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DIFFUSION BONDING OF TITANIUM TO SIMULATE S-IC FUEL TANKAGE COMPONENTS

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ABSTRACT

This publication presents the results of recent contractual and MSFC in-house efforts for the development of manufacturing methods applicable to the future production of large titanium alloy structures which simulate S-IC fuel tankage components.

The report shows that large simulated titanium alloy gores, skin, and Y-ring segments can be fabricated to the design requirements of the S-IC fuel tank utilizing advanced diffusion bonding technology. This should be directly applicable to the future production of launch vehicle structures which specify a titanium alloy material. Although the roll diffusion bonding technology in this report is concerned primarily with launch vehicle structural development, the information should also be of value to other applications such as the upper flight stages where strength to density ratio and integral construction are important considerations.

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MANUFACTURING ENGINEERING LABORATORY
RESEARCH AND DEVELOPMENT OPERATIONS

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DIFFUSION BONDING OF TITANIUM TO SIMULATE S-IC FUEL TANKAGE COMPONENTS

SUMMARY

This publication presents the results of recent contractual and MSFC in-house efforts for the development of manufacturing methods applicable to the future production of large titanium alloy structures which simulate S-IC fuel tankage components.

The report shows that large simulated titanium alloy gores, skin, and Y-ring segments can be fabricated to the design requirements of the S-IC fuel tank utilizing advanced diffusion bonding technology. This should be directly applicable to the future production of launch vehicle structures which specify a titanium alloy material. Although the roll diffusion bonding technology in this report is concerned primarily with launch vehicle structural development, the information should also be of value to other applications such as the upper flight stages where strength to density ratio and integral construction are important considerations.

INTRODUCTION

The work accomplished in the development of diffusion bonding techniques for large structural components using the basic S-IC aluminum alloy 2219-T87 fuel tank as a configuration concept is presented herein. The results of the processing required negate consideration of Ti-8Al-1Mo-IV in the development of large titanium structures, especially those structures such as simulated titanium S-IC tanks which require welding.

Three MSFC contractual programs were established early in 1965 to investigate solid-state diffusion bonding. North American Rockwell Corporation investigated roll diffusion bonding on the skin and Y-ring and The Boeing Company investigated static diffusion bonding of a port embossment on a gore part. This work has been completed and the final reports on these programs are available

[1 - 3]. In addition, because of the advanced diffusion bonding technology developed, a NASA-MSFC film titled "Roll Diffusion Bonding" sponsored by the Office of Technology Utilization is available.

The purpose of this report is essentially twofold: (1) review the technology that established the capability to fabricate typical large and complicated launch vehicle shapes from titanium alloys by diffusion bonding and (2) provide guidance for further titanium development from a manufacturing viewpoint. This report describes the manufacturing development and evaluation of the large titanium parts.

MANUFACTURING DEVELOPMENT

The objective of this titanium manufacturing program is the development of technology applicable to fabricating heavy gauge titanium alloys into structures such as the basic S-IC fuel tank. This welded structure currently employs 2219-T87 aluminum alloy. Should the need for a greater payload capability arise, reliable titanium manufacturing process data will be appropriate.

S-IC Fuel Tank Orientation

This structure has a 10 m (33 ft) diameter shell, monocoque design, which carries the stresses of the fuel, sustains the weight of the stages above, and endures the rather severe shock and vibrational stresses of the five F-1 engines. Because each engine generates at least 2.0×10^6 joules (1.5×10^6 ft-lb) of thrust, it is apparent that the S-IC fuel tank is a critical structural configuration. This is different from the usual concept of tanks because the S-IC fuel tank is primarily designed to resist buckling loads. Adaptation of this tank to the selected titanium alloy is ideal because there already exists base line aluminum alloy data for comparative purposes. Tooling and past experience gained from the aluminum tank assembly can be applied with modifications to titanium assembly tools and, in some instances, to the fabrication of the titanium tank components.

The welded cylindrical framework of the tank (Fig. 1) consists of tee type stiffened skin segments and the upper and lower bulkhead Y-rings. The bulkheads consist of welded ellipsoidal shaped, closed rib plate base and apex gores — with a centerpiece closure weld. The gores have various port embossments for attachment of the fuel inboard and outboard elbows, the five

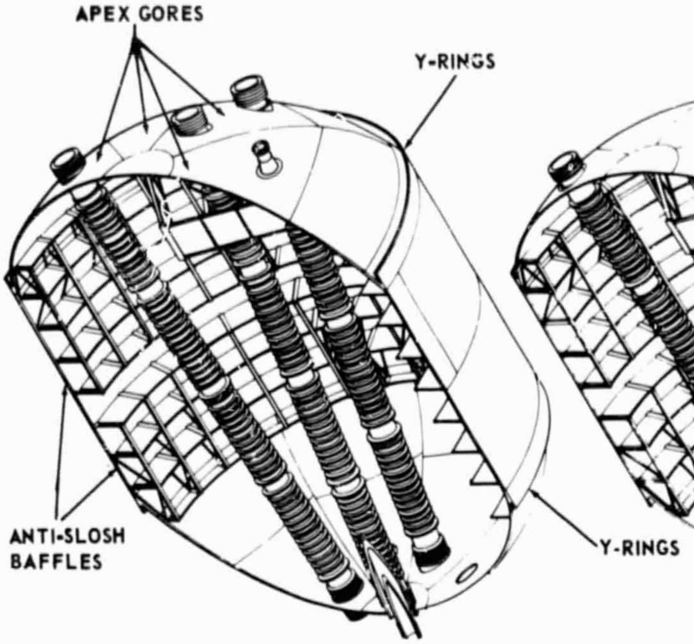

FIGURE 1. S-IC FUEL TANK

liquid oxygen (LOX) ducts which run through the tank, and other openings for relief and pressure sensing lines. Anti-slosh ring baffles are attached to the tee sections of the skin panels inside the tank. At the bottom of the tank are the cruciform support and fuel exclusion riser.

Program Approach

The approach to this titanium manufacturing development was to take the S-IC fuel tank and adapt it for the selected titanium alloy. For this purpose, contracted technology was undertaken in designing, manufacturing, and fabricating full-scale Ti-8Al-1Mo-IV skin, Y-ring, and base gore segments. The MSFC Propulsion and Vehicle Engineering Laboratory primarily directed design aspects and the Manufacturing Engineering Laboratory established a reliable manufacturing process.

