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Fabrication of Ceramic Substrate-Reinforced and Free Forms

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FABRICATION OF CERAMIC SUBSTRATE-REINFORCED AND FREE FORMS

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ABSTRACT

Components fabricated of, or coated with, ceramic have lower parasitic cooling requirements. Techniques are discussed for fabricating thin-shell ceramic components and ceramic coatings for applications in rocket or jet engine environments. Thin ceramic shells with complex geometric forms involving convolutions and reentrant surfaces were fabricated by mandrel removal. Mandrel removal was combined with electroplating or plasma spraying and isostatic pressing to form a metal support for the ceramic. Rocket engine thrust chambers coated with 0.08 mm (3 mil) of ZrO_2-$8Y_2O_3$ had no failures and a tenfold increase in engine life. Some measured mechanical properties of the plasma-sprayed ceramic are presented.

1. INTRODUCTION

Preventing excessive surface temperatures on rocket or jet engine components requires either parasitic cooling, an insulating layer such as a ceramic coating, or ceramic components.

In the early 1940's, Ernst Schmidt[1] was fabricating ceramic turbine blades of two basic types: a hollow core with coolant passages, and a membrane over a compliant porous substrate (Fig. 1). Apparently Schmidt recognized four basic needs: low thermal conductivity, a smooth exterior surface, blade cooling, and a strain relief mechanism.

In the late 1950's, steel and copper heat-sink rocket engine nozzles plasma spray coated with Rokide-A were used in engine performance testing of the liquid oxygen/ammonia-fueled X-15.[2] Although the engine was cycled from cryogenic temperatures to 4.1 MPa and 2200 °C (600 psi and 4000 °F) in 0.02 sec and ran for 0.2 sec to shutdown, the coatings did not spall but eroded away at the throat.

Since that time several investigations involving ceramic coatings on rocket nozzles have been carried out[3] and engine cooling requirements have been pushed to extremes. For example, the space shuttle main engine (SSME) operates at a chamber pressure of over 20.7 MPa (3000 psia), has a throat heat flux of 131 to 147 MW/m^2 (80 to 90 Btu/in^2 sec), and has a life of 55 thermal cycles. Beyond SSME, orbital transfer vehicles (OTV) may have life requirements of over 300 thermal cycles, in addition to being lightweight and high performing. It would appear that ceramics are necessary.

Recognizing the need for a ceramic-lined thrust chamber and the surface roughness limitation, an "inside out" process[3,4] was chosen to form a smooth ceramic liner. Details of the process and test results are discussed in section 5.

Niino et al.[5] fabricated a similar thrust chamber, without the ceramic, by cold isostatic pressing a 10-percent-tin OFHC copper powder mixture to form the closeout and using Wood's metal to preserve the milled coolant passages. Adding the ceramic should not have been a major problem as the loads are compressive and restrained by the mandrel, but as discussed in section 4, this was not so.

In Ref. 6, ceramic sheet-strips were fabricated by plasma spraying zirconia (YSZ) over NiCrAlY on a substrate and then using acid dissolution. This study extended this technique to complex free forms and surfaces requiring parting planes.

Free-form ceramics are discussed in section 2, properties in section 3, an outside-in process in section 4, and inside-out thrust chambers in section 5. The test results are summarized in section 6.

2. COMPLEX FREE FORMS

Following Ref. 6, complex thin-shell, plasma-sprayed free forms were fabricated with the concept that a series of these forms (Fig. 2) could be assembled to form a seal. Although the stresses are large on the individual components, ceramic seals and other components are possible if assembled with compliant cores.

A stainless steel mandrel in the form of a chevron seal was grit blasted with Al_2O_3 and plasma sprayed with 0.05 to 0.08 mm (2 to 3 mil) of NiCrAlY. Next a 0.38-mm (0.015-in) layer of ZrO_2-$8Y_2O_3$ was plasma sprayed to form a composite. The metal was dissolved in an HCl solution (typically 0.8 to 1.0 HCl/0.2 to 0.0 HNO_3) with no deleterious effects.[6,7]

A second method is described by Clingman[8] whereby ZrO_2-$8Y_2O_3$ (YSZ) is plasma sprayed directly onto SiC (Fig. 3). A ceramic free-form ring 22.5 cm in diameter by 1.5 cm high by 0.125 cm thick (8.86 by 0.59 by 0.049 in) was fabricated by plasma spraying a preparation coat 0.025 cm (0.01 in) thick followed by a 0.1-cm (0.04-in) thick coat of about 14 percent YSZ and 86 percent eccospheres (small ceramic spheres). The substrate temperature was held below 150 °C (300 °F). The ring contracted during spraying,[6-8] facilitating removal from the substrate. The ring could be easily flexed radially but was quite stiff in the other coordinate. Failure of the ring structure resulted in square-ended, full-width arc segments of random lengths less than 4 cm (1.57 in).

