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INVESTIGATION OF SOLUBILITY AND DIFFUSION OF OXYGEN IN REFRACTORY METALS

SECOND QUARTERLY REPORT

by

R. PAPE
L. REED

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-7626

Eimac

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ABSTRACT

Electrical resistivity measurements are to be used to determine the solubility limits and diffusion constants of oxygen in tantalum, Ta-10W, alloy T-222, columbium, Cb-1Zr, and alloy FS-85.

An oxygen solubility apparatus is operational. Three trial solubility experiments have been conducted with Cb specimens and five with tantalum specimens.

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I.   INTRODUCTION

This investigation concerns oxygen diffusion
and solubility in commercially pure refractory metals
and in advanced alloys for space power system components.
Oxygen contamination of refractory metals reduces their
resistance to alkali metal corrosion. Accurate know-
ledge of oxygen diffusion constants and solubility
limits are required in order to establish realistic
engineering and environmental specifications.

This investigation is divided into two phases;

Phase I. A determination of the limits of oxygen
solubility in commercially pure tantalum and columbium
and in the alloys Cb-1Zr, Ta-10W, FS-85 (Cb-28Ta-10.5W-
0.9Zr), and T222 (Ta-9.8W-2.4Hf-0.01C), at five tempera-
tures in the temperature range of 1000°F to 2400°F.

Phase II. The determination of average diffusion
constants of oxygen in the materials listed above across
four different concentration couples and at four tempera-
tures in the range of 1600°F to 2400°F.

Solubility limits and diffusion constants will be
determined from electrical resistivity data using commer-
cially obtainable wires for specimen samples. Absolute
oxygen contents shall be determined by neutron activation
analysis. Other methods of analysis such as electron
microprobe analysis, X-ray diffraction analysis, micro-

hardness and conventional chemical analysis will be
employed for obtaining corroborating data.

It is expected that the data obtained on the pure
metals will confirm the previously reported data in the
literature and provide the required confidence level
to pursue alloy solubility data collection.

II.   <u>SUMMARY</u>

1. Modifications and additions were made to the solubility apparatus.

2. A double vacuum 1500°C furnace has been designed and partially constructed. This furnace will supplement the 1000°C furnace presently used with the solubility apparatus.

3. Three trial solubility runs have been conducted with columbium specimens and five with tantalum specimens. Distinct inflection points in the data plots of "Resistance versus Oxygen Charges to the System" give oxygen solubility values which are in general agreement with published resistivity data. Test runs using the alternate technique of "Iso-Oxygen Concentration" have produced no apparent inflection points though statistical analysis of the data may reveal their existance.

III. EXPERIMENTAL PROCEDURE

Several techniques were considered for oxygen solubility determination during the first quarter and one was discussed in some detail (Iso-Concentration). However, in the light of experience gained with the trial solubility runs on tantalum and columbium performed during this quarter, it is felt that the Isothermal Technique is preferred, in some cases at least, and possibly for all the subject materials. Both techniques will be described again briefly.

A. Iso-Concentration Technique

With this technique the procedure is to hold the oxygen content in the wire specimen constant while the temperature of the vacuum furnace is reduced (or increased) in discrete steps, (See Figure Ia.) Resistance is measured at each temperature and a resistance value obtained. A plot of resistance vs corresponding temperature is then made (Figure Ib). While the absolute resistance is increased by oxygen in solution, i; the near vicinity of, but above the saturation temperature (T_s) for the oxygen content of the specimen (Z ppm), the slope of resistance versus temperature will reflect the temperature coefficient of resistivity for the oxygen-free material. In the near vicinity of, but below the saturation temperature, the slope of resistance versus temperature will be a function of both the temperature coefficient of the metallic phase
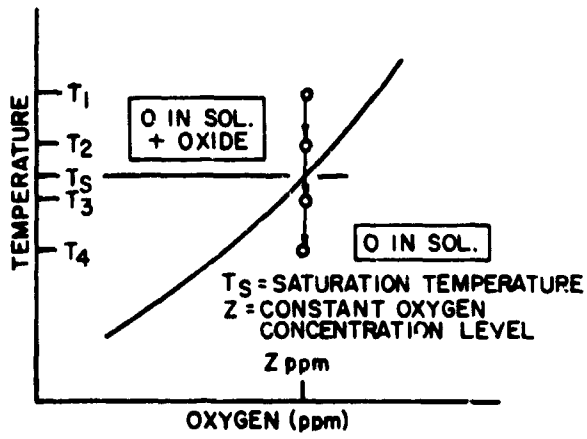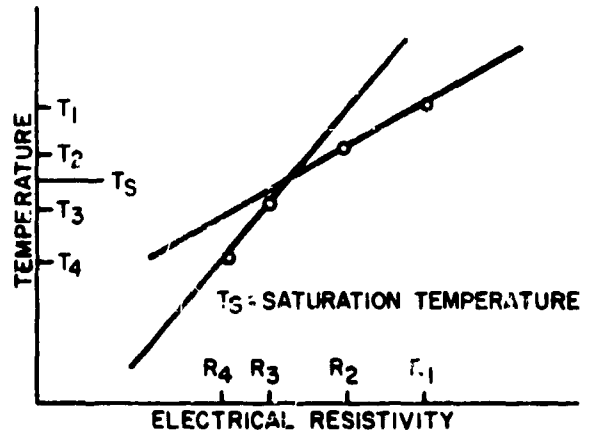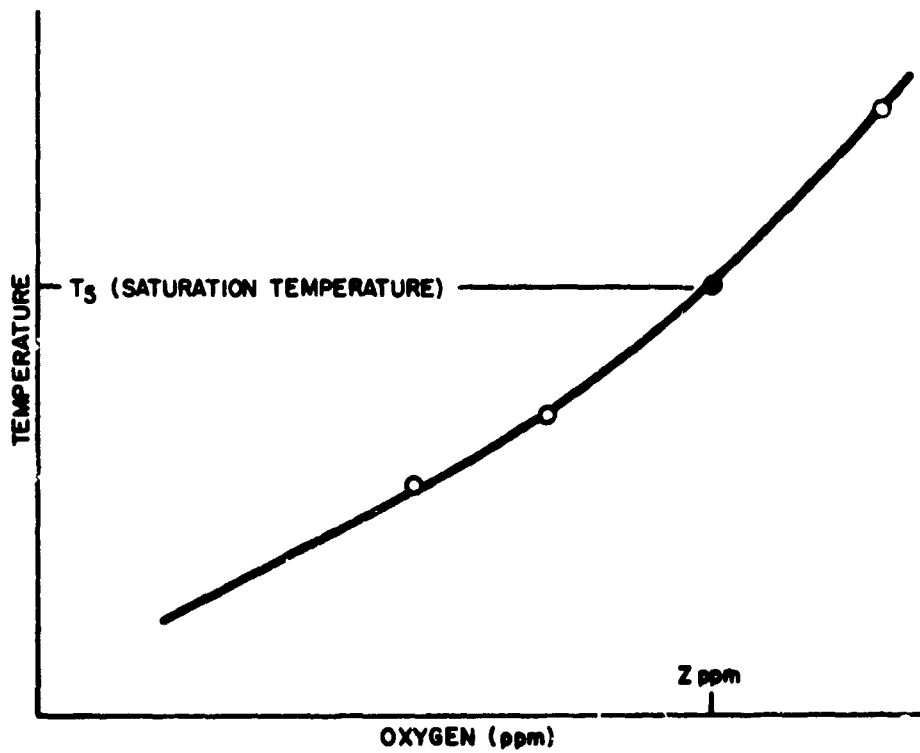
FIG. I. ISOCONCENTRATION TECHNIQUE

and that of the precipitated oxide phase(s). The two slopes will intersect at Temperature T_s where T_s represents the temperature at which the oxide phase will first precipitate. Oxygen content (ppm) is determined after the experimental run by means of neutron activation analysis. The procedure is repeated at various oxygen concentration levels and the curve for saturation temperature vs oxygen concentration can be plotted (Figure Ic).

