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SEG-TDR-64-19

UTILIZATION OF REFRACTORY METALS ON THE X-20A (DYNA-SOAR)

WILLIAM COWIE

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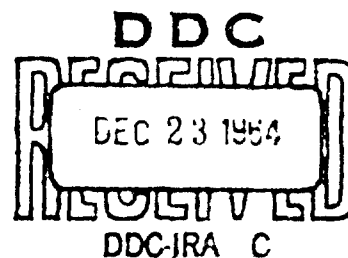
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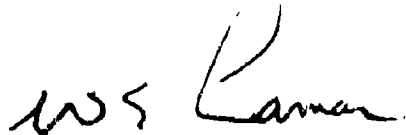
FOREWORD

This in-house report was prepared by personnel of the X-20 Engineering Office as a summary of the developmental efforts expended in utilizing refractory metals on the X-20A. The Boeing Company, Seattle, Washington, accomplished the work under Contract AF 33(657)-7132. William Cowie was Project Engineer for the Glider Division of the X-20 Engineering Office at Research and Technology Division, Wright-Patterson Air Force Base, Ohio. The work period reported is from May 1960 to the termination date of the program, 10 December 1963.

ABSTRACT

The utilization of coated refractory metals as heat shields and leading edges on the X-20 is discussed. Peculiarities and history of the materials used, designs, developmental tests, and problems are emphasized in this resume. Molybdenum alloys Mo-.5Ti and Mo-.5Ti-.07Zr, columbium alloy D-36, and silicide coatings are discussed in relation to their applicability and effectiveness in an X-20 re-entry environment. The practicality of a heat-protection system capable of resisting 3000°F for long periods of time is demonstrated.

This Technical Documentary Report has been reviewed and is approved.



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INTRODUCTION

The attempts to reconcile the characteristics inherent in refractory metals with the conditions imposed by an X-20 type re-entry environment represent a pioneering effort. The purposes of this report are to present a general summary of that effort, give highlights of specific problems, and outline recommendations that could influence future efforts to use refractory metals in re-entry environments.

The general philosophy toward the thermal protection system for the X-20 glider was that the major portion of the re-entry heat was to be rejected by the process of radiation. Aside from the nose cap, the X-20 "hot" areas (areas that exceeded 2000°F) would be the wing and fin leading edges, the bottom surface of the glider, and the control surfaces. If the general philosophy were to be applied successfully, such hot surfaces of the X-20 glider had to withstand high temperatures approximately one hour and have high-emittance characteristics.

Any material used in the thermal protection system for the hot areas of the X-20 would have to meet the following requirements which were believed necessary at the 1960 initiation date:

1. Have good resistance against oxidation at temperatures of 2700°F during re-entry;
2. Have reasonable strength at the use temperatures (at least 2700°F);
3. Have a highly emissive surface ($\epsilon = 0.7$, or better);
4. Have a good modulus of elasticity at room temperature and at elevated temperatures;
5. Thermal expansion should be low;
6. Creep characteristics should be good through the use temperatures, for at least one hour;
7. Have the ability to endure repeatedly the design environment with retention of required physical or mechanical properties;
8. Have low density;
9. Be capable of good elongation at room temperature through use temperature.

Early in the program, available candidate materials, along with oxidation-protective coatings, were screened to ascertain how practical their use would be to withstand the X-20 environments. All of the evidence indicated that refractory metals, in conjunction with a silicide oxidation-protective coating, would be a logical choice.

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MATERIALS

Of the refractory metals available at the beginning of the X-20 program, Mo-.5Ti (a molybdenum alloy), when coupled with a silicide oxidation-protection coating, was the metal that met nearly all of the requirements of an X-20 thermal protection system. Mo-.5Ti exhibited sufficient strength and possessed adequate creep resistance at the intended X-20 use temperatures. On the other hand, Mo-.5Ti possessed apparent drawbacks. It had characteristics of brittleness at room temperature, and it was difficult to machine, weld, and form without heated tooling. The adverse properties of Mo-.5Ti that would affect fabrication are given in Table 1 where it is compared to aluminum.

As the program proceeded, and despite the inherent brittleness and poor forming characteristics, good results were obtained when developmental components made of Mo-.5Ti were fabricated and tested. Appreciable knowledge was gained on the properties and parameters of reliability of the oxidation-protection coating. Early tests of the silicide protective coating on Mo-.5Ti pointed out the importance of a requirement for rounding the edges of all parts before the application of the first coating. The early silicide coatings were applied by the atmospheric retort process shown in Figure 1. This process did not lend itself to production assemblies or methods due to the rather long time-periods required for the application of the coating.

In December 1962, Boeing Company and the Air Force decided to utilize another alloy of molybdenum (Mo-.5Ti-.07Zr), TZM, in lieu of the Mo-.5Ti alloy. In the uncoated state, TZM possessed obvious advantages, as illustrated in Figures 2 and 3. In addition to the effort to use TZM, at this time, Boeing incorporated into the program a new production coating facility that they had developed. The facility, shown in Figures 4 and 5, was the "fluidized bed," which contributed substantially to advancing the state-of-the-art in coating applications. The use of the fluidized bed substantially reduced the amount of time necessary to apply a coating and made a more uniform and reliable silicide coating system possible.

Paralleling the developmental effort on molybdenum and its silicide coating, was Boeing's considerable effort on another refractory alloy - - columbium. Columbium was considered to be ductile, even after coating, and was rated close to aluminum in the ease with which it could be fabricated. However, there was no coating available for use with Columbium at temperatures above 2500°F. In addition, columbium exhibited low strength and poor creep characteristics at temperatures above 2500°F. Of the columbium alloys that could be made available in the time-period required, D-36 was selected as a prime alloy for development. The selection of D-36 was based primarily on the ease with which the material accepted a silicide coating and on the relatively low density of the material.

By early 1962, the contractor had developed a high emittance top coat that could be applied over the protective silicide coating of the columbium. This development was again a substantial step forward in the state-of-the-art for oxidation-protective coatings for refractory metals and resulted in a lowering of columbium temperatures, relative to those without the top coat, by 200°F.

A topcoat was also developed for the molybdenum alloys. However, since the molybdenum system was already adequate from a requirements point of view and additional funds were not available, implementation for the use of the topcoat for molybdenum was curtailed and eventually stopped.

By the end of 1963, because of the tremendous strides that were made in the state-of-the-art of refractory metals and their coatings, the capabilities of the available systems of molybdenum and columbium materials to withstand the X-20 re-entry environment were firmly established. As a matter of interest, Figure 6 illustrates those areas of the X-20 where TZM and D-36 could be used at the date of contract cancellation. In 1960, all of these areas utilized the Mo-.5Ti molybdenum alloy.

More specific information on materials may be found in Reference 1.

Table 1

Difficulties of Fabrication Molybdenum (Mo-.5Ti) Vs. Aluminum	
Process	Complexity Factor (AL = 1 as a base)
Cleaning	5.0
Chemical Blanking	10.0
Machining	---
Shearing	---
Abrasive Sawing	2.5
Bandsawing	2.6
Edge Milling	2.5
Radiusing	2.0
Coating	3.5
<u>Fusion Welding</u>	
Mechanized	3.0
Manual	---
Repair	---
Forming: (Brake; Roll; Joggle; and Hydro Press)	3.0
Stretch	1.5
Hammer and Draw	3.5
<u>Fastening</u>	
Drilling	5.0
Reaming	7.0
Dimpling	3.0
Riveting (Coated)	6.0

