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TECHNICAL NOTE

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STABILITY OF TITANIUM NITRIDE AND TITANIUM CARBIDE WHEN EXPOSED TO HYDROGEN ATOMS FROM 298° TO 1950° K

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SUMMARY

Titanium nitride and titanium carbide deposited on tungsten wires were exposed to hydrogen atoms (10^{-4} atm pressure) produced by the action of microwave radiation on molecular hydrogen. The results of these experiments in the temperature range 298° to 1950° K indicate that no appreciable reaction takes place between atomic hydrogen and TiN or TiC. The formation of reaction products (NH_3, CH_4, C_2H_2) should be favored at lower temperatures. However, because of the high catalytic activity of Ti for H atom recombination, the rate of such reactions with H atoms is controlled by the rate of evaporation of Ti from the surface, this rate being low at temperatures below 1200° K.

In order to interpret the stability of TiN and TiC in H atoms more fully, the stability of TiN and TiC in vacuum and H_2 gas was also studied. The thermodynamic computations conform in order of magnitude to the experimentally found rates of decomposition of TiN and TiC in vacuum and are also consistent with the fact that no appreciable reaction is found with these compounds in molecular H_2 at a pressure of 10^{-3} atmosphere in the temperature range 298° to 2000° K. When TiN or TiC was heated in atomic H or molecular H_2, no reaction products other than those obtained from the simple decomposition of the nitride and carbide were observed. The gaseous products were analyzed in a mass spectrometer.

INTRODUCTION

Because of the growing importance of rocket propulsion and space technology, interest in the chemical and physical properties of high-melting-point materials has increased in recent years. In the search for materials useful at high temperature, one must be concerned with, in addition to the mechanical properties, the stability of these materials in various gases that are present in the exhaust of missiles and in the upper atmosphere. With this in mind, a great deal of research has been done on the stability of refractory compounds such as the borides, carbides, and nitrides of titanium, hafnium, and tantalum.

This paper is concerned with the decomposition of titanium nitride and titanium carbide in an environment of hydrogen atoms. The stability of these compounds was experimentally determined between 298° and 1950° K and substantiated by calculations using recent thermodynamic data. An interesting aspect of this study is the thermodynamic calculation and experimental determination of the rates of decomposition of TiN and TiC in vacuum and in H_2 gas at 10^{-3} atmosphere.

EXPERIMENTAL PROCEDURE

Preparation of Materials

The samples of TiN and of TiC were in the form of crystalline deposits grown onto tungsten filaments from the gaseous phase. Tungsten wires (0.01-in. diam. and about $2\frac{1}{2}$ in. long) were U-shaped and attached at each end to heavy molybdenum leads. The method used for the preparation of TiN was similar to that used by Agte and Moers (ref. 1). A mixture of H_2 and N_2 in the volume ratio 1:3 was saturated with $TiCl_4$ vapor at 30° C and passed over the tungsten wire at about 1400° C. The titanium carbide deposits were made in a similar fashion (ref. 1) by passing a mixture of H_2 and CH_4 in the volume ratio 10:1 saturated with $TiCl_4$ vapor at 30° C over tungsten wire at about 1500° C. After about 0.1 gram of material had been deposited, the filament together with the two Mo leads was removed from the apparatus, immersed in water several times to clean the surface, and allowed to dry in air.

Apparatus and Procedure

A diagram of the test apparatus is shown in figure 1. Pure H_2 after passing through a Deoxo unit to remove traces of O_2 entered the system through a flowmeter and passed over water at 20° C in the trap. (Water vapor must be present if H atoms are to be formed.) The flow rate was adjusted by the needle valve so as to maintain the desired pressure in the test chamber, the system being open to a vacuum pump (264 liters/min). The H atoms were produced by exciting the H_2 (flowing through a 10-mm-diam. quartz tube) with microwave radiation (1000 w at 2450 megacycles). The 10-millimeter quartz tube was positioned in a slot of the tapered matching section of the wave guide.

