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The Sticking Coefficients of Carbon and Iron

Atomic Beams

Leo Brewer and Donald F. Mastick

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## The Sticking Coefficients of Carbon and Iron Atomic Beams

### 1. Introduction.

The accurate determination of the vapor-pressures of the elements is of great importance in the establishment of their thermodynamic properties. For many of the elements it is convenient to carry out vapor-pressure measurements using direct manometric methods which usually yield accurate results above 50 mm Hg. In the case of the refractory metals the use of pressure gauges is unsatisfactory because of the low vapor-pressures and the high temperatures involved in such determinations.

Two methods which are capable of yielding accurate results in the low pressure range are the effusion method and the measurement of rates of vaporization from a free surface in a vacuum.

In making effusion measurements, the vaporizing material is contained in an inert vessel which has a small hole through which the vapor may escape. In order that the equations of the kinetic theory of gases may be applied to this system the construction of this hole must meet two conditions: (1) the thickness of the edge of the hole must be much smaller than the diameter such that the probability that an atom arriving at the aperture passes through it is essentially unity, and (2) the diameter of the aperture must not be greater than the mean free path of the atom such that the velocity direction of the atoms in the containing vessel are not disturbed by the act of effusion. It is then a simple matter to collect a known fraction of the effusing atomic beam and calculate the total amount effusing from an ideal aperture in a given time

interval by applying the cosine dependence of the beam density. The relation given by gas kinetics for the calculation of the vapor-pressure is

$$p = \frac{z\sqrt{MT}}{44.383 \cdot a \cdot t} \quad (1)$$

wherein  $p$  is the pressure in atm,  $z$  the total number of moles effusing,  $M$  the molecular weight of the gaseous species,  $T$  the temperature in  $^{\circ}\text{K}$ ,  $a$  the hole area in  $\text{cm}^2$ , and  $t$  the time in seconds.

In these considerations of the equilibrium between a gaseous and condensed phase one must consider a factor  $\alpha$ , the sticking coefficient, such that if  $p$  is an apparent vapor-pressure which determines the rate of evaporation from a surface, and if  $p^{\circ}$  is the equilibrium vapor-pressure, then

$$p = \alpha p^{\circ}$$

This means the atoms in the gas phase do not have unit probability of condensing upon striking the surface if  $\alpha$  is not unity and that the rate at which atoms enter the gas phase is proportional to  $p$  rather than to the equilibrium pressure  $p^{\circ}$ . The factor  $\alpha$  may also be expressed as a fraction of the number of gaseous atoms which strike unit surface per unit time that condense.

Now it can be shown that measurements made by the effusion method are independent of the sticking coefficient within the effusion vessel if the area of the hole is considerably smaller than the vaporizing area. If  $a$  represents the area of the hole and  $s$  the vaporizing area, the relation

$$p = p^{\circ} \frac{\alpha}{\alpha + \frac{a}{s}}$$

expresses the dependence of the measurement of the equilibrium vapor-pressure upon the sticking coefficient within the vessel. It is obvious that in making measurements by the effusion method that one should vary his hole size in order to establish the fact that one is not dealing with an abnormally low sticking coefficient.

However, this factor is of importance at the collector where the effusing beam is condensed. Since the measured vapor-pressure is directly dependent upon the amount collected, a sticking coefficient which is quite small leading to considerable reflection will introduce serious errors into the final measurement.

In the determination of vapor-pressures by the measurement of rates of vaporization from a free surface in a vacuum the factor  $\alpha$  is of direct importance since the relation given by gas kinetics is

$$p\alpha = \frac{m}{44.383} \sqrt{\frac{T}{M}} \quad (2)$$

wherein  $p$  is the pressure in atm,  $\alpha$  the sticking coefficient,  $m$  the weight loss from the surface in gms/cm<sup>2</sup>/sec,  $T$  the temperature in °K and  $M$  the molecular weight of the gaseous species. In this type of measurement no collection is necessary and the sticking coefficient is of importance only in the above relation.

There is one possible situation in which equations (1) and (2) will give a calculated vapor-pressure which is greatly in error. That is when account is not taken of the actual vaporizing species. For instance, Melville and Gray<sup>(1)</sup> have shown that red phosphorous evaporates to give P<sub>2</sub> molecules and only P<sub>2</sub> molecules condense on red phosphorous even under conditions where P<sub>4</sub> is the predominate gaseous species. When P<sub>4</sub> molecules hit the red phosphorous surface they condense to

the more volatile white phosphorous which evaporates again to  $P_4$  gas without conversion to red phosphorous. Therefore, if the equilibrium pressure measured consists of  $P_4(g)$  with only a small amount of  $P_2(g)$ , the small equilibrium pressure of  $P_2(g)$  should be used to calculate the rate of vaporization. Such cases, however, are rare and in the case of the refractory metals discussed in this paper the vaporization is predominately to the monatomic gas.