Another method, under development, involves plasma spraying over a ceramic core or fiber-reinforced ceramic core materials. Core materials with various densities and reinforcements mitigate stresses engendered in heat engines.[9] Refiring in a controlled atmosphere can provide strain relief or densification. In some instances the core could be "fired" away. A 0.38-mm (0.015-in) thick layer of ZrO_2-$8Y_2O_3$ was plasma sprayed over the fibrous SiO_2 material of Ref. 9; the adhesive strength was poor.

In each of these methods surface distortions caused by residual stresses and dimensional stability must be considered in fabricating components.

3. PROPERTIES OF YSZ

For metal substrates with thermal expansion coefficients larger than those of ZrO_2-Y_2O_3 the ceramic flowed in extension; for those with smaller coefficients the flow was in compression (also see analysis of Ref. 10). The plastic flow appeared to be identical for ZrO_2-$8Y_2O_3$ and ZrO_2-$12Y_2O_3$ and similar for ZrO_2-8CaO and Al_2O_3-2.5TiO. Thin, free-form, plasma-sprayed ZrO_2-$8Y_2O_3$ annealed at 1200 °C (2200 °F) showed no plastic flow when subsequently loaded at 1200 °C; the as-plasma-sprayed ZrO_2-$8Y_2O_3$ showed significant plastic flow at the same load and temperature. These results were independent of the argon, hydrogen, and vacuum environments; plastic flow was insignificant with slow cooling. The annealed specimens were significantly harder than as-sprayed specimens.[7] In general the elastic modulus of sheet ZrO_2-Y_2O_3 ranged from 3.4 to 6.9 GPa (500 to 1000 ksi), with a bending (flexural) strength of 12.4 MPa (1.8 ksi) and a shear (torsion) strength of 15.5 MPa (2.25 ksi). Thermomechanical racketing was noted. Mechanical deformation tensile crack spacing w (in millimeters) equaled $0.51 + 0.113\ t^2$, for $0.12 < t < 1$ mm, where t is the sheet thickness.[11,12]

The large changes (microfracturing) evident in the initial thermal cycles of recent acoustic emission tests[13] indicate structural deformations perhaps similar to those noted in the flexure and creep studies[7] or that the thermophysical phase changes are not passive.

Annealing can be a practical method to control creep, but why creep occurs is a more fundamental issue.[7] The term "creep" can encompass such mechanisms as microcracking, sintering, and phase change including devitrificaton.

4. OUTSIDE-IN METHOD FOR COOLANT CHANNELS

4.1 Powder Metallurgy

Powder metallurgy, a concept still in the development phase, was used to form coolant channels (Fig. 4). The walls of the press form had nonstick surfaces. Stabilized ZrO_2 powder was placed in the press form and partially compacted; powdered copper, in an amount equivalent to the liner thickness, was added and partially compacted. An insert, contoured Wood's metal, was put in place to form the coolant passages, and copper powder was added to form the closeout.

The layers were not as uniform as expected. The powders were pressed to 0.176 GPa (25.6 ksi) in the press form and encapsulated in a rubber baggie then isostatically pressed to 400 MPa (60 ksi). Some of the zirconia powder stuck to the rubber. The samples were then sintered at 930 °C (1700 °F) overnight (12 hr) in a vacuum furnace. The component had a dusty surface, but the desired interface gradient layer existed. More effort is required.

4.2 Components with Parting Planes

Plasma spraying onto preformed mating mandrels and constructing the component superstructure by electroforming can be used for components requiring parting planes. To illustrate the process, a copper tube was sectioned and plasma sprayed on the inside with thin layers of YSZ and NiCrAlY (0.08 mm (3 mil) each). The copper was then dissolved in a nitric acid solution (Fig. 5(a)). No failures of either the YSZ or the NiCrAlY occurred, but the residual stresses of plasma spraying distorted the cylindrical shape to elliptical. "Butt" and "lap" joints were secured by electrical tape, which also prevented infusion of the electrolyte into the ceramic. The NiCrAlY bond coat formed the cathode in a copper sulfate/HCl solution (copper tube anode) with a plating current of 3 A.

Three of four critical requirements have been met: a cooled, smooth, approximately 16-rms, low conductivity surface has been achieved. The strain relief remains to be tested.

5.3 Post-Test Analysis

Figure 12, a summary figure, illustrates the thrust chamber and engine configuration used, the coolant channel geometry, and a tenfold increase in thrust chamber life over that of the standard uncoated OFHC copper thrust chamber. It is important to note that there were no failures of these thrust chambers. Furthermore the ceramic must be continuous and not expose the substrate. For even if the thrust chamber is designed to operate uncoated, hot spots can develop in a spalled or delaminated region from excessive upstream boundary layer temperatures and turbulence.