In order to determine the number of H atoms that would strike the sample, a platinum filament (2 cm long, 0.011 in. wide, and 0.005 in. thick) was substituted for the TiN or TiC sample. The temperature of the Pt filament in contact with H atoms was determined by measuring accurately the electrical resistance of the filament. The number of moles of H atoms striking and recombining on the filament per minute N was calculated by the following equation (see table I):

$$N = 14.3 \frac{P}{\Delta H}$$

where P is power in watts required to heat the filament electrically to the same temperature attained when the filament was heated by the recombining H atoms, and ΔH is the heat of recombination of the H atoms (52,089 cal/mole). Using the Knudsen equation discussed later in this report, the number of H atoms can also be described in terms of pressure (table I), given the area A of the object being struck by them.

For a typical run involving H atoms, the quartz reaction tube was first cleaned with 5 percent HF followed by a wash with dilute phosphoric acid. The excess acid was poured out, and the remainder was allowed to dry in the tube. The presence of a thin layer of phosphoric acid on the tube is claimed to render the quartz inactive as a catalyst for H atom recombination, according to J. W. Linnett, of Queens College, Oxford. The previously weighed sample was then inserted into the reaction tube as shown in figure 1. The system was evacuated and then filled with H_2, the process being repeated. Next, the H_2 flow rate was adjusted with the needle valve to correspond to a pressure of about 0.7 millimeter of mercury in the reaction tube. The pressure that corresponds to a given reading on the flowmeter was obtained from a calibration of the flowmeter against a McLeod gage. With the H_2 flowing through the system, the microwave discharge was turned on and the sample was raised to the desired temperature by passing an electric current through the coated wire. This temperature was measured by viewing with an optical pyrometer of the disappearing filament type, corrections being made for the viewing system (ref. 2) and for the emissivity of the TiN and TiC (the emissivity for both the nitride and the carbide being taken as 0.50, an estimate from the emissivity of similar materials). In general, the duration of each experiment ranged from 20 to 30 minutes, after which time the filament current was turned off. When the filament containing the coated sample had cooled, the discharge was also turned off.

The activated carbon trap immersed in liquid nitrogen was then closed off and the remainder of the system was filled with argon. This trap was then removed and connected to a Consolidated Model 21-620 mass spectrometer. As the trap was warmed, the gas evolved was passed into the mass spectrometer for analysis.

Next, the filament was removed from the reaction chamber and examined, and the weight change was determined. The rate of evaporation of Ti (g/(sq cm)(sec)) was then calculated (table II). The surface area of the TiN or TiC deposit was determined from the total weight of TiN or TiC, its density, and the geometry of the sample. The assumption was made that the sample had a smooth surface. Deposits, if any, on the cooled wall of the reaction tube were dissolved in aqua regia and analyzed for titanium. In some cases when a change in color of the crystalline deposit on tungsten wire was observed, X-ray and electron-diffraction patterns were obtained, but these gave no indication of reaction products.

Experiments designed to determine the stability of TiN and TiC in molecular H_2 were carried out in a way similar to those with H atoms, with the exception that the H_2, instead of being passed over water before entering the reaction tube, was dried in a liquid-N_2 trap. The weight-change data are given in table III. In the experiments used to determine the stability of TiN and TiC in vacuum, the same apparatus previously described was used. For these runs, no activated carbon was used in the trap between the reaction tube and the pump. The system was pumped down until the reading of the thermocouple gage indicated that the pressure of the system was a micron of mercury or less. The sample was heated electrically to the desired temperature. The rapidity at which the material sublimed and condensed onto the cooled wall of the reaction tube determined the duration of the experiment (from 2 min to several hours). The rate of decomposition was calculated from the weight loss in the sample (table IV) and compared with the amount of Ti condensed on the wall of the reaction tube.

THERMODYNAMIC CALCULATIONS

In order to discuss to better advantage the most probable behavior of TiN and TiC when exposed at high temperatures to H atoms, H_2, or vacuum, thermodynamic calculations were made to determine the equilibrium partial pressures of possible reaction products under the conditions of the experiments. By application of the Knudsen equation, the rates of evaporation of Ti from TiN and TiC were also estimated. These calculations were made possible with the aid of a recent compilation of high-temperature thermodynamic data published by the Bureau of Mines (ref. 3).