This technique is predicated upon the assumption that (a) oxygen pickup (or loss) after initial engassing is negligible; (b) the temperature coefficient of resistivity will be linear for the alloy materials; (c) the slope of resistance versus temperature will be distinct and different above and below the saturation temperature; (d) no oxide precipitated will have a high enough vapor pressure to effect significant loss of material over the period of time in which the specimen is at temperature in vacuum.

B.   Isothermal Technique

The procedure with this technique is to cross the oxygen solubility curve isothermally by reaction with successive discrete charges of oxygen. The resistivity will increase linearly in accordance with Nordheim's rule [I] for small increments of oxygen. When the saturation limit is exceeded, the slope of $\Delta R/Ro_T$ vs (number of charges admitted) will be altered due to the resistivity

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ɔf the oxides forming. Extrapolation of the two linear portions of the resistance versus oxygen charge curve will indica.e the saturation point for the given constant temperature in terms of the number, or fractions thereof of oxygen charges admitted and in terms of the corresponding increment of resistance. The next step is to determine the saturation point in terms of oxygen content.

If the quantity of oxygen in each charge admitted to the reaction tube were accurately measured and if each charge were reproducible, then the cumulative quantity of oxygen in solution corresponding to the saturation point could be determined, but only if all the oxygen admitted could be accounted for. This would require determining the quantity absorbed by the walls of the reaction tube, etc.

Alternatively, it is proposed to determine the oxygen content corresponding to the saturation point by relating the incremental resistance to oxygen in solution (in parts per million) by means of neutron activation analysis. A calibration of $\Delta P/P_O$ versus oxygen content in ppm will be obtained by preparing a series of calibration specimens (Figure 2a) which can then be used to correlate the incremental resistance at the saturation point to oxygen in solution (Figure 2b). The quantity of oxygen in each charge need not even be known nor the
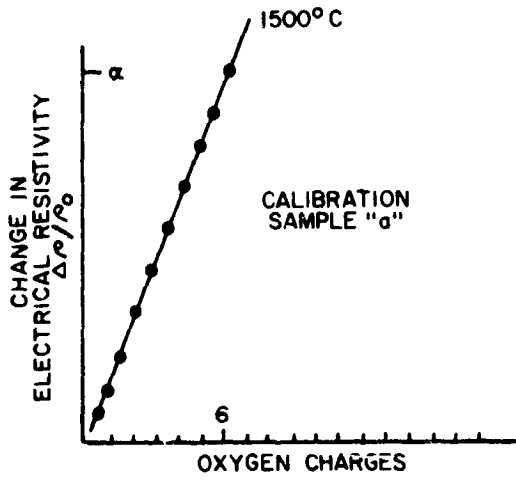
(a)
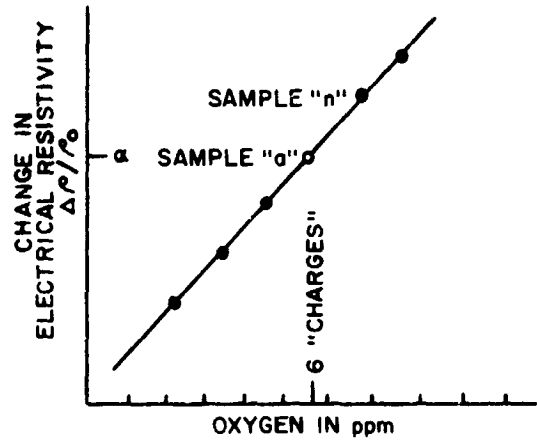
(b)
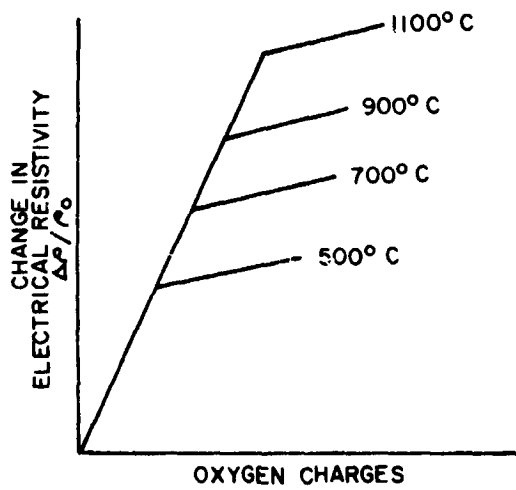
(c)
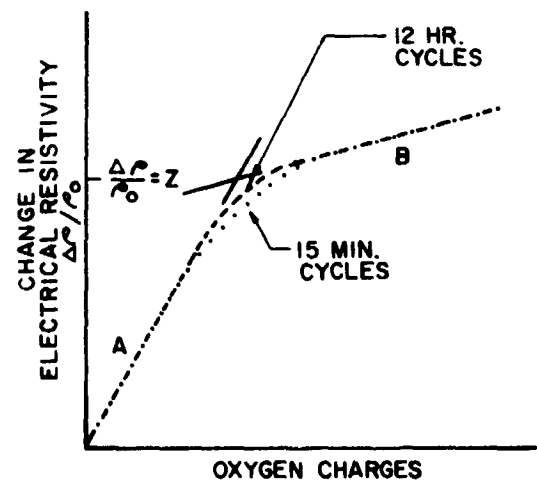
(d)

FIG 2. ISOTHERMAL TECHNIQUE

disposition of the oxygen in the reaction tube; it is only necessary to provide that the admitted charges are uniform.

Were the "Isothermal Technique" to be performed under equilibrium conditions the results of a series of runs would be as depicted in Figure 2c. Figure 2d is an approximate representation of two runs performed under non-equilibrium conditions; the resultant curve is not composed of two line segments but rather of two linear segments connected by a transition region. While the period of time between charges will determine the extent of this transition region, the saturation point will not be affected.

IV. APPARATUS

A. Modification and Additions to Solubility
Determination Apparatus

The solubility determination apparatus incor-

porating a 1000°C air furnace was described in the first

quarterly report.

1. Oxygen Feed System

The oxygen feed system was originally assembled

with Swag-lok fittings on the stainless steel tubing.

The object was to verify the effectiveness of the oxygen

feed system design and to evaluate the various component

parts before combining them in a permanent installation.

Early in this quarter, the oxygen feed system was

dismantled and rebuilt. The Swag-lok fittings were re-

placed by welded joints; three Hoke valves were replaced

by Nupro valves; and the system was rearranged for con-

venience (see Figure No. 3).

A Veeco TG-20 Thermocouple vacuum gauge (0 to 20

torr) was added to the expansion chamber and a Veeco

TG-6 Thermocouple vacuum gauge (1 to 1000 microns) was

installed to monitor pressure in the balance of the system.

The entire oxygen feed system was determined ఆ

be vacuum tight by testing on a Veeco helium leak detector.