5.3.1 Coating: ZrO_2-$8Y_2O_3$, 0.13 and 0.08 mm (5 and 3 mil)

A surface micrograph (Fig. 13) illustrates the ceramic, bond coat, and substrate layers. The ceramic surface is smooth and of uniform thickness. Figure 14 represents an axial sectioned chamber with some coating delamination in the chamber region (approximately one-third of the chamber length from the left end). The 0.08-mm (3-mil) thick coating delaminated to 0.025 mm (1 mil). Since the delaminated coating was in a low-heat-flux region, engine operations were not affected.

5.3.2 Coating: ZrO_2-CaO, 0.2 mm (8 mil)

The 0.2-mm (8-mil) thick coatings spalled to an average thickness of 0.064 mm (2.5 mil) without further deterioration. Correspondingly the temperature rose significantly to about 80 hot firing cycles, leveled off (when the coating reached average thickness), and remained nearly constant to over 650 cycles (Fig. 15).

Nonuniformities in either the coating or the engine injector can cause coating failures (Fig. 16). Figure 16(a) illustrates a structurally sound surface: Fig. 16(b) shows the opposite surface with significant erosion and delamination. However, the engine survived because the delaminated coating was still sufficient to protect the coolant channels.

These results also illustrate the fourth criterion - strain relief. If strain relief is not sufficient, the coating erodes or delaminates to 0.025 to 0.08 mm (1 to 3 mil) but with sufficient residual within the bond coat to protect the copper liner. Extensions to complex curvatures, other engine geometries, and such engine conditions as hard starts and off-stoichiometric operation are required.

6. Internal Plasma Spraying

Plasma spraying the internal surface of a 6.6-cm (2.6-in) diameter cylinder is, at best, difficult. A small torch and a spraying history had to be developed before several cylinders were fabricated and tested. The results were, by comparison with the inside-out process, poor. Furthermore the substrate oxidized, probably during fabrication. The coating was 0.08-mm (3-mil) thick ZrO_2-$8Y_2O_3$ over a 0.05-mm (2-mil) Ni-18Cr-6Al-0.5Y bond coat. Figure 17(a) illustrates the erosion of the plasma-sprayed surface. Recall that the smooth-surface criterion has not been met, and an appreciable increase in heat transfer and erosion can be expected. Similar results are noted in Ref. 2, with the steel nozzle throat being eroded away. A poorly bonded interface can lead to development of a hot spot (burnout) (Fig. 17(b)) from the flow of hotter boundary layer gases and augmented local heat transfer, or in some cases, to complete coating spall (Fig. 17(c)).

Post-test examinations led to the following recommendations: eliminate oxidation of the copper surface during the plasma-spraying process and maintain uniform (hopefully smooth) surface coatings. Further coating and testing is planned.

7. SUMMARY

Several methodologies for fabricating ceramic components and composite structures have been described. Among these are complex free-form, thin-shell, plasma-sprayed ceramic structures and the "inside out" process for fabricating ceramic-lined thrust chambers for rocket engines.

Direct plasma spraying of thin-shell surfaces, such as U-shaped surfaces of revolution, although a quick process, requires a sacrificial mandrel and is line of sight (hyperbolic) limited.

For components with parting planes the component assembly superstructure can be fabricated by electroplating. Electroplated plasma-sprayed sheets are quite flexible.

For the inside-out process 0.05- to 0.08-mm (2- to 3-mil) thick coatings of yttria-stabilized zirconia increased thrust chamber cycle life by a factor of 10 over that of the conventional uncoated OFHC chamber.

Direct plasma spraying of the chamber was not satisfactory because of surface oxidation during spraying and the significant increase in surface roughness. Further tests are planned.

Properties of free-form, plasma-sprayed YSZ are Young's modulus, 3.4 to 6.9 GPa (500 to 1000 ksi); bending stress, 12.4 MPa (1.8 ksi); shear stress, 15.5 MPa (2.25 ksi); flexural tensile crack spacing, $w = 0.51 + 0.113\ t^2$, for $0.12\ \text{mm} < t < 1\ \text{mm}$ thickness. Ratcheting, which is related to mechanical loading, occurs. Acoustic emission, which is most significant during initial thermal cycles, may be related to mechanical loading (ratcheting) or to annealing, which eliminates creep to the annealing temperature (another form of ratcheting).

