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CONTROLLED OXYGEN ADDITIONS TO REFRACTORY METALS

by Charles A. Barrett

Lewis Research Center

Cleveland, Ohio

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ABSTRACT

Controlled oxygen additions (doping) to refractory metals were carried out by a pressure-drop technique involving the use of an oxygen leak into a vacuum system at elevated temperatures. The method and apparatus are described, as well as the variability, precision, and accuracy of the method.

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SUMMARY

Refractory-metal alloys of columbium or tantalum can be conveniently doped to precise oxygen levels by exposure at elevated temperatures to oxygen at low partial pressures.

The specimens are heated under vacuum to elevated temperatures (1500° and 1700° F (1088 and 1200 K) in our studies) in a quartz or ceramic test section under a constant flux of oxygen in the molecular flow regime. The source of oxygen is through a mechanical leak valve between the specimen chamber and the vacuum pumping system. The rate of oxygen pickup can be monitored and controlled by measuring the pressure drop across a vacuum conductance leading into the test section.

Small samples of various shapes have been uniformly doped to levels of 100 to 5000 ppm oxygen added each, with the added levels within any group of samples doped together differing by less than 5 percent. In general, the runs take only 2 to 3 hours in the 10^{-4}-torr oxygen pressure range.

The pressures on either side of the conductance tube can be monitored continuously, and the pressure difference can be automatically converted to a digital readout which predicts the total weight gain of the specimen during a run with an error of just over 3 percent.

Chemical analysis for oxygen along the length of a specimen indicates no significant oxygen gradient, implying a uniform oxygen flux over the specimen.

INTRODUCTION

Controlled oxygen additions to refractory-metal specimens (commonly called "doping") are required in studying the effect of oxygen on various properties, such as alkali metal corrosion resistance and creep. Methods that have been used in the past include carrier gas techniques and volumetric oxidation techniques (refs. 1 and 2).

This report describes a readily controllable doping method that is an application of the recently developed pressure-drop technique for studying the low-pressure oxidation of metals (ref. 3). The general method and the apparatus are described, and the precision and variability of the results obtained by the method are evaluated.

The approach is to heat the specimens in a dynamic high-vacuum system and to allow the specimen to pump oxygen introduced through a controlled leak valve. All that is required, in principle, is to meter the gas being picked up by the specimen and to have enough control over the process so that reproducibility can be obtained. This can be done on a trial-and-error basis just by setting the leak so that a certain pressure is maintained above the baseline pressure and running for a certain period of time at a given temperature.

It is possible, however, to achieve better control by actually measuring the amount of gas being pumped within the specimen chamber by means of a pressure drop through a vacuum conductance. This technique is described in detail in reference 3. A schematic of the system is shown in figure 1. The oxygen leak valve is positioned between the vacuum pumps and the specimen chamber so that the flow is split between the two. The flow into the chamber is measured by the difference in pressure between the two ionization gages $P_0 - P_1$ multiplied by the molecular flow conductance between the two gages. This gives the volumetric flow rate in torr-liters per second. This value is converted to grams per hour by multiplying by $(M/T_0) \times 57.729$ derived from the ideal gas law:

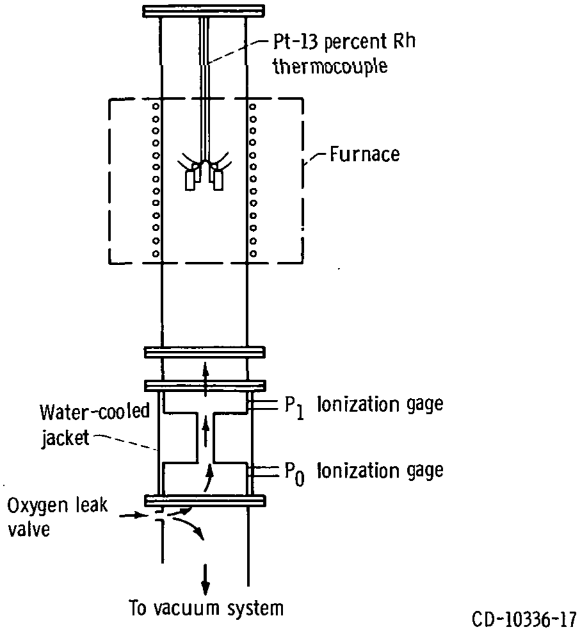

Figure 1. - Schematic of oxygen doping apparatus.