The first step was to calculate from these data the values of free-energy changes ΔF for the various reactions as listed in table V. The equilibrium constants K_f were derived from the free-energy values using the following well-known equation:

$$\ln K_f = -\frac{\Delta F}{RT}$$

where R is the gas constant and T is absolute temperature. The partial pressures p of the reaction products were calculated in the usual manner from the values of the equilibrium constants. In these computations it was assumed that solid components were at unit activity and that the activities of gaseous products under the conditions of the experiment were equal to their partial pressures.

The rate of evaporation of TiN and TiC expressed in terms of the rate of evaporation of Ti from the sample was then calculated from the partial pressure of Ti above its nitride or carbide at equilibrium by using the Knudsen equation (ref. 4):

$$G = ap \sqrt{\frac{M}{2\pi RT}}$$

where G is the rate of evaporation in vacuum (g/(sq cm)(sec)), M is molecular weight of the species in the gas phase, and a is the accommodation coefficient. This accommodation coefficient a represents the ratio between the rate at which molecules actually condense on the surface and the rate at which they strike the surface. Langmuir has shown that for metal atoms condensing on the surface of a metal the factor a may be assumed to be equal to 1 (ref. 4). Hoch, Blackburn, Dingledy, and Johnston have shown that the same assumption is justified for the evaporation of carbon from TaC and WC (ref. 5). The values of the rates of evaporation of titanium as given in table V were determined using a value of unity for the factor a, thereby setting a maximum value for the evaporation rate.

DISCUSSION

Based on the experimental results (tables II, III, and IV) and substantiated to some extent by the calculated values (table V), it is concluded that neither TiN nor TiC reacts appreciably with atomic or molecular hydrogen at 10^{-4} and 10^{-3} atmosphere, respectively, in the temperature range 298° to about 2000° K. The probable reaction products of such reactions (NH_3, N_2H_4, CH_4, and C_2H_2) could not be detected by means of a mass spectrometer. A comparison of the experimental results with calculated values is shown on a plot of rate of evaporation of Ti against temperature (fig. 2). According to thermodynamic calculations (table V), reaction of TiN and TiC with H atoms is favored at lower temperatures where the probable reaction products are not dissociated to any great extent, while at higher temperatures simple decomposition is the predominant process. The fact that no reaction of TiN or TiC with H atoms was detected at lower temperatures may be attributed to the fact that a thin film of Ti metal, which probably forms on the surface during such a reaction, is a good catalyst for H atom recombination (ref. 6); this metal film would simply promote the recombination of the atoms before they had a chance to react further with the nitride or carbide underneath. In view of this, the rate of reaction would be controlled by the rate of evaporation of Ti from the surface. As shown in table V(n) and figure 2, this rate is quite low at the lower temperature.

The experiments with H atoms are complicated by the fact that the presence of a small quantity of water is required for the production of satisfactory yields of atoms. Ti, TiN, and TiC have strong affinities for oxygen; thus, there is the possibility that oxidation of these materials by a small amount of water (2.3 percent) in the gas stream exerted some influence on the results of these experiments.

The calculated rate of evaporation of TiN and TiC in a vacuum represents a maximum value of this parameter. The actual rate could be somewhat less. In the calculations, the accommodation coefficient in the Knudsen equation was taken as unity, whereas in actuality the accommodation coefficient may have a value less than one.

With regard to the accuracy of the experimental values, the results represent an order of magnitude rather than an exact value. It should be noted that the values of the loss of Ti from the sample depend essentially on the weight loss of the sample that could be obtained to only two significant figures. Furthermore, the experimentally determined rates of evaporation depend upon an estimation of the exposed surface area of the sample. There may be some discrepancy between the estimated value and the true surface area.

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TABLE I. - HYDROGEN ATOMS RECOMBINING ON PLATINUM FILAMENT AS FUNCTION OF H_2 PRESSURE

[H_2 saturated with water at 20° C.]

