

Testing of Electroformed Deposited Iridium/Powder Metallurgy Rhenium Rockets

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SUMMARY

High-temperature, oxidation-resistant chamber materials offer the thermal margin for high performance and extended lifetimes for radiation-cooled rockets. Rhenium (Re) coated with iridium (Ir) allow hours of operation at 2200 °C on Earth-storable propellants. One process for manufacturing Ir/Re rocket chambers is the fabrication of Re substrates by powder metallurgy (PM) and the application of Ir coatings by using electroformed deposition (ED). ED Ir coatings, however, have been found to be porous and poorly adherent. The integrity of ED Ir coatings could be improved by densification after the electroforming process. This report summarizes the testing of two 22-N, ED Ir/PM Re rocket chambers that were subjected to post-deposition treatments in an effort to densify the Ir coating. One chamber was vacuum annealed, while the other chamber was subjected to hot isostatic pressure (HIP). The chambers were tested on gaseous oxygen/gaseous hydrogen propellants, at mixture ratios that simulated the oxidizing environments of Earth-storable propellants. The annealed ED Ir/PM Re chamber was tested for a total of 24 firings and 4.58 hr at a mixture ratio of 4.2. After only 9 firings, the annealed ED Ir coating began to blister and spall upstream of the throat. The blistering and spalling were similar to what had been experienced with unannealed, as-deposited ED Ir coatings. The HIP ED Ir/PM Re chamber was tested for a total of 91 firings and 11.45 hr at mixture ratios of 3.2 and 4.2. The HIP ED Ir coating remained adherent to the Re substrate throughout testing; there were no visible signs of coating degradation. Metallography revealed, however, thinning of the HIP Ir coating and occasional pores in the Re layer upstream of the throat. Pinholes in the Ir coating may have provided a path for oxidation of the Re substrate at these locations. The HIP ED Ir coating proved to be more effective than vacuum annealed and as-deposited ED Ir. Further densification is still required to match the integrity of chemically vapor deposited Ir coatings. Despite this, the successful long duration testing of the HIP ED Ir chamber, in an oxidizing environment comparable to Earth-storable propellants, demonstrated the viability of this Ir/Re rocket fabrication process.

INTRODUCTION

The material system that is currently used for radiation-cooled rockets, a niobium alloy (C103) with a fused silica coating, has an operating temperature of 1370 °C. Rockets composed of a rhenium (Re) substrate and an iridium (Ir) coating have demonstrated long lifetimes (over 10 hr) at 2200 °C operating temperature (ref. 1). The added thermal margin afforded by Ir/Re rockets allows the reduction or elimination of the large amounts of fuel film cooling required in C103 rockets. As a result, Ir/Re rockets have demonstrated greatly improved performance and reduced contamination over C103 rockets while still offering long chamber lifetimes (refs. 2 and 3).

The established process for fabricating Ir/Re rockets is chemical vapor deposition (CVD) (ref. 4). In the CVD process, a gaseous precursor of the material to be deposited is flowed over a heated mandrel that conforms to the inner contour of the chamber. The gas is thermally decomposed on the mandrel surface, depositing the material. In this manner, first Ir and then Re layers are deposited on the mandrel. After CVD processing, the mandrel is chemically removed leaving the Ir/Re chamber. Pure, dense, and adherent coatings of Ir have been successfully deposited using CVD. The CVD fabrication process has been scaled from 22- to 440-N class chambers.

An alternative fabrication process for Ir/Re chamber is to use powder metallurgy (PM) for the Re substrate followed by the electroformed deposition (ED) of the Ir. The PM process is a relatively low-cost and repeatable process for fabricating Re chambers. Thick, dense ED Ir coatings have been difficult to obtain, however, and have suffered from porosity and poor adherence (ref. 5). These coatings tend to blister and spall off the Re substrate when subjected to thermal cycling (ref. 6). It is possible that the integrity of the ED Ir could be improved by densification of the Ir layer through a post-deposition treatment process. Annealing could effectively sinter the ED Ir coating, closing off porosity. Application of high pressure and temperature through a hot isostatic pressure (HIP) treatment would consolidate the ED coating and perhaps provide a metallurgical bond between the Ir and Re layers.