$$R = (P_0 - P_1) \times C_0 \times \frac{M}{T_0} \times 57.729 \qquad (1)$$

where R is the pickup rate in grams per hour, C_0 is the calibrated conductance in liters per second, M is the molecular weight of the doping gas (in theory any gas which is absorbed or otherwise reacts with the specimen to form a nonvolatile reaction product can be used), and T_0 is the temperature of the conductance tube in Kelvin. It then becomes merely a matter of sizing the conductance so that, for a given anticipated pumping rate by the specimen (or specimens) in the chamber, the pressure drop $(P_0 - P_1)$ value is large enough that sizeable errors in measuring it are not introduced. If the conductance is too low, however, it becomes extremely difficult to lower the test chamber pressure into the molecular flow regime without auxiliary pumping. For a cylindrical tube between the two gages the conductance is (ref. 4)

$$C_0 = 3.638 W \pi r^2 \left(\frac{T_0}{M}\right)^{1/2} \qquad (2)$$

where W is the transmission probability for molecular flow for a cylindrical conductance tube, r is the radius of the conductance tube in centimeters, and T_0 is the mean temperature of the conductance tube. The transmission probabilities for cylindrical tubes are readily available (ref. 4). Thus, if the gages are once calibrated in oxygen and remain drift free, the only source of variation is the temperature of the conductance tube. This temperature can be held invariant by cooling the conductance tube.

This report describes an apparatus that was built at the Lewis Research Center according to this principle and discusses its use for doping columbium and tantalum alloys with controlled amounts of oxygen. The appendix by Vernon Klinect describes the oxidation weight gain computer.

APPARATUS AND PROCEDURE

The oxygen doping apparatus is shown in figure 2. The specimen chamber is a quartz glass tube 7.5 centimeters in internal diameter and 100 centimeters long. It is heated externally by a nickel-chromium alloy wound resistance furnace. The quartz tube required quartz-to-borosilicate graded transition sections to join the borosilicate-to-metal transition sections attached to the vacuum flanges. A 7.62-centimeter-diameter nonpermeable ceramic tube has also been used in this setup with top and bottom compression-fitted fluorocarbon rubber O-rings enclosed in water-cooled stainless-steel flanges. The bottom metal chamber section is joined to the pumping system by a stainless-steel conductance tube (6.39 cm by 1.40 cm i.d.) which is enclosed in a water-cooled jacket. Just above and below this conductance is each ionization gage station. Upstream of the

