiliary equipment was changed considerably, but the only change to the diode itself was in the placement of the cesium reservoir.

The testing and final examination of Diode No. 2 are recounted under Task B, Phase 4.

Diode No. 3

This diode is being built, using a welded thimble; and a larger cesium reservoir, filled with Ti-Zr alloy turnings, is being fitted to the diode to getter hydrogen.

TASK B - HEAT SOURCE DEVELOPMENT

Phase 2 - Fan-Powered Burner Development

During this report period, 10 different experimental setups were tested in the apparatus shown in Figure 14 of the last Quarterly Report.[1] Each setup differed from the others in the design of the flow baffle. Also, in some cases, the combustion chamber was augmented by the presence of chips of zirconium oxide. A brief description of the series of runs accomplished during this report period is given in Table I. Measurements made during the testing of each setup were assigned run numbers, as shown in Table I, and are given a symbol to identify the data in the following figures. Figure 3 shows the combustion chamber temperature plotted against the heat input for all the measurements made during this quarter's work. Notice that only rarely does the performance exceed that made during Runs 21 to 27, which is reported in the last Quarterly

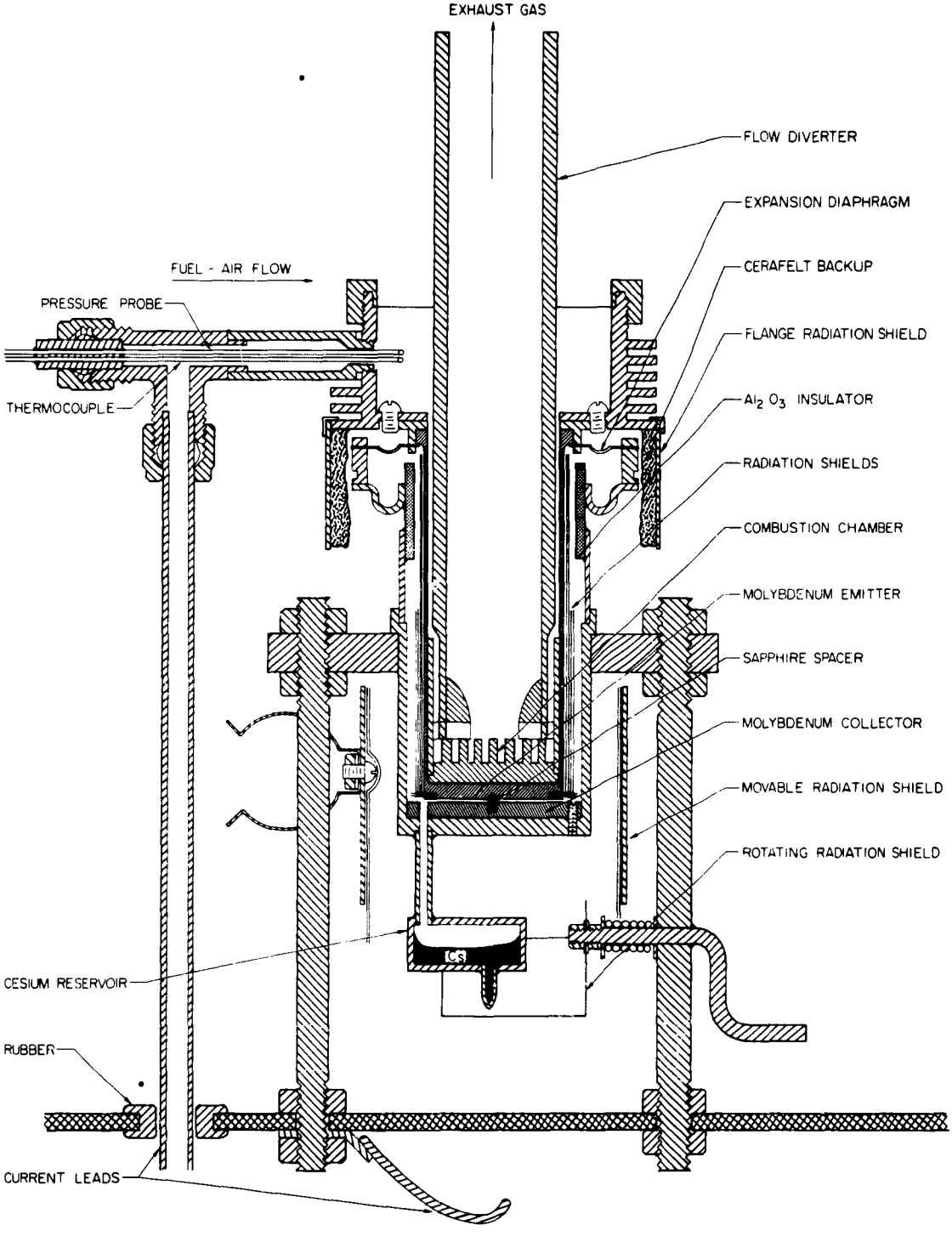

Figure 2. Second Sample Product Diode Test Apparatus

TABLE I

SUMMARY OF DEMONSTRATION DIODE BURNER TESTS

Symbol	Runs	Flow Divider	Combustion Chamber	Notes
○	28-32	Quartz glass tube, 0.78 in. OD, 0.70 in. ID	Zirconia chips covering standard combustion chamber.	Piercing whistles are produced unless outlet tube is partially blocked. Devitrifies
△	33-38	Same size quartz tube. Foam SiC plug in exit tube with 3/8 in. diameter by 1/2 in. long hole through it	4 or 5 zirconia chips (-6 +8 mesh), laying on standard combustion chamber	Whistle suppressed. Bottom of quartz flow divider devitrifies and deforms
□	39	Same size quartz tube, necked down to 1/2 in. ID at bottom	3 zirconia chips on standard combustion chamber	Quartz flow divider devitrifies and cracks
+	40-46	Quartz tube and flange, as above, with full-length insert of foam SiC. ID of insert, 3/8 in.	Standard	Loud whistle at high heat inputs. Low thermal performance. Does not deteriorate
X	47-55	Quartz flange replaced with flexible SS flange seal	Standard	Does not whistle. Still low thermal performance, but good durability
▽	56	0.78 in. OD, 0.70 in. ID quartz glass tube with foam SiC insert in bottom, 1-1/2 in. flexible SS flange	Standard	Quiet, high performance, but quartz flow divider devitrifies
◇	57-59	KT SiC tube, 3/4 in. OD, 1/2 in. ID	Standard	Low thermal performance. Sometimes noisy
●	60	Astroceram venturi	Standard	Noisy combustion. Some cracking of Astroceram
▲	61-62	Foam SiC used in Runs 21 to 27, coated on outside with Astroceram, 0.80 in. OD	Standard	Astroceram flakes off
■	63-72	KT SiC tube, 3/4 in. OD, 1/2 in. ID, fluted at bottom	3 zirconia chips added	80 hr continuous operation with no corrosion or fall in performance. Thermal performance is good.