2. Oxygen Source

The oxygen supply tank used previously was re-

placed by a new cylinder of research grade oxygen

FIG. 3. OXYGEN FEED SYSTEM

TO FURNACE

TO PUMP

FROM OXYGEN SUPPLY

V1

V5

V2

VL

MV

V3

V4

300cc EXPANSION CHAMBER

THERMOCOUPLE VACUUM GAGE 0-20 TORR

THERMOCOUPLE VACUUM GAGE 0-1000 MICRONS
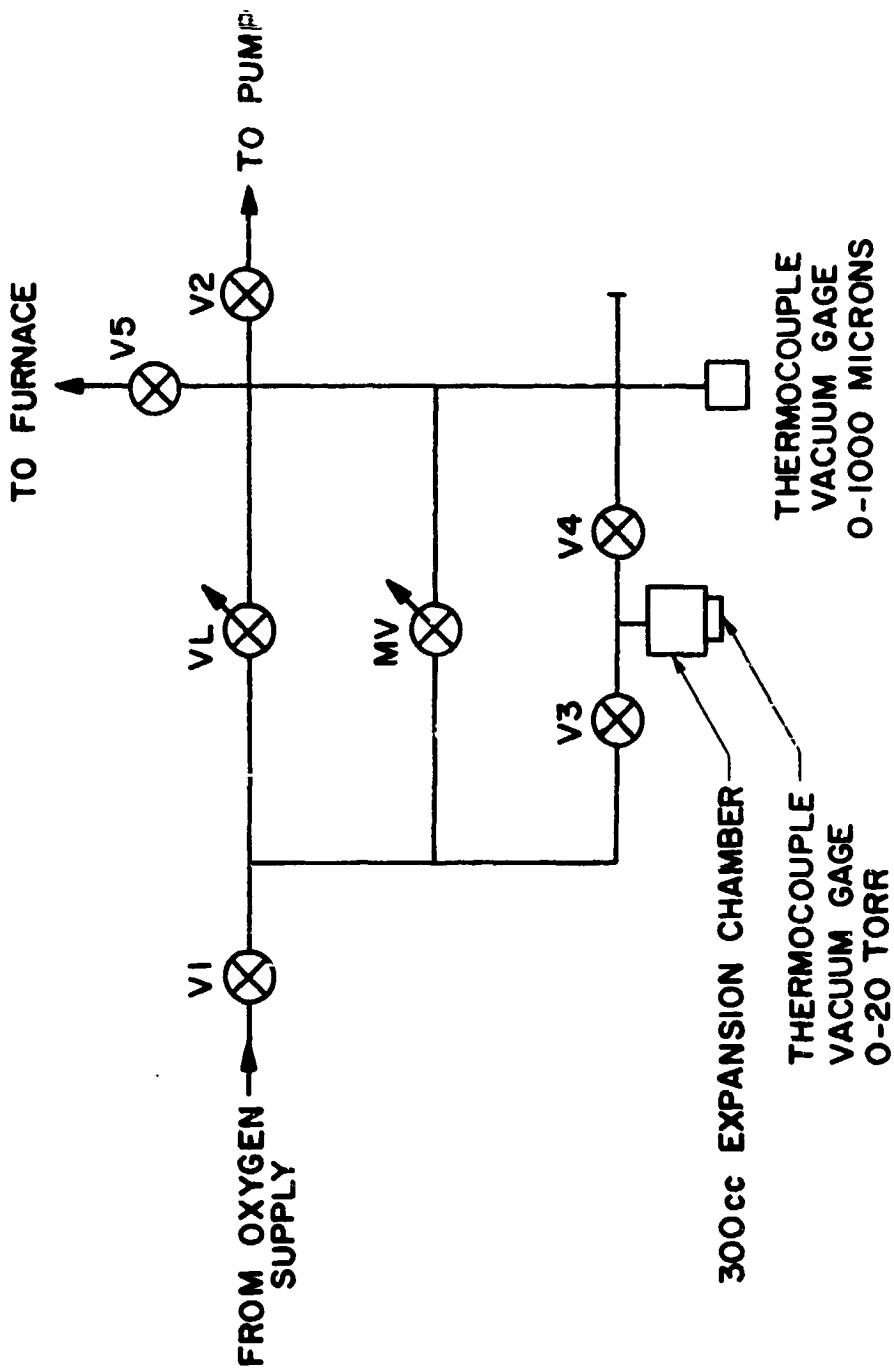
MV=MICROMETER METERING VALVE
VL=GRANVILLE-PHILLIPS VARIABLE LEAK
V =HIGH VACUUM BAKABLE VALVES

purchased from the Liquid Carbonic Division of General
Dynamics. This is 99.999% oxygen as determined by
mass spectrometer techniques at McClellan AFB. (See
Table 1 for analysis of impurities).

3. Milli-torr Gauge & Recorder

A Milli-torr high pressure ionization gauge
(10^{-6} to 1 torr) was mounted on the "Dutchman" (vacuum
manifold) and the output from its controller connected
to a Moseley Model No. 680 recorder. The recorder was
installed in the system in order to monitor the oxygen
pressure in the reaction tube of the furnace while sample
specimens are being engassed. The recorder provides a
permanent record of oxygen pressure; it also serves as an
elapsed time indicator, and is a time reference during
an experimental run.

B. Sample Holder

The sample holder and the top flange of the
"Dutchman" constitute the sample holder assembly depicted
in Figure 4. The high current leads are of copper and
support two molybdenum rods which extend into the hot
zone and provide a mounting for the wire specimen. The
two potential leads are of molybdenum wire and contact
the coiled wire sample a turn and a half from each end
so as to minimize the end heat loss effect due to con-
nection of the wire sample to the high current leads.

TABLE 1	
Impurity Analysis of 99.999% Oxygen (Cylinder #3710)	
Impurity	Content
Nitrogen	4 ppm
Hydrogen	3 ppm
Water	1 ppm
CO	0.8 ppm
CO_2	0.2 ppm
Halogenated Hydrocarbon (probably Trichlorethylene)	0.1 ppm

