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Advanced Liquid Rockets

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ADVANCED LIQUID ROCKETS

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ABSTRACT

A program to substitute iridium-coated rhenium for silicide-coated niobium in thrust chamber fabrications is reviewed. The life limiting phenomena in each of these material systems is also reviewed. Coating cracking and spalling is not a problem with iridium-coated rhenium as in silicide-coated niobium. Use of the new material system enables an 800 K increase in thruster operating temperature from around 1700 K for niobium to 2500 K for rhenium. Specific impulse of iridium-coated rhenium rockets is nominally 20 seconds higher than comparable niobium rockets in the 22 N class and nominally 10 seconds higher in the 440 N class.

INTRODUCTION

Liquid rockets are used for a variety of functions on many commercial, military and NASA space systems. Major roles include launch, orbit transfer, apogee, perigee, north-south stationkeeping, orbit control, drag makeup, and logistics (delivery and return). In many cases, it is the onboard propulsion which exerts a major influence on the characteristics of the space systems and their overall mission performance.[1] For example, several of the commercial geosynchronous (GEO) communication satellites have injected propulsion and payload mass fractions with onboard propulsion constituting greater than 50% of the injected mass, as shown in Figure 1. In another example, onboard propulsion system mass of the Space Transportation System Orbiter ranges from 13,000 kg to 18,000 kg, depending on Orbiter mission, as shown in Figure 2.[2] Technologies which advance the state-of-the-art of onboard propulsion, therefore, offer major potential mission leverage in terms of their impact on on-orbit payload mass and life.

The use of spacecraft onboard propulsion rather than an independent propulsion stage can also reduce cost and risk of the overall mission.[3] Major cost savings are achieved by eliminating the duplication of components and subsystems between independent propulsion stages. Low thrust onboard propulsion allows near-Earth deployment and checkout of spacecraft, operational flexibility during different missions phases, and a fail-safe approach to orbit transfer. Low thrust transfer has no great mission timeline impact in that it only increases the transfer time from approximately 6-8 hours to 2-5 days, depending on the rocket used for the transfer process. In a low-thrust ascent to GEO or some other high-energy orbit, orbit transfer is accomplished by a sequence of typically 10 to 30 extended burns centered on perigee, followed by a series of 1 to 3 apogee burns. Orbit transfer risk is reduced,[3] not by more reliable components in the low thrust process, but by fewer components and fewer nonredundant, critical components.

This paper reviews a basic research program in small liquid rocket technology. The materials technology which enables the fabrication of uncooled thrust chambers is reviewed first, followed by small rocket test results using these materials.

MATERIALS

Silicide-coated Niobium

Nearly every film and radiation cooled liquid rocket thrust chamber and exit nozzle presently is fabricated from niobium (C-103) with a fused silica coating (R-512A or R-512E) for oxidation protection. The life of the coating is limited by two modes of degradation. The first and most understood mode is the loss of the coating due to diffusion and vaporization of the material. This mode can be evaluated by available analytical techniques based on diffusion and vapor pressure relationships. The second mode of degradation is the result of differences in the coefficient of thermal expansion of the C-103 base material and the R-512 coating. The repeated cycling of the material system between room temperature and elevated temperature (1400 -1900 K) results in coating cracking and eventual spalling. In addition, the cracks formed result in substrate oxidation and this can result in additional spalling of the coating and eventual exposure of the C-103 to the combustion gases.

No definitive analytical technique has been found which can predict the onset of coating failure based on a combination of "time at elevated temperature" and number of thermal cycles. The recommended life limits (time at elevated temperature) used by several rocket manufacturers is given in Figure 3. These data are based on torch tests of materials and qualification tests of rocket engines conducted by these companies. Note a significant variability in recommended life, but a general agreement that there is 10 to 15 hours of life at 1640 K. One manufacturer also reports that life may be dependent on thruster size.

For cyclic applications, manufacturer recommended life limits are expressed as the number of full thermal cycles versus peak cycle temperature, as given in Figure 4. These data indicate that for high engine temperature, this coating cannot withstand many (100-1000) thermal cycles without failing. For mixed steady state and pulse duty operation, manufacturers recommend addition of the steady state and cyclic components of life. For example, using the most conservative data, a thruster life is defined as operation at 1670 K for 3 hours (50% of life) plus 500 cycles in pulse duty at 1470 K (50% of life).

Iridium-coated Rhenium

Rhenium coated with iridium for oxidation protection is the thrust chamber material chosen for development under this program.[4,5] This selection was made following a literature and vendor survey of potential materials with capabilities of operating at temperatures as high as 2470 K in an oxidizing rocket engine environment.

