HIGH CURRENT DENSITY CATHODES

G. Thornber

Spectra-Mat, Incorporated

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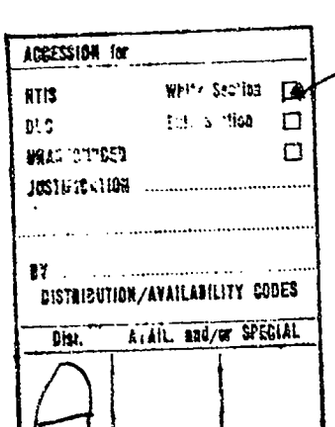
April 1974



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The procedures and equipment used are described together with the progress to date.
An emission level of 16 Amps./cm^2 has been obtained at 1050°C but cannot yet be consistently reproduced.

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U.S. ARMY ELECTRONICS COMMAND, FORT MONMOUTH, N.J.

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ILLUSTRATIONS

ABSTRACT

The objective of this program is to develop the expertise necessary to produce barium strontium tungstate cathodes with high levels of emission and of different physical configuration.

The procedures and equipment used are described together with the progress to-date.

An emission level of 16 Amps./cm^2 has been obtained at 1050°C but cannot yet be consistently reproduced.

OBJECTIVES

It is the objective of this study to develop the necessary expertise to fabricate and supply to USAECOM high current density tungstate cathodes as follows:

a. Strip beam cathode 0.060 inches long, 0.800 inches wide capable of delivering 8 A/cm^2 at a temperature of 1000°C and a life objective in excess of 6000 hours.

b. Planar cathode approximately 0.3125 inches in length with a diameter of 0.106 inches for an emission objective of 16 A/cm^2 at an operating temperature of 1075°C_t and a life objective in excess of 1000 hours.

c. Convergent cathode approximately 0.140 inches in diameter with a radius of curvature for 2 to 1 convergence and emission capability of 9 A/cm^2 at an operating temperature of 1025°C and a life objective in excess of 5000 hours.

d. Cylindrical cathode 0.025 inches in length and 1.200 inches in diameter for an emission objective of 8 A/cm^2 at an operating temperature of 1000°C_t and a life objective in excess of 6000 hours. Emission from this cathode is to be from the cylindrical surface area excluding end caps.

The above cathodes must be capable of meeting the emission objectives cited at the specified temperature. This capability has been demonstrated in planar tungstate cathodes developed on Government Contract DA28-043 AMC 02289(E) titled "High Current Density Cathodes".

EQUIPMENT

At the beginning of this program it was decided that to avoid contamination problems some special pieces of equipment should be constructed and used only for processing these cathodes and materials.

This equipment included the following:

1. A tungsten ball mill. Although a molybdenum ball mill had been used in the earlier program, poisoning of active cathodes by molybdenum had been recorded. In view of the fact that the greater part of the finished cathode is made from tungsten powder, this new ball mill could be expected to be an advantage.

2. An air firing furnace with a horizontal alumina tubing allowing the sliding in and out of alumina boats. Both tubing and boats were to be of high purity alumina. The heating element for this furnace was quoted for delivery in four months. On arrival it was fitted into the insulating brick and support structure and heated by alternating current supplied by a transformer. The power was that recommended by the manufacturers of the heating element but the design temperature of 1450°C was not reached.

In fact the furnace temperature did not quite reach 1000°C. The form of construction used is shown in figure (1) with porous alumina bricks forming the major portion of the surrounding thermal insulation. The heating element has a resistance of approximately one ohm and, consequently, has to be supplied by a transformer.

To reach the required temperature it was decided to supply twice the recommended input power and new transformers were procured. At this point over 3 kW was being supplied to the heating element but the operating temperature was still too low. At this stage advice was sought from the manufacturer's engineering staff.

3. Because of the four month delivery quoted for the heating element a temporary air firing furnace had to be constructed in order to allow the program to proceed.

This consisted of a hollow graphite crucible heated in air by a 10 kW induction heater and containing an upright alumina crucible. In this manner, a temperature of just over 1400°C was readily obtained and samples of tungstate powder were prepared. During the two hour firing the graphite crucible was almost completely consumed, a flame burning from the top of it all the time. This was producing carbon dioxide etc., and was presumably not strictly an air firing process. Firings were also carried out using a platinum crucible instead of the alumina one, but this gave a pink coloration to the fired mix.

