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COATED COLUMBIUM THERMAL PROTECTION SYSTEMS - AN ASSESSMENT OF TECHNOLOGICAL READINESS

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16. Abstract

The current state of the art for the coated columbium thermal protection system (TPS) was assessed. Coated columbium suffers some weight and cost disadvantages compared to the alternative ceramic reusable surface insulation being developed for the Space Shuttle Orbiter TPS. If problems related to emittance losses in the reentry environment and localized substrate embrittlement at coating breakdown sites on internal surfaces could be resolved and if panel and fastener design were optimized, coated columbium could again be considered for limited use in a 100-mission TPS.

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SUMMARY

Coated columbium was considered as one of several candidate materials for the Space Shuttle Orbiter thermal protection system (TPS). Further efforts to develop coated columbium for this purpose are being minimized. This system suffers from four liabilities. First, there are weight and cost disadvantages when the system is compared to ceramic reusable surface insulation, which is currently viewed as the primary TPS material choice. Second, decreases in coating emittance have been observed in as few as 10 exposures to high-velocity, low-pressure plasma-arcs simulating reentry conditions, and these decreases cause concern about the suitability of columbium coated with silicon - 20 percent chromium - 20 percent iron (R512E) for 100-mission reradiative TPS service as currently formulated and used. Third, there is the problem of internal, hard to detect, local coating breakdowns. These would require that the panels be removed and carefully inspected about every 20 reentries. Fourth, additional effort is still required in the area of optimum panel design and fastener technology. Progress in the latter three areas is required for this system to see limited use in a 100-mission, minimum-refurbishment TPS such as is required for the Space Shuttle Orbiter vehicle.

Evaluation and development to date show that of the coated columbium alloys FS-85 coated with R512E shows significant promise for a reusable TPS system as judged by environmental resistance and the retention of mechanical properties and structural integrity of panels upon repeated reentry simulation. Production of the alloy, the coating, and full-sized TPS panels is well within current manufacturing technology. Small defects which arise from impact damage or from local coating breakdown do not appear to have serious immediate consequences in the use environment anticipated for the Space Shuttle Orbiter TPS.

INTRODUCTION

Ablators, ceramic reusable surface insulation (CRSI), carbon-carbon composites, and metallic systems have all been considered as potential candidates for various parts of the reentry thermal protection system (TPS) for the Space Shuttle Orbiter (ref. 1). Each material class is in a different state of readiness, and each has different advantages and liabilities. Ablators are relatively well developed and have a proven record of accomplishment in the Mercury, Gemini, and Apollo programs. Such materials currently have reuse capability for only a single mission or, at most, a few missions rather than the desired 100-mission life needed to minimize Orbiter vehicle operating costs. Ceramic reusable surface insulation, while having considerable potential to minimize TPS cost and weight, is still being developed and optimized. Additional effort is required to assure its vehicle readiness. CRSI has had only one reentry flight - in 1968 on the NASA Pacemaker reentry vehicle. Carbon-carbon composites, with primary potential for leading-edge use, are in a similar, if not earlier, stage of development. The metallic heat shield materials: titanium alloys, superalloys, dispersion strengthened materials, and refractory metals - have a much larger technology base than any of the other reusable systems. Some single reentry vehicle experience exists for superalloy metal heat shields - primarily as shingles on Mercury, Gemini, and Apollo capsules - and heat shield components of the ASSET (Aerothermal Dynamic/Elastic Structural Systems Environmental Test) vehicle. Coated columbium (Cb) has primary use potential in those areas of the Orbiter experiencing temperatures from 1100° to 1350° C upon atmospheric reentry (fig. 1). In general, such areas can represent only 10 to 15 percent of the total heat shield area, but these higher temperature areas are, of course, critical to successful operation.

The purpose of this report is to review the readiness for use of only one of the potential metallic heat shield materials - coated columbium alloys. (A total assessment of the readiness for use of the alternative concepts is also needed but is beyond the scope of this report.) The primary factors which will be considered in this review are:

(1) Readiness to survive the reentry environment
  (a) Oxidation resistance in furnace and plasma-arc reentry simulation
  (b) Emittance stability
  (c) Mechanical property stability
  (d) Defect effects on oxidation, mechanical properties, and structural integrity
(2) Manufacturing readiness
(3) Relative weights and costs of coated Cb and RSI concepts

HISTORICAL SUMMARY

The fused slurry silicide coating R512E (silicon - 20 percent chromium - 20 percent iron (Si-20Cr-20Fe)) is the best available commercial coating for protecting columbium TPS panels.

The impetus for the development of coated columbium systems is primarily traceable to the U.S. Air Force interest in high-speed reentry vehicles which started in the late 1950's. The promise of easy fabricability for the complex high-temperature structures being designed, as illustrated in figure 2 (ref. 2), prompted work with columbium alloys. The fact that high-temperature use in air promoted oxidation of columbium alloys led to a considerable coating development effort on the part of a number of laboratories. The historical background of the various types of coating which resulted (pack-cementation, slurry, chemical vapor deposition, etc.) has been thoroughly reviewed by Gadd (ref. 3). Basically, TPS coating development history can be summarized as shown in figure 3. In four generations of development, the silicide coatings were made easier to apply and considerably more reproducible. These efforts were primarily supported by the Air Force ASSET program. Here aluminide slurry coated columbium alloy heat shields and duplex chromium-titanium/silicon (Cr-Ti/Si) coated leading edge segments as well as coated columbium fasteners were used. In the middle 1960's an ASSET vehicle was recovered after a single atmospheric reentry, and even at that early date the coatings appeared sound enough for another flight (ref. 4). This recovered vehicle is shown in figure 4. The Cr-Ti/Si pack coating (ref. 5) which protected the Cb leading-edge segments represented the first significant improvement in coating protection. However, the need for large retorts to apply this coating to full-sized heat shields as well as the continued lack of protection uniformity led to further efforts to develop improved systems and application techniques.

Subsequently, further coating improvements were achieved involving glass-sealed silicides (ref. 6), tin-aluminum (Sn-Al) impregnated silicides (ref. 7), and better surface prealloy chemistries (ref. 8). As far as the complex structure of heat shields is concerned, however, the best system developed was the fused slurry silicide coating system, R512E (Si-20Cr-20Fe) (ref. 9).

The Si-20Cr-20Fe system is applied as a green slurry by spraying or dipping. The part is then heated above the slurry fusion temperature. The coating melts and wets and covers the columbium alloy like a braze. As it reacts with the columbium alloy substrate, its remelt temperature increases and it solidifies. The resultant coating consists of a number of complex layers of metal-silicon (M_5Si_3) and (MSi_2) phases containing Cb, Fe, Cr, and various substrate elements. A typical photomicrograph of the Si-20Cr-20Fe coating is shown in figure 5. Since such coatings are applied by slurry techniques, they can be conveniently varied in thickness from 0.025 to 0.125 millimeter and be applied uniformly to large complex shapes.

Figure 5 also shows a typical distribution of silicide phases and electron microprobe concentration profiles for the major elements in the as-applied coating. Note that, during the formation of these coatings, about 0.030 millimeter of substrate is consumed for each 0.075 millimeter of coating. This consumption factor is important in dealing with thin (0.3- to 1.0-mm) columbium sheet needed to make the Cb reradiative TPS concept a viable system having minimal weight.

Of the various columbium alloys examined for TPS usage, three had the best combinations of fabricability, coatability, and overall performance under simulated reentry conditions. These are Cb-752 (columbium - 10 percent tungsten - 2.5 percent zirconium (Cb-10W-2.5Zr)), C-129Y (columbium - 10 percent hafnium - 10 percent tungsten - 0.1 percent yttrium (Cb-10Hf-10W-0.1Y)), and FS-85 (columbium - 28 percent tantalum - 10 percent tungsten - 1 percent zirconium (Cb-28Ta-10W-1Zr)). All three alloys were developed in the early 1960's.

READINESS TO SURVIVE REENTRY ENVIRONMENT

The R512E coated FS-85 system has the best combination of properties of any current coated columbium TPS material.

During atmospheric reentry the TPS of the Orbiter will be subjected to varying temperatures, pressures, and loads as it decelerates from orbital velocity. External surfaces will be exposed to high mass flow shear conditions and will radiate heat to the atmosphere. Internal surfaces will be exposed to lower pressures in a relatively stagnant atmosphere. Since a flying test bed is not available, simulation of this complex environment, as illustrated in figure 6, would be extremely difficult. In general, only temperature-pressure-stress profiles approximating either external or internal vehicle conditions during reentry are run in static furnace tests, or temperature-pressure profiles are run in plasma-arc tests. These tests generally vary from laboratory to laboratory.

FURNACE SIMULATION LIFE

Panels have survived up to 100 furnace reentry cycles with minimum coating breakdown and up to 175 cycles without structural failure.

Many hundreds of coupons, tensile specimens, and small panels of the most promising columbium alloys have been coated with Si-20Cr-20Fe and tested under various simulated reentry conditions. In general, protection well beyond 100 missions has been consistently achieved in furnace simulators where programmed temperature-pressure-stress profiles have been applied to simulate lift-off and reentry conditions. For example, 35 of 36 R512E coated Cb-752 and C-129Y specimens so tested survived 100 cycles (ref. 10). In addition, R512E coated FS-85 panels have survived up to 200 launch and furnace reentry simulations, as shown in table I. (The 7.5- by 30-cm rib-stiffened panels were loaded in flexure during simulated reentry exposure cycles to a 1320° C maximum temperature and external-pressure. About 6 min were spent at peak temperature, and the panels were above 880° C for about 30 min/cycle. Acoustic loading to 52 meganewtons per square meter (MN/m^2) was applied periodically to simulate boost.) Similarly, three corrugation-stiffened panels survived their scheduled 100 internal-pressure reentry exposures with no evidence of local coating breakdown (ref. 11).

The microstructure of R512E coated FS-85 after 100 external-pressure reentry simulation exposures is compared with the as-coated microstructure in figure 7. A thick oxide formed on the surface of the tested specimen and filled the widened natural coating cracks. Very little net reduction in coating thickness occurred and only a fraction of the potential coating life was used. Diffusion consumed only about 0.01 millimeter of substrate per side beyond that consumed by coating formation.

PLASMA-ARC SIMULATION

No tests were performed on stressed panels. Of the specimens tested, local coating breakdown was generally observed in the 20- to 50-cycle range.

While many plasma-arc tests have been conducted on coated columbium alloys (refs. 10 and 12 to 14), most of these tests either involved specimens with intentional defects or were stopped after only a few cycles to minimize costs in a multialloy, multicoating evaluation program. Some of the longer time arc test data obtained are shown in table II.

In the shear mode tests described in table II, a temperature profile approximating atmospheric reentry was used (ref. 10). Pressure and stress were fixed at levels characteristic of reentry. Half of the R512E coated specimens developed local coating breakdowns at times short of the 40-cycle goal in these tests. In some cases, the early appearance of local coating breakdown could be attributed to shock wave attachment at leading edge locations of the sheet tensile specimens. Otherwise, the coating was protective for over 29 cycles in these arc tests at a maximum temperature approximating the intended service conditions. Similarly tested columbium alloy specimens coated with VH-109 were not protected as consistently or for as long a time.

In shear tests of R512E coated Cb-752, C-129Y, and FS-85 (ref. 13), 66 specimens survived their scheduled 1 to 10 cycles of arc exposure at 1290° or 1370° C and 15 torr.