SPECIMEN

POTENTIAL LEAD

THERMOCOUPLE LEADS

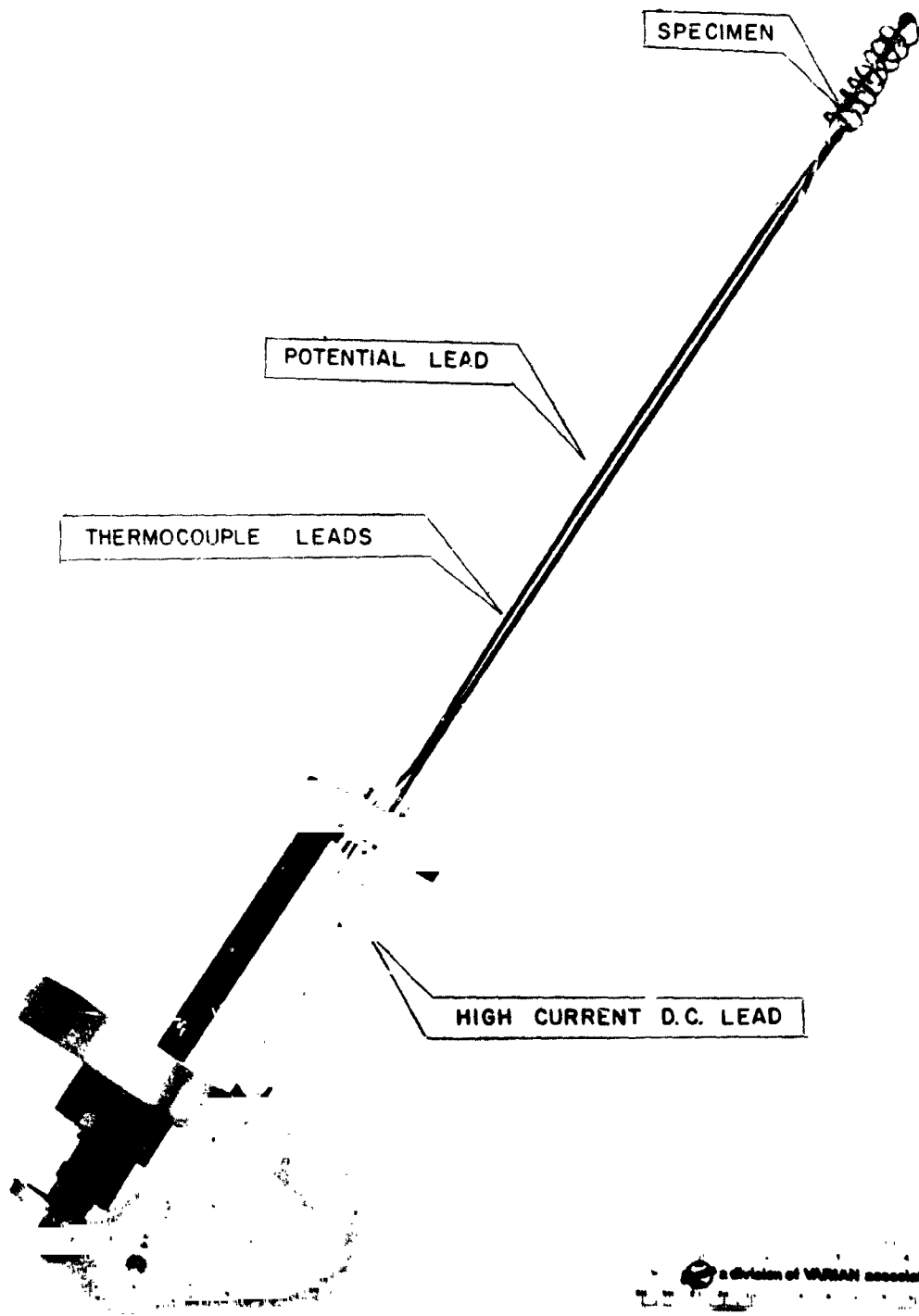HIGH CURRENT D.C. LEAD

a division of VARIAN associates

FIG. 4 SAMPLE HOLDER

C.   1500°C Double Vacuum Furnace

A double vacuum furnace has been designed and constructed during this quarter. This furnace will supplement the 1000°C air furnace currently in service with the oxygen solubility determination apparatus. (See Figures 5 and 6).

The 1500°C furnace is to be mounted vertically below the "Dutchman" and is resistance heated. Ten 0.050 diameter tungsten hairpin filaments are employed as heating elements. Each filament loop is suspended vertically by its ends and is maintained taut by a weight hung from the bend of the loop (Figure 7). While it is intended to operate the filaments on single phase alternating current, provision has been made to permit three phase operation. Between the filaments and the outer stainless steel furnace are six concentric molybdenum radiation shields (see Figure 8).

Power is supplied to the heater elements from a 100 amp. Silicon Controlled Rectifier power supply controlled by a Leeds and Northrup CAT Series 60 Controller which amplifies an error signal from a Leeds and Northrup Speedomax H Recorder. Target specifications call for a zone of uniform temperature at least 10 inches in length. The reaction tube is 30 inches long; the overall length of the outer furnace jacket is 38.5 inches and the filaments

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FIG. 5 REAR VIEW OF 1500°C DOUBLE VACUUM FURNACE
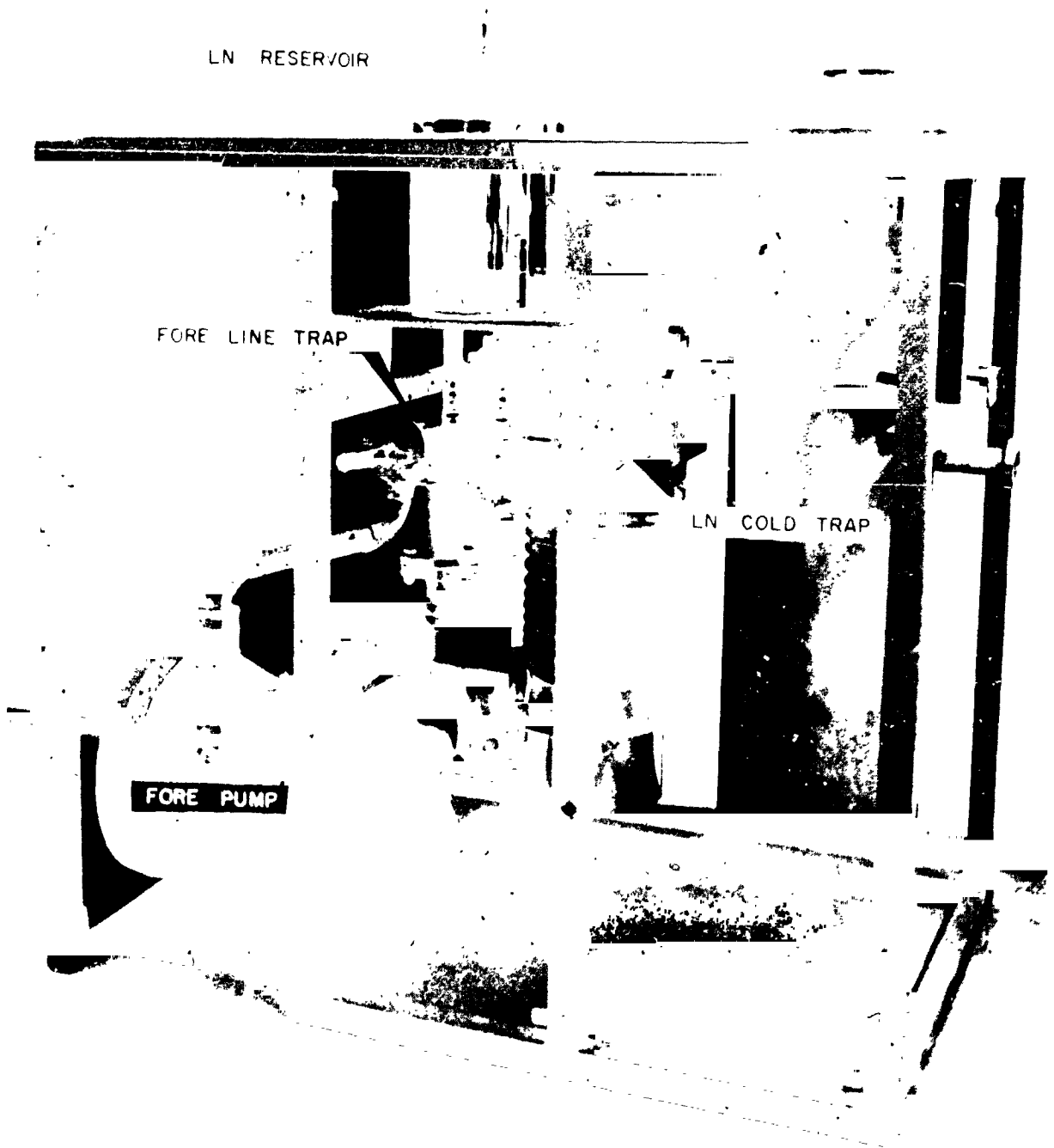
FURNACE JACKET TOP FLANGE

CURRENT ROD ASSEM.'S (20)

HEATER, PULLEY & WEIGHT ASSEM.'S (10)

TUNGSTEN HEATER ELEMENT (10)