Roll diffusion bonding is a method of joining individual titanium pieces (packed and oriented in a suitable matrix using machined low carbon steel filler bars and welded air-tight with steel facing sheets) under heat, time, and pressure by roll reduction of the pack. This was the principal developmental area selected for the skin and Y-ring programs. Static diffusion bonding was selected as one of the principal developmental areas for diffusion bonding a doubler ring to a reinforced port opening on a base gore. The other manufacturing area was the investigation of hot temperature forming for the gore part. Work conducted within the Manufacturing Engineering Laboratory was directed toward establishment of processes such as cleaning, joining, forming, thermal treating, and stress relieving. Assembly plans including test fixtures and procedures to load test an assembled partial structure were prepared cooperatively with stress personnel from MSFC's Propulsion and Vehicle Engineering Laboratory.

Plans to load test an assembled partial titanium structure have been indefinitely suspended; however, further work is planned relative to welding diffusion bonded components and investigation with non-thermal stress relief equipment.

ROLL DIFFUSION BONDING

The feasibility of preparing structural panels by the roll diffusion bonding process was established at Battelle Memorial Institute, Columbus, Ohio. Battelle Memorial Institute has proprietary interest in the process and has made available an excellent treatise on the Roll Diffusion Bonding of Structural Shapes and Panels [4].

A review of the skin and Y-ring segments of the simulated titanium S-IC fuel tank and the MSFC in-house support are presented in this section to delineate problem areas still remaining in the area of roll diffusion bonding.

Development of a Simulated Titanium S-IC Skin Section

Roll diffusion bonding was selected as the principal method of fabrication to be studied in the accomplishment of this program's objective. A secondary objective was to establish Ti-8Al-1Mo-1V design criteria and process parameters for the fabrication of simulated skin sections to fulfill design requirements of the S-IC fuel tank. At the time this investigation began, little was known concerning roll diffusion bonding of Ti-8Al-1Mo-1V. Laboratory data indicated that under controlled metallurgical conditions (heat, time, and pressure), quality acceptable joining with bond strength equal to or better than that of the parent metal can be readily accomplished.

The transition from the laboratory to subscale parts to full scale parts representative of the S-IC fuel tank was accomplished on this program and is described in the final report [1]. This report describes engineering studies, design effort, the fabrication of 8Al-1Mo-1V titanium alloy roll diffusion bonded, subscale and full-scale panels, the fabrication of a panel assembly for testing by NASA/MSFC, and the laboratory testing and evaluation of roll diffusion bonded material from the subscale and full-scale panels.

In preparation for rolling, individual machined titanium parts are arranged in a vacuum-tight welded steel pack, using machined steel to fill in the space and maintain arrangement around each titanium part. The manufacture of titanium full scale skin sections will be briefly described in this report.

<u>Synopsis.</u> In brief, this work is an advancement in the state-of-the-art in roll diffusion bonding of large titanium structures such as the simulated S-IC cylindrical skins. The results can be applied to other titanium alloys with adaptation of techniques developed; however, at the same time, it is clear that further work needs to be accomplished before the process is ready for quality production of full scale parts. The major problem area to be resolved is solution of thermal expansion and contraction problems which caused severe damage to titanium panels in the areas at both ends of the pack, especially the two full-scale packs. This may not be a major problem area if the titanium alloy were 6Al-4V or 5Al-2.5 Sn because they require considerably lower rolling temperatures than Ti-8Al-1Mo-1V. As a result, the entire reduction operation could be accomplished faster and with less heat loss. Also, there is a broader band for safety with Ti-6Al-4V between the beta transus and proper roll diffusion bonding temperature than there is for Ti-8Al-1Mo-1V.

A somewhat parallel MSFC in-house effort was conducted in 1966 and 1967 to extend the state-of-the-art of roll diffusion bonding.[1] Three extremely close fitting packs with different yoke configurations were fabricated, assembled, reduced 60 percent in thickness by hot rolling, and then evaluated. Results of the study showed end separation and deformation near the sides of the fabricated

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1. Wood, C. M.: Roll Diffusion Bonding Development, Internal Note R-ME-68-4, Manufacturing Engineering Laboratory, Marshall Space Flight Center, Huntsville, Alabama, June 1968.

part were not a result of yoke geometry or a loose fitting, but appeared to be inherent characteristics from the rolling operation.

Discussion. In order to weld quality acceptable Ti-8Al-1Mo-1V roll diffusion skin panels, it is preferable to have the material in the duplex annealed condition and free of residual stresses. Material that has been exposed above the beta transus temperature of 1310°K (1900°F) is ruined for welding purposes. Also, material that is procured in the duplex annealed temper will not remain in this condition because to roll diffusion bond Ti-8Al-1Mo-1V properly, temperatures close to the beta transus temperature must be employed [1255°K to 1295°K (1800°F to 1870°F)]. Any error in the Ti-8Al-1Mo-1V manufacturing sequence, such as exceeding the beta transus temperature, can be disastrous.

It is difficult to procure acceptable Ti-8Al-1Mo-1V in the sheet and plate thicknesses desired for this program. The starting stock should be in the mill annealed temper. Any other temper may initiate an ordering embrittling reaction when the material is reheated to a temperature such as that required for roll diffusion bonding. In addition, the cool down rate of the packs has a direct bearing on the welding performance. Failure to reduce the temperature of the rolled pack from 1060°K to 755°K (1450°F to 900°F) in less than one hour can also initiate the ordering embrittling reaction associated with Ti-8Al-1Mo-1V or ruin the material for welding purposes, unless it is re-solution heat treated and annealed properly.

Surface Contamination. To remove surface contamination and surface cracks to produce a quality acceptable part, it is necessary to flash chem-mill roll diffusion bonded parts to at least a depth of 0.005 cm (0.002 in.). Titanium has an affinity for elements such as hydrogen, oxygen and nitrogen, especially at high temperatures; consequently, it is necessary to minimize interstitial pick-up by careful manufacturing techniques. These techniques include the use of white gloves, proper cleaning and degassing of steel inserts, and the use of water-free Argon for purging the packs before they are vacuum sealed. With proper manufacturing techniques the interstitial contents of the finished parts can be kept within specified limits.

