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High-Temperature Oxidation Behavior of Iridium-Rhenium Alloys

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Space Administration**

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SUMMARY

The life-limiting mechanism for radiation-cooled rockets made from iridium-coated rhenium (Ir/Re) is the diffusion of Re into the Ir layer and the subsequent oxidation of the resulting Ir-Re alloy from the inner surface. In a previous study, a life model for Ir/Re rockets was developed. It incorporated Ir-Re diffusion and oxidation data to predict chamber lifetimes as a function of temperature and oxygen partial pressure. Oxidation testing at 1540 °C suggested that a 20-wt % Re concentration at the inner wall surface should be established as the failure criterion. The present study was performed to better define Ir-oxidation behavior as a function of Re concentration and to supplement the data base for the life model. Samples ranging from pure Ir to Ir-40 wt % Re (Ir-40Re) were tested at 1500 °C, in two different oxygen environments. There were indications that the oxidation rate of the Ir-Re alloy increased significantly when it went from a single-phase solid solution to a two-phase mixture, as was suggested in previous work. However, because of testing anomalies in this study, there were not enough dependable oxidation data to definitively raise the Ir/Re rocket failure criterion from 20-wt % Re to a Re concentration corresponding to entry into the two-phase region.

INTRODUCTION

The most common material system currently used for radiation-cooled rockets is a niobium alloy (C-103) with a fused-silica coating (R512) for oxidation protection. However, significant amounts of fuel film cooling are usually required to keep this material below its maximum operating temperature of 1370 °C. In addition, engine performance is degraded by the incomplete mixing of the film coolant with the core flow and some film coolant goes unreacted through the nozzle and becomes a source of contamination in the plume. Recently, high-temperature, oxidation-resistant materials have been developed for radiation-cooled rockets. These materials have the thermal margin to allow long life rocket operation up to 2200 °C, which enables the reduction or elimination of fuel film cooling. The most developed of these high-temperature materials is rhenium (Re) coated with iridium (Ir) (refs. 1 and 2).

There is an ongoing effort to develop flight-type iridium-coated rhenium (Ir/Re) rockets. Such rockets have been tested extensively without failure with Earth storable propellants at two operating conditions: a thrust level of 22 N for 15 hr (ref. 3) and a thrust level of 440 N for 6.3 hr (ref. 4). Complementing this effort is a more fundamental program (ref. 5) that includes characterization of the mechanical properties of Re materials, development of alternate methods for fabricating Ir/Re chambers, development of alternative methods for joining Re chambers to dissimilar metals, development of other material systems that may offer more thermal margin and/or oxidation resistance than Ir/Re, and determination of the life-limiting mechanisms for Ir/Re rockets. This study was conducted as a follow-on to earlier efforts (refs. 3 and 4) to determine the life-limiting mechanisms for Ir/Re rockets and to predict their useful lifetime as a function of temperature and propellant combination (i.e., oxygen partial pressure).

During high-temperature operation, it has been shown (ref. 6) that Re slowly diffuses along grain boundaries into the Ir coating; thus the concentration of Re at the inner surface slowly increases. Oxidation testing at 1540 °C showed (ref. 3) that the Ir oxidation rate increases significantly when the Re concentration exceeds 20 wt %. From this testing, the Re diffusion into Ir with subsequent oxidation of the resulting Ir-Re alloy was identified as the life-limiting mechanism for Ir/Re rockets, and a 20-wt % Re concentration at the inner wall surface was selected as the failure criterion. In addition, a life model incorporating Ir-Re interdiffusion and oxidation data was developed to predict chamber lifetimes. Currently, the empirical data base supporting this life model is sparse.

All of the Ir/Re rocket testing in the initial technology programs was performed with either a cooled section between the chamber and injector or with a platinum-rhodium (Pt-Rh) liner installed in the head end region of the chamber. No problems were experienced with the chambers tested in this manner (refs. 3 and 4). However, two Ir/Re chambers directly welded to injectors experienced erosion or pitting of the Ir layer, primarily in the head end (near the injector) region of the chamber (ref. 7). The reasons for this degradation of the Ir coating are not yet certain, but the phenomenon might pose a threat to the long-life, high-temperature advantages offered by this material system. Most of the Ir degradation occurred in the relatively low-temperature, head end region of the chamber, so the flowfield was probably still mixing and oxidizers and combustion radicals might have been present at the wall. When the tests were run using Ir/Re chambers with a cooled section or liner, the Ir was not exposed to the flowfield until it was well mixed and nearly completely combusted. Circumstantial evidence seems to indicate that the Ir degradation was a result of the reaction of Ir with combustion radicals, the oxidation of the underlying rhenium through the Ir grain boundaries, or the direct oxidation of Ir as a result of the high partial pressures of the oxidizers at the wall. However, testing has not yet been conducted to determine conclusively the specific causes of the Ir degradation.

This report discusses the results of oxidation testing that was performed in two oxygen environments at 1500 °C, with samples ranging from pure Ir to Ir-40 wt % Re (Ir-40Re). The purpose of the testing was to better define the failure criterion for Ir/Re rockets through a more detailed evaluation of the effect of Re concentration on Ir oxidation. The report also discusses the oxidation behavior of Ir as a function of oxygen partial pressure and Re concentration and reviews previous studies of high-temperature Ir oxidation. The Ir oxidation rates determined in this study were incorporated into the Ir/Re life model; the effect of these rates on predicted chamber lifetimes is reviewed.

