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RESEARCH MEMORANDUM

INVESTIGATION OF TANTALUM IN A 3,800° F
SUPERSONIC AIRSTREAM

By Otto F. Trout, Jr., and Jerry L. Modisette

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Langley Field, Va.

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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

WASHINGTON

January 9, 1958

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RESEARCH MEMORANDUM

INVESTIGATION OF TANTALUM IN A 3,800° F
SUPERSONIC AIRSTREAM

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SUMMARY

An investigation of tantalum was made in still air and in high-velocity air to determine its resistance to oxidation at high temperatures. Experiments show that, except for a narrow range of temperature, unprotected tantalum is not suitable for airframe parts exposed to air at high stagnation temperatures, because above this temperature range tantalum oxidizes or burns rapidly.

INTRODUCTION

As part of an investigation by the National Advisory Committee for Aeronautics to determine the ability of materials to withstand aerodynamic heating at hypersonic speeds, tests have been made of tantalum. The excellent resistance of tantalum to oxidation at low temperatures (refs. 1 and 2) and its high melting point make it of interest.

The present tests were made to determine the effect of oxidation on tantalum in a high-stagnation-temperature, high-velocity airstream. Oxidation tests were also conducted in still air to obtain quantitative measurements of the average oxidation.

TESTS AND METHODS

Hot-Air-Jet Tests

Models made of tantalum, shown in figures 1 and 2, were tested in the ceramic-heated jet (laboratory model) at a Mach number of 1.96 in an airstream having a stagnation temperature of 3,800° F and a stagnation pressure of 110 pounds per square inch absolute. A description of the testing facility is provided in reference 3. The models, initially

at room temperature, were placed in the high-temperature free-air jet and were heated until they burned or were ablated. Test results were recorded by means of a high-speed motion-picture camera. Temperatures of the jet were determined by optical pyrometer measurements of the pebble-bed temperature.

Still-Air Tests

Commercially pure tantalum wires of 1/16-inch diameter were tested in the apparatus shown in figure 3 at temperatures of 2,800° F, 3,300° F, and 3,500° F. At each temperature both clean wires and wires covered with a 0.05-inch-thick layer of oxide were tested. Temperatures were measured with an optical pyrometer, and the tests were timed with a stopwatch. The specimens were weighed before and after the tests on an analytical balance. At 2,800° F and 3,300° F, the time required for the specimens to be heated to the test temperature was approximately 5 percent of the total test time. At 3,500° F, the wires were completely oxidized in less time than was required for heating, and no oxidation rates are given.

RESULTS AND DISCUSSION

Hot-Air-Jet Tests

The models presented in figure 1 were tested in the $M = 1.96$ ceramic-heated jet (laboratory model) at a stagnation temperature of 3,800° F. The sharp-pointed model ignited almost immediately upon entering the hot airstream and was completely ablated in 6.9 seconds. Photographs of this test are presented in figure 2(a). During the tests on the rounded specimen (fig. 2(b)), the model remained in the airstream 3.4 seconds before ignition took place and was completely ablated in 8.4 seconds. Tests were also made on a 1/2-inch-diameter, solid, hemispherical, tantalum model (fig. 2(c)). In this case a layer of oxide formed on the face after about 5 seconds and ignition took place after 15 seconds in the jet. Tests on a 1/2-inch-diameter, flat-faced, solid, tantalum model (fig. 2(d)) produced a thick oxide film. This film along with a lower heat-transfer rate to the flat-faced model prevented ignition of the metal. Tests on tantalum in a high-temperature high-velocity airstream indicated that it is not suitable for use in high-temperature airstreams unless a protective coating or plating can be developed which will prevent oxidation, even though tantalum has a very high melting point. Tests on the flat-faced tantalum model indicate that the thick oxide film formed on the flat-faced model offers some protection against oxidation due to high heating rates.

Still-Air Tests

The references disagree as to the oxides present at the test temperatures. According to reference 1, tantalum pentoxide Ta_2O_5 is the only stable oxide. Reference 2 states that Ta_2O_5 decomposes, and it lists Ta_2O_4 as a lower oxide which oxidizes in air but does not give the temperatures of decomposition or oxidation. Another reference (ref. 4) says Ta_2O_5 melts at $3,400^\circ F$.