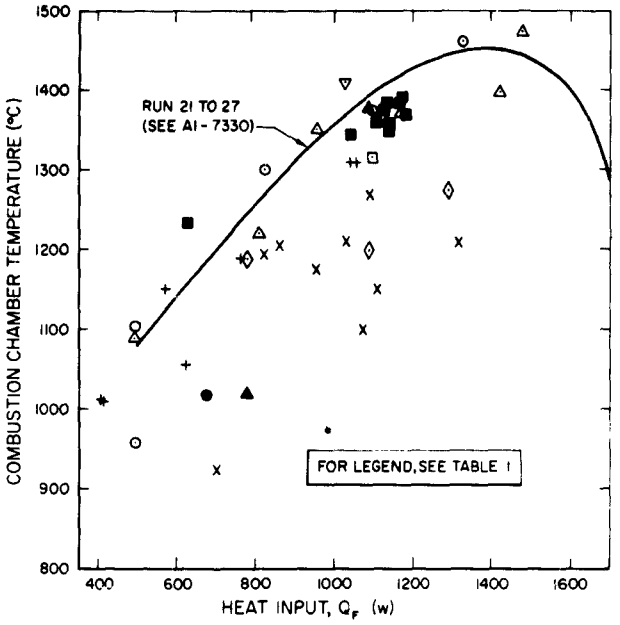

Figure 3. Combustion Chamber Temperature vs Heat Input

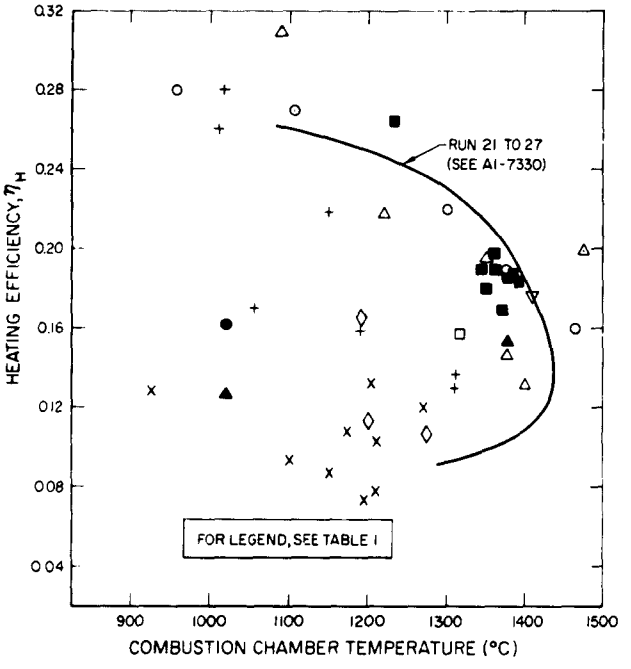

Figure 4. Heating Efficiency for Demonstration Diode Heater Experiments

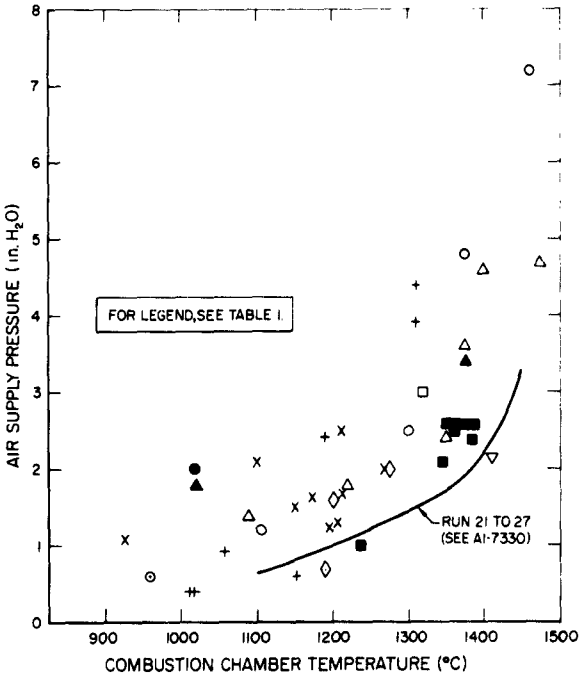

Figure 5. Air Supply Pressure for Demonstration Diode Heater Experiments

Report. However, the final configuration, Runs 63 to 72 (shown as the black squares), had almost as good performance, and was run for 80 hr without any apparent signs of deterioration, either in performance or the appearance of the parts of the combustion chamber and the heat exchanger.

Figure 4 shows the heating efficiency* plotted as a function of emitter temperature for the same series of runs. This figure also shows isolated instances where higher heating efficiencies were observed. If these conditions could be repeated, and if the furnace which produced them could run for a long period of time without deterioration, a significant advance in heating efficiency could be obtained. However, Runs 63 to 72 (black squares) showed efficiencies almost as high as the best of the earlier observations.

Figure 5 shows the relationship between the air supply pressure and the emitter temperature for the series of runs outlined in Table I. In order to alleviate the pumping problem, one is interested in obtaining the lowest air supply pressure for a given emitter temperature. Note the usual exponential increase in air supply pressure with emitter temperature. The life test runs with the SiC flow divider (Runs 63 to 72) exhibit a reasonably low pressure drop.

The conclusion drawn from this series of runs is that KT SiC is the best material to use for the flow diverter, because of its superior resistance to flame erosion and thermal shock.

A burner test was constructed to use an internally coated molybdenum thimble, so that the burner design could be checked out on a cavity of the exact size and thermal properties that would exist in the thermionic converter. Because of difficulties in obtaining satisfactory molybdenum thimbles, no test in this experimental apparatus could be carried out during this report period.

Phase 3 - Temperature Control

The problems of the temperature control of the emitter, collector, and cesium reservoir have been met in the sample product, as described in the next section, by the use of manual controls. After some experience is accumulated with the prototype diode, and the temperature control requirements are better defined, then automatic controls will be manufactured, at least for the cesium reservoir temperature. Some work was done this quarter (on a low

*The heating efficiency is defined as the heat picked up by the water cooled heat sink near the combustion chamber divided by the heating value of the fuel supplied.

priority basis) toward the development of a cesium reservoir temperature controller, based upon the principle of natural convection heat transfer of a critical fluid.[2] Freon 112 was sealed into a capsule made from 1/4 in. OD, 0.035 in. wall stainless steel tubing, 6 in. long. On testing, a temperature control function at 270°C was observed (critical temperature for Freon 112 is given as 278°C). However, this control was only in evidence over the central inch of the capsule, and the ends of the capsule were at much different temperatures, the bottom being much higher than the control temperature, and the top being much lower. From the temperature measurements, it appears that the bottom of the capsule attained about 450°C when the center of the capsule began to control at 270°C. As the apparatus heated up, even higher temperatures probably existed at the bottom of the capsule. These high temperatures caused the Freon 112 to decompose into a solid (probably consisting of a polymer and carbon) and a gas (probably consisting of hydrogen fluoride and hydrogen chloride). At the end of the experiment, the capsule was disassembled. The top of the capsule was found to be pressurized with a gas having the odor of hydrogen chloride. The bottom one inch of the capsule was found to be solid with a greenish-brown material. This experiment indicates that larger diameter capsules must be used with organics like Freon 112, since the material is inherently a poorer heat transfer medium than water. Since the Rayleigh number which governs this type of heat transfer involves the diameter to the fourth power, a small increase in diameter would greatly increase the natural convection heat transfer.