Ir/Re CHAMBER LIFE-LIMITING MECHANISMS

Wooten and Lansaw (ref. 3) identified Re coated with Ir as a long-life, high-temperature material system for radiation-cooled rockets. Chemical vapor deposition (CVD) was used to fabricate the Ir/Re rockets. The life-limiting mechanism for Ir/Re rockets is thought to be the diffusion of Re through the Ir layer and the subsequent oxidation of the resulting Ir-Re alloy. To determine the oxidation rate, thermogravimetric analyses (TGA) were performed at 1540 °C with Ir, Ir-20Re, and Ir-40Re samples in an environment of 0.5 vol % oxygen with water saturated argon at 0.028 atm (giving an oxygen partial pressure of 0.014 kPa) and with Ir and Ir-20Re samples in 67 vol % oxygen with 33 vol % nitrogen at 0.028 atm (giving an oxygen partial pressure of 1.93 kPa). The TGA results and the material recession rates calculated from them are shown in table I. At 0.014 kPa oxygen pressure, the Ir oxidation rate increased significantly as a function of Re concentration: from 0.361 μm/hr for Ir-20Re to 58.1 μm/hr for Ir-40Re. On the basis of this work, the failure criterion for Ir/Re rockets was established as 20-wt % Re at the Ir inner surface. An Ir/Re rocket was operated at a thrust level of 22 N for 15 hr at 2200 °C, on monomethylhydrazine/nitrogen textroxide (MMH/NTO) propellants, without any degradation or erosion.

The Ir-Re phase diagram (ref. 8) is shown in figure 1. The lower abscissa is weight percentage Re and the upper abscissa is atomic percentage Re. The dashed lines in the phase diagram represent the uncertainty of the locations of region boundaries. The Ir-Re phase diagram shows that the Ir-20Re sample is a single-phase solid solution, whereas the Ir-40Re sample is a two-phase mixture. The boundary between the single- and two-phase regions represents the solubility limit of Re in Ir. The Re-rich phase, which was reported (ref. 3) to have a composition of 29-wt % Ir and 71-wt % Re, accounts for over a third of the Ir-40Re alloy at 1540 °C. The sudden, large increase in oxidation rate between the two samples was attributed to the Re-rich phase, which is thought to be more susceptible to oxidation (ref. 3). The failure criterion for Ir/Re rockets, then, might be set at entry into this two-phase region, where a Re-rich phase would begin to appear in the alloy, rather than at the 20-wt % Re concentration. Setting the failure criterion at a higher Re concentration would result in the prediction of a significant amount of additional chamber life for Ir/Re rockets, particularly since the diffusion rates of Re into Ir are thought to be slow.

A follow-on technology program (ref. 6) was initiated to investigate the life-limiting mechanisms for Ir/Re rockets and to develop flight-type rockets with thrust levels of 62 and 440 N. Ir-Re samples were prepared by CVD and were vacuum annealed at either 1400, 1700, or 1900 °C for 8 hr. Electron microprobe analyses of the samples revealed significant diffusion of Re along Ir grain boundaries, whereas there was little diffusion of Ir into Re. Diffusion constants for the samples were determined by curve fitting of microprobe line scans, with the assumption that only Re diffused into Ir. The diffusion constant was found to have an Arrhenius dependence on temperature T and was fitted to an equation of the form

$$D = D_0 \exp(-E/kT)$$

where $D_0 = 3.14 \times 10^8$ cm^2/sec and $E/k = 1.42 \times 10^4$ K. The activation energy of diffusion E was found to be 1.23 eV. A first-order life prediction model was established for Ir/Re rockets by using the diffusion rate data in the one-dimensional diffusion equation

$$\partial C / \partial t = D(\partial C^2 / \partial x^2)$$

where C is the Re concentration, t is time, and x is the position normal to the Ir/Re interface. This equation was solved numerically by using the Crank-Nicholson differencing scheme, with the boundary conditions of zero Re flux at the Ir (inner) surface and 100-percent Re concentration at the Ir/Re interface. In the life model, the inner surface was assumed to be removed at a fixed recession rate; this assumption is supported by the Jassowski TGA data (ref. 6). The limit on chamber lifetime was established as the time required to reach 20-wt % Re concentration at the inner surface. Although the diffusion rate data could have been extrapolated to higher temperatures, the lack of Ir-Re oxidation data precluded applying the life model to higher temperatures and different oxygen environments.

Ir-Re ALLOY OXIDATION TESTING SURVEY

There has been extensive study of the oxidation characteristics of Ir and the other Pt-group metals, but less so of Ir-Re alloys. Recession rate data from different studies (with various temperatures, oxygen partial pressures, and flowrates) are summarized in table II for Ir and table III for Ir-Re alloys. On the basis of testing done with resistively heated Ir wires, Wimber (refs. 9 and 10) suggested that in a flowing gas, equilibrium is established at the metal-gas interface and the overall oxidation rate is limited, for all practical purposes, by diffusion through the boundary layer that forms adjacent to the surface. However, the partial pressures of the diffusing oxide species, which are the driving forces for the diffusion process, are determined by the chemical reaction's equilibrium constants and the oxygen partial pressures. In Wimber's study, the boundary layer consisted almost entirely of oxygen, and therefore, the inward diffusion of oxygen was not expected to be a controlling contributer to the oxidation rate. The limiting diffusion process, then, involve the outward diffusion of the oxide molecules, along with the unoxidized Ir atoms at higher temperatures. The oxidation rate was, therefore, believed to be controlled by the rates of evaporation of the gaseous species of Ir, IrO_2, and IrO_3, and by the rates of their subsequent diffusion through the gaseous boundary layer adjacent to the Ir surface. (The IrO was felt to have a negligible partial pressure compared to the other oxide species). At low pressures ($<1 \times 10^{-5}$ atm) and high temperatures (>1965 °C), the recession rate was independent of oxygen partial pressure.