Only one specimen suffered a natural local coating breakdown: an R512E coated Cb-752 specimen exposed for one cycle at 1370° C.

Stagnation model plasma-arc tests with a 30-minute square wave temperature profile were conducted to a temperature well above the expected service temperature (ref. 14). From the results of these tests on specimens without defects, it was concluded that R512E in combination with FS-85 performs better than the same coating with either of the other two alloys examined: Cb-752 or C-129Y. The failure of one FS-85 specimen after twelve 1/2-hour cycles was caused by breakage of a holddown tab and subsequent substrate erosion from this site. A second specimen suffered edge degradation at about 30 cycles. Testing of this specimen was continued to 50 cycles with edge recession of only about 1.6 millimeters. A third R512E coated FS-85 specimen survived fifty 1/2-hour cycles intact. The R512E coated C-129Y and Cb-752 systems suffered edge degradation in 6 to 18 cycles. All test results are probably conservative since the specimens were coated prior to the development of an improved edge striper coating technique which appears to triple times to first local coating breakdown at edges (ref. 11).

In all of the arc tests conducted, no evidence of coating breakdown was observed on surface locations. The microstructure of R512E coated FS-85 after 50 cycles of plasma-arc exposure at an average temperature of 1390° C is shown in figure 8. In addition to a greater amount of coating consumption by oxidation and interdiffusion with the substrate due to the longer time and higher temperature, the plasma-arc exposed specimen differs from the furnace exposed specimen (fig. 7(b)) in oxide morphology and composition. The plasma-arc test specimens formed an oxide layer consisting primarily of silicon dioxide (SiO_2) and columbium pentoxide (Cb_2O_5), whereas the furnace specimens formed an oxide rich in the chromium columbate ($CrCbO_4$) phase. As pointed out by Kohl and Stearns (ref. 15), Cr losses from Si-20Cr-20Fe in a dynamic environment can be quite high at about 1300° C and about 1×10^3 N/m^2. A second major difference is the relative absence of oxidation at natural coating cracks in the arc tested specimen exposed to a square wave temperature profile, whereas the furnace slow-cycle temperature profile produced extensive oxidation in natural coating cracks.

EMITTANCE STABILITY WITH REPEATED EXPOSURE

Limited test results indicate unacceptable emittance losses - to below 0.7 - in plasma-arc tests.

A stable emittance of at least 0.7 to 0.8 is extremely important for a reusable, radiative TPS. Columbium alloys coated with Si-20Cr-20Fe have exhibited these desirable traits in furnace reentry simulation exposure (refs. 11 and 16). However, Bartlett, Maykuth, Grinberg, and Luce (ref. 13) have observed a decline of emittance with time during plasma-arc exposures of R512E coated Cb alloys as illustrated in figure 9 for FS-85. Kaufman and Nesor (ref. 17) computed a total normal emittance of 0.58 for R512E coated Cb-752 exposed at 1405° C, 8×10^2 N/m^2, and Mach 3.2. Schaefer (ref. 14) also found low estimated emittance values (0.50 to 0.75) for R512E coated Cb alloys during plasma-arc exposures. These observed decreases may be partially due to errors in temperature measurement and increases in surface catalycity. The latter phenomenon results in higher heat inputs to the TPS and thus has consequences similar to an emittance decrease. There are significant differences in surface oxide chemistry and appearance between furnace and arc tested R512E coated columbium alloys. For example, the surface oxide formed on plasma-arc tested R512E coated Cb-752 is Cb_2O_5 with a reported emittance of 0.7 (ref. 18). This indicates that the emittance decreases observed in dynamic environments are real.

Thus, the as-deposited Si-20Cr-20Fe coating is questionable from an emittance standpoint. Since higher emittance oxides form on atmospheric furnace exposure, a static preoxidation treatment prior to the first reentry exposure might be beneficial. Modification of the coating with higher emittance oxides such as stabilized zirconia might also offer a way to minimize this problem (ref. 19).

MECHANICAL PROPERTY STABILITY WITH REPEATED EXPOSURE

Tensile Behavior

Coated columbium alloy sheet shows only modest, predictable losses in tensile strength and ductility after 100 simulated reentry exposures.

The degradation of the tensile properties of coated columbium alloys is slow and predictable with repeated reentry simulation exposure. The primary mode of strength loss involves substrate consumption as a result of inward silicon diffusion from the coating at high temperature (refs. 10 to 12). In the coating application step, about 0.03 millimeter per side of columbium alloy substrate is converted to the silicide coating for a nominal 0.075-millimeter coating thickness. In 100 reentry simulation cycles, an additional 0.01 millimeter per side is usually consumed through continued inward diffusion. Thus, for a 0.3-millimeter sheet, the coating step should result in a 20-percent loss in strength, while 100 exposures should produce an additional loss of about 10 percent. These estimates are close to actual observations, as shown in figure 10 for R512E coated Cb-752, FS-85, and C-129Y. Note here that, once the major loss in room-temperature ultimate tensile strength occurs because of substrate consumption during coating formation, exposure produces only minor further losses. Also note that the brittle coating does not significantly reduce the ductility of the coated columbium composite - even after 100 simulated reentries. Figure 11 presents a more detailed indication of the effect of test temperature on the ultimate and yield strengths and tensile elongation of the R512E coated FS-85 system (refs. 11 and 20). Above about 1100° C the as-coated specimens are stronger than bare sheet because the coating makes a positive contribution to the strength of the composite. After several reentry exposures, however, this contribution appears to diminish (ref. 13).

Black et al. (ref. 10) found that the efficiency of electron-beam welded and diffusion bonded joints is 100 percent in both the as-coated condition and after 100 reentry simulation exposures. No adverse effect of welded material on coating performance was observed.

Fatigue Behavior

More fatigue data are needed to ensure successful panel design.

The high noise level acoustic environment (to 165 dB) to be experienced by a TPS during Space Shuttle launch makes fatigue life an important design consideration. Only limited fatigue data, however, are available on R512E coated columbium alloys. Bartlett, Maykuth, Grinberg, and Luce (ref. 13) have conducted tension-tension fatigue studies on R512E coated FS-85 and, to a limited extent, on Cb-752 in a variety of conditions. Because of stress concentrations in the test specimen, the majority of the failures occurred at the radius between the ends and the gage section. Thus, while these data may be somewhat conservative, they are felt to represent the material behavior and are plotted in figure 12. Using the universal slopes equation (ref. 21) which gives reasonable predictions out to 10^5 cycles and adjusting for stress range by use of a Goodman diagram (ref. 22) allow a reasonable curve to be fit to the data as shown in figure 12. The scatter in these data is rather large.

In addition, data from two series of reentry simulation tests on rib-stiffened panels subjected to intermittent acoustic vibration (ref. 11) are also presented in figure 12. For each reentry cycle, 28.6 seconds of fatigue cycling at 265 cycles per second were applied. In one series of tests the maximum fiber stress was 69 MN/m^2 - a total stress range of 138 MN/m^2. At this stress level panels were expected to fail in 100 complete simulations. In the second series, the stress level was reduced to a more realistic 52 MN/m^2 - a total stress range of 104 MN/m^2. The higher stress level produced failures after 22 to 100 complete simulation exposures. The second series, with lower stresses, resulted in one panel failing in fatigue after 175 reentry simulations and acoustic exposures, while the remainder survived 200 exposures (table I). Since oxidation at coating cracks can produce localized substrate contamination, environmental interaction can greatly affect fatigue performance. This is especially the case at highly stressed rib edges. From the data available (ref. 13), the FS-85 alloy seems less sensitive to this problem than Cb-752 or C-129Y. However, a larger Cb TPS fatigue data base is needed for design purposes.

Creep Behavior

Cyclic creep strains are several times greater than those in isothermal creep, but proper panel designs can minimize this problem.

Creep data for coated columbium alloys have been accumulated in three ways: (1) conventional isothermal, constant-load experiments, (2) reentry simulation exposures on tensile specimens, and (3) measurements of panel deflection during reentry simulation exposure. The latter two methods are fraught with the difficulties of load-temperature synchronization and are not readily interpreted in terms of conventional creep analysis or transferred to other time-temperature-stress profiles.

Conventional creep measurements on R512E coated columbium alloys have been conducted by Black et al. (ref. 10), Bartlett, Maykuth, Grinberg, and Luce (ref. 13), Holloway (ref. 23), Fitzgerald and Rusert (ref. 24), and North American Rockwell (ref. 25). A composite of their results is presented in figure 13. The figure shows that coated FS-85 is the most creep resistant, while coated Cb-752 is only slightly more creep resistant than coated C-129Y.

Creep elongations were measured during temperature-pressure-stress furnace reentry simulation exposures by Black et al. (ref. 10). The significant point made by these tests is that cyclic creep is more severe than isothermal creep. The same creep extensions after 100 cycles were obtained in only one-quarter and one-eighth of the times required for similar extensions in isothermal tests on C-129Y and Cb-752, respectively. From these results it is apparent that panels must be designed on the basis of plots of cyclic creep strain against time, or conventional plots of creep strain against time must be obtained at various temperatures and a method of life fractions applied. The data in figure 13 indicate that, even for conventional creep data, the data base is too small, especially for coated FS-85.

Temperature-pressure-stress exposures of 2.5- by 10-centimeter rib-stiffened panels (ref. 11) resulted in calculated creep strains of 0.5 percent for R512E coated FS-85, 0.7 percent for R512E coated C-129Y, and 1.1 percent for R512E coated Cb-752 after 100 reentry simulation cycles. In these tests stress was applied by a four-point loading fixture. The panels were held at a maximum temperature of 1315° C for 6 minutes. The stress applied while at peak temperature was about 9 $\mathrm{MN/m^2}$. In similar tests, previously discussed, on 7.5- by 30-centimeter R512E coated FS-85 panels with acoustic fatigue applied at 22-cycle intervals (ref. 11), the creep strain at 100 cycles (table I) was about 0.15 percent, which is within acceptable limits for a TPS panel. Thus, cyclic creep strains can be minimized with proper panel design.

EFFECTS OF COATING DEFECTS - DAMAGE OR LOCAL BREAKDOWN

Defect Growth Rate

Small defect growth rates are observed in static and dynamic exposures up to about 1350° C.

Damage to the coating or local coating breakdowns have different consequences depending on where they occur. Internal damage during assembly or local coating breakdown during service are impossible to detect without panel removal and inspection. However, the pressures found in such locations during reentry are rather low, and thus oxidation at these sites will be less. Exterior surfaces can be damaged by impacts from meteoroids in orbit, stones on the runway during landing, or a dropped tool or bump while the vehicle is on the ground. External damage is easier to detect and must be repaired to prevent eventual ingestion of hot gases during reentry.

The majority of the tests conducted to evaluate the consequences of coating defects have been concerned with retention of structural integrity or with plasma ingestion. Testing has been done for the former in relatively static furnace tests, while for the latter plasma-arc tests (refs. 12 to 14) have been used. As shown in figure 14(a) the defect growth process consists of two parts: metal recession and interstitial oxygen contamination of the substrate. As shown in figure 14(b), under low-gas-flow isothermal conditions, the growth rates of defects are very slow. (Many reentry cycles would be required to produce a defect of significant size.) Compared to through-hole defects with initial diameters of 0.8 millimeter, the surrounding oxygen-contaminated zones after various isothermal and furnace reentry simulation exposures were also relatively small, as shown in figure 15 (unpublished data obtained at the Lewis Research Center by the author). Note that five cycles of reentry simulation at either internal- or external-pressure conditions produce an increase of only a factor of 2 to 3 in the diameter of the oxygen-affected area around an 0.8-millimeter through-hole defect. This factor influences strength and ductility retention, as will be discussed in the next section.

