THE VAPORIZATION AND PHYSICAL PROPERTIES OF CERTAIN REFRACTORIES

Quarterly Technical Summary Report No. 5

Prepared by

A. A. Hasapis
A. J. Melveger
M. B. Panish
L. Reif
C. L. Rosen

RESEARCH AND ADVANCED DEVELOPMENT DIVISION
AVCO CORPORATION
Wilmington, Massachusetts

ARPA Order No. 24-60, Task 6
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3 May 1961

Prepared for

WRIGHT AIR DEVELOPMENT DIVISION
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
Wright-Patterson Air Force Base, Ohio

Avco Research and Advanced Development

201 LOWELL STREET, WILMINGTON, MASSACHUSETTS · OLiver [illegible]

May 5, 1961

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Enclosed is a revision of "The Vaporization and Physical Properties of Certain Refractories", Quarterly Technical Summary Report No. 5, RAD-SR-61-54. Please destroy the previous copy which was issued 11 April 1961.

Yours very truly,

Research and Advanced Development Division
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Stanley Ruby

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Materials Research

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(Rev. 1)

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APPROVED

E Scala
E. Scala, Manager
Materials Department

M. E. Malin, Manager
Physics Research Department

Prepared for

WRIGHT AIR DEVELOPMENT DIVISION
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
Wright-Patterson Air Force Base, Ohio

ABSTRACT

I. VAPORIZATION STUDIES

1. Ruthenium. Preliminary data are presented.

2. Hafnia. Preliminary vaporization and thermodynamic data are presented for the reactions

$$HfO_{2(s)} \longrightarrow HfO_{(g)} + O_{(g)}$$

and

$$HfO_{(g)} \longrightarrow Hf_{(g)} + O_{(g)}$$

3. Rare Earth Oxides. A mass spectrometric survey of the mode of vaporization of all the rare earth sesquioxides is presented.

4. Molybdenum - Silicon system data are presented.

5. Tungsten - Silicon system data are presented.

II. VISCOSITY STUDIES

No new results during this quarter.

III. SURFACE TENSION

Summary of data is presented.

CONTENTS

ILLUSTRATIONS

I. VAPORIZATION STUDIES

A. RUTHENIUM

The first simple effusion data on the vaporization of ruthenium are now available. Ruthenium metal was vaporized from a 1/8-inch orifice in a thoria liner. Otherwise, the system was identical to that shown in figure 1 of reference 1. The total amount of material which had struck the targets was determined by weighing. The data are plotted in figure 1.

In earlier reports it had been noted that thoria appears to vaporize at an increased rate after being heated for awhile in tungsten. The two triangular points in figure 1 are for apparent Ru pressures in cells in which interaction between the thoria and the tungsten had apparently taken place. The high pressures indicated by these points may be due to thoria and not ruthenium. Further work with a radioactive tracer (Ru^{103}) is now underway. This will permit the use of shorter heating times and smaller orifices. The preliminary data from these studies has not yet been evaluated.

The data reported here are tentative. No detailed thermodynamic calculations have been made. Such calculations will be made when more vaporization data, taken at shorter exposure times and with smaller effusion orifices, are available.

B. HAFNIA

1. Simple Effusion Studies

Simple effuson studies in which hafnia, containing Hf^{181} as a tracer, was vaporized from effusion cells with iridium liners, have been undertaken. The effusion cells consisted of a relatively massive outer tungsten shell, a thin thoria separator, and an iridium inner liner. The cell is illustrated in figure 2. Orifice diameters of approximately 1/16 and 1/32 inch were used and a collimated portion of the effusion beam was caused to impinge upon liquid nitrogen cooled copper targets. The apparatus and procedures were essentially the same as those described previously for the rhodium and iridium studies.[1] The radioactivity of the material deposited upon the targets was compared to that of a calibrated sample containing a known amount of Hf with the same ratio of Hf^{181} to normal Hf as in the sample in the effusion cell.

The data from several runs was found to be very scattered. Inspection of the cells after the run revealed that the iridium cells were severely attacked in some instances, apparently from the outside. This may have been caused

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[1] Hasapis, A.A., M.B. Panish and C. Rosen, The Vaporization and Physical Properties of Certain Refractories. Part, I, WADD Technical Report 60-463, August 1960.