The oxide formed in the tests at $2,800^\circ F$ was observed to be white and very porous, and fitted the descriptions of Ta_2O_5 given in reference 5. At $3,300^\circ F$ the oxide was molten and very viscous, and at $3,500^\circ F$ the oxide was very fluid. On cooling, the oxides formed at $3,300^\circ F$ and $3,500^\circ F$ became a vitreous solid, which on being exposed to air at room temperature for several days changed to a white oxide, apparently the same as that formed at $2,800^\circ F$. This change was accompanied by a gain in weight. However, this weight gain did not exactly match the stoichiometric weight gain that would accompany the oxidation of Ta_2O_4 to Ta_2O_5 . The weighings seem to indicate that the oxide was a mixture of Ta_2O_4 and Ta_2O_5 . This would indicate that Ta_2O_4 oxidizes rapidly on first cooling, and then more slowly to completion. The time required for heating the specimens to test temperatures was about 5 percent of the total test time, and the time for cooling was slightly longer.

Average oxidation rates given in table I were calculated from the differences in weights at the beginning and end of the tests and from the average surface area of the wires. The mechanism of the reaction was not investigated. The oxide formed at $2,800^\circ F$ and was assumed to be Ta_2O_5 , and at $3,300^\circ F$ it was assumed to be Ta_2O_4 .

The heat inputs were calculated from heats of reaction determined from heats of formation and heat capacities given in references 2, 6, and 7.

Table I shows that the oxide coating provides no protection at $2,800^\circ F$, but retards the oxidation at $3,300^\circ F$. At $3,500^\circ F$, the heat input due to oxidation was greater than the heat losses due to radiation and conduction, and the test specimens immediately rose to their melting point of $5,100^\circ F$ and burned vigorously. The reaction was too rapid to permit any measurement at this temperature.

CONCLUSIONS

From an investigation made in still air and in high-velocity air to determine the resistance of tantalum to oxidation at high temperatures, the following conclusions are presented:

1. Tests of tantalum in a supersonic air jet having a stagnation temperature of $3,800^{\circ}$ F indicate that unprotected tantalum burns rapidly.
2. Tests of tantalum wires heated electrically in still air indicate that tantalum oxidizes rapidly above $2,800^{\circ}$ F and burns when heated above $3,500^{\circ}$ F.
3. Tantalum is unsuitable for airframe construction when exposed to high air temperatures or heated to high temperatures in still air unless a protective coating can be developed to prevent oxidation.

Langley Aeronautical Laboratory,
National Advisory Committee for Aeronautics,
Langley Field, Va., September 26, 1957.

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3. Purser, Paul E., and Hopko, Russell N.: Exploratory Materials and Missile-Nose-Shape Tests in a 4,000° F Supersonic Air Jet. NACA RM L56J09, 1956.
4. Anon.: American Institute of Physics Handbook. McGraw-Hill Book Co., Inc., 1957.
5. Peterson, Robert C., Fassell, W. Martin, Jr., and Wadsworth, Milton E.: High Pressure Oxidation of Metals - Tantalum in Oxygen. Tech. Rep. V (Contract DA-04-495-ORD-237), Univ. of Utah, Dept. Metallurgy, Oct. 10, 1953.
6. Orr, Raymond L.: High-Temperature Heat Contents of Tantalum and Niobium Oxides. Jour. American Chem. Soc., vol. 75, no. 12, 1953, pp. 2808-2809.
7. Perry, John H., ed.: Chemical Engineers' Handbook. Third ed., McGraw-Hill Book Co., Inc., 1950.