Even in high-mass-flow plasma-arc tests, catastrophic metal recession has not been observed at intentional coating defect sites on coated columbium alloys when exposed to the projected maximum use temperature range of 1250° to 1300° C. This observation is based on tests by several independent laboratories under a variety of flow, gas pressure, temperature, and defect conditions. The data from these tests are summarized in figure 16. Bartlett, Maykuth, Grinberg, and Luce (ref. 13) studied growth rates of intentional defects consisting of either a through-hole, 1 millimeter in diameter, or surface flaws produced by removing the coating down to the substrate at spots having diameters of 1 or 0.1 millimeter. Defect growth rates of the order of 0.01 millimeter per minute were observed up to a threshold temperature of about 1350° C with R512E coated Cb-752 and C-129Y and up to even higher temperatures with coated FS-85. These temperatures are 50° to 100° C above the projected use temperature for coated columbium.

Black et al. (ref. 10), in a second set of plasma-arc shear tests, measured metal recession rates at local coating breakdown sites found at specimen edges. Metal recession rates at these unconstrained sites ranged from 0.027 to 0.11 millimeter per minute for C-129Y and from 0.019 to 0.076 millimeter per minute for Cb-752. These higher rates are attributable to erosion of the growing substrate oxide from the defect site due to shock wave attachment and are thus not typical of anticipated Cb TPS service conditions.

In another study (unpublished data obtained at the Lewis Research Center by J. P. Merutka, S. Levine, and N. Vojvodich) defect growth rates were measured at edge notches, 0.9-millimeter-diameter through-holes and coating removal sites, and impact damage sites (15- to 30-m/sec impact with 0.33-g, 4.4-mm-diam pellets). These tests were conducted in stagnation-model plasma-arc exposures of up to five simulated reentry cycles (ref. 14). Edge notches and through-hole defects tended to fill and plug with substrate oxide. Defects introduced by removing coating from a 0.9-millimeter-diameter area or by impact damage did not develop through-holes in five cycles.

Stein, Bohon, and Rummler (ref. 12) also reported low growth rates at intentional coating defects when specimens were exposed in stagnation-model plasma-arc tests at 1320° C, 2.9×10^3 N/m^2, and a free stream Mach number of 5.

The composite of all these data (fig. 16) shows that low growth rates, similar to those for static or furnace reentry simulation exposure, are observed at constrained defects until a threshold temperature 50° to 100° C above the anticipated use temperature of coated columbium is reached. This figure also shows that, even if a temperature overshoot developed and a growth rate of 0.55 millimeter per minute was reached, during a 10-minute reentry, a nominal 1-millimeter defect could be expected to grow to a hole only 6 millimeters in diameter.

Thus, while coating damage or local coating breakdown can lead to the development of a through-hole or oxygen contaminated zone, for the environmental conditions projected for the Cb TPS the magnitude of this problem on external surfaces is much less severe than originally anticipated.

Mechanical Property Effects

The primary consequence of limited strength and significant ductility losses caused by oxidation at local coating defects in critically stressed areas is a reduction in fatigue life. Panels can survive several reentry cycles with locally damaged coatings.

A number of investigators have studied the effect of intentional coating defects on the mechanical properties of coated columbium alloys primarily by using standard 6-millimeter gage width tensile specimens (refs. 11 and 13). With such specimens the metal recession and area of oxygen contamination at the defect can become a significant fraction of the specimen gage width after as little as one exposure cycle. However, significant tensile strength and limited ductility were retained in Cb-752, FS-85, and C-129Y (ref. 11), as shown in figure 17. The R512E coated FS-85 system appeared to show the best tolerance to intentional defects in both tensile and fatigue tests (refs. 11 and 13).

More important than test data on small gage width specimens, however, is the ability of a panel to retain structural integrity in the presence of coating damage or local coating breakdown. The response of 7.5- by 30-centimeter FS-85 rib-stiffened panels with intentional defects to temperature-pressure-stress reentry simulation profiles with acoustic fatigue simulation applied periodically has been evaluated by Fitzgerald (ref. 11). The fiber stress in the fatigue exposure (69 MN/m^2) was selected to produce panel failure in 100 complete simulations. Under external pressure conditions, the panel with intentional defects shown in figure 18 survived 47 complete simulations and 60 reentry cycles. During the final acoustic exposures, however, the panel failed structurally. A second panel with a defect on the face sheet between each rib survived 77 complete cycles. A third panel with a single defect over the center rib survived 45 complete simulations. From the figure it is apparent that surface defects grow to readily observable dimensions long before they become structurally significant. This has been the general conclusion of all workers in the field. A simple walk-around inspection could detect coating defects and permit field repairs to be made long before serious externally induced structural degradation of a panel occurs.

Internal surfaces of TPS panels are not amenable to such an easy inspection and, in spite of less severe oxidizing conditions, defects may be more harmful. This is illustrated by the response of a panel with intentional defects exposed to reentry simulation using an internal-pressure profile (ref. 11). As shown in figure 19, structural failure occurred within 22 complete reentry simulation and acoustic cycles by crack propagation from the critically stressed rib edge in the defect area. Since significant substrate oxidation does not occur in the internal-pressure environment, small unintentional defects may be difficult to detect - even upon panel removal and visual inspection. Similar behavior was obtained with a pair of corrugation-stiffened 7.5- by 30-centimeter panels given 0.32-centimeter spot defects on the corrugations. One panel failed short of 22 cycles, and the second failed between 25 and 30 cycles; both failed in fatigue (ref. 11).

Defect Detection

At present there is no method for detecting coating defects on the internal surfaces of panels without removing the panels.

As previously mentioned, under low-velocity, external-pressure conditions local coating breakdown is clearly visible on surface members since the yellow-white columbium oxide contrasts well with the darker oxides formed on R512E. However, in plasma-arc exposures, more closely representing Orbiter service, the surface oxides are lighter in color and these failure sites are less clearly visible.

Internal surface defects would have to be discovered by panel removal and more sophisticated examination - perhaps using nondestructive evaluation (NDE) techniques, as will be discussed in the next section. Data accumulated to date indicate such examinations should be conducted about every 20 reentry missions. The problem of premature fatigue failures in full-scale panels having more than a dozen stiffening elements should be less severe than the problem observed in subscale laboratory specimens with only three stiffening elements when one such element suffers a premature local coating breakdown.

A continuing problem could be that of the effect and detection of defects in coated columbium fasteners which have been considered for joining the TPS to the primary or substructure. Designs other than conventional threaded columbium fasteners will be needed to ensure minimal coating damage during assembly and to ease the problem of removal for panel inspection.

MANUFACTURING READINESS

FS-85 PANEL MANUFACTURING

R512E coated FS-85 panels can be readily fabricated.

FS-85 is commercially available in foil, sheet, and bar stock. It is one of the easiest intermediate-strength columbium alloys to produce because of its good fabricability. FS-85 is available with the following guaranteed properties:

Ultimate tensile strength, $\mathrm{MN/m^2}$ (ksi)	550 (80)
Yield strength, $\mathrm{MN/m^2}$ (ksi)	450 (65)
Elongation in 2.5 cm, percent	20
Maximum interstitials, ppm	
Oxygen	300
Nitrogen	100
Carbon	100
Hydrogen	10

Electron-beam or tungsten-inert-gas welding, diffusion bonding, and brazing are all established processes having potential for joining TPS panels. In each case, there is a certain amount of art involved in producing successful joints. For most of the TPS designs considered in past studies, electron-beam welding was favored for simple butt joints because it used relatively inexpensive tooling, was more readily repaired, and resulted in lighter joints because of smaller joint areas (ref. 26). Bend tests show that FS-85 retains excellent bend ductility after electron-beam welding (ref. 11).

The authors consider electron-beam welding to be the primary joining choice except in cases of complex hardware where large surfaces are involved, no subsequent stress relieving is possible, large thickness differences exist between parts, and so forth. The majority of the test panels fabricated in NASA-supported studies (refs. 10 and 11) have been made by electron-beam welding. Thus, electron-beam welding of FS-85 is considered to be a process ready for use in the manufacture of TPS panels.

COATING APPLICATION

The R512E fused slurry coating process has been successfully scaled up to coat 50- by 50-centimeter columbium alloy rib-stiffened panels (ref. 11).

The R512E coating is applied by dipping in a slurry of controlled viscosity. Weight pickup of green slurry should be 19±3 milligrams per square centimeter. Uniformity of coverage is confirmed by NDE inspection with an eddy current device. The edges are given a second coating with slurry by using a paint striper. Then the part is vacuum fired at 1415° C for 1 hour. Uniformity of coverage on flat surfaces is again confirmed by eddy current NDE. Edges are checked by thermoelectric NDE. The coating process has been developed to the point that a process specification has been written. Thus, the coating process is ready for TPS production.

MONITORING PANEL MANUFACTURE AND SERVICE REUSABILITY

While NDE is adequate to monitor panel manufacture, no present NDE technique can detect oxidation attack on internal panel surfaces.

The need for nondestructive evaluation of the coated columbium TPS panels during manufacture falls into two general areas: general structural soundness of the as-manufactured panel and coating compositional and thickness uniformity. Structural soundness of welds, joints, and other parts can be established by more conventional NDE techniques such as transmission radiography and ultrasonic C-scan. Coating compositional and thickness uniformity can be established by eddy-current, thermoelectric, electron emission, and autoradiographic techniques (ref. 27).

One of the primary NDE techniques for confirming coating uniformity involves the use of induced electrical currents or eddy currents. The changes in phase or amplitude caused by the presence of a coating can be calibrated with metallographic measurements to give a good indication of both green slurry and fired coating thickness. This technique was developed by Stinebring and Sturiale (ref. 28) primarily for coated columbium alloys and is almost universally used in silicide coating quality control. The coating thickness at an edge - the critical area - is difficult to determine by this technique, and while thermoelectric measurements are somewhat useful, the problem has been circumvented by applying a second coating to the edges with the paint striping technique (ref. 11).

The majority deficiency in Cb TPS NDE involves the inability to monitor the integrity of the internal structure and fasteners after each flight. This means that some periodic removal of panels, inspection, refurbishment, and reassembly will be in order. The fact that under internal-pressure exposures localized coating breakdown causes only minimal FS-85 substrate oxidation means that such inspections might be necessary only every 20 cycles or so. Some additional NDE development might also be necessary to detect local coating breakdown on external areas since the general oxide scale color formed on reentry may approximate that of oxides formed at defects. Once local coating defects are detected, developed field repair techniques can be applied (ref. 29).

PANEL DESIGN AND FASTENER TECHNOLOGY

Panel and fastener designs need to be optimized.

While a number of panel design configurations have been cursorily examined, the design of coated columbium panels has not been optimized. This factor controls panel weight, maximum panel life, and reliability. A typical design concept is shown in figure 2. Several designs which cope successfully with the thermal expansion of uniformly heated coated columbium panels have been fabricated and tested (refs. 27 and 30). However, accommodation of bowing resulting from nonuniform heating of a panel may require additional consideration. Also, abrasion and wear of the coating as a result of panel vibration at slip joints could be a source of coating failures.