In oxidation testing of polycrystalline Ir discs, Jehn (ref. 11) found that the Ir recession rate is proportional to the oxygen pressure raised to 0.3 power, for oxygen pressures between 0.001 and 0.1 kPa and temperatures between 1500 and 2000 °C. At lower pressures and higher temperatures, the recession rate became pressure independent since the evaporation of the pure Ir metal predominated. Between 1500 and 1900 °C and at lower pressures, the recession rate was proportional to the oxygen pressure. Jehn noted that the theoretical treatment of the oxidation process at higher pressures was difficult since, in addition to the chemical surface reactions, the mass transfer in the gas phase and the natural or forced convection have to be taken into consideration.

Lee (K.N. Lee and W.L. Worrell, 1989, University of Pennsylvania, Philadelphia, PA, personal communication) conducted oxidation testing of Ir from 1675 to 2400 °C and of Ir-Re alloys from 2200 to 2500 °C. He found Ir oxidation rates of at least an order of magnitude lower than those reported by Wimber for similar oxygen pressures and comparable to those reported by Jehn, despite having tested in higher oxygen pressures and temperatures. Lee felt that this difference in Ir oxidation rates could not be accounted for by different procedures in measuring temperature or in establishing oxygen pressure. TGA data from Wooten (ref. 3) also indicate that Ir oxidation rates are an order of magnitude lower than those reported by Wimber. Direct comparison of Ir-Re oxidation data of Lee and Wooten is not practical, however, given the differences in temperature (2200 °C versus 1540 °C) and oxygen pressure (7.7 kPa versus 0.014 and 1.93 kPa). However, Lee's data do show fairly linear oxidation behavior among the three samples at or under 20-wt % Re and show a sharp increase in oxidation rate between the 20- and 30-wt % Re samples.

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NASA LEWIS Ir-Re OXIDATION TESTING

Ir-Re Samples

In an Ir/Re rocket, the percentage of Re at the inner surface increases over time as Re diffuses into the Ir layer. The present study used Ir-Re alloys of varying Re concentrations to represent the changing composition at the inner surface. Two groups of samples were procured for this testing. The first group ranged from pure Ir to Ir-40Re, in approximate increments of 5-wt % Re. The second group ranged from pure Ir to Ir-30Re, in approximate increments of 2.5-wt % Re. Compositional analyses of the scrap material with energy dispersive spectroscopy (EDS) revealed some variations from the intended compositions. Table IV lists the sample compositions as determined from the EDS analyses.

All of the Ir-Re samples were vacuum-arc-melted buttons, roughly 1.27 cm in diameter and 0.635 cm thick. They were irregularly shaped and had to be machined into a geometry with a surface area that could be readily measured. Electrical discharge machining (EDM) was used to cut rectangular samples out of the buttons. The buttons from the first group were cut with a brass electrode into two 0.953- by 0.953- by 0.318-cm rectangular slabs. The buttons from the second group were cut with a molybdenum electrode into three 0.953- by 0.318- by 0.476-cm rectangular slabs.

1500 °C Horizontal Tube Furnace Testing

The first group of samples were tested in a horizontal tube furnace operating at 1500 °C, which was the maximum operating temperature of the furnace. The samples were inserted slowly (i.e., over a period of 10 min) into the furnace at temperature. The samples had to be inserted slowly since they were held in an alumina boat, which had poor thermal shock resistance. One set of samples from the first group was tested in an oxygen environment of 0.69 kPa partial pressure oxygen (0.7 vol % oxygen in ultrahigh purity argon at 1 atm). This environment simulated the oxygen partial pressure of a chamber operating on MMH/NTO propellants at a mixture ratio of 1.65 and chamber pressure of 690 kPa. The other set of samples from the first group was tested in an oxygen environment of 6.90 kPa partial pressure oxygen (7.0 vol % oxygen in ultrahigh purity argon at 1 atm). This environment simulated the oxygen partial pressure of a chamber operating on gaseous hydrogen/gaseous oxygen (GH_2/GO_2) propellants, at a mixture ratio of 6 and chamber pressure of 690 kPa. The samples were weighed before and after testing to determine mass change. The material recession rates were calculated from the mass change, surface area, and density of the samples. Mass loss was assumed to have occurred from all of the sample surfaces. The raw oxidation data and calculated material recession rates from the first group of samples oxidized in the horizontal tube furnace at 1500 °C are shown in table V.

The furnace data exhibited a significant amount of scatter; oxidation rates were nonmonotonic with increasing Re concentration. The oxidation testing in the 0.69 kPa oxygen environment was repeated with a set of samples from the second group (results shown in table V). Again, anomalous data were found. For example, the recession rate of three samples between 11- and 12-wt % Re that were tested in the 0.69 kPa oxygen environment varied from 0.070 to 0.714 to 2.36 μm/hr. It seemed unlikely that these results were truly representative of the Ir oxidation behavior.

After the second group of samples were tested, some of them were found to have surface flaws in the form of gouges or pits. Table VI lists the surface conditions of the second group of samples, which were determined after furnace testing, and the corresponding recession rates from the furnace testing. A sample with at least one 0.5-mm-diameter pit was characterized as having moderate pitting. A sample with at least one 1.0-mm-diameter pit or larger was characterized as having major pitting. Gouges and pits were also found in a set of samples that had not been subjected to oxidation testing. The flaws were later identified as voids that formed in the Ir-Re buttons during arc-melting fabrication. Because the arc-melting process was not performed in a perfect vacuum, but in a low-pressure argon environment, void formation occurred.

It is important not to confuse the flawed samples discussed in this program with the pitting of the Ir layer that was found in some of the testing with Ir/Re rockets (ref. 7). In the present study, some of the Ir-Re alloy samples had flaws as-received, introduced in the arc-melting process. The pitting referred to in reference 7 was degradation of the pure Ir layer by some injector-related or combustion-related phenomenon occurring during rocket testing.