A water-filled capsule[2] was tested, to determine the combined effect of heat flux and temperature upon the control action of a naturally convecting critical fluid. A quarter-inch copper rod was bound against the bottom 13/16 in. of the 6 in. capsule with copper wire, and then was soldered. This rod was connected to a heat source. A similar copper rod was attached to the top of the capsule by the same method. This rod was connected to the cooler. Two thermocouples in the bottom copper rod were used to measure the heat input by observing the temperature gradient. Two thermocouples in the upper copper rod also measured the heat output. Fourteen thermocouples, spaced at approximately equal distances along the free length of the capsule, measured the temperature profile. Due to the fact that air was sealed into this capsule when it was prepared, it was necessary to gently tap the capsule, in order to start the convective action. A more detailed temperature profile was obtained, similar to that shown

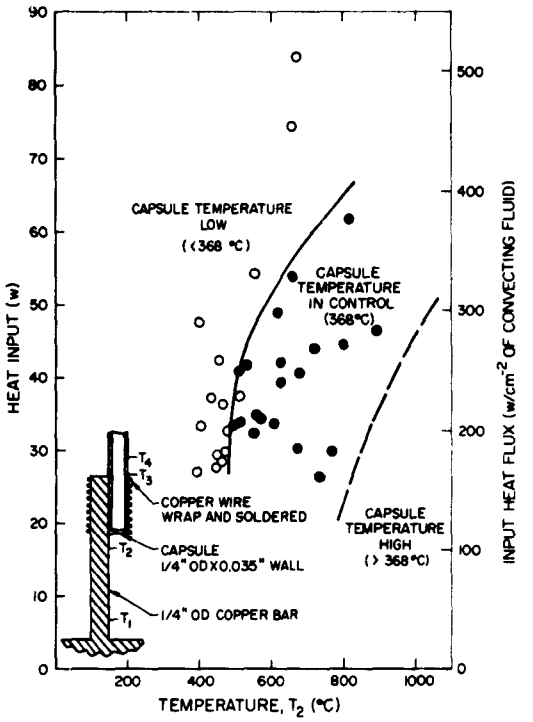

Figure 6. Control Range for Water-Filled Capsule

in Reference 2, Figure 11. Various heat inputs, in combination with different methods of cooling the top of the capsule, were employed, in order to map out the control region. For instance, Figure 6 shows a graph of the temperature of the copper bar, 1/4 in. below the stainless steel tube, plotted against the heat input into this capsule, as measured by the temperature gradient in the bottom copper bar. The black circles show instances where the center of the capsule was on control at 368°C. The open circles show instances where the cooling was too great and the center of the capsule was lower than 368°C. From previous qualitative experiments, it is known that it is possible to have so little cooling that the temperature of the stainless steel tube will ride up above the control temperature of 368°C. Notice that the limits of the control region appear to be a function of both temperature and heat flux. In preparing Figure 6, the point at which the temperature was recorded is arbitrary, and the heat flux of the capsule is a function of the effective heat transfer area at the bottom of the capsule and the capsule diameter, as well as of the fill liquid. Nevertheless, Figure 6 demonstrates that control is possible, at exactly the critical temperature, over a rather wide range of temperatures and heat fluxes. Notice that sufficient data were obtained at capsule temperatures which were lower than the critical point to fairly well delineate the boundary between control and noncontrol on the low side. Noncontrol on the high side was not experienced during this test.

Phase 4 - Prototype Construction and Testing

According to the terms of the contract, a sample product, illustrating the results of the year's research work, is required at the end of the fiscal year. The sample product was built to show that a thermionic converter can be heated by a flame and can be controlled with a very minimal amount of equipment. It was also built to show that the converter could be heated internally in a fairly

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Figure 7. First Demonstration Diode, Ready for Test

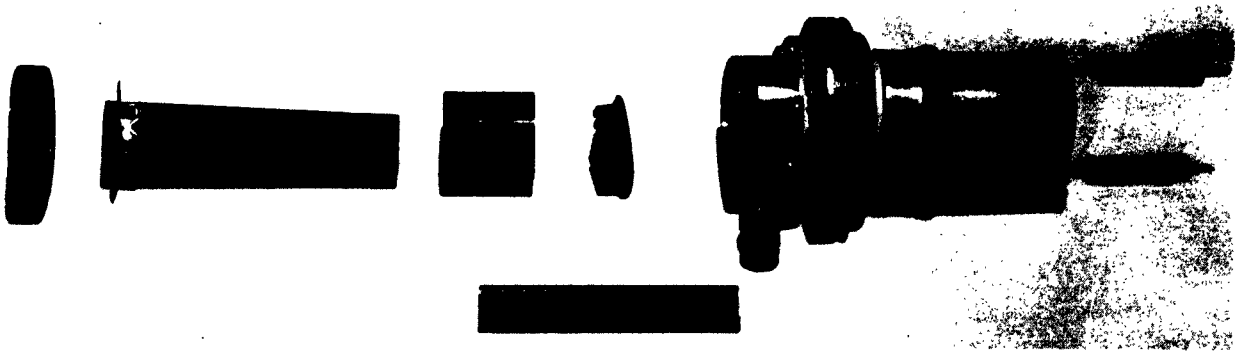

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Figure 8. Exploded View of First Demonstration Diode

efficient manner. High heating efficiency in this first converter is not intended, but this development will come later. The product was also built to show the electrical output of the converter, by measuring the output on a voltmeter and an ammeter, and by controlling the current with a variable resistance. The apparatus to maintain and control the temperature of the diode was built and tested with three runs, using the first diode. When it was concluded that the first diode had failed, a new holder and temperature controller was built for the second diode, to overcome the difficulties experienced in the first apparatus. This experimental work is described more fully in the following sections.

Diode No. 1

Figure 1 shows a section of the sample product diode that was first constructed. A photograph of the sample product diode, ready for test, is shown in Figure 7; and an exploded view of the diode, the inlet mixture manifold, and the furnace parts used in the first assembly are shown in Figure 8. In the setup shown in Figure 1, the mixture of propane and air comes up through the fuel line into the manifold, and then passes down through the annulus into the combustion chamber. Combustion is supposed to take place among the fingers of the combustion chamber, and the exhaust products pass out through the center opening. The temperature of the collector cup is regulated by two radiation shields which are slotted, so that half the area of the collector cup can radiate directly to the surroundings or radiation shields can cover all the area of the collector cups. This arrangement is best seen in Figure 7. The temperature of the cesium reservoir is controlled by a movable radiation shield, which either reflects radiant energy from the bottom of the collector cup onto the cesium reservoir or shields the reservoir from such radiation.

Run No. 1 was soon terminated, due to flashbacks which caused the flame to travel from the combustion chamber into the inlet manifold. The larger diameter molybdenum cup had not been available for burner testing, previous to this time, and the resultant larger inlet flow area gave a flow velocity too low to prevent flashback. In Run No. 2, a small insert of foam silicon carbide was fabricated to go between the protection tube and the flow diverter, in order to increase the velocity of the mixture entering the combustion chamber. With this modification, the diode would almost reach thermionic temperature before flashback would occur. During the early part of Run No. 2, some rectifier action was observed when the converter was driven with a transformer.

