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PERMEATION OF HYDROGEN THROUGH METALS IN
THERMIONIC-CONVERTER APPLICATIONS

John E. Creedon

January 1964

UNITED STATES ARMY
ELECTRONICS RESEARCH AND DEVELOPMENT LABORATORIES
FORT MONMOUTH, N.J.

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PERMEATION OF HYDROGEN THROUGH METALS IN
THERMIONIC-CONVERTER APPLICATIONS

John E. Creedon

DA Task No. 1G6-22001-A-055-02

Abstract

Permeation of hydrogen through the emitter surface of a thermionic converter is discussed. Approaches to eliminating a pressure buildup inside the converter are examined, and it is shown that a nickel window could be used to maintain a relatively low partial pressure of hydrogen.

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PERMEATION OF HYDROGEN THROUGH METALS IN THERMIONIC-CONVERTER APPLICATIONS

INTRODUCTION

Recent attempts^{1,2} to use thermionic converters with fossil-fuel heat sources have emphasized two major problem areas associated with the environment generated by the burner. One problem is the oxidation and subsequent burn-through of the emitter that has state-of-the-art protective coatings. In general, the protective coatings applied have had poor thermal-cycling characteristics, with failure occurring in times of 6 to 30 hours. The second problem is hydrogen permeation.^{2,3} At emitter temperatures of 1400°C the permeation rate for hydrogen through most metals is sufficiently large that it is conceivable that in approximately one hour the pressure of hydrogen inside the converter can be in the millimeter range. The permeation problem has been examined, and approaches to eliminating the hydrogen pressure buildup inside the converter are discussed.

THEORETICAL ANALYSIS

The amount⁴ of gas diffusion per unit time through a metal of area (A) can be expressed as

$$q_1 = D K \frac{A}{d} \sqrt{P} \quad (1)$$

where

D = diffusion coefficient

K = constant

A = area

d = thickness

and

P = pressure at the surface into
which the gas penetrates.

This equation assumes that the pressure on the exit side of the metal is approximately zero. At a constant pressure, the rate of diffusion theoretically increases exponentially with temperature⁵ as

$$q_1 = q_0 \sqrt{T} e^{-\frac{b_0}{T}} \quad (2)$$

where q_0 and b_0 are constants for a given gas-metal system.

Smithells⁶ has shown that for all practical purposes equation (2) can be expressed as

$$q = \frac{k_0}{d} \sqrt{P_{mm}} e^{-\frac{b_0}{T}} \quad (3)$$

where

q is in $\frac{\text{atm. cm}^3}{\text{cm}^2 \text{ sec}}$

d is in millimeters

P_{mm} is in mm Hg

k_0 and b_0 are constants.

Equation (3) can also be expressed in terms of micron-liters per minute (Q_{ul}) at 0°C by

$$Q_{ul} = 760 \times 60 q = 45,600 q. \quad (4)$$

If the pressure of the gas on the exit side of the material is appreciably different than zero, then equation (4) should be modified to

$$Q_{ul} = \frac{k}{d} e^{-\frac{b_0}{T}} \left(\sqrt{P_{mm1}} - \sqrt{P_{mm2}} \right) \quad (5)$$

where P_{mm1} and P_{mm2} are the pressures of the gas in millimeters on the entrance and exit sides of the material, respectively.

In Fig. 1 the dependence of the permeation rate of hydrogen through molybdenum on temperature is shown. Curve 1 shows the results obtained by Smithells^{4,6}. The dotted curves, 2 and 3, are recent results obtained by RCA and Atomics International, respectively. In the case of curve 3, the molybdenum was coated with Durak B, and Q_{ul} values were obtained by measuring the time rate of increase of hydrogen (P_{mm2}) on the exit side of the metal. One atmosphere of hydrogen was present on the entrance side for all three curves. Although the experimental values on curve 3 show a significant reduction in the permeation rate in comparison to the other results, the investigators² do not attribute the decrease to the Durak B coating. They suggest that the solubility of hydrogen in the metal requires more time than was allowed for in the experimental procedure and that this may account for the reduced rate.