In general the method of vaporization from a free surface yields results of greater accuracy than the effusion method when  $\alpha$  is known or is quite close to unity. This is a result of difficulties encountered in determining the true mean temperature within the effusion cell. Despite great care in experimental details, serious temperature gradients usually exist when runs are conducted at high temperatures. One immediate reason for this is that it is not possible to include radiation shielding in the space about the effusion cell where the exit gas atoms travel to the collector. Thus the area about the hole acts as a heat sink and not only introduces a factor which lowers the apparent vapor-pressure but leads to error in the measurement of the temperature within the cell by the optical pyrometer due to possible deviations from black body conditions when the temperature is not uniform.

If the emissivity of the material is known at the temperature of the measurement, the temperature of the free surface may be measured relatively accurately and the difficulties encountered in the effusion method are not encountered in the vaporization method.

Unfortunately very few direct determinations have been made of the sticking coefficient of the refractory metals. It has generally been assumed that  $\alpha$  is essentially unity in conducting



the vaporization measurements. In the first paper to report the determination of vapor-pressures by free surface vaporization, Langmuir<sup>(2)</sup> discusses this problem. Since Knudsen has shown the thermal accommodation coefficients of gases increase with increasing molecular weight, Langmuir concludes that it is extremely probable that the sticking coefficient of gaseous metallic atoms will be essentially unity. In a recent paper Holden, Spieser and Johnston<sup>(3)</sup> have determined the vapor-pressure of beryllium by both of the methods described above and find agreement within their experimental error, which indicates that the sticking coefficient is unity at the temperature of their measurement within the accuracy of their measurements.

Actually very few qualitative observations of the reflection of metallic atoms have been reported. It should be emphasized that we are interested only in those metals whose vapor-pressures are vanishingly small at room temperature, or the temperature of the target material. It is well known<sup>(4)</sup> that atomic beams of volatile metals such as mercury and cadmium show considerable reflection, because of re-evaporation from the collector. Such cases will not be discussed. Rudberg and Lempert<sup>(5)</sup> report no extensive reflection occurring when barium atoms strike a collector; Rudberg<sup>(6)</sup> also finds no evidence for the reflection of calcium atomic beams by visual inspection of surfaces adjacent to the collector. Weber and Kirsch<sup>(7)</sup> report no visual evidence for the reflection of bismuth atoms from a bismuth deposit on a collector. Johnston and Marshall<sup>(8)</sup> observed no condensation of nickel on re-entrant portions of their apparatus during measurements of the vapor-pressure of nickel. Of not direct bearing on the reflection of metallic atoms, but of

interest from view of large molecular weight are the experiments Verholk and Marshall<sup>(9)</sup> in which the vapor-pressures of tri-m-cresyl phosphate, tri-p-cresyl phosphate, dibutyl phthalate and dibenzyl sebacetate were determined at 100°C by both the effusion method and the measurement of the rate of vaporization of the free surface. The sticking coefficient was found to be essentially unity for each of these compounds since in all cases the two methods gave the same vapor-pressure.

The vapor-pressures of several of the refractory metals have been investigated by at least one of these methods. In a review of the vapor-pressures of monatomic vapors Ditchburn and Gilmour<sup>(10)</sup> give the pressure ranges which have been measured, and in the case of the more refractory metals give the vapor-pressures for Cu, Ag, Au, Be, Al, Si, Pb, Bi, Cr and Mn. Kelley<sup>(11)</sup> has given vapor-pressure data for these elements and also C, Fe, Mo, Ni, Pt, Sn, and W. Brewer<sup>(12)</sup> summarizes the thermodynamic properties of the elements, giving vapor-pressures at high temperatures for the elements given above and also for Ti, N, Th, Cb, Ta, U and other elements determined by these methods.

It is seen that the vapor-pressures of many of the refractory metals have been measured by either of two methods, both of which are subject to error if the sticking coefficient is not close to unity. Furthermore, essentially only one measurement has been made to determine these sticking coefficients to test the validity of the reported vapor-pressures.