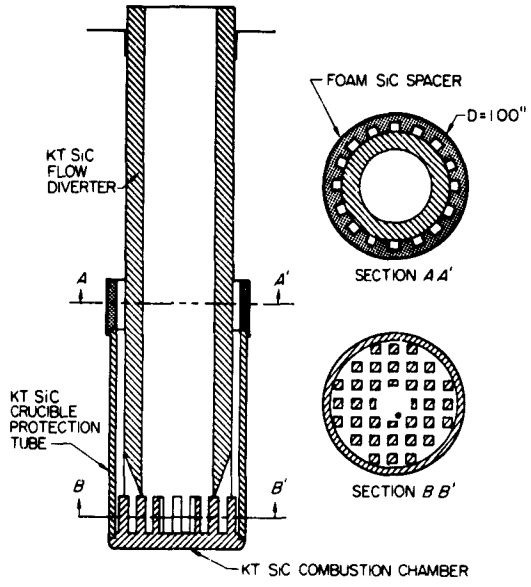

Figure 9. Flame Heater Used During Sample Product Run No. 3

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Figure 10. Emitter Thimble of Diode No. 1, Showing Failure

In Run No. 3, a new heater, shown in Figure 9, was used inside the converter. This heater design operated satisfactorily; and stable heater operation, at what appeared to be good thermionic conditions, was obtained. However, no electrical output of any sort was observed from the converter.

After removing the diode from its holder, and removing the combustion chamber and flow divider from the inside of the diode, the diode was inspected thoroughly for obvious flaws and none were found. The diode was then opened at the cesium reservoir, in order to test the diode for leaktightness. The diode began to pump down; and then a pop occurred, and a very large hole opened up in the emitter thimble. Figure 10 is a photograph of the emitter thimble, taken after it had been removed from the diode. The converter thimble seems to have had a small pinhole in its coating. Early in the experiment, the hole let the vapor space of the diode down to air. The cesium metal gettered all the oxygen in the air, leaving an inert blanket of nitrogen in the interelectrode space. After this, changes in temperatures of the gas inside the diode, and changes in pressure on the inlet manifold of the burner, caused some passage of air through the pinhole in the emitter thimble. The oxygen in the air that entered through the pinhole consumed the molybdenum of the thimble itself and of the molybdenum radiation shields in the immediate vicinity of the pinhole. At the end of the run, all the molybdenum in a 1/4-in. diameter circle around the pinhole had been consumed. Outside this circle, the molybdenum appeared to have been attacked very little. Therefore, inside this 1/4-in. circle, the only thing that protected the internal parts, made of molybdenum, from the air was the free-standing molybdenum disilicide coating. Thus, when the vacuum was applied to the internal parts of the diode to test for leaks, the unsupporting coating gave way, and produced the hole that is shown in Figure 10.

Diode No. 2

The second design of a test apparatus for the thermionic diode is shown in Figure 2. The finished sample product, using this design, is pictured in Figure 11. Changes were made for the following reasons:

1) A larger finned inlet manifold was employed to prevent preignition of the mixture due to overheating of the manifold.

2) A narrower flow passage to the combustion chamber was constructed to prevent flashback.

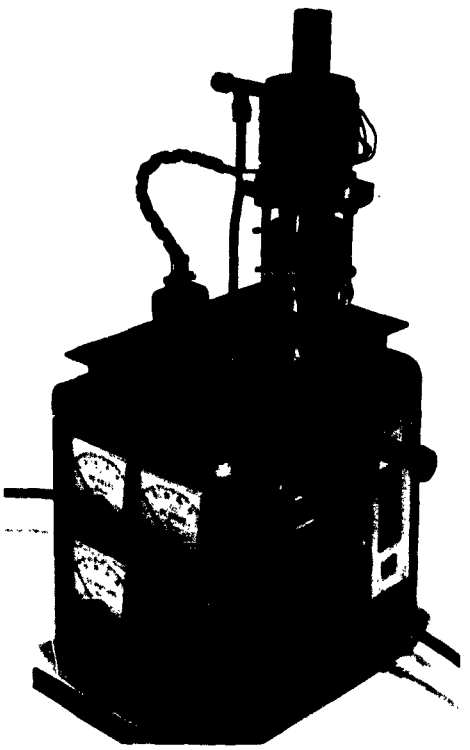

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Figure 11. Sample Product

3) A massive clamp at the top of the collector cup was employed to prevent overheating of the bottom of the seal. This change in the method of holding the diode made possible a more compact and simpler design of the temperature control system for the collector and the cesium reservoir. The temperature of the collector cup is regulated by a cylindrical radiation shield which moves up and down on one of the support columns. The temperature of the cesium reservoir, now located directly below the collector cup, is controlled by a rotatable radiation shield. This shield can be either below the cesium reservoir (to assist in its heating) or above it (to shield the cesium reservoir from direct radiation from the collector cup, so that its temperature is lowered).

4) A probe was inserted into the inlet manifold to determine gas temperature and pressure at that point.

Run No. 4 was conducted with the setup shown in Figure 2. During this run, 0.7 v of open circuit voltage, but essentially no current, were produced. The temperature of the combustion chamber reached 1325°C. The collector reached 545°C, and the cesium reservoir temperature was controllable from 290 to 400°C.

For Run No. 5, only minor modifications were instituted in the setup, such as the attachment of additional thermocouples and rework of the cesium reservoir shield. After the diode had attained possible thermionic conditions, the diode was observed to be shorted, apparently by the crushing of the sapphire spacer. The diode was cooled, removed from its holder, pried apart, and shimmed, so that the short between the emitter thimble and the collector thimble was eliminated. The diode was then re-installed, and Run No. 6 was conducted. In Run No. 6, a combustion chamber temperature of 1415°C was observed, and

an open circuit voltage was obtained, although no appreciable current could be drawn without the use of applied voltage. The converter was driven with a transformer, and volt-ampere curves were obtained, but the curves did not extend into the power quadrant. The temperature of the cesium reservoir could be controlled from 200 to 400°C, apparently without affecting the electrical characteristics of the converter. At the end of the experiment, an open circuit voltage could be obtained, but no volt-ampere curves were observed, using the transformer drive. We concluded that, by the end of Run No. 6, the diode had been let down to air. However, on disassembly, the diode was found to be helium leaktight, and cesium metal was present. The inability of the diode to operate may have been due to one or more of the following causes:

1) Cesium metal overfill, causing the entire cesium reservoir to be full of cesium, so that the pressure of cesium in the interelectrode space was not that expected from the observed cesium reservoir temperature reading.

2) Low emitter temperature, due to unexpected high thermal resistance between the combustion chamber and the emitter surface. This may have been due to a possible air gap between the SiC combustion chamber and the molybdenum thimble.

3) Hydrogen permeation, causing a high pressure of hydrogen gas to fill the interelectrode space.

Diode No. 3

Diode No. 3 is being prepared with a hydrogen getter made from turnings of a zirconium-titanium alloy. The amount of cesium, and the volume of the cesium reservoir, have been chosen so that there is no possibility of overfilling the cesium reservoir. More attention will be directed toward eliminating any possible gaps in the conduction path from the combustion chamber to the emitter surface.