Two 22-N class rocket chambers were fabricated using the ED Ir/PM Re process. Each chamber was subjected to a different post-fabrication treatment in an effort to densify the ED Ir coating. One chamber, procured directly by NASA Lewis, was vacuum annealed for 30 min at 1480 °C. The other chamber, procured under a technology program with TRW (Space Storable Rocket Technology, Option 3, Final Report, Contract NAS3-26246, to be published) was subjected to a hot isostatic pressure (HIP) treatment. Both rocket chambers were tested on gaseous oxygen/gaseous hydrogen (GO_2/GH_2) propellants at mixture ratios (MR's) of 3 to 4. The low MR GO_2/GH_2 combustion environment has an oxygen and water vapor content comparable to Earth-storable propellant, although GO_2/GH_2 is higher temperature. This report summarizes the testing and post-test examination of the chambers.

ROCKET FABRICATION PROCESS

Rocket Chamber Geometry

The rocket chambers used in the testing described herein had the nominal geometry shown in figure 1. This chamber geometry had been used by NASA as a standard for screening advanced chamber materials (refs. 1 and 7). The barrel had an inner diameter of 0.894 cm and a length of 3.5 cm. The contraction ratio was 4.3:1 with a gradual converging contour to the throat. The throat inner diameter was 0.432 cm. The nozzle was an 8:1 area ratio, 15° half-angle cone. A 37° angle cone at the head end was used for attachment to the injector assembly. The rocket was sized for mass flowrates corresponding to a 22-N thrust level.

Rhenium (Re) Substrate Fabrication

The PM Re chambers for this program were fabricated by Rhenium Alloys, Inc. In the PM process, high purity Re powder is pressed and sintered, producing 95 to 97 percent dense Re ingots. The density of the pressed and sintered Re can be increased above 99 percent by cold working it into a rolled sheet or subjecting it to HIP treatment. Samples of pressed and sintered, rolled sheet, and HIP PM Re samples, along with CVD Re samples were previously evaluated in tensile, low-cycle fatigue, and creep testing (ref. 8). All of the PM Re samples had strength levels comparable to or better than the CVD Re samples. For the chambers fabricated in this program, the PM Re ingots were HIP and found to have a density of at least 99 percent. After the HIP process, the ingots were electrically discharge machined and ground into the final configuration. The nominal thickness for the Re substrates was 0.125 cm.

Application and Densification of Iridium Coating

The ED Ir coatings for both chambers were applied by Electroformed Nickel, Inc. In the ED process, a plating bath, composed of either an aqueous or molten salt solution, acts as an electrolytic cell. The workpiece is immersed in the bath to serve at the cathode, while the anode is composed of the material to be coated on the workpiece. Passing current through the anode transfers ions of the coating to the workpiece. To date, the only thick Ir coatings that have been successfully deposited have used molten salt plating baths (ref. 5). The nominal thickness of the Ir coatings for both Re chambers in this program was 100 μm . The surface of the ED Ir coatings were fairly rough. Densification of the Ir coating was conducted in two different ways for the two chambers. One chamber was vacuum annealed at 1480 °C for 30 min. Mass and hardness measurements showed no changes as a result of the annealing process. The other chamber was subjected to a HIP treatment. The HIP process details and parameters are proprietary to TRW.

High Emissivity Exterior Coating

A hafnia coating was plasma-sprayed to the exterior of the HIP ED Ir/PM Re chamber for high emissivity. A high emissivity coating on the exterior provides better heat rejection from the chamber. The HIP ED Ir/PM Re chamber, after application of the hafnia coating and before rocket testing, is shown in figure 2. No high emissivity coating was applied to the exterior of the annealed ED Ir/PM Re chamber.

TEST APPARATUS

Injector

The chambers were attached to an assembly consisting of a water-cooled transition section and injector, as shown in figure 3. The injector body had a center annulus for a spark plug. Oxygen was injected radially in the center annulus, where it was energized by the spark plug. A small amount of hydrogen was injected radially downstream of the spark plug to ignite the energized oxygen. Six impinging elements injected hydrogen into the center annulus flow, while six elements injected hydrogen axially into the chamber for fuel-film cooling. The 5.08-cm-long, water-cooled section had a trip ring 2.54 cm downstream of the injector face to promote mixing of the fuel film into the core flow and elimination of streaking. The water-cooled section also protected the front end from thermal soakback from the chamber. The conical head end of the chamber was clamped to a cone on the face of the water-cooled section by a split ring and sealed using a flexible graphite gasket material.