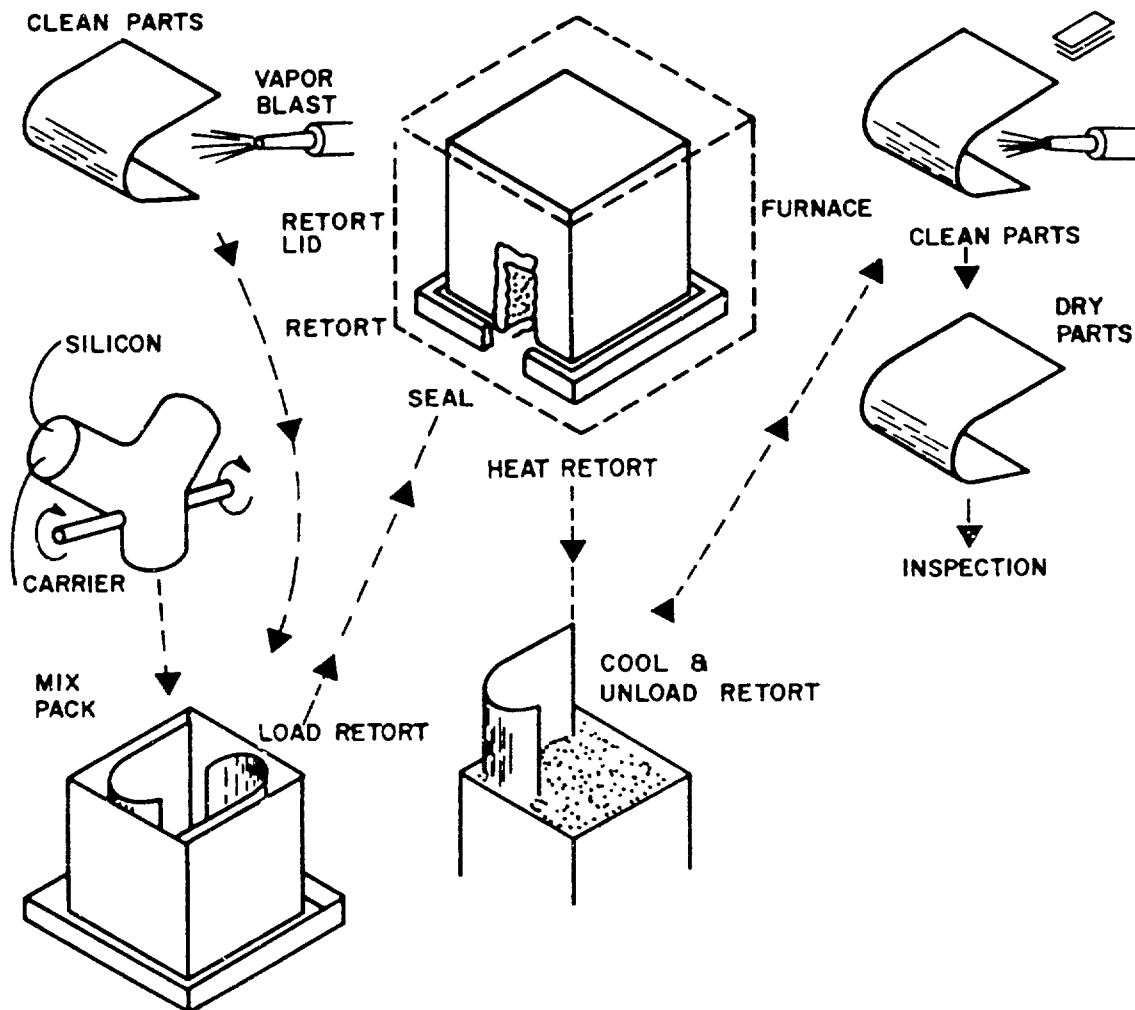


Figure 1. Atmospheric Retort Process

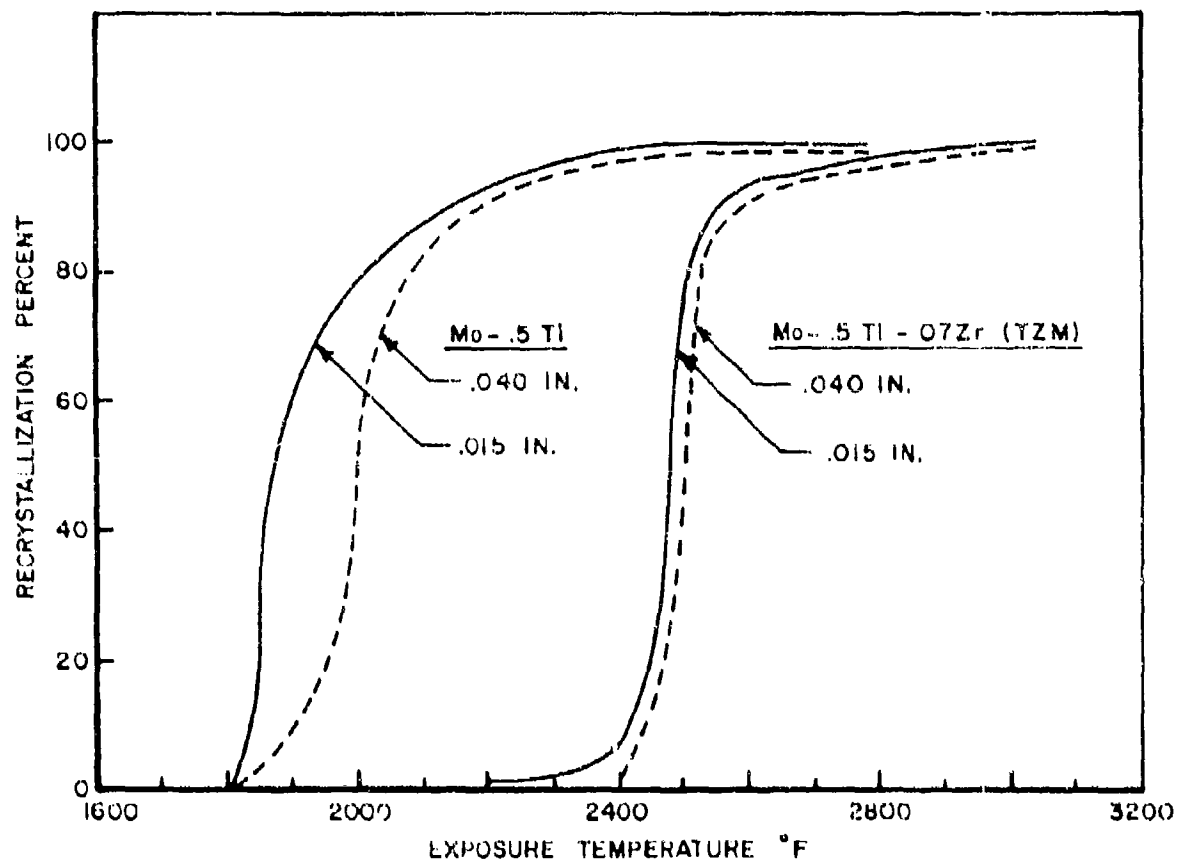


Figure 2. Mo-.5Ti and TZM Recrystallization Curves Based on One-Hour Exposure

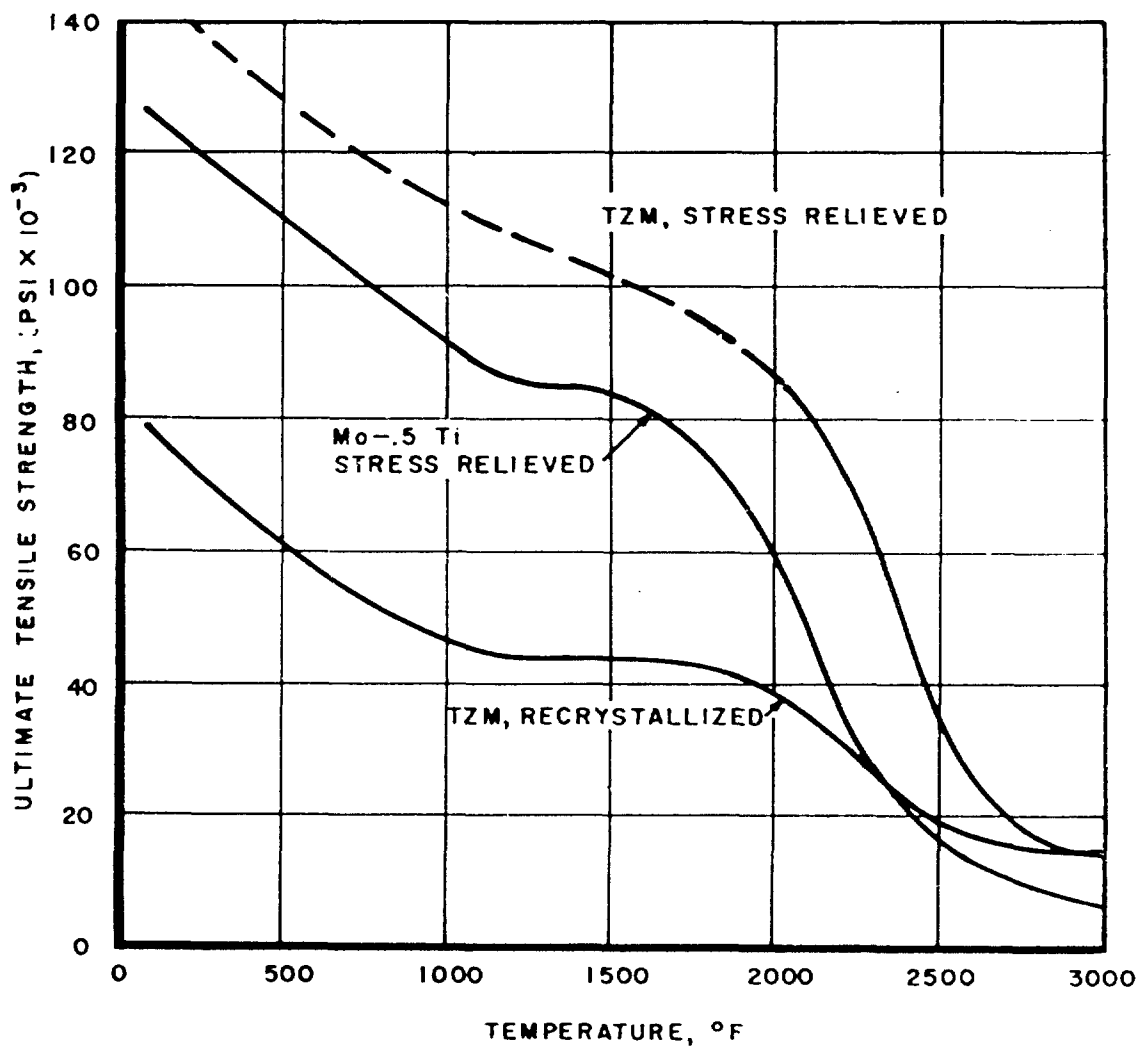


Figure 3. Tensile Strength of Mo-.5Ti and TZM in Vacuum

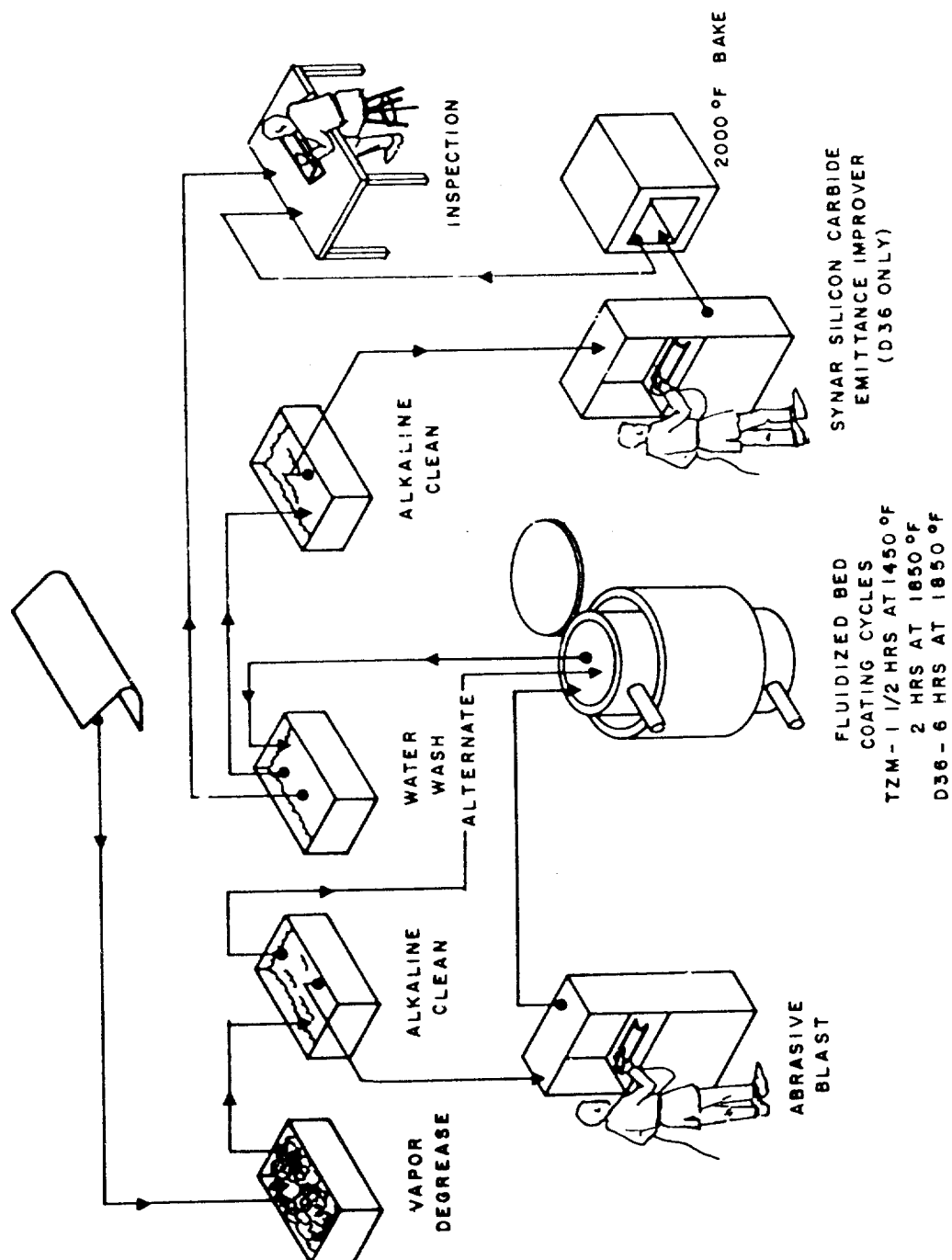


Figure 4. Fluidized Bed Process for Coating Refractory Alloys

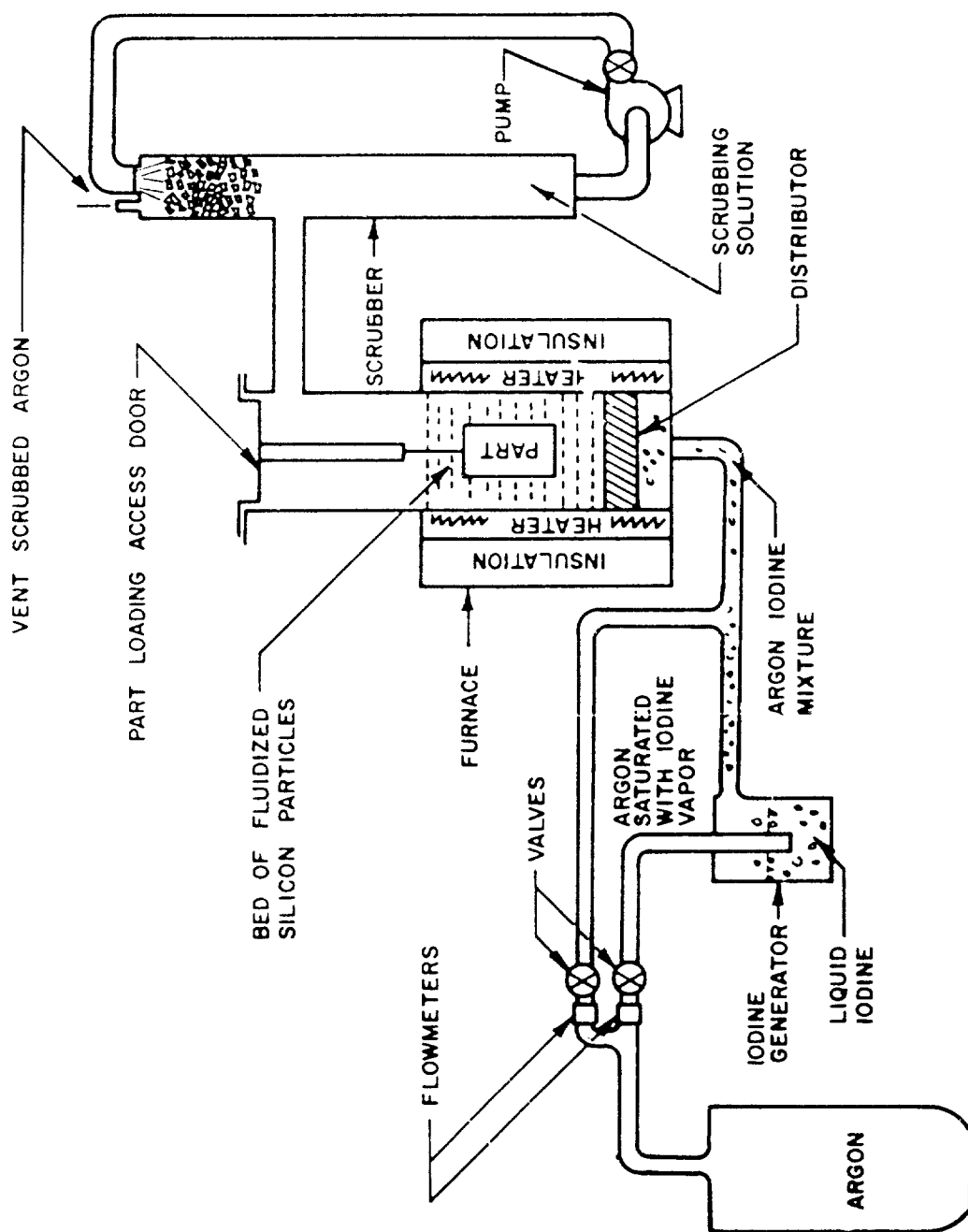


Figure 5. Fluidized Bed

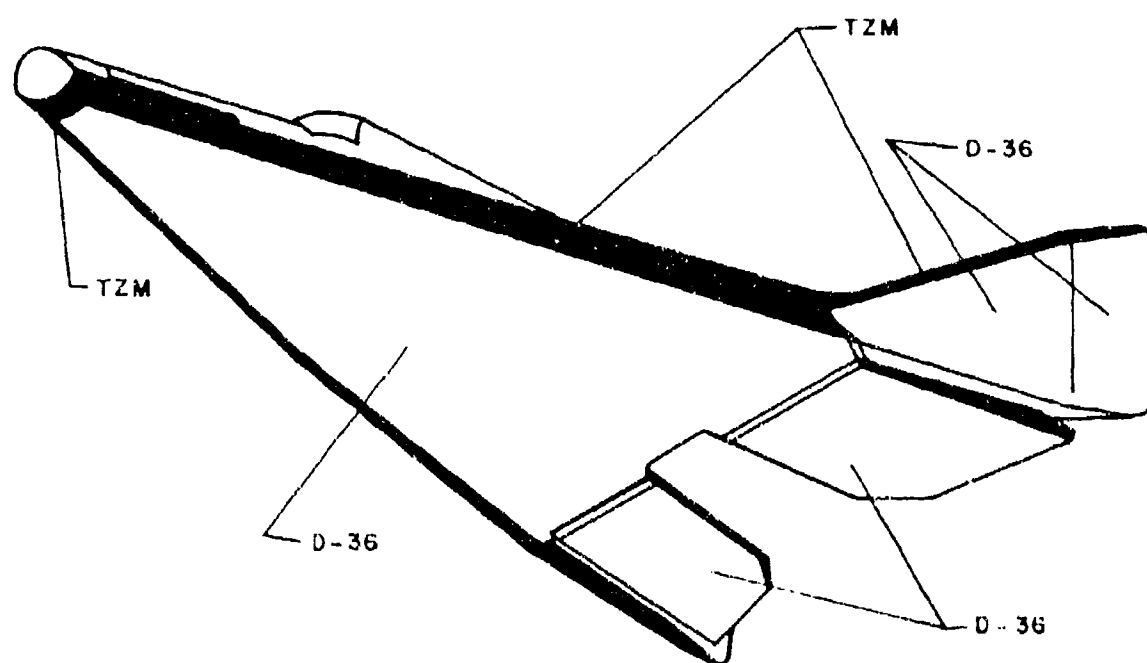


Figure 6. Areas on X-20 Where TZM and D-36 Could be Used

NOTE: Due to the high temperatures expected, the use of D-36 in lieu of TZM on the bottom surface of the elevon was questionable.

HEAT SHIELD

HEAT SHIELD AND INSULATED PANEL DESIGN

Insulated panels, composed of a refractory metal surface, a high-temperature insulation, and thin-gage Rene 41 corrugations were utilized in all areas of the X-20 glider where temperatures would exceed 2000°F. The external portion of the insulated panels was made of small, overlapped, coated, refractory metal tiles mounted on refractory metal clips in a manner that would minimize thermally induced stress. These clips were riveted to the external surface tiles. The assembly of attachment clips plus the tile is commonly referred to as a heat shield. The heat shields were bolted to Rene 41 super alloy corrugations that were protected by a 6 lb/ft³ "Q" felt insulation. This insulation was sandwiched between the heat shield and the corrugation. A typical insulated panel is shown in Figure 7.

Aside from the requirement to cool by radiation, the heat shield portion of the insulated panel was used to contain and protect the insulation from the high-velocity air stream. Figure 8 depicts the basic concept of the heat shield that was initially developed by Boeing so that preliminary thermal work and structural design could proceed. This concept was adaptable to material systems of either molybdenum or columbium.

Because of the large areas of the X-20 surface that had to be covered by heat shields, a minimum-weight design for a flight-type article was required. Considerable effort was expended in the design area to define a light-weight heat shield. Figures 9 through 15 are but a few of the heat shield designs that were investigated. Figures 16a and 16b show a design generated by the X-20 Systems Program Office. In addition, limited effort was expended on possible light-weight refractory non-metal heat shields. Some of these designs are shown in Figures 17 through 20. The final production design concept is shown in Figure 21.

In the final production design concept, the surface tiles possessed slightly outward-facing corrugations (approximately .040 in. in depth) that were intended to add stiffness. The refractory standoff clips consisted of four support legs each. These legs were angled to increase their length and as a result, increase the conduction path, thereby reducing heat shorts. The legs of the clips were spread so as to transmit more effectively the local surface air loads and to minimize the concentration of radiant energy to the superalloy corrugations. A typical clip design is shown in Figure 22.

Important factors, which influenced the design of the X-20 heat shields, were: (1) location of heat shield; (2) emittance values; (3) relative thermal growth between the Rene 41 corrugations and the refractory heat shields; (4) aerothermal requirements.

- (1) The heat shield location on the vehicle was important because the insulation requirements, temperatures, and pressures differed from area to area.

Since the coating and, therefore, the emittance values, were changing during the coating development work, temperatures and allowables were adjusted and readjusted. Typically, conservative lower emittance values (curve 1) of Figures 23 and 24 were used in heat shield design due to local unexpected hotspots; whereas the higher values (curve 2) were used for calculating internal temperature since the net effect of external local hotspots would be negligible internally.

- (3) The relative thermal growth between the Rene 41 corrugations and the refractory heat shields was considered because it particularly affected the gaps between the heat shields. A peculiar characteristic of the X-20 insulated panel system was that during a re-entry, and at maximum heating, the heat shields would grow together; while later, during the re-entry, when the heat shields were cooler, the peak heat flux to the Rene 41 corrugations would occur causing the heat shield gaps to open. This was due principally to thermal expansion through the Q-felt insulation and the relative thermal growth characteristics of the two materials.
- (4) Aerothermal requirements, such as maximum permissible sizes of protrusions, steps, gaps, and leakage, were considered. These requirements are specifically pointed out in Reference 2.

Generally speaking, the majority of the columbium heat shields were designed for the re-entry condition. Molybdenum heat shields were designed primarily for the boost and/or approach phase of flight.

DEVELOPMENT OF HEAT SHIELD AND INSULATED PANEL

The development of the insulated panel was centered around the structural evaluation of a panel design that incorporated the following materials: heat shield and support clips -- molybdenum (Mo-.5Ti) and columbium (C-36) (Figure 25); insulation -- stabilized Q-felt (quartz fiber); and support panel -- Rene 41. The object of this program was to develop a coated, refractory alloy thermal protection system that would be capable of withstanding temperatures up to 3000°F. Concept studies were centered principally on the heat shield shown in Figure 25.

A series of 6-inch by 6-inch and 12-inch by 13-inch Mo-.5Ti insulated panels was built and tested under simulated Dyna-Soar thermal and acoustical environments. These environments (Figure 26) consisted of a simulated re-entry, three cycles from room temperature to 2750°F, and a thermal exposure to 3000°F.

Of significant note during the testing was the problem of measuring temperatures of silicide-coated refractory materials at temperatures above 1800°F. This was solved by the development of a platinum-platinum rhodium thermocouple attached to a platinum disc. This disc was flame-sprayed with alumina to prevent reaction with the silicide coating.

To establish the effect of material recrystallization on the ability of the panel to resist sonic excitation, the contractor subjected additional panels to an acoustical environment of 152 db overall for 5 minutes, then to 2750°F for 15 minutes, followed by 55 minutes at 152 db overall, then 30 seconds at 157 db overall and 162 db overall. The acoustical acceleration was random, involving frequencies from 25 to 7000 cps.

Problems encountered during all of the testing were limited primarily to oxidation pitting of the erosion shield and sonic failure of the .012-gage erosion shield support clips. All .020 erosion shield clips passed all sonic testing. No oxidation failures occurred during the initial simulated re-entry heat cycle.

The evaluation of the intersection of a series of erosion shields, under conditions of high temperature, high air-flow velocity, and transverse pressure, required the development of a major facility. The types of problems requiring empirical evaluation under these

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environments were: free edge deflection under combined temperature gradients and loads; internal erosion of the insulation; boundary layer leakage into the insulation cavity which could result in excessive insulation and Rene 41 temperatures; and oxidation in an environment involving approximate flight temperature, pressure, and moving air mass.

A one-megawatt plasma jet tunnel was developed by the contractor to test insulated panels under the above mentioned conditions. This facility was used to test a series of 4-inch by 10-inch test specimens (representing the intersection of four erosion shields) in the environment shown in Figure 27.

Detailed information on the development of the heat shield and the insulated panel can be found in Reference 3.

