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COMPATIBILITY EXPERIMENTS OF FACILITIES, MATERIALS, AND PROPELLANTS FOR ELECTROTHERMAL THRUSTERS

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ABSTRACT

Experiments were performed to determine the compatibility of materials and propellants for electrothermal thrusters. Candidate propellants for resistojet propulsion include carbon dioxide, methane, hydrogen, ammonia, and hydrazine. The materials being examined are grain stabilized platinum for resistojets for space station and rhenium for high performance resistojets for satellites. Heater mass loss and deterioration of materials were evaluated.

A coiled tube of platinum, with yttria dispersed throughout the base material to inhibit grain growth, was tested in carbon dioxide at 1300 °C for 2000 hr. Post-test examination indicated the platinum-yttria heater would last over 100 000 hr with less than 10 percent mass loss.

Short-term compatibility tests were conducted to test the integrity of the platinum-yttria in hydrogen, methane, carbon dioxide/methane mixtures, and ammonia environments. In each of these 100 hr tests, the platinum-yttria mass change indicated a minimum coil life of 100 000 hr.

Facility related effects were investigated in materials tests using rhenium heated to high temperatures. Refractory metals, such as rhenium, are readily oxidized and affected by facility cleanliness. Because as little as 1 ppm of oxygen can cause significant oxidation of rhenium, several methods of reducing oxygen and water in the vacuum facility were evaluated. The methods included titanium used as a getter for oxygen, a liquid nitrogen system simulating freon (-50 °C), and a liquid nitrogen system (-120 °C). In each case, the mass change and condition of the rhenium was examined. Vacuum facility water reduction was monitored using a mass spectrometer. In vacuum environments obtained using only diffusion pumping and those obtained with the assistance of cryogenic equipment there were mass gains in the rhenium heaters. These mass gains were the result of the combination of carbon (from the backstreaming pumping system) with the rhenium test articles. In a research grade nitrogen environment there was a 10 percent mass loss in a period of 700 hr. This high mass loss rate was primarily the result of the high amount of oxygen and water contained in the gas. Propellant purity and preferred test facility environments are discussed.

INTRODUCTION

Electrothermal thrusters are candidates for space station auxiliary propulsion and satellite propulsion. Resistojet propulsion systems, in particular, have characteristics that make them attractive for many of these applications.

The space station resistojet requires long life and multipropellant capability. A multipropellant resistojet can allow use of fluids onboard the space station for propulsion, providing a potential reduction or elimination of propellant resupply. Satellites require resistojets that maximize propellant performance. These demanding requirements are limited by available materials and their properties, and the compatibility of materials with candidate propellants has become a key consideration.

The materials being investigated in this study are grain-stabilized platinum for the multipropellant resistojet and rhenium for high performance, advanced resistojets. Candidate resistojet propellants include carbon dioxide, methane, steam, hydrogen, ammonia, and hydrazine.

Platinum and alloy-strengthened platinum were considered in the early 1970's as materials for biowaste resistojets in the Manned Orbital Research Laboratory program.[1-3] Platinum was chosen because of its excellent resistance to corrosion and high temperature oxidation.[4] Pure platinum was found to lack adequate strength. Alloying the platinum with 10 to 40 percent rhodium improved the material strength; rhodium, however, does not possess the corrosion resistance of platinum. In CO_2 above 1200 °C, it was found that rhodium forms a volatile carbonyl compound. Volatilization of this compound weakens the platinum alloy through loss of the rhodium.[5] Grain growth was also found to be a major concern in extended high temperature operation of platinum. Grain growth can cause a reduction in the stress-rupture performance, formation of voids, and physical distortion of materials.

A platinum material grain stabilized with 0.6 percent thoria was developed in the early 1970's for use in biowaste resistojets.[3] This material was found to be compatible with carbon dioxide, but its compatibility with other candidate propellants was not reported.

Rhenium has been investigated in the past as a material for high performance resistojets.[6-10] Several designs using rhenium heater elements and heat exchangers have been developed. In addition to having a high melting point of 3180 °C, rhenium has good high temperature strength and is chemically compatible with hydrogen and hydrazine, two candidate propellants for satellite applications. A major concern in the use of rhenium is the possible loss of structural integrity caused by grain growth at elevated temperatures. Rhenium oxidation is also a concern. Rhenium readily oxidizes at temperatures above 600 °C in environments with greater than 1 ppm of oxygen. Therefore, the purity of test gases and the conditions of test facilities must be considered when evaluating rhenium.

This paper presents preliminary data on the compatibility of platinum-0.6 percent yttria with candidate space station resistojet propellants. Platinum-yttria was chosen because of its grain-stabilization and availability. The objective was to determine, in short-term tests, the effects of different propellants on the grain stabilized platinum. Particular attention was given to mass loss and the propellant effects on the material structure and surface. The results are intended to verify and expand existing platinum compatibility data.

Information about facility effects on material tests is also presented. Rhenium was used in these investigations because it is readily oxidized and affected by facility cleanliness. Several methods of reducing the water and oxygen present in the vacuum facility were evaluated. Effects of gas purity and vacuum pumps are discussed. The results are used to recommend a preferred test facility environment.