Figure 2 shows the recession rates for all of the low-oxygen-environment testing, including samples with smooth surfaces or only minor pitting, samples with moderate or major pitting, and samples from the first group, whose condition was not known. (The condition of the first group of samples was not noted before part of their surfaces were abraded for EDS analysis). For the most part, samples with a moderate or major amount of pitting corresponded with anomalous or higher oxidation rates than expected. There were also anomalous data points from the first group of samples, but the oxidation rates were generally below those from the second group.

It is not clear why the flawed samples had high oxidation rates. The flaws in the samples could have had locally high Re concentrations, which caused accelerated oxidation. Since samples with identifiable flaws seemingly corresponded to anomalous data, conclusions about Ir-Re oxidation behavior were drawn only from samples in this study with smooth surfaces or minor pitting. This necessarily restricts the results to data from the second group of samples, since they were the only samples whose surface condition was identified.

The low-oxygen-environment furnace data are replotted in figure 3; this time only the samples with smooth surfaces or minor pitting are represented. There is a sharp increase in oxidation rate between the ~20-wt % Re sample and the ~28-wt % Re sample (from 0.369 to 4.29 μm/hr). There is a still sharper increase in oxidation rates as the Re concentration rises above 30 wt %. The 32-wt % Re sample oxidized at a rate of 7.57 μm/hr and the 35-wt % sample at a rate of 20.8 μm/hr. The oxidation data from the first group of samples indicated similar behavior.

The samples above 30-wt % Re were clearly in the two-phase region of the Ir-Re phase diagram (fig. 1). At 1500 °C the Re-rich phase is composed of approximately 59-wt % Re. For alloys with increasing percentages of Re, the amount of this Re-rich phase in the alloy increases. The sharp rise in oxidation rate seems to indicate that even a small amount of the Re-rich phase leads to accelerated oxidation rates. Although not definitive from the phase diagram, the 28-wt % Re sample falls just short of entry into the two-phase region. The absence of any other credible data from samples with concentrations between 20- and 30-wt % Re makes it difficult to draw any conclusions about Ir oxidation behavior there. This set of tests, then, did not offer enough dependable oxidation data to determine conclusively whether the failure criterion for Ir/Re rockets should be raised from 20-wt % Re at the inner Ir surface to a Re percentage corresponding to entry into the two-phase region.

Figure 4 plots the recession rate data from the first group of samples, tested in both low- and high-oxygen environments. The oxidation rate was generally two to three times faster in the higher oxygen environment for alloys with less than 20-wt % Re and three to five times faster for those with more than 20-wt % Re. However, the unknown condition of the first group of samples and the presence of apparently anomalous data make it difficult to interpret the differences in Ir oxidation behavior between the two oxygen environments.

IMPACT ON Ir/Re CHAMBER LIFE MODEL

The Ir/Re chamber life model described earlier was modified to incorporate the results from the oxidation testing in this program. Since there were not enough credible data from this program to alter the failure criterion, it remains at 20-wt % Re concentration at the inner surface. A linear curve fit was made of the data obtained from the second group of samples with compositions up to 20-wt % Re. The recession rate at 1500 °C and 0.69 kPa oxygen partial pressure was determined from the following equation:

$$\text{recession rate (μm/hr)} = (0.157 \times \text{wt \% Re concentration}) + 0.0182$$

It is important to note that the relevant temperature in the model is the temperature at the inner surface and not that at the outer wall. The temperature drop through the Re wall could be as great as 100 °C, depending on the wall thickness. Usually, the temperature drop through the Ir wall is not significant (5 °C).

The chamber lifetime for silica-coated niobium (R512/C–103) rockets, based on rocket engine qualification tests conducted by various rocket manufacturers (ref. 12), is plotted versus temperature in figure 5. At 1370 °C, which is the maximum operating temperature for R512/C–103 rockets, the chamber lifetime is 10 to 15 hr.

Figure 5 also shows the predicted chamber lifetimes at 1500 °C for Ir/Re rockets with Ir coating thicknesses of 50.8 and 101.6 μm. An Ir/Re rocket with a 50.8-μm Ir coating is predicted to have an order of magnitude increase in chamber lifetime over R512/C–103 rockets. Ir/Re rockets operated at 1500 °C would sacrifice performance gains for greatly extended chamber lifetimes. This application of Ir/Re rockets was proposed for the Space Shuttle Orbiter vernier engines (ref. 13).

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The life model is valid only within the range of oxygen partial pressures and temperatures for which Ir-Re oxidation rate data exist. Although the data gathered in this study had an oxygen pressure representative of the oxygen content in an MMH/NTO propellant combustion environment, the 1500 °C temperature was far below the operating temperatures of Ir/Re chambers (1900 to 2200 °C). Extrapolating this data to make life model predictions above 1500 °C probably yields unrealistically long chamber lifetimes.

There has not been sufficient life testing of Ir/Re rockets to correlate life model predictions with empirical data. An Ir/Re rocket using MMH/NTO was tested at a thrust level of 22 N for 15 hr, primarily at outer-wall temperatures of 2200 °C. There was no evidence of any degradation or dimensional change in the chamber. This data point is shown in figure 5. An Ir/Re rocket using MMH/NTO was also tested at a thrust level of 440 N for 6.3 hr at 1900 °C. Again, there was no evidence of any material degradation. To date, neither of these chambers have been sectioned to determine the amount of Re diffusion into the Ir layer. There is ongoing testing of Ir/Re rockets operating at thrust levels of 22, 62, and 440 N on Earth-storable and GH_2/GO_2 propellants (refs. 5 and 12). Analyses of these rockets should begin to provide an empirical data base with which to compare fundamental diffusion and oxidation data.

CONCLUDING REMARKS

This study was conducted to better define the effect of Re concentration on Ir oxidation behavior and to provide more Ir-Re alloy oxidation data for an Ir/Re rocket life model that predicts useful lifetime as a function of temperature and oxygen partial pressure.