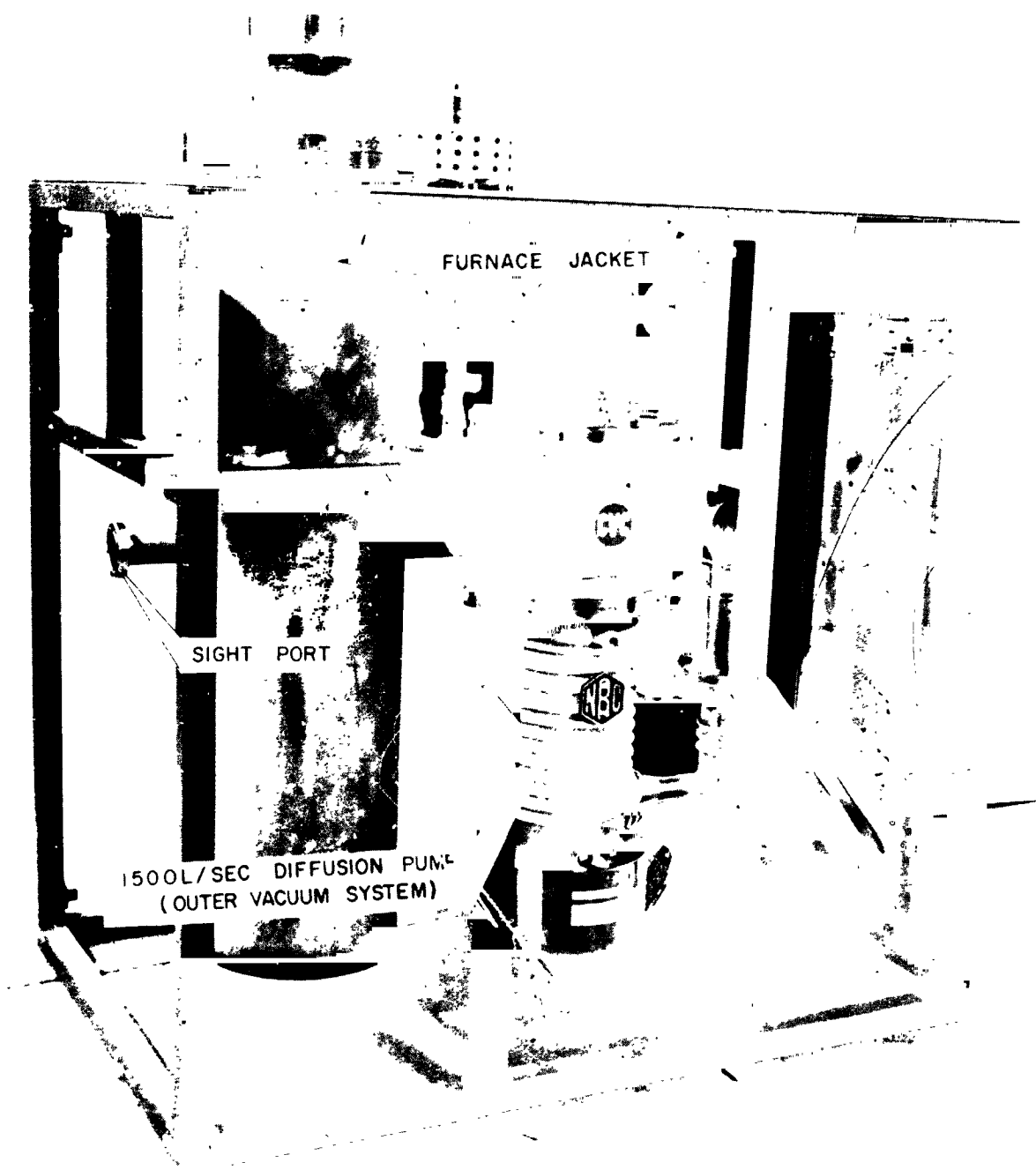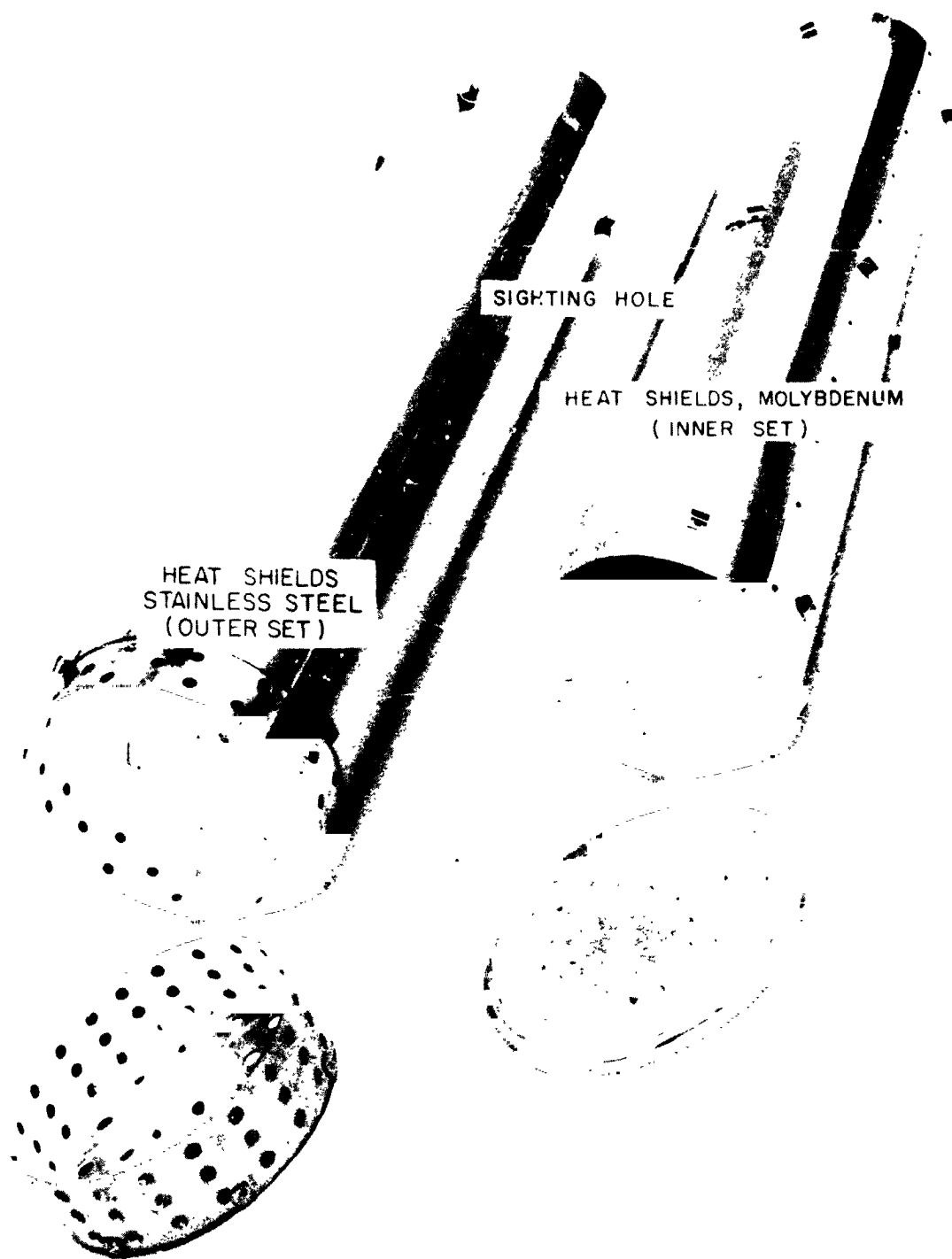FIG. 7 HEATER ELEMENTS FOR 1800°C DOUBLE VACUUM FURNACE

FIG. 6 FRONT VIEW OF 1500 °C DOUBLE VACUUM FURNACE

SIGHTING HOLE

HEAT SHIELDS, MOLYBDENUM
(INNER SET)

HEAT SHIELDS
STAINLESS STEEL
(OUTER SET)

FIG. 8 HEAT SHIELDS FOR 1500 °C DOUBLE VACUUM FURNACE

are approximately 20 inches long (40 inches wire length).

The double vacuum furnace employs two independent vacuum systems. The outer vacuum protects the tungsten filaments from oxidation and isolates the sample specimen from gasses which might otherwise permeate through the dense alumina reaction tube from the surrounding atmosphere at elevated temperatures. (While dense alumina is impervious at room temperatures, it becomes pervious at temperatures of 1500°C and above. There is little or no data available between these two temperature ranges, although some work has recently been started in this region*). The present arrangement for vacuum bake out of the alumina reaction tube is judged to be adequate.

The reaction tube volume of the double vacuum furnace is pumped through the existing "Dutchman" by a 40 liter/sec Vacion pump. The reaction tube itself is brazed to a sealing ring which is heliarc welded to a double conflat vacuum flange. Reaction tubes are thus interchangeable and may easily be replaced without dismantling more than the Dutchman.

A quartz reaction tube may be substituted for the dense alumina for the 1000°C-and below range.

The reaction tube can be eliminated entirely should its outgassing prove to be a problem. The standard

*R. Fulrath, Univ. of Calif., Berkeley

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isothermal technique would have to be modified, however.

A viewing port with a glass window has been provided for sample viewing with the latter two arrangements.