APPARATUS

PLATINUM-YTTRIA EXPERIMENTS

Two facilities were used in the stabilized platinum compatibility tests. Figure 1 is the test chamber used in the tests with flowing H_2, CH_4, and the CO_2/CH_4 mixtures. A schematic of the typical apparatus is shown in Fig. 2. The 2.54 cm od (1.0 in) x 20.3 cm (8.0 in) chamber was constructed of a quartz tube with metalized ends. The test chamber was mounted in the bell jar of a vacuum tank. A mechanical roughing pump was used in purging the chamber and maintaining the cell pressure. A current-controlled dc power supply with a 100 A, 100 V maximum capacity was used in each experiment. The platinum-yttria test piece was coiled from 0.203 cm od (0.080 in) x 0.025 cm (0.010 in) wall tubing. It was supported by 0.635 cm (0.25 in) diameter power leads. The platinum-yttria experiment in ammonia incorporated a similar test cell, except the chamber was stainless steel with a quartz viewing window.

An additional experimental facility employed a 45.7 cm (18 in) diameter stainless steel bell jar. This bell jar was used for the platinum-yttria coil tested in stagnant CO_2. A roughing pump was used in initial purging and in test shutdown procedures. The same type of power supply and similar specimen mounting arrangement were used in each experiment.

FACILITY EFFECT TESTS

Each of the tests to determine facility effects were conducted in a 91.4 cm (36 in) diameter bell jar of a vacuum facility capable of 1.33×10^{-5} N/m^2 (10^{-7} torr) vacuum. The test pieces were 0.203 cm od x 0.025 cm wall (0.080 in od x 0.010 in wall) rhenium tubes of 15.24 cm (6.0 in) lengths. The tubes were mounted between two copper blocks, which also served as power leads.

Experiments were conducted in the bell jar using 99.9995 percent purity nitrogen cover gas. Flow meters and a mechanical roughing pump were used to maintain a 6.7 N/m^2 (50×10^{-3} torr) background pressure of nitrogen.

Tests were also conducted with a titanium sublimation pump. The sublimation pump consists of a hollow sphere of titanium with an internal resistance heater. The heater radiated from 200 to 760 W of power causing the outer surface of the titanium to sublime. A control unit was used to adjust the sublimation rate to a value which eliminated the oxygen peaks observed on a mass spectrometer. The rhenium test piece was shielded to prevent line-of-sight contamination by the subliming titanium.

Rhenium test pieces were tested in a vacuum environment of 1.3×10^{-4} N/m^2 (10^{-6} torr) with diffusion pumps providing the vacuum. Additional experiments were operated with the diffusion pumps being augmented by a cryopump system. The cryopump system consisted of copper tubing coiled to 91.4 cm (36 in) (the id of the bell jar), a liquid nitrogen supply and a temperature controller maintained the liquid nitrogen at -120 °C.

TEMPERATURE MEASUREMENT

A two color pyrometer was used to measure temperatures above 700 °C. Temperatures below 700 °C were measured using a calibrated chromel-alumel thermocouple.

PROCEDURE

Pt/Y_2O_3 COIL PREPARATION

The Pt/Y_2O_3 tube was vacuum annealed at 1000 °C for 30 min. After annealing the material was ductile enough to be coiled.

Each coiled tube was cleaned with both NA-500 (a degreaser) and ethyl alcohol and then dried with high purity nitrogen. The mass of each coil was measured with an analytical balance readable to 0.1 mg.

PLATINUM-0.6 PERCENT YTTRIA TESTS

A series of experiments to determine the compatibility of platinum-0.6 percent yttria metal with H_2, CH_4, NH_3, and 2:1 and 1.5:1 mixtures of CO_2 and CH_4 was performed. Each Pt/Y_2O_3 coil was maintained in a flowing gas environment. The test cell was brought to the operating pressure of 1.38×10^5 N/m^2 (1030 torr) by closing the gas outlet valve and flowing gas into the chamber. Next, the mass flow was set by opening the outlet valve and adjusting the flow controllers to maintain the cell pressure and allowing a mass flow rate of 100 standard cm^3/min. The Pt/Y_2O_3 coils tested in H_2, CO_2, and NH_3 were heated to 1300 °C and those tested in CH_4 and mixtures of CO_2/CH_4 were heated to 500 to 600 °C. The current and voltage ranges were 20 to 40 amps and 1 to 10 V and depended on the desired operating temperature. The coil was brought to the desired operating temperature by passing a current through it and employing resistive self heating. A chart recorder recorded the current, voltage, and flow rate throughout the experiment. Each of these experiments lasted approximately 100 hr.

A long-term experiment was done with a Pt/Y_2O_3 coil in a stagnant CO_2 environment. The bell jar was pumped using a roughing pump. The chamber was purged several times with CO_2 before filling it to the operating pressure of 6.7×10^4 N/m^2 (500 torr). Power was then applied to the coil and adjusted for a coil temperature of 1300 °C. The current, voltage, chamber pressure, and coil temperature were recorded. The Pt/Y_2O_3 coil accumulated 2000 hr in the stagnant CO_2 environment.

