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AUTHORITY

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(6) RESEARCH TO DETERMINE THE EFFECTS OF SURFACE CATALYTICITY ON MATERIALS BEHAVIOR IN DISSOCIATED GAS STREAMS.

(9) QUARTERLY PROGRESS REPORT No. 1, 1 Jun-15 Sep 66,

(10) Joan B. Berkowitz

Contract No. AF 33(615)-3922
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(11) 15 Sep 66

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September 15, 1966

(14) C-68410

Arthur D. Little, Inc.

Introduction and Summary

On 1 June, 1966, Arthur D. Little, Inc., initiated work on a program to determine the rates of reactions of various aerospace vehicle leading edge and nose cap materials with dissociated oxygen over a range of temperatures and pressures of 300-1500°K and 10^{-4} -10 torr. The materials to be investigated are ThO_2, Mo, Ta, Ti, and $MoSi_2$. The primary purpose of the program is to evaluate the effect of atom-surface interaction on heat transfer and surface degradation.

Atom recombination rates will be determined in a fast flow system using NO_2 titration and air afterglow to measure changes in oxygen atom concentration due to reactions with the sample under study. It is important to note that changes in the surface of the sample by chemical reaction with oxygen may result in changes in measured recombination coefficient. Identification of the actual surface whose catalytic efficiency for recombination is being measured is therefore of major significance. For ThO_2 recombination should be the only important reaction occurring in the temperature range of interest. For Mo, oxidation, with the formation of both solid and volatile oxides, will be competitive with recombination, and we will want to know whether our recombination results are characteristic of bulk metal, bulk oxide, or oxide modified by juxtaposition with the metal. For tantalum and titanium an additional factor that may influence the results is the high solubility of oxygen in the metal lattice.

During the current report period, 1 June, 1966--15 September, 1966, we have completed construction of the fast flow apparatus, and have put both the oxygen atom generation system and the NO_2 titration system into operation. We did our first NO_2 titration, using a visual end point, the morning of 15 September.

In the next quarter we plan to calibrate the NO_2 flowmeter by adsorption in Indacarb and to verify the known recombination coefficient for quartz. Before the end of the quarter we hope to have made recombination measurements at 300°K for ThO_2 and all of the metals and to have started work at higher temperatures.

