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**Westinghouse Electric Corporation
Fusion Power Systems Department**

**EXPERIMENTAL STUDIES OF
ZIRCONIUM/ALUMINUM GETTER PUMPS**

MASTER

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EXPERIMENTAL STUDIES OF
ZIRCONIUM/ALUMINUM GETTER PUMPS

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ABSTRACT

Getters of the type to be utilized as tritium pumps in the foreline of the TFTR torus vacuum system, and presumably in those of the neutral beam injector arms, were evaluated using hydrogen and deuterium. The capacity and speed as rated by the manufacturer (SAES) are found to be conservative. In addition, the getters are shown to continue to pump hydrogen even in the presence of chemically active, impurity gases, provided the getter temperature is maintained at $>300^{\circ}\text{C}$. Since hydrogen pumping is reversible, the getters can be used over and over again after reactivation to release the previously pumped hydrogen. By operating a tungsten filament to crack methane, and using the getter to pump the hydrogen produced, relatively high speed and capacity for methane is demonstrated.

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1.0 INTRODUCTION

The current design of the TFTR tritium handling system uses getter pumps to remove gaseous tritium from the main toroidal vessel and to store it in a solid form. The getters are designed primarily for hydrogen pumping which is reversible and proceeds via adsorption and diffusion into the bulk. Chemically active gases such as oxygen, nitrogen, the carbon oxides, and water vapor are pumped irreversibly forming highly stable oxides and nitrides. Because adsorption is a precursor to diffusion into the bulk, species such as the inert gases and methane which do not adsorb are not pumped.

These pumps consist of a zirconium alloy⁽¹⁾ and serve as backing pumps for the large mercury diffusion pumps which compose the primary pumping system on the toroidal device. In addition to and in conjunction with the getters, "crackers" in the form of incandescent filaments will also be incorporated to dissociate tritium from gaseous hydrocarbons which may be formed. The purpose of this report is to present experimental results on a getter/cracker system under conditions which simulate the most severe conditions anticipated in TFTR operations. While the experiments were not carried out with tritium, the processes involved are dominated by chemical characteristics so that the data obtained using hydrogen and deuterium can be expected to be applicable to tritium.

2.0 EXPERIMENTAL DETAILS

The experimental arrangement is shown schematically in Fig. 1. The system was constructed using 304 stainless steel, ultrahigh vacuum valves, and Conflat⁽²⁾ flanges. This system is connected via a 2.5 cm valve and defined orifice (0.1 mm diam. hole in a 0.56 mm thick copper disc having a conductance of 1.75×10^{-4} liter/sec for air at 25°C) to a small ultrahigh vacuum system equipped with a mass spectrometer. High purity hydrogen is supplied from "boil off" of liquid hydrogen. The hydrogen is pulsed into the system using two compressed-air-actuated valves⁽³⁾, controlled by two recycling electric timers. A vacuum regulator⁽⁴⁾ is used to maintain a constant hydrogen pressure of ~ 100 Torr at the first air-actuated valve. With the volume between the valves equal to 10 cc, a fixed dose of ~ 1 Torr liter is admitted to the system by sequentially closing the first valve, V_1 , and opening the second, V_2 . The 100 cc buffer volume between the vacuum regulator and V_1 is provided to assure that the 10 cc volume between V_1 and V_2 is filled to a reproducible pressure during cyclic operation. Hydrogen doses were admitted once a minute for almost all of the data obtained, but in several tests the dosing was done at five minute intervals to duplicate the planned TFTR operation and to check for any unexpected differences in getter performance.

The volume of the system, ~ 20 liters, was chosen such that the maximum pressure at which the getter would operate would equal the maximum anticipated in TFTR at full power operation; i.e., for a 2.5 MA plasma the total quantity of hydrogen isotopes to be pumped, including that introduced via neutral beam injection, would be 50 Torr liter which would be pumped into the 4 foreline volumes having a total volume of nearly 1000 liters. Table 1 gives the important parameters of the present study together with those expected in TFTR. Note that we are scaling by a factor of about 1000 except that in the present work the getter is required to pump a much larger fraction of its total capacity per dosage.