A current case of interest is the determination of the vapor-pressure of graphite by Brewer, Gilles and Jenkins.<sup>(13)</sup> In this work the beam of carbon atoms was collected on a water-cooled

platinum disc. The primary purpose of the determination was to allow the fixing of the heat of sublimation of graphite, there being three values "allowed" by spectroscopic data: 170.39, 141.25 and 125.03 kcal/mole. While the results of this measurement gave close agreement to the first value, it can be shown that this would result fortuitously if the true heat were 141.25 kcal/mole and the sticking coefficient on the collector were  $10^{-2}$  or if the true heat were 125.03 kcal/mole and the sticking coefficient on the collector were  $10^{-4}$ . Considering the orders of magnitude involved it should be possible to determine the amount of reflection of the carbon atoms from the graphite deposit on the collector in order to distinguish between these possibilities. Such is done in this paper under essentially the same experimental conditions as used by Brewer, et al, in their determinations.

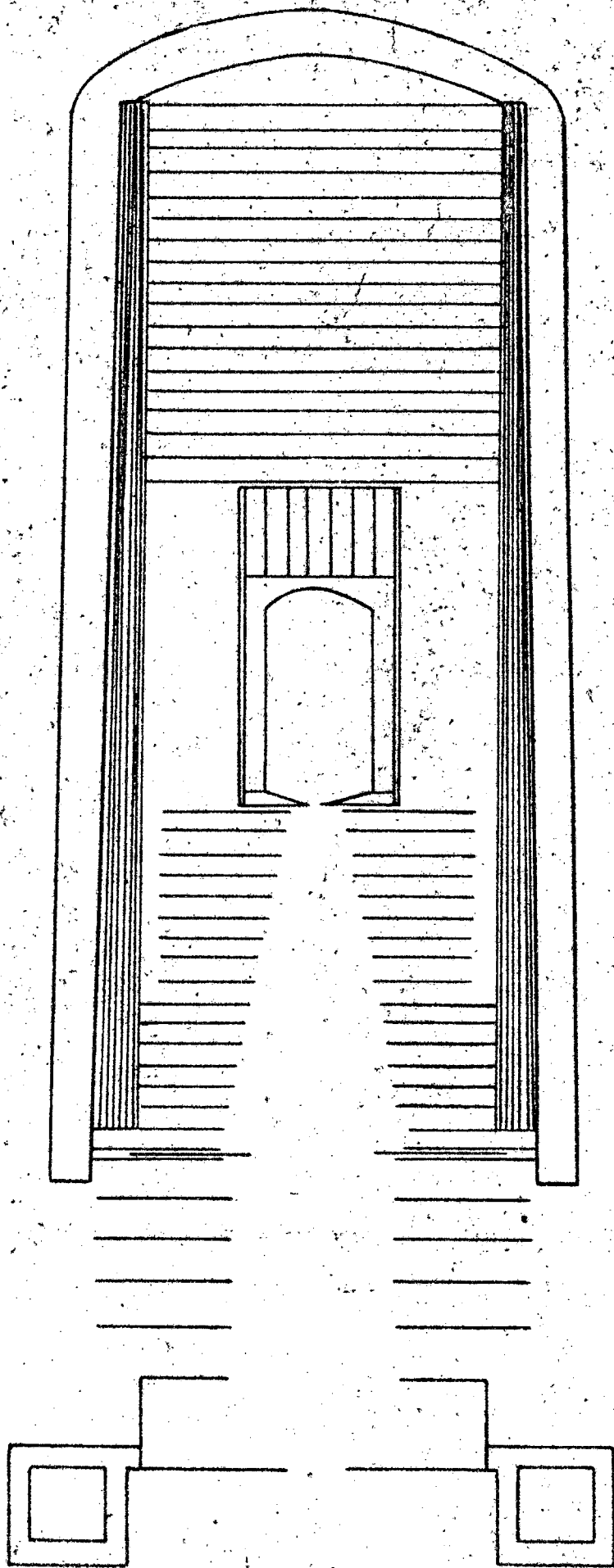
The sticking coefficient is also reported for iron atoms on the collector under the same experimental conditions used by Brewer and Mastick<sup>(14)</sup> in the determination of the vapor-pressure of iron by the effusion method. If the iron atoms condense completely it would be possible to compare the vapor-pressure values obtained by the effusion with those obtained by the measurements of rates of volatilization from free iron surfaces<sup>(15), (16)</sup> and thus obtain a measure of the sticking coefficient at the temperature of the actual measurements.