Experimental

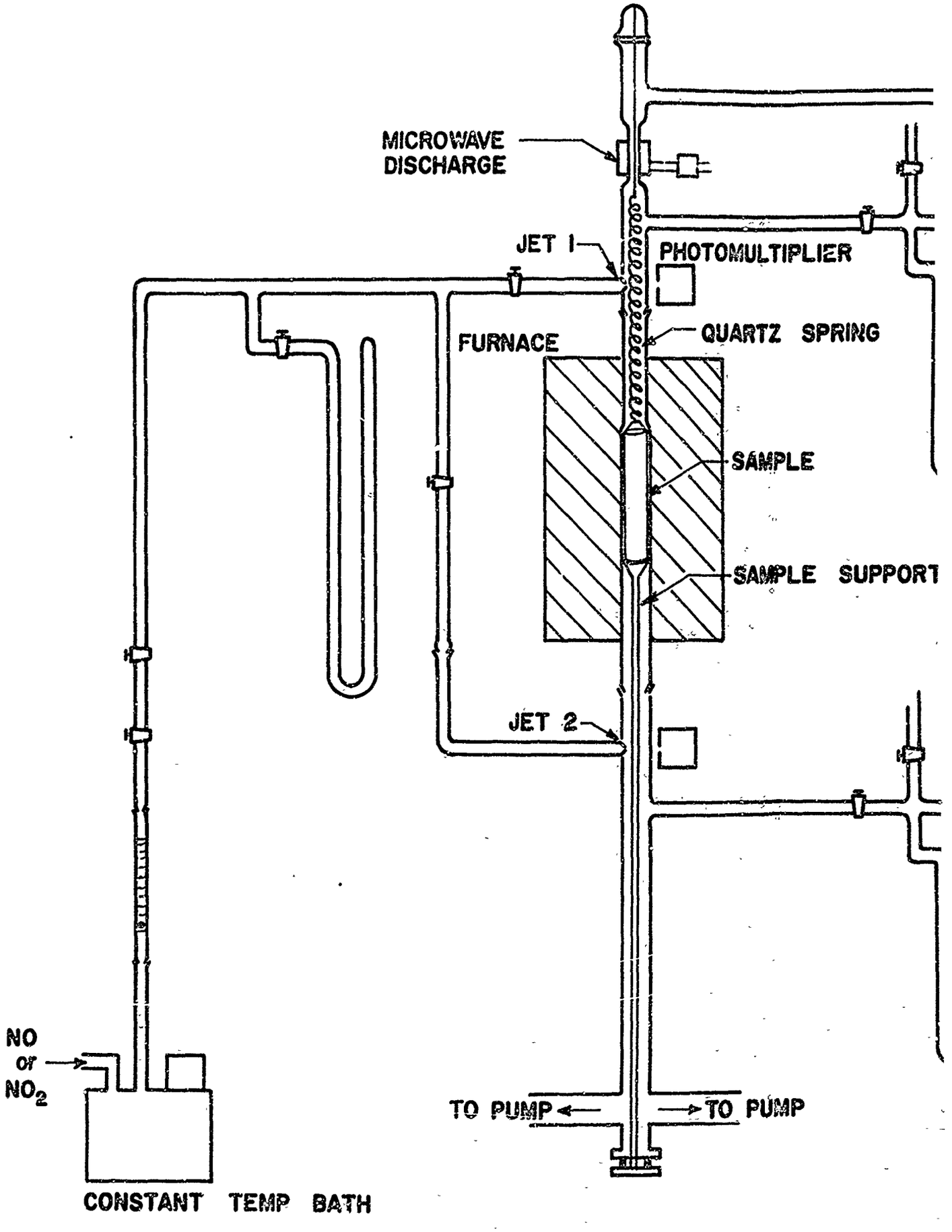
A schematic diagram of the apparatus is shown in Figure 1. Construction has been completed except for the installation of the tube furnace. The oxygen and argon flowmeters have been calibrated. The fast flow system is operative, the oxygen and argon flowmeters have been calibrated with a wet test meter. The microwave discharge has been turned on and the presence of oxygen atoms has been demonstrated by the green afterglow produced upon introduction of NO_2. The intensity of the glow could be varied from zero to a maximum by adjustment of the NO_2 flow rate.

In Figure 1 oxygen and argon are separately metered through sapphire ball-type flowmeters into a common line. Argon flow rates can be varied from 200-12,000 cc/min, and oxygen flow rates can be adjusted between 1-260, 10-1,900, and 200-12,000 cc/min by appropriate choice of flow line. A Wallace-Tiernan guage is used to measure line pressure. An oxygen-argon mixture of desired composition is passed through a Raytheon 2450-Mc/sec powered Ophthos 125-w Evenson microwave discharge cavity where oxygen atoms are generated. Dowstream of the discharge tube there is a jet for introduction of NO_2(g) or NO(g) and a photomultiplier tube. For calibration purposes absolute atom concentrations in the gas stream under steady state conditions will be determined by the method of NO_2 titration. During the atom recombination experiments, relative atom concentrations will be monitored along the flow system by the afterglow intensity observed upon introduction of known very small amounts of NO(g) into the gas stream.

Tubular samples will be supported from a balance within the middle third of the heated zone. Clearance of approximately 1 mm will be provided between the outer wall of the sample and the inner wall of the quartz housing. For the preliminary runs the sample will be held rigidly in place by means of the quartz plunger during the measurement of recombination rates. The gas stream will flow only over the inner sample wall. The quartz plunger will be released periodically so that the sample hangs freely from a quartz spring balance for intermittent weighing. Later in the program a microbalance will be incorporated into the system for continuous weight change measurements on those systems for which oxidation is competitive with atom recombination.

At the exit of the sample chamber, a second jet and photomultiplier tube are located for measuring the atomic oxygen in the exit stream, again by NO_2 titration or nitric oxide afterglow. The ratio of the afterglow intensities at the two photomultiplier positions, with appropriate corrections, can be related to an effective oxygen recombination coefficient for the system under study.