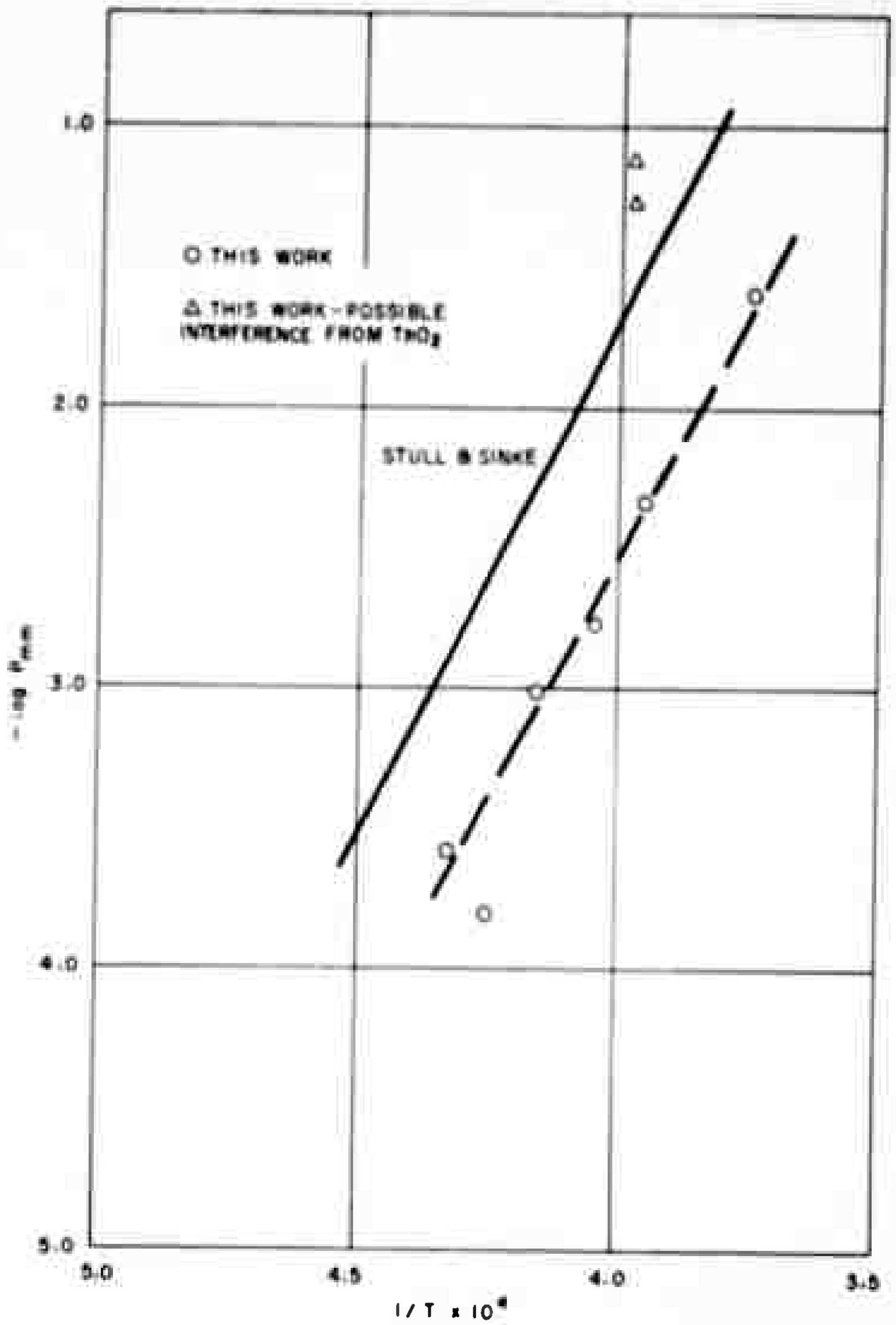

Figure 1 VAPOR PRESSURE DATA FOR RUTHENIUM
61-2036

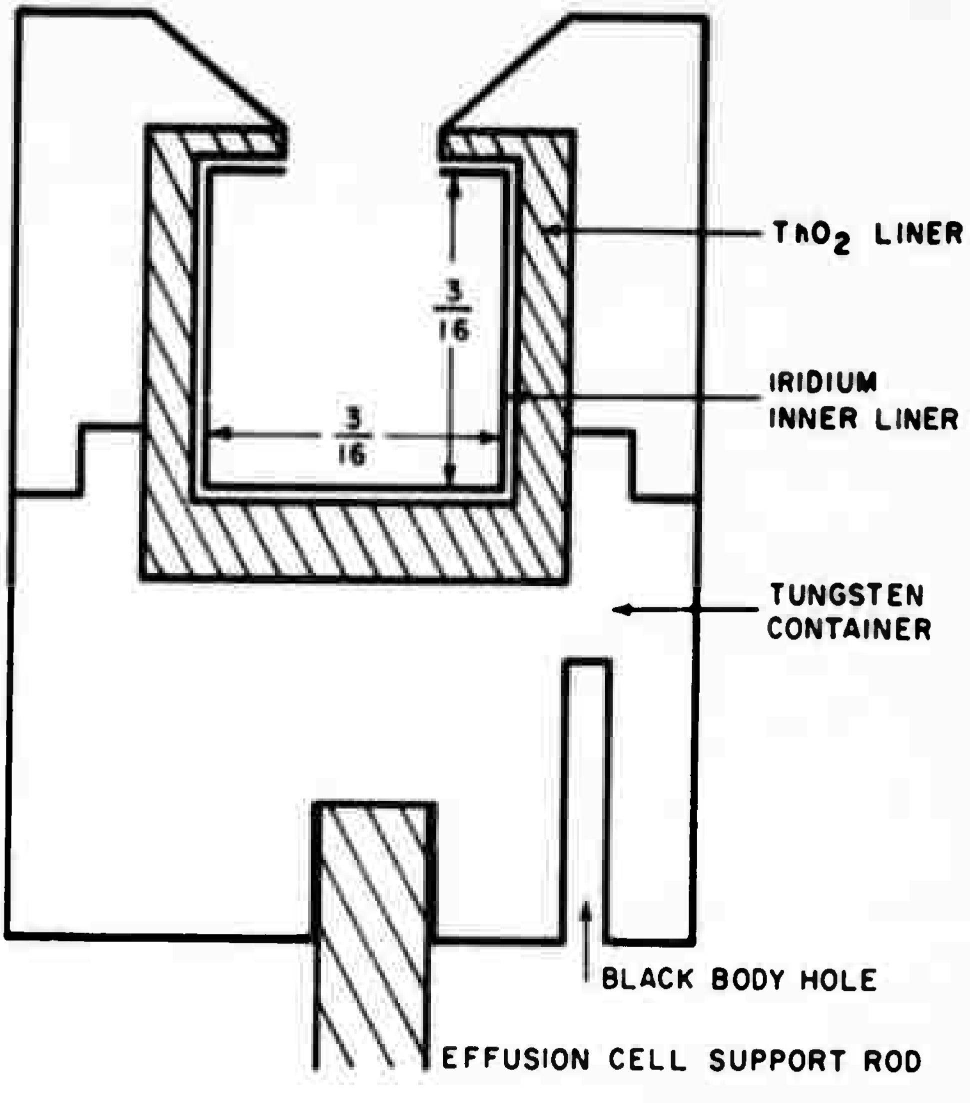

Figure 2 THE EFFUSION CELL
61-2037

by alloying with thorium metal resulting from the interaction of thoria and tungsten. Where the iridium cell had been punctured, the thoria liner was highly contaminated with Hf^{181}. Where there was no puncture, no activity of Hf^{181} was observed outside the iridium liner.

A number of points which were obtained when the effusion cell was apparently undamaged are given in figure 3. Where possible, these points were selected from runs in which the iridium liner was inspected periodically throughout the run. Third law calculations, for which the free energy functions tabulated by Brewer[2, 3] and Stull and Sinke[4] were employed, were used to determine the ΔH_{298} for reaction (1).

$$(1) \qquad HfO_{2(s)} \longrightarrow HfO_{(g)} + O_{(g)}, \quad \Delta H_{298} = 357 \pm 5 \text{ Kcal}.$$

It was assumed that the vaporization is stoichiometric and that reaction (1) is the principal reaction in the cell. This assumption is supported by the fact that $HfO_{(g)}$ is the species observed mass spectrometrically[5] when hafnia vaporizes. The reaction is, however, not completely stoichiometric (see below), and reaction (1) can only be an approximation to the true vaporization reaction.

Stull and Sinke's tabulated data for the heat of vaporization of Hf and the heat of dissociation of O_2 may be used with the heat of formation of $HfO_{2(g)}$[3] and ΔH_{298} for reaction (1) to obtain

$$(2) \qquad HfO_{(g)} \longrightarrow Hf_{(g)} + O_{(g)}, \quad \Delta H_{298} \approx 196 \text{ Kcal}.$$

The value of ΔH_{298} is considered approximate because in addition to the rather large uncertainty in ΔH_{298} for reaction (1), the ΔH_{298} for the vaporization of hafnium metal is quite uncertain.

It should be noted that these data are strictly tentative. The scatter in the data is large and work is underway at present on the elucidation of the actual processes which occur within the effusion cell.

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[2] Brewer, L., and M.S. Chandrasekharaiah, Free Energy Functions For Gaseous Monoxides, UCRL-8713, April 1959.

[3] Brewer, L., Chapter 7 of an unpublished compilation, Revised September 1960.

[4] Stull, D.R., and G.C. Sinke, Thermodynamic Properties of the Elements, American Chemical Society, Washington (1956).

[5] Hasapis, A.A., A.J. Melveger, M.B. Panish, L. Reif, and C.L. Rosen, Quarterly Technical Report No. 4, RAD-SR-16-61-1 on Contract AF33(616)-6840, January 1961.