## 2. Experimental.

The equipment and experimental techniques involved in the carbon measurements will not be described in detail as they have been given elsewhere.<sup>(13)</sup> Graphite powder (Acheson grade 38) was

contained in a graphite crucible which was fitted with a snug graphite lid. Surrounding this was a heavy tantalum crucible and an 8 mil tantalum sheet with a  $\frac{1}{8}$  inch circular hole which rested on top of the graphite lid whose circular hole was  $\frac{1}{4}$  inch in diameter. Figure 1 indicates the tantalum crucible surrounded by tantalum radiation shielding and collimator plates. The tantalum assembly is contained in a zircon crucible which is about 5 inches high and 2.5 inches in diameter. Suspended above the zircon crucible, in line with the effusing beam is a water cooled copper ring on which a 2 mil platinum collector disc rests. This disc has a central hole  $\frac{1}{4}$  inch in diameter to allow sighting on the effusion hole with an optical pyrometer. A short platinum cylinder with one end open and the other end closed with a large hole cut in this closed end was slip-fitted into the bottom of the ring so that the open end just touched the collector disc. The interior of this cylinder was lined with 1 mil platinum in the form of a ring resting on the bottom and a strip around the walls. These surfaces acted as collectors for the atoms reflected from the collector disc. During the runs the portion of the disc intercepting the beam was heated to about 900°K.

Table I gives the essential results of the carbon reflection measurements. Runs 4 and 5 were blank runs in which there was no collector disc in order that the amount of in-beam scattering to the 1 mil ring and strip could be determined. This amount was subtracted from the weight found on these surfaces during reflection runs 1, 2, 3, and 6. In view of the relatively large amount of reflection observed in these runs, run 7 was conducted without the lower 1 mil surfaces to determine the amount of reflection from them back to the collector disc. This was necessary to determine



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Table I

<u>Run</u>	<u>°K</u>	<u>Time secs. x 10<sup>-4</sup></u>	<u>Wt. C on disc (mg)</u>	<u>Wt. C on ring and strip (mg)</u>	<u>Corrected ratio of C on disc to total C</u>
I Collector disc and ring and strip reflection collectors in place:					
1	2775	1.374	1.535	1.029	0.547
2	2765	1.440	2.042	1.164	0.585
3	2760	1.440	2.496	1.247	0.616
6	2674	1.800	1.292	0.774	0.581
II Ring and strip reflection collectors in place only:					
4	2760	1.440	————	0.088	————
5	2760	1.440	————	0.049	————
III Collector disc in place, no reflection collectors under disc:					
7	2760	1.440	1.705	————	————

the magnitude of single reflection from the deposit on the collector. Since the experimental conditions of runs 2, 3 and 7 are the same, the first two runs give the total amount of the beam collected and the ratio of the weight on the collector in run 7 to the total weight striking the collector as determined by runs 2 and 3 gives the desired sticking coefficient. To determine the total amount of carbon in runs 2 and 3, it is necessary to correct for the fraction of the atoms which escape back through the hole in the bottom of the cylinder. This factor, calculated from the geometry of the system, is 1.32 times the weight on the collector disc. Column six of Table I gives the ratio of the corrected weights of carbon found on the collector to the sum of the corrected weights of carbon found on all surfaces. This ratio is not the sticking coefficient, but approaches this coefficient in the limit as the amount of reflection approaches zero. The sticking coefficient calculated from runs 2, 3 and 7 carbon atoms on the deposit is 0.45.

The iron reflection runs were made with the same equipment and geometry with two exceptions: the radiation shields and collimators were constructed of molybdenum, and the iron was contained in a beryllium oxide effusion cell.<sup>(14)</sup> Table II gives the results of these runs. Runs 4 and 5 were made to determine the blank correction for the amount of iron found on the 1 mil ring and strip when the collector disc was left out of the system. Because of the slight reflection observed in runs 1, 2 and 3 it was considered to be of no further advantage to attempt to determine the back reflection due to the 1 mil surfaces. The sticking coefficient of iron atoms on the iron deposit is given as 0.98.