The spent plasma gas in TFTR can be expected to contain some carbon monoxide and methane,

and the effects of these gases on the getter performance were a part of this study. Because any methane or other gaseous hydrocarbons produced in TFTR may contain tritium, it is highly desirable to remove these gases in a manner similar to that used for tritium. Consequently, methane was used to examine the effectiveness of an incandescent tungsten filament as a methane cracker used in conjunction with the getter for pumping of the resulting hydrogen. The impurity gases are introduced from reagent grade flasks via a leak valve⁽⁵⁾. The tungsten filament for methane cracking is mounted on the same feedthrough flange as the getter. The filaments used are of the coiled-coil design such as are used in 100 W incandescent light bulbs and consist of 585 mm of 0.064 mm diam. wire. The temperature is maintained at 2500°K using 100 V ac.

A getter cartridge was mounted by spot-welding to a demountable flange equipped with electrical feedthroughs and was surrounded by a radiation shield 2.6 cm diam. made from 0.075 mm tungsten sheet. This shield does not affect the pumping speed of the getter; it does result in a more uniform getter temperature and enables reliable temperature measurements to be made using a thermocouple inserted between the "pleats" of the getter (see Fig. 2). An axially mounted heater in the getter cartridge is used for operation at elevated temperatures and for reactivation of the getter. Current and voltage monitoring of this heater input permits reproducible operation at selected temperatures. For most of the data reported below, a fixed heater current was used to give a temperature of ~300°C. Actual thermocouple measurements gave values from 310°C to 325°C.

Pressure measurements are made using a capacitance manometer⁽⁶⁾, Bayard-Alpert type ionization gauge⁽⁷⁾, and a mass spectrometer. The capacitance manometer is particularly convenient because of its range, 10^{-4} to 10 Torr, and because it is an absolute gauge whose output is independent of gas species. The ionization gauge (B.A.G. in Fig. 1) is primarily used to measure lower pressures, 10^{-6} to 10^{-7} Torr, obtained during the preparation of the system. The mass spectrometer provides continuous monitoring of partial pressures via a continuous "leak" through the defined orifice separating the two portions of the overall system.

After installation of a getter and pumping down to $\sim 10^{-5}$ Torr with a liquid-nitrogen-trapped rotary mechanical pump⁽⁸⁾, the getter is activated. This initial activation results in the release of some gas and diffusion of chemisorbed gases into the bulk of the getter. Because hydrogen sorption by the getter is reversible, the previously pumped hydrogen is released during reactivation of the getter. This hydrogen is removed from the system by the liquid-nitrogen-trapped mechanical pump. Reactivation is carried out at 875°C for approximately one hour after which the pressure is $\sim 1 \times 10^{-4}$ Torr. At this point the getter heater power is removed and the pressure falls rapidly into the 10^{-6} Torr range in ~ 2 min. The valve to the trap and pump is then closed and the getter continues to cool. The pressure bottoms out, typically at $\sim 2 \times 10^{-6}$ Torr, while the getter is still quite hot, $\sim 500^{\circ}\text{C}$. Further decrease in the base pressure is minor and occurs very slowly over many hours. During activation of the getter, the walls of the 18 liter tank, which is ~ 25 cm diam. $\times$ 35 cm long, are non-uniformly heated by radiation to a maximum of $\sim 55^{\circ}\text{C}$.

A total of six different getter cartridges were employed in this study. All were of the same manufacturing lot and nominally identical. Their size and construction are indicated in the photograph of Fig. 2. The zirconium/aluminum getter powder is mechanically embedded on iron sheet, which is then bent into the pleated cylinder shown in the photo by SAES. The heater and glass mounting press is added by the Electron Tube Division of Westinghouse in Elmira, New York

3.0 EXPERIMENTAL RESULTS

Data was obtained on the getter performance at several temperatures in pure hydrogen, hydrogen with controlled quantities of carbon monoxide and methane, and in pure deuterium. In addition, the effectiveness of an incandescent tungsten filament for methane cracking and the resulting overall pumping of methane by the cracker/getter combination was examined.