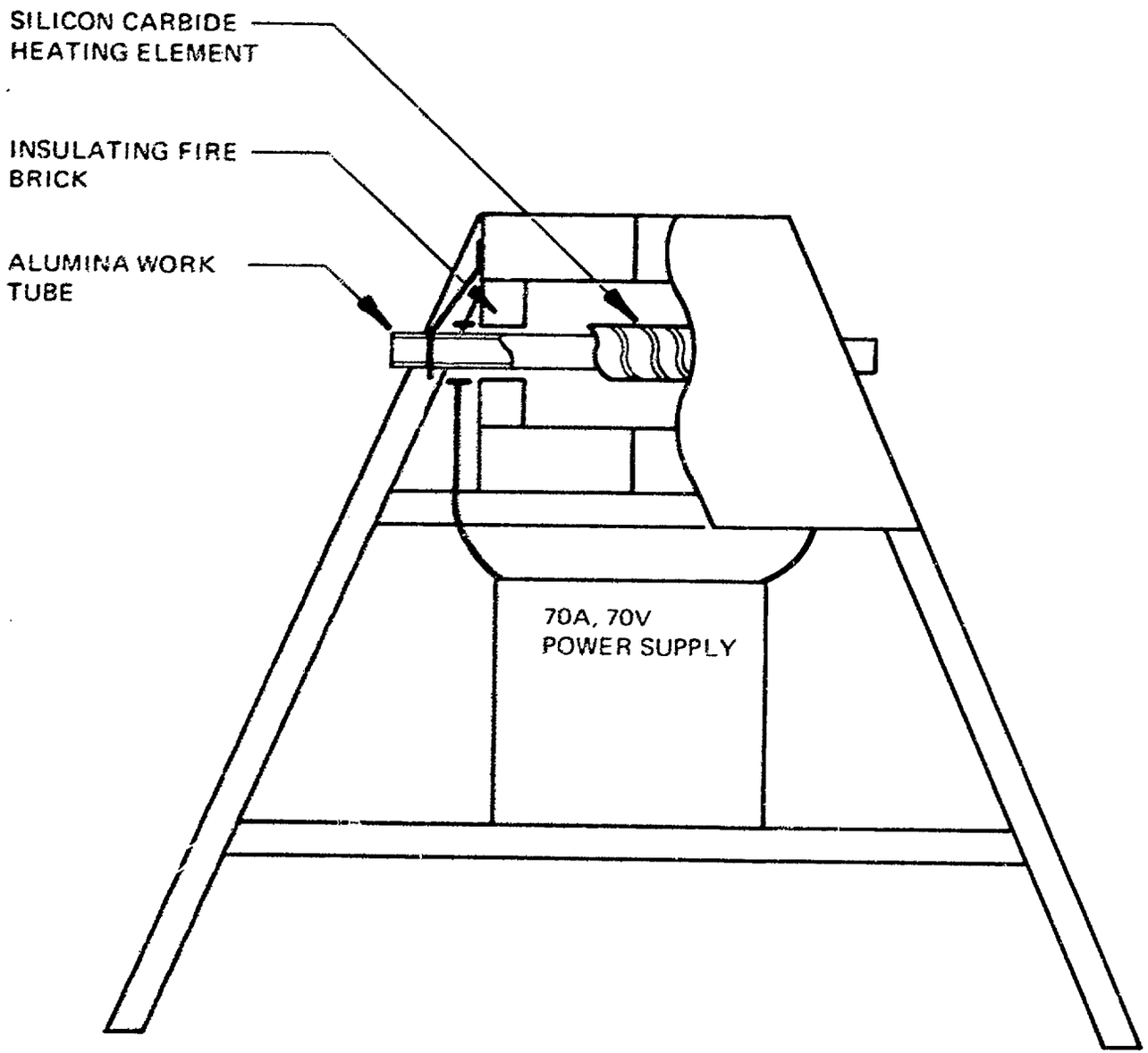

FIGURE 1 HIGH TEMPERATURE AIR FURNACE

4. A special furnace chamber was built for the hydrogen firing at 1840°C. This consisted of a molybdenum cylinder as shown in figure (2). The whole assembly is placed inside a resistive heating element made from molybdenum rod which is inside multiple heat shields. A line of sight to the inside of the molybdenum cylinder is provided to allow for optical pyrometer measurements of temperature. The hydrogen is passed through a catalytic unit converting any oxygen to water which is removed by a dryer. It has been standard practice to run through a heat cycle immediately before firing a cathode.