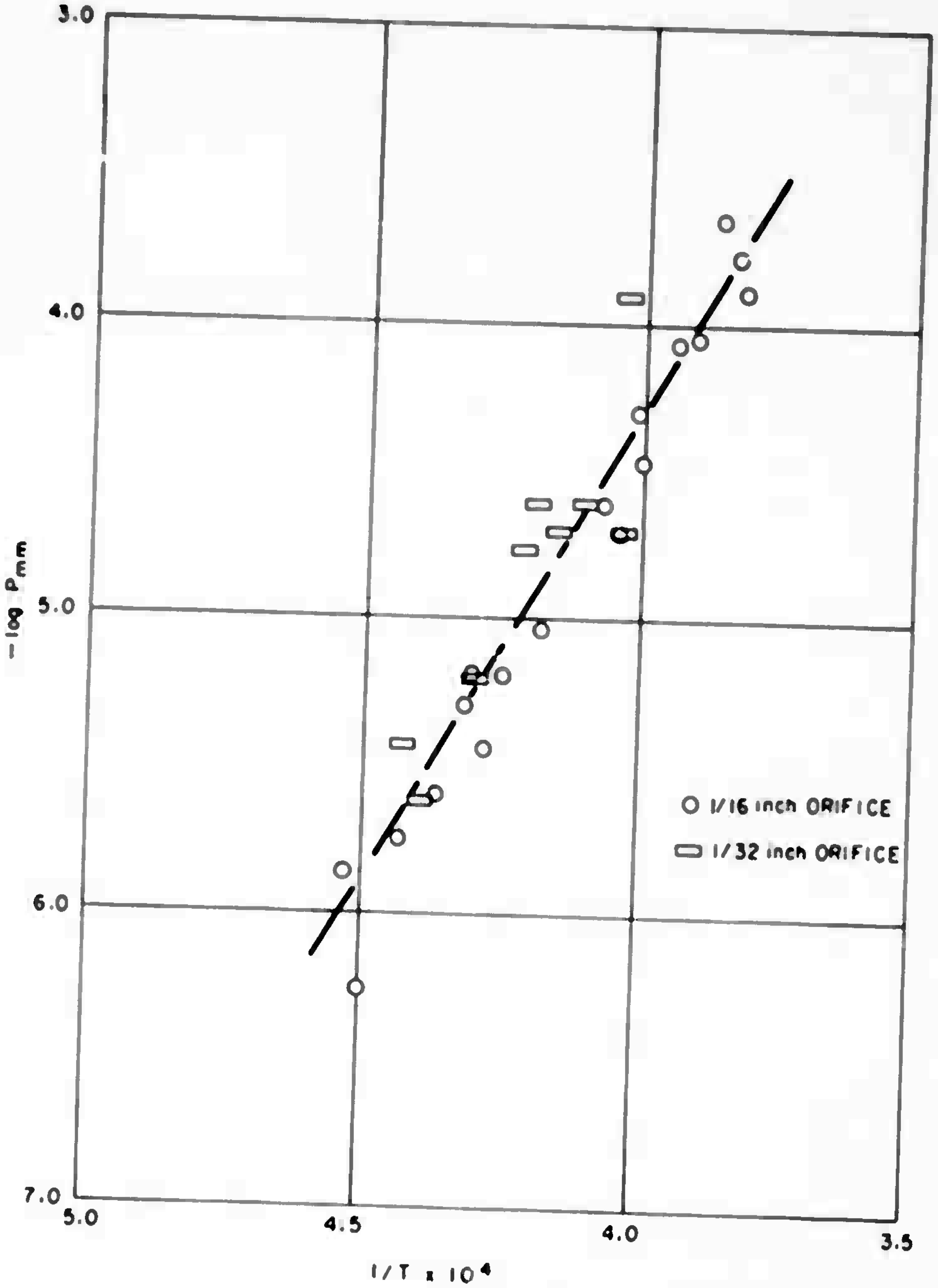

Figure 3 LOG P OF $HfO_{(g)}$ VERSUS $\frac{1}{T}$ FOR THE VAPORIZATION OF HAFNIA

61-2038

2. Stoichiometry Studies

Studies have been undertaken to determine what changes occur in the condensed phase when hafnia is vaporized. Samples of spectroscopic grade hafnia were heated in tungsten and iridium crucibles to temperatures of from 2300 to 2600°K for various lengths of time. The samples which were heated in the tungsten container turned a uniform grey. Those in iridium appeared to consist of white and dark phases. When these samples were reheated in air they become white again and showed a small weight gain. For the samples heated in tungsten, this corresponded to an average composition of $HfO_{1.997 \pm .001}$ for the oxygen poor material if it is assumed that the white material is $HfO_{2.000}$. The samples heated in iridium samples lost considerably less oxygen. This work is being continued.

X-ray studies of the material heated in the iridium crucibles are underway. The white phase has been identified as the normal monoclinic phase HfO_2. A darker colored phase has been found to be cubic. Only very small quantities of the cubic material were available. This work is continuing.

C. THE RARE EARTH OXIDES

A preliminary mass spectrometric survey of the vaporization of the rare earth sesquioxides has been completed. The results of these studies, with the exception of the work on Ce_2O_3, are given in Appendix I which will appear as a note in the Journal of Chemical Physics.

Preliminary studies of the vaporization of cerium oxides indicated that $CeO_{2(s)}$ vaporizes principally as $CeO_{2(g)}$, and that Ce_2O_3 vaporizes principally to yield CeO with appreciable amounts of $CeO_{2(g)}$ present. Not enough work has been done on cerium oxide to determine whether the $CeO_{2(g)}$ from the sesquioxide vaporization arises from actual vaporization of Ce_2O_3 or from some residual CeO_2 present in the sample. No Ce vapor was observed. Work on these systems is being continued.

D. MOLYBDENUM-SILICON SYSTEM

The studies on the rates of evaporation of silicon from silicon-molybdenum composites of various compositions have been continued during this quarter and are nearing a state of completion.

The purpose of these experiments is to determine the thermodynamic properties of the various equilibrium phases in the molybdenum-silicon system and to reconfirm the phase diagram of this system. The thermodynamic properties are being determined by measuring several isothermal rates of evaporation and converting these measurements into pressures[6] and using these pressures to calculate the ΔF, ΔH, and ΔS of the reactions of interest. The phase diagrams are being determined by using the isothermal rates of evaporation to determine the variation of the activities of the volatile components as a function of composition.[7]

In the previous quarterly report[5], an experimental technique was described in which molybdenum and silicon powders were mixed and allowed to react in the evaporation cell, shown in figure 4, while the rate of evaporation from the cell was being monitored. During the present quarter, a modification of this technique was employed in which silicon powder was placed in a molybdenum crucible and the rate of evaporation of material from the condensed phase was monitored as a function of time. If the net rate of transfer of material to the evaporating surface is sufficient to replenish the material lost due to evaporation then the rates of evaporation measured can be related to the equilibrium decomposition pressure of the various phases in the molybdenum-silicon system. The results of such an experiment are shown in figure 5. On the basis of these experiments, we now interpret the changes in volatility of the various regions of the graphs shown in figures 5 through 9 as follows:

1. The initial region exhibits a decreasing volatility which corresponds to the interaction and disolution of molybdenum in the silicon.

2. The second region is due to the evaporation of silicon from the two-phase region containing liquid silicon (of the prescribed molybdenum concentration) and $MoSi_2$.

3. The subsequent regions correspond to the two-phase regions containing $MoSi_2$-Mo_5Si_3, Mo_5Si_3-Mo_3Si and Mo_3Si-Mo respectively.

We are now identifying the second intermediate compound as Mo_5Si_3 instead of Mo_3Si_2.[8] The X-ray diffraction analysis, where available, of the material remaining in the crucible after an experiment are included in figures 5 to 9.

One of the most serious sources of error in these experiments is the measurement of temperature. All attempts to sight an optical pyrometer directly on the sample or to sight it on a black body hole in the crucible have proved unreliable; therefore, an attempt is being made to calibrate the sample temperature in terms

---

[6] a. Knacke, O. and I.N. Stranski, Progress in Metal Physics, 6,181 (1956);
b. Speiser, R. and H.L. Johnson, Trans. Amer. Soc. Metals, 42,283 (1950);
c. Motzfeldt, K., J. Phys. Chem; 59,139 (1955).