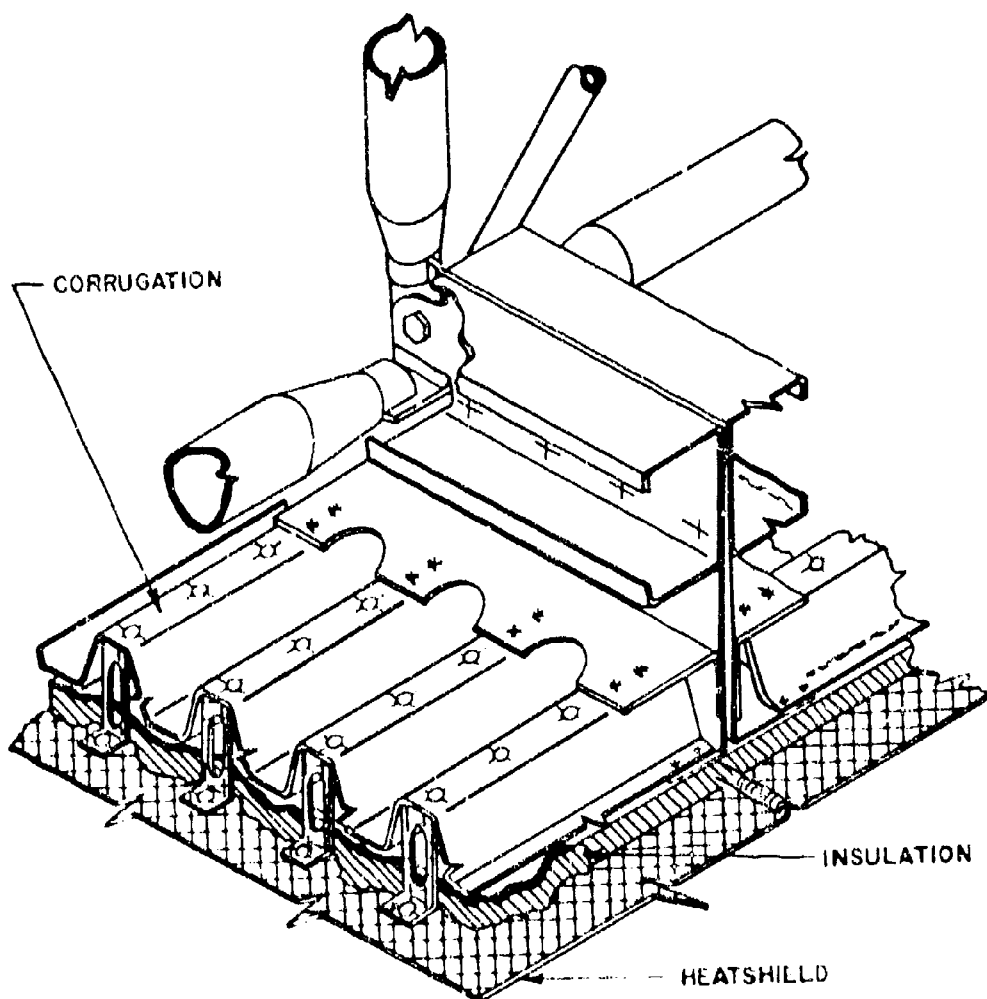


Figure 7. Insulated Panel

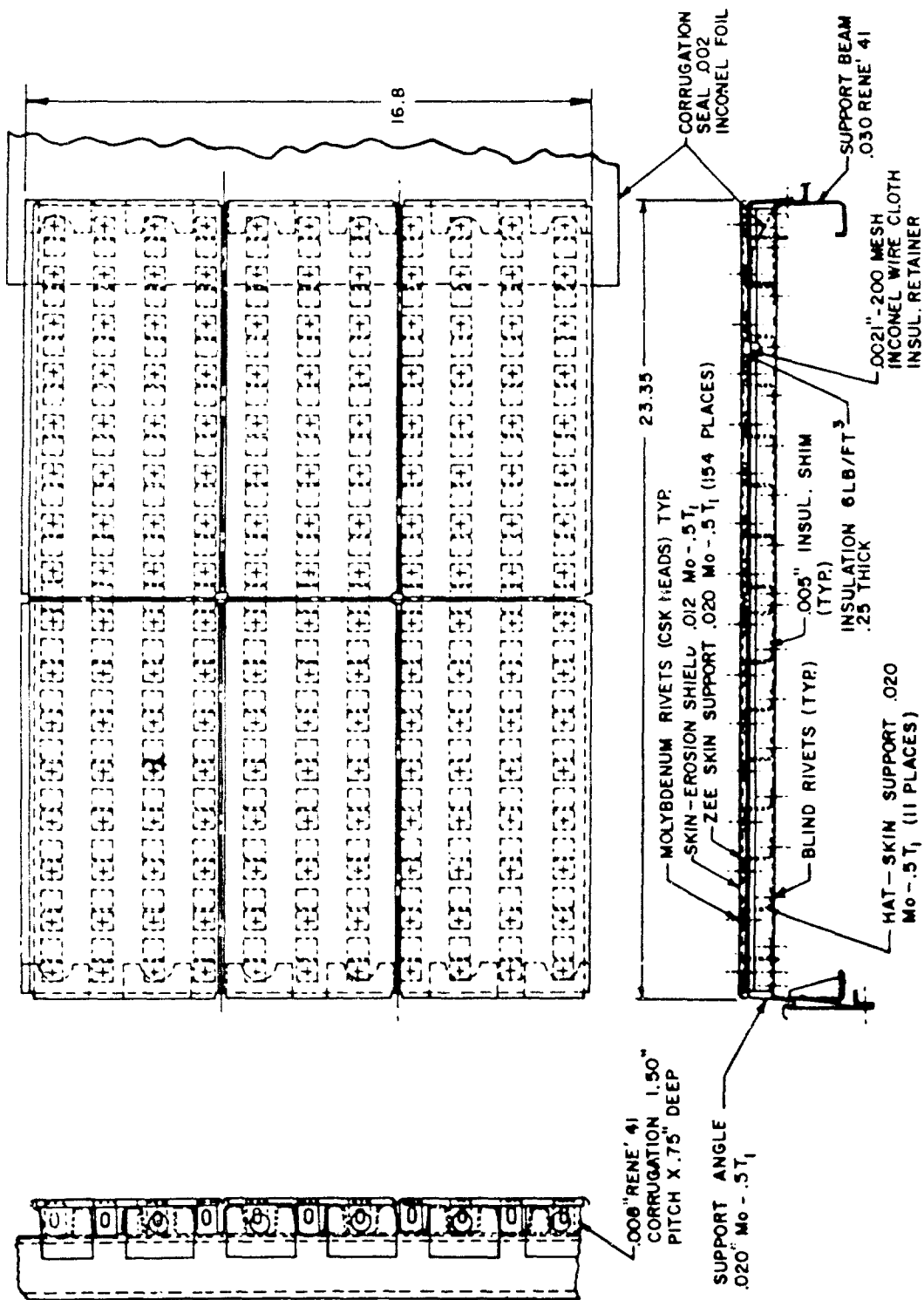


Figure 8. Basic Heat Shield Concept

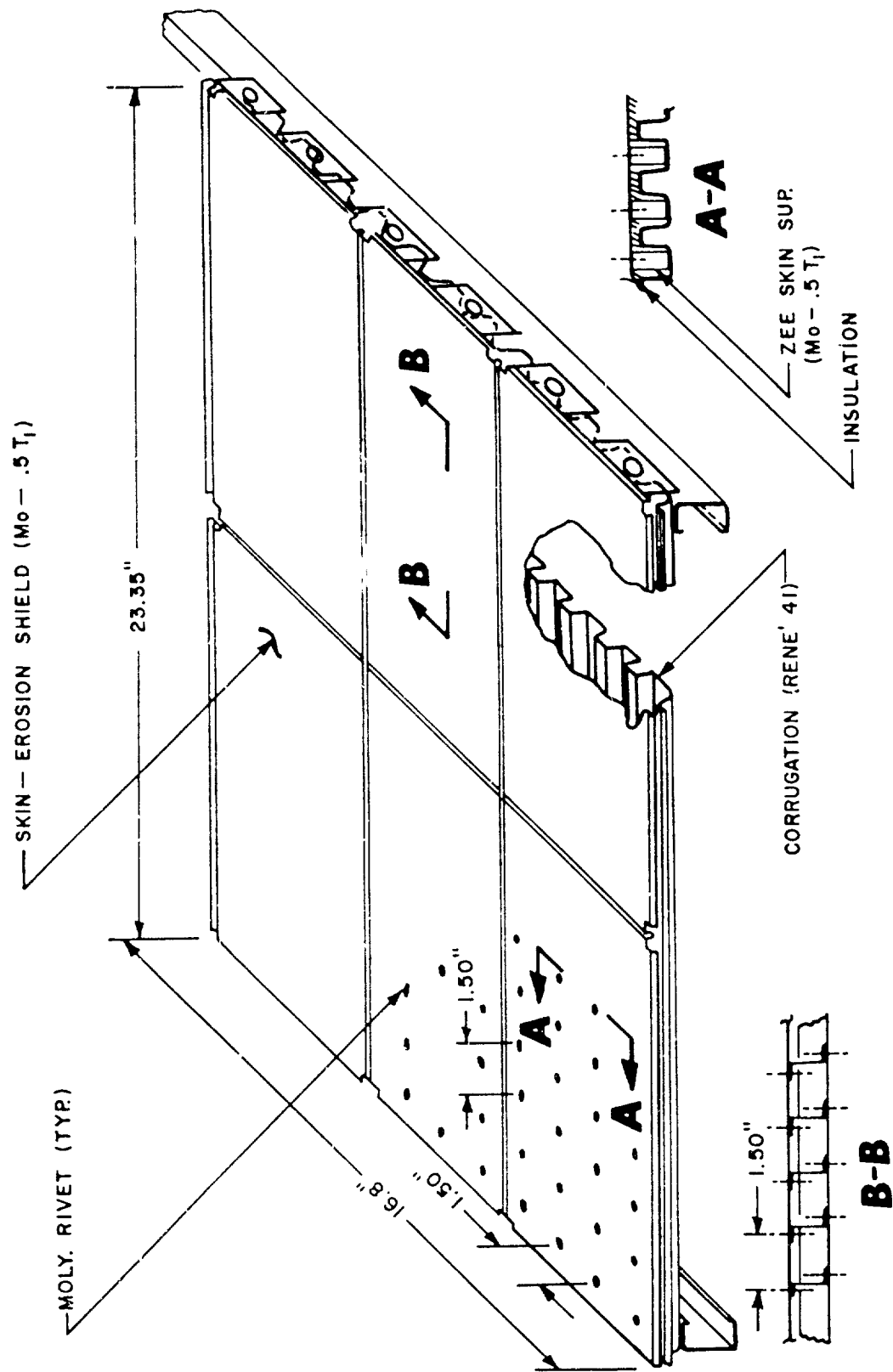


Figure 9. Molybdenum Heat Shield Concept

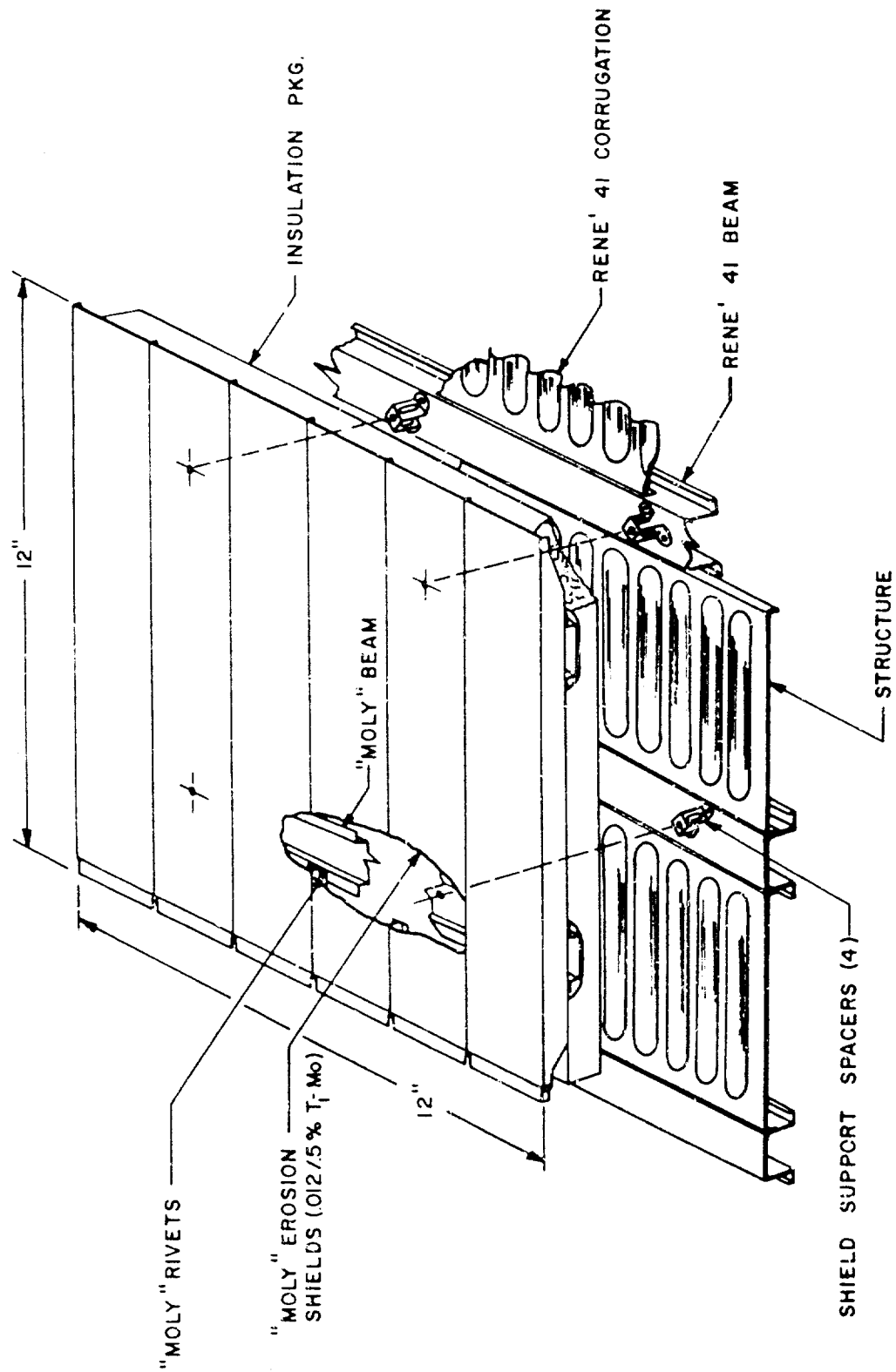


Figure 10. Molybdenum Heat Shield Concept

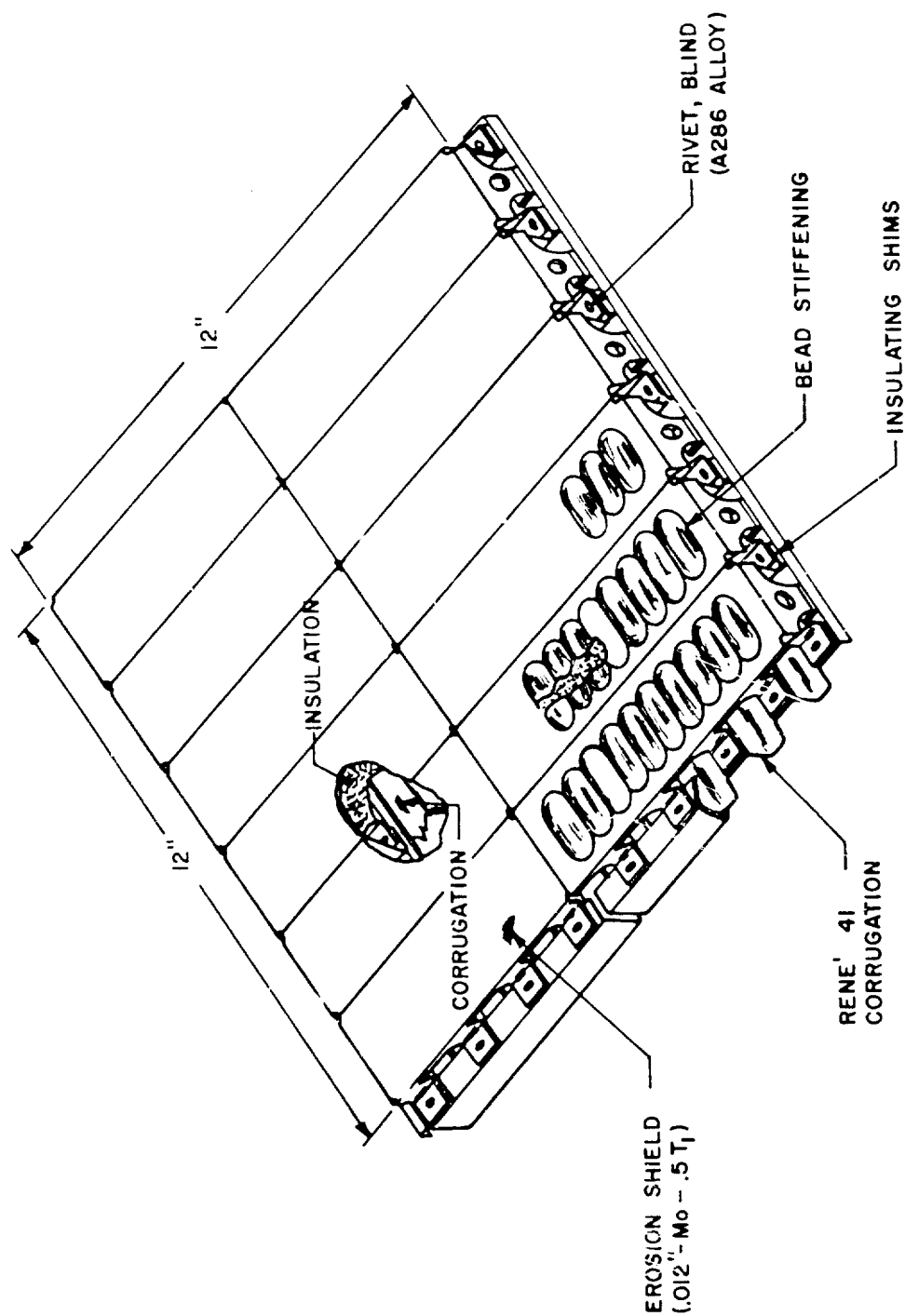


Figure 11. Molybdenum Heat Shield Concept

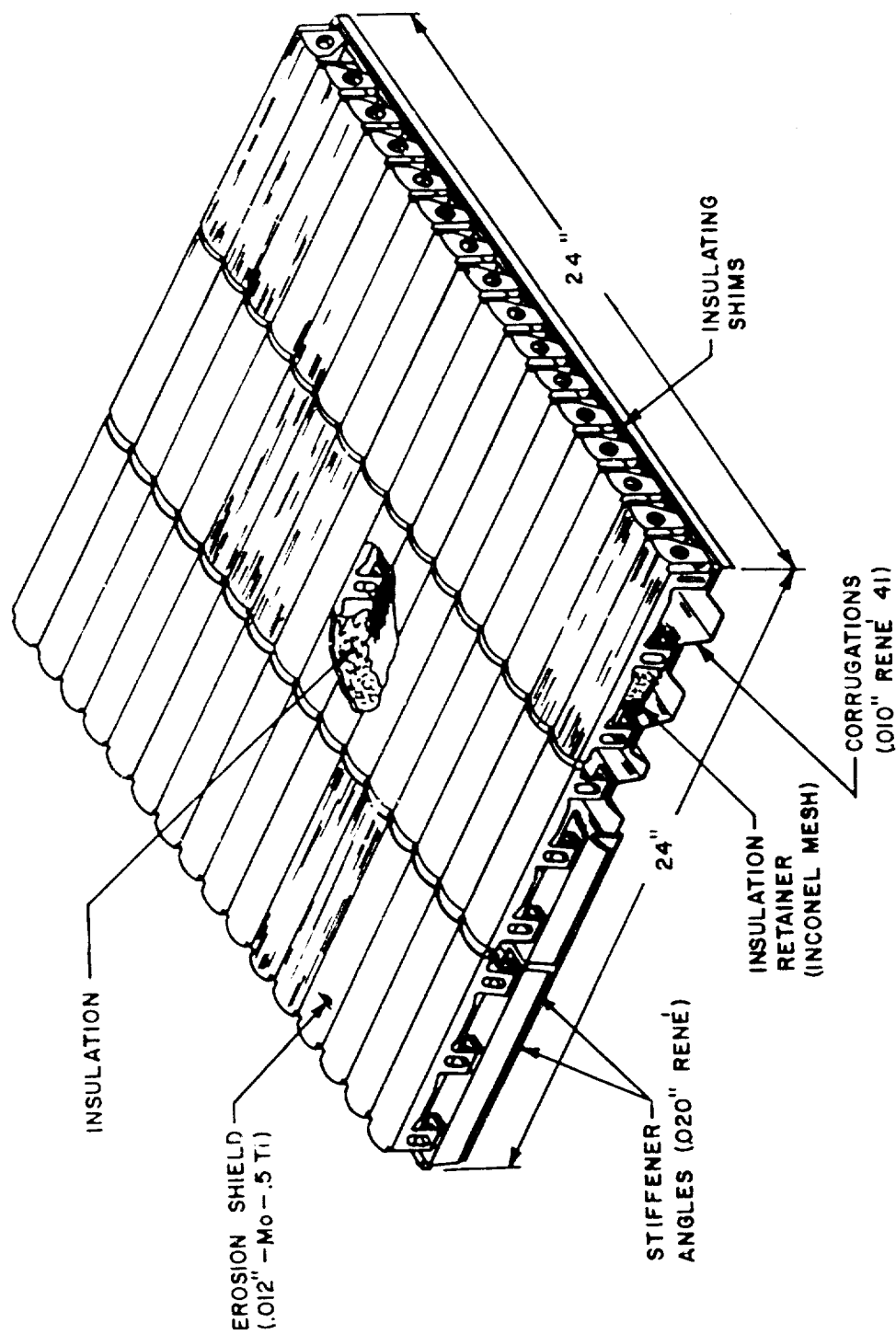


Figure 12. Molybdenum Heat Shield Concept

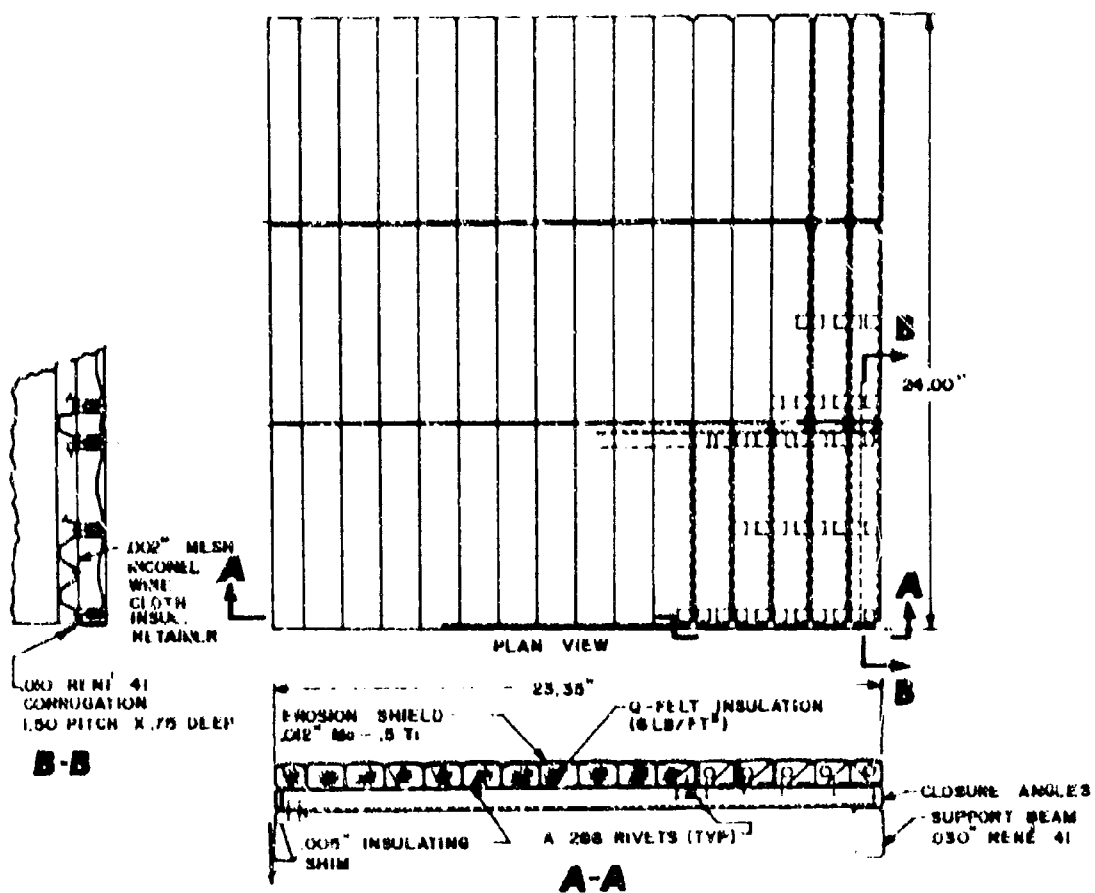


Figure 13. Molybdenum Heat Shield Concept

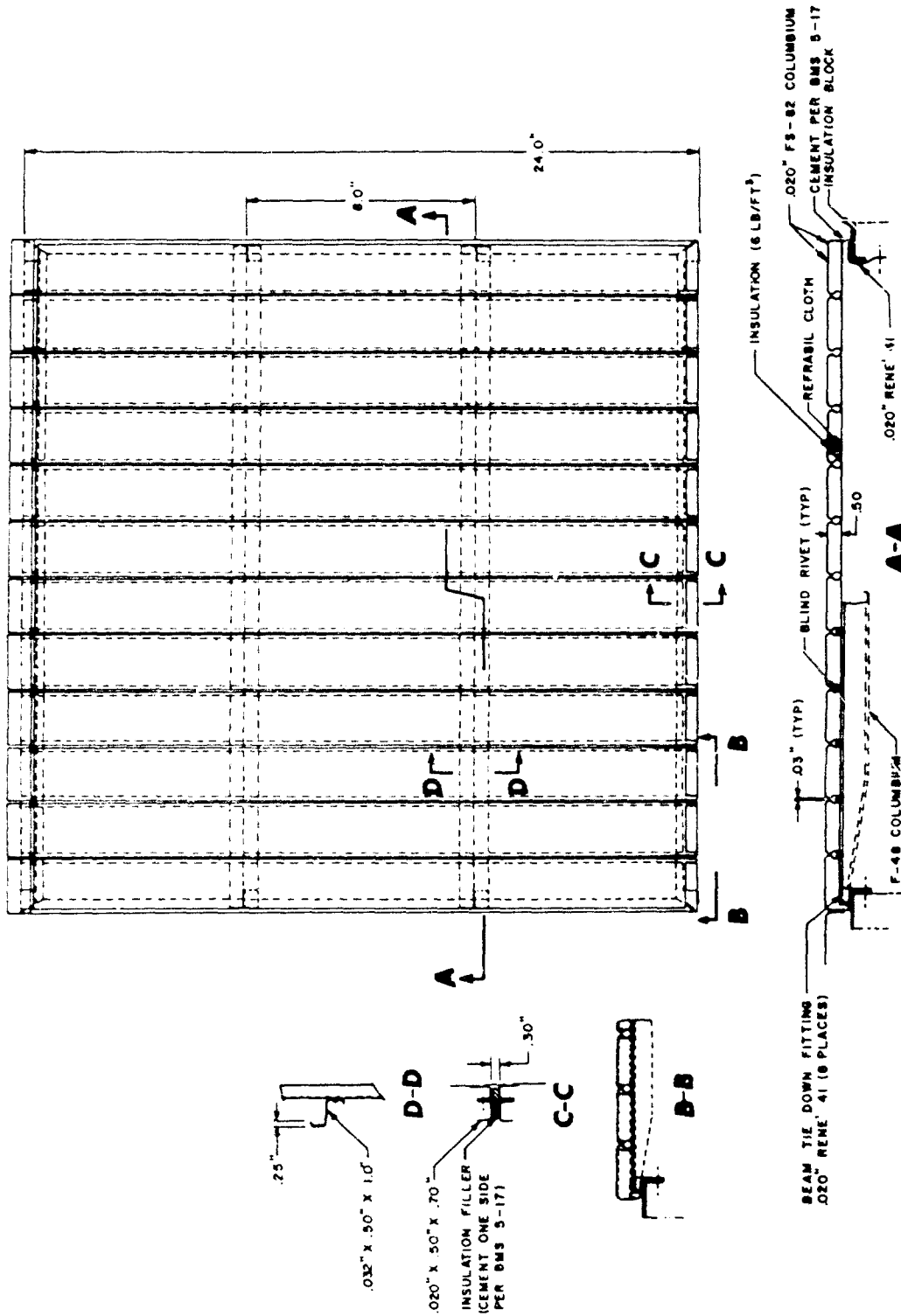


Figure 14. Columblum Heat Shield Concept

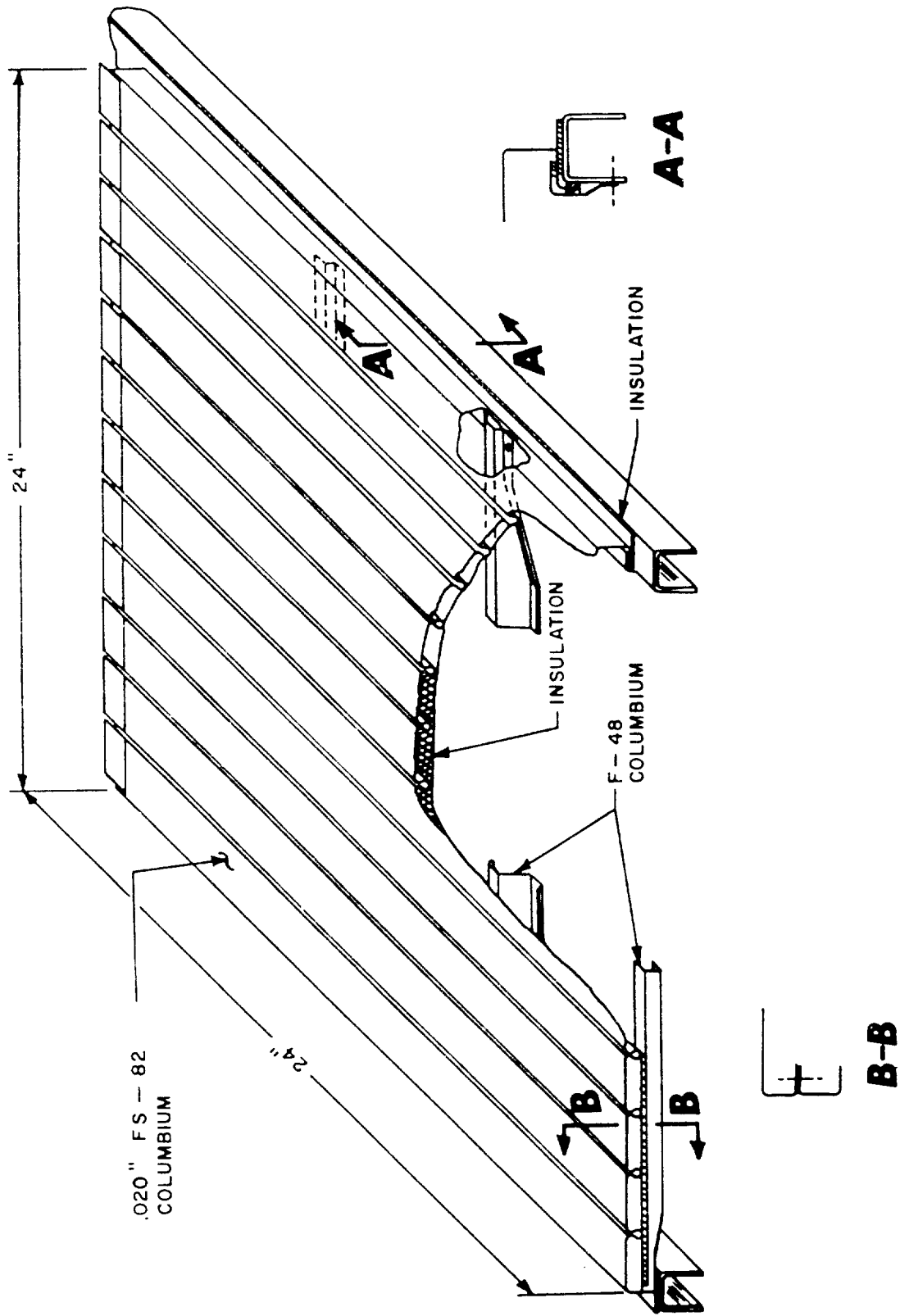


Figure 15. Columbia Heat Shield Concept

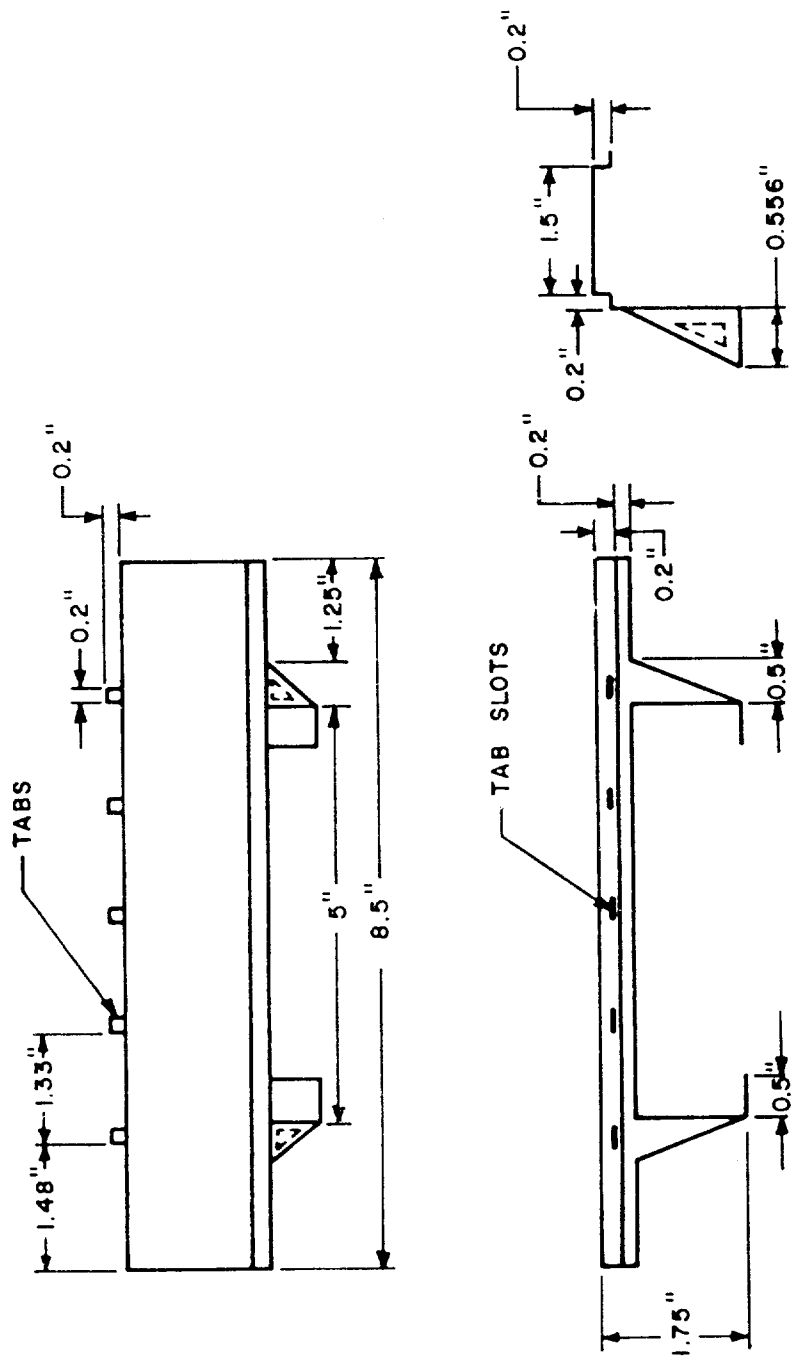


Figure 16a. X-20 SPO Engineering Heat Shield Concept

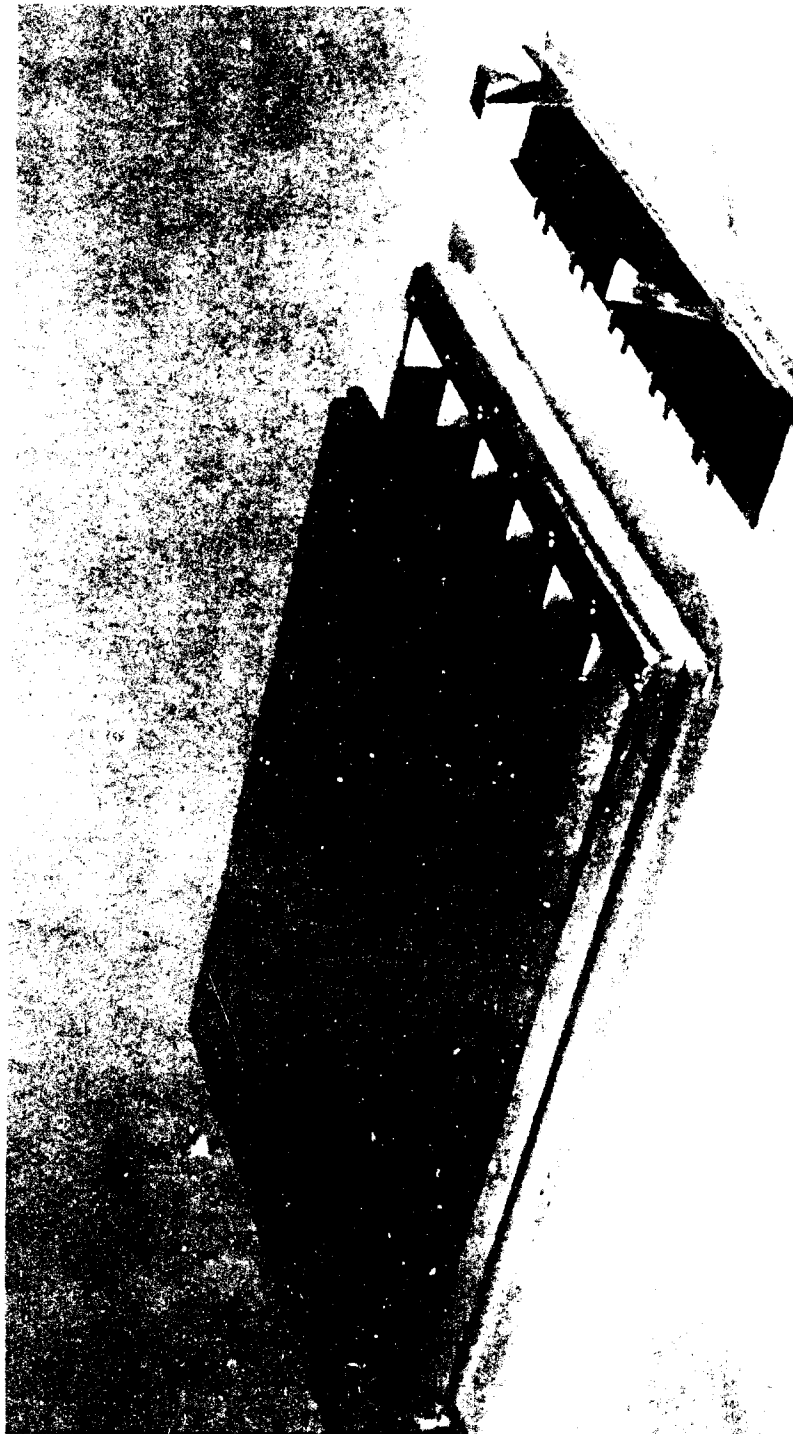


Figure 10. N-20 SPO Engineering Heat Shield Concept

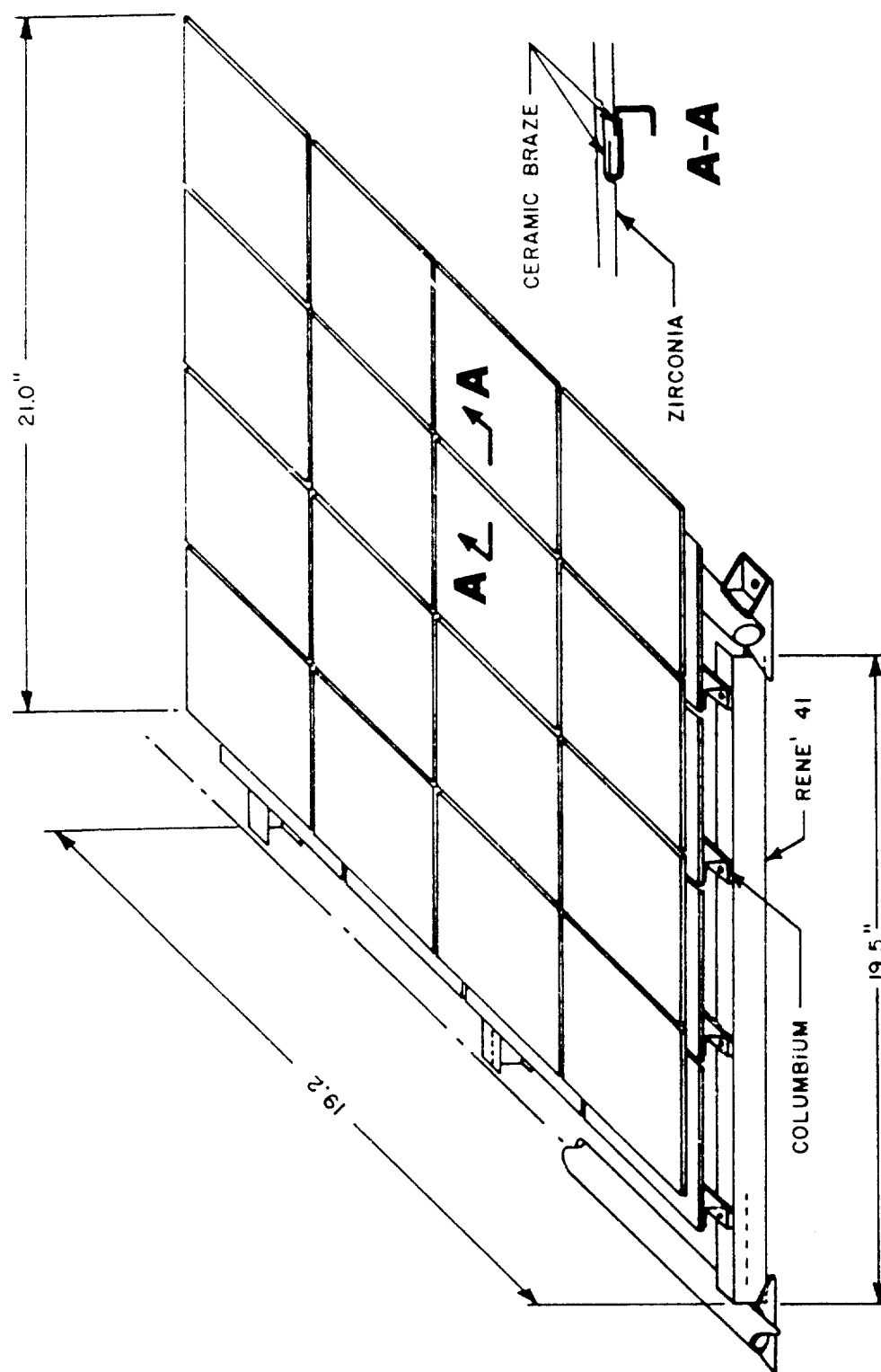


Figure 17. Ceramic Lower Surface Panel

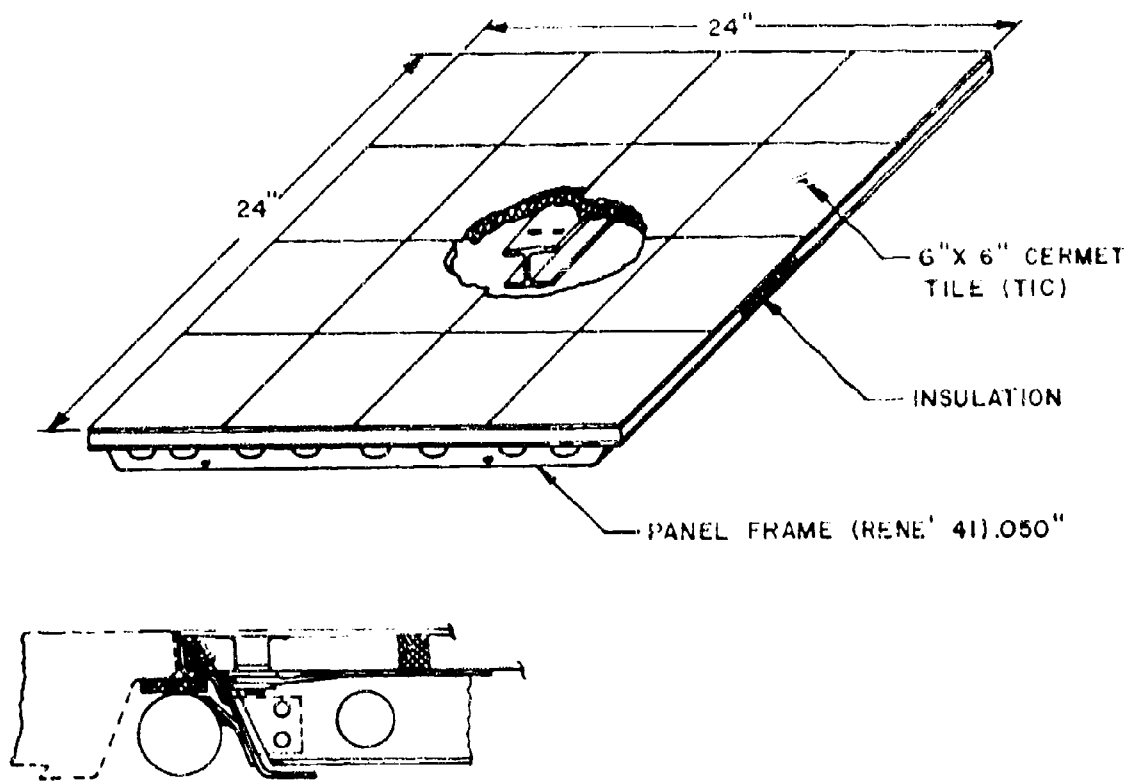


Figure 18. Cermet Lower Surface Panel

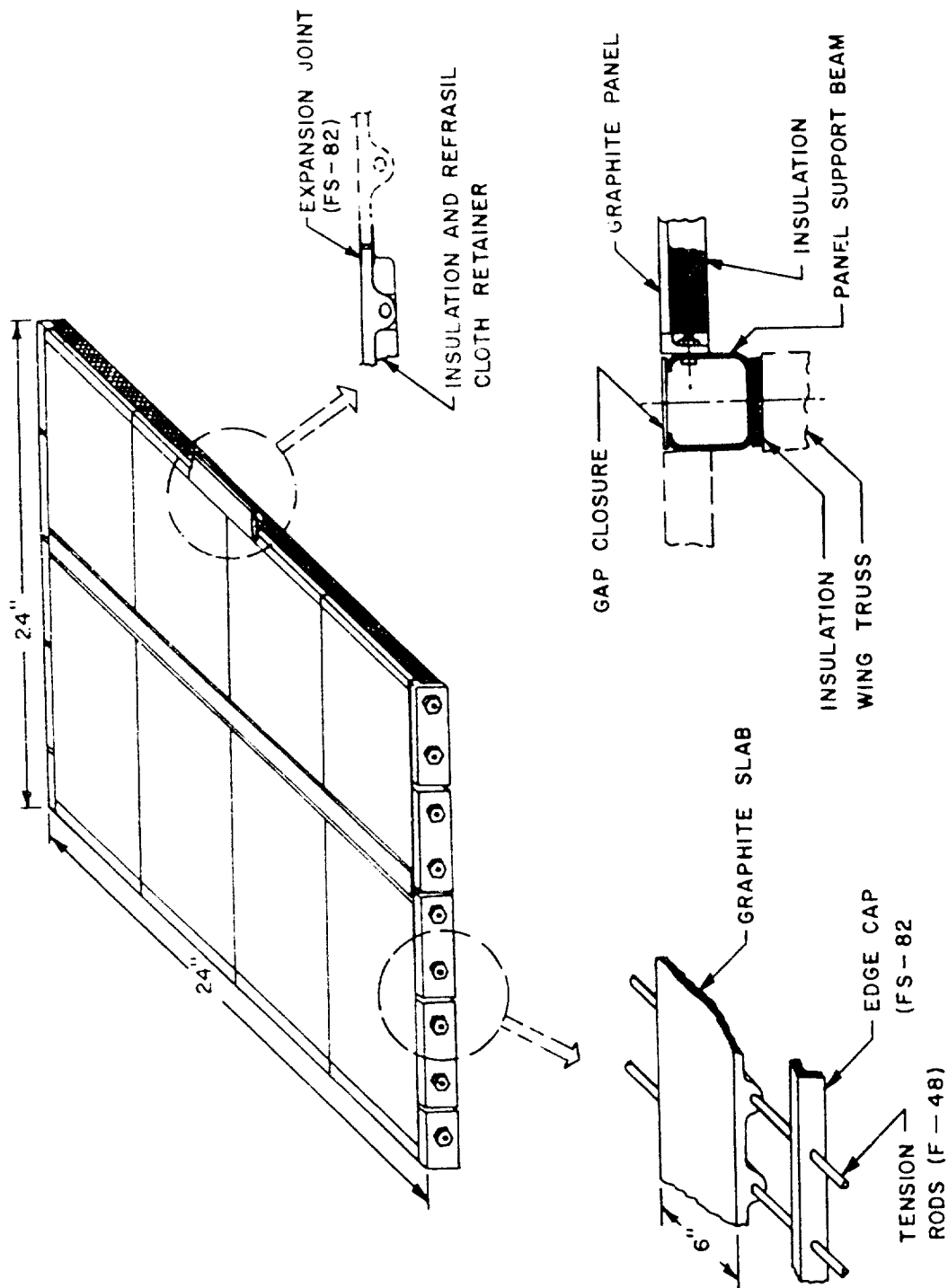


Figure 19. Graphite Lower Surface Panel

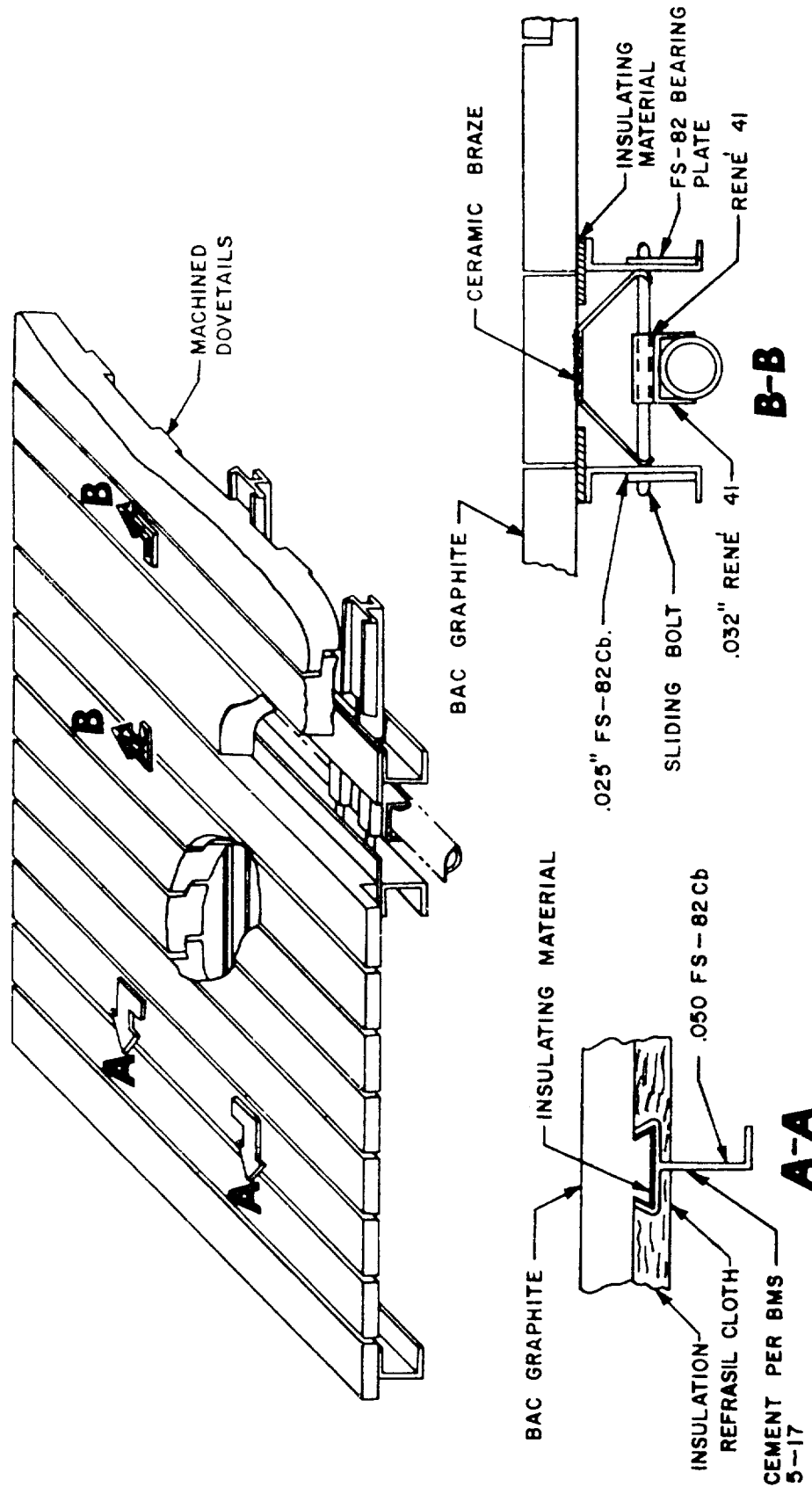


Figure 20. Graphite Skin Panel

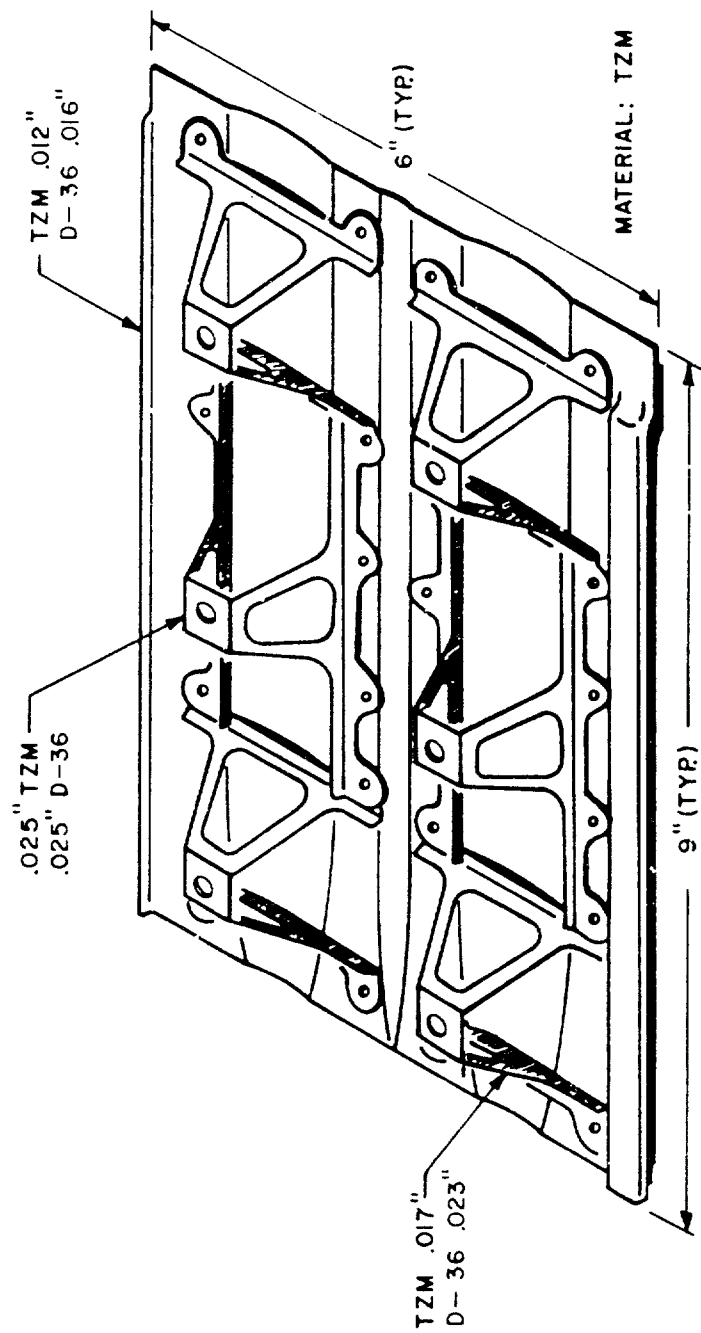


Figure 21. Production X-20 Heat Shield

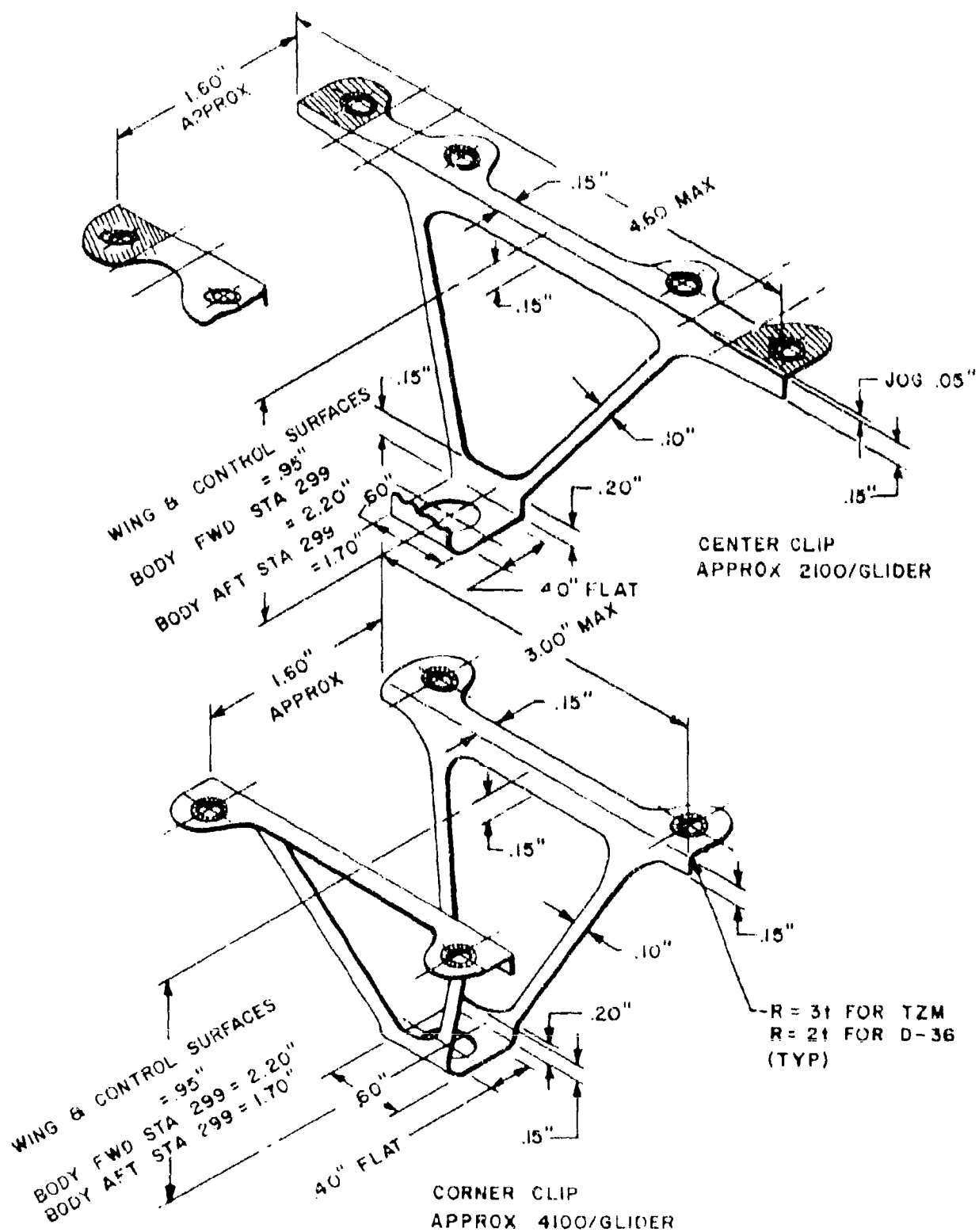


Figure 22. Production X-20 Heat Shield Clip

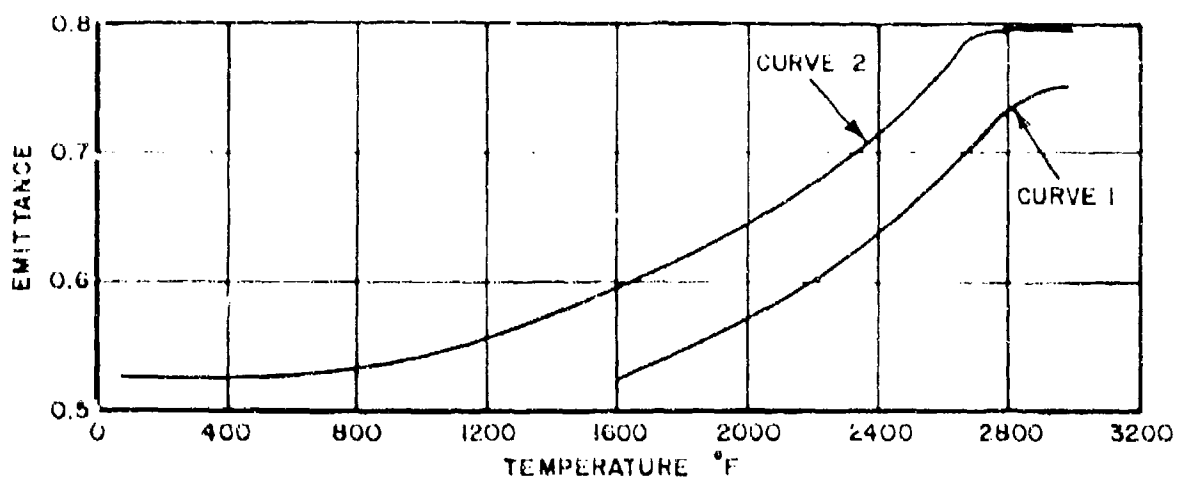


Figure 24. Coated TZM Emittance Values

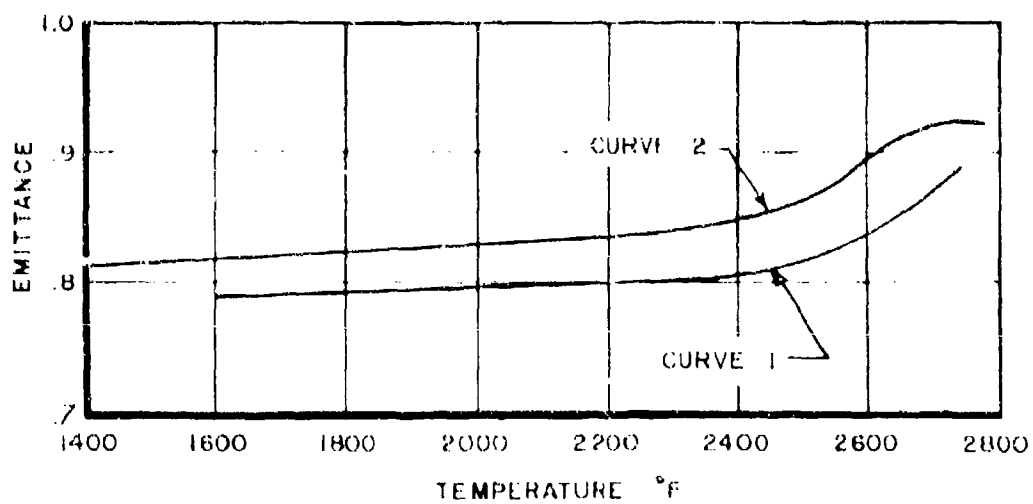