In the same vein, columbium threaded fastener technology is not adequate for this application. Preliminary studies indicated that coated fasteners are not reliable for long time use above 1000° C (unpublished data obtained at North American Rockwell Corporation by L. K. Crockett). The sharp edges, thread crests, and wrenching slots found in normal threaded fasteners lead to coating chipping and oxidation failures. Also, in order to prevent excessive creep, only relatively low levels of torque are applied to threaded fasteners. This leads to loosening under vibratory conditions. Finally, oxidation of internal and external threads can be significant and must be accounted for in preexposure fitting. New fastener designs or approaches, such as superalloy or thoria dispersion strengthened Ni-Cr (TD-NiCr) fasteners, are needed if Cb TPS panels are to be joined to primary structures or substructures at junctions where temperatures above 1000° C are encountered. A recent design employing TD-NiCr fasteners has been successfully fabricated, tested, disassembled, and reassembled (ref. 30). Thus, current fastener technology for a coated columbium TPS appears to be adequate.

RELATIVE WEIGHTS AND COSTS OF COLUMBIUM THERMAL PROTECTION SYSTEMS

Compared with CRSI, currently designed Cb TPS panels have some weight and cost disadvantages.

A direct comparison of TPS weights and costs is quite difficult. First, each organization has a different level of experience upon which to estimate fabrication costs. Second, the designs of metallic and CRSI type TPS panels are still far from being optimized. And third, such estimates do not incorporate consideration of profit, refurbishment and recertification for another flight, percent of the vehicle covered, panel size and shape, scrappage, and installation on the vehicle. Thus, at best, such comparisons give only a rough estimate of production costs, and total costs could be many times these values.

However, because McDonnell-Douglas has had considerable experience with the manufacture of coated columbium hardware and since they have also manufactured CRSI, their cost and weight data are presented in table III. On the basis of cost, both subpanel-with-standoff concepts appear equal, but a weight penalty is charged to the coated columbium system. A distinct cost advantage goes to CRSI if a design concept involving direct attachment to a 150° C primary structure is used.

Other columbium TPS designs using Cb-752 in either tee-stiffened or open-corrugation panel configurations gave panel weights of 28.2 and 29.0 kilograms per square meter, respectively (ref. 30). Although these panels were designed for attachment to a 315° C primary structure, a direct comparison with the FS-85 single-faced corrugation TPS listed in table III should be made with caution because of significant differences in the weight proportioned between panel structure and insulation. In reference 28, a 5-percent saving in structure weight was realized by using Cb-752 instead of FS-85. The Cb-752 alloy is about 5 percent stronger than FS-85 on the basis of strength-density ratio. This alloy substitution results in a 2- to 3-percent saving in total TPS weight. A 10- to 15-percent weight saving for either alloy may be accomplished with further optimization of the panel design (ref. 30). Thus, the current tee-stiffened Cb-752 TPS may be reduced in weight to the point where it is 50 percent rather than 90 percent heavier than the CRSI concept designed for direct attachment to a 315° C primary structure. Similarly, the corrugation-stiffened FS-85 TPS designed for attachment to a 150° C primary structure may be reduced to a weight of 27 kilograms per square meter by optimization of the panel design (10 percent reduction) and use of Cb-752 (3 percent reduction). Further weight reduction (10 percent) by utilization of a rib or tee-stiffened panel design could put the coated columbium metallic TPS on a par with CRSI in the current Space Shuttle Orbiter concept, where the TPS will be directly attached to a 150° C primary structure. Thus, with further effort, only a cost advantage might be retained by mullite RSI. Silica RSI, which has been selected as the primary Space Shuttle Orbiter TPS enjoys an even more favorable cost advantage over a metallic TPS incorporating coated columbium. Use of 144-kilogram-per-cubic-meter (9-lb/ft^3) silica rather than 240 kilogram-per-cubic-meter (15-lb/ft^3) silica, as is currently being considered, could again also restore a weight advantage to CRSI.

SUMMARY OF STATE OF READINESS FOR THERMAL-PROTECTION-SYSTEM SERVICE

The state of TPS use readiness of R512E coated FS-85 and the considerations upon which this evaluation is based are presented in the following tabulation:

Consideration	Readiness	Comments
Reentry survival		
Furnace simulation	To 175 cycles	FS-85 rib-stiffened panel
Arc simulation	To 20 to 50 cycles	Stressed panels have to be tested
Emittance stability	Not ready	25 Percent loss in 10 plasma-arc cycles
Tensile-strength retention	To 100 cycles	-----------------------------------
Fatigue resistance	Limited data, to 175 cycles	More data needed
Creep resistance	To 175 cycles	Controlled by panel design
Oxidation at defects	To 10 to 20 cycles	Checking needed for internal defects
Effects of defects on properties	To 10 to 20 cycles	-----------------------------------
Defect detection	Not ready	Defects on internal panel surfaces cannot be detected
Manufacture		
Alloy	Ready	-----------------
Coating	Ready	-----------------
Joining	Ready	-----------------
NDE	Not ready	Internal coating breakdowns not detectable
Panel-fastener design	Needs optimization	Weight still appears to be high Thermal expansion under nonuniform heating needs additional attention TD-NiCr fasteners work
Cost and weight	-----------------	Costlier than CRSI
Overall	Additional effort still needed	Suitable for limited applications - at least 10-mission reuse

In spite of the many positive aspects of coated columbium, as shown in the table, this system shows disadvantages in weight and cost compared to CRSI for a similar location on the Space Shuttle Orbiter vehicle. In addition, advances in technology are still needed to deal with the following:

(1) Emittance losses in as few as 10 exposure cycles in high-velocity gas streams

(2) Detection of local coating breakdowns on internal panel surfaces

In its current state of development coated columbium is suitable for application in limited areas of the Space Shuttle Orbiter TPS. For example, coated columbium may be competitive with carbon-carbon and ablators for leading edge applications. Solution of the remaining problems could extend the reusability of coated columbium to 100 missions.

Lewis Research Center,
National Aeronautics and Space Administration,
and
U.S. Army Air Mobility R&D Laboratory,
Cleveland, Ohio, April 12, 1973,
502-21.

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TABLE I. - RETENTION OF PANEL STRUCTURAL INTEGRITY AFTER OCCURRENCE OF LOCAL COATING BREAKDOWNS

[Data from ref. 11; panels of 7.5- by 30-cm rib-stiffened R512E coated FS-85; furnace reentry simulation consisted of temperature-pressure-stress and acoustic fatigue cycles; maximum temperature, 1320° C; external-pressure profile; acoustic fatigue, 7600 cycles/reentry cycle; fatigue stress, 52 MN/m^2.]

Cycles to first local coating breakdown (a)	Cycles to structural failure (b)
66	>200
66	↓
110	↓
110	↓
110	>200
110	175

[a] Breakdown occurred at edges.

[b] Creep deflection after 100 cycles, 0.093 cm.

TABLE II. - CYCLES TO FIRST LOCAL COATING BREAKDOWN IN PLASMA-ARC TESTS

Flow mode	Pressure, N/m^2 (torr)	Maximum temperature, °C	Time at maximum temperature per cycle, min	Stress during test, MN/m^2 (ksi)	Substrate	Cycles to first local coating breakdown
Shear (ref. 10)	5.2×10^2 (4)	1315	10	10.3 (1.5)	Cb-752 C-129Y	29, >40 9, 35, >40, >40
Stagnation model (ref. 14)	6.1×10^2 (4.6)	1390	30	--------	Cb-752 C-129Y FS-85	6, 12, 12 12, 12, 18 12, 30, >50

TABLE III. - COMPARISON OF AVAILABLE WEIGHT AND COST ESTIMATES OF COATED-COLUMBIUM AND REUSABLE-SURFACE-INSULATION THERMAL PROTECTION SYSTEMS

[Data from ref. 2; data for vehicle area heated to 1260° C.]

	Attachment to 315° C primary structure		Attachment to 150° C primary structure		Subpanel with standoffs, 150° C tank structure	
	R512E coated FS-85 (a)	Mullite reusable surface insulation (b)	R512E coated FS-85 (a)	Mullite reusable surface insulation (b)	R512E coated FS-85 (a)	Mullite reusable surface insulation on graphite polyimide subpanel (b)
Weights,[c] kg/m^2	30.2	16.0	31.6	24.4	31.6	24.0
Normalized weights	1.9	1.0	2.0	1.5	2.0	1.5
Normalized cost	3.2	1.0	3.2	1.2 to 1.4	3.2	3.2

[a] Weight of panel, support beams, retainer, support struts, links, fittings, and insulation, 31 kg/m^2 (6.5 lb/ft^2); cost of basic columbium thermal protection system, approximately \$32 000/m^2 (\$3000/ft^2) (ref. 30). Cost does not include evaluation, installation, nondestructive testing, profit, or allowance for 100-mission costs.

[b] Mullite density, 240 kg/m^3 (15 lb/ft^3).

[c] Weight includes 1.1 nonoptimum factor for structural members and 1.1 contingency factor for total weight.

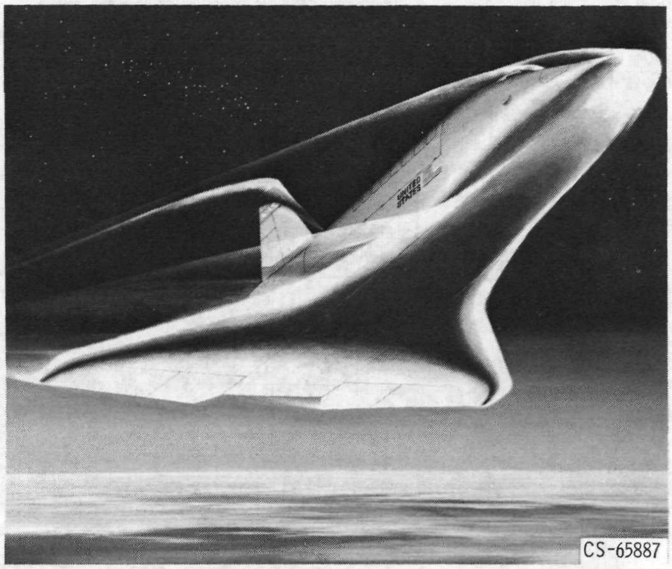


Figure 1. - Artist's conception of Space Shuttle Orbiter on atmospheric reentry. Heat shield area, approximately 1000 square meters; primary opportunity for coated columbium is the 10 to 15 percent of vehicle area heated to 1100° to 1350° C.

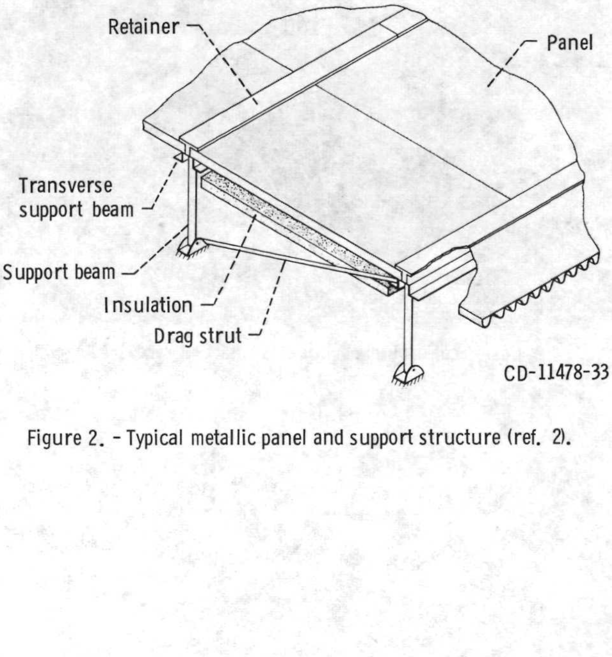


Figure 2. - Typical metallic panel and support structure (ref. 2).