FIG.1 PERMEATION RATE AS A FUNCTION
OF TEMPERATURE FOR H₂-Mo

The buildup of hydrogen pressure inside a device where the initial hydrogen pressure was zero can be predicted from the rate values shown in Fig. 1. The time dependence is given by

$$P_{mm2} = \frac{Q_{u1} \times A \times \sqrt{P_{mm1}}}{V \times d \times 10^3 \times \sqrt{760}} \frac{T}{273} t \quad (6)$$

where

P_{mm2} = pressure in mm inside the device

P_{mm1} = pressure in mm of the hydrogen on the entrance side of the material

Q_{u1} = permeation rate at a given temperature on the material

A = area of heated area in cm^2

d = thickness of heated area in mm

V = volume of device in liters

T = temperature of envelope in degrees Kelvin

t = time in minutes.

This equation assumes that Q_{u1} is independent of P_{mm2} , and this should be a reasonable assumption for values of P_{mm2} in the millimeter range if P_{mm1} is an appreciable part of an atmosphere. The potentially rapid buildup of hydrogen, even for the low permeation rates indicated by curve 3 on Fig. 1, is illustrated by putting nominal converter design values in equation (6). For example, at a molybdenum cathode temperature of 1400°C ($Q_{u1} = 0.4$), the pressure dependence would be

$$P_{mm2} = 0.16 t \quad (7)$$

for a 10 cm^2 cathode, $\frac{1}{2}$ mm in thickness, P_{mm1} equal to one atmosphere, and a converter volume of 0.1 liter at an ambient temperature of 250°C .

At the present time it is not known what partial pressure of hydrogen could actually be tolerated before degradation of converter performance occurs. However, the presence of hydrogen could influence the operation of the converter from either electron-hydrogen collisions or heat-conduction-loss considerations. If electric collisions between electrons and hydrogen molecules were the dominant degradation effect, then an upper pressure limit could be obtained from mean-free-path considerations. It is fairly well established that the energy distribution of the electrons in the device is Maxwellian, with an average temperature equal to that of the emitter. For an emitter of 1400°C , the electron energy would then be 0.14 volt. From

scattering measurements of low-energy electrons in hydrogen, reported in the literature,⁷ the probability of collision (P_c) would be equal to a constant value of $50 \text{ (cm - mm Hg)}^{-1}$. The average distance (mean-free-path λ_{el} in cm) that an electron will travel before colliding with a gas molecule is given by

$$\lambda_{el} = \frac{1}{p_0 P_c} \quad (8)$$

where p_0 is the reduced pressure in mm of Hg and

$$p_0 = 273 \frac{p}{T} . \quad (9)$$

For a converter oven temperature of 250°C

$$\lambda_{el} = \frac{T}{273 p P_c}$$

$$\lambda_{el} = \frac{0.038}{p} \text{ cm} . \quad (10)$$

The onset pressure at which degradation would occur would be given by a mean-free-path equal to the diode spacing. For a nominal spacing of 0.020 inch (0.05 cm), the maximum pressure before collision damping would occur would be 0.76 mm or 760 microns. Since the energy of the electron after an elastic collision is proportional to the square ratio of the difference in masses divided by the sum of the masses, a very small change in energy would result. On this basis many collisions could be tolerated that would then increase the allowable hydrogen pressure proportionately.

The power conducted by the gas⁴ can be estimated from

$$P_{cg} = \alpha \wedge_o P_u \frac{273}{T_o} (T_i - T_o) \frac{\text{watts}}{\text{cm}^2} \quad (11)$$

and, for hydrogen

$$\alpha \approx 0.3$$

$$\Lambda_0 \approx 61 \times 10^{-6}$$

P_u = pressure in microns

T_0 = temperature of cold sink in $^{\circ}\text{K}$

T_1 = temperature of source in $^{\circ}\text{K}$.

At an emitter temperature of 1400°C (envelope temperature of 250°C) the power conducted away by hydrogen would be approximately 15 watts per cm^2 . Λ_0 value for cesium could not readily be found in the literature, but, by comparing values quoted for other gases (Ne, N_2 , O_2 , A, Co_2 , and Hg), the cesium value would be less than that of hydrogen by a factor of 4 to 15. Assuming that α is not too much different in the case of cesium, it is seen that the presence of a millimeter of hydrogen would have a pronounced effect on the emitter temperature for a given heat input in consideration of conduction losses.

Several approaches appear potentially capable of reducing or minimizing the permeation of hydrogen. The first and best solution would be to prevent the diffusion of the gas through the emitter surface by using materials and/or coatings that are impervious to hydrogen. This approach will be investigated in an external contractual program.