Diode Heaters

During the six diode runs described in the previous sections, some data was collected which can be compared with the demonstration diode burner tests described under Phase 2. In particular, the combustion chamber temperature was observed in the same way as it was during the demonstration diode

burner tests. That is, the apparent temperature of the bottom of the combustion chamber was recorded. Also, in the diode heater tests, the heat input is known, although the heat flux through the combustion chamber is not known. Therefore, Figure 12, which is directly comparable with Figure 3, was prepared from the burner tests data. We did notice that, during Runs No. 4, 5, and 6, the combustion chamber actually ran hotter (for a given heat input) than was experienced during the best of the burner tests. In Run No. 3, the temperature for a given heat input was considerably lower, due to the fact that the diode had, by this time, filled with air. When a diode which will produce an appreciable amount of current is prepared, its effective electron cooling should bring the combustion chamber temperature down to agree substantially with the burner tests previously conducted. This indicates that the burner tests so far conducted have been a good stand-in for diode heater experience.

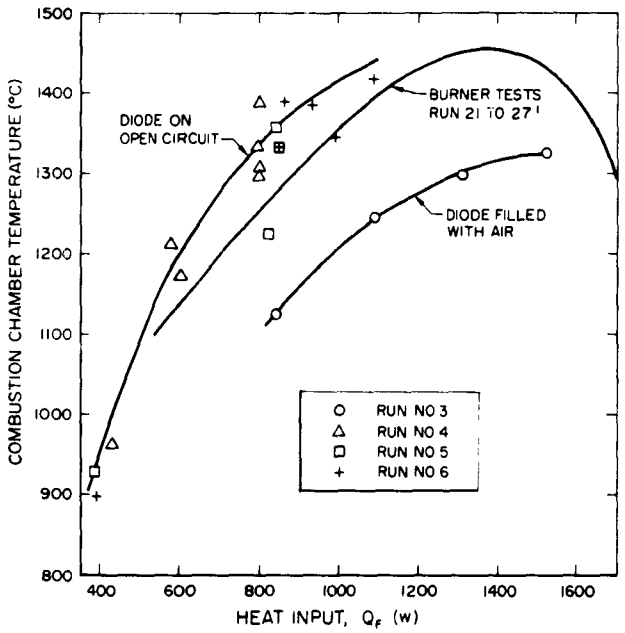

Figure 12. Thermal Performance of Diode Heaters

TASK C - MATERIALS DEVELOPMENT AND EVALUATION

Phase 1 - Protection of Molybdenum

Summary of Durak-B Data

The studies of the Durak-B coating on molybdenum have continued, as outlined in earlier Quarterly Reports.[1, 2, 3] The first batch of 1.5-mil and 2.5-mil coated samples is expended. A second batch of 2.5-mil and 4-mil samples were received in April, and is currently being tested. In addition to the life tests in the various media, a new test was initiated during the quarter to establish the capability of the coatings to withstand the temperature cycling expected in a series of startup and shutdown sequences.

The experimental data obtained during the report period are shown in Table II, and all of the pertinent data obtained to date are shown on Figures 13 and 14. Figure 13 shows the air-exposure data. The 1.5-mil data are seen to fall above the reference curve, but the 2.5- and 4-mil data tend to fall on or below the curve. This indicates that the heavier Durak-B coatings are better protection agents than those for which literature data are available. However, a wide range of failure times is observed for all four groups of samples. On the basis of these test results, it appears unlikely that any significant increase in protection life will be obtained from the present Durak-B coating. However, there is still room for improvement in the control of "premature" low-temperature failures.

Protection From Flame Components

Figure 14 shows the flame-component exposure data. Again, there is a large variation in the observed service lives, but an "average" line through these data would lie a factor of three above the line shown (i.e., the protection-life would be about one-third of that indicated by the "literature value" line[1, 4, 5, 6, 7]). It is of interest to note that the samples tested in flames tend to fail at or near their centers. This seems to be consistent with the corrosion chemistry of the SiO_2-flame component system. The chemical equilibria indicate that reducing components, such as CO, H, or H_2, should react with SiO_2 to form gaseous products, particularly in the presence of water vapor. In air, however, the equilibria are more favorable to the SiO_2 protective coating; so that other processes, such as the transformation of silica glass to a crystalline SiO_2, may become important contributors to failure. In the flames, the SiO_2 attack-rate

TABLE II

SUMMARY OF CORROSION EXPERIMENTS ON DURAK-B COATED MOLYBDENUM SAMPLES

Sample Number	Nominal Coat (in.)	Test Environment	Brightness Temperature (°C)	Corrected Temperature* (°C)	Time (hr)	Notes	Remarks
227	0.0025	Air	1750	1840	20	(1)	Failed at center
228	0.0025	Air	1750	1840	78	(1)	Test terminated by power failure
229	0.0025	Air	1750	1840	10	(1)	Failed at center
230	0.0025	Air	1500	1570	22	(1)	Test terminated by power failure
231	0.0025	Air	1500	1570	273	(1)	Failed at quarter-point
232	0.0025	Air	1300	1350	355	(1)	Failed at quarter-point
233	0.0025	Air	1750	1840	12	(1)	Failed at center
234	0.0025	Air	1500	1570	80	(1)	Failed at center
235	0.0025	Air	1500	1570	117	(1)	Failed at center
236	0.0025	Air	1500	1570	161	(1)	Failed at center
237	0.0025	Air	1300	1350	408	(1)	Failed at center
238	0.0025	Air	1400	1460	269	(1)	Failed at center
239	0.0025	Air	1500	1570	204	(1)	Failed off-center
258	0.004	Air	1750	1840	156	(1)	Timed test, no failure
259	0.004	Air	1750	1840	25	(1)	Timed test, no failure
261	0.004	Air	1500	1570	311	(1)	Failed at quarter-point
266	0.004	Air	1300	1350	549	(1)	Failed off-center
268	0.004	Air	1500	1570	140	(1)	Failed at center
277	0.004	Air	1500	1570	167	(1)	Failed off-center
815	0.0025	Air	1750	1840	20	(2)	Timed test, no failure
262	0.004	Red. Flame†	1750	1840	3	(1)	Failed at center
263	0.004	Red. Flame	1500	1570	77	(1)	Failed at center
264	0.004	Red. Flame	1300	1350	556	(1)	Failed off-center
265	0.004	Red. Flame	1500	1570	163	(1)	Failed at center
812	0.0025	Red. Flame	1500	1570	144	(2)	Failed at center
814	0.0025	Red. Flame	1300	1350	545	(2)	Failed at center
807	0.0025	CO	1700	1790	0.1	(2)(3)	Failed at center
809	0.0025	CO	1750	1840	0.6	(2)(3)	Failed at quarter-point
382	0.0015	Air	1500	1570	238	(2)(4)	Failed at center

*Temperature corrected for an assumed emissivity, $\epsilon = 0.6$
†Obtained with a Fischer burner using natural gas and air.
Notes
(1) Sample from the batch received from Chromizing Corp. in April 1962
(2) Sample from the batch received from Chromizing Corp. in August 1961
(3) Pre-oxidized in air at 1500°C
(4) Na-Ca glass wash over entire sample