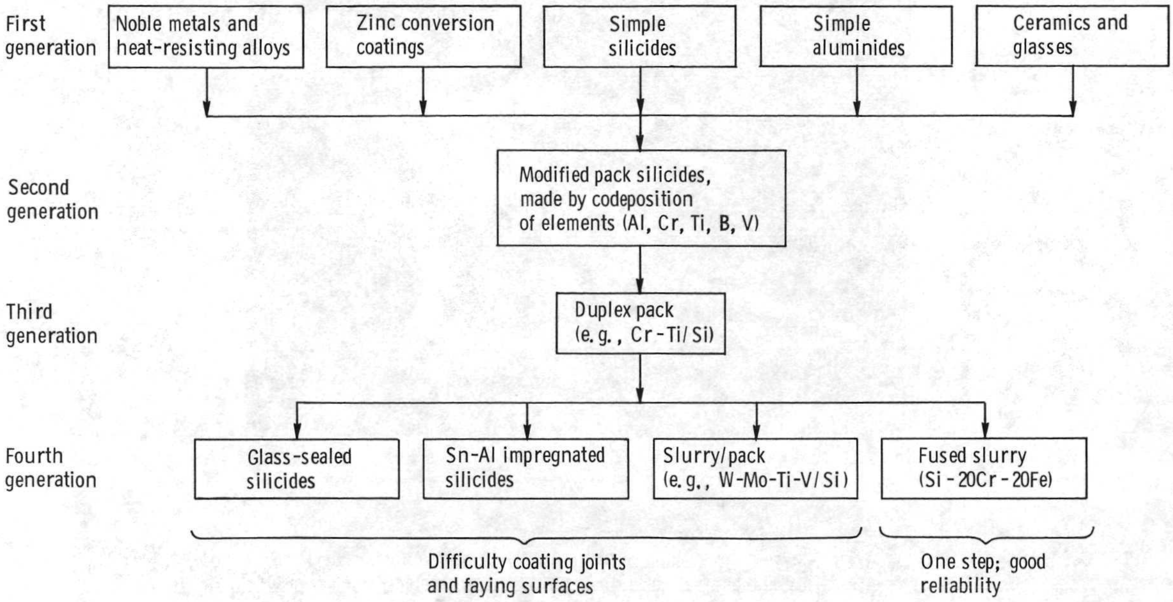


Figure 3. - Historical development of coatings for columbium alloys.

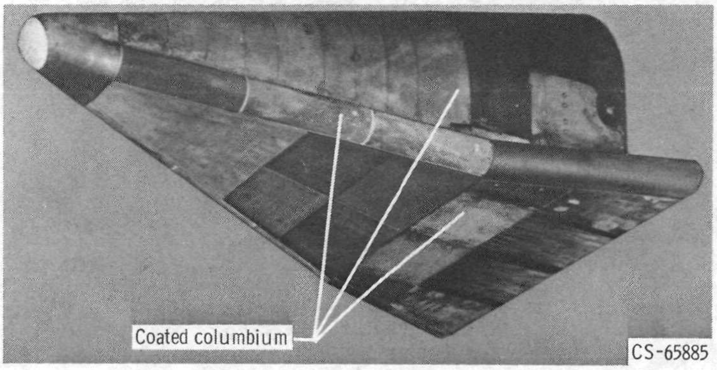


Figure 4. - Coated-columbium components intact on recovered ASSET vehicle.

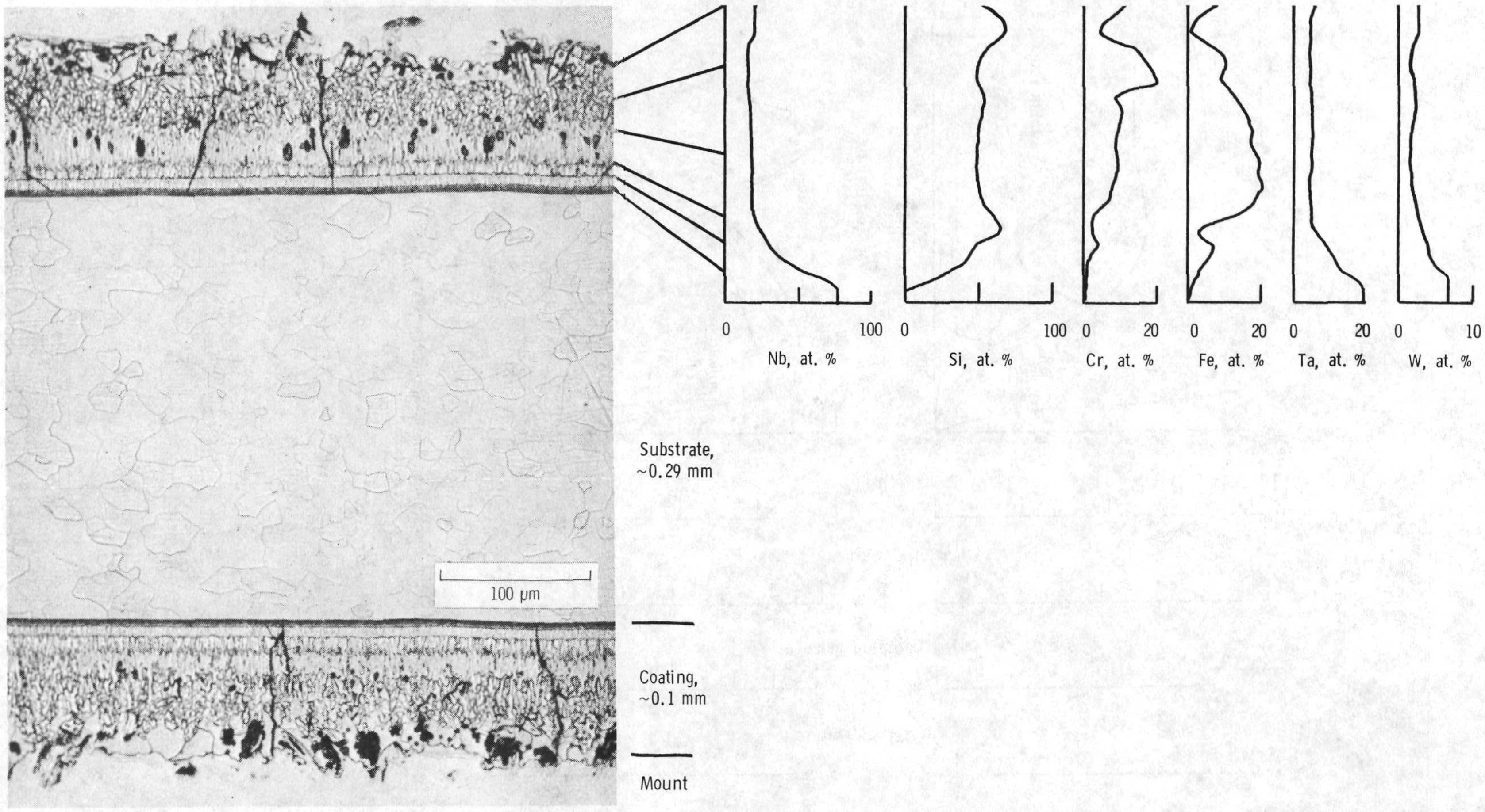
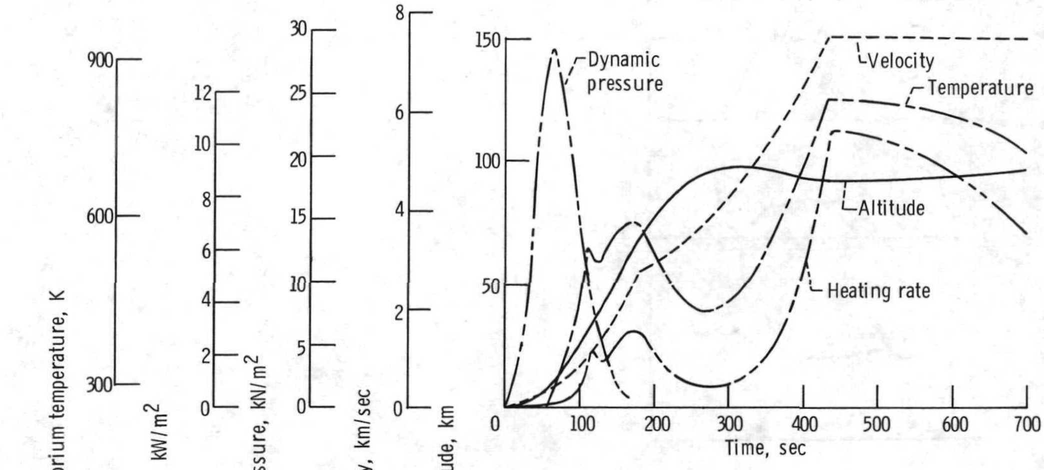
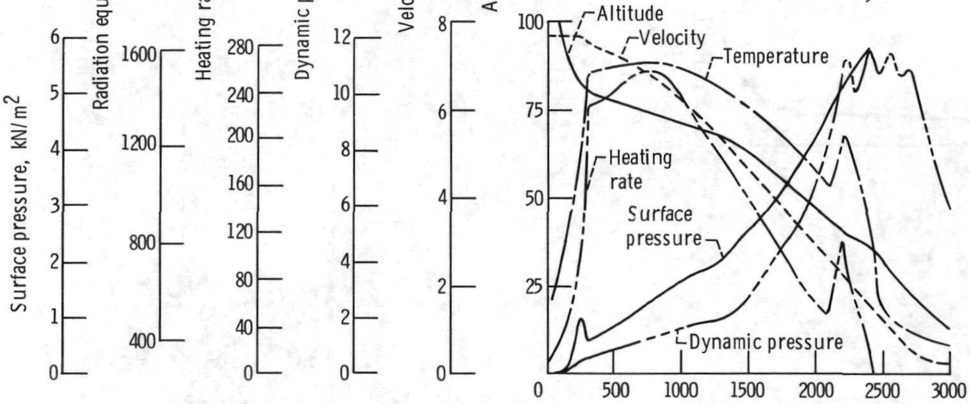


Figure 5. - Microstructure and composition profiles of R512E (Si-20Cr-20Fe) coated columbium alloy FS-85.