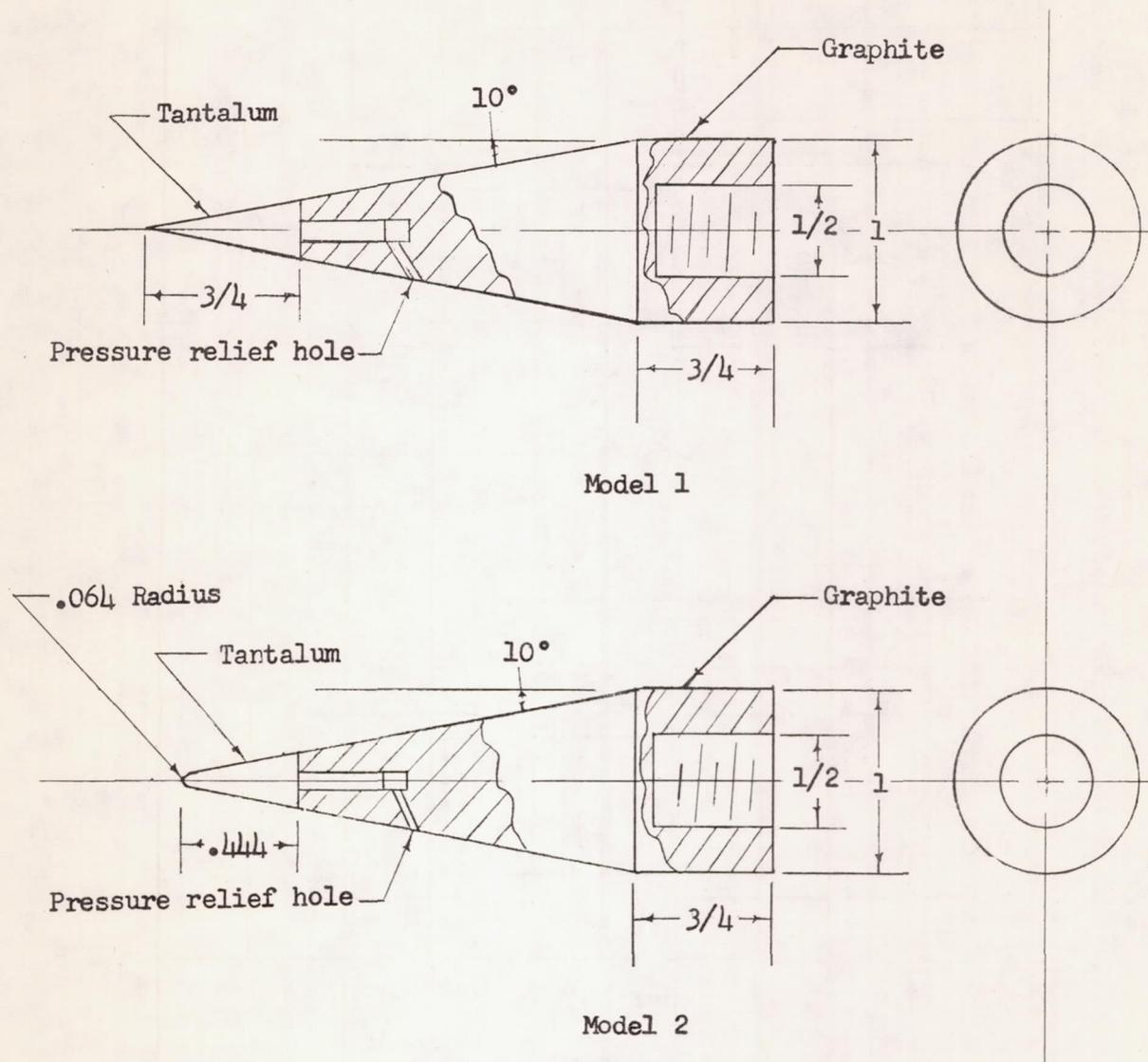
TABLE I.- AVERAGE OXIDATION RATES OF TANTALUM IN STILL AIR

Temperature, °F	Surface condition	Probable oxide formed	Average oxidation rate, $\frac{\text{lb}}{\text{ft}^2\text{-hr}}$	Oxidation heat input, $\frac{\text{Btu}}{\text{ft}^2\text{-hr}}$
2,800	Clean	Ta ₂ O ₅	76	340
	Oxidized	Ta ₂ O ₅	76	340
3,300	Clean	Ta ₂ O ₄	83	Heat of formation of Ta ₂ O ₄ is not known
	Oxidized	Ta ₂ O ₄	65	
3,500	Clean	-----	Temperature rises immediately to 5,100° F; melting is greater than 1,000 $\frac{\text{lb}}{\text{ft}^2\text{-hr}}$ (the limit of the instrument)	
	Oxidized	-----		