Refractory metals, ceramics, composites, and carbon-carbon materials were evaluated for substrate materials. Platinum group metals, Engle-Brewer compounds and ceramics were considered for oxidation resistant coatings. Iridium-rhodium-rhenium alloys and ceramic/metal (CERMET) alloys were considered as monolithic materials. Many candidate materials were available, but most had very little information available about their fundamental properties of interest such as strength, shock resistance, and oxidation resistance. In addition, some very promising materials required extensive development of their fabrication technologies.

Rhenium (Re) was selected as the substrate material because of its high melting point (3400 K) excellent strength at high temperature and absence of a ductile-to-brittle transition common in other refractory metals. Iridium (Ir) was chosen as the oxidation resistant coating for rhenium because of its adequate melting temperature (2720 K), good oxidation resistance (3 orders of magnitude better than Re), close coefficient of thermal expansion to rhenium, adherence to rhenium, and ductility.

These materials were first fabricated for the Air Force Rocket Propulsion Laboratory[6,7] in the form of 3 mm diameter tubes by the Chemical Vapor Deposition (CVD) technique. Oxidation tests were performed by induction heating of the samples in air to 2270 K for over 20 minutes. The iridium coating, however, was porous and did not afford the requisite oxidation protection to the underlying rhenium. Further development[8] of the iridium CVD process has yielded uniform, non-porous coatings that offered excellent oxidation protection to the rhenium. Small 22 N rocket chambers were fabricated to evaluate this material. They were mated to a water cooled injector and fired with nitrogen tetroxide/monomethylhydrazine (N_2O_4/MMH) propellants as a material demonstrator. Over fifteen hours[4,9] of operation and 2684 thermal cycles at temperatures around 2500 K were demonstrated without failure. A summary of test time versus mixture ratio is given in Table I. Following these tests, the measured throat diameter was only about 0.01 mm larger and chamber weight loss was less than 1%.

In order to understand the performance limits of Ir-coated Re thrusters, an effort[5,10] was undertaken at the Sandia Combustion Research Facility to measure interdiffusion and oxidation kinetics of Ir-coated Re. Gas phase measurements were obtained near the surface of heated samples in an atmospheric pressure air/H_2 flame by laser-induced fluorescence.[10,11] Hydroxyl radical (OH) measurements significantly above equilibrium were measured for almost all test conditions and no difference in profiles near iridium or platinum samples was observed. This suggests that the OH concentrations near the surface are determined primarily by a radical recombination process in the post-flame gases, or else these surfaces have the same reactivities.

Surface reaction phenomena on samples heated in a furnace were examined using Raman spectroscopy, Auger spectroscopy, and x-ray diffraction.[10,12,13] Analysis of samples shows that Ir is attacked and etched by oxygen by the formation and desorption of IrO_2. An Ir recession rate of 0.15 micrometers/hr was measured[4] by thermogravimetric analysis (TGA) at 1810 K in Ar+0.5%O_2 at 190 Pa. Ir-Re interdiffusion was examined by annealing Ir-coated Re samples in a vacuum furnace at temperatures between 1670 K and 2170 K. The samples were cross-sectioned and polished and electron microprobe analysis was used to determine the distribution of Re and Ir in these annealed coatings. Re was observed to diffuse preferentially along grain boundaries into the Ir coating with very little diffusion of Ir into the Re. Diffusion constants were obtained by a model of diffusion into a semi-infinite medium where the boundary was held at constant concentration. Measured diffusion constants are given in Figure 5. They have an Arrhenius dependence with an activation energy for diffusion of 1.23 eV. This activation energy is well below that expected for bulk diffusion, suggesting that grain boundary diffusion is the dominant diffusion mechanism.

Failure of the Ir-coated Re material system was therefore projected to occur by diffusion of the Re through the Ir, followed by subsequent oxidation and removal at the Ir surface. Thermogravimetric analysis[4] (TGA) was used to measure the oxidation rates of Ir and Ir-Re alloys. Rapid oxidation of specimens with greater than 20 atomic percent Re was observed. This suggests imminent failure when the Re concentration at the surface reaches 20 atomic percent. A life limit model[14] was then developed, as shown in Figure 6. The life model has functional dependence on operating temperature, Ir-thickness and surface recession rate.

Efforts are underway[15] to develop enhanced oxidation protection for Ir coated Re engines. A combination thermal/diffusive barrier using oxide coatings was chosen for development. The prime candidates are hafnia (HfO_2), zirconia (ZrO_2), and yttria (Y_2O_3).

Other Materials

Another material system of high interest is mixed hafnium carbide (HfC) and tantalum carbide (TaC) ceramic composite reinforced with graphite fibers. HfC, TaC, and graphite are among the highest melting point materials known (4200 K, 4150 K, and 3800 K, respectively). These temperatures exceed the flame temperature of most propellants and could enable uncooled operation of hydrogen/oxygen rocket chambers. Mixed HfC/TaC coating on graphite fibers were successfully formed and oxidized[16] into protective oxide layers of HfO_2/Ta_2O_5. Some degree of stabilization of the HfO_2 was observed by the inclusion of the Ta_2O_5. The melting point of HfO_2 (3110 K) limits the operating temperatures in oxidizing environments. This temperature is well above the melting point of Ir, however, some cooling of rocket chambers fabricated with these materials may be required. Compositional variations of HfC/TaC are to be examined in order to determine that which provides the most adherent oxide coatings.