The manufacturer has difficulty avoiding the beta phase contamination as a result of exceeding the beta transus temperature. This is a result of the direct contact of the steel filler bars generating additional heat under pressure when rolling at a temperature near the beta transus. Thus, it was mandatory to develop a rolling schedule which would give satisfactory bonding while never exceeding the beta transus temperature.

Manufacture and Evaluation of K Pack Skin Section. This was one of the two full-scale packs rolled at the Homestead Works, U. S. Steel, Homestead, Pennsylvania. Considering that this was the second full-scale pack of this size ever rolled, the accomplishment was indeed an advancement in the state-of-the art even though further development is required to minimize or eliminate the steel shrinkage problem and to effectively prevent the titanium from adhering to the steel filler bars. An illustration of Pack "K" as rolled is presented in Figure 2. Testing for void areas as revealed by sounding examination and

FIGURE 2. PACK "K" AS ROLLED

surroundings of the pack is shown in Figure 3. Void areas at one end of the rolled pack are shown in Figure 4. Figure 5 shows the withdrawal of the titanium panel from the etchant tank used to leach the already rough machined steel filler bars. This pack was machined into specific sizes for use in the final assembly, for backup parts, and for laboratory test pieces of various sizes. The panel sections were then sent to Automation Industries for flash chemical-milling, a process which cleaned the titanium by etching 0. 005 cm (0. 002 in.) from all surfaces. The sections were then wrapped in a clear plastic material for protection until needed for welding operations.

MSFC In-House Metallurgical Evaluation of Pack "K" Material. The material from a test section of "K" pack with two longitudinal welds was sent to MSFC's Manufacturing Engineering Laboratory for evaluation. Radiographic examination of the welds January 1968 and April 1968 showed that Class I and

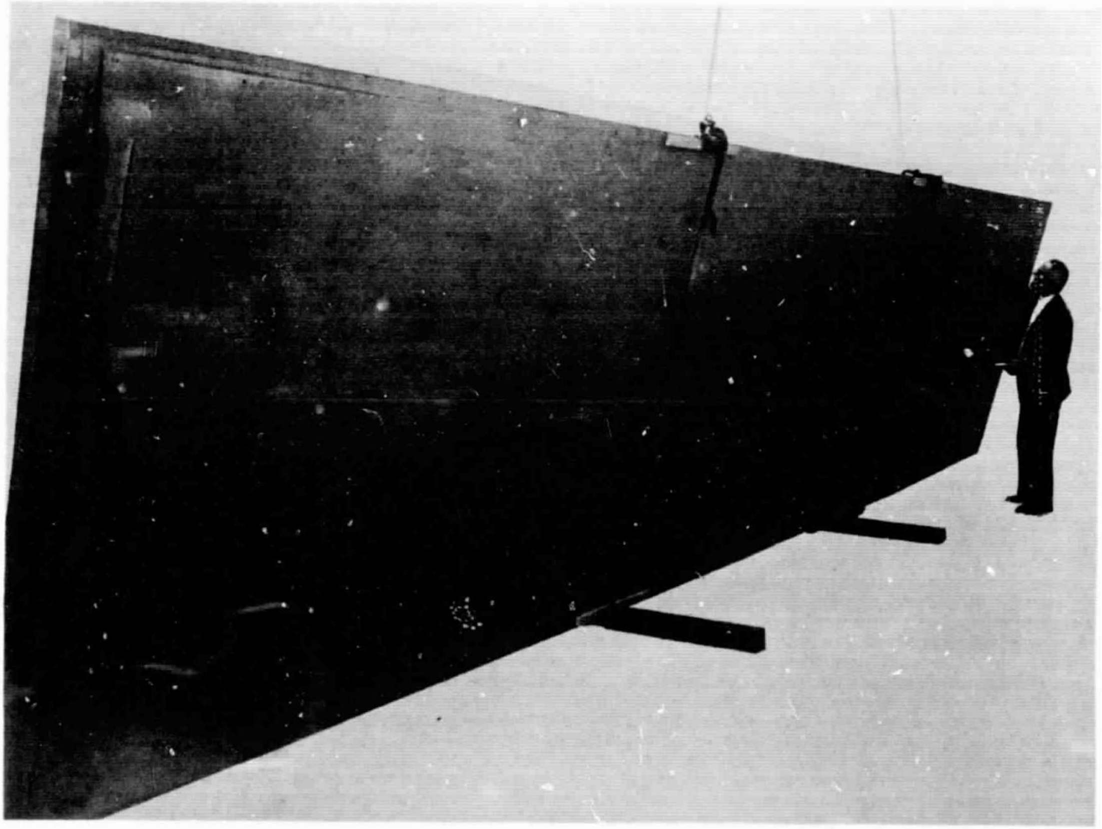

FIGURE 3. TESTING FOR VOIDS

II welding was accomplished and that no cracks developed while exposed to the atmosphere encountered during this period. Based on mechanical properties and equiaxed alpha-beta structure, it was also determined that the material was essentially duplex annealed. Inspection of the welding data revealed that this panel was stress-relieved after welding. This was accomplished in a stainless steel vacuum retort with the panel contoured between blankets of stainless steel honeycomb core and the requirements of the duplex annealing process were adhered to 1060° K (1450° F) and cooled to 755° K (900° F) in less than one hour.