Purge Assembly

The exterior surface was purged with an inert gas to prevent the oxidation of the exterior Re. A 24-element ring was used for injection of the purge gas around the chamber exterior. The purge was contained in a 5.20-cm diameter fused silica tube, with a 2.54-cm inner diameter, stainless steel end cap. Springs were used to hold the tube to the purge ring, while allowing some movement due to thermal growth. The purge gas was either helium (~71 lpm) or argon (~47 lpm), based on availability of the gas. An oxygen absorbing purifier was used in the purge line to ensure that the purge gas had less than 0.1 ppm oxygen. The exterior purge was initiated prior to the first test in each series and maintained until after each series was completed and ambient pressure had been reestablished in the test tank (by which time the chamber had cooled to ambient temperature). An analysis was performed to show that the purging had a negligible effect on cooling the chamber.

Instrumentation

A pressure tap in the injector face was used to measure the static chamber pressure (P_c). This static P_c measurement was corrected for momentum pressure loss across the combustion zone and converted to total pressure. Hydrogen and oxygen mass flowrates were calculated using the inlet pressures, inlet temperatures, and discharge coefficients of critical flow venturis, with corrections for real gas effects (ref. 9). The measurement uncertainties of the mass flowrates and MR were related from measured parameters by using a Taylor series expansion according to the JANNAF recommended procedure (ref. 10). The measurement uncertainties were less than ± 2 percent for all of the mass flowrates and MR's.

Outer wall temperature of the chamber was measured using an Ircon Modline Plus two-color pyrometer. The two-color pyrometer viewed two wavelength bands and used the ratio of the two signals to calculate temperature. As such, the absolute emissivity of the target was not required but rather the change in emissivity over the two wavelength bands. The emissivity was assumed to be constant over the two wavelength bands. The pyrometer was aimed at the converging section on the chamber (typically the highest temperature) and had an approximately 1.0-cm-diameter spot size. The temperature range of the pyrometer was 982 to 3315 °C with a measurement error of ± 27 °C.

Test Facility

Testing of the chambers was conducted in a propulsion test facility designed for research of low-thrust rockets operating on GO_2/GH_2 propellants. The rocket was mounted in a 0.91-m, cylindrical test tank with viewports for optical access. A two-stage air ejector system maintained a 1.4 kPa pressure in the tank, equivalent to an altitude of 36.6 km. The rocket was mounted horizontally and fired into a water-cooled diffuser. All data were recorded on a PC-based data acquisition system and performance parameters were calculated in real time. A more detailed description of the test facility is available in reference 11.

TEST RESULTS

Annealed ED Ir/PM Re Chamber

Testing of the annealed ED Ir/PM Re chamber is summarized in table I. MR's were chosen to simulate the oxygen and water vapor partial pressure of Earth-storable propellant combustion environments. Combustion temperatures with GO_2/GH_2 propellants are higher than Earth storables, so a more severe test environment was provided. Two series of tests were conducted on the annealed chamber. In the first series, a total of 9 firings were conducted, with a total test time of 2.01 hr, at a nominal MR of 4.2 and P_c of 552 kPa. Initially, two 10-sec tests were performed for checkout of the chamber and hardware, followed by longer duration tests, varying from 10 to 30 min. The outer wall temperature measurement varied from 2040 to 2110 °C. On the ninth firing, P_c rose from 552 to 621 kPa, indicating a change in the throat diameter. Visual inspection showed blistering of the Ir coating in the barrel section of the chamber. Also, the Ir layer was spalling in the converging section; this revealed an underlying darker material. It was not clear from visual inspection whether this underlying material was Re or an Ir-Re alloy. Because of the chamber geometry, the composition of the underlying material could not be determined nondestructively. Instead of examining the chamber destructively, testing was continued to determine whether more time could be accumulated.