3.1 PUMPING OF PURE HYDROGEN

The rated capacity for hydrogen pumping for the getters used here is 6 Torr liter/gm of getter alloy and the maximum capacity permitted to avoid flaking of getter material off the substrate is 15 Torr liter/gm⁽⁹⁾. For the AP-10 getter cartridges employed in this study, the above mentioned capacities are 60 and 150 Torr liters of hydrogen, respectively. Provided the hydrogen is pure (estimated chemically active impurities $\lesssim 10$ ppm), the getters pump the rated 60 Torr liters of hydrogen at high speed, even at room temperature. Figure 3 shows the pumpdown curves obtained under these conditions following introduction of hydrogen in ≈ 1.2 Torr liter quantities. Following a run to $\sim 150\%$ of the rated capacity (75 such doses of hydrogen, i.e., 90 Torr liter) the getter is reactivated (875°C for 1 hour) with the desorbed hydrogen being pumped away by the liquid-nitrogen-trapped mechanical pump. Two getters were run to 150% rated capacity and reactivated for 10 such cycles each. The performances, as measured by the pumping speed and equilibrium hydrogen pressure, were found to be identical for each successive run. It is clear from the results in the presence of carbon monoxide impurity presented below, that such behavior with the getter at room temperature can only be obtained for relatively pure hydrogen. In Fig. 3 it is seen that the getter pumping speed decreases from ≈ 30 liter/sec, the rated speed at 400°C , to ≈ 15 over the course of a run. This decrease observed with the getter at room temperature is attributed to the presence of trace impurities which chemisorb on the getter surface. While in pure hydrogen the pumping speed is determined by the diffusion rate of hydrogen into the bulk, chemisorbed species such as oxygen and nitrogen in effect reduce the area of the getter available to hydrogen sorption with

the result that the rate determining step in hydrogen pumping is its adsorption. By maintaining the getter at an elevated temperature the chemisorbed species also diffuse into the bulk so that hydrogen sorption is not impaired. This is demonstrated by the data of Fig. 4 in which the getter is operated at 325°C and the hydrogen speed remains in excess of the rated speed.

3.2 PUMPING HYDROGEN IN THE PRESENCE OF CARBON MONOXIDE IMPURITY

Carbon monoxide impurity can be expected to occur in TFTR as it invariably does in virtually every vacuum system. Consequently, a constant influx of carbon monoxide was established into the getter system and hydrogen again introduced in approximately 1 Torr liter doses. The rate of carbon monoxide influx, 1.5×10^{-4} Torr liter/sec, was chosen such that it would scale to nearly 100 x that which the getters in TFTR would be exposed to (an average of 1.7×10^{-3} Torr liter/sec for a 1% concentration in the spent plasma gas) and at the same time would be well below the rated carbon monoxide capacity of the small getter cartridges used here, ≈ 6 Torr liter per activation or 35 Torr liter total. Note that the rated capacity for carbon monoxide (and other chemically active gases) are much less than for hydrogen and that such gases are not released upon reactivation.

An objective of the present work was to operate the getter at the minimum required temperature to pump hydrogen in the presence of carbon monoxide. Virtually no hydrogen pumping was measured at a temperature of 30°C and severely limited speed and capacity were found at 210°C. However, the results at 310°C were indistinguishable from those found at 410°C. In each of ten successive runs at 310°C, the getter pumped ~ 90 Torr liters of hydrogen and $\lesssim 1$ Torr liter of carbon monoxide. Figures 5a and 5b show the hydrogen pressure as a function of time for the first and last of these 10 runs. The pumping speed for hydrogen is seen to decrease during the course of a run as well as with increasing number of runs in carbon monoxide. However, no decrease in hydrogen capacity as measured by the hydrogen equilibrium pressure was detectable. In spite of the significant decrease in hydrogen speed in the presence of the carbon monoxide influx and the simultaneous pumping of carbon monoxide, the hydrogen speed remains much

higher than required for the current TFTR design. If the lowest hydrogen speed measured here, viz., 4 liter/sec, is extrapolated to the large, C-500 SAES getter cartridges in the TFTR design, the resultant speed is still two orders of magnitude larger than that required for pumpdown between pulses.