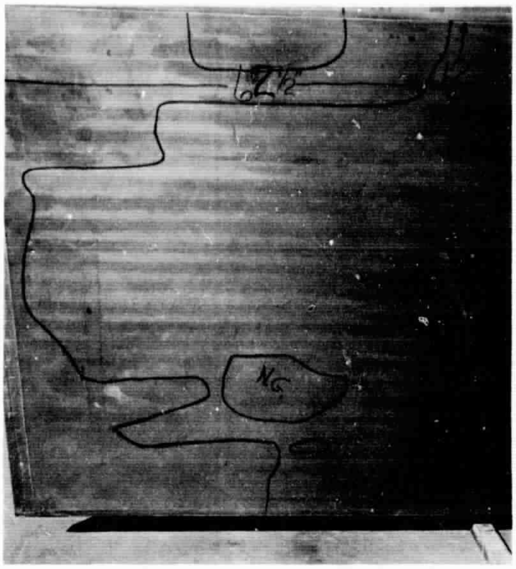

FIGURE 4. VOID AREAS AT ONE END OF A ROLLED PACK

However, incongruous but now somewhat explainable data was found

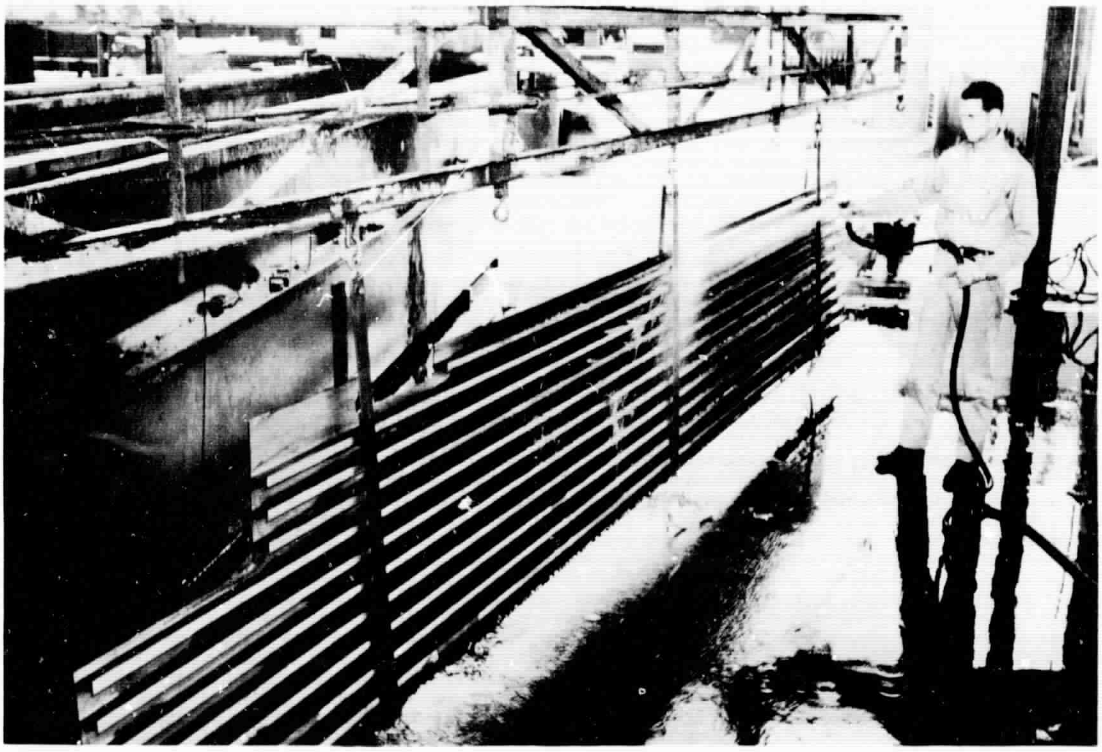

FIGURE 5. WITHDRAWING PACK FROM LEACHING TANK

when a section of this panel was given an additional heat treatment of the duplex type involving solid solution equilibration between 1172°K (1650°F) and 1228°K (1850°F). This was followed by stabilization annealing for eight hours at 810°K (1000°F) to 866°K (1100°F) employing a vacuum furnace and Argon as the cooling gas. No cooling rate was established. However, it is known that intermediate cooling rates result in mechanical properties which are intermediate to those of mill and duplex-annealed material.

Fine cracks or fissures were noted approximately two weeks after the thermal treatment. This experiment was repeated to verify that the cracks were caused by the thermal treatment and were not present beforehand. This is explained by the embrittlement ordering that occurred in the slow cool down. These fine cracks or fissures were readily removed by a MSFC in-house flash chem-milling etch to give a quality acceptable surface finish. The material probably remained of non-quality acceptable weldability based on the ordering embrittlement reaction and experience of North American Rockwell when attempting weld repairs. This was listed as one of the several major obstacles which must be overcome before roll diffusion bonding of Ti-8Al-1Mo-1V is acceptable as a production process.

Development of a Simulated Titanium S-IC Y-Ring Segment

North American Rockwell was given the task of establishing design criteria and process parameters for a simulated Ti-8Al-1Mo-1V Y-ring which met all the requirements of the 2219-T81 aluminum alloy S-IC fuel tank Y-ring. This resulted in a weight savings of 32 percent over the current aluminum Y-ring and the subsequent final report [2] outlines the details of the process.

Some of the processing problems requiring resolution are discussed in this extraction from the final report:

Thermal Treatment. Ti-8Al-1Mo-1V alloy sheet in the duplex annealed condition exhibits an excellent combination of fracture toughness, strength, and weldability. The use of duplex annealed material also permits fusion welding the alloy without cracking and potentially eliminates the need for postweld stress relieving.

The duplex annealing treatment is performed as a two-step operation, involving achievement of metallurgical equilibrium at 1060°K (1450°F) to obtain the desired alpha-beta phase relationship and cooling from 1060°K (1450°F) at a rate sufficient to prevent initiation of the embrittling ordering reaction associated with Ti-8Al-1Mo-1V and other high aluminum titanium alloys. In sheet gages this rate is readily achieved by air cooling. The nature of the time temperature ordering reaction in high aluminum titanium alloys is such that the material, even if properly duplex annealed, will undergo ordering when reheated to moderately high temperatures for sufficient lengths of time. This effect is shown for Ti-8Al-1Mo-1V in data obtained from Titanium Metals Corporation of America presented in the time-temperature embrittlement curve shown in Figure 6. Notch tensile tests using NASA sharp notch tensile specimens with a maximum root radius of 2.54 μm (0.0001 in.) were used to indicate loss in toughness or degree of embrittlement associated with the ordering phenomenon. The time dependency of the ordering reaction is evident from the decreasing values of notch tensile strength shown in parentheses and expressed in ksi at several temperature levels. Selection of a notch tensile strength of 827.4 MN/m^2 (120 000 psi) as the criterion for toughness permits developing the curve, as indicated, describing the limiting time and temperatures for heating duplex annealed Ti-8Al-1Mo-1V to prevent initiation of ordering embrittlement. The curve also permits development of the critical cooling rate necessary for cooling the material from the duplex annealing temperature, 1060° K (1450° F).