[7] a. Kubaschewski, O. and E. W. Evans, Metallurgical Thermochemistry, Pergamon Press, New York 1958 p. 60, section G;
b. Dimkerley, F.J. and G.J. Mills, Thermodynamics in Physical Metallurgy, American Society for Metals, Cleveland, (1950) pp 47-84.

[8] Aronsson, B., Acta Chem. Scand., 9, 137 (1955).

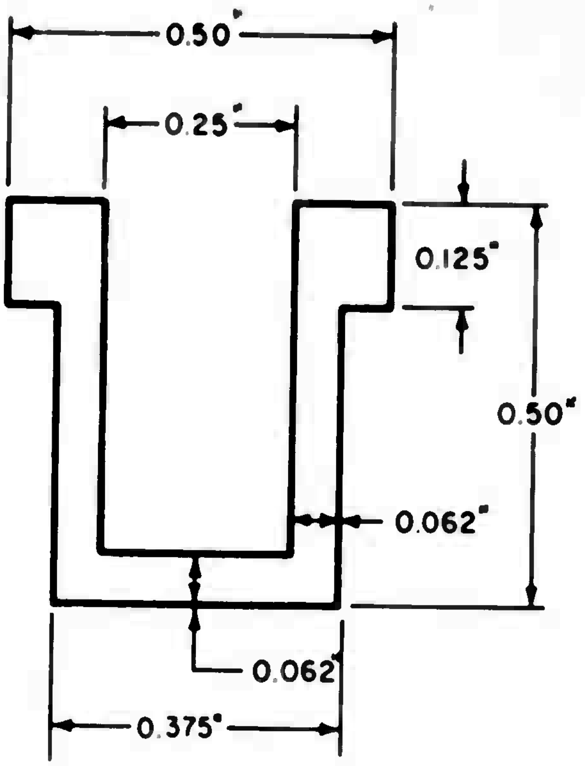

Figure 4 CRUCIBLE SHAPE FOR SILICON EVAPORATION EXPERIMENTS
61-2041

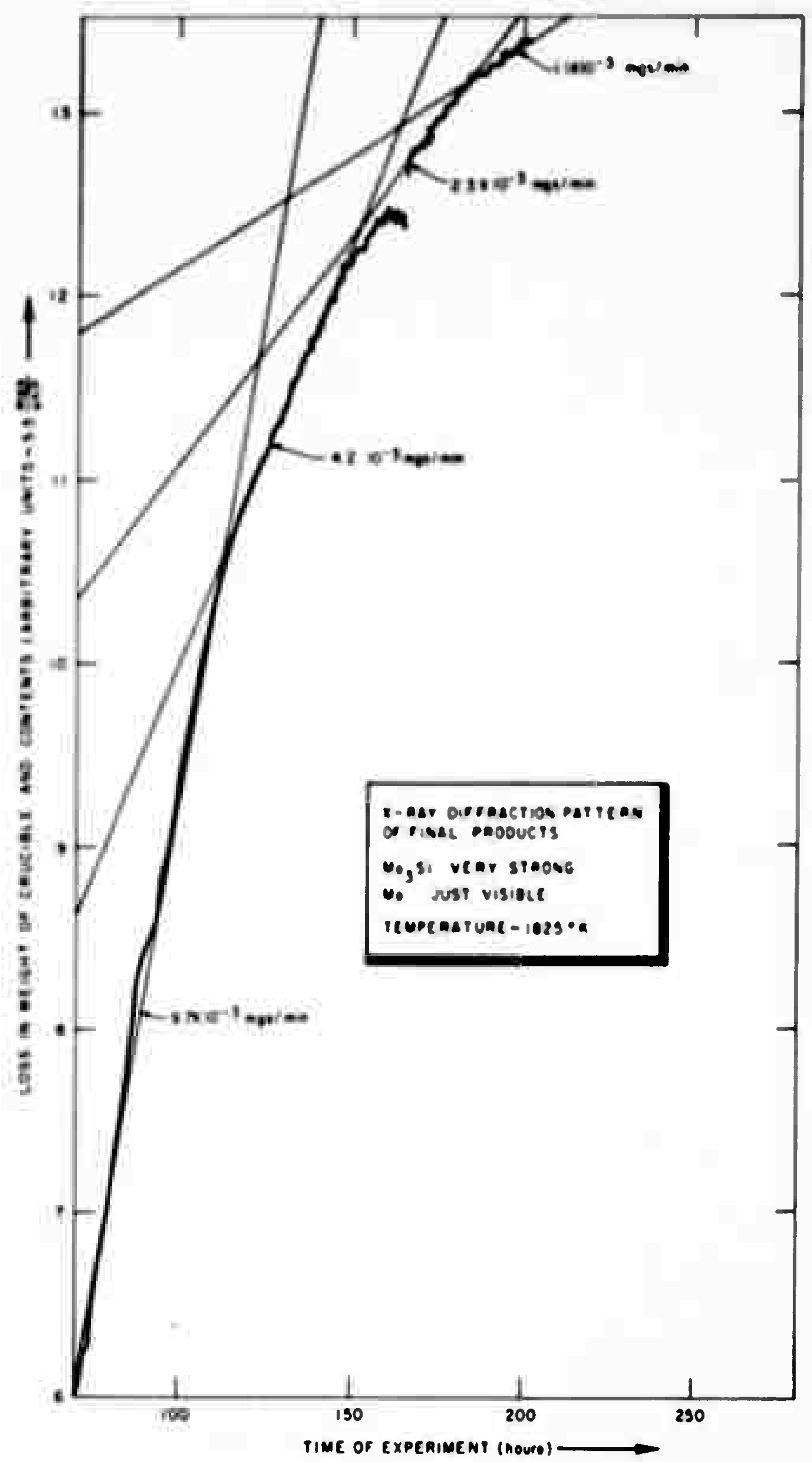