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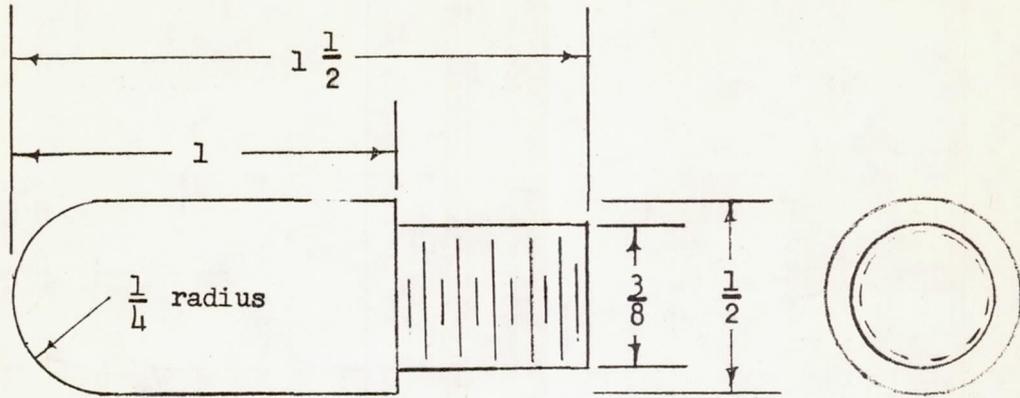
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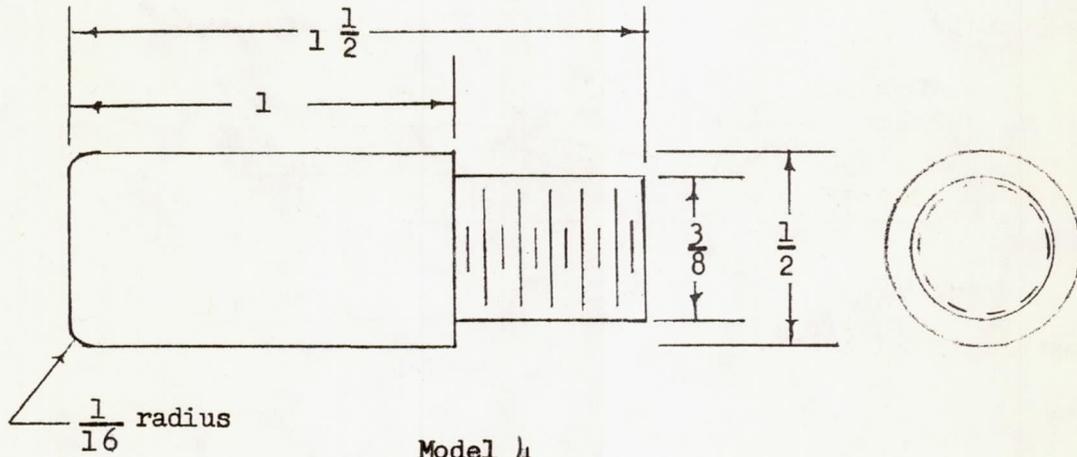


(a) Models 1 and 2.

Figure 1.- Sketches of models. All dimensions are in inches.



Model 3



Model 4

(b) Models 3 and 4.

Figure 1.- Concluded.



0.4 second



3.9 seconds



5.7 seconds

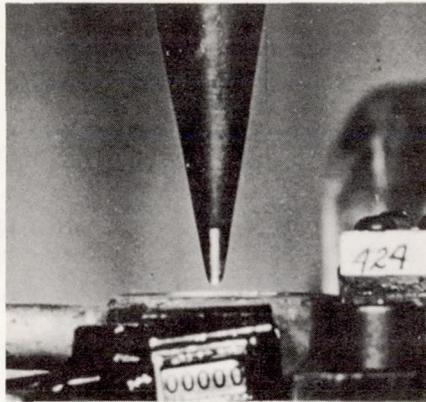


6.9 seconds

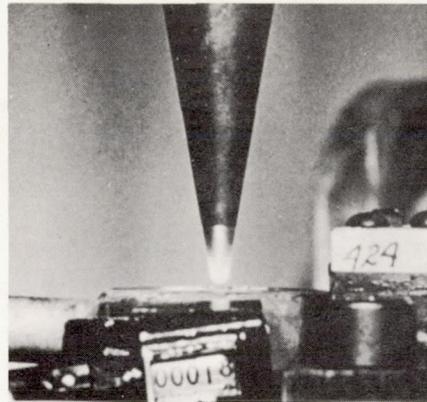
(a) Sharp-pointed tantalum model.

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Figure 2.- Photographs of tests on tantalum models. Air stagnation temperature, 3,800° F.