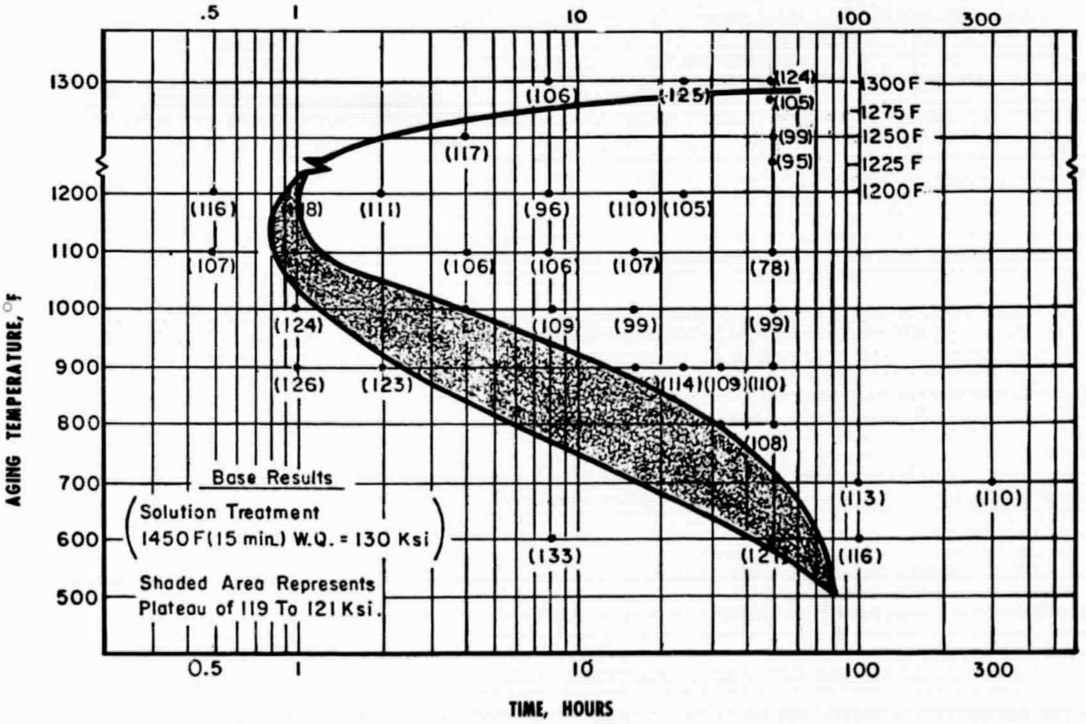

FIGURE 6. TIME-TEMPERATURE-EMBRITTLEMENT CURVE FOR Ti-8Al-1Mo-1V

Attainment of duplex annealed properties in the Ti-8Al-1Mo-1V structure necessitates the development of appropriate thermal processing cycles either during or subsequent to bonding due to the erasure of prior thermal history by the high bonding temperatures. This has permitted procurement of raw material in the less expensive mill-annealed condition.

The need for the use of heat in excess of 810° K (1000° F) for substantial lengths of time to form design contours in the postbonded structure eliminates the practicality of attaining duplex annealed properties during the bonding cycle because subsequent hot forming would disrupt the duplex annealed condition and order-embrittle the material. This has led to development of combined hot forming/thermal processing cycles to produce the properties desired.

The approach taken involves heating the roll-bonded pack to the forming temperature, progressively contouring the pack through successive stages of dies and permitting the final formed segment of the structure to cool in air as it emerges from the furnace. The forming temperature, 1060° K (1450° F), is selected to provide ease in contouring. The sequence of 10-minute increments at 1060° K (1450° F) in each die stage is designed to permit soaking at 1004° K (1350° F), as indicated in Figure 6. It is recognized that a stepwise temperature gradient will exist between 1060° K (1450° F) and lower temperatures. However, it is believed that 10-minute maximum sojourn at intermediate temperature will result in the accumulation of less than 30 minutes at temperatures in excess of 755° K (900° F), well within the limit for beginning of ordering embrittlement.

Y-Ring Segment Program Conclusions.

1. Large titanium structures up to at least 12.2 m (40 ft) in length can be successfully roll diffusion bonded to produce useful structures for application to booster vehicles.

2. Large titanium structures can be designed to yield weight savings as compared to conventional aluminum structures.

3. Large titanium structures can be made to be cost competitive with conventionally fabricated titanium structures.

4. Incremental hot creep forming using relatively small tooling dies can be effectively utilized to form large titanium structures into complex shapes. The iron filler material in intimate contact with the titanium serves a very useful purpose in stabilizing the titanium structures so that they can be shaped with relative ease.

5. Titanium alloy 8Al-1Mo-1V is more difficult to fabricate reliable structures from than the titanium alloy 6Al-4V. This includes the roll diffusion bonding process.

6. The procedures used and established for fabricating and processing large titanium structures in this program using titanium alloy 8Al-1Mo-1V can be successfully applied to other titanium alloys such as 6Al-4V with only small changes in processing details.

7. A small loss in mechanical properties occurs during the roll diffusion bonding process as compared to as-received material.

8. Diffusion bond joint strengths are equivalent to the parent material.

9. Chemical milling to remove surface contamination and improve fillet radii is an important processing technique in fabricating roll diffusion bonded structures.

In summation, serious consideration should be given to the use of 6Al-4V titanium alloy in lieu of 8Al-1Mo-1V. As a result of the increased aluminum content of Ti-8Al-1Mo-1V over Ti-6Al-4V, this alloy is more difficult for the producers to roll into sheet and to maintain good finish gage control. Although mill producers have produced Ti-8Al-1Mo-1V sheet within the required gage tolerances, they have not been able to obtain required gage tolerances each time they roll into sheet. This fact could result in a delay in procurement of the needed material programs.

Ti-8Al-1Mo-1V sheet is available in three conditions of heat treatment: single, duplex, and triplex annealed. Duplex annealed sheet is the most advantageous for use due to its increased fracture toughness characteristics despite a lower yield strength than single annealed material. Utilization of duplex annealed material, however, presents some problems. A diffusion bonding temperature of approximately 1251° K (1835° F) destroys the desired duplex annealed properties. As a result, parts have to be reheat-treated following diffusion bonding. To restore the duplex annealed properties, the parts require reheating to 1060° K (1450° F) then must be air cooled. The air cooling step is critical and ideally should be done by reheat-treating only the bonded part. Reheat-treatment results in increased costs.