Pressure of H_2, mm Hg	H atoms, moles/min
0.8	[a]4.16×10^{-4}
1.0	4.32×10^{-4}
2.0	5.09×10^{-4}

[a] H atom press. on Pt filament, about 10^{-4} atm.

TABLE II. - RATE OF DECOMPOSITION IN H ATOMS

[H atoms, 4.16×10^{-4} mole/min equivalent to 0.07 mm Hg; 2.3 percent H_2O in gas stream.]

Material	Temp., °K	Time, min	Weight loss, g	Rate of weight loss of Ti, g/(sq cm)(sec)
TiN	298	30	0	0
	1383	30	0	0
	1644	30	0	0
	1962	20	.0070	2.86×10^{-6}
TiC	298	180	0	0
	1216	150	0	0
	1894	20	0	0
	1940	20	0	0

TABLE III. - RATE OF DECOMPOSITION IN DRY H_2

[H_2 pressure, 0.7 mm Hg.]

Material	Temp., °K	Time, min	Weight loss, g	Rate of weight loss of Ti, g/(sq cm)(sec)
TiN	1645	12	0	0
	1966	10	.0064	5.2×10^{-6}
	2120	10	.0299	2.44×10^{-5}
TiC	1650	30	0	0
	1970	30	0	0

TABLE IV. - RATE OF DECOMPOSITION IN VACUUM

Material	Temp., °K	Time, min	Weight loss, g	Rate of weight loss of Ti, g/(sq cm)(sec)
TiN	1245	1080	0	0
	1428	120	0	0
	1567	70	0	0
	1588	90	.0004	4×10^{-8}
	1779	10	.0007	6×10^{-7}
	1891	10	.0038	3.1×10^{-6}
	2012	10	.0072	5.9×10^{-6}
	2209	2	.0133	5.44×10^{-5}
TiC	1650	25	0	0
	1903	25	0	0
	2139	20	.0007	2.9×10^{-7}
	2389	20	.0073	3.1×10^{-6}

TABLE V. - VALUES DERIVED FROM THERMODYNAMIC DATA PERTINENT TO TiN OR TiC IN VACUUM, H_2, AND H ATOMS

(a) $TiN \rightleftarrows Ti(s) + \frac{1}{2} N_2$

Temp., °K	$Log_{10}K_f$	Vapor pressure of N_2, atm
298	-48.51	9.55×10^{-98}
700	-17.82	2.29×10^{-36}
1000	-11.03	8.70×10^{-23}
1200	-8.39	1.66×10^{-17}
1500	-5.74	3.31×10^{-12}
1800	-3.97	1.15×10^{-8}

(b) $TiN \rightleftarrows Ti(g) + \frac{1}{2} N_2$

Temp., °K	$Log_{10}K_f$	Vapor pressure of Ti, atm $\left(p_{N_2} = \frac{1}{2} p_{Ti}\right)$	Evaporation rate of Ti, g/(sq cm)(sec)
298	-122.8	1.71×10^{-82}	3.05×10^{-81}
1000	-27.79	3.75×10^{-19}	3.64×10^{-18}
1200	-21.13	3.87×10^{-16}	3.43×10^{-15}
1500	-14.50	2.71×10^{-10}	2.15×10^{-9}
1800	-10.11	1.74×10^{-7}	1.26×10^{-6}
2000	-7.93	2.44×10^{-5}	1.67×10^{-4}

(c) $TiN + \frac{3}{2} H_2 \rightleftarrows Ti(s) + NH_3$

Temp., °K	$Log_{10}K_f$	Pressure of NH_3, atm (at $p_{H_2} = 10^{-3}$ atm)
298	-45.55	8.91×10^{-51}
700	-19.84	4.57×10^{-25}
1000	-14.27	1.70×10^{-19}
1200	-12.11	2.46×10^{-17}
1500	-9.93	3.72×10^{-15}
1800	-8.53	9.33×10^{-14}
2000	-7.80	5.01×10^{-13}