The outer (furnace cavity) vacuum system is pumped through an arm on the outer stainless steel (304S.S.) jacket of the furnace. A 1500 liter/sec diffusion pump (NRC #HKL 1500), a liquid nitrogen cold trap (CVC3BC-60A) and a mechanical pump (Welch #1397-B) constitute the pumping system. This arrangement is also adequate in the event that the alumina tube is omitted.

V.   RESULTS

During this quarter trial solubility runs were
conducted to check out the solubility apparatus with
the 1000°C air furnace and to try out the proposed
experimental procedures. All segments of the apparatus
functioned satisfactorily with the exception of the
oxygen feed system. A slow leak made it impossible to
introduce controlled reproducible amounts of oxygen into
the reaction tube; the subsequent failure of a micrometer
metering valve further complicated matters. Three trial
solubility runs with columbium and one trial solubility
run with tantalum were carried out, nevertheless, in
order to check the performance of the balance of the
apparatus before shutting down for repair. As a result
of these initial runs, several oxygen introduction methods
were eliminated as impractical and one promising technique
was developed. The oxygen feed system was then rebuilt
(see Section IV above) after which four more trial solu-
bility runs were completed with tantalum.

A.   Trial Solubility Runs With Columbium

Three trial solubility runs were conducted with
0.020 diameter columbium wire specimens for the purpose
of testing out the equipment. See above.

It had been anticipated that oxidation of the
surface of the columbium wire would be a critical factor

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because (a) the formation of CbO_2 layer may inhibit further reaction with oxygen and (b) the presence of the surface oxide may alter the apparent volume resistivity of the specimen. It was found that the minimum pressure at which oxygen could be introduced was 10^{-4} torr (due to a defective valve) under which condition a grey oxide formed on the surface of the specimen. This oxide, probably CbO, was easily absorbed into the volume of the specimen at 1400° C or above.

A deep purple oxide (Cb_2O_3?) formed at higher admittance pressures was very difficult to absorb even at 1600° C.

The reaction tube used in the 1000° C Air Furnace has a bore of approximately 0.5 inch. Oxygen introduced at pressures above 10^{-4} torr reacted preferentially with the portion of the sample that was nearest the gas inlet. One of the engassing procedures investigated was that of admitting gas with the walls of the reaction tube and the specimen at room temperature and then heating the specimen by passing a current through it. After engassing the specimen the furnace is raised into position and the reaction tube brought up to temperature. While this method solves the above problem, it creates many others, such as non-uniform engassing due to temperature gradients in the self-heated specimen. The problem can be eliminated

by introducing gas at lower pressures and using a
larger reaction tube bore. (The 1500°C Double Vacuum
Furnace has a 3.5 inch bore.)

B. Trial Solubility Runs With Tantalum

Five trial solubility runs were completed with
0.020 inch diameter tantalum wire specimens. The first,
run number TA01 was undertaken before the oxygen feed
system was rebuilt and served mainly to check out the
solubility apparatus, as previously noted above.

RUN TA01 (Iso-Concentration Technique)

The procedure on Trial Run TA01 was to engas the
sample at 900°C to an oxygen concentration considerably
below saturation for that temperature. (The ratio of
the change in resistance of the specimen at temperature
to the "oxygen free" resistance of the specimen at
temperature, $(\Delta R/Ro)_T$ = const. is a function of the
amount of oxygen in solution[1,2].)

Engassing was terminated when the ratio $\Delta R/Ro$
reached 0.074. The specimen temperature was then de-
creased by steps and the resistance was measured at each
step in accordance with the procedure described above
under "Iso-Concentration Technique" (Section III A).
Data from this phase of the run was plotted (See Figure 9)
No inflection was observed visually.

OXYGEN ENGASSED:
$\Delta R/R_0 = 7.4\%$

TRIAL SOLUBILITY RUN TA 01
SAMPLE: Ta WIRE .020 DIA.
NOTES
ATTEMPT TO FIND INFLECTION
POINT BY ISO-CONCENTRATION
TECHNIQUE
ELAPSED TIME: 120 MIN.
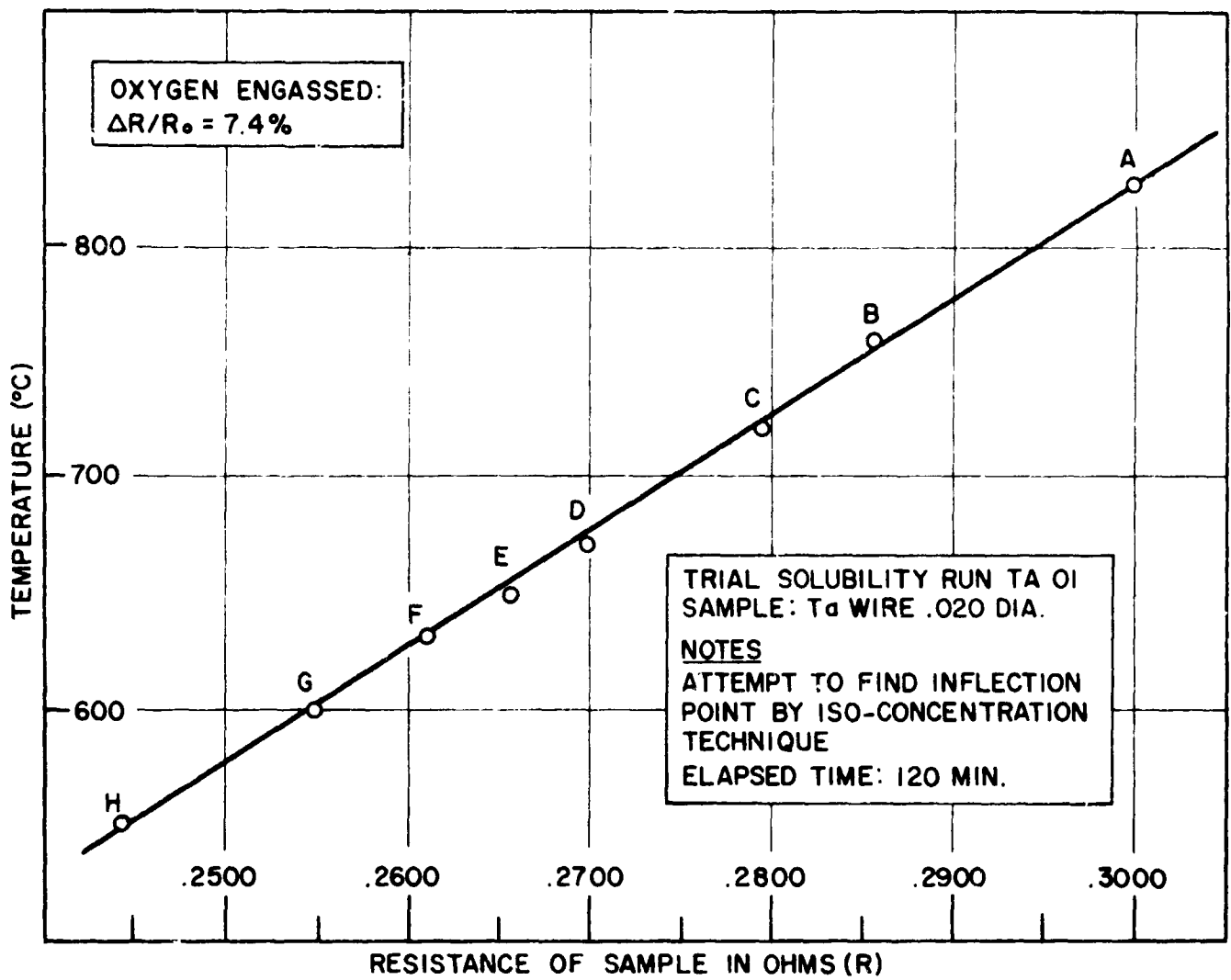
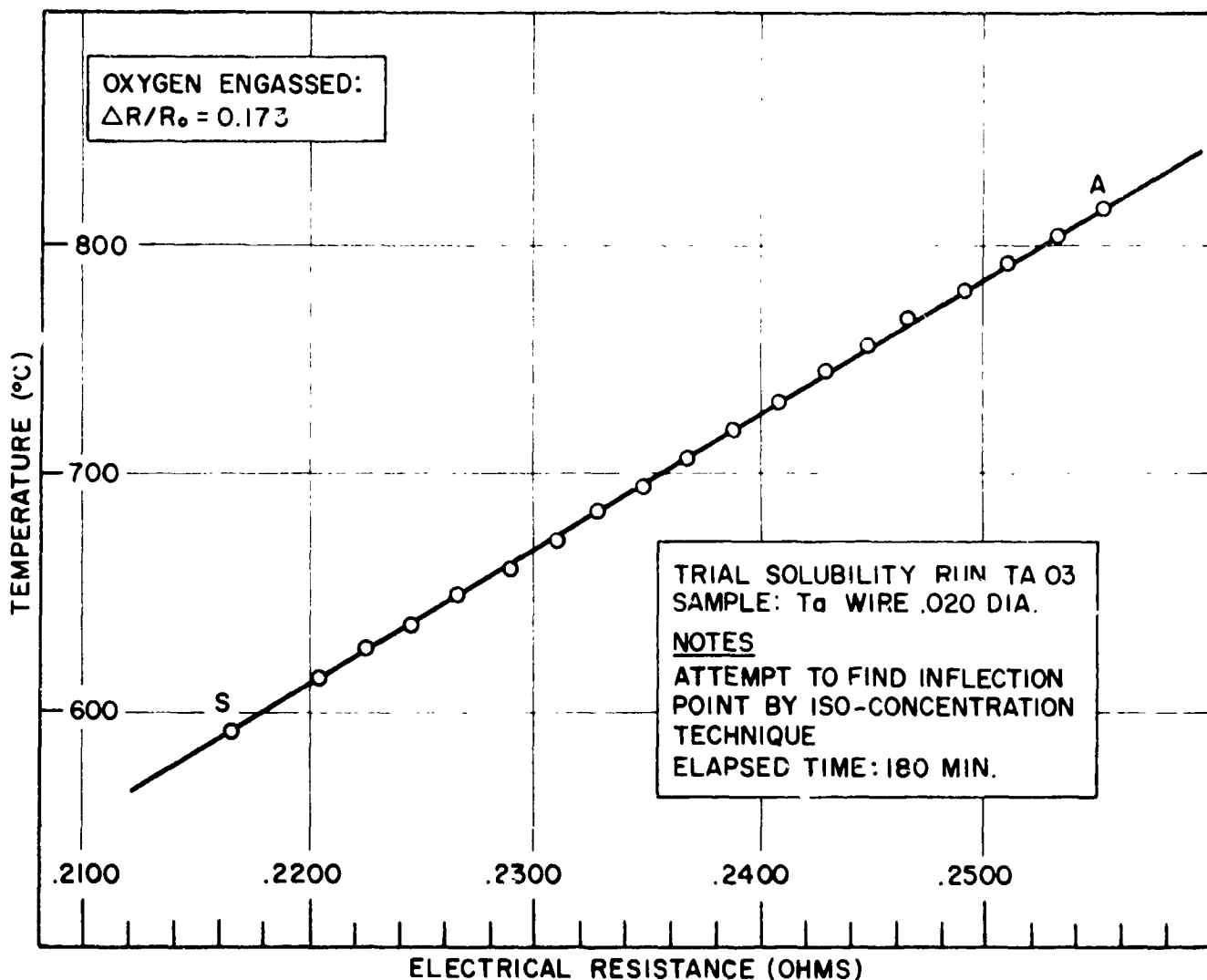| TRIAL SOLUBILITY RUN TA 01		
POINT	RES.(OHMS)	TEMP.(°C)
A	.2999	825
B	.2857	758
C	.2796	720
D	.2699	670
E	.2655	650
F	.2613	630
G	.2550	600
H	.2440	550