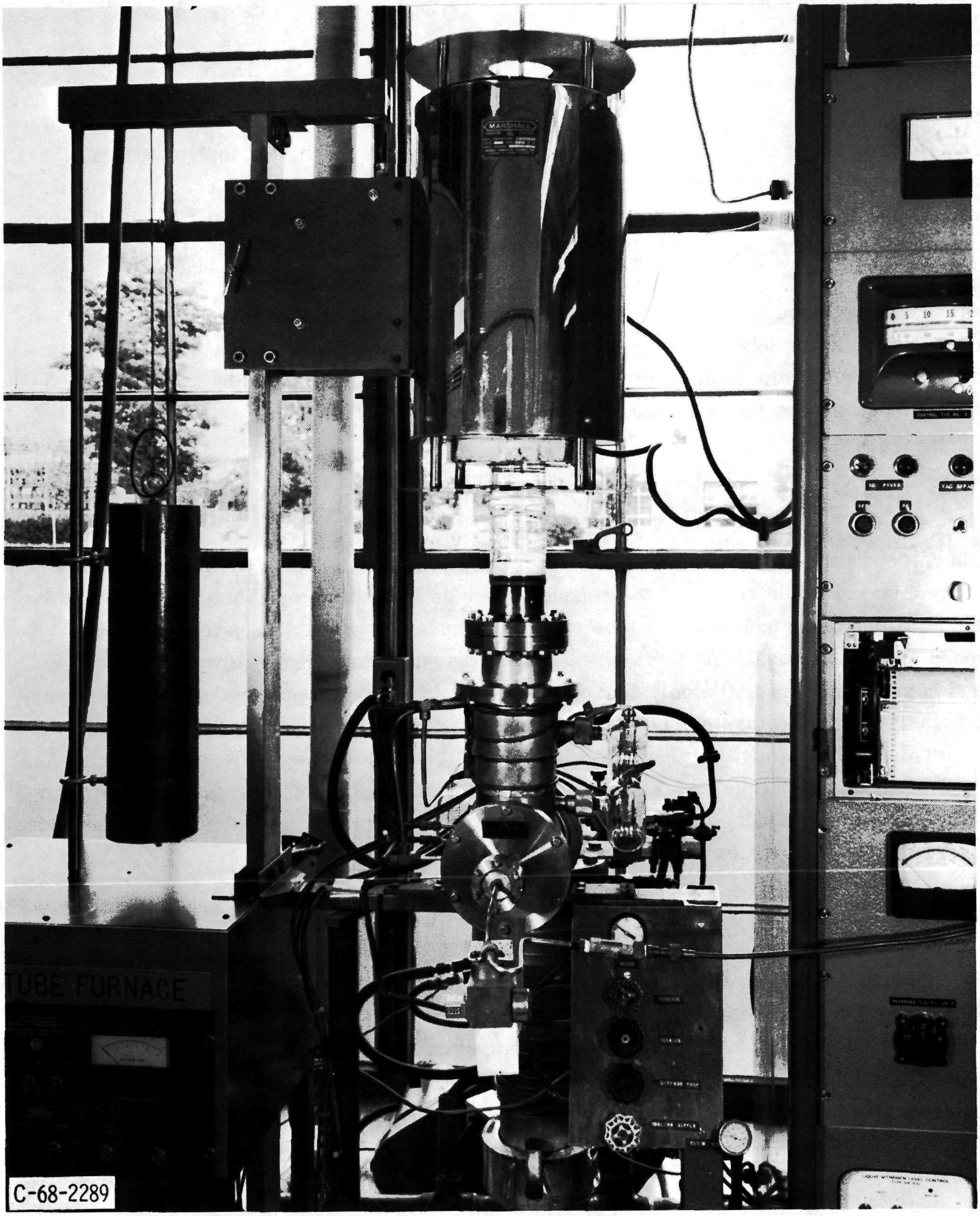

Figure 2. - Oxygen doping apparatus.

P_0 ionization gage is a manually adjustable variable oxygen leak valve, leading to a tank of high-purity oxygen. The vacuum system upstream of the oxygen leak consists of a mechanical pump backing a 6-inch (15.24-cm) oil diffusion pump with a water-cooled baffle. Because of the reactivity of oxygen, nonburnout-type ionization gages with thoriated iridium filaments were used.

In using this pressure-drop technique, it is critical that the only sources of oxygen pumping upstream of the conductance tube are the specimens themselves. For this reason, only ceramic and/or quartz and platinum and its alloys were used in the hot furnace zone for specimen support and thermocouples. A third ionization gage was mounted at the top of the test apparatus to detect any pressure difference across the specimens. In general, this top gage read nearly the same as the P_1 pressure.

The specimens to be doped are loaded through the top flange and supported by a platinum holder joined to platinum rods attached to the top flange. The requirements of the specimens are that for any given run they be the same alloy (or alloys with the same sticking probability for oxygen, ref. 3), have nearly the same dimensions, hang to the same level in the furnace, and be supported as symmetrically as possible with the long center axis of the furnace. When these requirements have been met, the oxygen flux will be equal for all the specimens, ensuring equal pickup for each specimen. To date, sheet samples, cups, springs, wires, tubes, and capsules have been successfully doped (ref. 5). The longest sample was 12.70 centimeters long, and most diameters or sheet widths have been 1.27 centimeters. The samples are individually weighed prior to doping and after doping to cross-check the weight gain estimates made from the pressure drop. (Blank specimens run with no oxygen leak at the test temperatures (1500° or 1700° F) in the doping apparatus in the 10^{-6}-torr range for several hours showed no significant weight change.)

The specimen pumping rates are determined by the total specimen area in the chamber as well as the inherent sticking probability of the alloy. The greater the total specimen area in the apparatus, the greater the precision in the doping levels for a given alloy. This relation is due to the greater pressure drop, $P_0 - P_1$. Likewise, large weight changes (i.e., high doping levels) are more precise than small weight changes (i.e., low doping levels) because of the sensitivity in weighing the samples.