Table II

<u>Run</u>	<u>°K</u>	<u>Time secs. x 10<sup>-3</sup></u>	<u>Wt. Fe on disc(mg)</u>	<u>Wt. Fe on ring and strip (mg)</u>	<u>Corrected ratio of Fe on disc to total Fe</u>
I Collector disc and ring and strip reflection collectors in place:					
1	1864	3.180	7.35	0.35	0.0174
2	1864	3.600	9.90	0.41	0.0208
3	1864	3.660	10.27	0.39	0.0182
II Ring and strip reflection collectors in place only:					
4	1864	3.600	_____	0.27	_____
5	1863	3.600	_____	0.22	_____



### 3. Discussion.

A sticking coefficient of 0.45 for the beam carbon atoms on the collector deposit indicates that the vapor-pressures reported by Brewer, et al.,<sup>(13)</sup> are low by about a factor of two, and therefore the values for the heat of sublimation of graphite which they give are high by about 4 kcal/mole. Excluding their outgassing runs, their average value for the heat of sublimation is 169.36 kcal/mole. Correcting for the reflection at the reflector, this value becomes 165.25 kcal/mole. Since the primary purpose of their research was to distinguish between the possible values 170.39, 141.25 and 125.03 kcal/mole, it is seen that the evidence for the value 170.39 kcal/mole is quite conclusive.

The probable explanation for their experimentally determined value being slightly low is based upon the uncertainty in establishing the true mean temperature within the effusion cell. It can be shown that the temperature gradients which may exist in the cell can introduce an error in the reading of the temperature with an optical pyrometer of sufficient magnitude to account for most of the observed deviation. This error should become more pronounced the higher the temperature of the effusion cell. Such was found to be the case in measurements reported in this paper which were conducted at temperatures 160°K higher than those reported by Brewer, et al.<sup>(13)</sup>

Kelley<sup>(11)</sup> gives  $\frac{\Delta F - \Delta H_0}{T} = -36.88$  for the reaction  $C(\text{gr}) = C(\text{g})$  at 2760°K; from this datum, the data in Table I and the geometry of the system we obtain as an average value for the heat of sublimation 157.1 kcal/mole.

The results given in Table II indicate that essentially complete condensation of the gaseous iron atoms takes place upon striking

the reflector. Thus the vapor-pressures reported by Brewer and Mastick<sup>(14)</sup> are not in error as a result of this factor. The vapor-pressures reported by these authors were determined by the effusion method and agree within experimental error with those determined by Jones, et al,<sup>(15)</sup> and Marshall, et al,<sup>(16)</sup> who utilized the method of measuring rates of vaporization from free iron surfaces in a vacuum. Within the accuracy of the measurements involved one can conclude that the sticking coefficient of gaseous iron atoms on an iron surface which is in thermal equilibrium with the gas is essentially unity.

It has been noted that careful distinction has been drawn between the sticking coefficient on a deposit on a cold collector and on a surface in thermal equilibrium with the gas, the former being of importance in the effusion method and the latter in the method of measuring rates of vaporization. That these two factors may not be immediately equated follows from the fact that the temperature dependence of the reflection is unknown and, secondly, it must be shown that the nature of the solid surface is the same in both cases.

In regard to the temperature dependence a qualitative discussion of the interaction of the atom with the surface is of help in estimating the probable importance of the dependence. As the gaseous atoms approach the surface it must be accelerated by the attractive surface forces. During impact with the surface the atom will lose a certain fraction of the total translational energy which it possesses at the moment of impact. Since this total translational energy is greater than that which the atom had when far from the surface, the energy loss may exceed that which the atom possessed originally. If this occurs the atom is held in an attractive potential field of the

surface bonding forces and thus condenses to become an integral part of the surface. Since the fractional energy transfer upon collision may be taken essentially independent of the initial translational energy of the atom, and for strong surface bonding forces the maximum translational energy at the point of collision is a slowly varying function of the initial translational energy of the atom, there must exist a certain translation energy possessed by the atom upon leaving the effusion cell which is equal in magnitude to the energy loss during collision. For initial translational energies greater than this critical value the atom will rebound from collision with residual kinetic energy equal to or greater than zero and may be considered reflected. It follows from Maxwell-Boltzmann statistics that some reflection will always occur for atoms which have extremely high velocities even though the temperature of the effusion cell is relatively low and the surface forces high. This effect rapidly becomes negligibly small as the mean velocity or temperature of the beam decreases. It is very difficult to reach conclusions regarding the effect of an increased surface temperature on the interaction between the atom and the surface, but one is led to conclude that it will be of secondary importance and that the two major factors in the reflection of atomic beams will be the strength of the surface forces which attract the impinging atom and the mean velocity of the atoms in the beam. Thus in conducting effusion measurements one should construct the collector of a material which has strong surface bonding forces for the effusing atoms and should keep the beam velocities as low as possible.