Some data indicated that the Ir-Re alloy oxidation rate increases significantly when the alloy composition crosses the boundary between a single-phase solid solution and a two-phase mixture with a Re-rich phase; however, there were not enough credible data to raise the Ir/Re rocket failure criterion from 20-wt %-Re concentration at the inner surface to a Re concentration corresponding to entry into the two-phase region. More empirical data are needed to make the diffusion-oxidation life model for Ir/Re rockets useful for predicting chamber lifetimes. In particular, Ir-Re oxidation data at temperatures and oxygen partial pressures representative of Ir/Re rocket operating conditions are required.

ACKNOWLEDGMENT

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TABLE I.—WOOTEN'S THERMOGRAVIMETRIC
ANALYSIS OF IRIDIUM/RHENIUM ALLOYS
[From ref. 3.]

Sample composition, wt %	Oxygen partial pressure, kPa	Surface area, cm^2	Mass change rate, $\dfrac{mg \cdot min}{cm^2}$	Recession rate, μm/hr
Ir	0.014	1.27	–0.0057	0.153
Ir-20Re	.014	.70	–.0133	0.361
Ir-40Re	.014	.45	–2.1100	58.1
Ir	1.93	1.28	–.0323	.865
Ir-20Re	1.93	.70	–.0897	2.44

TABLE II.—SURVEY OF IRIDIUM OXIDATION RATE DATA

Temperature, °C	Oxygen partial pressure, kPa	Flowrate, cm/sec	Recession rate, μm/hr
From Wimber (ref. 9)[a]			
1675	0.134	-------------	9.3
1965	.134	-------------	23
2260	.134	-------------	76
1675	1.34	-------------	20
1965	1.34	-------------	76
2260	1.34	-------------	202
1675	13.4	-------------	43
1965	13.4	-------------	97
2260	13.4	-------------	313
1675	134	-------------	202
1965	134	-------------	313
2260	134	-------------	972
From Wimber (ref. 10)			
1675	17.7 (0.83 atm air)	16.03×10^{-3}	259
1965		6.10	230
1965		6.17	428
2260	↓	15.95	1062
1965	.76	2.32	131
	1.34	.99	104
	13.4	.109	182
		.122	207
		.171	213
	↓	.220	207
	33.4	.43	688
	36.1	.60	562
	53.7	.94	954
	62.8	1.08	1724
From Jehn (ref. 11)[a]			
1500	0.010	--------------	0.79
1600		--------------	1.0
1700		--------------	1.4
1800		--------------	3.0
1900		--------------	4.0
2000		--------------	5.3
2100	↓	--------------	7.9
1500	.100	--------------	1.1
1700		--------------	3.2
1900		--------------	7.9
2000		--------------	12
2100	↓	--------------	22
From Lee (personal communication)			
1675	7.7 (0.36 air-argon)	--------------	1.06
2200		--------------	6.98
2295		--------------	13.9
2400	↓	--------------	26.6

[a]Data approximated from curve.

TABLE III.—LEE'S SURVEY OF IRIDIUM-RHENIUM ALLOY
OXIDATION RATE DATA

[From personal communication.]

Alloy composition, wt %	Temperature, °C	Oxygen partial pressure, kPa	Recession rate, μm/hr
Ir-10Re	2200	7.7 (0.36 atm air-argon)	16.4
	2295		31.0
	2400		58.8
	2500		103
Ir-20Re	2200		29.0
	2295		54.8
	2400		103
	2500		183
Ir-30Re	2200		66.0
	2295		122
	2400		236
	2500	↓	412

TABLE IV.—COMPOSITION OF SAMPLES

Nominal composition, wt %	Actual composition			
	wt %		at. %	
First group				
Ir	98.89	1.11	98.85	1.15
Ir-5Re	94.42	5.58	94.25	5.75
Ir-10Re	88.19	11.81	87.85	12.15
Ir-15Re	88.28	11.72	87.95	12.05
Ir-20Re	79.39	20.61	78.86	21.14
Ir-25Re	74.19	25.81	73.58	26.42
Ir-30Re	70.15	29.85	69.48	30.52
Ir-35Re	64.37	35.63	63.64	36.36
Ir-40Re	61.01	38.99	60.25	39.75
Second group				
Ir	98.86	1.14	98.82	1.18
Ir-2.5Re	96.39	3.61	96.28	3.72
Ir-5Re	96.92	3.08	96.82	3.18
Ir-7.5Re	91.30	8.70	91.05	8.95
Ir-10Re	90.29	9.71	90.01	9.99
Ir-12.5Re	88.85	11.15	88.53	11.47
Ir-15Re	86.55	13.45	86.18	13.82
Ir-17.5Re	83.14	16.86	82.69	17.31
Ir-20Re	80.21	19.79	79.70	20.30
Ir-22.5Re	77.10	22.90	76.54	23.46
Ir-25Re	74.23	25.77	73.62	26.38
Ir-27.5Re	72.39	27.61	71.75	28.25
Ir-30Re	67.96	32.04	67.26	32.74
Ir-32.5Re	65.21	34.79	64.49	35.51
Ir-35 Re	64.76	35.24	64.03	35.97

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TABLE V.—RAW DATA FROM 1500 °C HORIZONTAL
TUBE FURNACE TESTING

[Temperature, 1500 °C; oxygen partial pressure, 0.69 kPa; atmosphere (0.7 vol % oxygen in ultrahigh purity argon at 1 atm).]