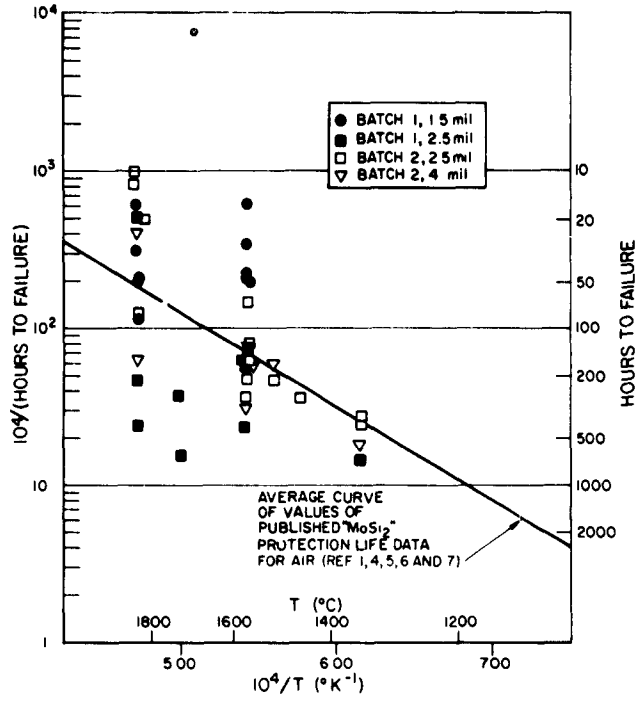

Figure 13. Protection Life of Durak-B Coating in Air

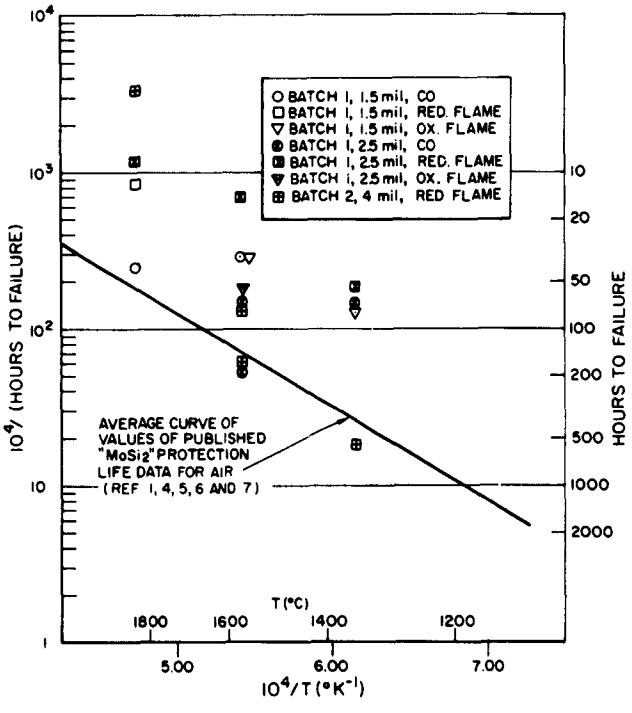

Figure 14. Protection Life of Durak-B Coating in Flame Components

might well dominate, so that the wires fail at their hot centers. Therefore, the observed lifetimes shown on Figure 14 are probably about as great as one should expect from the Durak-B coating in a flame environment. Because these times are so short, the silicon carbide cups, presently used as auxiliary protection agents, must be considered essential components of burners.

Alternate Materials

Table III is a summary of the corrosion experiments on KS coated tantalum samples, provided by the Chromizing Corp. of Hawthorne, Calif. The coating is presently under development by the Chromizing Corp., and is useful only at lower temperatures than those needed in the thermionic process. The data verify this, and suggest that the useful service temperature limit is, at most, 1300°C.

TABLE III

SUMMARY OF CORROSION EXPERIMENTS ON KS COATED TANTALUM SAMPLES

Sample Number	Test Environment	Brightness Temperature (°C)	Time (hr)	Notes	Remarks
551	Air	1300	6.7	(1)	Failed off-center
552	Air	1300	2	(1)	Failed off-center
553	Air	1300	26	(1)	Failed near end
554	Air	1400	3	(1)	Failed at quarter-point
555	Air	1000	21	(1)	Failed at quarter-point
556	Air	1500	0	(1) (2)	Failed upon reaching temperature

Notes
(1) Samples of an experimental tantalum coating obtained from the Chromizing Corp., Hawthorne, Calif.
(2) Pre-oxidized in air at 1300 and 1400°C

Thermal Cycling

Table IV is a summary of the thermal cycling experiments. Both Durak-B coated molybdenum and KS coated tantalum were tested. The test is carried out by rotating a Variac from zero to full output by a reversible geared-down motor. The output of this Variac is fed to a second Variac which is set to provide the desired maximum temperature level in the samples. The total cycle time is 3.2 min, and the sample is within 50°C of the set temperature for 0.5 min. The data show that the Durak-B coated molybdenum samples are quite

TABLE IV

SUMMARY OF THERMAL CYCLING EXPERIMENTS OF DURAK-B COATED MOLYBDENUM AND KS COATED TANTALUM SAMPLES EXPOSED IN AIR

Sample Number	Nominal Coat (in.)	Maximum Brightness Temperature (°C)	Corrected Temperature* (°C)	Cycles	Notes	Remarks
269	0.004	1300	1350	3120	(1) (2)	Test terminated, no failure
270	0.004	1500	1570	1651	(1) (2)	Failed at quarter-point
278	0.004	1750	1840	1147	(1) (2)	Failed at center
279	0.004	1300	1350	3170	(1) (2)	Test terminated, no failure
556		1500		0	(3) (2) (4)	Failed at quarter-point
557		1400		32	(3) (2)	Failed off-center
559		1300		124	(3) (2)	Failed at quarter-point

*Temperature corrected for an assumed $\epsilon = 0.6$

Notes

(1) Mo sample from batch received from Chromizing Corp. in April 1962

(2) Total cycle time 3.2 min, with time at $(T\ ^{+0}_{-50})$ of 0.5 min

(3) Samples of an experimental tantalum coating obtained from the Chromizing Corp.

(4) Pre-oxidized in air at 1300 and 1400°C

TABLE V

THE SURFACE LAYER OF SUMMARY OF X-RAY ANALYSES OF SAMPLES OF EXPOSED DURAK-B COATED MOLYBDENUM

Sample Number	Nominal Coat (in.)	Brightness Temperature (°C)	Time (hr)	X-Ray Analysis Presence* of		
				$MoSi_2$	Mo_5Si_3	Mo_3Si
369	0.0015	As rec'd		++	-	-
378	0.0015	1500	9	-	-	-
360	0.0015	1500	48	-	-	-
377	0.0015	1750	16.5	-	++	-
357	0.0015	1750	86	-	-	-
387	0.0025	As rec'd		++	-	-
804	0.0025	1300	550	-	++	-
801	0.0025	1500	155	-	++	-
815	0.0025	1750	20.5	-	0	++
396	0.0025	1750	211	-	-	-
808	0.0025	1750	422	-	-	-
266	0.0025	As rec'd		++	-	-
229	0.0025	1750	10	-	++	+
228	0.0025	1750	78	-	-	++
257	0.004	As rec'd		++	-	-
259	0.004	1750	25	-	+	++
258	0.004	1750	156	-	-	-

*The x-ray analyses were performed on filings from the surface of the samples. The symbol ++ indicates the compound is a major component, + indicates it to be present in a smaller amount 0 indicates the possible presence of the compound, and - indicates the compound is absent within the limits of the method.

resistant to this sequence, and in all cases withstood over 1000 cycles. The KS coated tantalum samples showed an increasing ability to withstand thermal cycling at lower temperatures, but were markedly inferior to the molybdenum samples.