FACILITY EFFECT TESTS

The experiments involving rhenium were conducted to determine the effect of the test environment. The rhenium tubes were cleaned and weighed using the procedure described for the Pt/Y_2O_3 coils.

The rhenium tube was mounted in the bell jar, and the chamber was outgassed for three days using a diffusion pump to 1.3×10^{-4} N/m^2 (10^{-6} torr) vacuum range. After three days, the chamber was purged several times using nitrogen. The nitrogen flow was then adjusted to maintain a 6.7 N/m^2 (50×10^{-3} torr) nitrogen background pressure with the roughing pump operating. The rhenium tube was then resistively heated to 1800 °C for 70 hr of testing.

In the next test, after mounting the sample and outgassing the port for three days, the titanium sublimation pump was run at maximum sublimation rate until a mass spectrometer reading indicated no water was present. This procedure took about 30 min. The tank was then filled with 99.9995 percent purity nitrogen to a pressure of 6.7 N/m^2 (50×10^{-3} torr) with the sublimation pump operating. The rhenium tube was operated in this environment for 70 hr at 1800 °C.

The final tests were operated in vacuum, allowing the pump oil to backstream into the chamber. The rhenium was run at 1800 °C for 70 hr at a pressure of 1.3×10^{-4} N/m^2 (10^{-6} torr) after three days of diffusion pumped outgassing. Comparisons were made between diffusion pumping and a combination of diffusion pumping and cryopumping on carbon absorption by rhenium.

RESULTS AND DISCUSSION

PLATINUM-YTTRIA PROPELLANT COMPATIBILITY

A summary of compatibility experiments is presented in Table I. The mass change in each experiment is presented in terms of test length. In each 100 hr compatibility experiment, mass loss was measured. The greatest mass loss occurred in the ammonia environment. Based on this mass loss and using 10 percent loss as the failure criterion, the minimum expected life in any of the candidate propellants is in excess of 100 000 hr.

The platinum-yttria coils in CO_2, H_2, and NH_3, were resistively heated to 1300 °C. This is the selected typical operating temperature for a resistojet heater fabricated from platinum. Platinum-yttria coils were heated in CH_4 and mixtures of CO_2/CH_4 to temperatures of 500 to 600 °C. This temperature range was chosen so that there would be no carbon deposition.

Methane decomposes to hydrogen and solid carbon at about 670 °C.[1] Mixtures of CO_2/CH_4 have a carbon deposition threshold temperature of 727 to 782 °C.[1] These temperatures indicate initial carbon deposition; the actual rate of carbon deposition depends on the rate of CH_4 decomposition. In previous testing it was found that the decomposition rate is a function of pressure as well as temperature.[11] Determination of the effect of temperature and pressure on the decomposition of methane may allow higher operating temperature of CH_4 and CO_2/CH_4 mixtures.

These tests were operated at a cell pressure of about 1.38×10^5 N/m^2 (1030 torr). The effect of varying pressure should be evaluated in any further testing.

Figure 3 shows the grain growth of pure platinum after 100 hr of operation at 1300 °C. This test illustrates grain growth in high temperature operation. The loss of strength and structural integrity that may result from grain growth, as well as relative movement and dislocation of adjacent grains, led to the investigation of platinum stabilized with 0.6 percent yttria reported herein.

The test results indicate that platinum-yttria will meet the life requirements for a space station resistojet with the candidate propellants. Another area of concern is the effect of the propellant on the stabilizer added to the platinum to control grain growth.

Figure 4 shows the structure of the surface of the platinum-yttria before testing. The series of Figs. 5 to 9 show the platinum yttria surface after heating in CH_4, 2:1 and 1.5:1 mixtures of CO_2 and CH_4, H_2, and NH_3, respectively. The coils heated in CH_4 and the CO_2/CH_4 mixtures appear unaffected. The surface structure looks like the untested Pt/Y_2O_3 coil, and there is no evidence of change in the grain size.

Figure 8 shows the surface of the platinum coil after heating in H_2. The surface appears roughened, with evidence of some slight pitting. As indicated in Table I, the platinum coil experienced no measurable mass change in its exposure to H_2. The material change may only be a surface reaction, but has yet to be verified. The material shows little change in grain size. Long term experiments ($\geq$1000 hr) will enable better determination of platinum-yttria reactions to H_2. In long term experiments, grain growth may become more evident because of loss of the stabilizing oxides in the reducing environment.

The surface of the Pt/Y_2O_3 coil heated in NH_3 showed an extreme change from the untested coil, as seen in Fig. 9. The coil surface is pitted and rough and the coil showed a mass loss of 0.001 g. This indicates that for short term exposure NH_3 does cause some corrosion of Pt/Y_2O_3. Surface analytical methods must be employed to determine if this corrosion has changed the chemical composition of the material. Long term testing will determine if this corrosion increases with time and if the life and strength properties of the material are affected.