Re concentration, wt %	Density,[a] g/cm³	Area, cm²	Mass change, g	Recession rate, μm/hr
First group of samples tested for 2 hr				
1.11	22.38	3.057	0.00003	0.0022
5.58	22.32	2.799	.00311	.249
11.72	22.24	3.115	.00989	.714
11.81	22.23	3.040	.00095	.0703
20.61	22.11	3.072	.00390	.287
25.81	22.04	3.043	.02314	1.73
29.85	21.98	3.071	.02530	1.87
35.63	21.91	3.017	.12502	9.46
38.99	21.84	3.069	.25266	18.8
First group of samples tested for 2.5 hr				
1.11	22.38	3.069	0.00062	0.036
5.58	22.32	3.165	.03725	2.11
11.72	22.24	3.010	.03231	1.93
11.81	22.23	3.068	.00222	.130
20.61	22.11	3.100	.00692	.404
25.81	22.04	3.101	.17113	10.0
29.85	21.98	3.069	.12852	7.62
35.63	21.91	3.059	.50880	30.4
38.99	21.84	3.050	.84122	50.5
Second group of samples tested for 2 hr				
1.14	22.38	1.746	0.00033	0.042
3.08	22.36	1.761	.00066	.084
3.61	22.35	1.789	.00047	.059
8.70	22.28	1.800	.00129	.161
9.71	22.26	1.762	.00467	.595
11.15	22.24	1.803	.01893	2.36
13.45	22.21	1.755	.06936	8.90
16.86	22.16	1.835	.00192	.236
19.79	22.12	1.762	.00288	.369
22.90	22.08	1.792	.02166	2.74
25.77	22.04	1.788	.06092	7.73
27.61	22.01	1.754	.03310	4.29
32.04	21.95	1.388	.04618	7.57
34.79	21.91	1.758	.15989	20.8
35.24	21.91	1.745	.17462	22.8

[a] Ir density = 22.4 g/cm³; Re density = 21.0 g/cm³.

TABLE VI.—CHARACTERIZATION OF
1500 °C HORIZONTAL TUBE
FURNACE SAMPLES

[Second group of samples.]

| Re concentration, wt %	Sample condition	Recession rate, μm/hr
1.14	Minor pitting	0.042
3.08	Minor pitting	.084
3.61	Minor pitting	.059
8.70	Major pitting	.161
9.71	Moderate pitting	.595
11.15	Major pitting	2.36
13.45	Major pitting	8.90
16.86	Smooth	.236
19.79	Smooth	.369
22.90	Major pitting	2.74
25.77	Major pitting	7.73
27.61	Minor pitting	4.29
32.04	Smooth	7.57
34.79	Smooth	20.8
35.24	Moderate pitting	22.8
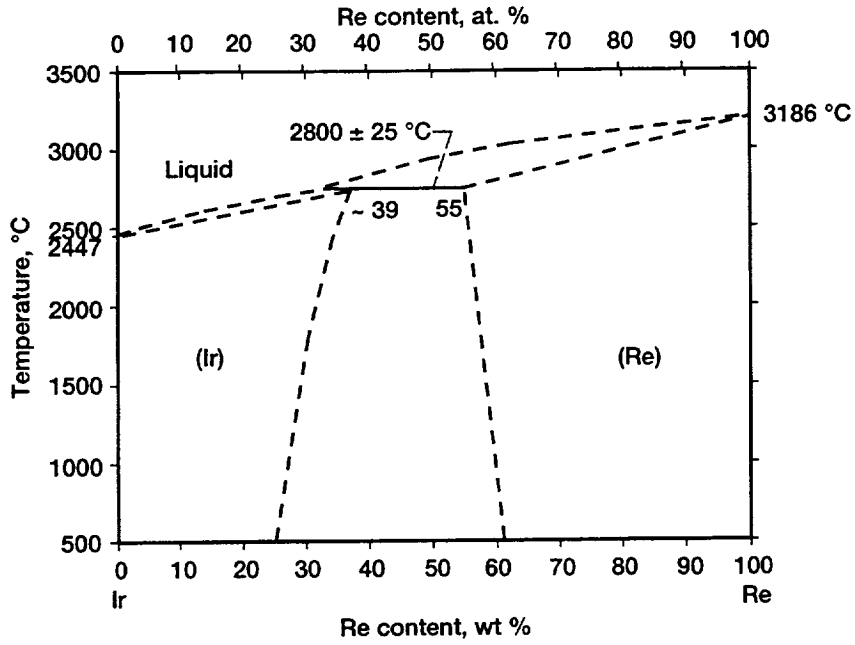
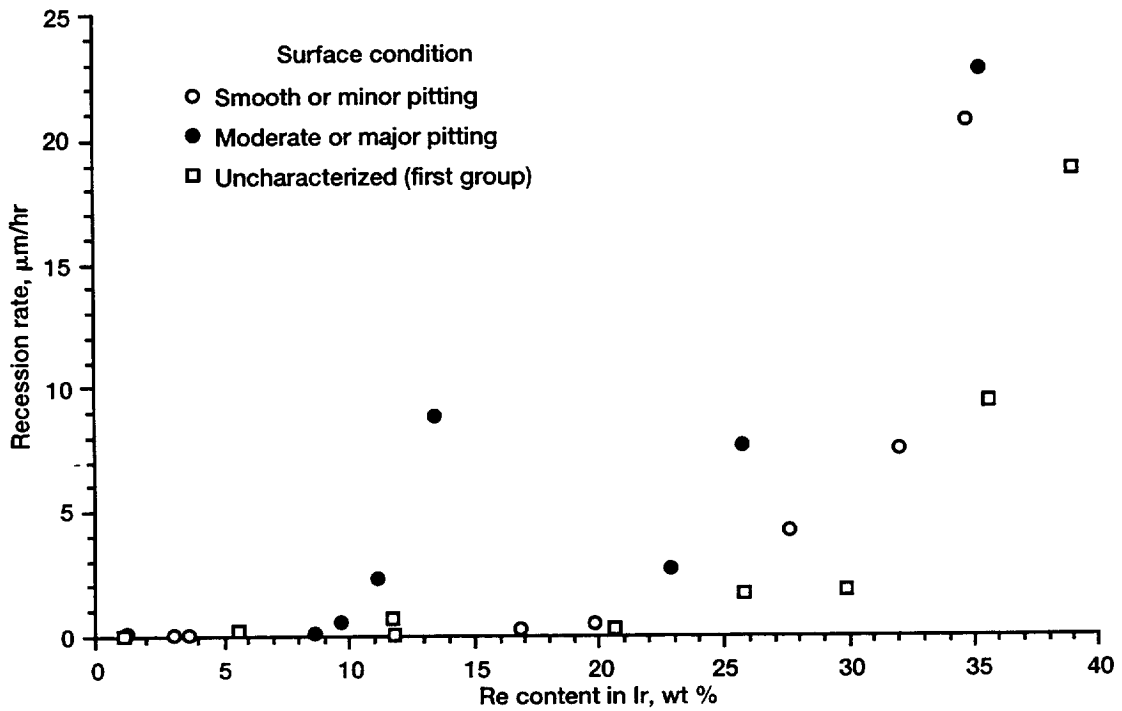Figure 1.—Iridium-rhenium phase diagram (from ref. 8).