THRUSTER RESULTS

Design and Fabrication

The design and fabrication of rockets using Ir-coated Re materials requires knowledge of materials properties and metallurgical joining technologies. Much of the basic work on measurement of properties was conducted in the 1960's and 1970's. This work was reviewed,[4,17] but sources were reported to have considerable variability. The method of fabrication of the Re was one uncertainty. A comparison of the high temperature creep and tensile properties of rhenium fabricated by arc cast, CVD, and powder metallurgy was recently conducted.[18] The results of testing indicate that the creep-rupture properties of CVD rhenium are similar to those of powder metallurgy rhenium. An investigation of metallurgical joining techniques of rhenium to dissimilar metals[4] produced furnace brazing with Palcusil 25 or Nioro (BAU-4) and a form of electron beam (EB) welding called parent metal braze as suitable joining techniques.

The high operating temperature of Ir-coated Re (2500 K) allows the elimination of fuel film cooling and its associated combustion/performance losses in thrusters. Small rockets require larger percentages of their fuel for cooling and, therefore, benefit the most from this high temperature material technology. Thruster design issues which arise from the use of these materials include thermal management of the injector-chamber interface and design for adequate fatigue strength during launch. Thermal management can be accomplished by the use of fuel film coolant, mixture ratio control near the wall, or injector regenerative cooling. Design for adequate fatigue life can be accomplished by the use of lighter weight materials, such as silicide coated niobium, for nozzle skirts where temperatures do not require rhenium and/or by providing adequate throat thickness.

<u>22 N Rocket</u>

Results from performance and life testing of a 22 N rocket design with N_2O_4/MMH propellants were reported first.[4,19] The thruster was designed with a 150:1 area ratio nozzle for the direct comparison of performance of the Ir-coated Re engine with that of a flight qualified niobium engine of 690 kPa chamber pressure. Heat transfer to the injector due to soakback from the Ir-coated Re chamber was managed by using 30 to 40% fuel film cooling along with a patented staged combustion device to mix the film with the core flow further downstream such that the Ir-coated Re chamber runs essentially uncooled. This mixing of the film with the core flow resulted in a significant increase in combustion efficiency with no modification of the injector. A high emissivity dendritic rhenium surface on the outside of the chamber enhanced radiation heat transfer from the chamber. The measured vacuum specific impulse for this engine was 313 seconds at a mixture ratio of 1.66. This is nominally 20 seconds higher than that obtained with the comparable niobium chamber. During these tests, the maximum wall temperatures were around 2200 K. The high emissivity external surface reduced observed temperatures by about 250 degrees below those of the material demonstrator. Duty cycle tests ranging from 10 to 90 percent on-time with each pulse being 0.050 seconds in duration were also conducted. Over 100,000 pulses were accumulated on the chamber. Based on temperature rise data, duty cycles of about 60-70% would have exceeded injector or valve temperature limits of 480 and 380 K, respectively. A rocket test summary of the 1.77 hrs of operation is given as a function of mixture ratio in Table II. Inspection of the chamber after these tests revealed an Ir coating failure at the throat. This failure occurred at a sharp expansion in the flow (0.8 mm axial radius of curvature of the nozzle contour). For comparison, the successful material demonstrator chamber had a milder expansion with a 7.6 mm axial radius of curvature. Stress analyses indicated that the combination of small radius of curvature and high axial temperature gradient contributed to the coating failure.

A further series of tests was also conducted to determine thermal behavior with an expanded operating envelope of chamber pressure from 590 to 1100 kPa, 320 K propellants, and mixture ratios of 1.65 and 1.90. The more benign, low chamber pressure, low duty cycle tests were successfully completed, but other tests led to overheating of the injector/valves. Additional thermal design of this thruster is, therefore, required for this operating envelope.

<u>440 N Rocket</u>

Following the successful material demonstration, the Jet Propulsion Laboratory undertook to demonstrate a 440 N thruster[20,21] on N_2O_4/MMH propellants at a nominal chamber pressure of 6900 kPa. Fuel regenerative cooling of the injector was employed to manage the soakback heat transfer and no high emissivity dendritic surface was employed. A total firing time of 4.2 hours and 33 cycles were accumulated on one of the chambers with temperatures ranging from 2100 to 2200 K. A performance of 292 seconds was measured at a mixture ratio of 1.65 and an area ratio of 22:1. This performance is about a 10 seconds higher than a similar Nb engine. A summary of these tests is given in Table III as a function of mixture ratio. This fabrication required a significant scale up of the CVD chamber fabrication technology and issues involving low deposition rates for the iridium coating resulted in local blistering of the coating. A metallurgical investigation of the coating revealed contamination sandwiched between the multiple layers of iridium in the 50 micrometer thick coating.