Coating Behavior Analysis

In an effort to verify the hypothesis that one of the methods by which corrosion protection is lost by a $MoSi_2$ coated surface is by the inward diffusion of silicon to form compounds of lower silicon content, a number of samples were submitted for x-ray analysis of their outer surfaces. The analysis was to be performed on scrapings or filings of the sample surfaces, and the compounds sought were $MoSi_2$, Mo_5Si_3, and Mo_3Si. The preparation of this type of sample is difficult at best, and particularly so if the surface layer is abrasive-resistant, as is $MoSi_2$. The results obtained are shown in Table V, together with a summary of the sample history. Unfortunately, in several of the analyses, no Mo-Si compound was detected. Since microscopic examination clearly shows the presence of a surface layer other than the silica glass, it must be concluded that good test samples were not obtained. However, those samples showing positive results do show a pattern consistent with the diffusion hypothesis. The two series of samples, 226-229-228 and 257-259-258, are the most illustrative of this pattern. From the data for Samples No. 377, 815, 229, and 259, one must conclude that, at 1750°C, the silicon moves rather quickly into the molybdenum; so that, after 10 to 15 hr, very little $MoSi_2$ remains. However, the $MoSi_2$ is present at the outset, as evidenced by the data for Samples No. 369, 387, 226, and 257. Although these data cannot be considered as firm proof, the diffusion hypothesis is at least supported by them.

Phase 2 - Corrosion of Silicon Carbide

No additional work on this phase was done during this quarter. Since earlier results indicate that KT silicon carbide is superior to the other compositions tested, and is adequate for the present application, no additional tests are planned for the immediate future.

Phase 3 - Gas Permeation

A study of the permeation of flame components through a Durak-B coated molybdenum wall was carried out. A block diagram of the apparatus used

is shown in Figure 15. The test capsule used was made by welding a 0.010-in. wall, 3/4-in. diameter, molybdenum tube to 1/4-in. thick end pieces. The

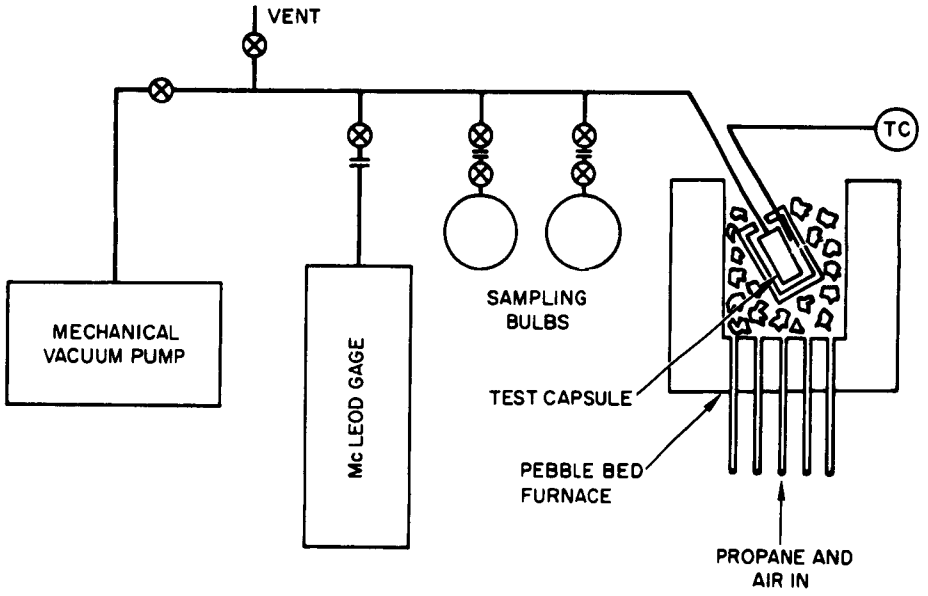

Figure 15. Apparatus for Gas Permeation Measurement

upper end piece has a drilled, 1/4-in. diameter molybdenum rod welded into it, to serve as a lead tube through which the samples are taken. The effective area of the thin walled section is 12.8 cm^2. The capsule and lead tube were coated externally with a 2-mil thick Durak-B coating (applied by the Chromizing Corporation, Hawthorne, California).

The coated capsule was brazed to a copper tube which was affixed, through a Kovar-glass seal, to a vacuum system manifold (see Figure 15). In addition, two gas sample bulbs, a Mc Leod gauge, a vent, and a mechanical vacuum pump were connected to the manifold. The capsule was inserted into a KT cup as an auxiliary flame protector, and both were imbedded in the pebble bed furnace for heating. A Chromel-Alumel thermocouple was inserted inside the cup to provide temperature measurement.

The measurements of gas permeation rate were performed by pumping the system down to about 1 μ Hg pressure and determining the time rate of increase of pressure for a number of test capsule temperatures. Using the known volume of the system, the leak rate in micron-liters per hour was determined. The data are summarized in Figure 16.

During Runs No. 13 and 15, gas samples were taken. The analyses of the samples are shown in Table VI. Room temperature leak-rates were obtained prior to Run 1, after Run 9, and after Run 19. These results are given on Figure 16.

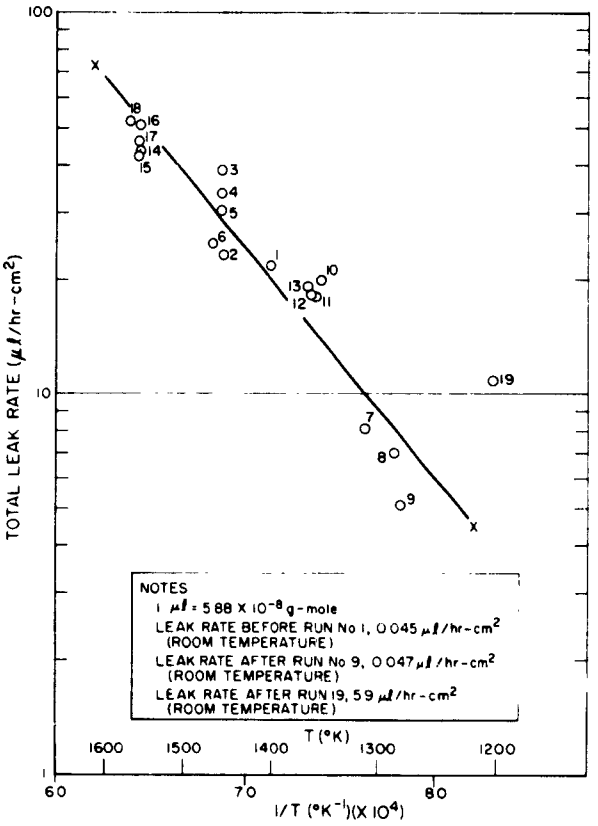

Figure 16. Permeation Rate Through Durak-B Coated Molybdenum Wall (0.010 in. Thick) Exposed to Propane-Air Flame

Examination of the data on Figure 16 shows that the gas permeation rate is temperature sensitive, as expected. However, there is so much scatter in these data that it is difficult to establish a firm value for the average activation energy of the diffusion process. Further, the fact that the system developed a leak during the second series of runs complicates the analysis and evaluation of the data. Clearly, more data must be obtained before any firm quantitative conclusions can be reached. However, tentative conclusions regarding the process can be drawn from the data in Figure 16.