5. The demountable test diode is shown in figure (3). It consists of two cathode supports, the exhaust tubulation and water cooled anode brazed to the base with copper-gold alloy and a cover with a sapphire viewing window.

The use of silver and glass has been deliberately avoided to reduce contamination. Mounting of the cathode is accomplished by welding three rhenium wires to the main support plate which is rigidly held by the two threaded support rods. An anode to cathode spacing of 0.005" to 0.007" is held and the two faces are set parallel to each other.

All pumping is carried out on a 50 l/sec. Vac-ion pump with initial pump-down by a mechanical oil pump through a bakeable trap.

6. In order to measure sublimation rates a Sloan Model 200 instrument was purchased and the detecting head can be mounted on an alternative cover fitted to the test diode with the cathode in an inverted position.

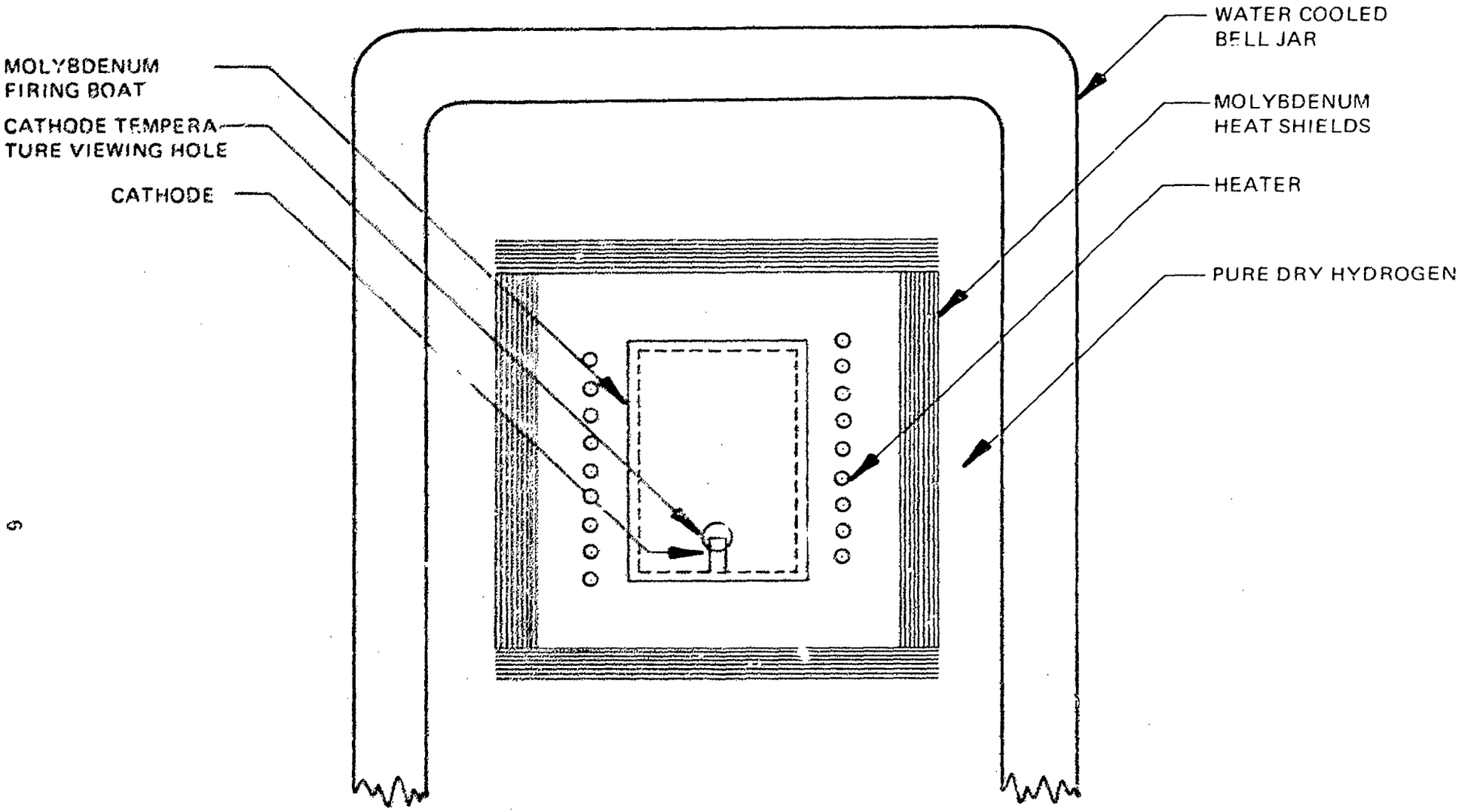

FIGURE 2 HIGH TEMPERATURE DRY HYDROGEN FURNACE

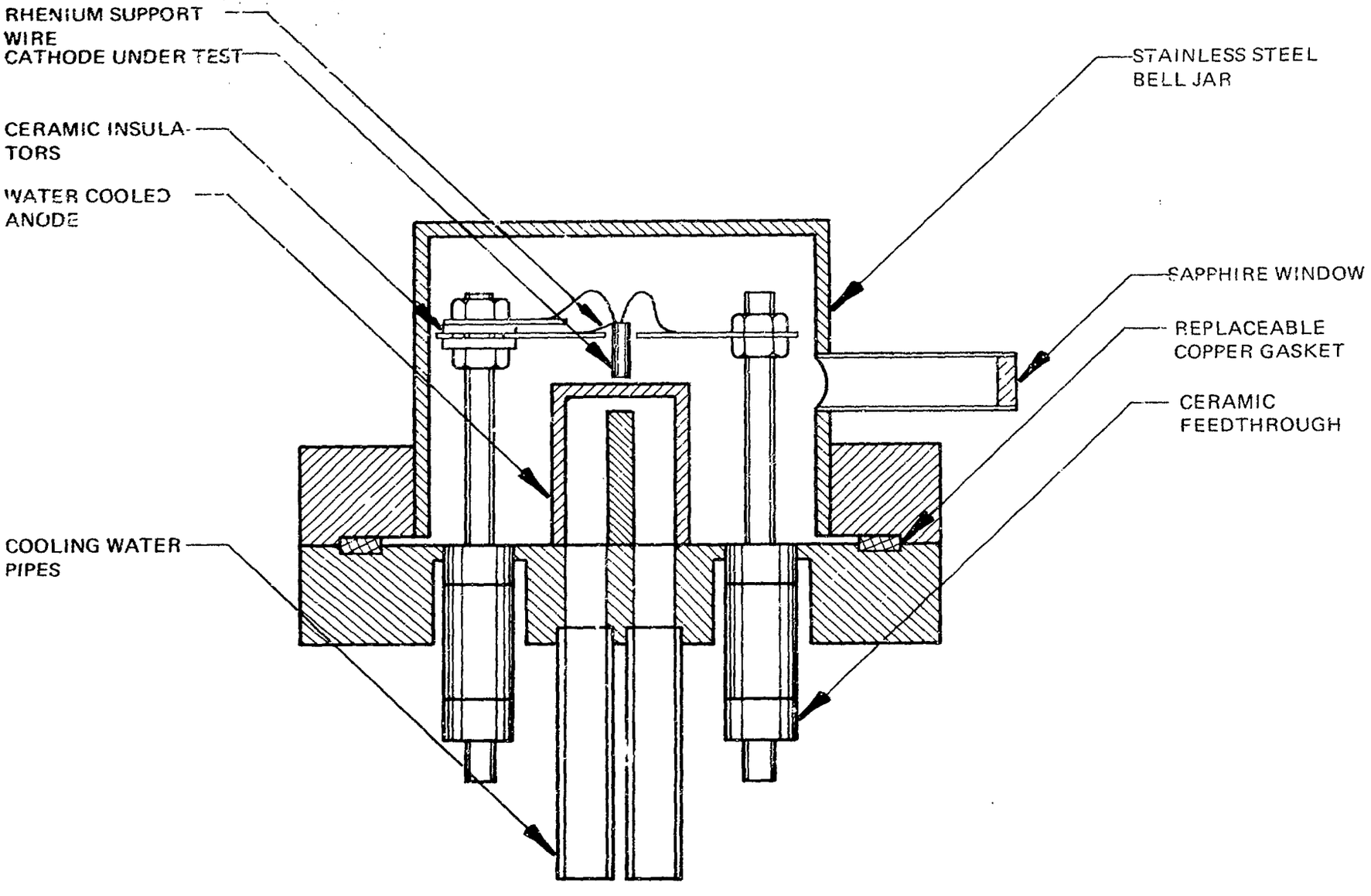

FIGURE 3 WATER COOLED DIODE

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CATHODE FABRICATION

The dimensions of the cathodes fabricated to demonstrate basic emission performance are shown in figure (4). Emission is from the flat circular end of 0.106" in diameter. Tungsten heaters are specially wound and cataphoretically coated with a commerical alumina coating which is then fired on in wet hydrogen.