The second series of tests totaled 15 firings and 2.57 hr, at the same nominal MR. In the first six tests, the chamber reached the two-color pyrometer redline (2315 °C) before thermal steady-state (approximately 10 sec). In order to conduct longer duration tests, P_c was reduced from 621 to 414 kPa by reducing mass flowrates. As a result, outer wall temperatures dropped to 2160 °C. Test durations for the remaining tests ranged from 10 to 30 min. The Ir layer continued to spall as test time was accumulated, which made it appear as if it were being peeled back. The spalling occurred in the converging section at first, but continued into the barrel of the chamber. The dark material underneath the spalled Ir showed no signs of degradation. It would seem unlikely that this material was pure Re, since it would have been attacked by the oxidizing environment it was exposed to.

Testing was stopped after a total (including both test series) of 4.5 hr was accumulated, although the chamber was still capable of more runs. The Ir layer was completely removed from the barrel section, at about 1.3 cm upstream of the converging section, through the converging section itself, and down to the throat. There were two thin strips of the Ir layer removed from the throat downstream into the nozzle. There were also three small regions in the barrel where coating was removed. In the regions where it was being removed, the inner layer had curled up and was hanging down into the chamber, particularly so just downstream of the throat. Metallography was not conducted on this chamber. The blistering and spalling of the annealed ED Ir coating was similar to that experienced with testing of an as-deposited ED Ir/PM Re chamber (ref. 6). From this data it was concluded that the vacuum annealing process as performed was not effective in improving the adherence of the ED Ir coating.

HIP ED Ir/PM Re Chamber

Testing of the HIP chamber is summarized in table I. A total of 91 firings and 11.45 hr of test time were accumulated on the HIP chamber, at a nominal P_c of 496 kPa. For the first 9.04 hr the chamber was tested at a nominal MR of 3.2, followed by 2.41 hr at MR = 4.2. After an initial 5-sec checkout test, the next 60 firings were 30-sec duration tests. This was done to rapidly accumulate thermal cycles, cycling being better than time at temperature in determining the adherence of the coating. Most of the remaining tests were 30 min in duration. For the MR 3.2 testing, outer wall temperature measurements varied from 1700 to 1800 °C. For the MR 4.2 testing, the outer wall temperature was 1900 °C. This chamber ran cooler than the annealed chamber because of the high emissivity coating.

The chamber was visually inspected several times during testing. Throughout the testing, the Ir coating appeared unchanged from the pretest condition. No significant mass or dimensional changes were observed.

Following testing, the HIP chamber was sectioned and examined metallographically. The Re substrate survived the testing largely intact. An Ir/Re interdiffusion zone, with a width averaging between 5 and 20 μm , was found to run the length of the chamber. Diffusion of Re into the Ir layer and the oxidation of the resulting Ir-Re alloy from the inner wall is the known, and relatively slow, life limiting mechanism for Ir/Re rockets (ref. 2). A line of porosity was observed in the Ir coating down the length of the chamber. The Ir coating thinned from the barrel section of the chamber to the throat. The Ir layer thinning was most pronounced in the converging section, generally the hottest portion of the chamber. Downstream of the throat, where the chamber cools off rapidly, the Ir layer was thick. Some regions of the Ir coating contained approximately 5 μm grains of contaminants near the inner surface. Spectra of silicon, chlorine, oxygen, and carbon were found in the grains, probably introduced during the application of the ED Ir coating. Contaminates were thought to be in the as-deposited ED Ir coating tested in an earlier program (ref. 6).

From the barrel section to the throat, occasional, discrete porosity was observed in the Re layer, just below the Ir/Re diffusion zone. This feature was evident from optical differential interference contrast (DIC) micrographs, taken approximately 40 mm downstream of the attachment cone. The DIC micrographs are shown in figures 4 and 5. These pores in the Re layer formed near the thinning regions of the Ir coating. The pores indicated an oxidative or chemical attack of the Re substrate. Since Re material was removed, but the coating was still adherent (although thinned), a path through the Ir layer must have been present for Re to outgas. The path was likely provided by pinholes in the Ir layer, which also would provide a direct path for oxidation of the Re layer. Further testing would have probably led to the compromise of the Re substrate and chamber burnthrough. The time to failure would be a function of the severity of the oxidizing environment.