A long term evaluation of platinum-yttria in CO_2 has been completed. A coiled tube was run at 1300 °C in a stagnant CO_2 environment for 2000 hr without any measurable mass loss. Figure 10 is the cross-section of a platinum-yttria tube before and after 2000 hr at 1300 °C in CO_2. The grain structure does not appear significantly changed. The platinum-yttria can be considered compatible with CO_2, with no mass loss and no loss of material integrity from grain growth.

FACILITY COMPATIBILITY

Rhenium was selected for these compatibility experiments because it is more readily affected by small amounts of contaminants, such as oxygen and carbon.

One facility problem encountered was water and oxygen in the vacuum chamber. It was found that as little as 1 ppm of oxygen can oxidize rhenium at pressures as low as 1.3×10^{-3} N/m^2 (10^{-5} torr). In an attempt to minimize oxidation, a nitrogen cover gas was introduced into the chamber. A rhenium tube heated to 1800 °C was exposed to this environment for 70 hr. Figure 11 compares the results of mass loss measurements of rhenium tubes in the N_2 environment, N_2 with liquid nitrogen simulating freon, and N_2 with a titanium sublimation pump. The sublimation pump was added to determine if use of an oxygen getter would further reduce oxidation. Freon temperatures were simulated to determine if a freon system would eliminate oxidation. In the 6.7 N/m^2 (50×10^{-3} torr) N_2 environment, the rhenium lost 1.4 percent of its mass. Addition of the sublimation pump reduced the loss to about 0.3 percent. The sublimation pump did significantly reduce the oxidation. The simulation at freon temperatures showed that a freon system would be inadequate. It is interesting to note that with a higher N_2 pressure (200 mtorr) and the sublimation pump, the mass loss is further reduced (see Fig. 11). A mass spectrometer reading indicated that the N_2 reduced the volume percent of water in the tank. Another important consideration in these tests was the purity of the gas. Analysis of gases for water and oxygen was performed as the problem of contaminated ultrapure gases was encountered.

Migration of hydrocarbon oils from vacuum chamber pumps can be a problem at chamber pressures near the vapor pressure of the oils. The pump oils can decompose when they contact rhenium at high

temperatures. The resulting carbon can diffuse into the grain structure of the rhenium and cause embrittlement. Rhenium does not form a carbide, but the carbon infiltration weakens the crystal structure.

Tests were conducted to determine carbon infiltration and its possible reduction. Figure 12 shows the effect of pump type on rhenium mass gain. When using only diffusion pumps, the rhenium tube mass increased 0.17 percent in 70 hr. Chemical analysis of the tube indicated 0.2 percent carbon by weight which is the saturation point for rhenium at 1800 °C. With the cryopumping, the rhenium mass gain was reduced to 0.05 percent.

It became evident from these tests that the effects of vacuum facilities on material tests must be considered. At low pressures, when using diffusion pumps, oil backstreaming must be considered. The use of cold traps in the pumping train may be sufficient to eliminate oil migration. The purity of the gases being used should be known. In testing rhenium this became critical because rhenium is so easily oxidized. In long term tests, facility effects play a major role and must be taken into account.

CONCLUDING REMARKS

Experiments to determine the compatibility of materials and propellants for resistojets were performed. Platinum-0.6 percent yttria chosen for the space station application was evaluated in CO_2, H_2, NH_3, CH_4, and mixtures of 2:1 and 1.5:1 of CO_2 and CH_4. Platinum-0.6 percent yttria coils were operated at 1300 °C in H_2 and NH_3, and at 500 to 600 °C in CH_4 and CO_2/CH_4 mixtures for approximately 100 hr each. Mass loss measurements indicated a minimum coil life of 100 000 hr, which exceeds the 10 000 hr life requirement of space station. The grain stabilization did not appear affected in these short-term tests; however, there was some corrosion of the material surface in the H_2 and particularly in the NH_3 environment. Long-term exposure to reducing environments is a concern as grain growth may become more evident due to loss of stabilizing oxides in these environments. Further experiments in these environments are necessary to determine the long-term effects of H_2 and NH_3 on platinum-yttria material. Platinum-0.6 percent yttria was also run in a stagnant CO_2 environment for 2000 hr at 1300 °C. In this extended test, the platinum-yttria showed no measurable mass loss (to 0.1 mg), no carbon deposition, and no grain growth.

Facility effects were investigated in material tests using rhenium. Rhenium tubes were resistively heated to a temperature of 1800 °C for 70 hr in vacuum and flowing nitrogen gas. Since rhenium is rapidly oxidized by as little as 1 ppm of oxygen, measuring its mass loss for each test was a good indication of the amount of oxygen present in a vacuum system.

The tests have shown that diffusion pumping was sufficient to eliminate oxidation effects in most cases. Backstreaming of oil vapors from the pumping train should be minimized because rhenium samples have absorbed carbon when tested in an environment that permitted oil backstreaming. The samples were embrittled and fractured along the grain boundaries with very little applied force. Slightly contaminated facilities require additional pumping. The best tested additional type of pumping is use of liquid nitrogen cryopumping.