Figure 24. Coated D-36 Emittance Values

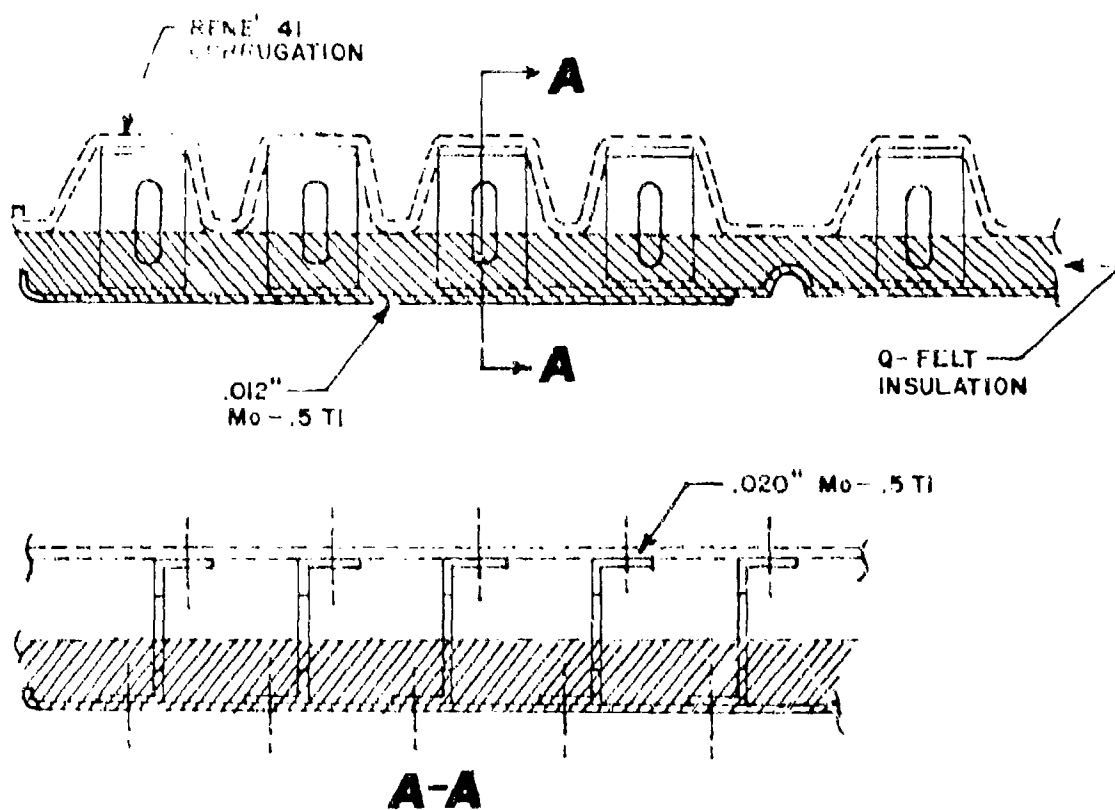


Figure 25. Basic Heat Shield Used In Development Tests

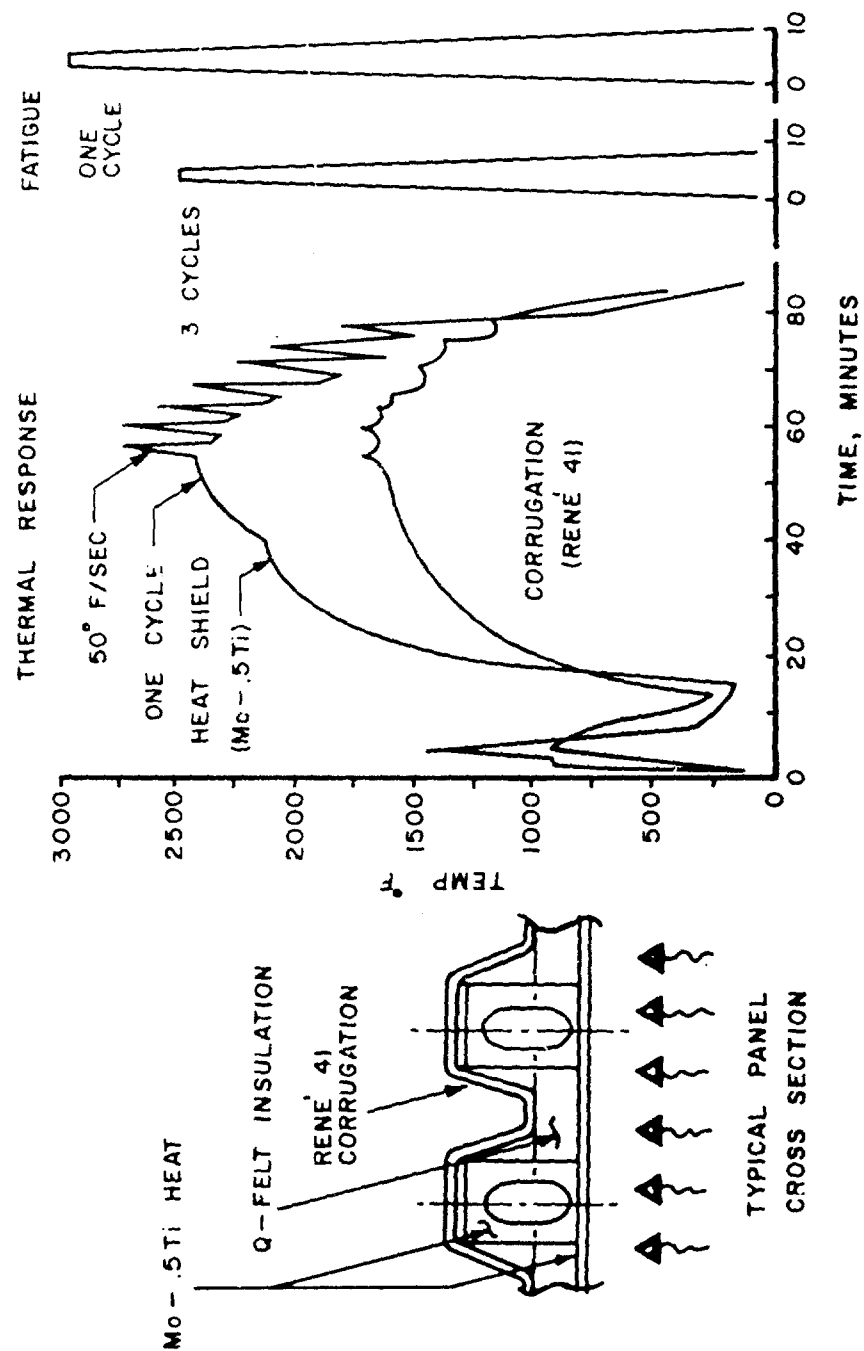


Figure 26. Radiant Heat Environment for Heat Shield Development Tests

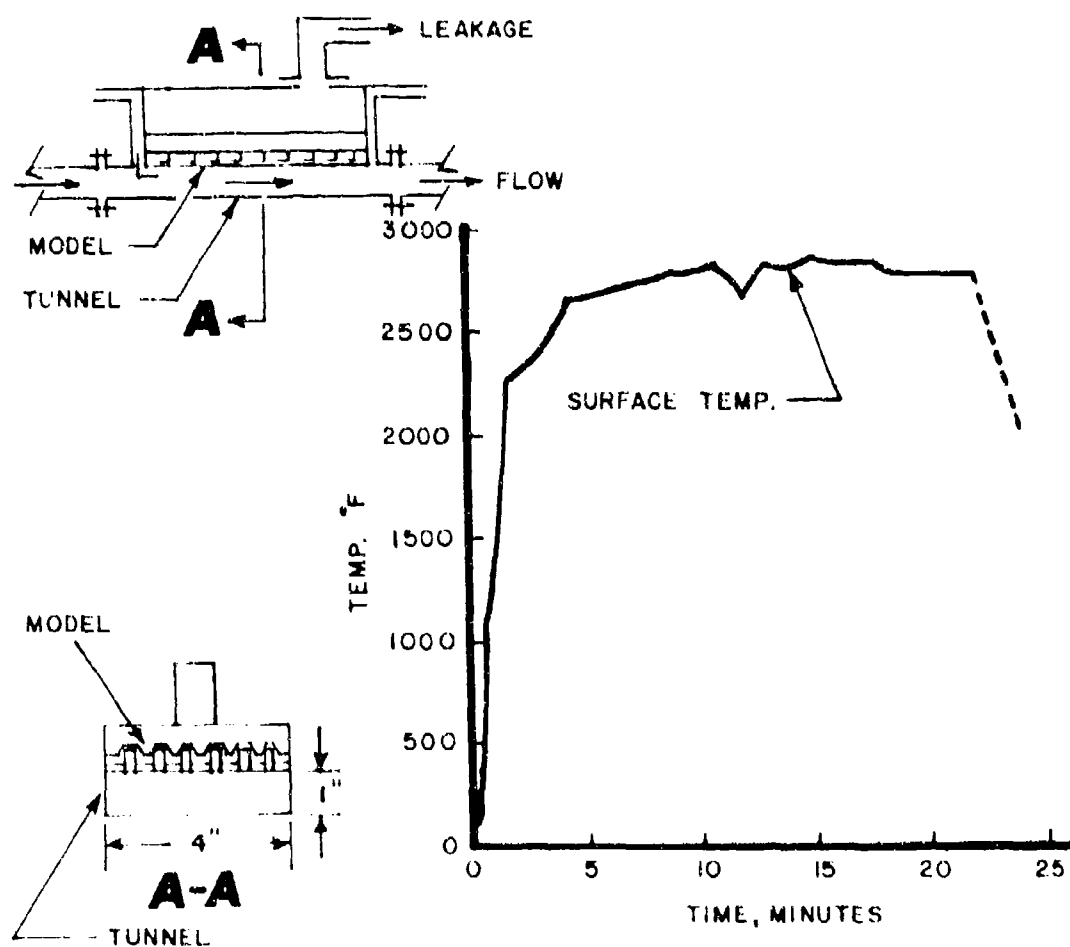


Figure 27. Test Environment of Plasma Jet Test Facility

LEADING EDGE

LEADING EDGE DESIGN

The X-20 leading edges were composed of thin shells fabricated from molybdenum and protected by an oxidation-resistant coating. They were segmented and possessed no longitudinal structural continuity and, therefore, did not contribute to the overall strength of the vehicle.

The configuration of the leading edge that received appreciable study was a double-shell design, constructed with inner shells approximately 3 inches in width, which had die formed stiffening beads to carry all pressure and inertia loads. Additional shells, also 3 inches in width, were staggered over the load-carrying shells and shingled in the downstream direction to provide aerodynamic smoothness, partial sealing and coating redundancy. All riveting was eliminated so as to obtain maximum protection from the coating. A sketch of the double-shell design is shown in Figure 28.

The leading edge shells were attached to a Rene 41 leading edge beam with frequently spaced bolts. The large number of attachment points minimized adverse effects from the boost sonic environment and reduced stress levels in the refractory alloy shells. The attachments in the cooler areas of the leading edges were superalloy bolts, but those exposed to temperatures above 2000°F were of refractory metal.