FIG. 9. ISOCONCENTRATION SOLUBILITY RUN TA 01

RUN TAO2 (Iso-Thermal Technique)

The second run, TAO2 was an attempt to find an inflection point associated with saturation, by means of the "Isothermal Technique" (Section III B) at 900°C. An indication of the presence of an inflection point was obtained at $\Delta R/Ro = 0.11$, but operational errors and equipment failure rendered the results quantitatively inconclusive.

RUN TAO3 (Iso-Concentration Technique)

The third run, TAO3, employed the Iso-Concentration Technique. In view of the failure of the Iso-Concentration Technique to produce a distinct inflection point in run TAO1, at the oxygen concentration level corresponding to $\Delta R/Ro = 0.074$, the specimen was engassed to a higher oxygen content level ($\Delta R/R_O = 0.173$). According to published resistivity vs oxygen concentration data[2], the solubility limit at 900°C occurs at approximately $\Delta R/R_O = 0.205$, so that an inflection indicating saturation should have occurred at some point in the temperature vs resistance plot (over the range 600 to 840°C) for one or another of the two concentration levels. As with run TAO1, no inflection was observed visually in the temperature vs resistance data plot for run TAO3. (See Figure 10).

FIG.10. ISOCONCENTRATION SOLUBILITY RUN TA 03

Within the graph:

OXYGEN ENGASSED:
$\Delta R/R_0 = 0.173$

TRIAL SOLUBILITY RUN TA 03
SAMPLE: Ta WIRE .020 DIA.

NOTES
ATTEMPT TO FIND INFLECTION POINT BY ISO-CONCENTRATION TECHNIQUE
ELAPSED TIME: 180 MIN.

TEMPERATURE (°C) vs ELECTRICAL RESISTANCE (OHMS)

TRIAL SOLUBILITY RUN TA 03					
POINT	RES.(OHMS)	TEMP.(°C)	POINT	RES.(OHMS)	TEMP.(°C)
A	.2550	817	K	.2347	697
B	.2530	805	L	.2327	685
C	.2509	793	M	.2308	673
D	.2489	781	N	.2289	661
E	.2464	768	O	.2265	649
F	.2447	757	P	.2245	637
G	.2428	744	Q	.2224	626
H	.2408	733	R	.2203	614
I	.2388	721	S	.2165	590
J	.2367	709			

RUN TAO4   (Iso-Thermal Techniques)

Trial solubility run number TAO4 was programmed

to find an inflection point by the isothermal technique

at 900°C in order to confirm the results of Run TAO2.

Oxygen was admitted, in discrete charges, to the reaction

tube of the furnace where the specimen was maintained at a

constant temperature; resistance measurements were made

after each charge admittance. The series of charges

was interrupted by equipment failure so that the

resultant resistance vs oxygen charge plot was discon-

tinuous. However, data showed a definite inflection,

(at $\Delta R/R_O$ = 0.23) but was quantitatively inconclusive.

RUN TAO5   (Iso-Thermal Technique)

The fifth trial run, TAO5, had the specific

objective of determining the oxygen saturation point

of tantalum at 790°C in terms of the ratio $\Delta R/R_O$ by the

isothermal technique. Figure 11 shows resistance plotted

against number of charges admitted to the reaction tube.