When flowing a gas through a vacuum chamber while running oxidizing materials, the gas purity is critical. Each bottle of gas should be analyzed for water and oxygen content. Ultrapure gases can become contaminated when they are bottled. This was prevalent in over 50 percent of the gases used. The above effects must be taken into account when running vacuum tests on materials affected by carbon, water, and oxygen.

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TABLE I. - SUMMARY OF PLATINUM/0.6 PERCENT YTTRIA EXPERIMENTS

Propellant	Test cell pressure, N/m^2	Coiled heater temperature, °C	Coiled heater mass loss, g/100 hr[a]	Mass loss, percent
CO_2	6.7×10^4	1300	[b]<0.0001	<0.0008
CH_4	1.4×10^5	500	.0003	.003
CO_2/CH_4 (2:1)	1.4×10^5	600	.0007	.006
CO_2/CH_4 (1.5:1)	1.4×10^5	600	.0002	.002
H_2	1.4×10^5	1300	<.0001	<.0008
NH_3	1.4×10^5	1300	.0010	.008

[a]After 100 hr operation, mass measurement accuracy ±0.0001 g.
[b]CO_2 results after 2000 hr of operation.

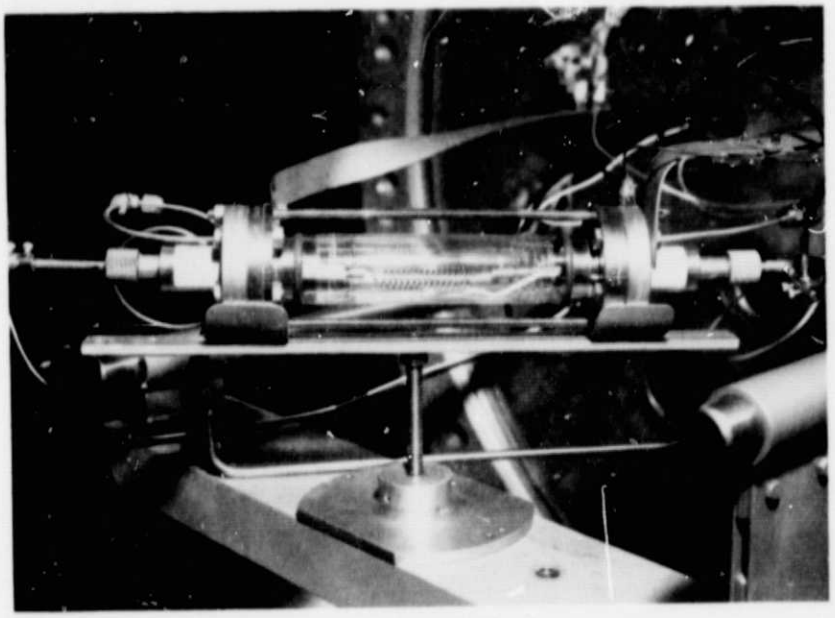


Figure 1. - Material test cell.