A program was then undertaken to improve the CVD iridium deposition process.[22] The deposition rate was improved by taking advantage of natural convection flows within the CVD chamber to deliver more precursor material to the surface of the mandrel. In addition, a fluidized bed evaporator was developed to enable a continuous feed of evaporated iridium precursor material. Continuous iridium deposition at about 15 μm/hr then enabled the elimination of the previously experienced contamination in the scaled up CVD process by depositing the coating in one continuous deposition run.

62 N Rocket

This improved CVD fabrication technology was first demonstrated on a 62 N chamber with N_2O_4/MMH propellants at a nominal chamber pressure of 6900 kPa and a 75:1 area ratio.[5,23,24] This chamber was chosen to demonstrate that iridium-coated rhenium chambers could be retrofited on existing Nb rockets without modification of the injector. Soakback heat transfer to the injector was managed by the fuel film cooling along with a patented staged combustion device to mix the film with the core flow such that the Ir-coated Re chamber runs essentially uncooled. This mixing of the film with the core flow resulted in a significant performance increase with no modification of the injector. The high emissivity dendritic rhenium coating was used to lower chamber temperatures. A total firing time of 600 sec and 263 cycles were accumulated on one of the chambers with no degradation. Chamber temperatures ranged from 2050 to 2150 K. Measured specific impulse was 305 seconds at a mixture ratio of 1.65. This compares to 286 seconds for the flight qualified Nb design yielding a 19 second performance increase. Thruster performance was successfully demonstrated over the flight qualification inlet pressure, mixture ratio operating envelope. When comparing the Re chamber with a Nb chamber, post test chamber discoloration streaks suggest more complete combustion and less plume contamination with the Re chamber. The final hurdle for this chamber was an acceptance test to determine whether the material and design had the requisite fatigue properties to survive the launch vibration environment. This test was successfully passed although analyses indicate the design is marginal. Increasing the material thickness at the throat alleviates this concern and a demonstration of this fabrication technology is needed.

440 and 550 N Rockets

The improved CVD fabrication technology was also demonstrated on 440 N and 550 N chambers. Performance and durability tests are underway on the 440 N engine[5] with N_2O_4/MMH propellants. Performance achieved on the prior program[20,21] was duplicated at an area ratio of 22:1. Also, performance data was obtained on a high area ratio (286:1) flight type engine. Preliminary evaluation of the data indicates a nominal vacuum specific impulse of 319 seconds, with data ranging from 318 to 321 seconds. An effort is underway to prequalify this flight type thruster to a planetary program operating envelope. Analyses indicate, however, that significantly thicker throats are required to survive the launch vibration environment.

Preliminary test results from a second thruster manufacturer with the 550 N chamber on N_2O_4/N_2H_4 propellants were obtained.[25] Vacuum specific impulses in the 326 to 328 second range are estimated at an area ratio of 204:1. A combustion efficiency in excess of 98% theoretical was achieved with rhenium chamber temperatures below 1900 K. Further injector optimization is projected to yield a specific impulse of 330 seconds.

SUMMARY

Recommended life limits of silicide-coated Nb thrust chambers used by several manufacturers of state-of-the-art thrusters were given as a function of operating temperature and duty cycle. Significant variability in recommended life was noted along with a general agreement that there was about 10 to 15 hours of life at 1640 K. For cyclic applications, a limit of between 100 and 1000 thermal cycles was indicated, dependent on operating temperature. A materials technology program to fabricate and substitute iridium-coated rhenium thrust chambers was reviewed. These new materials enable the fabrication of uncooled thrust chambers with significant performance increases due to the elimination of fuel film cooling. Over fifteen hours of operation and 2684 cycles at operating temperatures of 2500 K were demonstrated on these materials without failure with N_2H_4/MMH propellants. The life limiting process in iridium-coated rhenium material was evaluated from fundamental measurements. The process of failure is described as the diffusion of rhenium through the iridium coating until an alloy composition in excess of 20% Re occurs on the surface which results in catastrophic material loss to oxidation.

The design and fabrication of rockets using iridium-coated rhenium materials was outlined and the results of four different rocket test programs with these materials were reviewed. Rockets in the thrust classes of 22 N, 62 N, 440 N, and 550 N were tested by two different contractors with excellent results. Performance of iridium-coated rhenium rockets is nominally 20 seconds higher than comparable niobium rockets in the 22 N class and nominally 10 seconds higher in the 440 N class. Design and fabrication of chambers to survive the launch vibration environment is underway.