3.3 HYDROGEN PUMPING WITH METHANE IMPURITY

Methane pumping by these getters occurs only as the result of a small amount of cracking by the coaxial heater. With the getter maintained at 310°C we obtain speeds $\sim 3 \times 10^{-3}$ liter/sec (1.5×10^{-5} liter/sec cm^2). For comparison, SAES literature gives a methane speed of 5×10^{-2} liter/sec for this size getter when run at 400°C at which the heater temperature is correspondingly higher. In either case, the speed is very low because methane does not chemisorb and therefore does not dissociate or diffuse into the getter at these low temperatures. Our interest in this particular aspect of the getter evaluation was in the ability of the getter to pump hydrogen in the presence of impurities and not primarily in pumping the impurities. Consequently, the ≈ 20 liter volume in which the getter is located was filled with methane to a pressure of 1.5×10^{-2} Torr prior to the normal hydrogen dosing. The getter was maintained at 310°C for these runs and again 10 successive runs were taken. Some decrease in hydrogen speed was found, as can be seen from Fig. 6. This is not significant for the intended TFTR application and has not been investigated.

3.4 HYDROGEN PUMPING WITH MECHANICAL PUMP VAPOR IMPURITY

A getter at room temperature was exposed to the vapors of an untrapped mechanical pump for approximately 15 hours. Subsequently, the getter was isolated from the pump and exposed to the normal hydrogen pulses as well as 1.5×10^{-2} Torr of methane. The getter was incapable of pumping the hydrogen until its temperature was raised to 310°C for about 15 min. Hydrogen pumping then proceeded normally.

3.5 PURE DEUTERIUM PUMPING

Since there is no data available for the isotopic effect with zirconium/aluminum getters, a few runs were taken using pure deuterium. A paper from SAES¹⁰ quotes data comparing pumping speeds of hydrogen and deuterium by zirconium and aluminum⁽¹¹⁾ and tacitly suggests those results may be applicable to their getter. It was found in that study⁽¹¹⁾ that, instead of the ratio of pumping speeds for deuterium and hydrogen being in the ratio of the square roots of the masses, i.e., 0.71, the experimental value was 0.56. There was little effort in the present work to obtain highly accurate pumping speed measurements since the speeds are orders of magnitude higher than required for the foreseeable applications. However, speeds were obtained for deuterium and hydrogen under the same conditions of temperature (325°C) and gas loading. Figure 7 shows representative data for the two isotopes. The speeds, approximately 45 and 30 liters/sec for hydrogen and deuterium, respectively, yield a ratio closer to that expected than to that reported in Ref. 11. It should be noted that impurities can decrease the speed significantly as demonstrated here with carbon monoxide (see Fig. 5).

3.6 PUMPING OF METHANE BY CRACKING

A tungsten filament can be operated at high temperature to effectively crack methane to hydrogen which is then pumped by the getter. Several experiments were conducted using coiled-coil tungsten filaments to explore this method for removing tritium from methane. A filament was mounted from the same flange as was the getter with the axes approximately co-linear. The filament was operated at $\approx 2500^\circ\text{K}$, the getter at 315°C , and a defined influx of methane was established. The overall pumping speed is given by the quotient of the known methane influx and the measured equilibrium pressure. As in the other experiments, the gas composition is obtained using the mass spectrometer. The resulting pumping speed for methane is ~ 10 liter/sec. The total quantity which can be pumped is limited by the filament life. Further, the filament life appears to be strongly affected by the methane pressure in which it is operated, i.e., if the filament is turned on at relatively high pressure, $\sim 5-10 \times 10^{-3}$ Torr,

arcing between turns of the filament leads to filament burn out. This problem, presumably, could easily be avoided by utilizing filaments of larger diameter which would operate at the same temperature at lower voltage. With the filament at $\approx 2500^{\circ}\text{K}$, the getter at 315°C , and a methane influx of $\approx 1 \times 10^{-3}$ Torr liter/sec, up to 90 Torr liter of methane can be pumped. Since two hydrogen molecules are assumed to be formed, and pumped by the getter, for each methane molecule cracked at the incandescent filament, the getter sorbs up to 180 Torr liter of hydrogen or twice the quantity pumped in the hydrogen experiments discussed above. Thus, the capacity for methane pumping by this method is limited by the capacity of the getter for hydrogen. No flaking of the getter was evident, even at the highest, 180 Torr liter, loading of hydrogen.

An analysis for carbon was performed on two filaments run to failure. The carbon contents were found to be 1.09 and 1.55 percent by weight, corresponding approximately to W_5C , well below complete carbiding.