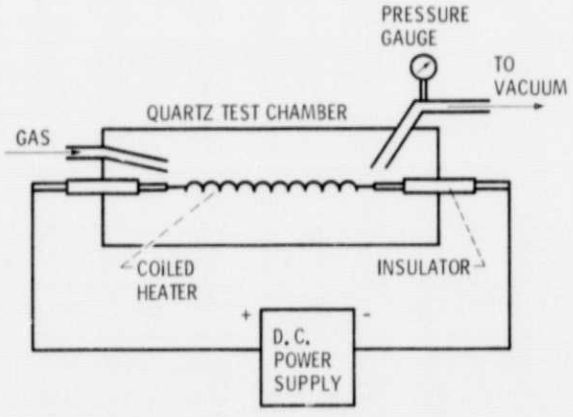
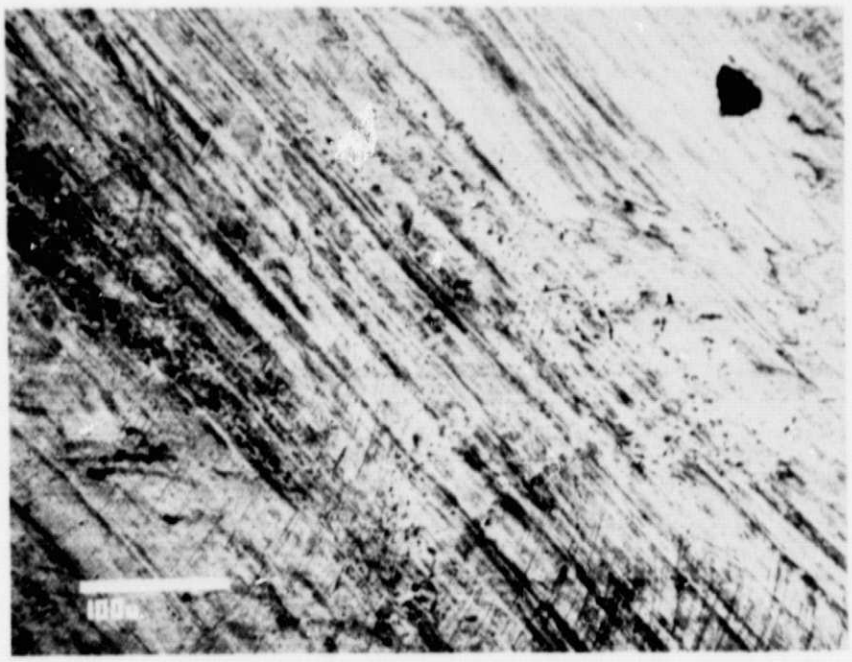
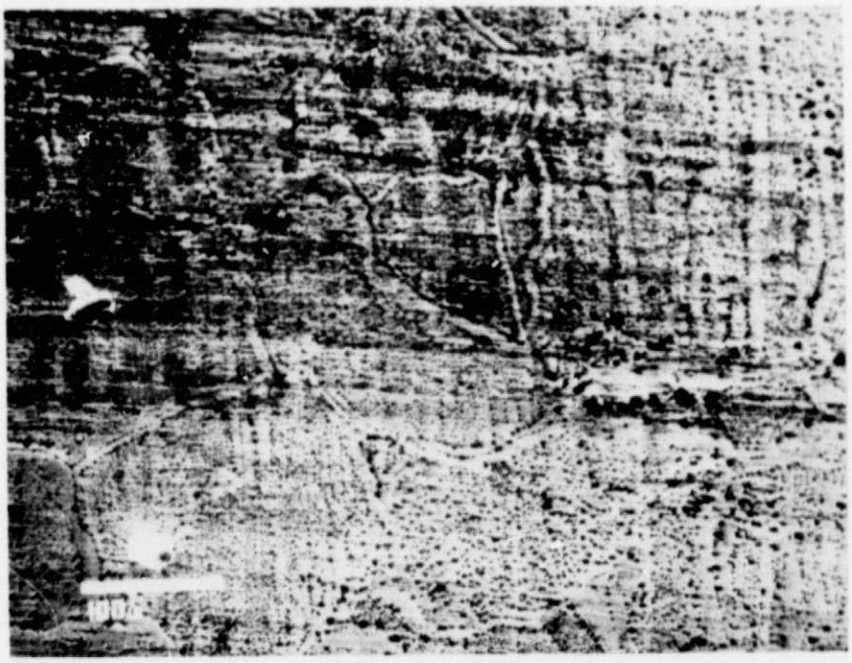


Figure 2. - Material experimental system.



Before 200X



After 200X

Figure 3. - Pure platinum tube surface before and after 100 hr in CO_2 at 1300 ^{0}C.

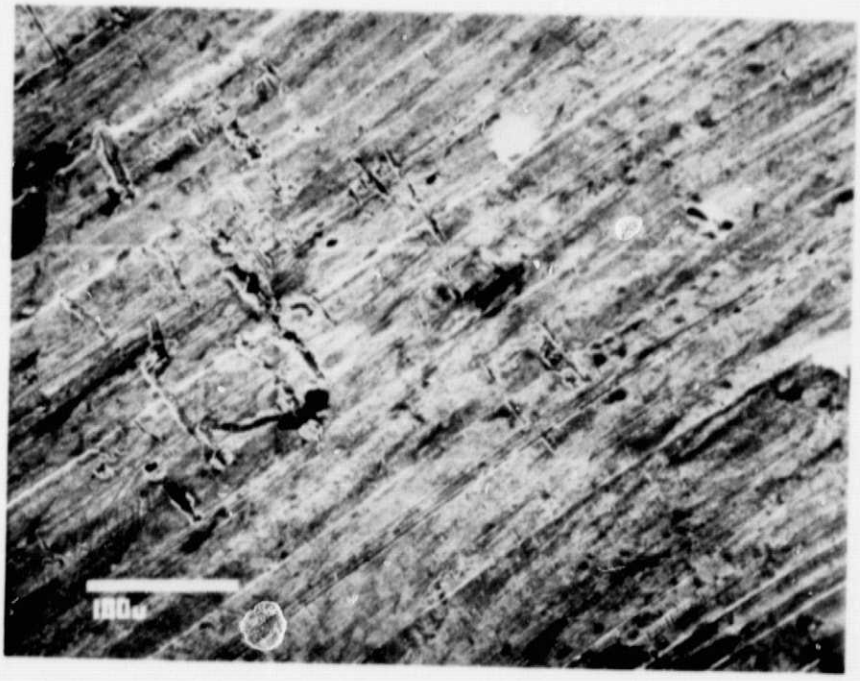


Figure 4. - Platinum-yttria surface before testing (200X).