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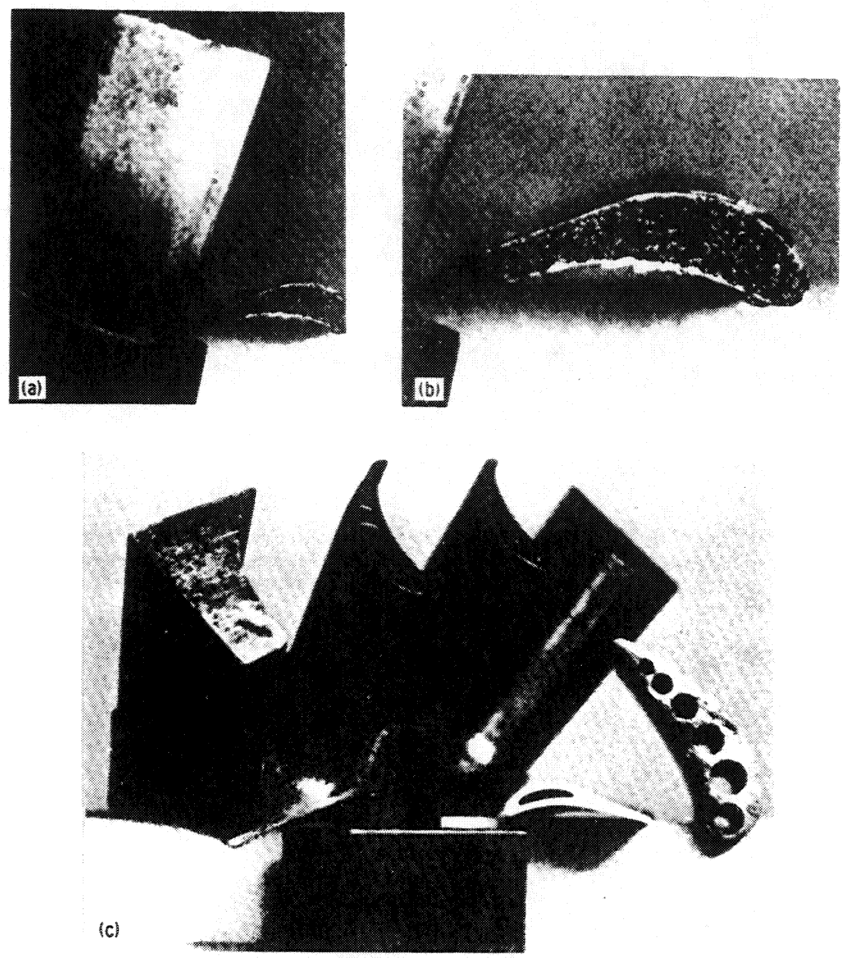


(a) Structure of porcelain enamel compliant core.
(b) Closeup of shell and compliant core structure.
(c) Turbine blade configuration and experimental ceramic blades.

Figure 1. - Prof. Schmidt's experimental ceramic vanes and blades.

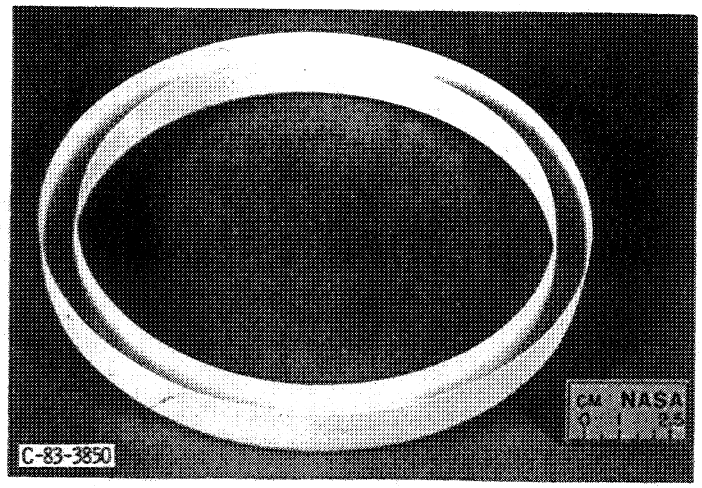


Figure 2. - Complex plasma-sprayed free form.

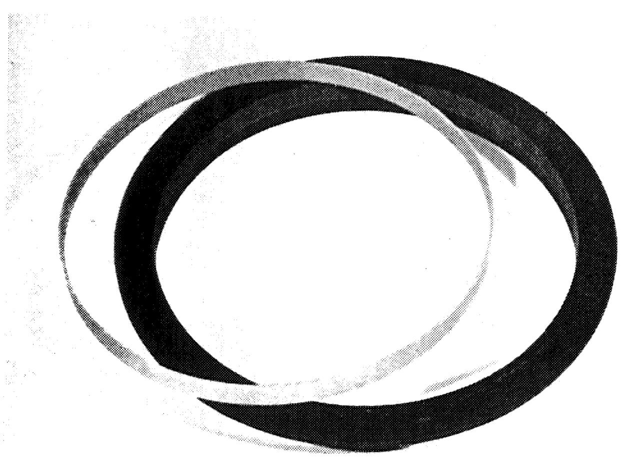


Figure 3. - Plasma-sprayed free-form ring. (From ref. 9.)