Another approach would be to include a hydrogen gettering material in the device. Atomics International² is planning on using titanium or a titanium-zirconium alloy in such an approach. Results of previous internal work on reservoir materials⁸ indicate that hafnium-free zirconium would be a better material to use. This material is relatively easy to load with hydrogen in comparison to the rare-earth metals; does not react with conventional electron-tube materials as the rare earth metals do; and, in the α and β regions, the vapor pressure of hydrogen above the metal is independent of pressure for atomic hydrogen concentrations in the metal up to 30 per cent. Titanium shows a rather strong pressure-concentration dependence in the two-phase region, although otherwise its handling and loading characteristics are excellent and well known. Pressure-loading data of zirconium⁸ indicate it should be possible to maintain the hydrogen pressure below 50 microns at ambient temperatures as high as 450°C up to hydrogen concentrations in the metal corresponding to loadings of 60 liter-millimeters per gram.

The effectiveness of the reservoir approach can be illustrated from the following example: using one gram of zirconium occluder in a converter whose volume is 0.1 liter and where the permeation process results in a hydrogen pressure dependence given by equation (7), the time at which the partial pressure of hydrogen would exceed 50 microns would be approximately 62 hours. Although this approach has some merit as an immediate solution, it is a limited solution where long life requirements are desired.

Since there are very few materials in which hydrogen is insoluble at emitter temperatures of current interest, and since it is desirable from

efficiency considerations to operate the converter at temperatures higher than 1400°C , it is expedient to consider the possibility of continuously exhausting the hydrogen as it permeates. After examining the literature for a material suitable as a hydrogen window and compatible with converter operation, it appeared that nickel had many desirable features.

EXPERIMENTAL PROCEDURE AND RESULTS

Some preliminary work was performed to evaluate the permeation characteristics of nickel as a pumping mechanism. A solid nickel rod, 0.5 inch in diameter and $4\frac{1}{2}$ inches in length, was drilled out to give a tube of 0.060-inch wall thickness. One end of the rod was left solid. A kovar section was then brazed to the open end and a glass tubulation was sealed to the kovar. The assembly was sealed to a vacuum station containing a McLeod gauge for measuring pressure and a palladium thimble for admitting hydrogen. An electrically heated oven was used to heat the nickel tube.

The experimental setup is shown in Fig. 2. Hydrogen was admitted to the system (total volume equal to 2.5 liters) and the decrease in hydrogen pressure as a function of time was measured at a constant oven temperature as measured by a chromel-alumel thermocouple.

Fig. 3 shows the result obtained at an oven temperature of 750°C . The system was initially filled to 620 microns of hydrogen and the hydrogen pressure was measured as a function of time. From the slope of this curve the permeation rate can be obtained, and, in Fig. 4, the rate expressed in terms of micron-liters per hour is plotted as a function of pressure in microns. The slope of the line is approximately one-half, which is the theoretical pressure dependence predicted by equation (1). Also shown on this figure are a few points obtained at reduced oven temperatures. Although the lower temperature data appear reasonable in this presentation, insufficient time was allowed for the metal to saturate, and the data are presented mainly to illustrate the temperature dependence.

In order to compare these types of data with results in the literature, it is necessary to know the area and temperature of the nickel used in the experiment. Fig. 5 shows the temperature distribution as measured by platinum-platinum 10% rhodium thermocouples attached to the tube. The thermocouples were located at the following places: 1) center of solid end face; 2) 0.38-inch from the solid end; and 3) 1.25 inches from the solid end. The measured temperature distribution is nearly linear, and, as a first approximation, the area of the tube inside the oven can be assumed to have an average temperature over an area where the temperature drop does not exceed 50°C . This is a fairly good approximation, since, for a 50° change, the rates quoted in the literature differ by approximately a factor of two. Using this approximation, the area and average temperature were determined from Fig. 5, for an oven temperature of 735°C , to be 21 cm^2 at a nickel temperature of 465°C . The 735°C oven curve of Fig. 4 would then give a Q_{ul} equal to 1.5 micron-liters/ $\text{cm}^2/\text{mm}/\text{min}$ at atmospheric pressure. This point is plotted on Fig. 6, which shows the values of Q_{ul} as a function of temperature for molybdenum and nickel, which have been reported in the literature.⁴ For nickel, the solid line was obtained by averaging the rates from five sources as given in the reference. The experimental value measured agrees quite well with the literature.