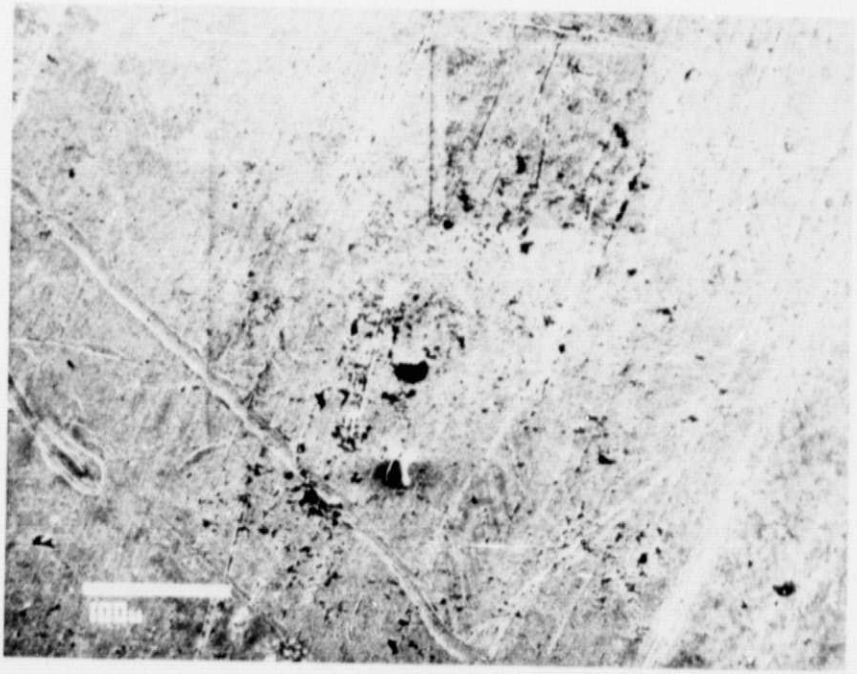


Figure 5. - Platinum-yttria surface after testing 100 hr in CH_4 at 500 ^{o}C (200X).

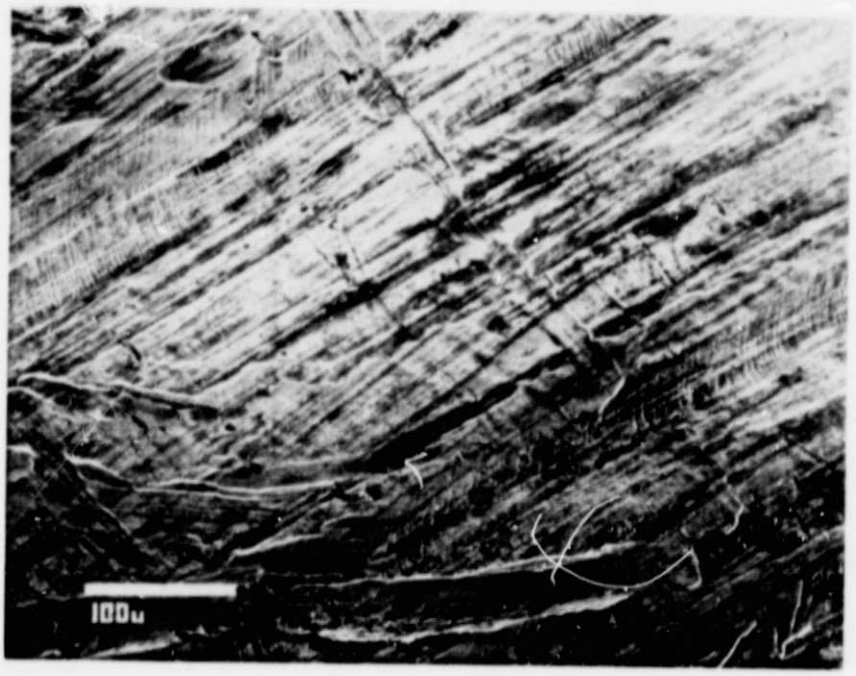


Figure 6. - Platinum-yttria surface after 100 hr in CO_2/CH_4 (2 to 1) at 600 ^{0}C (200X).