All runs with columbium and tantalum alloys were performed at 1500° or 1700° F (1088 or 1200 K). These temperatures were high enough for these materials so that oxygen diffusion away from the specimen surface was rapid enough to keep the specimen clean (i.e., low oxygen surface coverage). Higher temperatures tend to raise the permeability of the ceramic tube, cut down heater life, and raise the background pressure. If oxide surface films are desired instead of dissolved oxygen, the furnace temperature is lowered.

The specimens to be doped are first measured and weighed and then loaded into the platinum or quartz holder. The holders are designed to contact as little specimen area as possible while maintaining the samples at the same height in the furnace and symmetrical with the center axis. Once loaded, the system is sealed and evacuated. Typically, the system pumps down in a few hours until the P_0 gage reads in the low 10^{-7}-torr range and the P_1 gage above the conductance is in the 10^{-6}-torr range. The furnace is then heated to temperature, which takes a few hours. During heating, the gage pressures rise over the heating cycle on the order of a decade as a result of outgassing of the specimen and chamber walls at the lower temperatures and hydrogen outgassing of the Ta and Cb alloys above 921 K. The specimens are then held at temperature, usually for about 1 hour, until the pressures level out. Typically, the P_0 pressure is now in the mid 10^{-7}-torr range, while P_1 is of the order of 1×10^{-5} to 2×10^{-5} torr. Then, the oxygen leak is opened until P_0 is approximately 6×10^{-4} torr. The P_1 gage is typically 1×10^{-4} torr but is dependent on the type of specimens and their total area. This pressure differential is established within seconds of opening the leak to the setting where the desired P_0 level is reached. After a few minutes of initial instability, the pressures level out so the ΔP is nearly constant.

The ΔP value can be converted to weight gain and then plotted against time to estimate the total weight gain of the specimens. For greater convenience and precision in following the weight gain, it was decided to devise a weight-gain integrating device based on use of the expression

$$W = C \int (P_0 - P_1)dt \tag{3}$$

where W is the accumulated weight gain in the test chamber, P_0 and P_1 are the two gage pressures in torr, C is the constant for any test temperature and reacting gas for the conductance and its conversion factor to grams. Since P_0 and P_1 values can be read as voltages (e_0 and e_1), an instrument to give a time integral of $e_0 - e_1$ was devised. This instrument is described in detail in the appendix. This voltage difference is fed into a voltage-to-frequency converter which produces a series of pulses, the frequency of which is proportional to the input voltage. Thus, the frequency of the output pulses is proportional to the pressure difference (i.e., weight), and the total counts read out on a digital counter are directly proportional to the total weight pickup.

A typical run for pure tantalum of adding 0.012 gram of oxygen for a total specimen area of 80 square centimeters takes a little over 1 hour. The leak is then closed, and the furnace cooled. The turnaround time is approximately 24 hours. The actual volume of oxygen gas used in such a run is so small that well over 100 runs can be gotten from a standard 79 286-liter- (2800-cu-ft-) capacity bottle.

RESULTS AND DISCUSSION

After a group of specimens are doped, they are removed and individually weighed. These individual weights are totaled to give the total weight gain. In the range of 0.010 to 0.050 gram, the total weight gain can be estimated from the counts to less than 0.0005 gram (i.e., 1 to 5 percent).

Figure 3 is a log-log plot of weight pickup against counts for the complete set of samples for various runs (i.e., doping runs from the same set of ionization gages). This plot is for specimens doped at both 1500° and 1700° F (1088 and 1200 K) for times as long as about 3 hours. The straight line is the regression fit which estimates the weight gain from the counts with a standard deviation of 3.31 percent. This shows how relatively insensitive to furnace temperature the estimates were as long as the conductance tube was cooled to the same temperature (T_0).