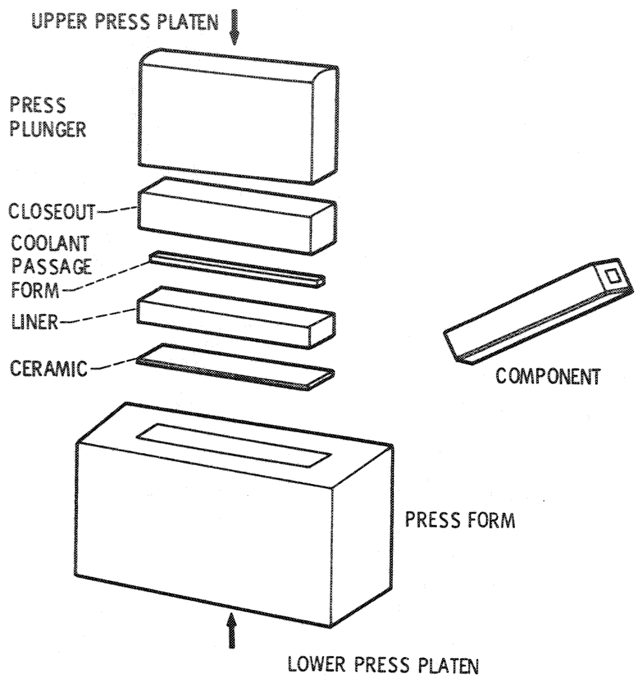
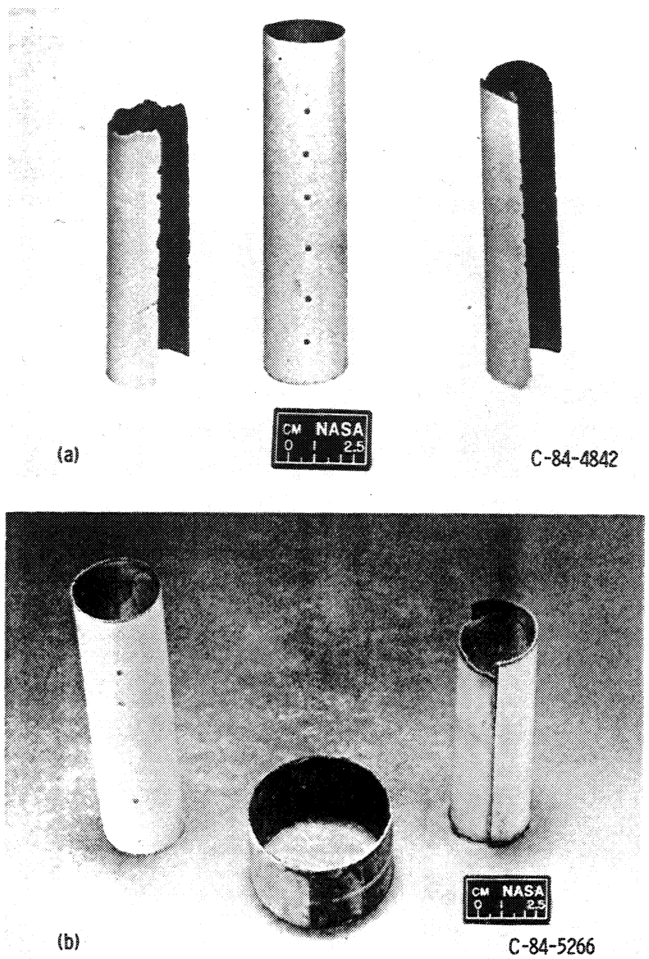
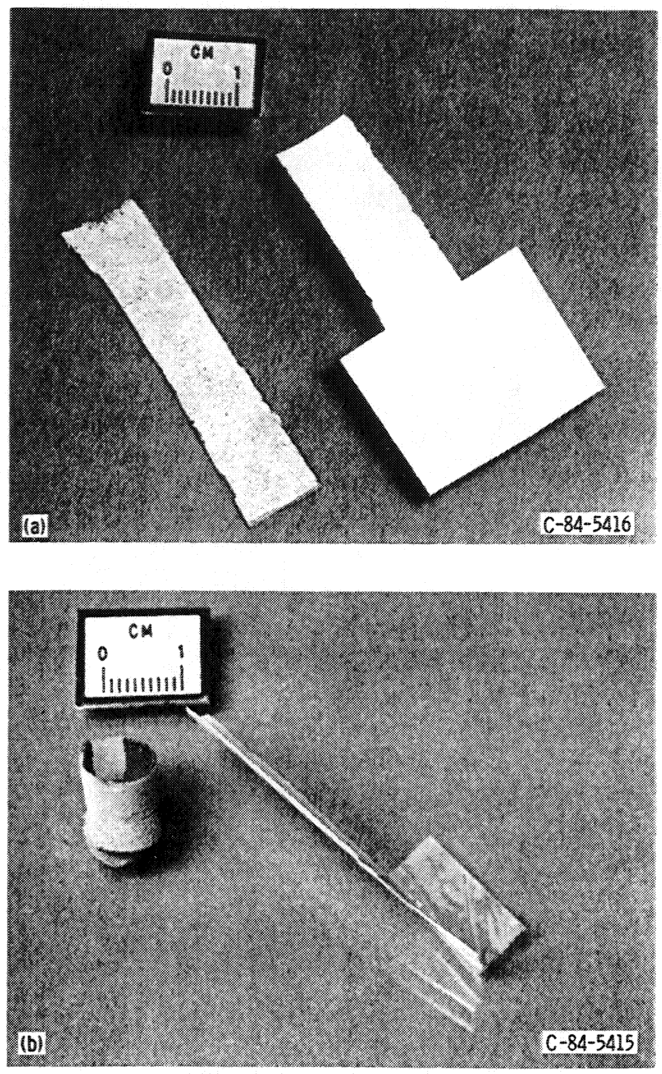