The first heater gave trouble and the method of support was modified to remove the physical strain on the alumina coating. Since then no heater problems have been encountered.

Fabrication of the cathodes was according to the attached list of procedures with the following additional details:

It is important to carefully control both the temperature and time during the sintering operation. Inadequate firing can usually be detected during the subsequent machining operation because of the low mechanical strength of the cathode pellet. Firing for too long or at too high a temperature can result in an excessive loss of barium and a short life for the finished cathode.

The control of the 1840°C sintering temperature has been more difficult than expected owing to the uncertainties of the method of measurement. The calibration of the optical pyrometer had to be carried out three separate times and was finally checked against the melting points of known elements and alloys before being acceptable.

During the preparation of the pellet it is important that the pressing tool has short unsupported lengths. At the compaction pressure of 90 tons per square inch, small diameter rods which were unsupported repeatedly fractured. After reducing the unsupported length of the main compacting rod no further fracturing occured. However, some cathodes were built and were unsatisfactory in various respects. It turned out that these had been insufficiently compacted owing to a restriction in the movement of the compacting die. Further modification resulted in compaction which was satisfactory and did not cause any fracturing after repeated use.

The finishing of the emissive surface is done by carefully machining with a carbide tipped tool as described in the procedures. It is important to use a sharp tool and a slow feed. This very slow feed was produced by a geared brush type motor controlled by an electronic speed controller. A special cross-slide with the drive permanently attached was always used and various combinations of depth of cut and rate of feed are readily obtained with this arrangement. At the present time the adequacy of the