During flight, the X-20 leading edge components would be subjected to sonic vibration and inertia loads; however, local air pressure loads constituted the major source of mechanical loading. The magnitude and distribution of this air-loading was a function of the flight corridor through subsonic, transonic, and hypersonic speed ranges. Airloads of the greatest magnitude occurred during transonic flight, however, air loads during hypersonic flight were associated with high structural temperatures and were the most critical.

Except for the nose cap, the leading edges of the glider had to withstand higher heating rates than any other portion of the X-20 vehicle. The difference in coefficients of thermal expansion and differences in temperature of the leading edge and leading edge Rene 41 beam dictated that the leading edge be attached with freedom in the longitudinal direction. Transverse differential thermal growth was accommodated by shell flexing. A blanket of insulation, which completely covered the front face of the corrugated leading edge beam and any exposed fasteners, protected these components from the back face radiation of the hot leading edge.

Since the leading edges were subjected to an environment that combined loads and high temperatures, a thorough analysis of both thermal and load-induced stresses was required. The fact that maximum thermal stresses and maximum load-induced stresses do not occur simultaneously suggested that separate analyses be performed for each type of stress at several design conditions to find the critical design stress. In a restrained or loaded beam with thermal gradients, the thermal strains of free beam were computed separately from the strains due to restraints or loads and then combined algebraically. The combination is accomplished by adding the strains at limit and at ultimate load. The structure was not allowed to yield at limit load nor fail at ultimate load. The stress level was determined from the total strain. The strains may be combined provided buckling is not a consideration. This restriction was valid for the X-20 leading edges.

Late in 1963, the leading edge concept was changed from the aforementioned double-shell concept. The reason for this change is explained in the section on Problems. The most recent leading edge concept is shown in Figure 29. The concept was a single-shell design with integral chemical milled stiffeners. Attachment mechanisms of columbium were to be employed. More detailed information on leading edge development tests can be found in Reference 4.

LEADING EDGE DEVELOPMENT

The development program for the refractory alloy leading edge was directed principally toward determining and improving the following: (1) an adherent oxidation-resistance coating capable of withstanding temperatures to 3000°F; (2) the design and evaluation of an attachment scheme that would be adequate for load and sonic environments but would not impose excessive restraint for differential thermal growth between the leading edge segments and the leading edge beam; and (3) sealing requirements.