Titanium alloy diffusion bonding studies and bonding procedures have been carried out and developed primarily using Ti-6Al-4V on other programs. The results of these programs indicate a much easier dimensional control is possible. This is a result of the lower roll bonding temperature with 6Al-4V titanium, thereby allowing the steel to provide better support during the deformation process. At the 8Al-1Mo-1V roll bonding temperature the steel deforms more severely, distorting the titanium details being joined.

MSFC In-House Effort

Details of the roll diffusion bonding development conducted at MSFC are described in a separate report.[1] This program was established to extend the state-of-the-art in roll bonding as a fabricating technique for high strength alloys by fabricating simulated skin sections from Ti-8-1-1. This work supplemented the work done by North American Rockwell Corporation on the titanium skin panel program.

The test was to design, fabricate, and test three 305 × 122 cm (12 × 48 in.) skin panels using two yoke designs and one conventional design for comparison. The subscale packs measured 21.6 × 45.7 × 60.7 cm (8.5 × 18 × 24 in.) prior to rolling. When heated to 1275°K (1835°F) and rolled to 60 percent reduction in thickness, the packs measured 8.25 × 45.7 × 152 cm (3.25 × 18 × 60 in.).

The two yoke concepts were especially designed to determine whether end separation and deformation of the tee stiffeners were caused by yoke design, loose fitting packs, or inherent characteristics from the rolling operation. In addition, yoke and filler bars were made of AISI-1050 carbon steel rather than AISI-1018 carbon steel used by North American Rockwell Corporation. Also, high purity titanium sheets were omitted under the MSFC effort while North American Rockwell Corporation used these sheets to prevent bonding of the face plates to the titanium parts and filler bars.

While the AISI-1050 carbon steel matrix did prevent bonding of the titanium to the steel, all three of these packs encountered end separation during the rolling operation. Consequently, the conclusion reached on this program was that the end separation and deformation of the part at the outside edge were caused by the rolling operation and not by a loose fitting pack. It was determined that the design of the yoke had no significant effect on the end separation and straightness of the final parts in this program.

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1. Wood, C. M.: Roll Diffusion Bonding Development, Internal Note R-ME-68-4, Manufacturing Engineering Laboratory, Marshall Space Flight Center, Huntsville, Alabama, June 1968.

STATIC DIFFUSION BONDING

Rather than joining materials by roll diffusion bonding which involves gross plastic flow, such as rolling a pack to 60 percent reduction in thickness, static diffusion bonding involves only microscopic plastic flow. For joint efficiencies approaching 100 percent, it is necessary to employ temperatures close to the beta transus when diffusion bonding the titanium alloy 8Al-1Mo-1V. The results of the development of a simulated titanium S-IC base gore segment program conducted by The Boeing Company have been reviewed and are presented in the following paragraphs.

Development of a Simulated Titanium S-IC Base Gore Segment

The present S-IC base gores for the lower bulkhead of the fuel tank are bulge formed from 2219-T37 aluminum alloy sculptured panels. These panels are machined from a plate on a numerically controlled skin mill at a constant temperature to maintain dimensional requirements, then aged to the -T87 temper. The objective of this program was to establish design requirements and process parameters for fabricating similar base gore segments from Ti-8Al-1Mo-1V mill annealed plate instead of from 2219-T87. State-of-the-art advancements were made in (1) diffusion bonding a 1.27 cm (0.50 in.) doubler ring to a basic plate thickness of 1.27 cm (0.50 in.) to provide the required reinforcement at a fuel line port, (2) the rolling of large titanium plates, (3) the development of a low cost, high temperature ceramic die for titanium forming, and (4) technical improvements for titanium hot flattening and the use of ultrasonics to inspect diffusion bonded port reinforcement.

Highlights of the basic feasibility work accomplished were extracted from the final report [3] for this report.

1. Processing methods were developed for producing the largest 8Al-1Mo-1V titanium plates made to date [2.44 × 3.96 × 0.0127 m (96 × 156 × 0.50 in.)].

2. The titanium basic gore design, sized to have an equivalent load carrying ability of the present 2219-T87 aluminum alloy gore, has a calculated weight of 95.71 kg (211 lb). This is a weight reduction of 29.48 kg (65 lb) or 23.6 percent. Material costs, assuming a straight-line cost per pound, were

kept at one-half the cost of a plate thick enough to provide reinforcement around the fuel line hole by diffusion bonding a 1.27 cm (0.50 in.) titanium ring to a 1.27 cm (0.50 in.) titanium plate.

3. Time, temperature, and pressure cycles were developed for diffusion bonding titanium to titanium in air, eliminating the need for protective atmospheres. A semi-portable, highly versatile tooling concept was also devised.

4. Hot flattening and stress relief cycles were developed to alleviate stresses and distortion caused by localized heating during bonding. Correlation of ultrasonic tests with metallographic examination of diffusion bonds provides a basis for development of a reliable production proof-test.

5. A full size, high temperature ceramic die was designed, fabricated and cycled to 1060°K (1450° F) eight times for a total operating time of 60 hours with no malfunction or indication of degradation.

6. The design, tooling, and fabrication methods were demonstrated by diffusion bonding a ring doubler to a full size 8Al-1Mo-1V titanium plate, then sculpturing this assembly in the flat and "hot" vacuum forming it to contour.

Naturally, before the process is ready for quality acceptable production of titanium base gore segments, further research and development is required. In 1962 and 1963, during the production of the first 2219-T87 parts for the component test structure of the fuel tank for the S-IC stage, an extensive research and development program was undertaken before quality acceptable parts were produced. The parts used at that time would have been marginal quality acceptable parts for flight stages of the current Saturn V vehicles.

Titanium Base Gore Fabrication Sequence. Figure 7 illustrates the fabrication sequence for the full size titanium gore, and Figure 8 illustrates a cut-away view of the integrally heated ceramic gore forming die.