Table I shows some typical results from doping runs for tantalum samples of various shapes and sizes. These data give some indication of the within-run variability. Includ-

TABLE I. - TYPICAL OXYGEN DOPING RUNS

(a) Run 193. Three tantalum sheet specimens, 1.27 by 0.069 by 5.08 centimeters; total weight gain, 0.01202 gram; gain estimated from counter, 0.01252 gram

Original weight, g	Added weight, g	Oxygen added, ppm
8.35986	0.00401	479.7
8.42945	.00403	478.1
8.36970	.00398	475.5

(b) Run 203. Three tantalum cups: diameter, 1.27 centimeters; height, 2.54 centimeters; wall thickness, 0.127 centimeter; total weight gain, 0.01490 gram; gain estimated from counter, 0.01410 gram

Original weight, g	Added weight, g	Oxygen added, ppm
21.52486	0.00505	234.6
21.27987	.00493	231.7
21.21289	.00492	231.9

(c) Run 220 (see fig. 3). Six tantalum wire coils: length, 20.96 centimeters; wire diameter, 0.051 centimeter; turns, approximately 5; total weight gain, 0.01081 gram; gain estimated from counter, 0.01070 gram

Original weight, g	Added weight, g	Oxygen added, ppm
0.72741	0.00188	2585
.72452	.00174	2402
.72286	.00186	2573
.71976	.00180	2501
.72441	.00170	2347
.71991	.00183	2542

ed are the number of samples for each run and the original weight for each sample as well as the weight pickup and the ppm oxygen pickup when this weight is converted to oxygen. The weight of individual samples varied from 7 to over 21 grams with nearly uniform weight changes in the milligram range for each sample in a run. The runs covered pickups of nearly 100 ppm per sample to over 5000 ppm per sample, with the degree of variability of the samples within a given run well under 5 percent.

Two sets of four specimens of S-291 alloy (Cb-10Ta-10W) were run especially to check out all phases of variability. In particular, it was desired to know

(1) Was there an oxygen gradient from the top to the bottom of the specimen?

(2) If so, could it be minimized by some method such as heat treatment?

(3) Could sets of identical specimens be repeatedly doped to the same desired oxygen level in successive runs?

(4) Would comparing the oxygen uptake estimated by weight gain against that derived from chemical analyses give some indication of the accuracy of the method?

Eight samples were prepared, all 1/2 inch by 2 inches by 0.030 inch (1.27 cm by 5.08 cm by 0.076 cm) with an initial oxygen content of 80 ppm. Samples 1 to 4 were run to a count level of 6000 to give an estimated uptake of at least 2000 ppm per sample. Samples 4 to 8 were run next at nearly the same conditions to the same number of counts. Samples 1 to 4 were then rerun identically as before to the same count level with the intent to double the oxygen uptake. Samples 4 to 8 were then rerun to double their uptake, but this time the samples were inverted. This was done in an attempt to balance any oxygen flux gradient that might be present along the specimen. Then two samples from each set (samples 1, 2, 5, and 6) were vacuum homogenized ($<10^{-7}$ torr) for 1 hour at 2200° F (1477 K). The samples were then sectioned into 1/4-inch- (0.635-cm-) long segments along the length of the specimen starting with the initial top position. The segments were then analyzed for oxygen by vacuum fusion.

The data are presented in factorial form in table II. The four successive runs doped to the same count level gave weight gains of 0.04774, 0.04442, 0.04398, and 0.04859 gram, respectively, a range of about 5 percent.

These values are also plotted in figure 3 but are displaced from the curve since they represent data for a different set of ionization gages at P_0 and P_1. This difference is due to the slight differences in oxygen sensitivities for each gage. The curve must be recalibrated for each set of gages used although it will still be linear and nearly parallel to the established curve.

The overall average for all the data is 4730 ppm of oxygen with a standard deviation of 157 ppm. This gives a coefficient of variation of just over 3.3 percent. There appeared to be no significant difference between samples that were not reversed (average of 4775) and those that were (average of 4684). Likewise, heat treatment did not appear to be significant. Sample averages also did not differ appreciably. The samples were