Figure 6 shows a DIC micrograph taken in the nozzle. Here the Ir layer is thick and intact. While there were pores in the Ir layer (as there were throughout the chamber), the porosity was not as extensive as the porosity in the Re layer that was experienced in the barrel and converging sections. The lack of Re pores and presumably Ir layer pinholes in the nozzle would seem to indicate that the flaws upstream of the throat formed during testing, rather than as a result of the fabrication process.

The HIP chamber used in this program was compared to a reference CVD Ir/Re chamber tested in an earlier program (ref. 12). The CVD chamber was of the same geometry and thrust class as the PM chambers and was tested in the RL-11 facility with an injector assembly similar to the one used in this program. A total of 128 firings and 14.2 hr of test time was accumulated on the chamber. Most of this testing was conducted at $MR = 3$ to 5 and $P_c = 655$ to 690 kPa. Under those conditions, the outer wall temperature was below 1900 $^{\circ}\text{C}$. Testing was stopped because of a facility-related incident that resulted in the overheating of the water-cooled section between the injector and chamber. Metallography of the chamber revealed no measurable change in the Ir layer thickness from pretest measurements. The amount of Re diffusion into the Ir layer of the CVD chamber was low, leading to the conclusion that the chamber could have run for a significantly longer time. The Ir/Re diffusion zone for the HIP chamber was small, indicating that it could have also accumulated more test time. The pinholing observed in the ED Ir layer of the HIP chamber provides a more rapid path to failure than the slow Re diffusion/Ir-Re oxidation life limiting mechanism. Thus, where HIP treatment of ED Ir resulted in a more adherent coating than annealed or as-deposited ED Ir coatings, further densification of the ED Ir coating is required to yield test results comparable to the CVD Ir coatings.

CONCLUDING REMARKS

Two 22-N ED Ir/PM Re rocket chambers were tested on GO_2/GH_2 propellants. Two different post-deposition treatments were performed on the chambers in order to densify their ED Ir coatings. One chamber was vacuum annealed, while the other was HIP. Both chambers were tested in combustion environments that were comparably oxidizing to and higher temperature than Earth-storable propellants. When subjected to thermal cycles, the annealed ED Ir coating began to blister and spall in the converging section of the rocket. This behavior was similar to Re chambers tested with unannealed, as-deposited ED Ir coatings. The HIP ED Ir coating remained adherent to the Re substrate under significant thermal cycling. Metallography of the HIP chamber indicated that pinholes developed in the Ir coating in some places, allowing a direct path for oxidation of the Re substrate. While the HIP ED Ir coating was more effective than vacuum annealed and as-deposited ED Ir, further densification is still required to match the integrity of CVD Ir coatings. The demonstrated durability (91 firings and 11.45 hr) of the HIP chamber, though,

indicates that the ED Ir/PM Re fabrication process may be a viable, low-cost alternative to CVD and further investigation is warranted.

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TABLE I.—TEST SUMMARY FOR ED Ir/PM Re CHAMBERS

| Chamber | Firings | Time, sec | Mixture ratio, MR | Chamber pressure, P _c , kPa | Outer wall temperature, °C |
|--------------|---------|-----------|-------------------|--|----------------------------|
| Annealed ED | 9 | 7 237 | 42 | 552 | 2040 to 2110 |
| Ir/PM Re | 15 | 9 255 | 42 | 414 to 621 | 2160 to 2315 |
| Total | 24 | 16 492 | | | |
| HIP ED Ir/PM | 84 | 32 534 | 32 | 496 | 1700 to 1800 |
| Re | 7 | 8 685 | 42 | 496 | 1900 |
| Total | 91 | 41 219 | | | |