Figure 5 EVAPORATION OF SILICON FROM A MOLYBDENUM CRUCIBLE
61-2040

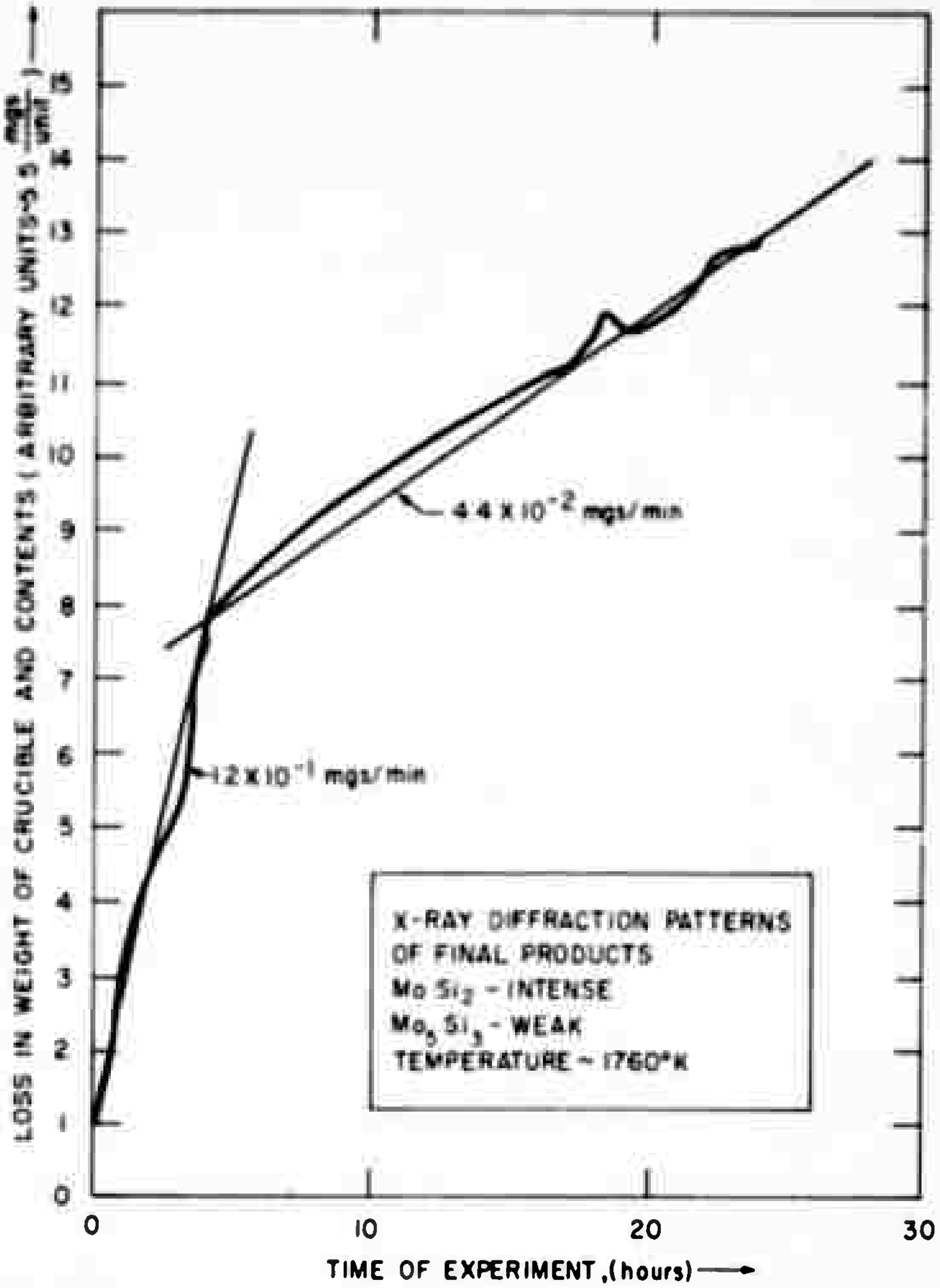

Figure 6 EVAPORATION OF SILICON FROM AN EQUAL WEIGHT MIXTURE OF SILICON AND MOLYBDENUM POWDERS IN A TUNGSTEN CRUCIBLE (II)
61-2041

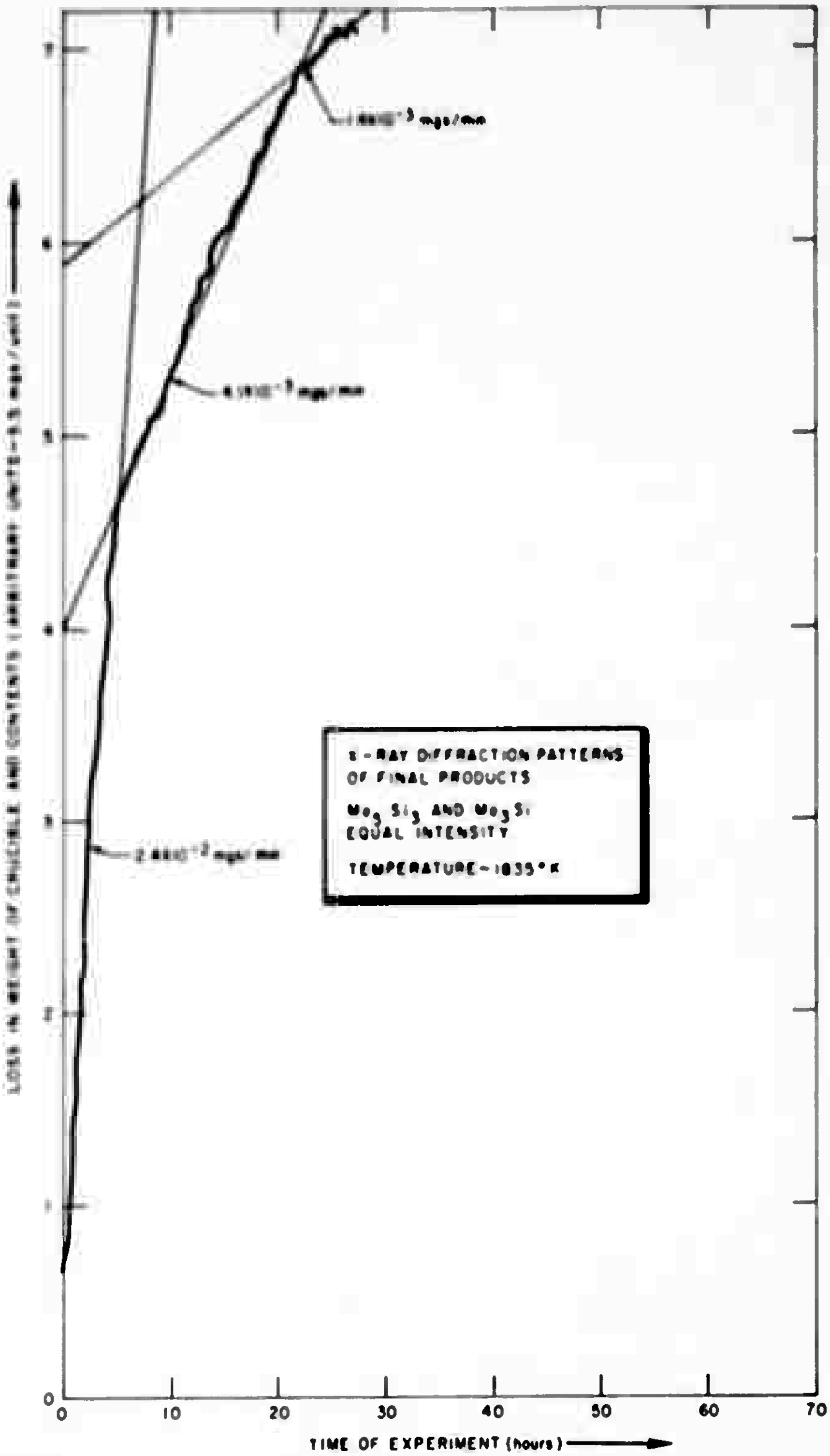

Figure 7 EVAPORATION OF SILICON FROM AN EQUAL WEIGHT MIXTURE OF SILICON AND MOLYBDENUM POWDERS IN A MOLYBDENUM CRUCIBLE (III)
61-2043