(d) $TiN + 3H \rightleftarrows Ti(s) + NH_3$

Temp., °K	$Log_{10}K_f$	Pressure of NH_3, atm	
		$p_H = 10^{-3}$ atm	$p_H = 10^{-4}$ atm
298	61.24	1.74×10^{52}	1.74×10^{49}
700	20.95	8.91×10^{11}	8.91×10^{8}
1000	11.67	4.68×10^{2}	4.68×10^{-1}
1200	8.00	1.00×10^{-1}	1.00×10^{-4}
1500	4.31	2.04×10^{-5}	2.04×10^{-8}
1800	1.82	6.61×10^{-8}	6.61×10^{-11}
2000	.563	3.66×10^{-9}	3.66×10^{-12}

TABLE V. - Continued. VALUES DERIVED FROM THERMODYNAMIC DATA PERTINENT TO TiN OR TiC IN VACUUM, H_2, AND H ATOMS

(e) $TiN + 3H \rightleftarrows Ti(g) + NH_3$

Temp., °K	$Log_{10}K_f$	Pressure of Ti, atm $(p_{NH_3} = p_{Ti})$	
		$p_H = 10^{-3}$ atm	$p_H = 10^{-4}$ atm
298	-13.04	9.55×10^{-12}	3.02×10^{-13}
700	-6.23	2.43×10^{-8}	7.67×10^{-10}
1000	-5.10	8.91×10^{-8}	2.82×10^{-9}
1200	-4.74	1.35×10^{-7}	4.27×10^{-9}
1500	-4.46	1.86×10^{-7}	5.89×10^{-9}
1800	-4.32	2.19×10^{-7}	6.92×10^{-9}
2000	-4.28	2.29×10^{-7}	7.24×10^{-9}

(f) $NH_3 \rightleftarrows \frac{1}{2} N_2 + \frac{3}{2} H_2$

Temp., °K	$Log_{10}K_f$	Pressure of NH_3, atm	
		at $p_{H_2} = 10^{-3}$ atm and $p_{N_2} = 10^{-3}$ atm	at $p_{H_2} = 10^{-3}$ atm and $p_{N_2} = 1$ atm
298	-2.916	8.24×10^{-4}	2.61×10^{-2}
700	2.016	9.63×10^{-9}	3.05×10^{-7}
1000	3.238	5.78×10^{-10}	1.83×10^{-8}
1200	3.723	1.89×10^{-10}	5.99×10^{-9}
1400	4.080	8.32×10^{-11}	2.63×10^{-9}
1800	4.554	2.79×10^{-11}	8.83×10^{-10}
2000	4.720	1.91×10^{-11}	6.03×10^{-10}

(g) $TiC \rightleftarrows Ti(g) + C(s)$

Temp., °K	$Log_{10}K_f$	Pressure of Ti, atm	Evaporation rate of Ti, g/(sq cm)(sec)
298	-113.2	6.31×10^{-114}	1.12×10^{-112}
700	-43.5	3.16×10^{-44}	3.67×10^{-43}
1000	-28.0	1.00×10^{-28}	9.70×10^{-28}
1200	-22.0	1.00×10^{-22}	8.87×10^{-22}
1500	-16.0	1.00×10^{-16}	7.92×10^{-16}
1800	-12.1	7.94×10^{-13}	5.75×10^{-12}
2000	-10.1	7.94×10^{-11}	5.45×10^{-10}
[a]2400	-----	-----------	2×10^{-6}

[a] Extrapolated to 2400° K.

(h) $TiC + 2H_2 \rightleftarrows Ti(s) + CH_4$

Temp., °K	$Log_{10}K_f$	Pressure of CH_4, atm $\left(\text{at } p_{H_2} = 10^{-3} \text{ atm}\right)$
298	-30.08	8.32×10^{-37}
700	-15.34	4.57×10^{-22}
1000	-12.26	5.50×10^{-19}
1200	-11.08	8.32×10^{-18}
1500	-9.88	1.32×10^{-16}