Five leading edge concepts (Figure 30) were designed, built, and tested under temperature, load, and acoustical excitation. From the tests, the various designs, including their ability to be coated, were evaluated structurally. Pertinent design details of each concept are as follows:

- Concept I - .051 Mo-.5Ti unstiffened shell 12 inches long, attachment by bolts in oversized holes;
- Concept II - .040 Mo-.5Ti 12-inch long shell, riveted corner reinforcement, and attachment by tilting bolts;
- Concept III - .030 Mo-.5Ti long shell with .030 Mo-.5Ti riveted stiffeners, and attachment by tilting bolts;
- Concept IV - .012 Mo-.5Ti outer shell with .030 Mo-.5Ti beaded inner shell with an 0.40 Mo-.5Ti corner reinforcement, and attachment by flexible lugs (specimen was 12 inches long);
- Concept V - .012, Mo-.5Ti outer shell overlapping an .030 Mo-.5Ti beaded inner shell with .030 corner reinforcement, 3-inch long segments, and attachment by bolts in standard size holes.

Each of the concepts was subjected to the thermal and acoustical environments shown in Figure 31. All concepts survived the test environments without sustaining structural damage. Oxidation was, in general, limited to local pitting and did not significantly alter the structural integrity of the parts.

After the thermal and acoustical tests, each concept was loaded at room temperature, using a radially applied load. All concepts carried a load greater than 100 percent of the calculated static failure load.

In formulating a refined design, various features from each of the original five designs were incorporated. The end result was double-shell design (Figure 32). Corner gussets were riveted as shown. The attachment was by TZM bolts in standard size holes. This concept was not tested due to a fabrication problem mentioned later in this report.

Because leading edge leakage could affect the temperature of the leading edge beam, an empirical evaluation of the leading edge sealing concept was necessary.

A plasma jet with a shroud-nozzle (Figure 33) that envelops a full-scale leading edge was developed. It was capable of generating structural surface temperatures to 3000°F and associated dynamic pressures to approximately 1 psi. This facility was also used to test the silicide coating for oxidation in high temperature and high-velocity air. Figure 33 also presents a cross section of the Mo-.5Ti leading edge tested in the plasma tunnel shroud. Basic construction of the leading edge was an .020 beaded inner shell with a joint overlapping the .012 outer shell. Figure 34 presents the pressures, temperatures, and leakage rates versus time obtained during the tests in the plasma shroud nozzle facility.