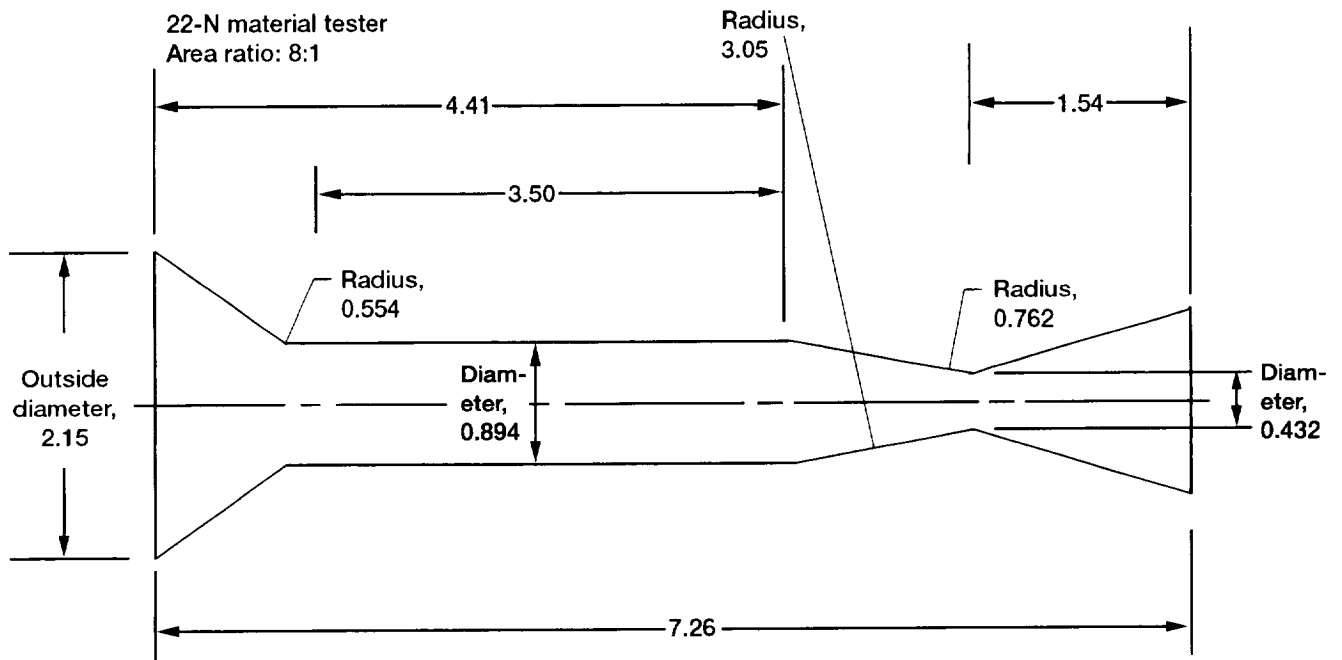


Figure 1.—Rocket geometry for 22-N class material tester. (All dimensions are in cm.)

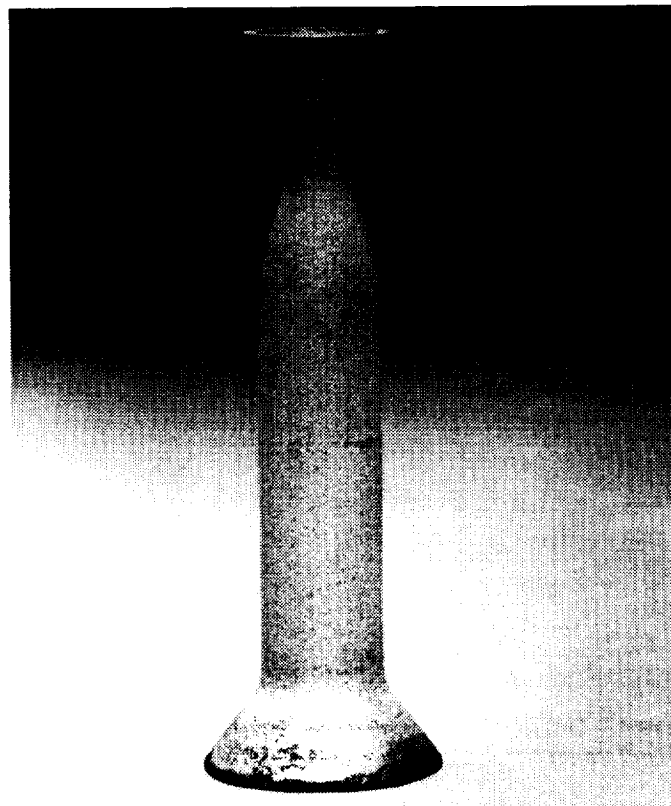


Figure 2.—HIP ED Ir/PM Re chamber, after application of high-emissivity exterior coating.