The basic process consists of:

1. Obtain raw material 2.44 × 3.96 × 0.0127 m (46 × 156 × 0.50 in.),
2. Machine fitting hole in plate,
3. Machine fitting ring out of excess material from corner of plate,

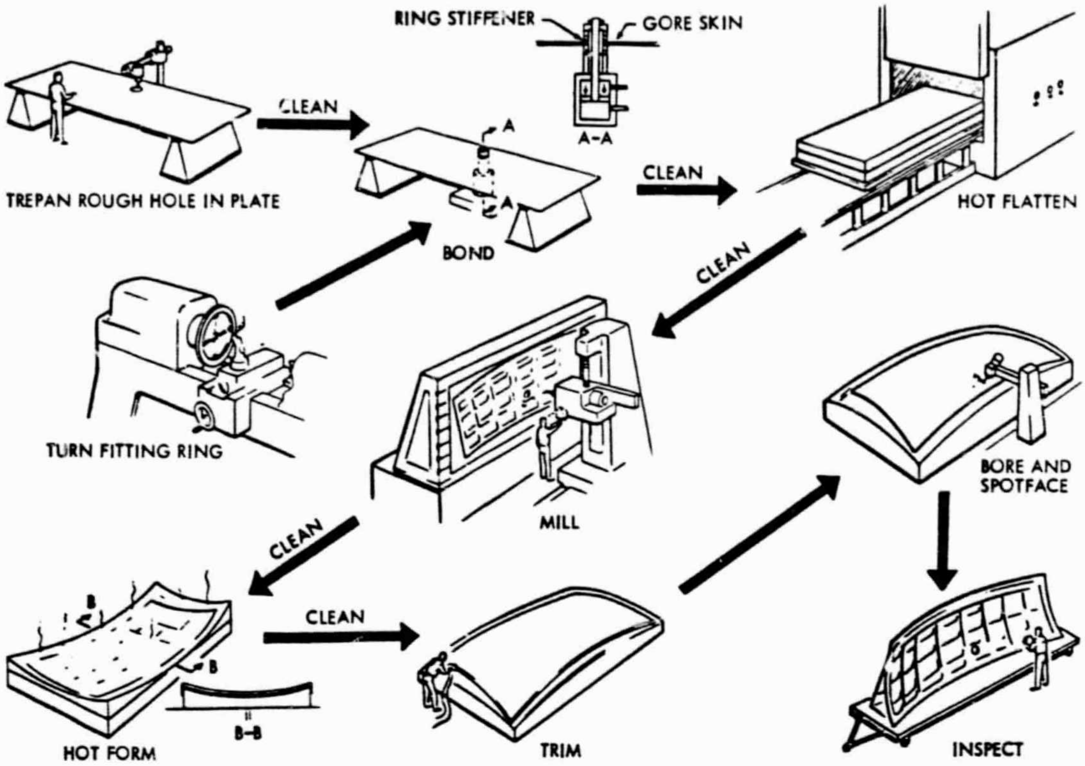

FIGURE 7. FABRICATION SEQUENCE CHART

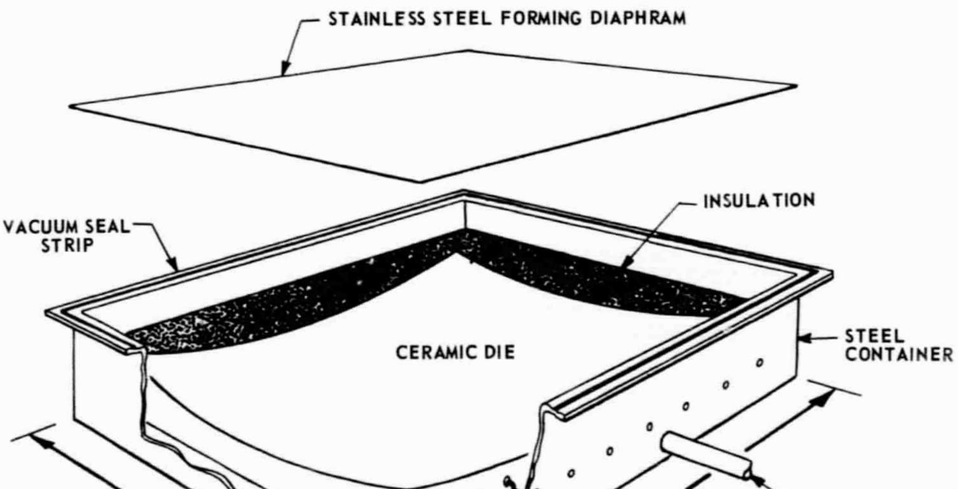

FIGURE 8. INTEGRALLY HEATED CERAMIC GORE FORMING DIE

4. Clean plate and ring with methyl ethyl ketone (MEK) prior to bonding,

5. Diffusion bond,

6. Clean bonded assembly and hot flattening tools, wipe with MEK and apply 3 coats of Formcoat (Everlube) T-50 protective coating to the part and tools before stress relieving and hot flattening,

7. Hot flatten,

8. Clean scale off the part by pickling in a nitric hydrofluoric bath and rinse before machining,

9. Profile mill pockets in plate,

10. Alkaline clean and rinse prior to forming,

11. Hot vacuum form to contour,

12. Pickle scale off and rinse,

13. Trim, bore, and spotface, and

14. Alkaline clean and inspect.

Figure 9 depicts a formed gore in the ceramic forming die and Figure 10 shows a view of the other side of the gore part prior to pickling to remove scale.