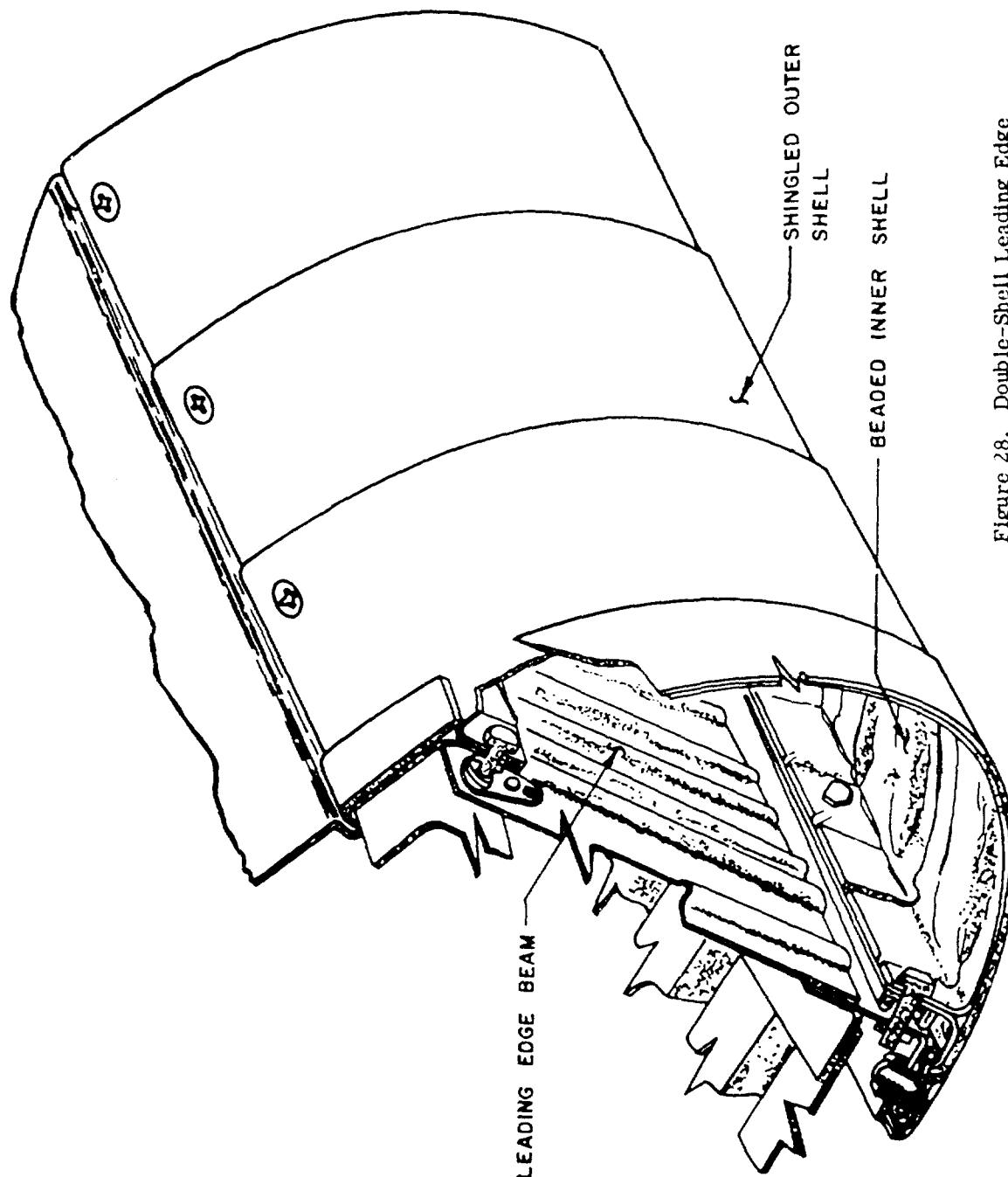


Figure 28. Double-Shell Leading Edge

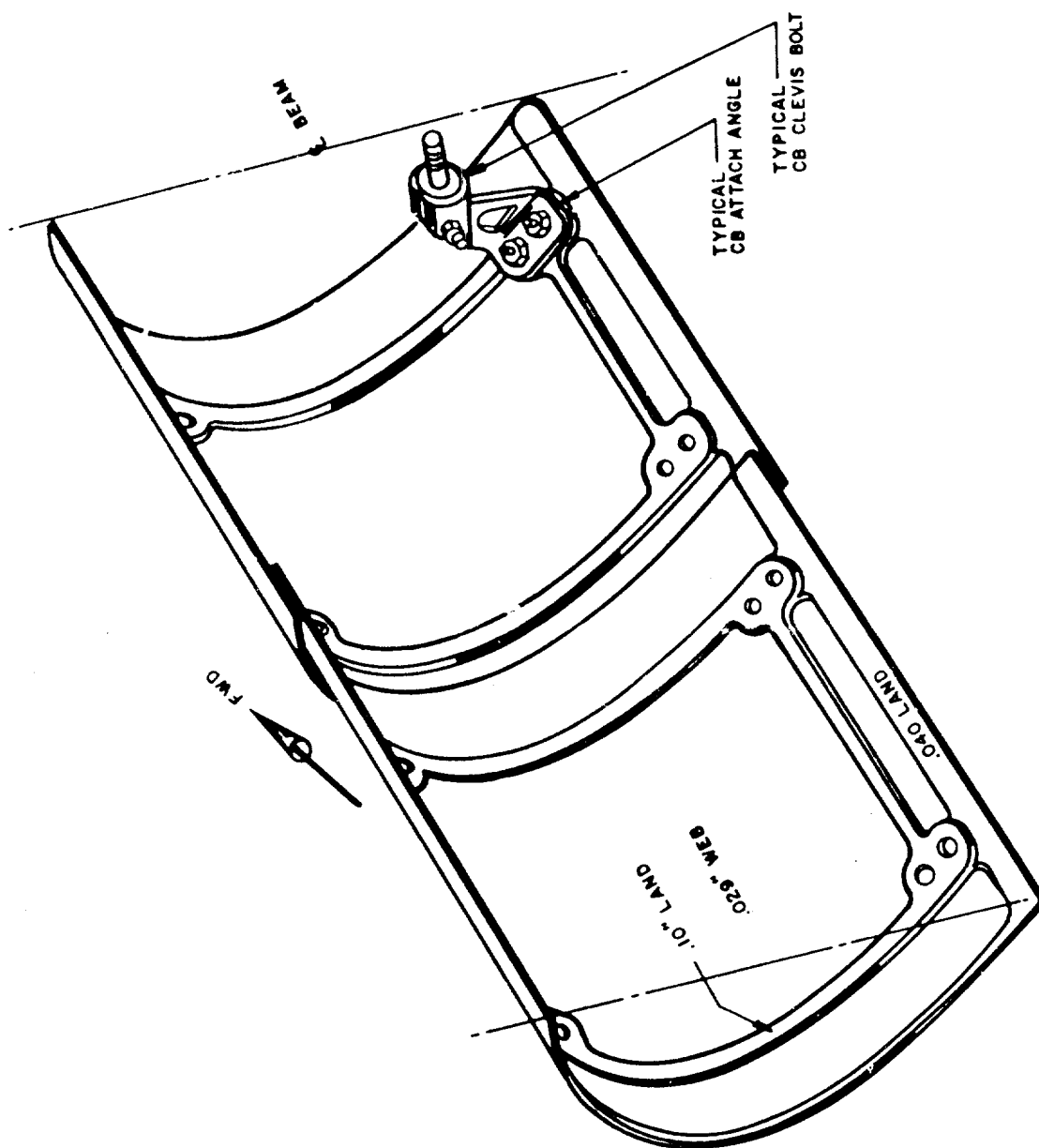


Figure 29. Chem Milled TZM Leading Edge

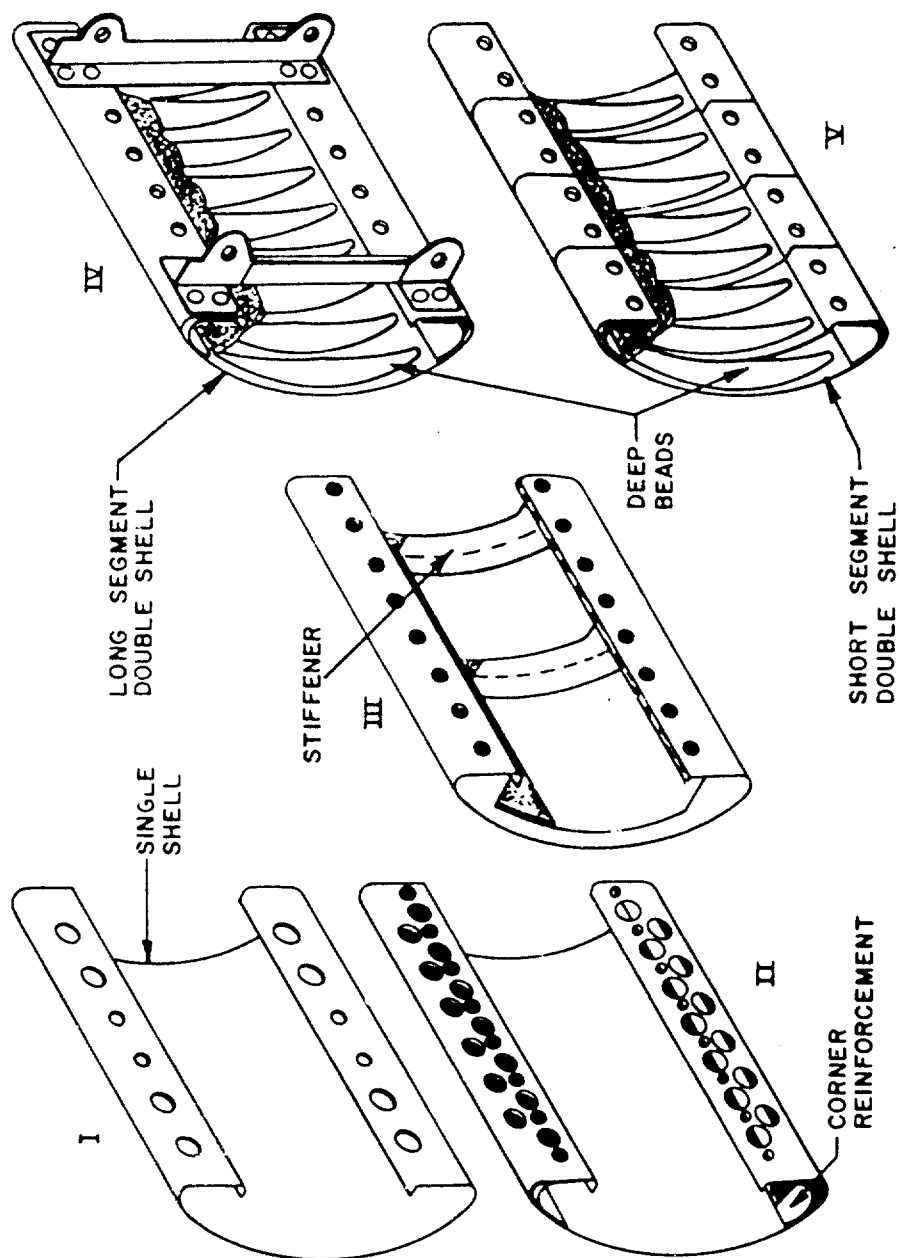


Figure 30. Mo-.5T1 Leading Edge Development Concepts

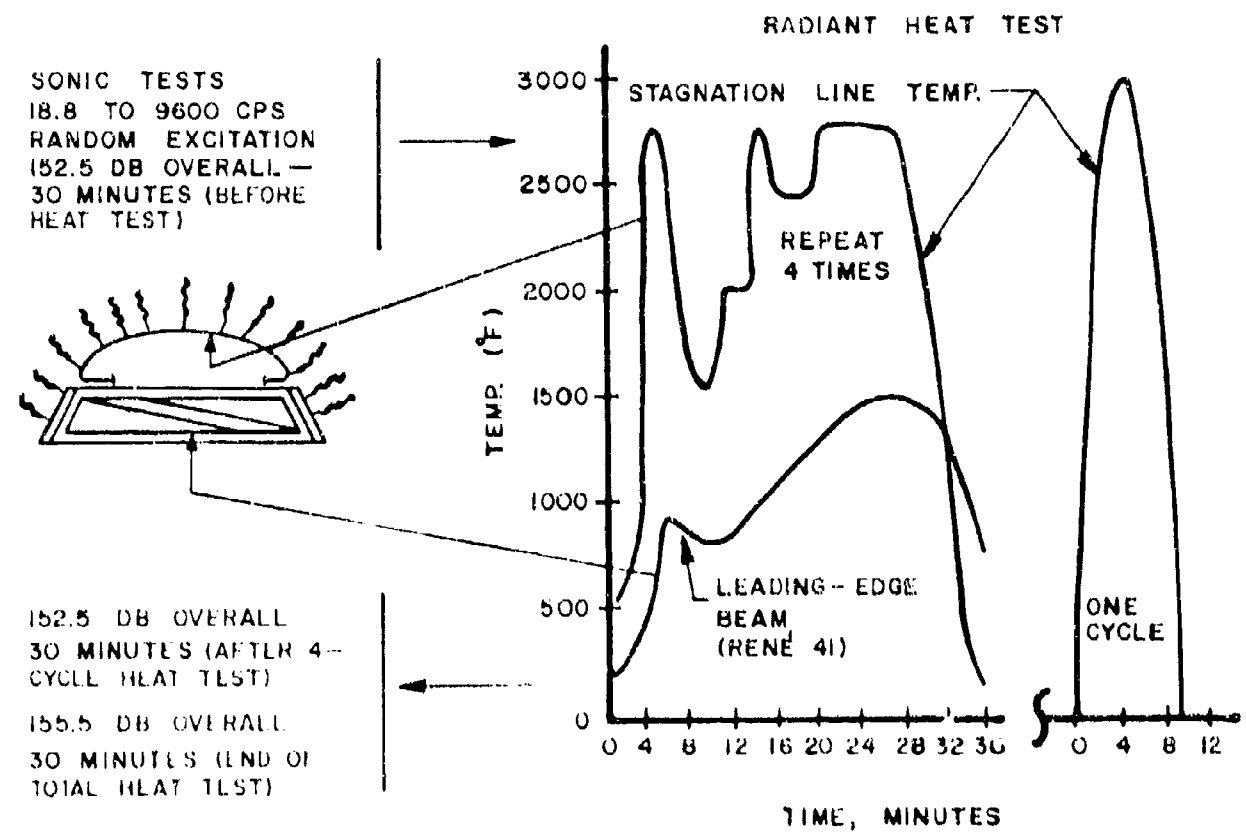


Figure 31. Radiant Heat Environment for Leading Edge Development Tests

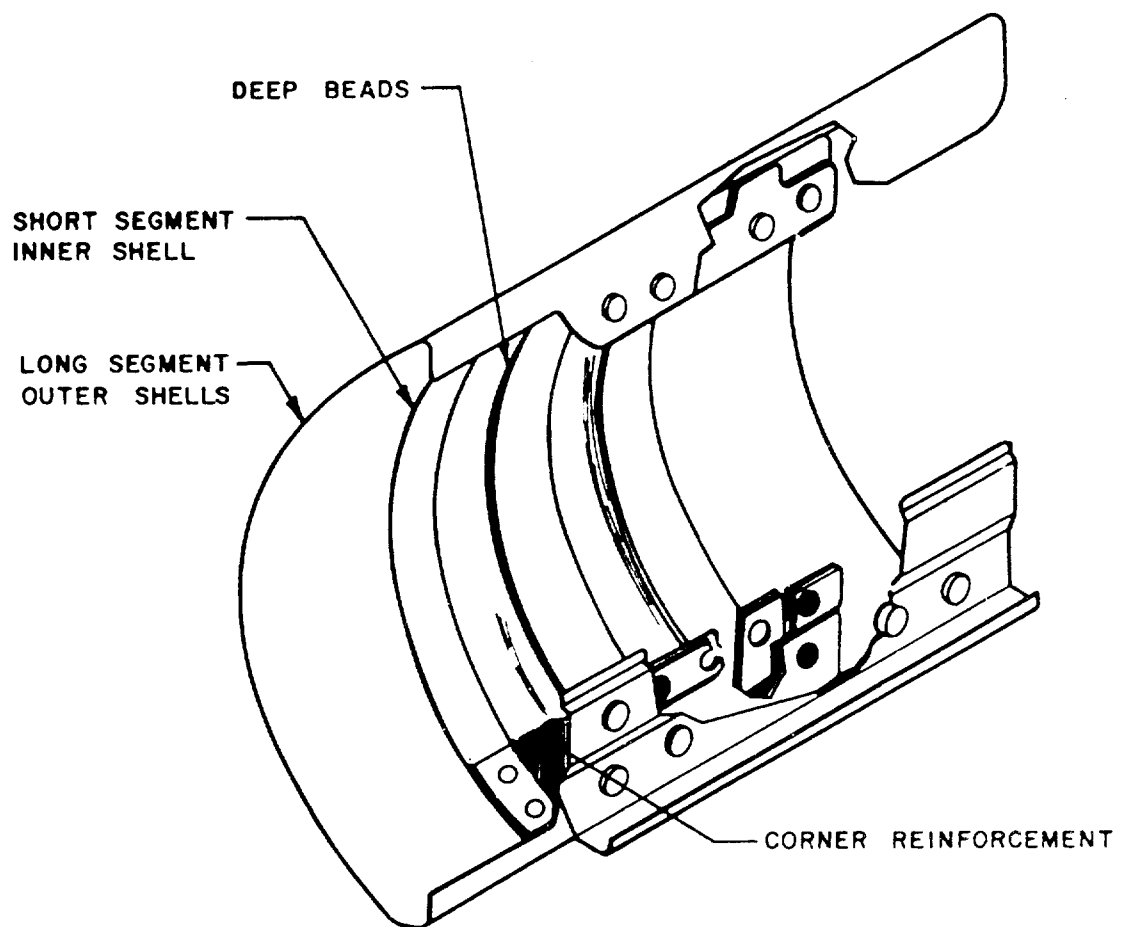


Figure 32. TBM Double-Shell Leading Edge Development Concepts

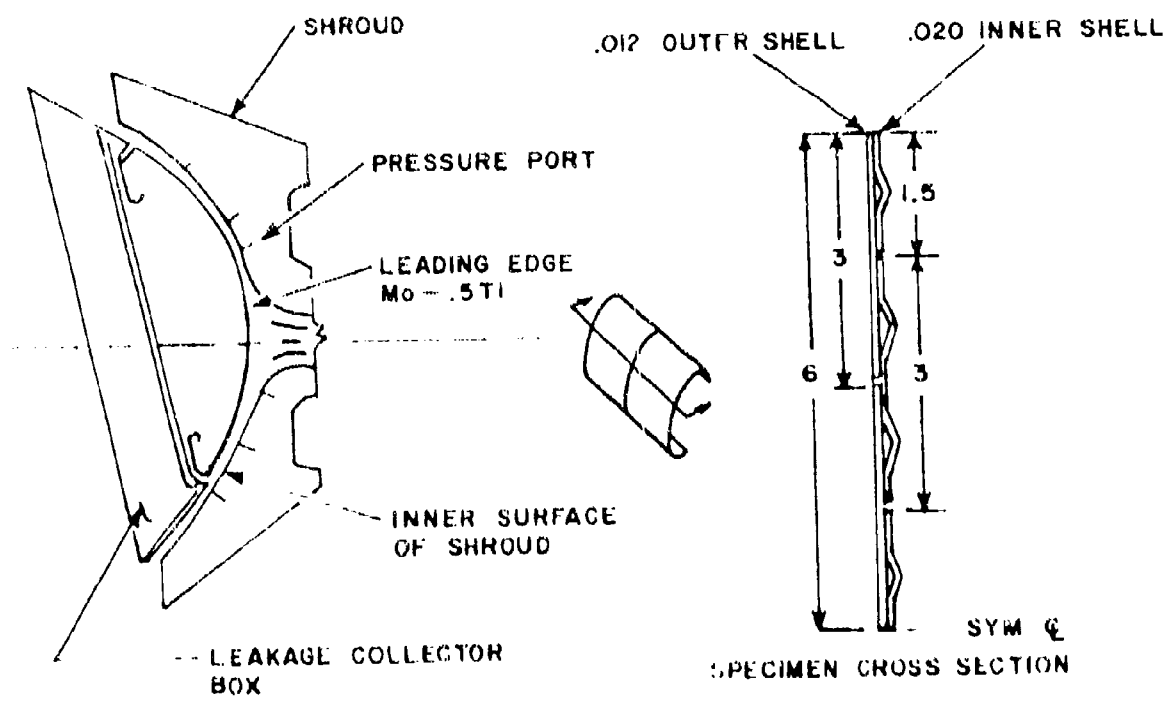


Figure 33. Plasma Jet Test of Leading Edge

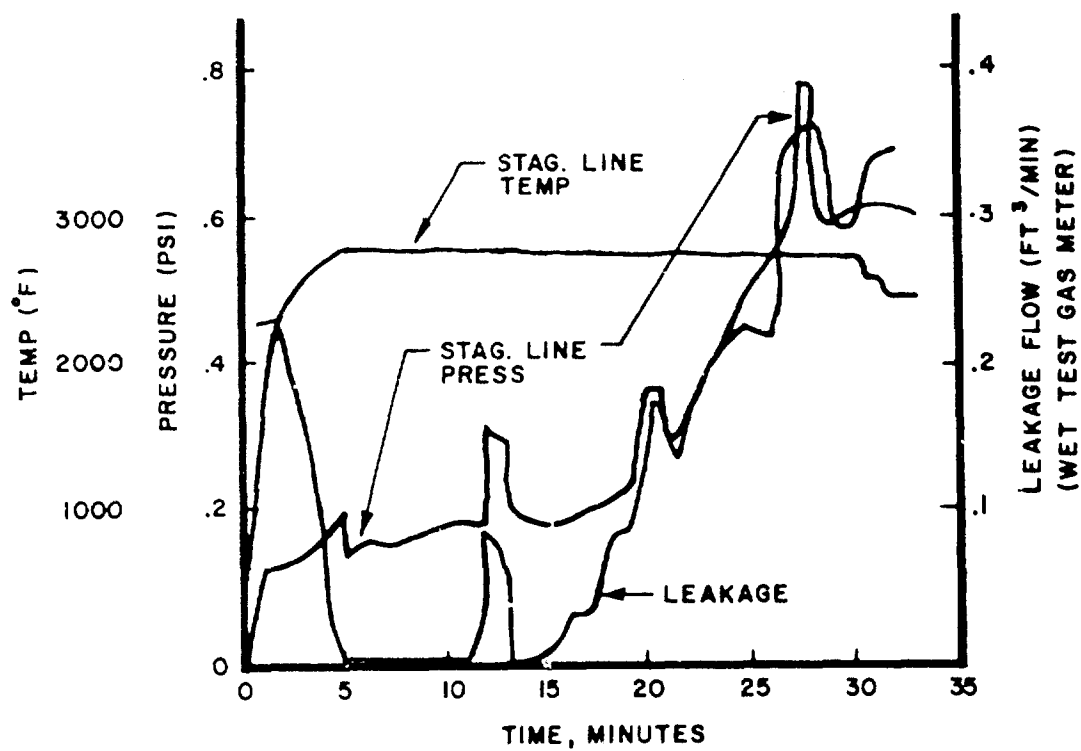


Figure 34. Plasma Jet Test Environment for Leading Edge

PROBLEMS

As is well known, refractory materials rarely have been used in aircraft design and have never been used on a scale approaching that necessary for the X-20. It was to be expected, therefore, that this pioneering effort in refractory material design and fabrication would introduce many new problems. Some of the more significant of these problems are presented in the following paragraphs.

In early 1962, the use of refractory metals on the X-20 was being questioned. This occurred after a number of tests on the silicide-coated refractory metal specimens had been accomplished at re-entry temperatures in a reduced pressure environment. The protective properties of the silicide coating were severely limited and were time-dependent in an environment of high temperature and reduced pressures.

In response to this potentially serious problem, the contractor developed and built the environmental simulator, shown in Figure 35, which permitted testing of silicide coatings in a X-20 re-entry environment. These tests substantiated that the effectiveness of the silicide coating protective mechanism was appreciably reduced at partial pressure on both the molybdenum and columbium alloys but was still adequate to survive the most severe of X-20 type re-entries. From these tests, the life capability curves of Figures 36 and 37 were derived.

Tolerance control was found to be a problem during the fabrication of a refractory metal heat shield. This was particularly true of the standoff clip heights. This problem was solved through the use of shims for positive tolerance control.

In the latter part of 1963, Boeing attempted to build a flight-type coated TZM leading edge (double-shell concept shown in Figures 28 and 32). This attempt did not succeed. During the fabrication of this article, TZM parts warped while undergoing the first application of the coating in the fluidized bed. Apparently, relaxation and/or stress relieving was occurring.

During the fabrication sequence of refractory metal components, the first of two coating operations occurs after part "fit-up" has been accomplished; and as a result of the warpage, subsequent part fit-up during assembly requires forced fitting. Due to the brittle nature of coated TZM, leading edge parts were being fractured during assembly. To solve this problem, the contractor started work on a simplified single shell leading edge concept (shown in Figure 29) and investigated various aspects of the techniques for fabricating molybdenum, such as hot resizing, control of grain size, and shot peening. Of these, hot resizing of parts after the first coating cycle was thought to be the best solution to the problem.

An attempt to fabricate the simplified leading edge (shown in Figure 29) ended when the program was terminated.

In the summer of 1963, analysis showed that, in particular areas, deflections during boost of the X-20 forward wing primary structure were causing excessive rotation and vertical motion of the leading edge segments. These movements were of such magnitude that interference was occurring with adjacent leading edge segments. To solve this problem, the contractor initiated a redesign effort on the X-20 forward wing and initiated planning for a component test involving an aft section of the X-20 wing and leading edge segments and backup structure (shown in Figure 38). Due to the problems in funding the program, the test of the wing component was cancelled eventually. At the time the program terminated, this problem was not entirely solved.

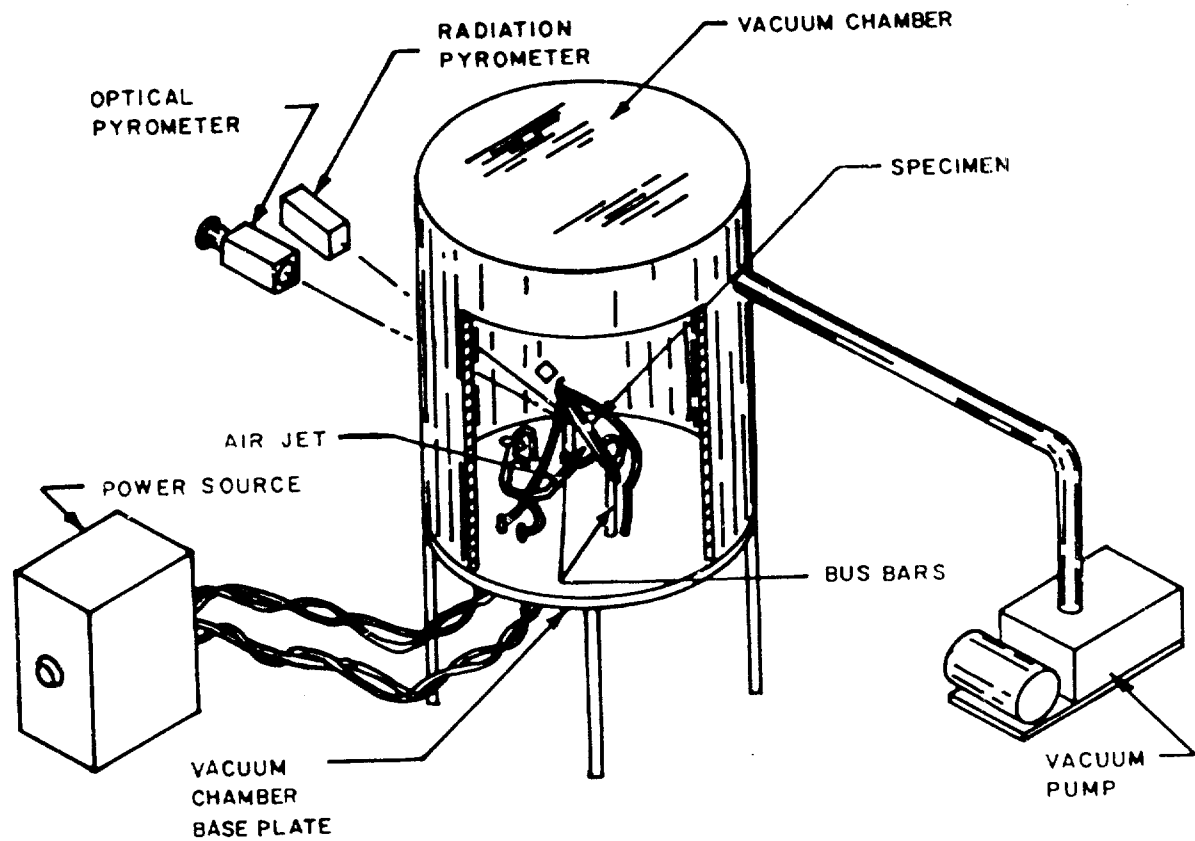


Figure 35. Environmental Simulator

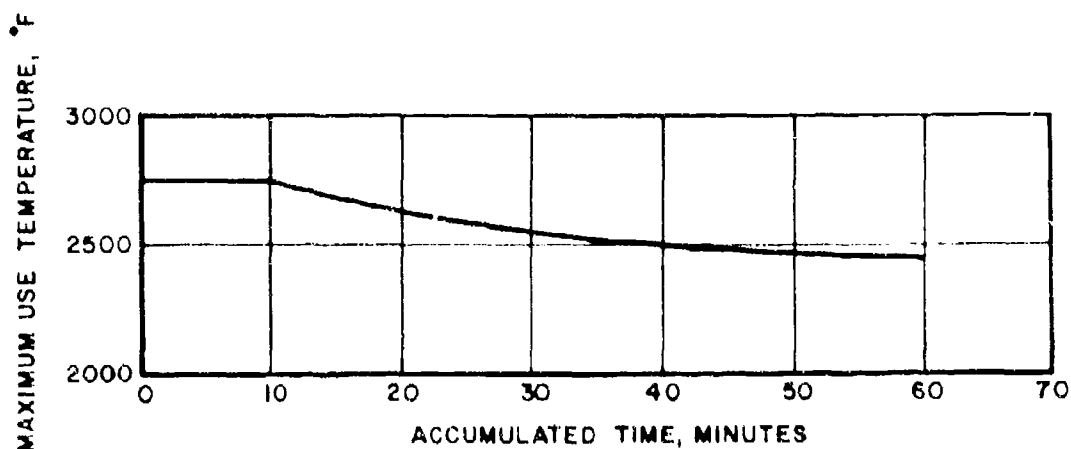


Figure 36. D-36 Maximum Use Temperature Vs Time

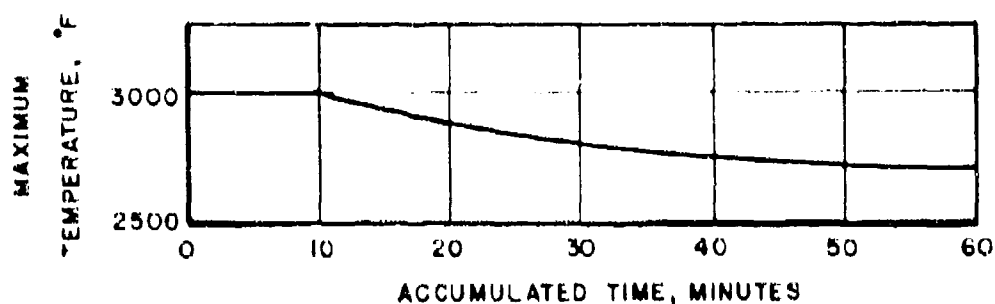


Figure 37. T2M Maximum Use Temperature Vs Time

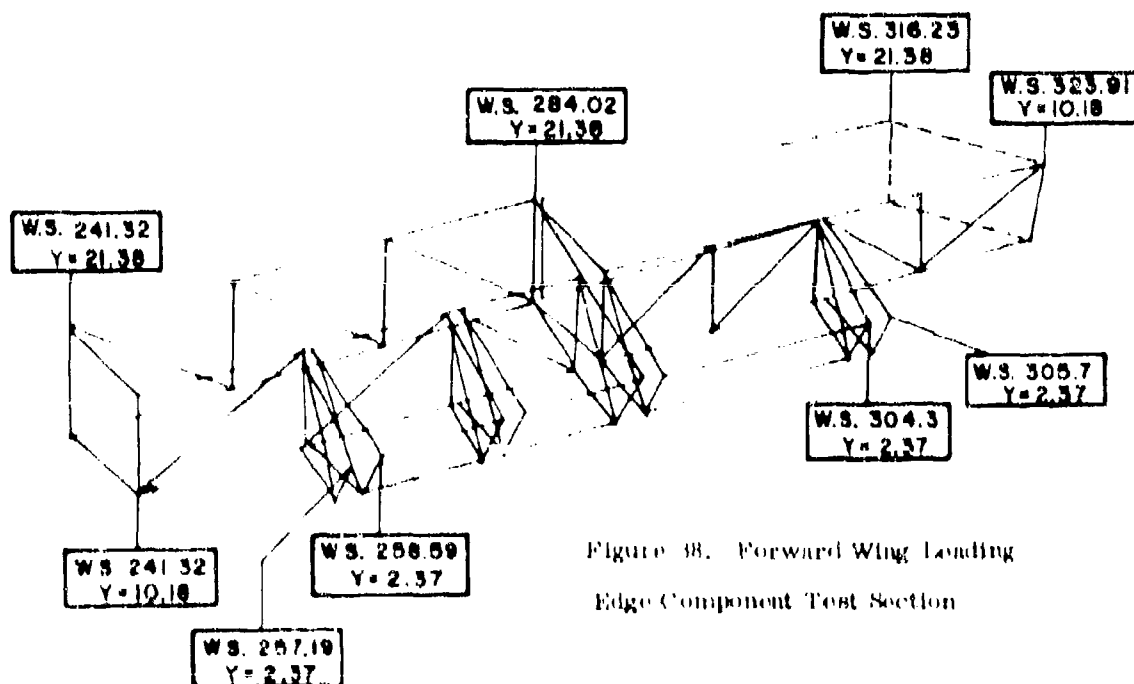


Figure 38. Forward Wing Leading Edge Component Test Section

CONCLUSIONS

The state-of-the-art in high-temperature heat-protection systems and in utilization of refractory metal was advanced greatly during the life of the X-20 program. A heat-protection system capable of resisting 3000°F for long periods of time was developed. A production facility for coating, which produced uniform and reliable coatings, was developed and utilized. A test facility, which tested coated refractory specimens under conditions of high temperature, reduced pressures, and mass flow, was developed and utilized.

RECOMMENDATIONS

1. Because of the interplay between the refractory base metal and the silicide coating and the resultant detrimental effect on the properties of base metals, the combination of material and coating should be investigated as a system, not independently of each other.
2. The investigation of the oxidation behavior of silicide coatings on refractory metals for re-entry applications should be accomplished in reduced pressure environments.
3. The problems of manufacturing should be considered when coated refractory metal components are designed. Early fabrication and testing of proposed flight-type designs should be accomplished so as to eliminate excessive design development.
4. Ductile coated refractory metals, such as the advanced columbium alloys and tantalum alloys, should be investigated as substitutes for molybdenum alloys.
5. Due to the brittle nature of the coated refractory metal system, this type of heat protection system should be utilized only with a basic structure that exhibits small deflections under conditions of load and thermal environments.

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