Figure 2.—Furnace oxidation data taken from all samples oxidized at 1500 °C in an oxygen partial pressure of 0.69 kPa.
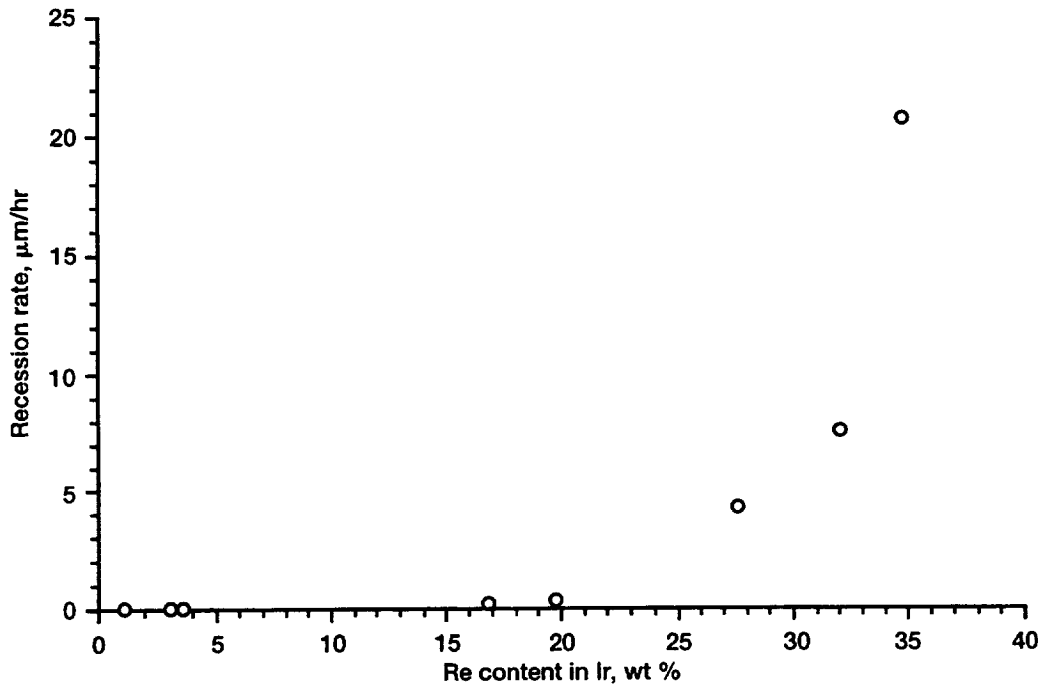
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Figure 3.—Furnace oxidation data from samples with smooth surfaces (oxidized at 1500 °C in an oxygen partial pressure of 0.69 kPa).
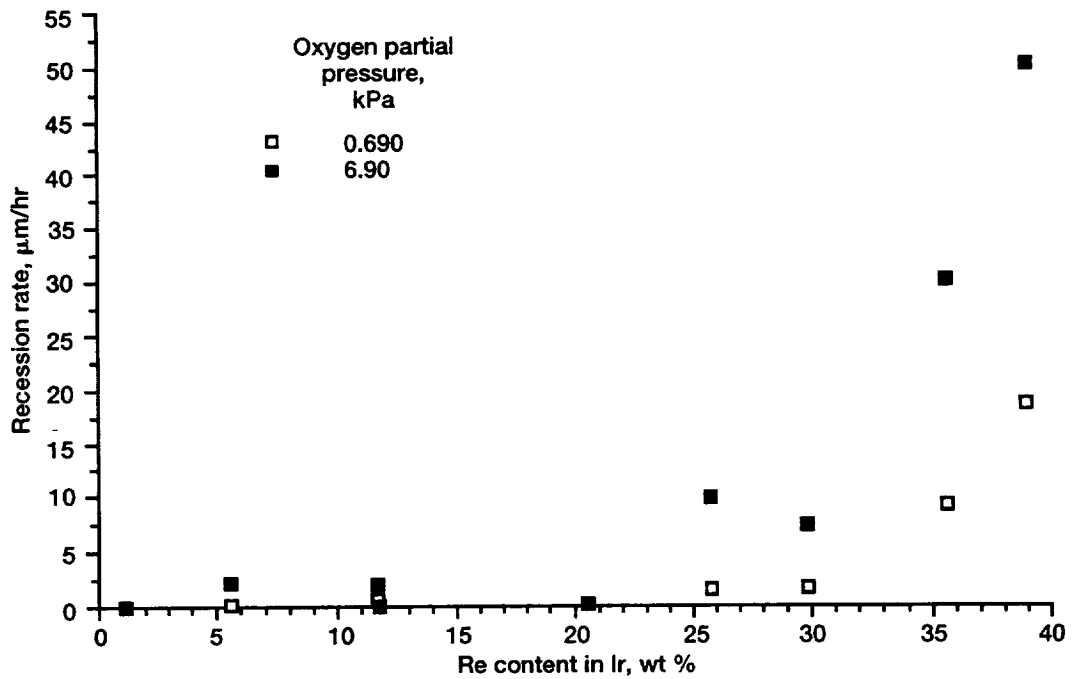