Figure 4. - Illustration of powder metallurgy concept.



(a) Thin-shell cylinder and split halves with holes.
(b) Electroplated superstructure including butt and lap joints.

Figure 5. - Electroforming superstructure to mating ceramic components.



(a) Electrodeposited and uncoated ceramic free-form sheets.

(b) Coiled, coated ceramic sheet and edge view of uncoated sheet.

Figure 6. - Flexibility of thin, ceramic free forms with electrodeposition.

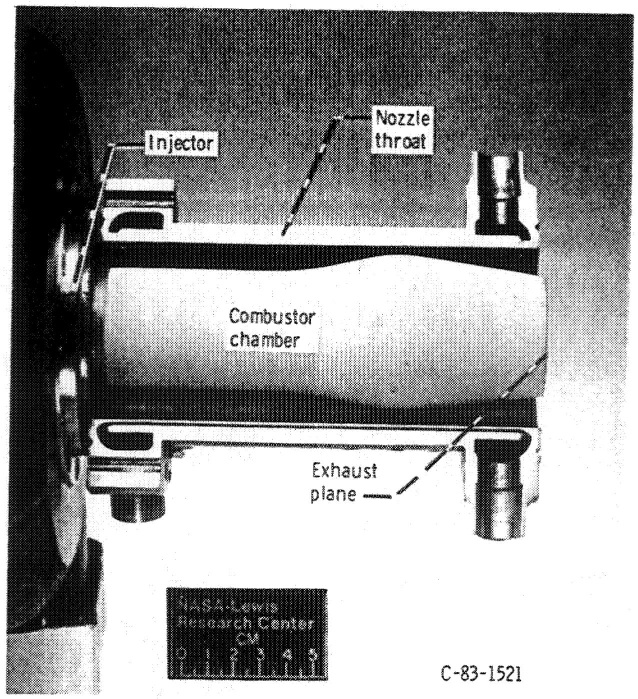


Figure 7. - Cutaway view of test thrust chamber.

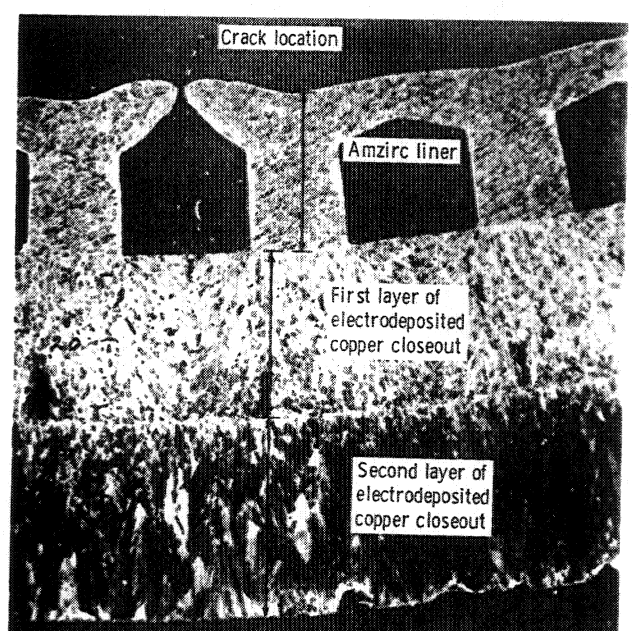


Figure 8. - "Doghouse" failure of coolant channel wall.