TABLE V. - Continued. VALUES DERIVED FROM THERMODYNAMIC DATA PERTINENT TO TiN OR TiC IN VACUUM, H_2, AND H ATOMS

(i) $TiC + \frac{1}{2} H_2 \rightleftarrows Ti(s) + \frac{1}{2} C_2H_2$

Temp., °K	$Log_{10}K_f$	Pressure of C_2H_2, atm $\left(at\ p_{H_2} = 10^{-3}\ atm\right)$
298	-57.31	2.40×10^{-108}
700	-23.24	3.31×10^{-50}
1000	-15.70	4.00×10^{-35}
1200	-12.75	3.17×10^{-29}
1500	-9.80	2.50×10^{-23}
1800	-7.82	2.50×10^{-19}
2000	-6.82	2.50×10^{-17}

(j) $TiC + 4H \rightleftarrows Ti(s) + CH_4$

Temp., °K	$Log_{10}K_f$	Pressure of CH_4, atm	
		at $p_H = 10^{-3}$ atm	at $p_H = 10^{-4}$ atm
298	112.5	3.16×10^{100}	3.16×10^{96}
700	39.04	1.10×10^{27}	1.10×10^{23}
1000	22.31	2.04×10^{10}	2.04×10^{6}
1200	15.74	5.50×10^{3}	5.50×10^{-1}
1500	9.17	1.48×10^{-3}	1.48×10^{-7}

(k) $TiC + 4H \rightleftarrows Ti(g) + CH_4$

Temp., °K	$Log_{10}K_f$	Pressure of Ti, atm $\left(p_{Ti} = p_{CH_4}\right)$	
		at $p_H = 10^{-3}$ atm	at $p_H = 10^{-4}$ atm
298	38.21	1.27×10^{13}	1.27×10^{11}
700	11.86	8.51×10^{-1}	8.51×10^{-3}
1000	5.55	5.96×10^{-4}	5.96×10^{-6}
1200	3.00	3.16×10^{-5}	3.16×10^{-7}
1500	.402	1.59×10^{-6}	1.59×10^{-8}

(l) $CH_4 \rightleftarrows C(s) + 2H_2$

Temp., °K	$Log_{10}K_f$	Pressure of CH_4, atm at $\left(p_{H_2} = 10^{-3}\ atm\right)$
298	-8.90	7.94×10^{2}
700	-.95	8.91×10^{-6}
1000	1.01	9.78×10^{-8}
1200	1.79	1.62×10^{-8}
1500	2.58	2.63×10^{-9}

TABLE V. - Concluded. VALUES DERIVED FROM THERMODYNAMIC DATA PERTINENT TO TiN OR TiC IN VACUUM, H_2, AND H ATOMS

(m) $C_2H_2 \rightleftarrows 2C(s) + H_2$

Temp., °K	$Log_{10}K_f$	Pressure of C_2H_2, atm $\left(\text{at } p_{H_2} = 10^{-3} \text{ atm}\right)$
298	36.66	2.19×10^{-40}
700	13.90	1.26×10^{-17}
1000	8.88	1.32×10^{-12}
1200	6.97	1.07×10^{-9}
1400	5.55	2.82×10^{-9}
1800	3.87	1.35×10^{-7}
2000	3.12	7.59×10^{-7}

(n) $Ti(s) \rightleftarrows Ti(g)$

Temp., °K	$Log_{10}K_f$	Vapor pressure of Ti, atm	Evaporation rate, g/(sq cm)(sec)
298	-74.3	5.01×10^{-75}	8.91×10^{-74}
700	-27.2	6.31×10^{-28}	7.32×10^{-27}
1000	-16.8	1.59×10^{-17}	1.54×10^{-16}
1200	-12.7	2.00×10^{-13}	1.77×10^{-12}
1500	-8.77	1.70×10^{-9}	1.34×10^{-8}
1800	-6.14	7.25×10^{-7}	5.25×10^{-6}
2000	-4.84	1.45×10^{-5}	9.93×10^{-5}

(o) $H_2 \rightleftarrows 2H$

Temp., °K	$Log_{10}K_f$	Fraction of H_2 dissociated at total press. of 10^{-3} atm
298	-71.22	3.88×10^{-35}
700	-27.19	4.02×10^{-13}
1000	-17.29	3.58×10^{-8}
1200	-13.41	3.12×10^{-6}
1400	-10.63	2.42×10^{-5}
1800	-6.90	5.61×10^{-3}
2000	-5.58	2.56×10^{-2}
3000	-1.61	.927
4000	.404	.9997