During the deposition of effusing atoms the first few atomic layers will be distorted from their normal crystal habit by the

surface forces of the collector, and the consequent mass of the deposit will be of a crystal structure appropriate to the element at the temperature of the collector unless an unusual case is encountered where the rate of crystal formation is extremely slow. In the case of the carbon deposit the structure is presumably graphitic. While no direct studies were made at least two facts support this conclusion: (1) the deposit had a metallic luster and was quite brittle, and (2) it has been shown<sup>(17)</sup> that carbon black is a mixture containing particles which range from single graphite layers up to graphite crystals several layers thick. Deposits of iron have been inspected by X-ray diffraction methods and are found to assume the normal cubic close-packed structure whose lattice constant was in excellent agreement with the best literature values.<sup>(18)</sup>

In view of these considerations one is probably justified in drawing a comparison between sticking coefficients measured at the collector and on the vaporizing surface. This may be done in comparing the value  $\alpha = 0.45$  determined in this paper with the results of Marshall and Norton<sup>(19)</sup> obtained by measuring the rate of vaporization from carbon rings in a vacuum. They report the heat of sublimation of graphite as 177 kcal/mole, based on their vapor-pressure measurements and the apparent assumption of unit sticking coefficient. Using the correct value 170.39 kcal/mole for the heat of sublimation one calculates that at the temperature of their determinations a sticking coefficient of  $\alpha = 0.18$  would account for their high value of  $\Delta H_0$ . It is difficult to conclude whether or not the difference in the values for the sticking coefficient may be attributed to the experimental error or to an actual temperature

dependence. However, it is interesting to note that direct determinations made by Simpson, et al,<sup>(20)</sup> of the sticking coefficient of carbon atoms on optically thin deposits on hot quartz (ca. 700°C) collectors is 0.18 for an effusion cell temperature of 2400°K and 0.22 for a cell temperature of 2216°K.

#### 4. Conclusions.

The sticking coefficient of atomic beam carbon atoms issuing from an effusion cell at 2760°K on a deposit at 900°K is 0.45. Since this measurement was conducted under essentially the same experimental conditions as used by Brewer, et al,<sup>(13)</sup> in the determination of the heat of sublimation of graphite, the factor  $\alpha = 0.45$  may be applied directly to their data. This correction leads to an average experimental value  $\Delta H_0 = 165.25$  kcal/mole instead of the average value  $\Delta H_0 = 169.36$  kcal/mole which they calculated assuming no reflection at the collector. Despite the lowering of the experimental value the evidence is conclusive that 170.39 kcal/mole and not 141.25 nor 125.03 kcal/mole (the three values "allowed" spectroscopically) is the correct value for the heat of sublimation of graphite.

A measurement has also been reported for the sticking coefficient of atomic beam iron atoms on the collector plate, the value being  $\alpha = 0.98$ . It is concluded from this evidence and the agreement between the determination of the vapor-pressures of iron by both the effusion method<sup>(14)</sup> and the method of measurement of rates of vaporization from free surfaces in a vacuum<sup>(15), (16)</sup> that the sticking coefficient is essentially unity at the temperature of the iron surface during the vaporization measurement.

It is indicated that the value of sticking coefficients

measured at the temperature of the collector may serve as a close approximation to sticking coefficient which obtains between the gaseous atoms and the surface when the two phases are in thermal equilibrium at the temperature of an actual measurement.

The determination of the sticking coefficient is of direct importance in the determination of vapor-pressures by both the effusion method and the method of vaporization from a free surface. In the former method reflection at the collector will cause a direct error in the measured apparent vapor-pressure and in the latter method the sticking coefficient enters directly into the expression given by gas kinetics for the calculation of vapor-pressures from the measured rate of vaporization.

The methods applied in this paper for the measurement of sticking coefficients may be applied in general for the determination of the extent of reflection of the atoms from a deposit and be incorporated with vapor-pressure measurements by either of the methods mentioned in direct support of the validity of the values obtained.

From a consideration of the experimental evidence and qualitative theoretical discussion given above one is led to the conclusion that the sticking coefficients for all refractory elements which vaporize to monatomic gases will have sticking coefficients close to unity at temperatures at which the vapor-pressure of the element is less than about  $10^{-6}$  atm. This conclusion is supported by direct experimental evidence in the case of beryllium and iron. Actually, it is not expected that values as low as that reported herein for carbon will be found for the refractory metallic elements. The sticking coefficient for carbon is probably low because of the random orientation of the graphitic planes in the deposit such that some

collisions of the gaseous atoms occur on sites where the bonding forces are small.

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