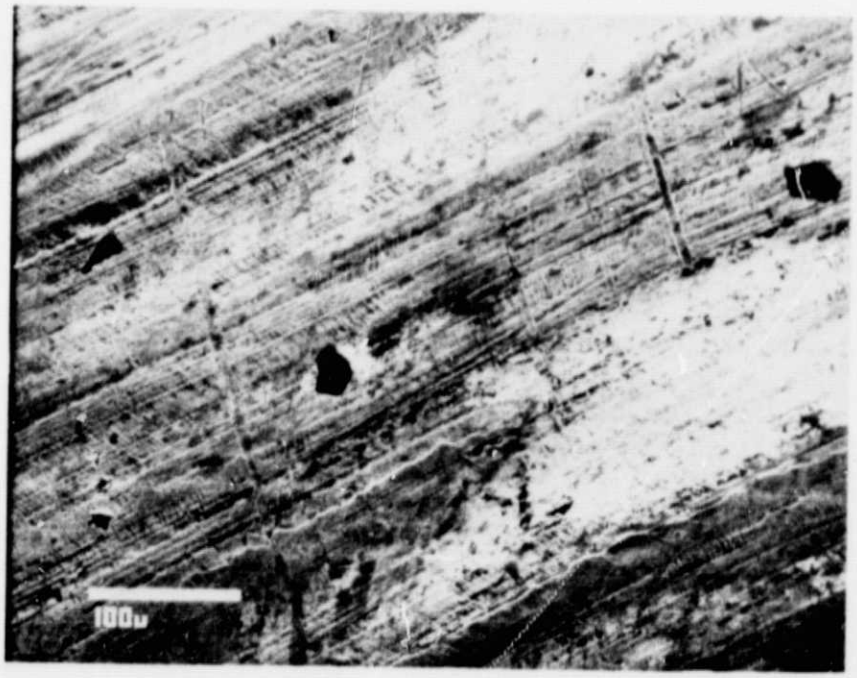


Figure 7. - Platinum-yttria surface after 100 hr in CO_2/CH_4 (1.5 to 1) at 600 ^{0}C (200X).

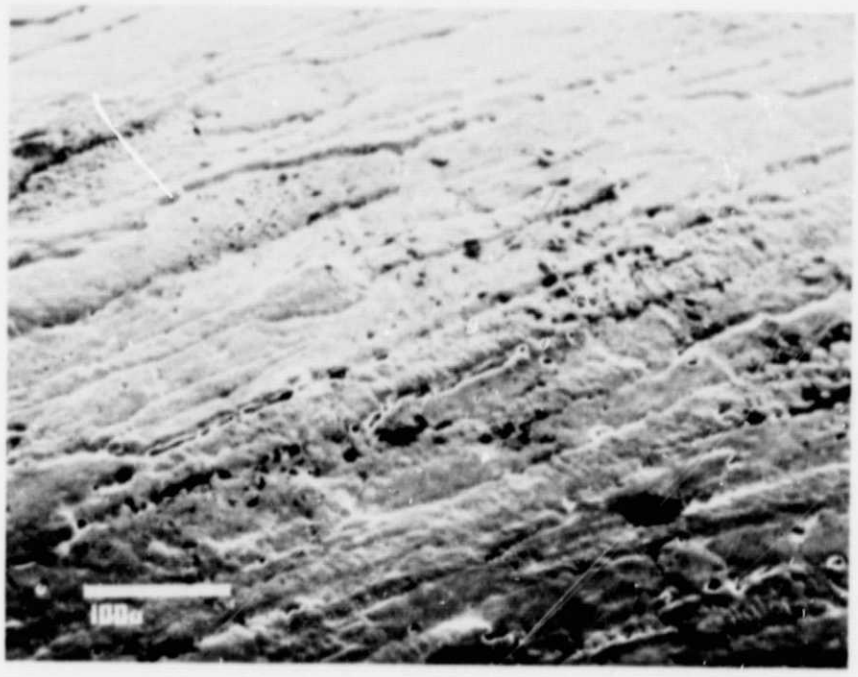


Figure 8. - Platinum-yttria surface after 100 hr in H_2 at 1300 ^{0}C (200X).

Figure 9. - Platinum-yttria after 100 hr in NH_3 at 1300 ^{0}C (200X).

Before 200X

After 200X

Figure 10. - Cross section of Pt/Y_2O_3 tube before and after testing for 2000 hr at 1300 ^{o}C in CO_2 (200X).

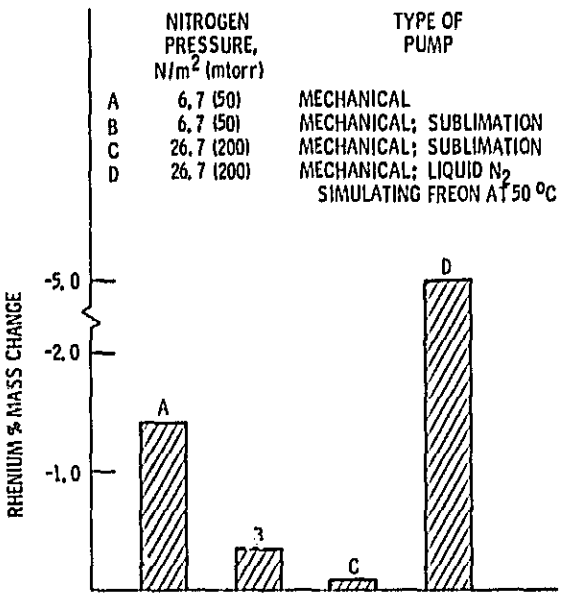


Figure 11. - Oxidation of rhenium under N_2 cover gas, with sublimation pumping or liquid nitrogen system.

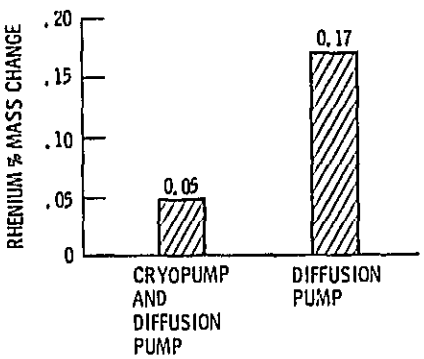


Figure 12. - Effect of pumping system on rhenium mass change