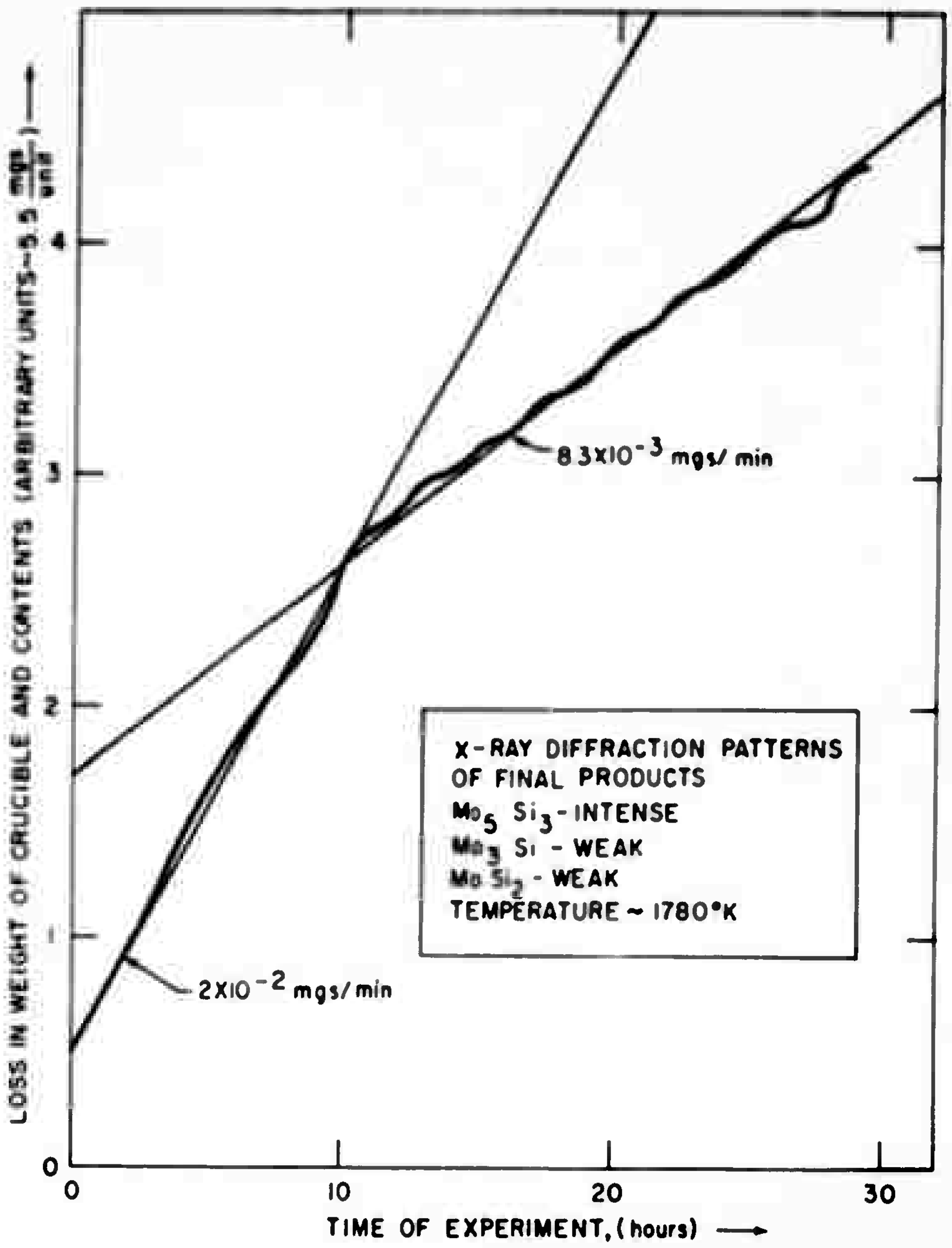

Figure 8 EVAPORATION OF SILICON FROM A MIXTURE OF SILICON AND MOLYBDENUM POWDERS IN THE ATOMIC RATIO Mo:2Si IN A TUNGSTEN CRUCIBLE (IV)
61-2068

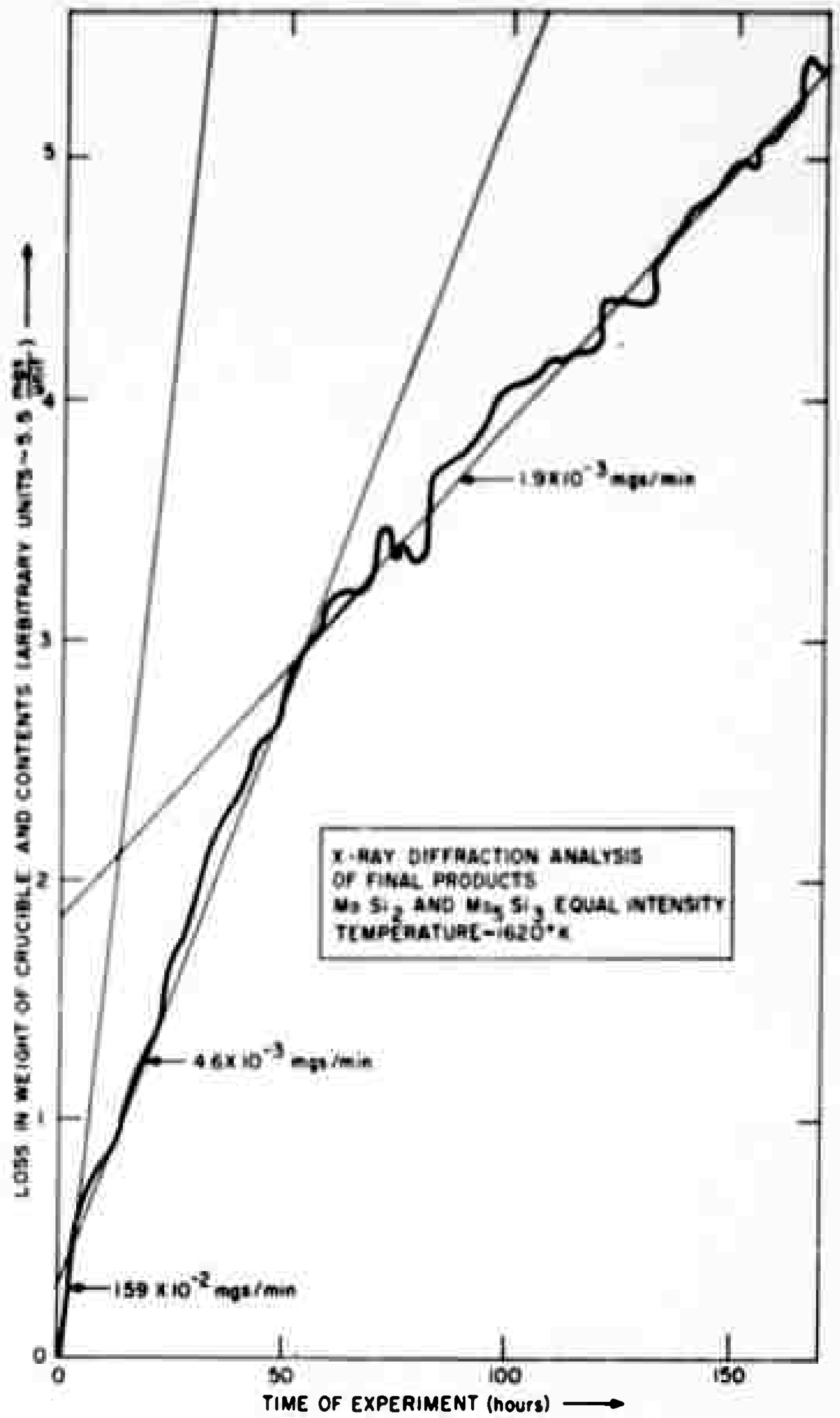

Figure 9 EVAPORATION OF SILICON FROM AN EQUAL WEIGHT MIXTURE OF MOLYBDENUM AND SILICON POWDERS IN A TUNGSTEN CRUCIBLE (V)

of the electrical power fed into the furnace. The power input versus sample temperature will be determined in two ways; first, by comparing the power input to the reading on a thermocouple and also by comparing the power input with the rates of evaporation of materials whose rates of evaporation as a function of temperature have been established.