TABLE II. - SPECIAL OXYGEN DOPING RUNS TO DETECT OXYGEN GRADIENT EFFECTS - VALUES IN PPM OXYGEN

Segment	Sample not reversed[a]				Sample reversed[b]				Segment average
	Heat-treated[c]		Not-heat-treated[d]		Heat-treated[c]		Not-heat-treated[d]		
	Sample								
	1	2	3	4	5	6	7	8	
1	4770	4920	5070	4570	4820	4960	4850	5090	4881
2	4560	4650	4820	4570	4490	4740	4690	4880	4675
3	4550	4700	4870	4660	4620	4710	4670	4750	4691
4	4620	4710	4810	4730	4460	4700	4660	4680	4671
5	4630	4760	4790	4730	4480	4600	4650	4620	4658
6	4720	4750	4830	4760	4480	4520	4620	4510	4649
7	4780	4770	4810	4740	4490	4560	4640	4580	4671
8	5100	4950	5120	4990	4860	4840	4840	4830	4941
Sample average	4716	4776	4890	4719	4588	4704	4702	4742	----
Oxygen pickup, g:									
Gravimetric	0.02284	0.02299	0.02352	0.02346	0.02227	0.02329	0.02321	0.02295	----
Chemistry	.02196	.02187	.02295	.02202	.02139	.02209	.02195	.02224	----

[a] Average, 4775.
[b] Average, 4684.
[c] Average, 4696.
[d] Average, 4763.

actually analyzed in eight separate vacuum fusion runs (one for each sample) over a period of several weeks so that analytical run-to-run variations could be confounded with sample differences. The only real effect seemed to be caused by end effects on the samples (segments 1 and 8) which were consistently higher than the intermediate segments.

These end values were, on the average, 5.2 percent higher than those values from the inner segments. The available area on the end segments was about 5.7 percent greater than the inner segments, however. This would seem to explain the difference since surface flux is thought to be rate controlling (ref. 3). If these values (segments 1 and 8) are dropped, the mean of all the remaining values becomes 4670 with a standard deviation of 109, which drops the coefficient of variation to just over 2.3 percent.

The accuracy of the method can be inferred by comparing the estimated oxygen pickup in grams both by weighing the samples and by chemical analyses. These values agree to about 5 percent, although the values derived from the chemical analyses are consistently lower.

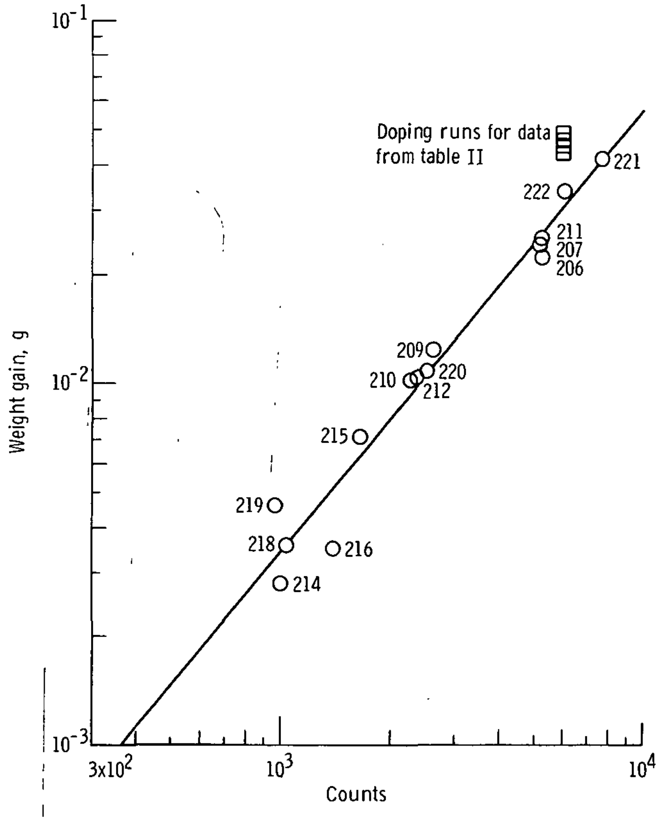

Figure 3. - Typical calibration run for given set of P_0 and P_1 ionization gages. Each data point represents a doping run (runs 206 to 222).

In general, the overall variability of the doping process is low with good reproducibility, precision, and accuracy. The associated overall error is less than the 10 percent expected from running straight oxygen vacuum fusion analyses on identical specimens.

SUMMARY OF RESULTS

Controlled oxygen additions to refractory metals or alloys can be conveniently made by exposure at elevated temperatures to oxygen at low partial pressures. A method used successfully at this center is summarized below.

The specimens are heated under vacuum to high temperatures (1500° and 1700° F (1088 and 1200 K) in our study) in a quartz or ceramic test section under a constant flux of oxygen in the molecular flow regime. The source of oxygen is a mechanical leak between the specimen chamber and the vacuum pumping system. The rate of oxygen pickup is monitored and controlled by measuring the pressure drop across a vacuum conductance leading into the test section.