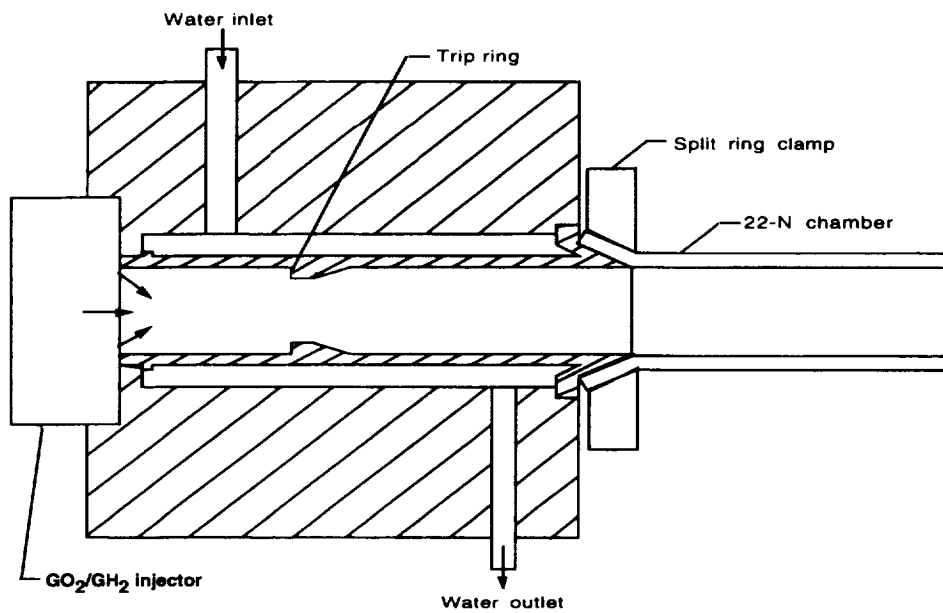


Figure 3.—Injector assembly for testing 22-N rocket chambers. (Sketch not drawn to scale.)

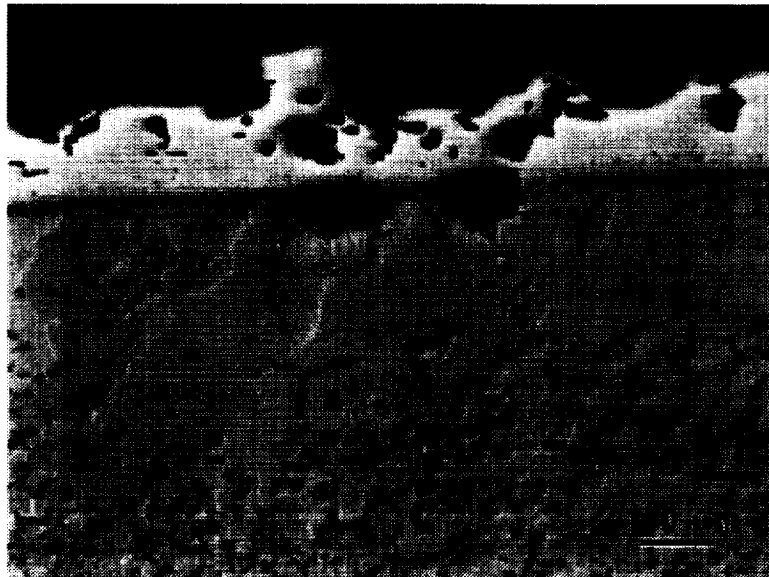


Figure 4.—DIC micrograph of HIP ED Ir/PM Re chamber, 40 mm downstream from attachment cone.