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MR	Max Temp (K)	No of Thermal Cycles	Duration (sec)
1.45		2	61
1.50		3	70
1.55	-	2	10
1.60	-	2	2705
1.65	2478	581	31457
1.70	2511	571	15856
1.75	-	1	2
1.80	2496	503	2210
1.85	-	2	10
1.90	2513	551	1105
2.00	2519	366	738
2.05	2519	100	200
Totals		2684	54430

Table I 22 N Iridium Coated Rhenium Material Demonstrator Rocket Test Summary

MR	Max Temp (K)	Max Specific Impulse (sec)	Duty Cycle	No of Pulses	Duration (sec)
1.59	2209	307	Steady	1	300
1.61	2156	304	Steady	1	5
1.62	2200	301	Steady	1	90
1.63	2229	310	Steady	1	350
1.64	2214	314	10%	2	95
1.64	1689	-	20%	1000	50
1.64	1700	-	40%	86,800	4340
1.64	1906	-	50%	1800	90
1.64	1972	-	60%	1400	70
1.64	2047	-	70%	2855	143
1.64	2094	-	80%	1706	85
1.64	2128	-	90%	2240	112
1.64	2153	-	Steady	2500	125
1.65	2237	311	Steady	1	90
1.66	2246	313	Steady	1	319
1.68	2259	318	Steady	2	110
Totals				100,311	6374

TABLE II 22 N Iridium-Coated Rhenium Rocket Test Summary at 150:1 Area Ratio

MR	Max Temp (K)	Max Vacuum Specific Impulse (sec)	No of Cycles	Duration (sec)
1.54	-	290	1	15
1.60	-	289	3	609
1.62	2100	292	3	1927
1.63	2123	291	2	1310
1.64	2094	292	4	2950
1.65	2222	292	8	4102
1.66	2169	293	5	3000
1.67	2144	293	6	1052
1.69	2144	294	1	35
Totals			33	15,000

TABLE III 440 N Iridium-Coated Rhenium Rocket Test Summary at 22:1 Area Ratio

MR	Max Temp (K)	Max Vacuum Specific Impulse (sec)	Duty Cycle	No of Pulses	Duration (sec)
1.41	-	300	Steady	1	20
1.54	2056	300	Steady	1	15
1.54	-	-	10%	80	8
1.57	2116	306	Steady	3	41
1.59	2113	303	Steady	2	25
1.60	2117	305	Steady	1	20
1.62	-	-	Steady	1	5
1.63	2128	301	Steady	2	15
1.63	2194	-	10%	80	8
1.64	2186	306	Steady	7	280
1.64	-	-	10%	80	8
1.65	2155	305	Steady	1	10
1.66	2182	305	Steady	2	115
1.68	2159	305	Steady	1	10
1.82	-	303	Steady	1	20
Totals				263	600