Small samples of various shapes have been uniformly doped at uptake levels of 100 to 5000 ppm of oxygen each, with uptake levels within any group of samples doped together differing by less than 5 percent. In general, the runs take only 2 to 3 hours in the 10^{-4}-torr oxygen pressure range with less than 1 percent of the volume of a standard 79 286-liter- (2800-cu-ft-) capacity oxygen bottle consumed.

If the pressures on either side of the conductance tube are monitored continuously, the pressure difference can be automatically converted to a digital readout which predicts the total weight gain of a specimen during a run with an error of just over 3 percent.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, March 3, 1969,
129-03-03-01-22.

APPENDIX - WEIGHT GAIN COMPUTER

by Vernon Klinect

The weight gain of a test sample as a result of oxidation is proportional to the pressure drop by the following relation

$$W = C \int (P_0 - P_1)dt \tag{3}$$

where W is the weight gain in grams and C is a function of the conductance, temperature, and molecular weight of the reactant gas. It is assumed that C is constant for any test run, although it can vary for different tests and test systems.

A block diagram of the system for electrically computing weight gain is shown in figure 4. The upstream and downstream pressures across a known conductance (P_0 and P_1, respectively) are measured by ionization gages. The output voltage signals from the ionization gage control units are proportional to the pressures by the factors K_0 and K_1, which are the transfer functions of the ionization gage control units. Dimensionally, these transfer functions are in volts per torr. For example, in the 1×10^{-4}-torr range, the full-scale output is 2 volts so the transfer function would be 2×10^3 volts per torr.

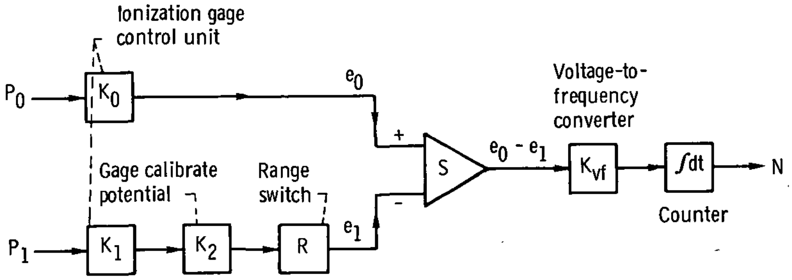

Figure 4. - Block diagram of system for electrically computing weight gain.

The downstream signal is modified by the factor K_2 so that the voltage signal e_1 is equal to P_1K_0. This is necessary to compensate for differences in ionization gage sensitivity. It is also necessary to provide the ability to scale down the P_1 signal when the downstream ionization gage control unit is operated on a lower range than the upstream one. This is done by the range switch R which provides a gain of 1 or 1/10, depending on whether the same or different ranges are used. The downstream signal is then subtracted from the upstream signal providing a voltage proportional to the pressure drop across the conductance.

The voltage-to-frequency converter produces a train of pulses, the frequency of which is proportional to the input voltage. The counter then records a number of pulses proportional to the time integral of the pressure difference.

$$N = \int f\,dt = K_{vf} \int (e_0 - e_1)dt = K_{vf}K_0 \int (P_0 - P_1)dt \tag{4}$$

By combining equations (3) and (4), we get

$$W = N \frac{C}{K_0 K_{vf}} \tag{5}$$

$$\frac{W}{N} = \frac{C}{K_0 K_{vf}} \tag{5a}$$

Since C and K_0 are determined by the test system and its instrumentation, adjustment of K_{vf} so that W/N becomes an integral power of 10 makes N a direct readout of the weight gain. The instrument has been designed so that for the expected range of C and K_0, W/N can be made equal to 1×10^{-6}. Then N is a direct readout of weight gain in micrograms.

The voltage-to-frequency converter is simply an operational amplifier used as an integrator with a trigger circuit which applies a discharge pulse to the summing junction when the integrator output reaches a predetermined value. By varying the width of the discharge pulse, the pulse rate per volt input, or K_{vf}, can be determined. For each discharge pulse, an output pulse is sent to the counter. Since each discharge pulse is of known voltage and duration, it is equivalent to the number of volt seconds integrated, and the number of these pulses is then equivalent to the integral of the input voltage.

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