The incandescent filament temperature was varied over the range $2100\text{--}2500^{\circ}\text{K}$ with the getter temperature constant at 315°C . The results are shown in Fig. 8 where the resultant pumping speed is plotted against Inverse temperature. The dotted line at 18.5 liter/sec represents the maximum speed which would be obtained if every incident methane molecule were cracked into two hydrogen molecules which were subsequently and instantaneously removed by the getter. The slope of a line fitted to the experimental points obtained at filament temperatures below the knee of the curve gives an activation energy of 120 kcal/mole for the cracking reaction.

By removing the getter, operating the filament alone, and recording the changes in methane and hydrogen partial pressures, the effect of the filament alone can be demonstrated. Figure 9 clearly shows the conversion of methane to hydrogen. The system volume was filled with methane to a pressure of 1.5×10^{-2} Torr prior to heating the filament. Operation of the filament

at $\approx 2500^\circ\text{K}$ results in a reduction of the methane partial pressure to essentially zero. Simultaneously, the hydrogen partial pressure rises. The hydrogen pressure would be expected to rise to 3×10^{-2} Torr. The fact that a smaller increase, to 2.3×10^{-2} Torr, is observed is attributed to pumping of atomic hydrogen at the walls of the chamber, and to experimental uncertainty, more specifically to lack of precision for the relative sensitivities of methane and hydrogen in the mass spectrometer during this particular portion of the study.

3.7 GETTER TEMPERATURE RISE WITH SORPTION

At the typical hydrogen loading rates used here, ~ 1 Torr liter/min, the getter temperature was found to increase approximately 10°C . When carbon monoxide and methane were present as impurity gases at the levels used in this work, this temperature rise was not measurably affected, although in the case of carbon monoxide one could expect an additional temperature increase if more of this impurity gas were present and pumped per unit time.

After pumping hydrogen to 150 percent of rated capacity, the getters were sometimes exposed at room temperature to room air. The temperature rise under such conditions was measured to be 4°C . The reason for this measurement was to assure that there is no explosive danger in exposing a getter saturated with hydrogen to ambient. No test was performed to explore the possibility of tritium release upon exposure to ambient which may occur as a result of isotopic exchange or simple displacement.

3.8 HISTORY OF GETTERS USED

Three getters were loaded to ~ 700 Torr liter of hydrogen, i.e., more than ten times the rated capacity. Under such condition flaking of some getter material off the substrate was observed. This is expected since embrittlement occurs when the limit of solubility (approximately one hydrogen atom per zirconium atom) is approached as is the case with sorption of 700 Torr liter of hydrogen with the small getter cartridges used here. Under all other conditions in which the maximum hydrogen sorbed was ≤ 200 Torr liter no flaking was found.

One getter was used for 10 runs in pure hydrogen with 90 Torr liter sorbed per run. It was then removed from the system and left exposed to room air for 26 days. After re-installing in the system, it was used for 24 additional runs in which hydrogen was pumped in the presence of methane impurity. A total of 34 activations were carried out, each at 875°C for one hour. No deterioration of the getter was evident from either the sorption data or from its physical appearance. A second getter was used for a total of 24 runs pumping hydrogen to 150 percent rated capacity. In half of these runs, carbon monoxide was present as an impurity. The getter was reactivated a total of 27 times and again no deterioration was detected. A third getter was used for only 7 runs in hydrogen and deuterium but was used for much of the methane work. In that portion of the work it pumped larger quantities of hydrogen, up to 180 Torr liter, cracked from methane. In addition, it was exposed to ambient some 20 times in the process of replacing filaments, etc., and was reactivated 25 times. It, too, showed no deterioration. Based on the present results obtained under rigorous test conditions we concluded that the getters are capable of withstanding the projected service in TFTR.

4.0 CONCLUSIONS

The SAES getter alloy provides an excellent means for pumping hydrogen and its isotopes. In particular, the conversion of tritium from the gaseous phase to a solid form having a low equilibrium pressure is a practical means for safely handling this gas. Use of an incandescent cracker to dissociate gaseous light hydrocarbons such as methane, in conjunction with the getter, is demonstrated to be an effective and practical means of pumping such impurities. Operation of the getter above room temperature, specifically at $\geq 300^{\circ}\text{C}$, is necessary to prevent passivation of the getter for hydrogen pumping by chemisorbed gases.