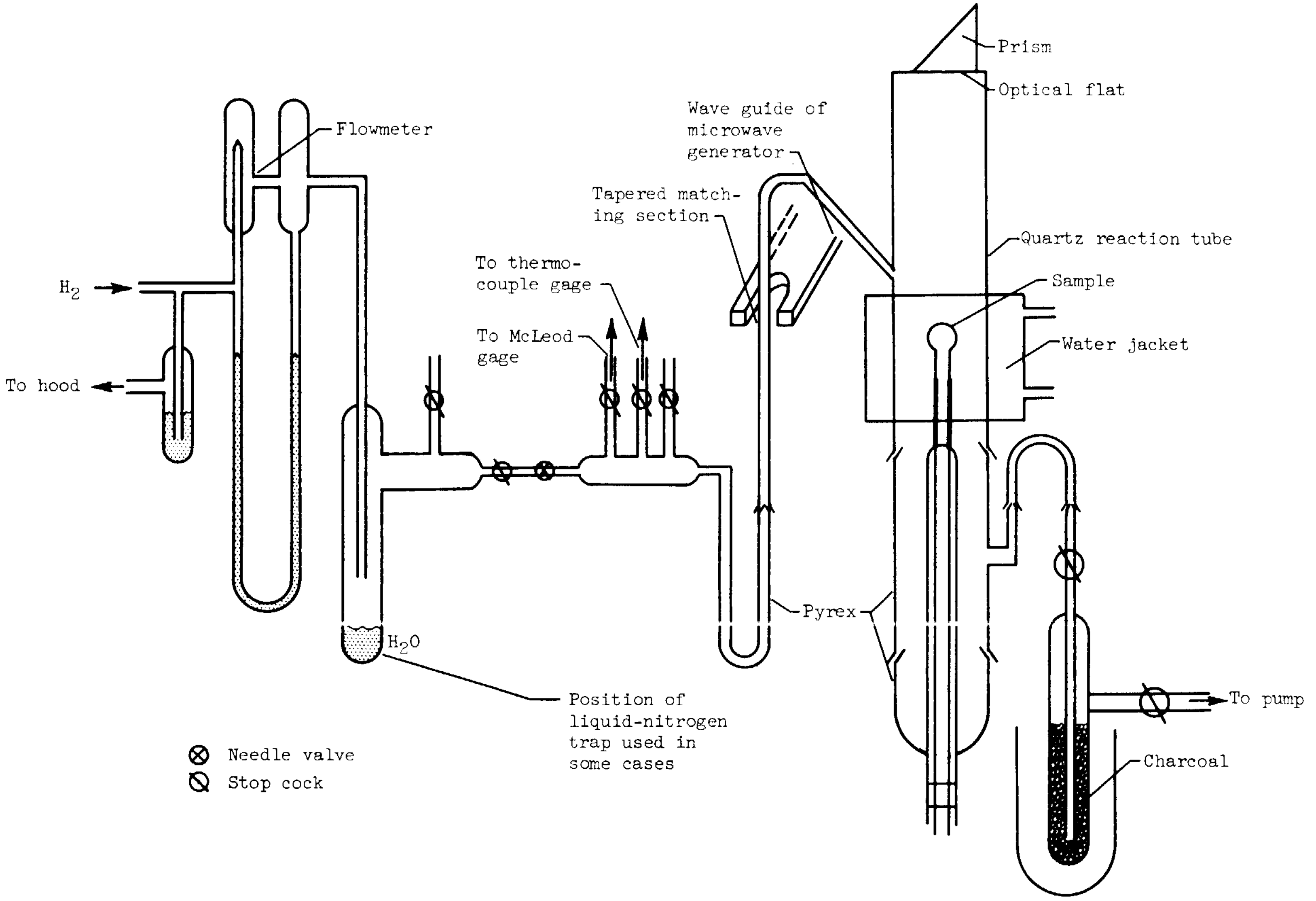

Figure 1. - Apparatus.