TABLE IV 62 N Iridium-Coated Rhenium Rocket Test Summary at 75:1 Area Ratio

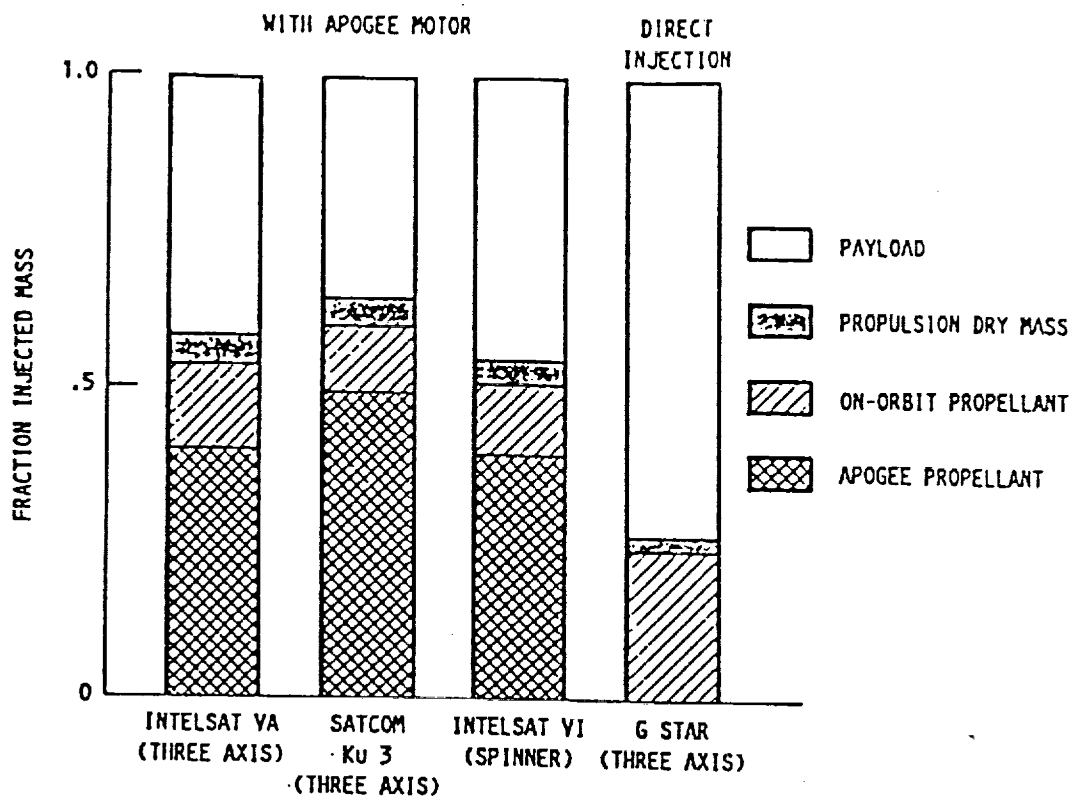

FIGURE 1. INJECTED PROPULSION AND PAYLOAD MASS FRACTIONS FOR GEOSYNCHRONOUS SATELLITES

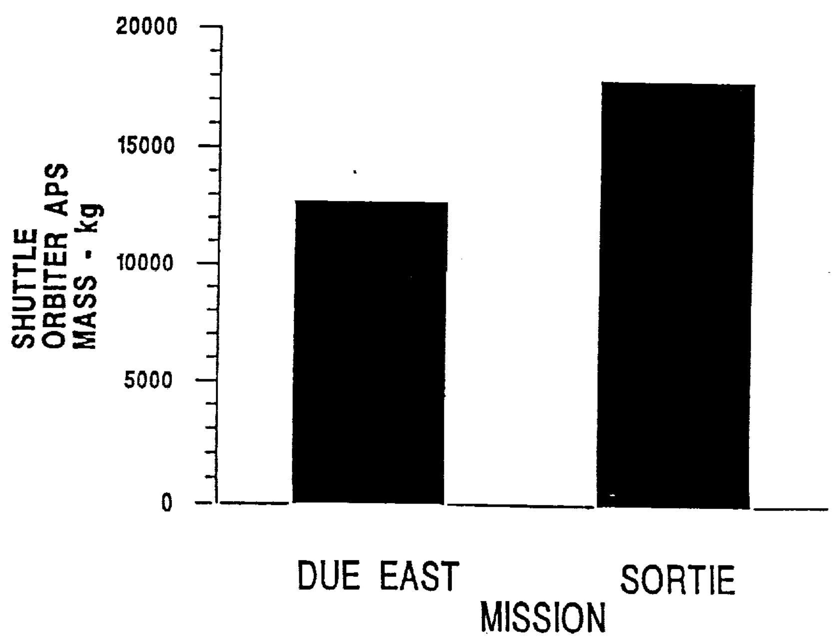

FIGURE 2. STS ORBITER ONBOARD PROPULSION SYSTEM MASS