The vapor species above the various molybdenium-silicon compositions has been determined to be monomeric silicon by means of a Bendix Time of Flight Mass Spectrometer.

Experiments still in progress, or not completed in time to be evaluated for this report, are the following:

1. Simultaneous simple Knudsen effusion experiments on $MoSi_2$, Mo_5Si_3, and Mo_3Si. Three molybdenum effusion cells will be placed inside a larger tungsten crucible. The three samples will be run simultaneously in order to try to avoid any temperature error in determining the ratios of the decomposition pressures of the various two-phase regions.

2. Determination of the phase boundries of the Mo_5Si_3 phase.

3. Temperature calibration.

4. Analysis of the metallographic examinations of the cross-sections of the crucibles after the evaporation experiments.

5. Analysis of the X-ray diffraction patterns of the sample products remaining after the evaporation experiments.

During this quarter, Searcy and Tharp[9] have published the results of a Knudsen effusion investigation of the dissociation pressures of the molybdenum silicides. They found the heats of dissociation per gram atom of silicon at 298°K to be: Mo_3Si 131.9; Mo_5Si_3 131.1; and $MoSi_2$ 117.2 kcal.

Searcy and Tharp and Brewer and Searcy[10] make the point that when data is very scattered a "third law" calculation would be preferable to the "second law" calculation that was performed on our data last quarter[5]. Accepting their reasoning, and with the reservation that our temperatures have still not been calibrated, and using $(\Delta F_T - \Delta H_{298})/T$ given by Searcy we calculate the following heats of dissociation at 298°K: liquid + $MoSi_2$ 117.8; $MoSi_2$-Mo_5Si_3 121.5; Mo_5Si_3-Mo_3Si 128.3; Mo_3Si-Mo 132.9.

As soon as all our experiments are analyzed, our results will be compared with those of Searcy and Tharp.

---

[9] Searcy, A.W. and A.G. Tharp, J. Phys. Chem., 64, 1539 (1960).

[10] Brewer, L. and A.W. Searcy, J. Chem. Educ. 26, 548 (1949).

E. TUNGSTEN-SILICON SYSTEM

Studies similar to those of the molybdenum-silicon system have been conducted on the tungsten-silicon system by measuring the isothermal rate of evaporation of silicon from tungsten crucibles. One such experimental result is shown in figure 10. The same temperature measurement difficulties as those mentioned in the molybdenum-silicon system have been experienced in these experiments and the same remedy will be applied.

F. TANTALUM-BORON SYSTEM

Serious difficulties have been encountered in obtaining continuously monitored effusion data of the tantalum-boron system. In those experiments where boron was evaporated from a tantalum crucible, the final products were found to be boron powder with a coating of tantalum boride on the crucible wall. In the experiments where boron was evaporated from TaB_2 in a tantalum crucible, the final products were non-equilibrium mixtures of various borides, e.g. TaB_2, Ta_3B_4 and TaB. Also in the experiments where the evaporation of boron was monitored from a reacting mixture of tantalum and boron powders, the final products were non-equilibrium mixtures, e.g. Ta, TaB_2, TaB, Ta_3B_4. It appears as if the most promising approach to the determination of the dissociation pressures would be to do Knudsen effusion experiments with very slow rates of evaporation. Such experiments will be conducted in the near future.

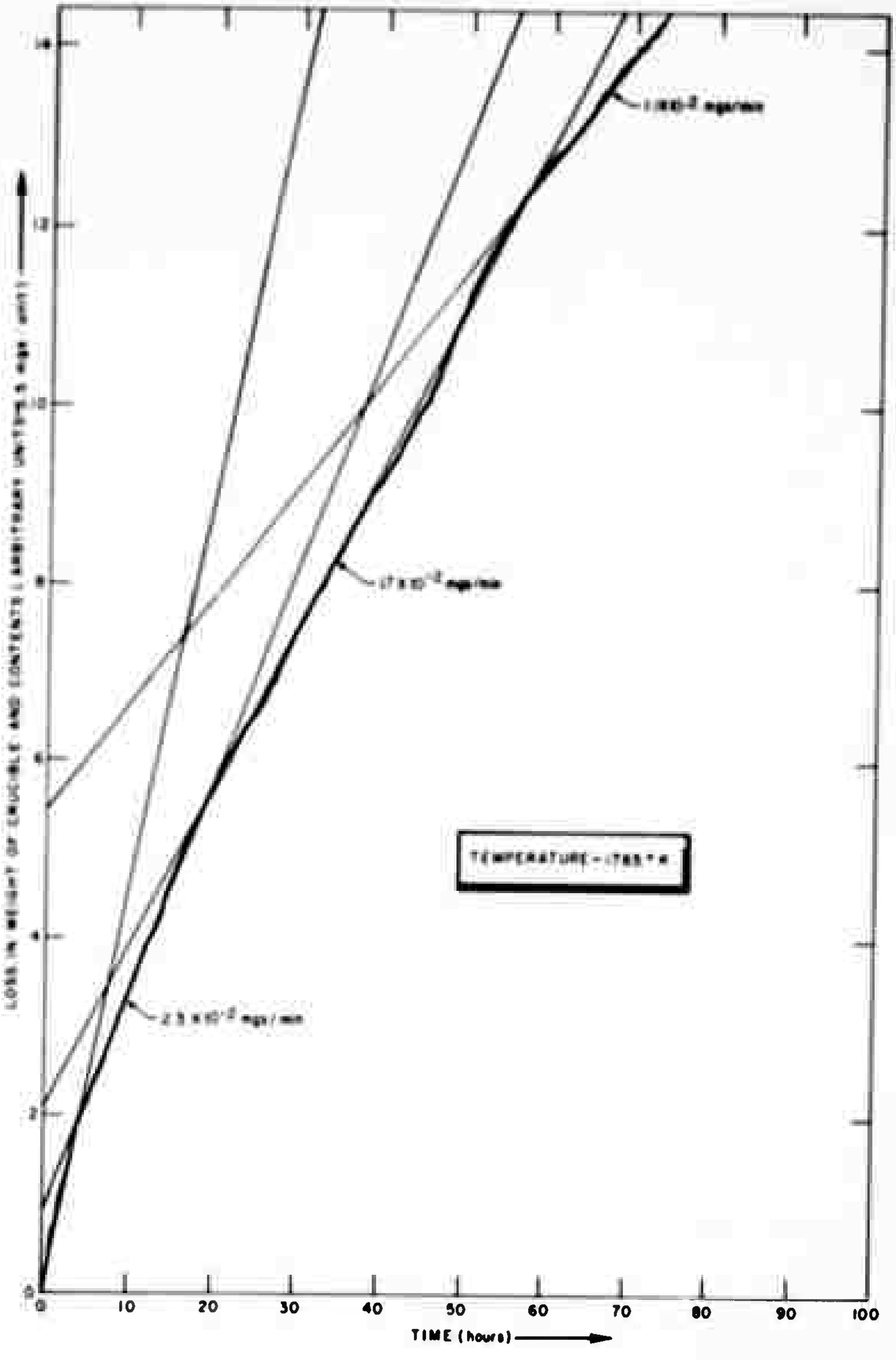

Figure 10 EVAPORATION OF SILICON FROM A TUNGSTEN CRUCIBLE
61-2045

II. VISCOSITY STUDIES

Furnace difficulties have slowed down the effort leading toward a successful conclusion of the determination of the viscosities of silica and alumina and additive additions to silica and alumina. The furnace elements will be replaced and the determinations will be attempted during the next quarter.