The flow system is coupled to a 25ℓ/sec mechanical pump. The static pressure can be measured up and dowstream of the sample by means of two Meriam fluid (ρ=1.04) differential manometers. The NO_2(g) is introduced into the system from a constant temperature reservoir. The rate of flow is monitored by a ball-type flowmeter, and will be calibrated by quantitative adsorption on Indacarb. The NO(g) can be introduced through the same line. A mercury manometer can be used to measure pressure in the NO-NO_2 line.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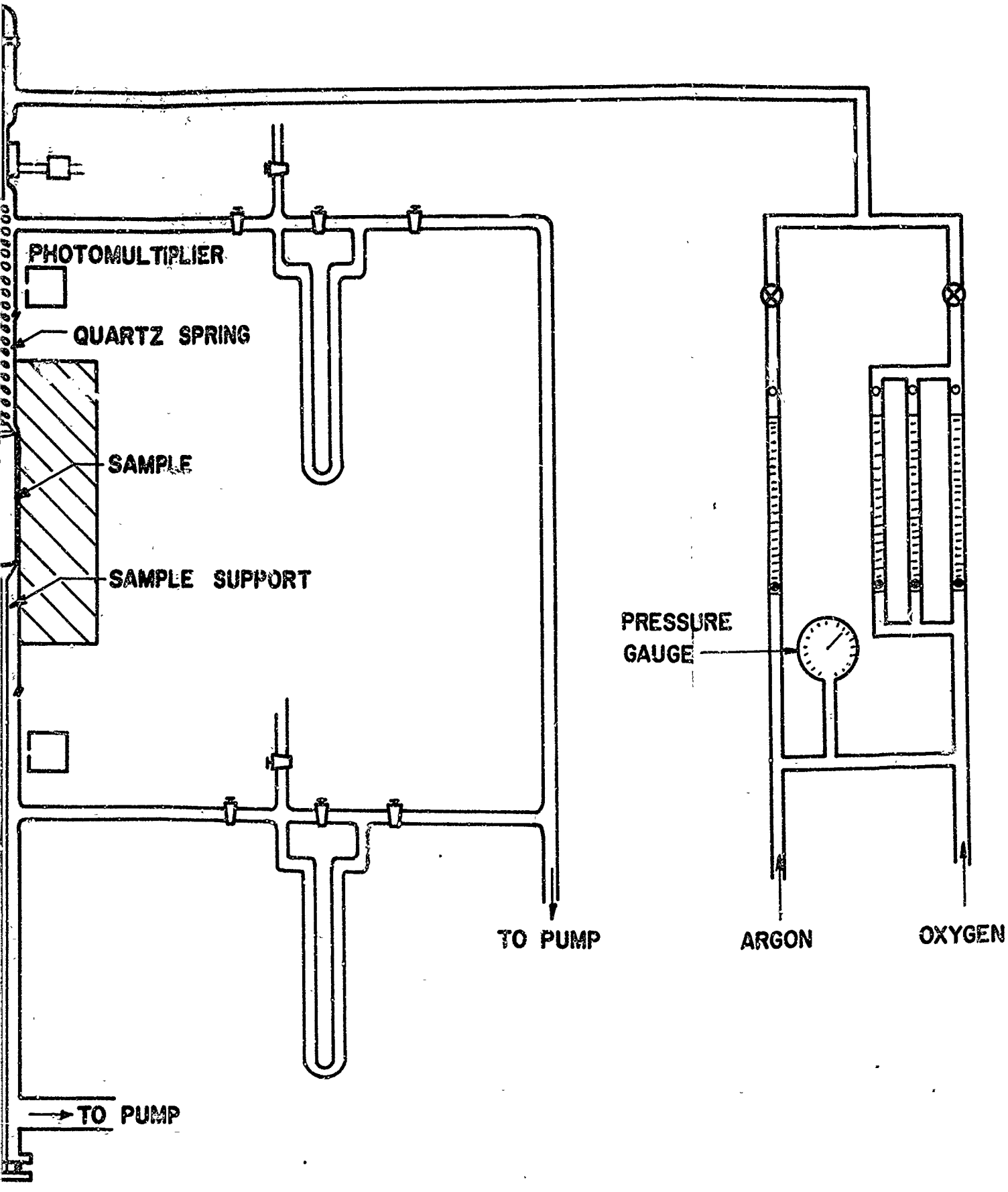
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2b. GROUP

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11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY

13. ABSTRACT

A fast flow apparatus for measurement of oxygen atom recombination rates in which surfaces at temperatures of 300-1500°K and pressures of 10^{-4} - 10 torr has been constructed. Oxygen atom concentrations are measured by means of NO_2 titration and air afterglow.

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14. KEY WORDS	LINK A		LINK B		LINK C	
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