The extrapolated inflection point occurs at a value of

$\Delta R/R_O$ = 0.205 at a specimen temperature of 780°C while

Gebhardt[2] reports a value of 0.20 at 900°C. (The

oxygen charge masses used, the time cycle, and apparatus

are considerably different from those employed in this

study as is the initial oxygen content of the specimen

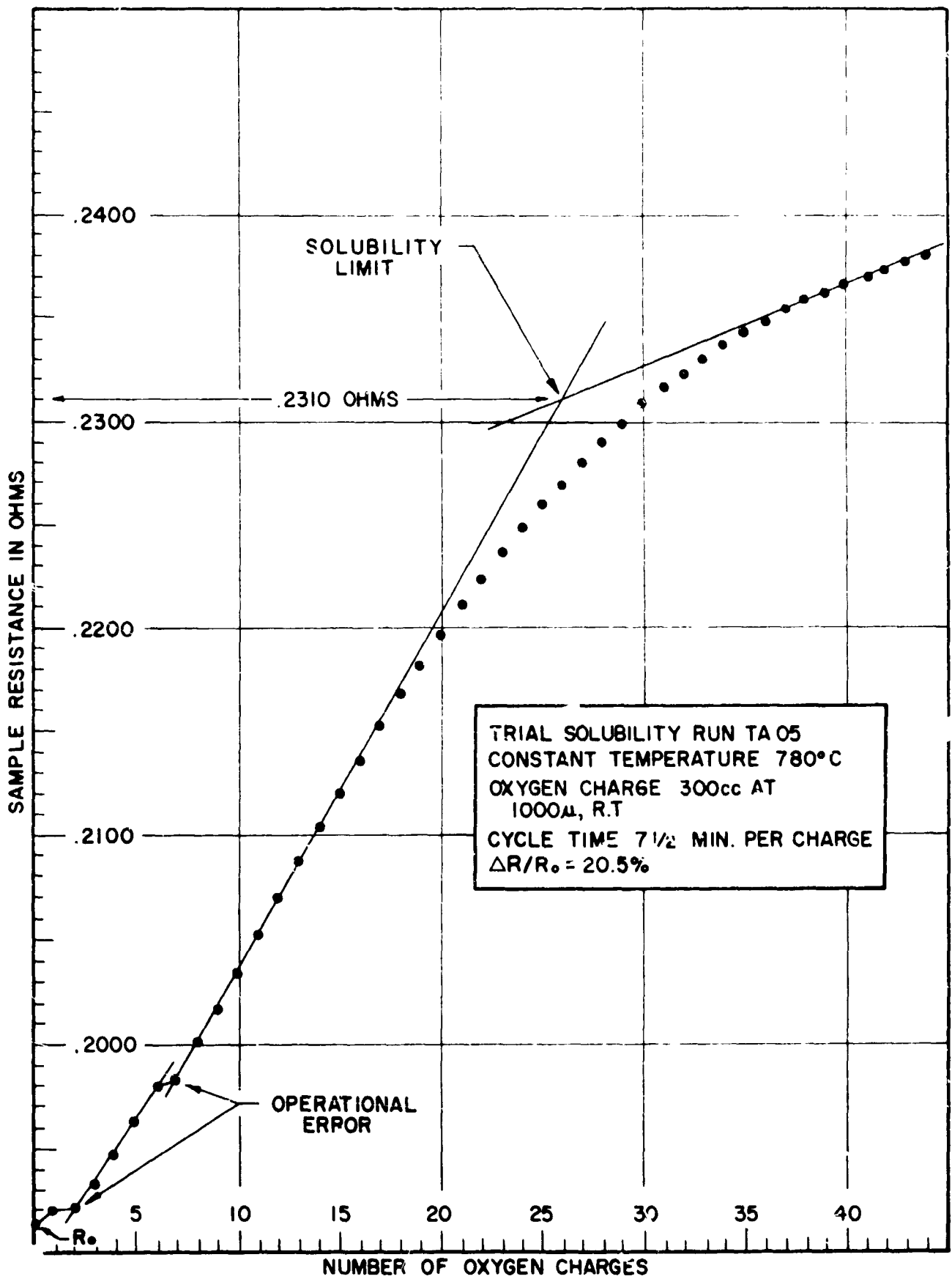wire.) The exact degree of agreement can be determined

FIG. II. ISOTHERMAL SOLUBILITY RUN TA 05

after the initial oxygen content of the commercial

purity wire being used has been determined and an

oxygen content vs $(\Delta R/R_o)$ calibration has been obtained

(See Section III B). This data is considered to be

quantitative and reliable.

VI.   DISCUSSION AND CONCLUSIONS

1.   The columbium runs, CbO1 CbO2. and CbO3 estab-
lished that the pressure of oxygen admittance had to be
below 10^{-4} torr in order to avoid the difficultly ab-
sorbed deep purple Cb_2O_3? oxide to form. The preferred
oxide to form is the grey CbO which is easily absorbed
No problems were encountered with the $Ta-O_2$ system. It
is not known whether the oxide absorbed is Ta_4O or TaO.
It is assumed that under the conditions of the experiment
that TaO is the oxide being precipitated in the iso-
thermal experiments.

2.   The iso-concentration technique (Runs TaO1, TaO3
and TaO4) produced no inflection points observable by
visual graphic techniques. However, statistical and
mathematical analysis of the data from these runs can
be considered if further iso-concentration runs do not
yield statistically valid graphically observable inflec-
tion points.

3.   The iso-thermal techniques yielded positive, though
qualitative, results in runs TaO2, TaO4, and quantitative
results in TaO5.

4.   The availability of two solubility determination
techniques may prove valuable in the study of complex
alloy systems. The utilization of the iso-concentration
technique is (1) dependent on significant precipitation

occuring under the conditions of a realistic experimental procedure and (2) dependent upon whether a suitable mathematical-statistical technique can be developed to determine the presence and co-ordinates of inflection points within acceptable limits of confidence.

5.   There are many critical factors relative to the two solubility determination techniques which should be considered in addition to the experimental results obtained above (2), (3), (4).

A.   Data produced by the iso-thermal technique is not adversely affected by non-linearily, of, or abrupt changes in, the temperature coefficient of resistivity of the alloys under study. In contrast, data from iso-concentration experiments will be very difficult to interpret under such circumstances.

B.   In order to carry out the iso-concentration procedure the approximate solubility curve must be previously known.

C.   The iso-concentration technique relies heavily on the assumption that the absolute oxygen content of specimens can be held in vacuo, at elevated temperature, for hours at a time (eg·volatile tungsten oxide which will be evolved during experiments on the alloys).

D.   Data produced by the iso-concentration technique

will be difficult to interpret in the case of the
alloys due to the appearance c. multiple inflection
points corresponding to the precipitation of the
several oxides obtained from each alloy or each con-
stituent of an alloy, (eg. oxides of Cb, Zr, Hf, Ta,
etc.) In view of this, it may be that the iso-
concentration _echnique pre-supposes a prior knowledge
of that which it seeks to determine.

E.   In the case of the iso-thermal method the
multiple inflection points still appear. (See D above)
However, the determination of the oxygen content
associated with the appearance of the first precipitate
constitutes fulfillment of the objective of the solu-
bility determination phase of this program so that an
individual experiment may be terminated after the
appearance of the first inflection point (or when an
oxygen level of 10,000 ppm is reached, whichever occurs
first).

F. The iso-concentration technique offers some
distinct advantages in cost and efficiency as it lends
itself to use of multiple specimens in a single experi-
mental run.

The use of multiple specimens assures identical
handling and history for all specimens in a given batch.
By contrast, the iso-thermal technique implies a separate

experimental run for each value of each of the two

variables, material and temperature.

G.   In the iso-concentration procedure the maximum

temperature reached will be 100-200°C above the

equilibrium, or frozen-in psuedo-equilibrium, that

would obtain during the course of an iso-thermal run.

Thus, the "solubility" data obtained will differ from

that obtained by the iso-thermal procedure (especially

at lower temperatures and in the case of the alloys) and

will not be easily relatable to the temperature condi-

tion that the alloy materials will see in service.

VII. PROGRAM FOR NEXT QUARTER

Solubility

1. Determine solubility limits of oxygen in
   Ta and Cb.

2. Place # 1500°C furnace in operation.

3. Obtain calibration plot of resistance versus
   oxygen content for Ta and Cb utilizing the
   neutron activation technique for analysis of
   oxygen content in calibration specimens.

Diffusion

1. Design Anodic Deposition apparatus.

2. Design resistance measurement fixture for
   diffusion specimens

3. Design diffusion furnaces, investigate RF.
   and D.C. electrical measurement techniques.

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