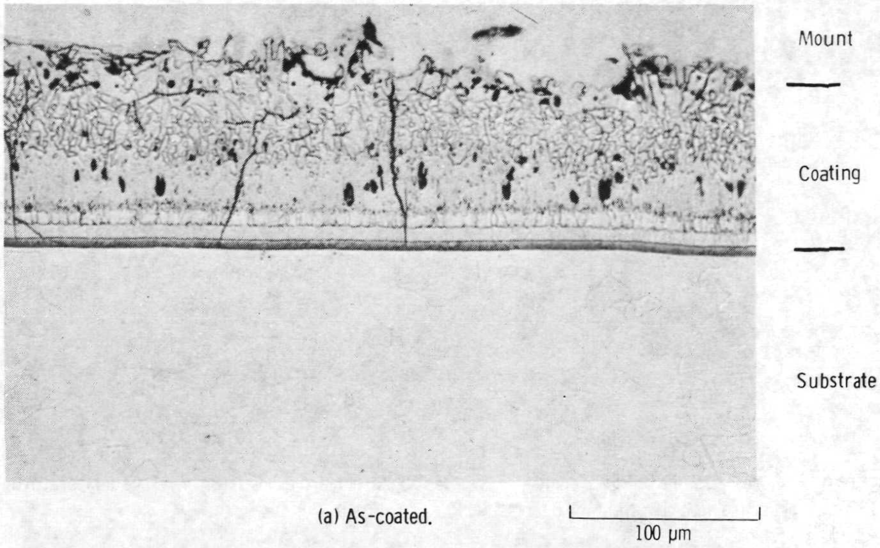


(a) Boost history.

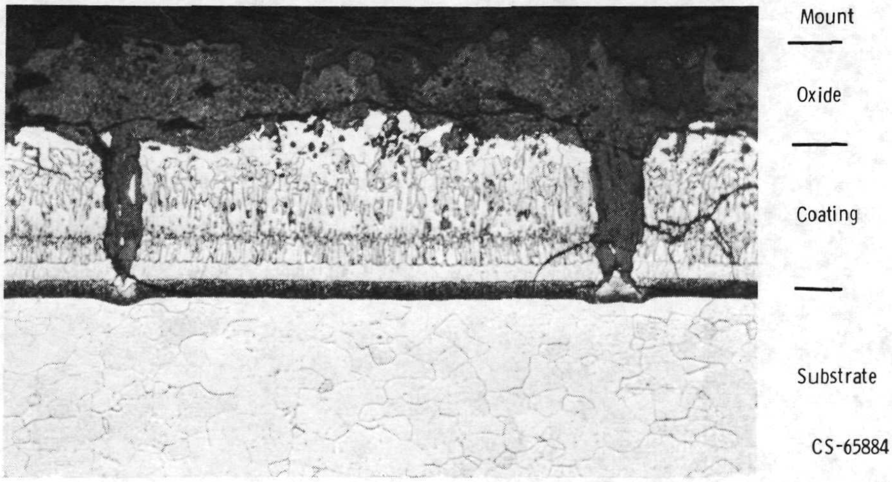


(b) Reentry history.

Figure 6. - Estimated conditions at one location on thermal protection system of conceptual orbiter vehicle (ref. 10).



(a) As-coated.



(b) After 100 external pressure furnace reentry simulation exposures.

Figure 7. - Comparison of microstructure of R512E coated FS-85 after 100 external-pressure furnace reentry simulation exposures with as-coated microstructure.

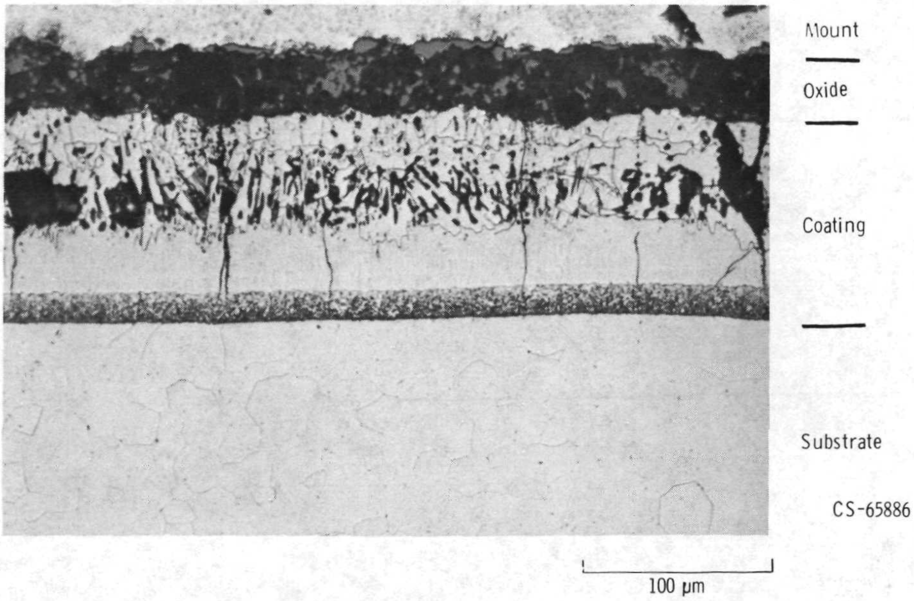


Figure 8. - Microstructure of R512E coated FS-85 after 50 plasma-arc exposures. Average temperature, 1390° C; pressure, 6.1×10^2 N/m^2 (4.6 torr). X250.

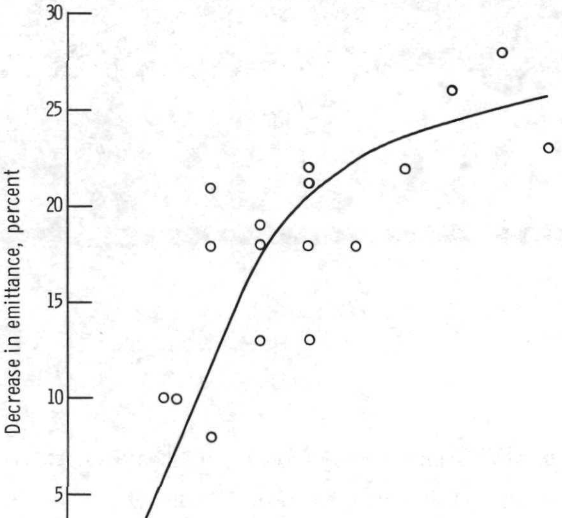
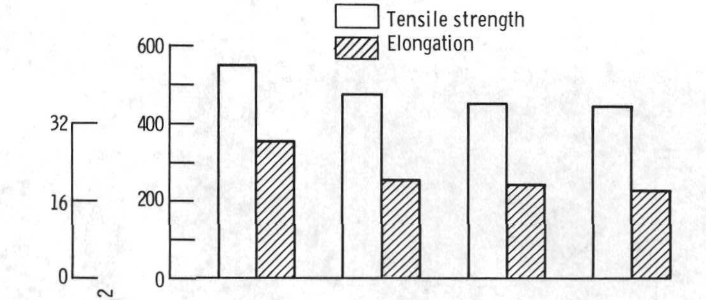
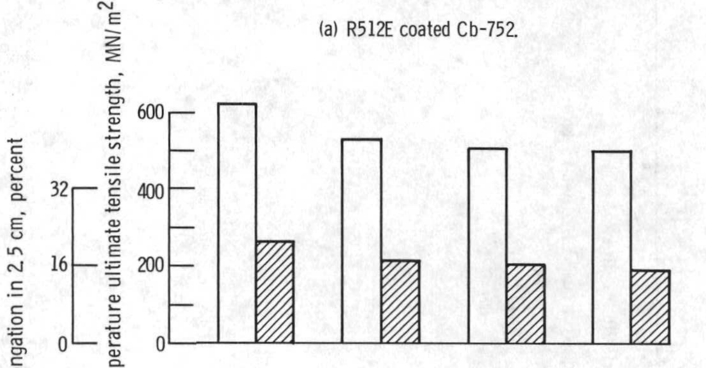


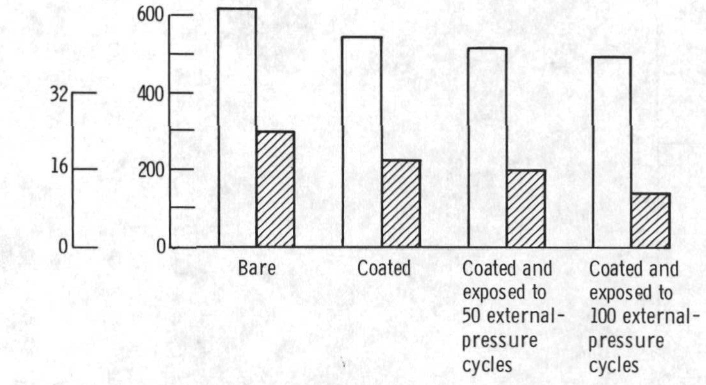
Figure 9. - Effect of 1290° C plasma-arc simulated reentry exposure on emittance change of R512E coated FS-85 (ref. 13). Assumed initial emittance, 0.85; emittance after 10 cycles, approximately 0.63.



(a) R512E coated Cb-752.



(b) R512E coated FS-85.



(c) R512E coated C-129Y.

Figure 10. - Retention of strength and ductility in columbium alloys after coating and furnace reentry simulation exposure (ref. 11). Room temperature ultimate tensile strength based on specimen cross section prior to application of 3-mil coating.

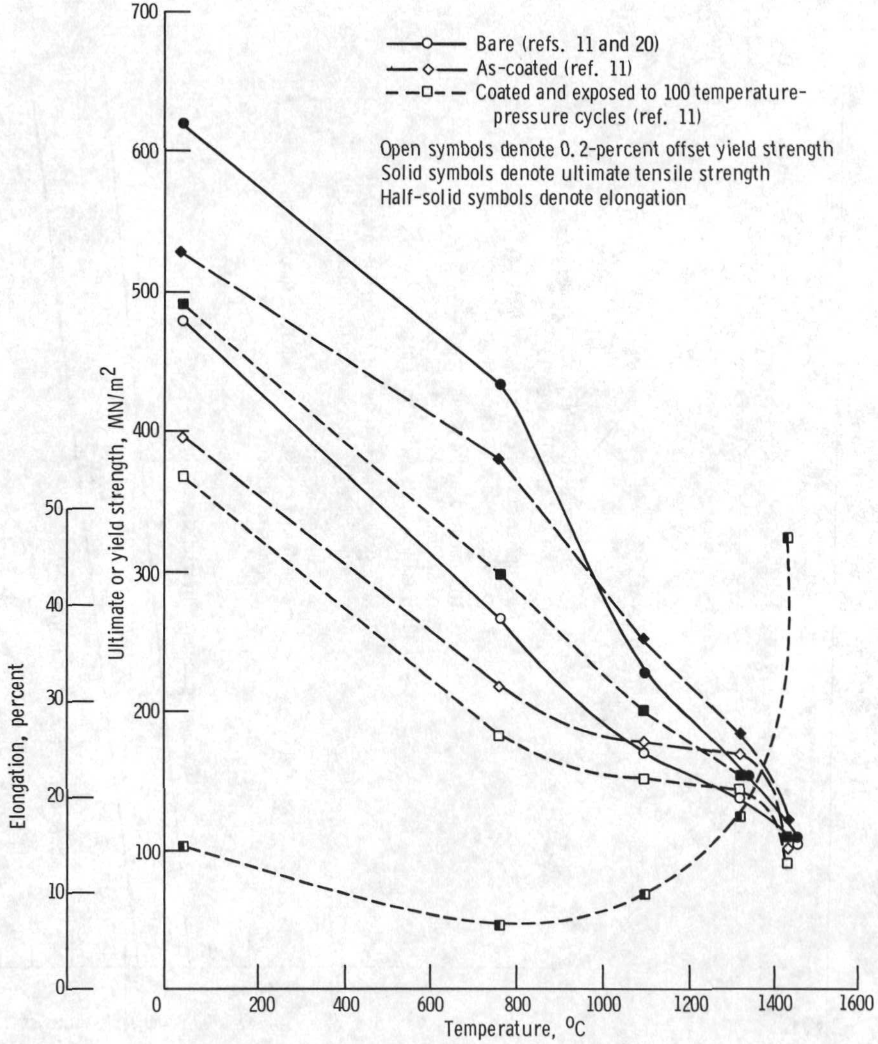


Figure 11. - Variation of ultimate tensile strength of R512E coated FS-85 with temperature and external-pressure reentry simulation exposure.