0 second



1.8 seconds



3.4 seconds



5.9 seconds



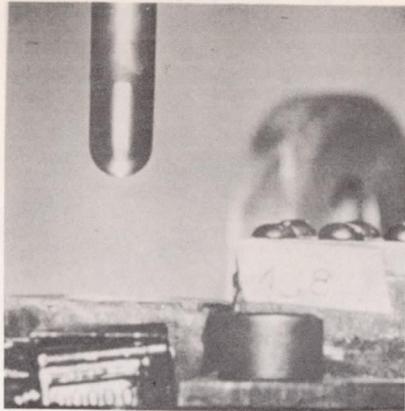
8.4 seconds

(b) Rounded-point tantalum model. L-57-2760

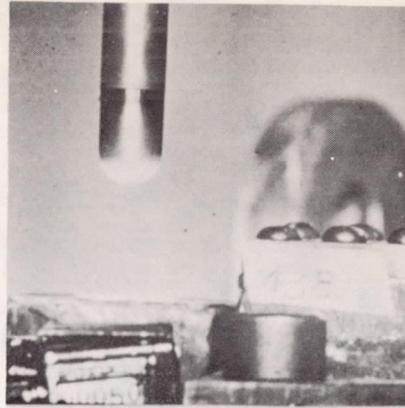
Figure 2.- Continued.

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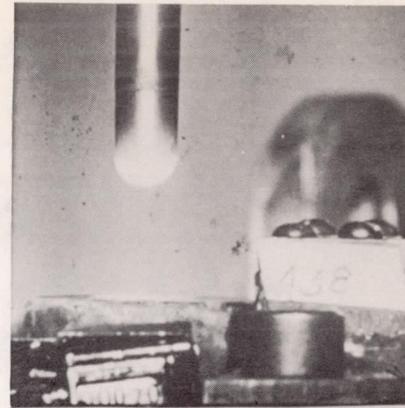
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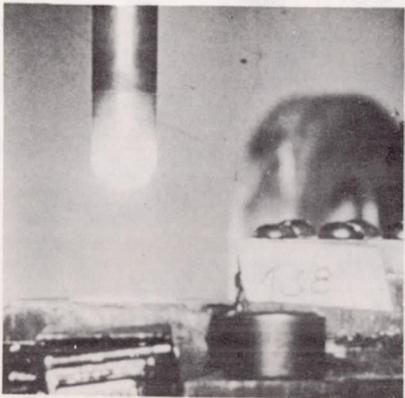
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5.0 seconds



7.0 seconds



9.0 seconds



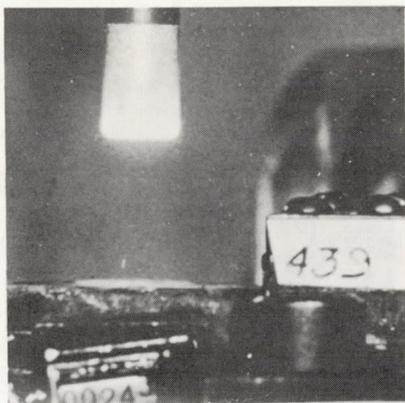
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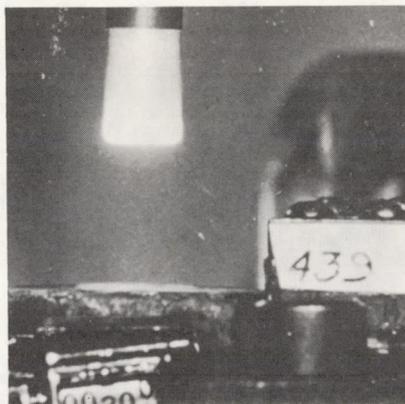
15.0 seconds

(c) 1/2-inch-diameter, solid, hemispherical, tantalum model. L-57-2761

Figure 2.- Continued.



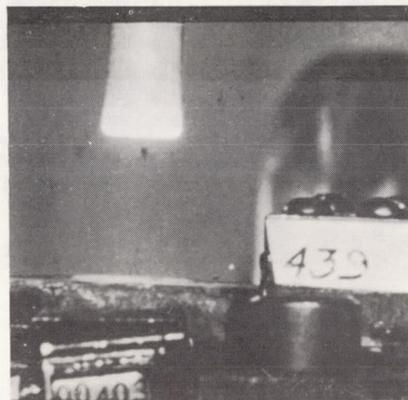
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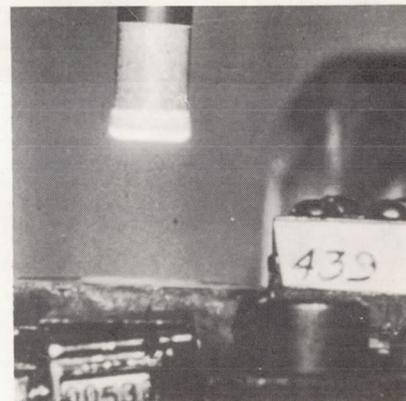
30.0 seconds



35.3 seconds



40.3 seconds



53.0 seconds

(d) 1/2-inch-diameter, flat-faced, solid, tantalum model.