TABLE VI

ANALYSES OF GAS PERMEATION SAMPLES

Gas	Sample I Run No. 13 (1368°K)	Sample II Run No. 15 (1558°C)
H_2 (mol %)	99.5+	90+
(N_2 + CO) (mol %)	0.2	10-
O_2	ND	trace
A	ND	trace

Runs No. 1 through 18 are thought to have been made with a leaktight system, and that the leak (which was found at the joint between the molybdenum tube and the copper lead tube) developed when the system was cooled down between Run No. 18 and Run No. 19. Run No. 19 is therefore neglected.

A tentative activation energy for the overall diffusion process was obtained, using the slope of the line defined by the X's in Figure 16. The tentative activation energy, E, given by the simple Arrhenius equation,

$$\frac{d\,\ell n\,(\text{rate})}{d\left(\frac{1}{T}\right)} = \frac{E}{R}$$

is 27,500 cal/mol.

This value is of the expected order of magnitude, for hydrogen and nitrogen permeation of metals. Stearn and Eyring[8] give a table of E values for gas permeation through bare metallic walls. They quote: for H_2 through Mo at 1200°K, E = 20,200 cal/mol; and for N_2 through Mo at 1500°K, E = 45,000 cal/mol.

An extrapolation of the X-line of Figure 16 to 1500°C (1775°K) indicates the expected gas permeation rate to be 1600 μ-ℓ/cm^2-hr. This amounts to $(5.88 \times 10^{-8})(1600)$ or about 10^{-4} moles/cm^2/hr through an 0.010-in. wall of Durak-B coated molybdenum.. At 1000°C (1275°K), the expected permeation rate is 4.5×10^{-6} moles/cm^2/hr.

An estimate of the integrated leak rate of the demonstration diode emitter thimble has been made, using the data of Figure 16. The diode has 10.4 cm^2 of thin-walled cylindrical area for each cm of length. A linear temperature

gradient, ranging from 1500 to 500°C, was assumed along the 7-cm length of the emitter thimble. On this basis, the estimated total permeation rate is 1.25×10^{-3} g-moles/hr, or 27.8 STP cc/hr. Since the internal volume of our present diode is about 40 cc, this leak rate would be expected to cause a troublesome buildup of gas in just a few minutes of 1500°C operation. There is a chance that an equilibrium pressure of hydrogen might be developed within the operating diode. If this pressure were not too high, it might be tolerable, and the diode could operate in spite of it. However, this circumstance seems an unlikely one, so that the prudent course is to provide a getter to absorb the permeating gas. For 100-hr service, there would be about 0.25 g of hydrogen permeating the thimble, and about 6 g of titanium or 8 g of titanium-zirconium alloy are required to getter it.

It should be borne in mind that these estimates are still rather crude. However, it does seem certain that a hydrogen-absorbing alloy must be installed in the flame-heated diode.

Phase 4 - Welding of Molybdenum

The retort welding technique previously described[3] has now been used successfully in the fabrication of emitters for the flame-heated diode. No difficulty was experienced in coating these emitters with Durak-B, and one is being incorporated in Diode No. 3. This type of weld can now be made on a routine basis.

CONCLUSIONS

1. Satisfactory individual diode heaters can be made, using propane as the fuel and air as the oxidizer.
2. Permeation of hydrogen through Durak-B coated molybdenum metal appears to be a serious problem.
3. Temperature control by critical fluid natural convection is possible, over a wide range of thermal conditions.

PROGRAM FOR NEXT QUARTER

TASK A - DIODE DEVELOPMENT

The final two of the first four demonstration diodes will be assembled and tested. Basic performance tests and life tests will be performed, with these and other similar diodes. Adaptation of low-temperature thermionic converters to flame heating will be initiated.

TASK B - HEAT SOURCE DEVELOPMENT

Work on the adaptation of the demonstration diode heater to the use of leaded gasoline will begin.

Development of high-efficiency heaters will be resumed, using vacuum-insulated heating experiments. These will be essentially converters with the collector replaced by a water-cooled calorimeter.

Development of automatic temperature-control devices will be continued.

TASK C - MATERIALS DEVELOPMENT

Results on the permeability of Durak-B coated molybdenum will be verified and analyzed. The equilibrium pressure of permeating gas will be measured.

Another survey of emitter thimble materials and seal materials will be made, to obtain materials for evaluation. Whenever possible, evaluation will be in conjunction with the heat transfer experiments.

TASK D - PROTOTYPE DEVELOPMENT AND TEST

A design of a portable power source, based upon the demonstration flame-heated thermionic converter, will be worked out in detail to guide future development.

When possible, the triggered diode concept will be tested with two converters.

KEY PERSONNEL ASSIGNED TO PROJECT

		Hours Worked During Fourth Quarter
D. H. Adair	Senior Mechanic	505.2
E. V. Clark	Research Engineer	487
R. G. Cole	Mechanic-Engineering Laboratory, Junior	484.4
W. R. Martini	Project Engineer	441
R. L. McKisson	Project Engineer and Supervisor	229.5

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Atomics International, Canoga Park, Calif.

FLAME HEATED THERMIONIC CONVERTER RESEARCH

by W. R. Martini, R. L. McKisson, and E. V. Clark

4th Quarterly Report, 1 Apr. - 30 June 1962

50 pp, 16 illus., 8 refs.

(Report No. AI-7490)

(Contract DA 36-039 SC-88982)

Unclassified Report

The purpose of this research is to develop the technology required for portable, flame-heated, thermionic power sources. Active development in thermionic diodes, heat sources, and materials is underway.

Two of the first four flame-heated thermionic diodes were built and tested. The first diode failed, due to a flaw in the protective coating for the molybdenum thimble. The second diode suffered a crushed sapphire spacer before proper operating conditions could be found.

A satisfactory heat source was developed for the demonstration flame-heated diode. A combustion chamber temperature of 1400°C, a heat flux of 20 w/cm^2, a pressure drop of 2.5 in. of water, and an indefinitely long burner life were attained in a space 1 in. in diameter by 2 in. long.

Testing of Durak-B coating for molybdenum metal continued. Tests on thermal cycling of this coating were initiated. Preliminary measurements

(over)

UNCLASSIFIED

1) Combustion Chambers
2) Combustion Chamber Liners
3) Molybdenum Compounds and Silicides
4) Silicon Compounds and Carbides
5) Inverter Rectifiers
6) Diodes (Electronic Tube Devices) – Cesium Vapor
7) Power Supplies (Power Equipment)

I Thermionic Converter Research
II Martini, W. R.
III McKisson, R. L.
IV Clark, E. V.
V U. S. Army Signal Research & Development Laboratory, Ft. Monmouth, New Jersey
VI Contract No. DA 36-039 SC-88982

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I Thermionic Converter Research
II Martini, W. R.
III McKisson, R. L.
IV Clark, E. V.
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Thermionics
Converters
Diodes
Molybdenum
Molybdenum Disilicide
Protective Coatings
Combustion
Durak-B
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