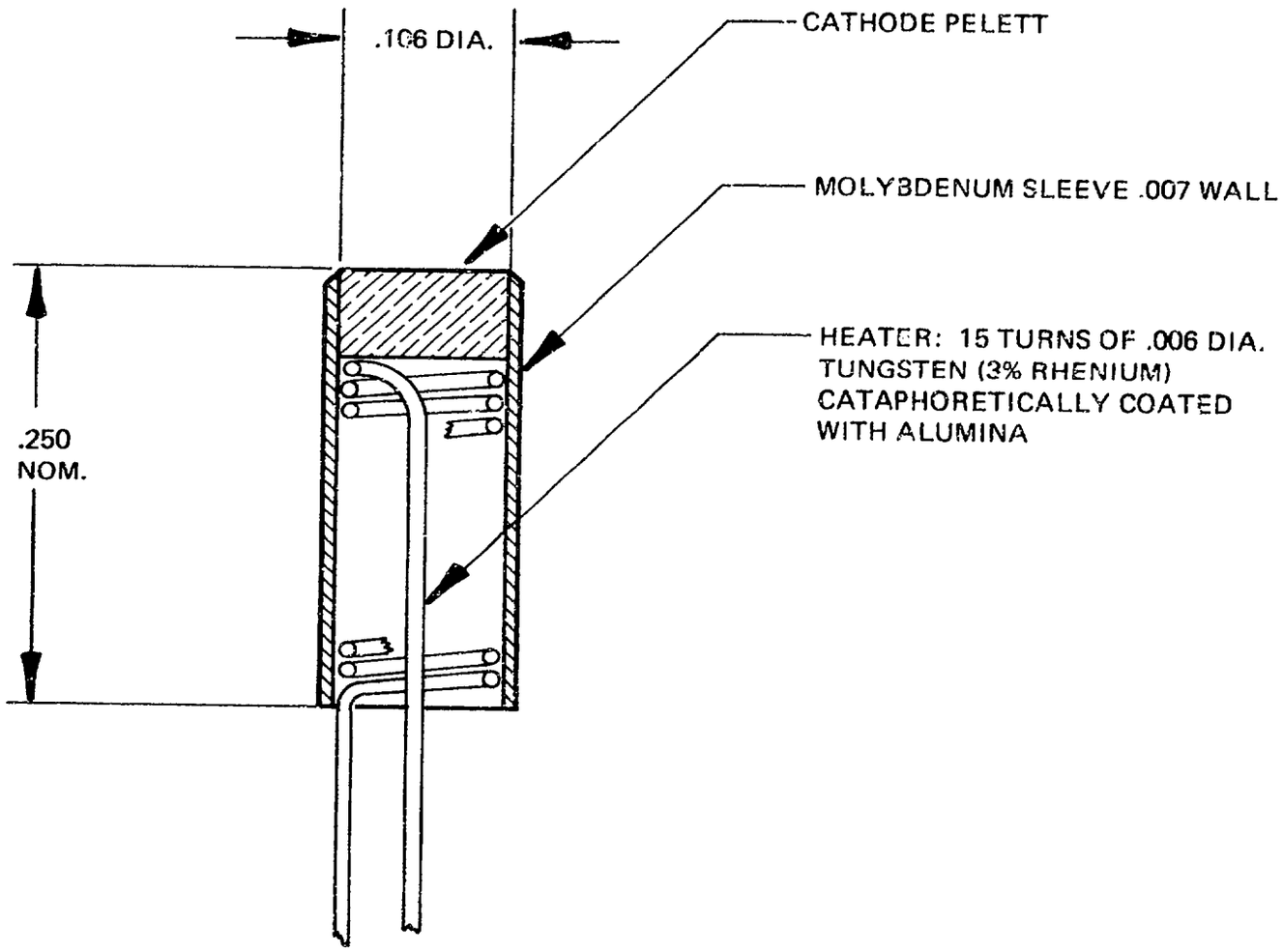

FIGURE 4 CATHODE/HEATER ASSEMBLY

machining operation is judged by the optical appearance of the surface as seen through a binocular microscope. This is felt to be inherently unsatisfactory for continued cathode production and improved methods are being investigated. For instance a metallurgical microscope is being tried with photographs used as a permanent record for comparison of emission performance and surface detail.

CONCLUSIONS

The results so far obtained have at times been promising, but, on the whole, have to be regarded as unsatisfactory.

An early cathode had the current-voltage diode characteristics as shown in figure (5). This data is plotted on "two-thirds" paper and 16 Amps. per cm^2 corresponds to 911 mA.

The activation process appeared normal, consisting of ten minutes at 1200°C., followed by tens of hours of further aging at a lower temperature whilst drawing current. After operation for a few days emission generally fell off, but this is attributed to the test diode still being connected to the main pumping station and thus exposed to various sources of contamination and higher pressure because of the long exhaust tubulation.

Further attemps to repeat and, hopefully, to improve these early results were unsuccesful. Every step of the procedures was checked many times and various problem areas were were discovered and the appropriate corrections made. Even after these corrections it was not possible to meet the emission requirements with subsequently fabricated cathodes.

In an attempt to obtain the required emission levels, emphasis is to be placed in three areas:

1. Completion of the air furnace.

2. Improved purification of the hydrogen.

3. Better definition and measurement of the machining operation.

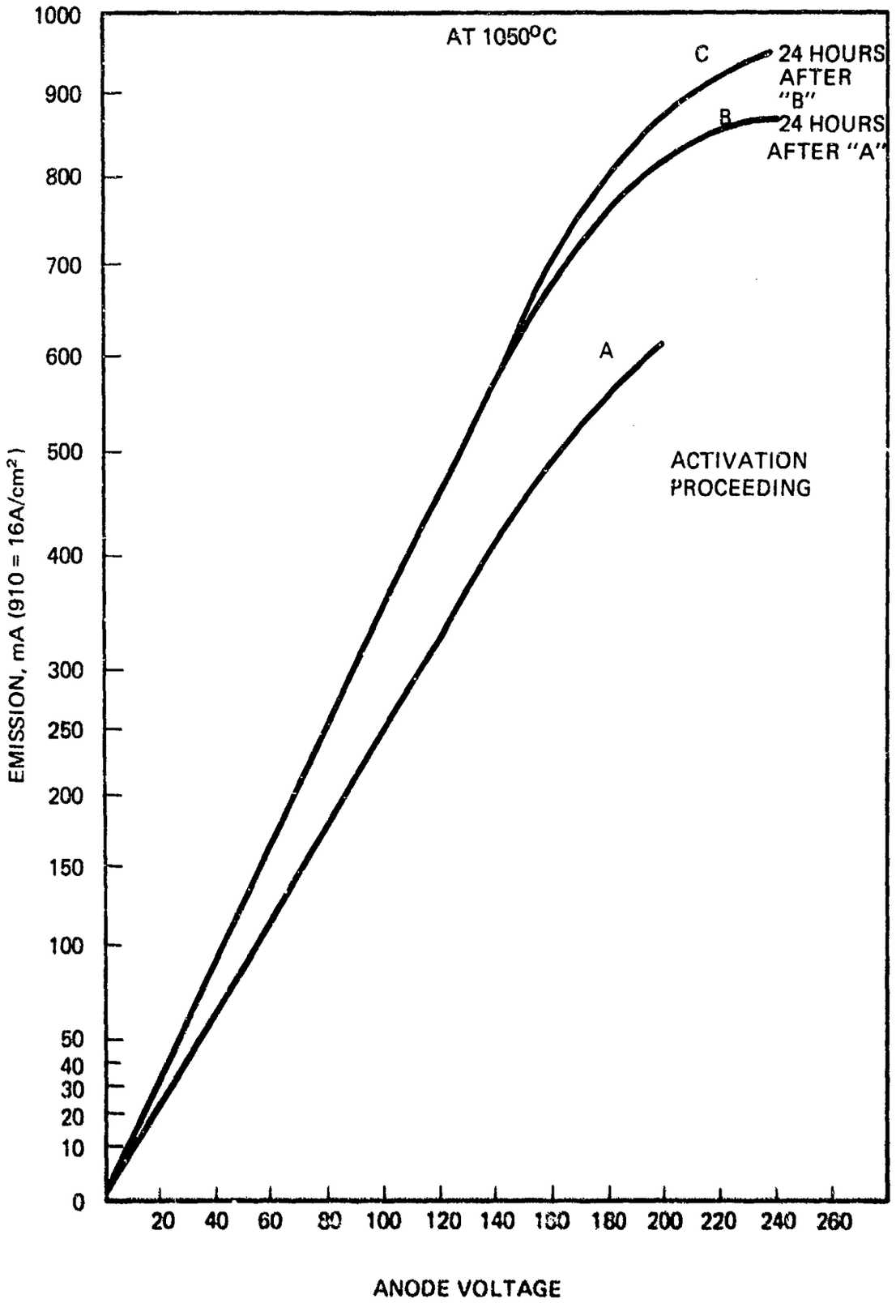

FIGURE 5 EMISSION PLOT – ACTIVATION

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PROCEDURES FOR THE MANUFACTURE OF

BARIUM TUNGSTATE CATHODES

1. PREPARATION OF ACTIVATOR COMPOUND

a. Use stoichiometric quantities of $BaCO_3$, $SrCO_3$ and WO_3:-

$BaCO_3$	18.51g.
$SrCO_3$	2.76g.
WO_3	8.73g.
Methanol	75 c.c.

b. Ball mill for 2 hours.

c. Filter the mixture using a fine Buchner funnel forming a cake.

d. Fire the cake in air at between 1400°C and 1450°C for two hours.

e. Grind dry in a mortar and screen through a 325 mesh sieve.

f. Repeat (d) and (e) above. The powder produced is $Ba_5Sr(WO_6)_2$.

2. PREPARATION OF CATHODE PELLET

a. Dry ball mill for 15 hours:-

1.	$Ba_5Sr(WO_6)_2$	- 325 mesh	2.73g.	9.14wt.%
2.	ZrH_2	- 325 mesh	0.36g.	1.22wt.%
3.	Tungsten	- 5 micron powder	26.892g.	89.64wt.%

b. Compact into oversized pellet at 90 tons per square inch using a press. Always use a vibrator during this operation.

3. SINTERING THE CATHODE PELLET

a. Use an atmosphere of pure hydrogen passed through an oxygen removing unit and a condensing unit immersed in liquid nitrogen. Dry the condensing unit each time before use.

b. Raise the temperature slowly to 1500°C_B and hold for five minutes.

c. Raise the temperature rapidly to 1840°C_B and hold for 2 1/2 minutes.

d. Allow to cool

4. MACHINING THE CATHODE PELLET

a. Use suitably prepared Carboloy tool.

b. Use a rough cut of 0.005".

c. Use a second cut of 0.002".

d. Use a final cut of 0.00025".

e. Use approximately 200 r.p.m.

f. Heat the cathode with a hot air gun.

g. Photograph the surface using a metallurgical microscope at X700 magnification.