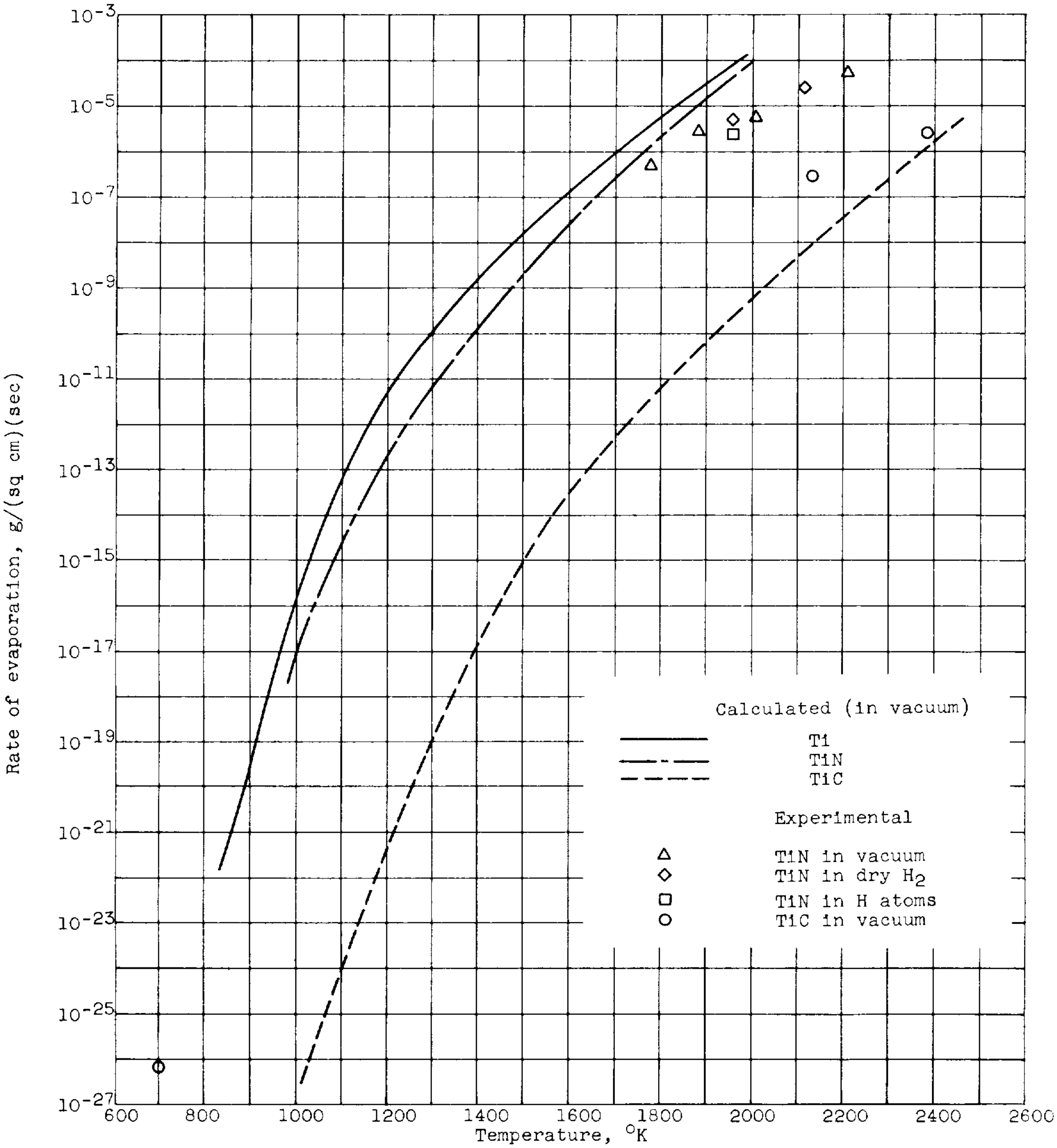

Figure 2. - Rates of evaporation and decomposition.