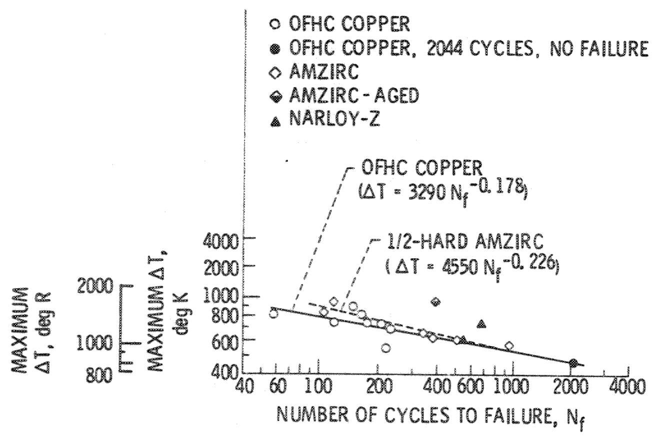


Figure 9. - Strainrange partitioning using maximum wall temperature difference between hot-gas side and back side as a function of cycles to failure for OFHC, Amzirc, and NARloy-Z cylinders.

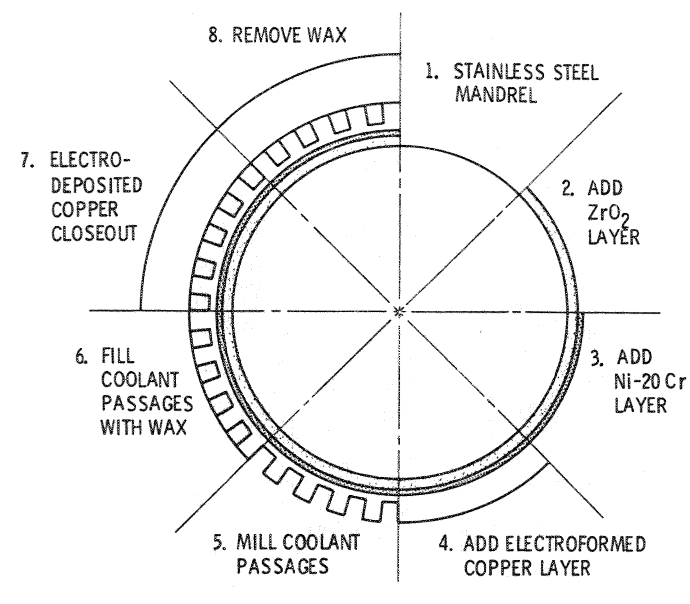


Figure 10. - Inside-out fabrication sequence for ceramic-lined thrust chambers.

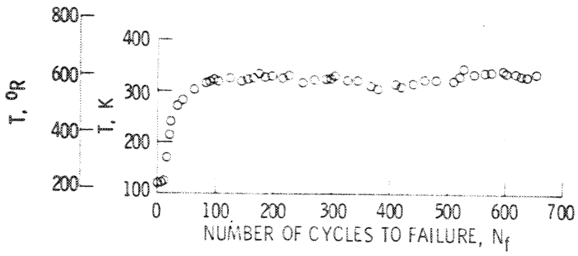
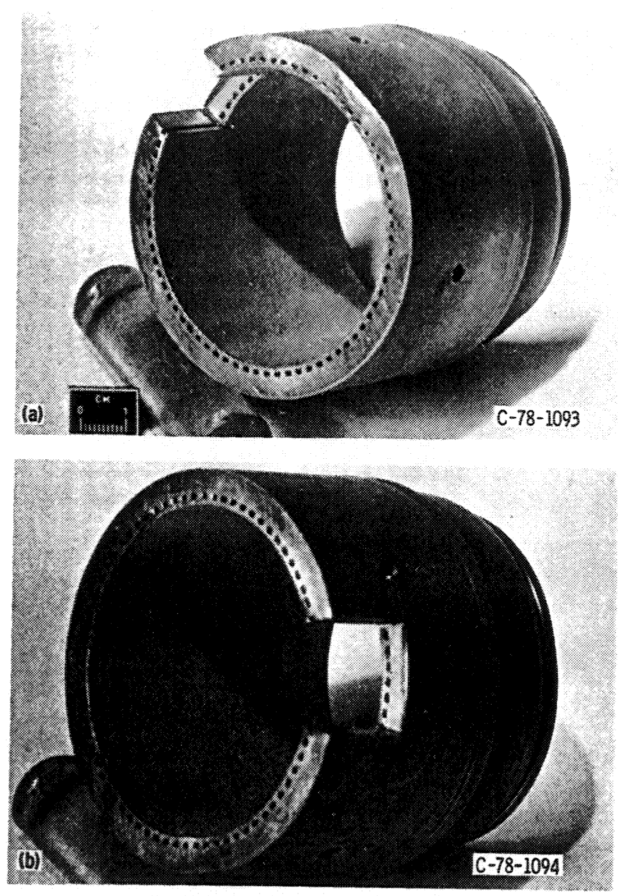
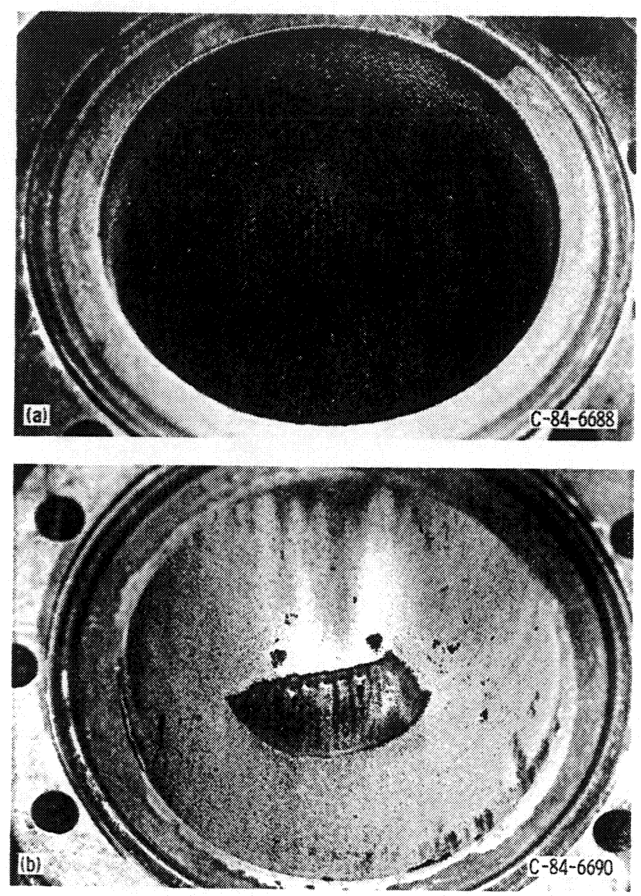