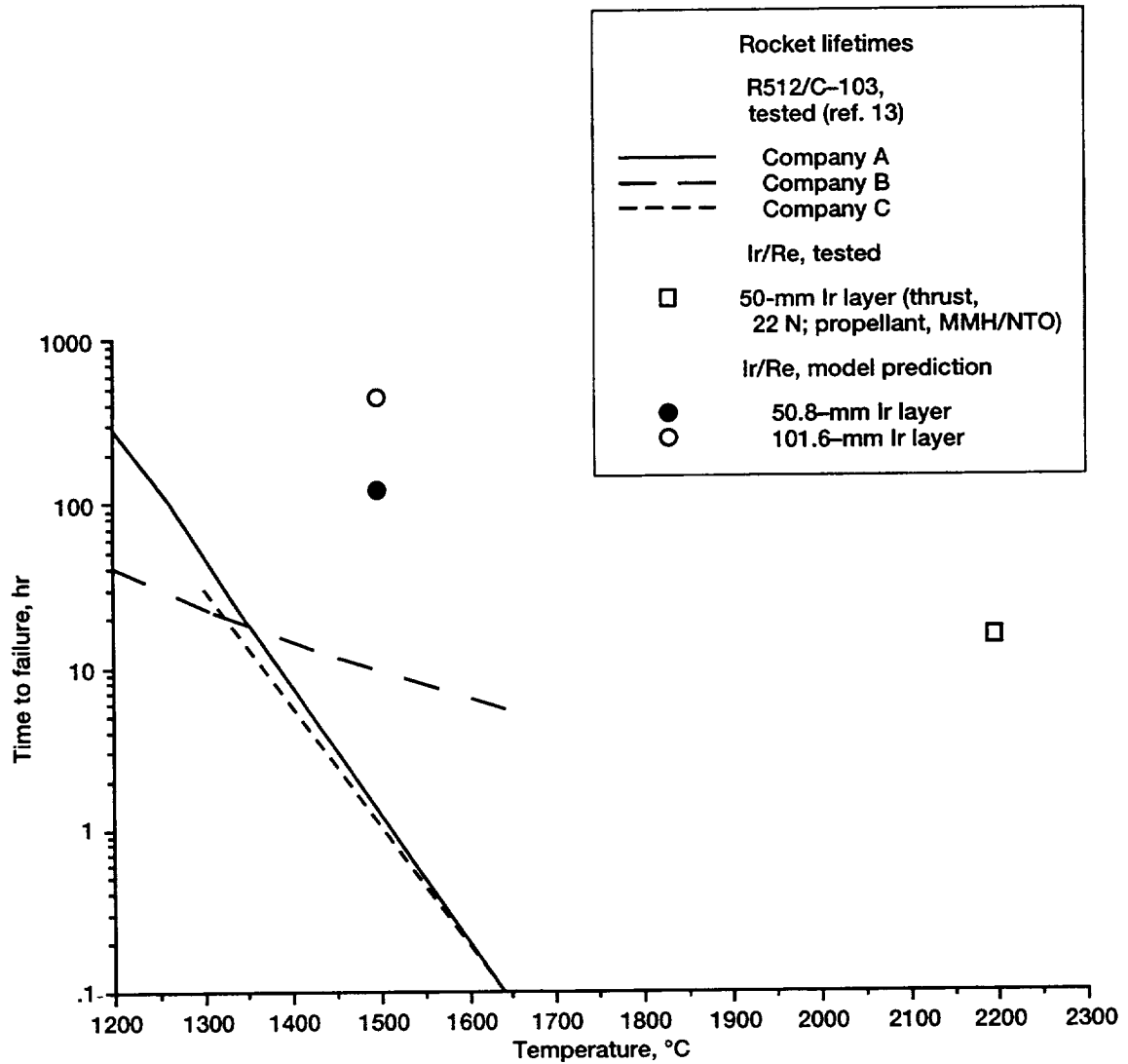Figure 4.—Furnace oxidation data taken from the first group of samples oxidized at 1500 °C.

Figure 5.—Chamber lifetime for R512/C–103 rockets and Ir/Re rockets as a function of temperature.

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13. ABSTRACT (Maximum 200 words)

The life-limiting mechanism for radiation-cooled rockets made from iridium-coated rhenium (Ir/Re) is the diffusion of Re into the Ir layer and the subsequent oxidation of the resulting Ir-Re alloy from the inner surface. In a previous study, a life model for Ir/Re rockets was developed. It incorporated Ir-Re diffusion and oxidation data to predict chamber lifetimes as a function of temperature and oxygen partial pressure. Oxidation testing at 1540 °C suggested that a 20-wt % Re concentration at the inner wall surface should be established as the failure criterion. The present study was performed to better define Ir-oxidation behavior as a function of Re concentration and to supplement the data base for the life model. Samples ranging from pure Ir to Ir-40 wt % Re (Ir-40Re) were tested at 1500 °C, in two different oxygen environments. There were indications that the oxidation rate of the Ir-Re alloy increased significantly when it went from a single-phase solid solution to a two-phase mixture, as was suggested in previous work. However, because of testing anomalies in this study, there were not enough dependable oxidation data to definitively raise the Ir/Re rocket failure criterion from 20-wt % Re to a Re concentration corresponding to entry into the two-phase region.

14. SUBJECT TERMS

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