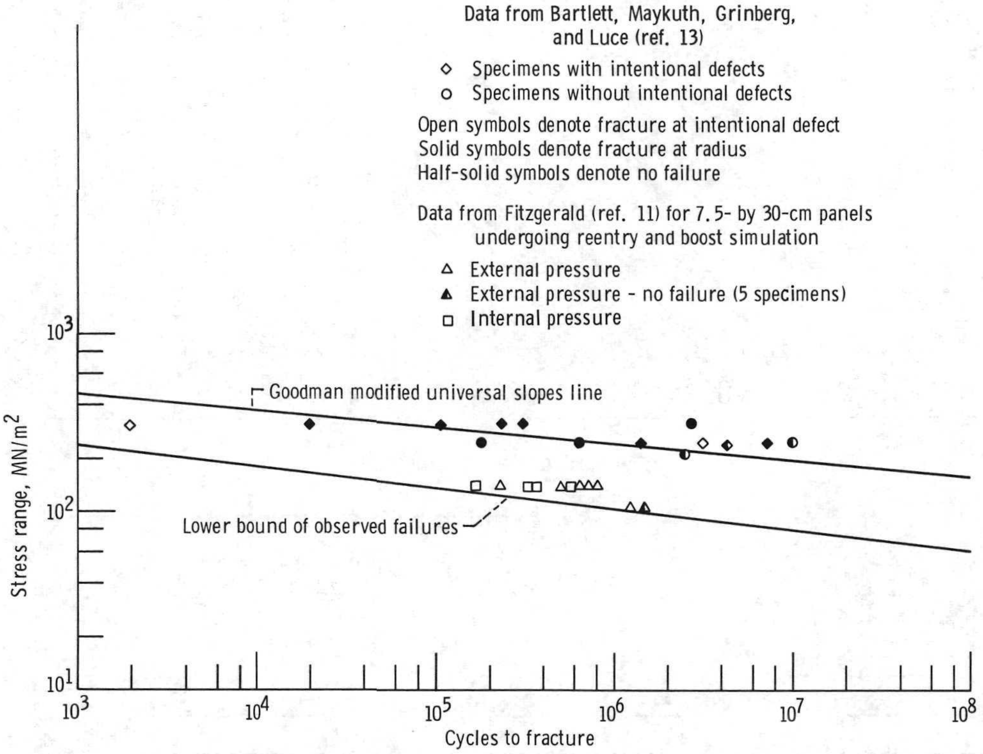


Figure 12. - Fatigue life of R512E coated FS-85.

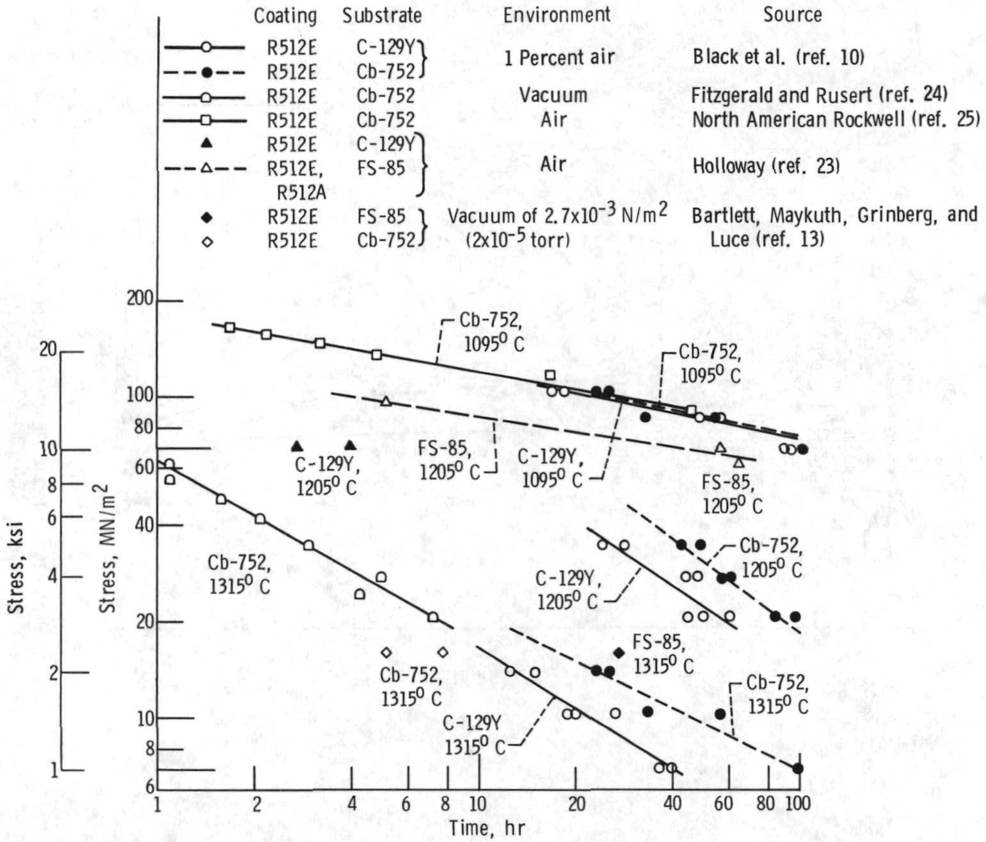
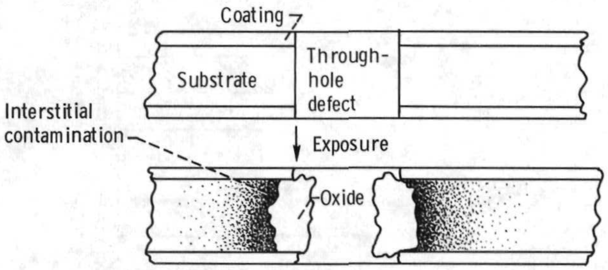
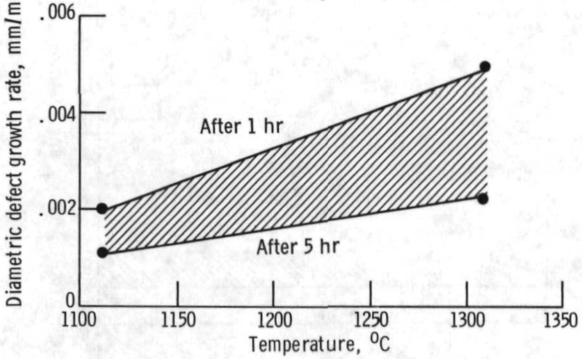


Figure 13. - Summary of isothermal 1 percent creep data for fused-slurry-silicide-coated Cb-752, C-129Y, and FS-85.



(a) Defect growth process.



(b) Growth of 0.8-millimeter through-hole defects in R512E coated FS-85. Isothermal conditions; pressure, 1.3x10^3 N/m^2.

Figure 14. - Defect growth in coated columbium alloys.

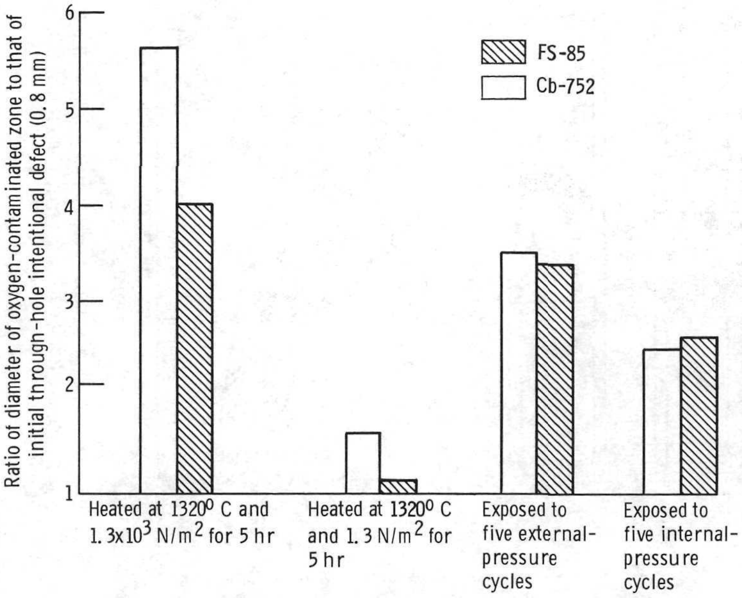


Figure 15. - Contamination-zone growth at through-hole defects in R512E coated FS-85 and Cb-752.