FIG. 2 EXPERIMENTAL SETUP

FIG. 5
TEMPERATURE PROFILE

FIG. 6 PERMEATION RATE AS A FUNCTION
OF TEMPERATURE

DUSHMAN VACUUM TECHNIQUES PG. 611

The data shown on this curve can be used to design a nickel window to allow hydrogen to pass out of the converter at a rate equal to the input if a residual partial pressure of hydrogen can be tolerated. For example, a molybdenum emitter of 1-cm² area and 1-mm thickness, operating at 1200°C, would have a permeation rate of 8.4 micron-liters per minute if one-half an atmosphere of hydrogen was present on the burner side of the cathode surface. A nickel anode of 3-cm² area and 0.010-inch thickness at 760°C would maintain the partial pressure of hydrogen inside the diode at 1 mm under these conditions.

Table I shows some anode temperatures that would be required to achieve and maintain selected partial pressures of hydrogen inside the converter. In this tabulation it is assumed that the anode area is equal to the cathode area; that one-half an atmosphere of hydrogen is present in the burner area; and that the H₂ permeation rate is equal to that obtained for molybdenum by RCA (Fig. 1, curve 2). For these conditions, only an extremely thin anode would be potentially capable of pumping out the hydrogen permeation so as to maintain a low partial pressure inside the converter. It is anticipated, however, that the permeation rate through a coated emitter will be one or more orders magnitude less than the value for uncoated molybdenum. Also, in many thermionic converter designs the anode area will be significantly larger than the cathode and this will give a more effective pumping system than the equal area assumption used for the calculations shown in Table I. For example, if a 10-mil coating of aluminum oxide is used on the emitter, cathode temperature is 1350° to 1400°C, and a 10-mil-thick nickel anode at 600°C is used with an area ten times greater than the cathode, then the pressure of hydrogen inside the converter will be maintained at approximately 10⁻⁵ mm Hg. In the absence of the window, the hydrogen pressure would rise to 1 mm in approximately 280 hours for a converter with a 10-cm² cathode and a volume of 0.1 liter.

CONCLUSIONS

Analysis of the hydrogen permeation problem in fossil-fuel-heated thermionic converters indicates that a nickel window could maintain the partial pressure of hydrogen inside the device at a level that would not be detrimental to the operation of the device. Initial results of the diffusion of hydrogen through nickel have been obtained and agree reasonably well with values quoted in the literature.

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TABLE I - NICKEL ANODE TEMPERATURE REQUIREMENTS										
Equilibrium H ₂ Pressure in Converter (mm)			0.05			0.02			1.0	
Thickness of Anode (inches)			0.001	0.01	0.02	0.001	0.01	0.02	0.001	0.01
Emitter T°C	Emitter Thickness (inches)									
1200	0.020					568	876	1025	487	730
	0.040	568	876	1025		502	763	876	436	650
1300	0.020					612	962	1134	520	796
	0.040	612	962	1134		540	825	961	462	698
1400	0.020					653	1042	1241	557	856
	0.040	653	1042	1241		574	896	1042	496	747
1500	0.020					698	1135	1367	589	917
	0.040	698	1135	1367		612	970	1135	520	796
1600	0.020					732	1218	1495	620	977
	0.040	732	1218	1495		654	1025	1218	546	844

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PERMEATION OF HYDROGEN THROUGH METALS IN
THERMIONIC-CONVERTER APPLICATIONS, by

J. E. Creedon. January 64, 15p. incl. illus.
table, 9 refs. (USAEIRD Technical Report 2431)
(DA Task 106-22001-A-055-02) Unclassified report

1. Thermionic converters
2. Emitters
3. Permeation

- I. Creedon, J. E.
- II. Army Electronics Research and Development Laboratories, Fort Monmouth, N.J.
- III. DA Task 106-22001-A-055-02

UNCLASSIFIED

Permeation of hydrogen through the emitter surface of a thermionic converter is discussed. Approaches to eliminating a pressure buildup inside the converter are examined, and it is shown that a nickel window could be used to maintain a relatively low partial pressure of hydrogen.

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