III. SURFACE TENSION

The following surface tensions of silica and silica plus additives have been determined so far in this program by means of projecting the image of the profile of sessile drops on "Metallographic" plates[1] and the use of the Bashforth and Adams tables.[11]

SUBSTANCE (Silica + 1%)		SURFACE TENSION ($Ergs/cm^2$)
V_2O_5	Temperature > Melting Point	498
CoO		351
Cr_2O_3		424, 500
Al_2O_3		473, 580
MgO		590
ZrO_2		800
Pure Silica (Temperature °C)		
1830		370
1850		351
1865		281
2140		320
2250		278

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[11] Bashforth and Adams, An Attempt to Test the Theories of Capillary Action, Cambridge, (1883).

APPENDIX A

THE VAPORIZATION OF THE RARE EARTH OXIDES*

Morton B. Panish

RESEARCH AND ADVANCED DEVELOPMENT DIVISION
AVCO CORPORATION
Wilmington, Massachusetts

The vaporization modes of most of the sesquioxides of the lighter rare earth metals have previously been shown to change from

$$M_2O_{3(s)} \longrightarrow 2MO_{(g)} + O_{(g)} \qquad (1)$$

to

$$M_2O_{3(s)} \longrightarrow 2M_{(g)} + 3O_{(g)} \qquad (2)$$

with increasing atomic number of the metal.[1]

In this work, we have studied the vaporization of Gd_2O_3, Tb_2O_3, Dy_2O_3, Ho_2O_3, Er_2O_3, Tm_2O_3, Yb_2O_3, and Lu_2O_3 at temperatures ranging from 2000 to 2500° K by analyzing the species effusing from an iridium effusion cell with a Bendix time-of-flight mass spectrometer. Experimental details of the heating and temperature measuring techniques have been described elsewhere.[2]

The ratios of the ion currents due to the metal and the monoxide, I_{M^+}/I_{MO^+}, are shown in figure A-1 for ions produced with a 20 ev electron beam. The previously reported data on La_2O_3, Pr_2O_3, Nd_2O_3, Sm_2O_3, and Eu_2O_3[1, 3] are also included in the figure. The effusing species were observed at several temperatures and electron beam energies for each of the compounds studied, and the variation of the ion current ratio with temperature over several hundred degrees was found to be within the limits shown about each point in figure A-1. The limits shown for the vaporization of Eu_2O_3 and Tm_2O_3 are quite large because the low concentration of MO obtained with these oxides necessitated measurements at low signal/noise levels. In the case of Yb_2O_3 only Yb^+ ions were observed.

The possibility that the results might have been seriously influenced by dissociative ionization or ion molecule re-combinations within the ion source was examined in a manner identical to that described previously,[1] and these processes were found to be insignificant. It thus appears, that the rare earth sesquioxides

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*This work was supported by the Advanced Research Projects Agency and by the Materials Central, Wright Air Development Division, Wright-Patterson Air Force Base, Ohio.

[1] M.B. Panish, J. Chem. Phys., 34, 1079 (1961).

[2] M.B. Panish and L. Reif, J. Chem. Phys., 34, (1961).

[3] P.N. Walsh, H.W. Goldstein and D. White, J. Am. Ceram. Soc. 43, 229 (1960).

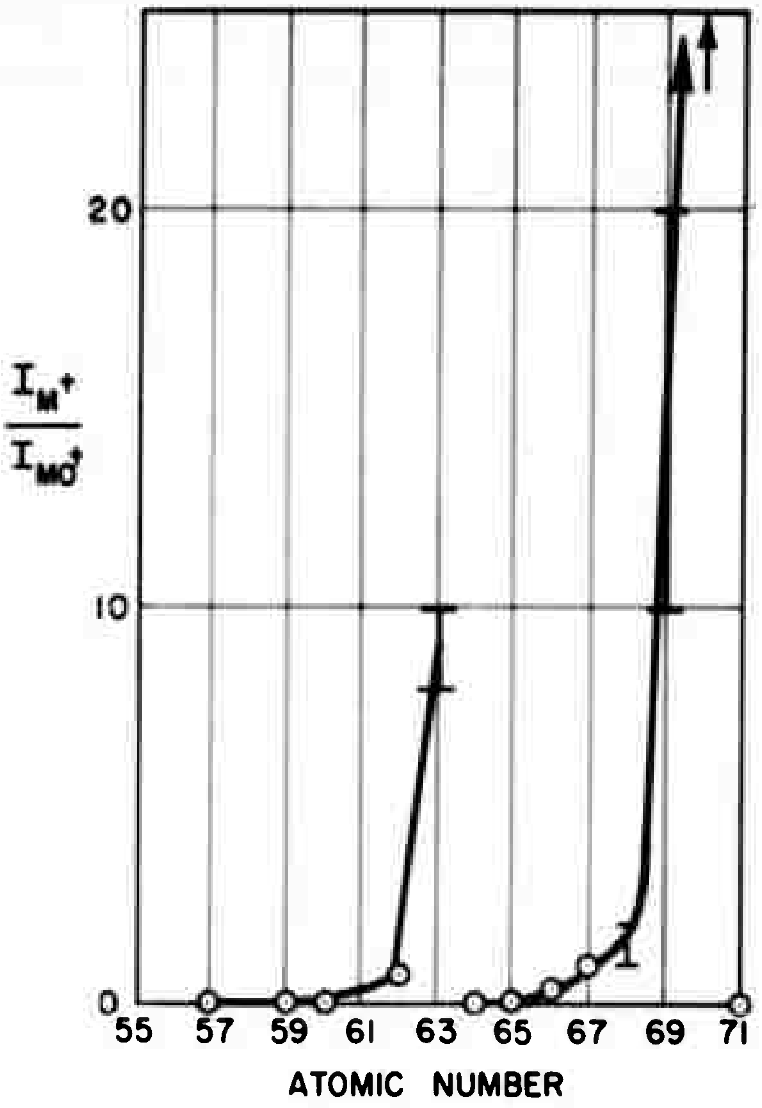

Figure A-1 I_M^+ / I_{Mo}^+ VERSUS ATOMIC NUMBER
61-2046

may be divided into two groups within each of which the vaporization mode changes from that shown in equation (1) to that shown in equation (2) with increasing atomic number of the rare earth metal. It is of interest to note the abrupt change in the vaporization mode which occurs after Eu and Yb, the rare earth metals in which the 4f electron shell is half and completely filled respectively. It is also of interest to note that, except for Lu_2O_3, the classification of the oxides into groups according to their vaporization modes is identical to the separation of the rare earths into so-called cerium and yttrium groups according to their behavior in various separation schemes.[4]

The approximate vapor pressures of the major species vaporizing from the yttrium group of oxides were determined in a manner similar to that described previously for the cerium group. The equilibrium partial pressures over $M_2O_{3\,(s)}$ at 2300 °K were: TbO, DyO, HoO, ErO, LuO $\approx 10^{-7}$ atm; Tm and Yb $\approx 10^{-6}$ atm. In order to obtain the approximate pressures, the iridium ion current from the vaporization of the iridium effusion cell was used for the calibration of the M^+ and MO^+ ion currents.

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[4] D.M. Yost, H. Russel, Jr., and C.S. Garner, The Rare Earth Elements and Their Compounds, John Wiley & Sons, New York (1947).

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