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Figure 2.- Concluded.

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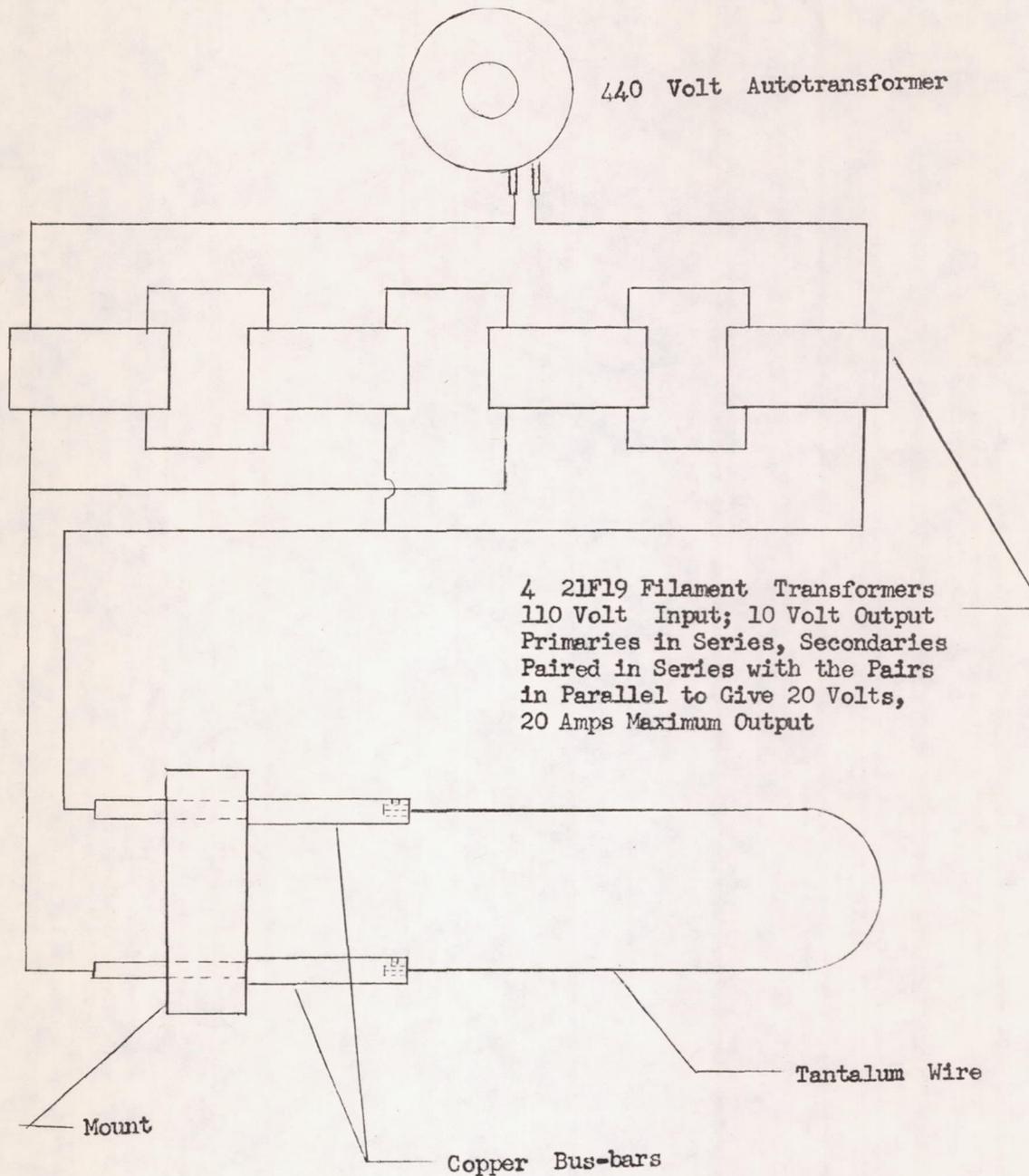


Figure 3.- Diagram of apparatus for still-air oxidation study.

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