Figure 15. - Steady-state rib temperature-time (cycle) history with ceramic liner erosion.



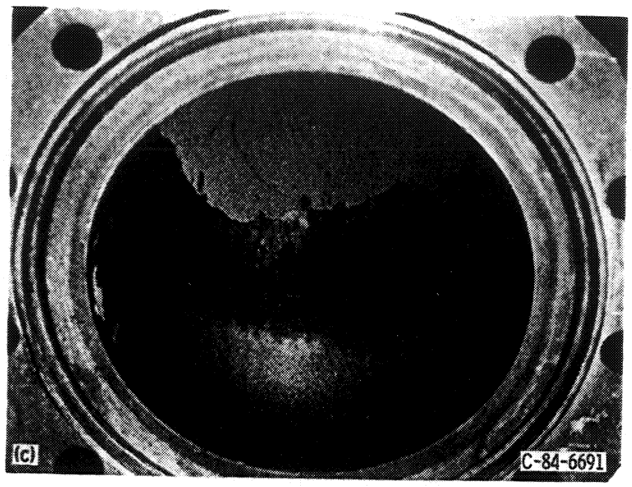
(a) Structurally sound surface.
(b) Opposite side, illustrating erosion and delamination.

Figure 16. - Sectioned ceramic-lined thrust chamber after 650 cycles.



(a) Erosion of thrust chamber 100 after 237 cycles.
(b) Hot-spot failure.

Figure 17. - Cylindrical thrust chamber plasma sprayed on inner surface.



(c) Spallation.

Figure 17. - Concluded.

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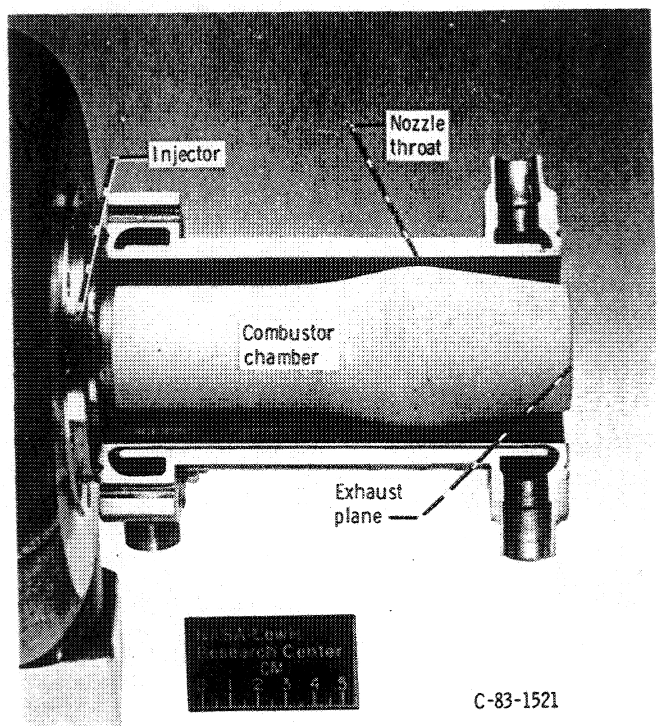

Figure 7. - Cutaway view of test thrust chamber.