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TABLE 1
SIMULATION OF GETTER REQUIREMENTS FOR TFTR

	<u>THIS WORK</u>	<u>TFTR DESIGN</u>
Getter Cartridges	one AP-10	16 C-500
Cartridge Size (cm)	2 diam. x 3	10 diam. x 13
Total Getter Area (cm ²)	1.7×10^2	2×10^5
Total Getter Mass (gm)	10	6.4×10^3
Temperature (°C)	30-400	325
Maximum Pressure (Torr)	5×10^{-2}	5×10^{-2}
Fraction of Rated Capacity Used for Pulse	1/60	1/800

Figure 1 Getter evaluation system consisting of the primary system, in which the getter is located in an 18 liter volume, and the mass analysis system which can sample gas from the primary system via a calibrated conductance, 6.6×10^{-4} liter/sec for H₂ at 25°C. Bayard-Alpert ionization gauges (B.A.G.) and a capacitance manometer are used for total pressure measurements. The operation of the air actuated valves V₁ and V₂, which admit hydrogen to the primary system in doses, is controlled by electric cyclic timers.

Figure 2 Photograph of one of the getter cartridges used in this study

Figure 3 Pumpdown by the getter, at room temperature, following pulses of ~ 1 Torr liter of H_2 at $t = 0$. The two curves show the initial faster speed (≈ 30 liter/sec at the third H_2 pulse) and the slower speed (≈ 15 liter/sec) which occurs after the getter has absorbed $\approx 150\%$ of its rated H_2 capacity (75 pulses, i.e., 90 Torr liter). The reduction in speed for the getter at room temperature results from adsorption of impurities during the course of the run, 75 min. total time.

Curve 684294-A

Figure 4 Pumpdown by the getter, when maintained at 325°C , following pulses of ~ 1 Torr liter of H_2 at $t = 0$. Comparison with Fig. 3 shows little effect on the speed by impurity sorption during the course of the run. The data for the third and the 75th pulses are shown. The H_2 speed is ≈ 45 liter/sec.

Figure 5a Pumpdown of H_2 by the getter, maintained at $310^\circ C$, in the presence of a continuous CO leak of 1.5×10^{-4} Torr liter/sec. The three curves show the progressively lower pumping speed from 30 to 15 liter/sec during the course of the run to 150% rated H_2 capacity. The data for the 3rd, 38th, and 75th pulses are shown.

Figure 5b Conditions are identical to those for the data of Fig. 5a except that the total quantity of CO pumped by the getter has increased from ≈ 1.6 Torr liter to ~ 9.6 Torr liter. Again the three curves show the progressive decrease in pumping speed from 25 to 4 liter/sec during the run. This is the twelfth such run to 150% rated H_2 capacity in the presence of CO. The getter was reactivated to $875^\circ C$ for 1 hour between runs.

Figure 6 Pumpdown of H_2 by the getter, maintained at $310^\circ C$, in the presence of a constant methane pressure of 1.65×10^{-2} Torr. The three curves show the small decrease in pumping speed during the course of the run from the 3rd to 38th to 75th H_2 pulse. The H_2 speed, ≈ 15 liter/sec, is about one third that in pure H_2 , cf., Fig. 4

Figure 7 Comparison of pumpdown curves for H_2 and D_2 with the getter at 325°C . The speeds are ≈ 45 and 30 liter/sec, respectively.

Figure 8 The overall pumping speed of the cracker/getter combination is plotted against inverse temperature. The data points and error bars represent the average values and standard deviations for approximately ten sets of data obtained using different but nominally identical tungsten filaments. The dashed horizontal line at 18.5 liter/sec corresponds to the maximum possible speed. The difference between this speed and that at the plateau of the experimental curve suggests that the process of accommodation and dissociation of methane at the incandescent surface occurs at a maximum efficiency of $\approx 50\%$. Previous measurements of hydrogen pumping by the getter in the presence of methane show the getter speed is high enough that it is not the limiting factor. The slope of the data points at the three lowest filament temperatures given an effective activation energy of 118 kcal/mole (5 eV/reaction).

Figure 9 Conversion of methane to hydrogen. Upon operating a tungsten filament having an area of 1.18 cm^2 at 2500°K , 1.5×10^{-2} Torr of methane in a 20 liter volume is converted to hydrogen.