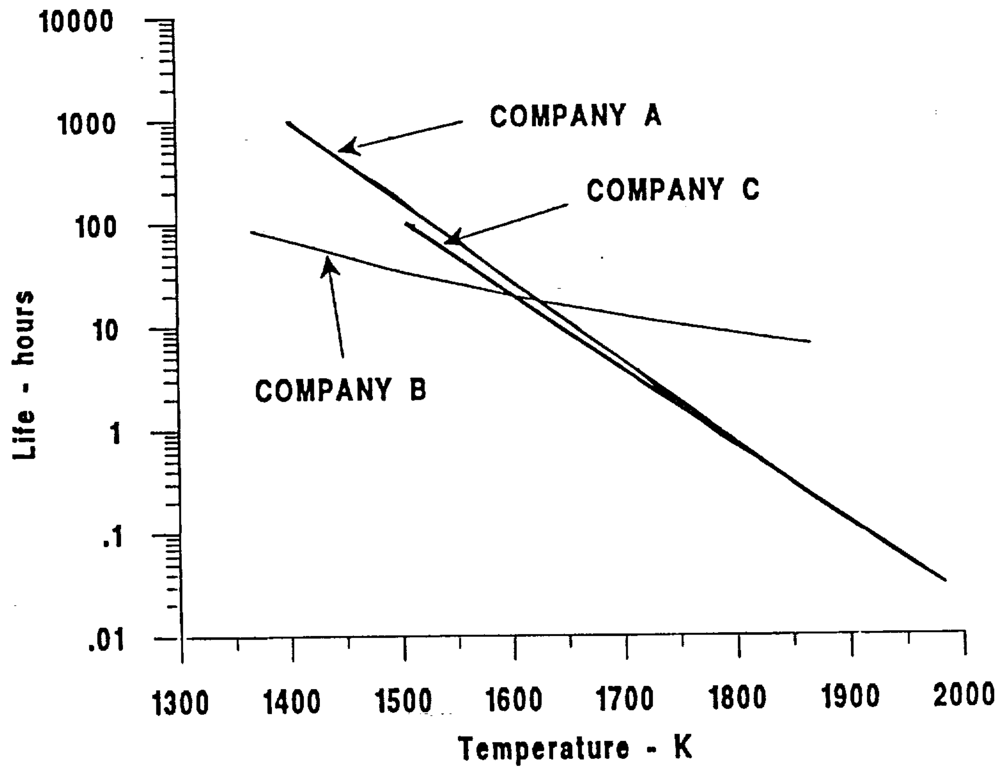

FIGURE 3. RECOMMENDED LIFE LIMITS OF SILICIDE COATED Nb USED BY SEVERAL ROCKET MANUFACTURERS

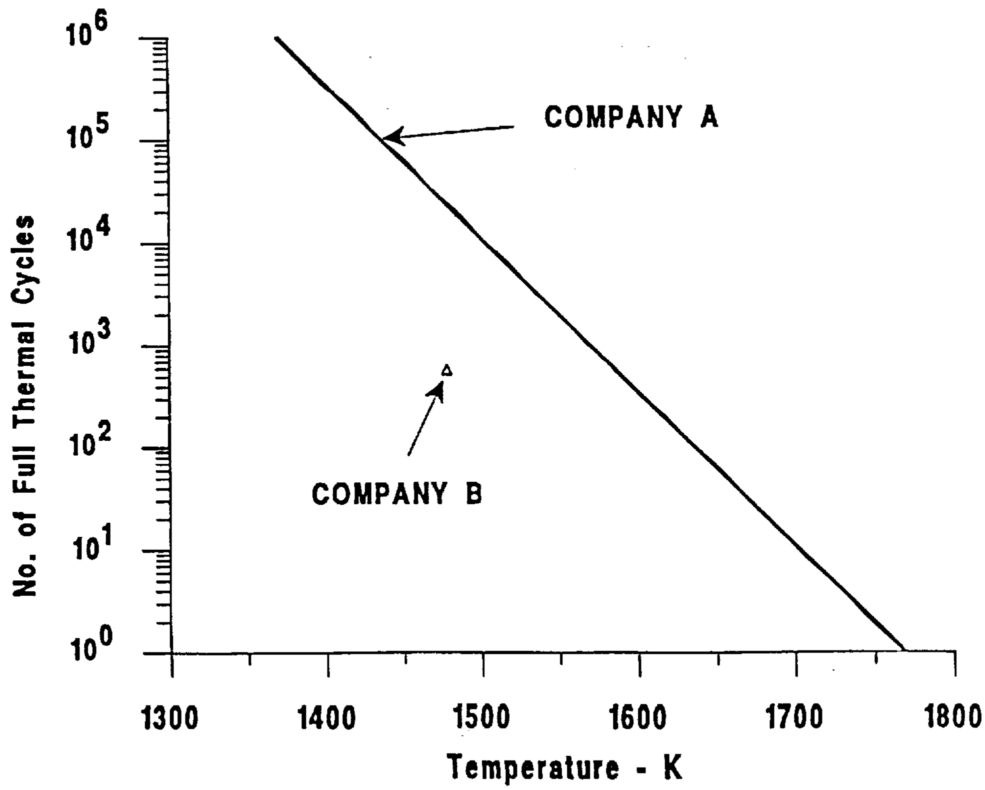

FIGURE 4. RECOMMENDED NUMBER OF FULL THERMAL CYCLES VS PEAK CYCLE TEMPERATURE FOR SILICIDE COATED Nb USED BY SEVERAL ROCKET MANUFACTURERS