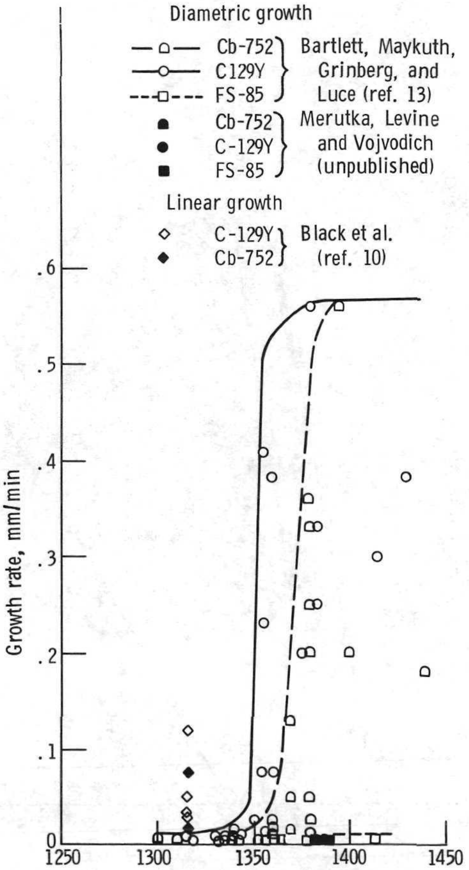
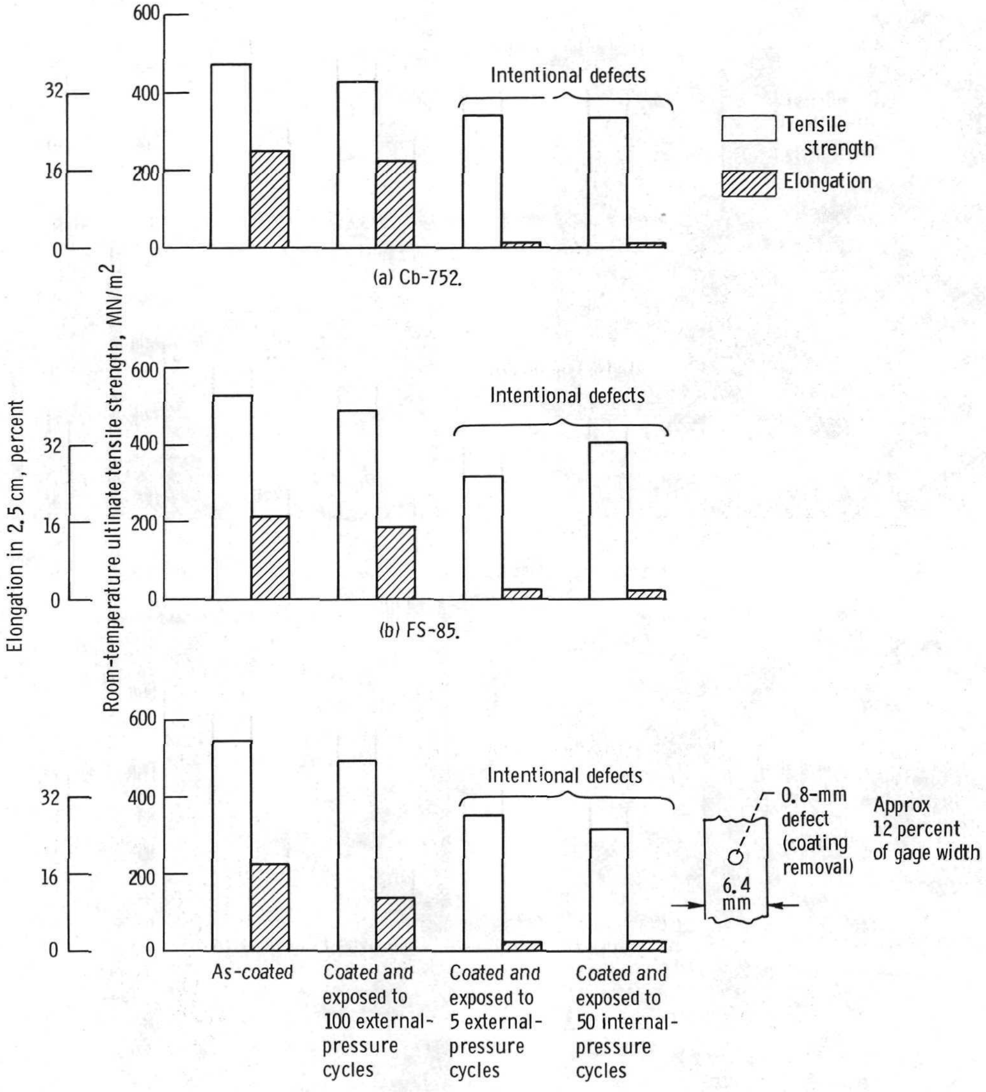


Figure 16. - Effect of plasma-arc exposure temperature on coating detect growth rate.

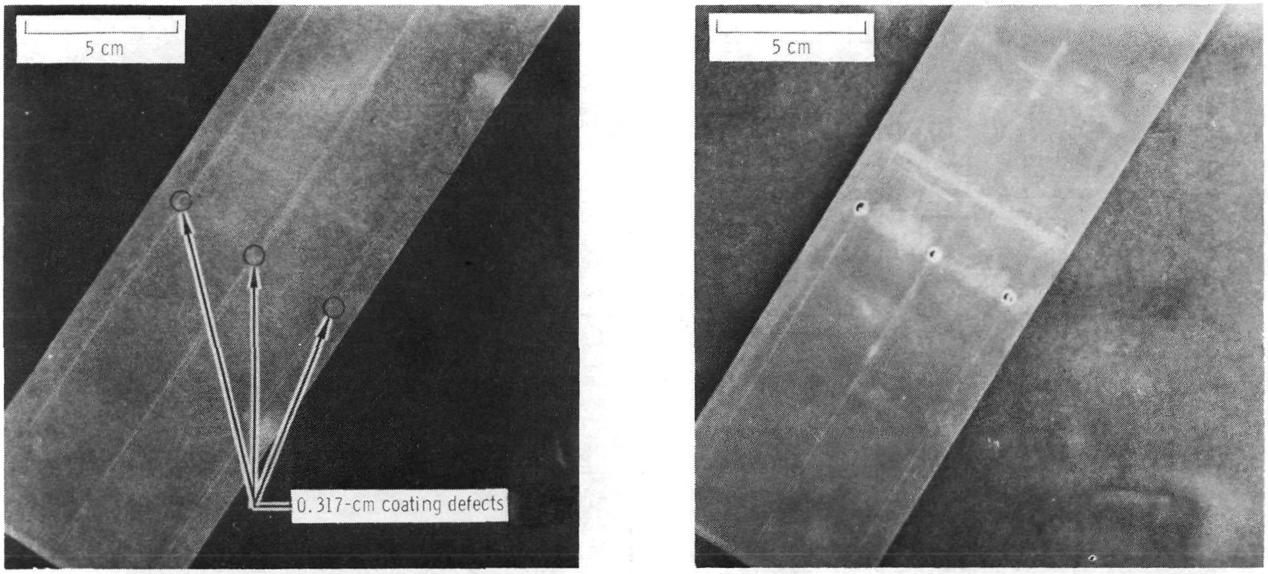


(a) Cb-752.

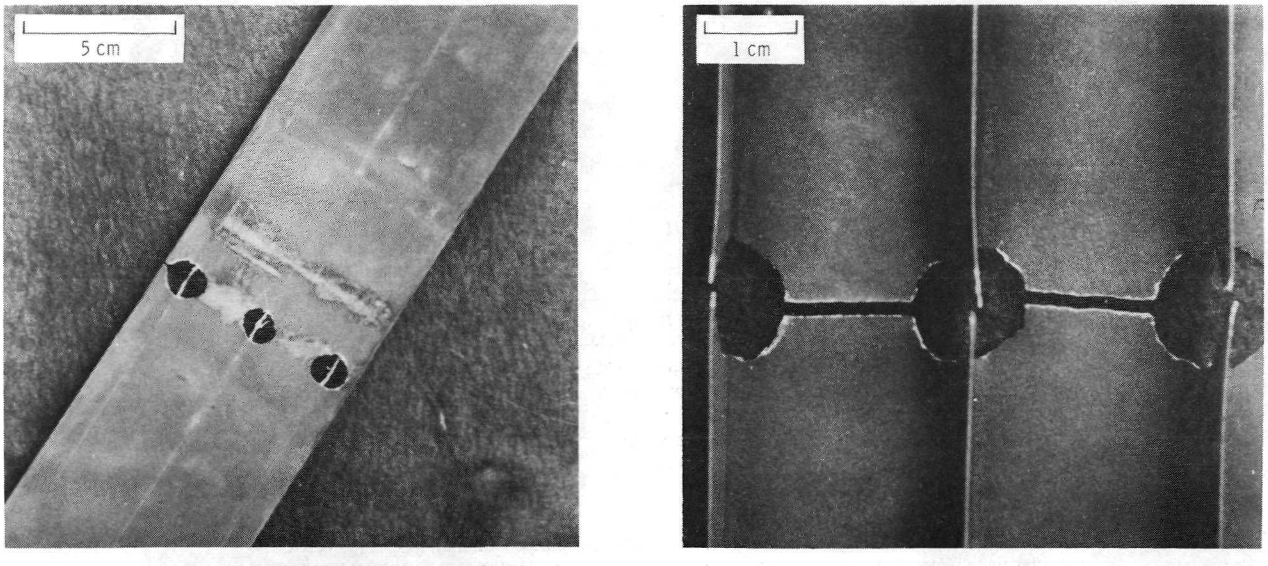
(b) FS-85.

(c) C-129Y.

Figure 17. - Tensile strength and elongation of R512E coated columbium alloy specimens with intentional defects after exposure to reentry simulation compared to tensile strength and elongation of as-coated and exposed specimens without intentional defects. Room-temperature ultimate tensile strength based on original cross section prior to coating.

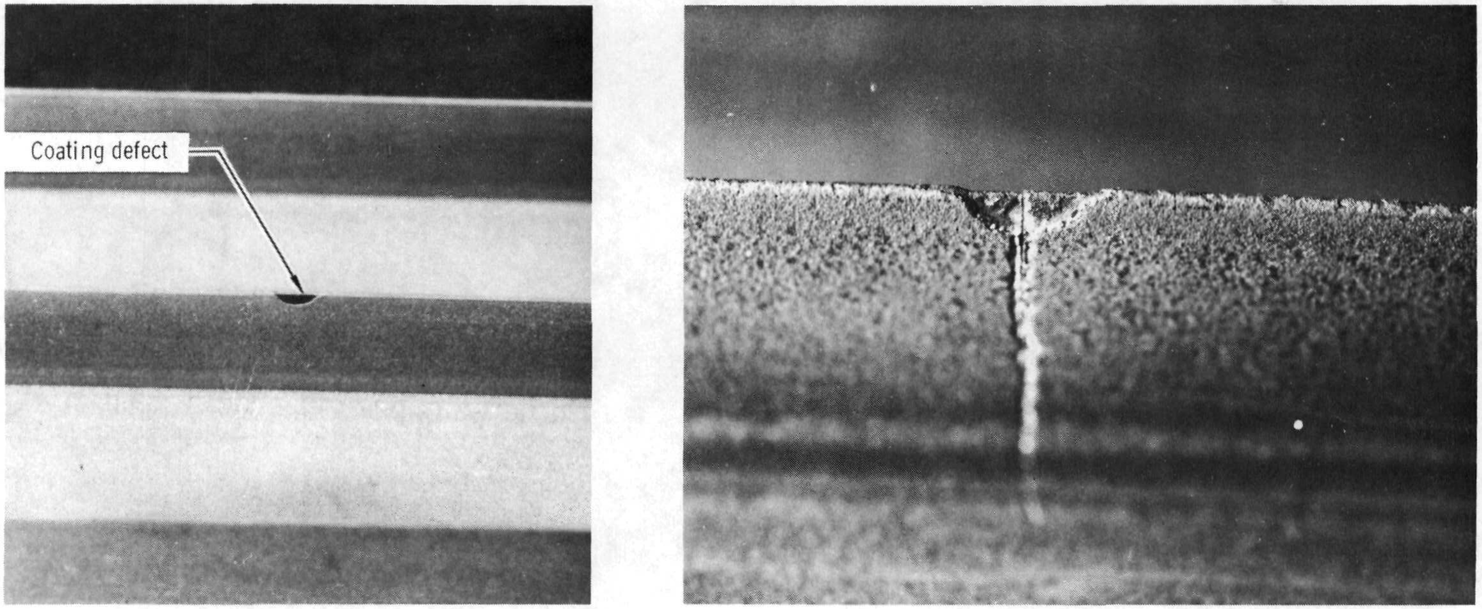


CS-65896 (a) Prior to testing. (b) After 16 reentry cycles and 16 acoustic cycles.



(c) After 60 reentry cycles and 47 acoustic cycles. (d) After 60 reentry cycles and 60 acoustic cycles.

Figure 18. - Condition of R512E coated FS-85 panel with intentional defects after external-pressure - temperature-stress reentry simulation plus boost acoustic fatigue simulation (ref. 11).



(a) Prior to testing.

(b) After 22 reentry cycles and 22 acoustic cycles.

CS-65895

Figure 19. - Condition of R512E coated FS-85 panel with intentional defect after internal-pressure - temperature-stress reentry simulation plus boost acoustic fatigue simulation (ref. 11).

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