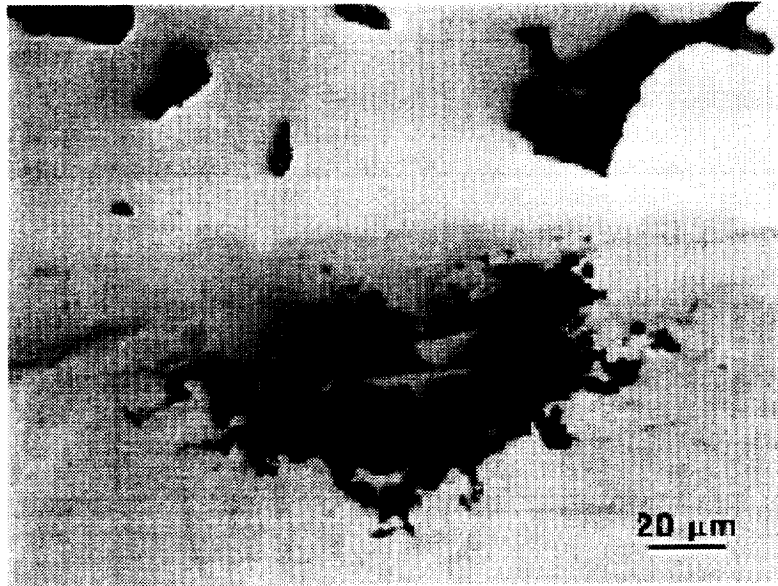


Figure 5.—DIC micrograph of HIP ED Ir/PM Re chamber, 40 mm downstream from attachment cone.

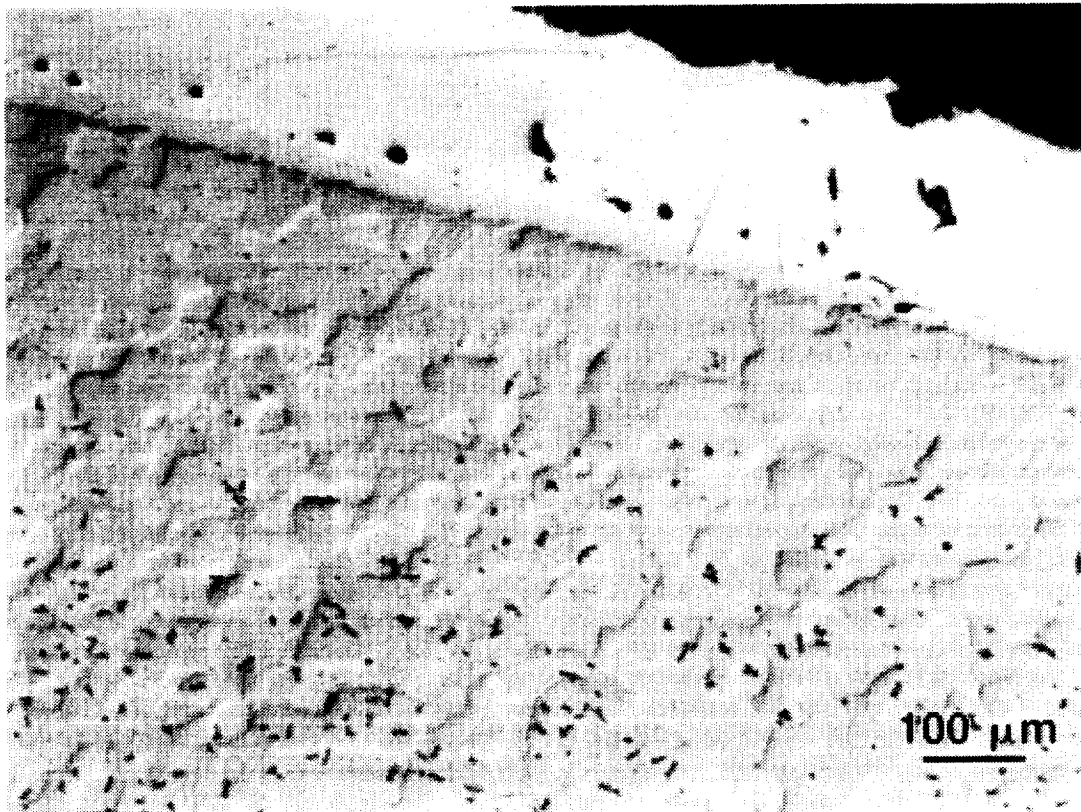


Figure 6.—DIC micrograph of HIP ED Ir/PM Re chamber, downstream of throat (nozzle).

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