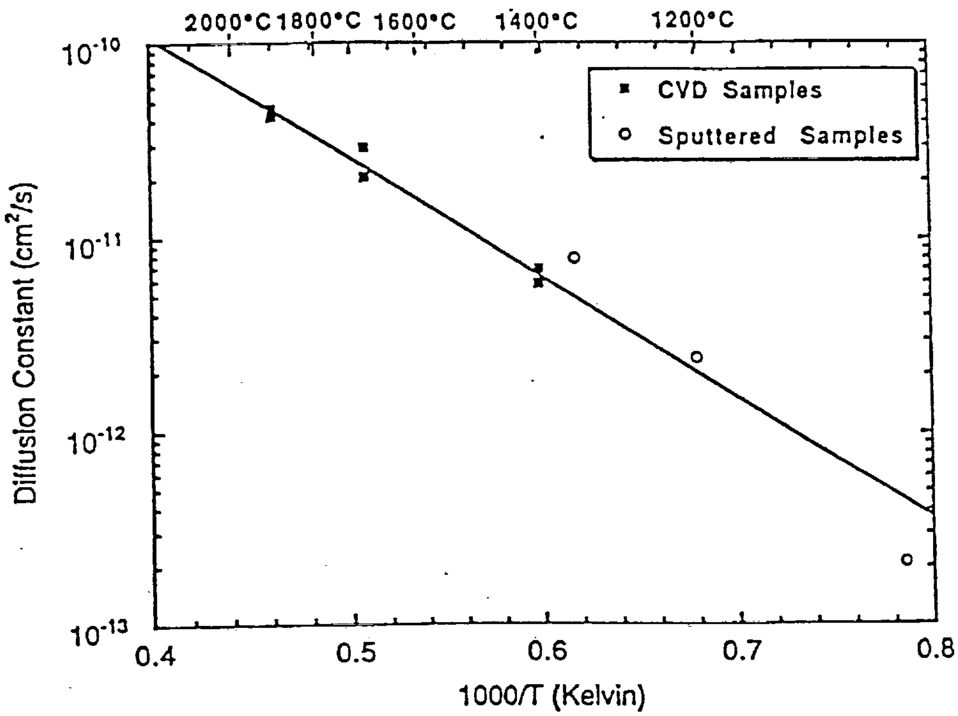

FIGURE 5. MEASURED DIFFUSION CONSTANTS OF IRIDIUM-RHENIUM COUPLES SHOWING ARRHENIUS DEPENDENCE (REFERENCE 13)

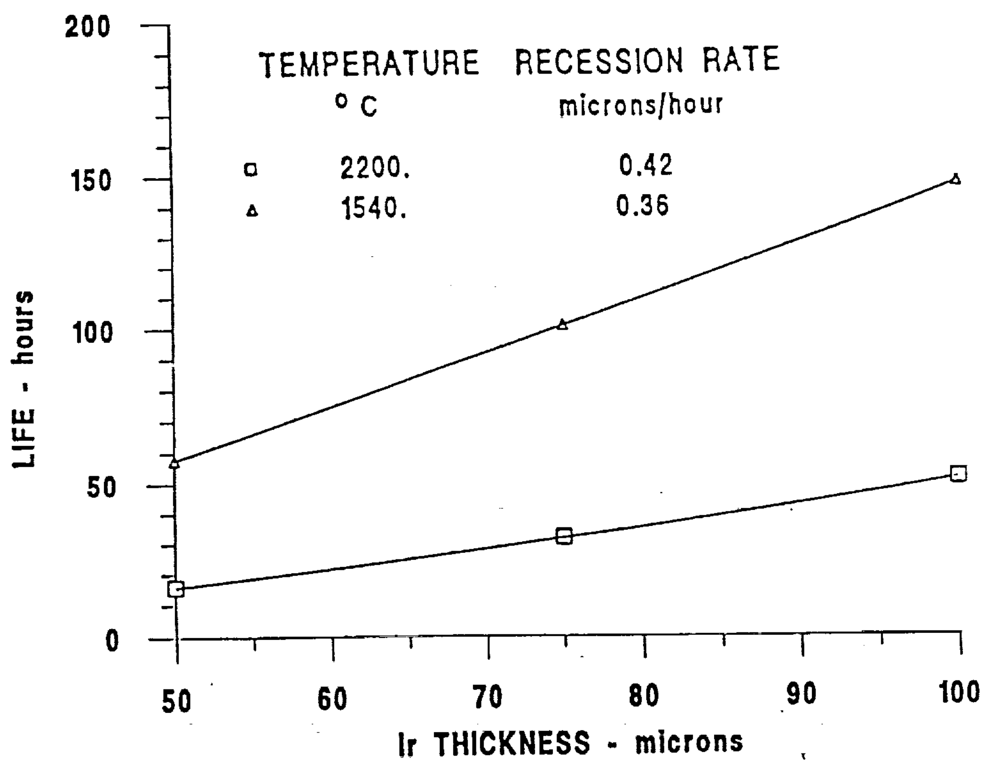

FIGURE 6. LIFE PREDICITON MODEL FOR IRIDIUM COATED RHENIUM THRUSTERS

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13. ABSTRACT ***(Maximum 200 words)***

A program to substitute iridium-coated rhenium for silicide-coated niobium in thrust chamber fabrications is reviewed. The life limiting phenomena in each of these material systems is also reviewed. Coating cracking and spalling is not a problem with iridium-coated rhenium as in silicide-coated niobium. Use of the new material system enables an 800 K increase in thruster operating temperature from around 1700 K for niobium to 2500 K for rhenium. Specific impulse of iridium-coated rhenium rockets is nominally 20 seconds higher than comparable niobium rockets in the 22 N class and nominally 10 seconds higher in the 440 N class.

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