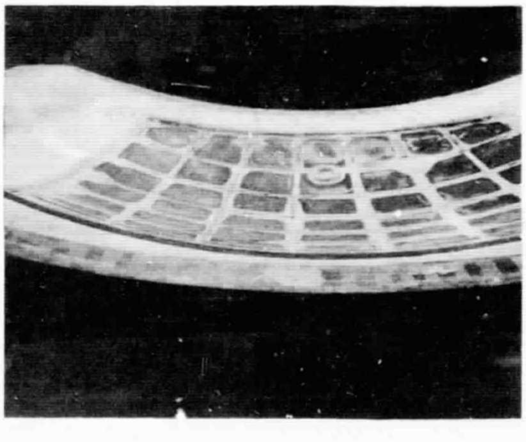

FIGURE 9. FORMED GORE IN THE DIE

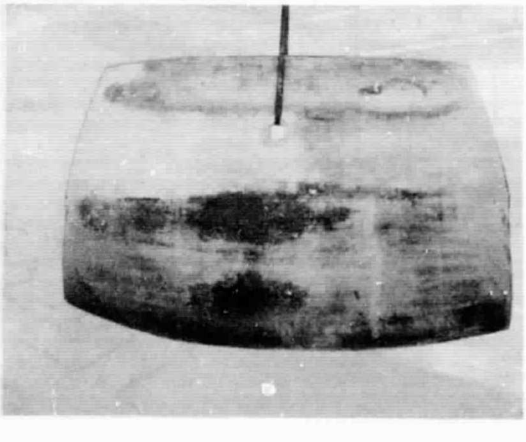

FIGURE 10. SIDE VIEW OF FORMED GORE

Base Gore Material Experience. The choice of Ti-8Al-1Mo-1V early in 1965 proved from the outset of this program inadequate from the viewpoint of availability and material quality acceptance. It must be remembered that this was pushing the state-of-the-art in rolling large plates and created a new frontier for Ti-8Al-1Mo-1V. Initially, no forewarning of major difficulties was foreseeable based on The Boeing Company's experience in the use of experimental samples of the material. This was substantiated by the MSFC in-house work [5] and the fact that two suppliers provided quotations to provide the material to Boeing's material purchase specifications. However, rolling the material to 1.27 cm (0.50 in.) thick, the 2.4- × 4.9-m (8- × 13-ft) Ti-8Al-1Mo-1V alloy plate in the mill annealed condition was at best a compromise. An appreciation of this materials experience is believed best cited by the following excerpts from the final report [3].

Following negotiations with two vendors for the purchase of the three titanium plates, a vendor was selected and approval was received from MSFC. The order was placed with delivery scheduled in January 1966.

The week before the material was to arrive the vendor informed The Boeing Company of a problem in rolling the material at the mill. In satisfying the purchase specification of not allowing the material to be heated above the beta transus temperature of 1310°K (1900°F), the material had been rolled at too low a temperature and surface cracks had appeared on the plates when they were still 0.64 m (2.5 in.) thick. The vendor gave Boeing a choice of accepting the present material or waiting three months for a new run of material. Boeing, with the concurrence of MSFC, elected to wait three months because the plates with the cracks would not clean up to the 2.4- × 4.9-m (8- × 13-ft) dimension after the cracks were ground out and rolling was continued to the 1.27 cm (0.5 in.) dimension. The vendor also requested and received approval to roll subsequent material at a higher temperature [1337°K (1950°F)] to preclude cracking.

The three 8-1-1 titanium plates were received at Boeing on May 16, 1966. Prior to shipment, the vendor advised that the material failed to satisfy surface finish, thickness, and properties requirements. To reject this material would mean another three or four months delay in the program, and with this material the major objectives of the program could be met. On this basis permission was requested and granted from MSFC to accept the below specification material.

The material was tested for properties by the vendor before it was shipped, it received a thorough receiving inspection at Boeing before it was processed, and Part No. 002 was further tested for mechanical properties after forming.

The properties of the three plates of material, as tested by the vendor, are presented in Table I.

TABLE I. PROPERTIES INSPECTION RESULTS

		Yield Strength	Tensile Strength	Percent Elongation
Vendor Inspection	Typical	896 MN/m^2 (130 000 psi)	979 MN/m^2 (142 200 psi)	15.0
	Low	849 MN/m^2 (123 100 psi)	907 MN/m^2 (131 500 psi)	13.5
	High	947 MN/m^2 (137 400 psi)	1065 MN/m^2 (154 400 psi)	12.5
Boeing Inspection	Typical	924 MN/m^2 (134 000 psi)	996 MN/m^2 (144 400 psi)	16
	Low	896 MN/m^2 (130 000 psi)	965 MN/m^2 (140 000 psi)	8
	High	980 MN/m^2 (142 100 psi)	1070 MN/m^2 (155 200 psi)	13
Post-Forming Inspection	Typical	924 MN/m^2 (134 000 psi)	986 MN/m^2 (143 000 psi)	16
	Low	924 MN/m^2 (133 700 psi)	969 MN/m^2 (140 600 psi)	15
	High	930 MN/m^2 (134 900 psi)	1002 MN/m^2 (145 600 psi)	11

The Boeing-specified properties (D6-6051-1) were 931 MN/m^2 (135 000 psi) yield strength, 999 MN/m^2 (145 000 psi) ultimate strength, and 10 percent elongation. This would indicate that the 8-1-1 titanium plate received was from 10- to 20-percent low on strength properties and the elongation was satisfactory. It also shows that elevated temperature forming had no discernible effect on the properties of the material.

The original plan was to purchase 1.27-cm (0.50-in.) thick material to a tolerance of 10 percent and machine only one side, thus saving the cost of the extra thickness of titanium and the cost of machining the outside surface of the material. This plan was followed even though the material received was as much as 0.51 cm (0.20 in.) over thickness tolerance, out of flatness as much as 0.635 cm (0.250 in.) in a 0.457-m (18-in.) span, and unsatisfactory in surface finish due to surface pits and gouges. These discrepancies had no measurable effect on the success or failure of the diffusion bonding and hot vacuum forming process development. However, the discrepancy did have a significant effect on the cost of the program and the surface finish and thickness quality of the titanium gore delivered to MSFC.

MSFC Metallurgical Investigation of Gore Shipped to MSFC. The microstructure of the gore was found to be essentially a course, large grained, transformed beta structure rather than the desired equiaxed alpha-beta structure. This should have been expected because approval was given to the mill to roll the "as received" plate above the beta transus temperature realizing deformation from the rolling operation would effect grain reduction. Numerous small cracks or fissures were reported on the gore structure; therefore, the material was not acceptable for welding purposes.

The reason for this terse statement can best be appreciated by the fact that for quality acceptable welding of Ti-8Al-1Mo-1V, a fine grained equiaxed alpha-beta structure is desired. Quality acceptable welding can be accomplished on duplex annealed material from the mill but to-date no practical manufacturing technology has been developed to transform the aforementioned ruined gore material to the material acceptable for welding purposes. Indeed, one of the recommendations cited in the Boeing final report [3] is worth citing to emphasize this key point. The success of a production program involving titanium gores will depend upon consistent material properties and a reliable manufacturing process. Material properties data is lacking, especially as related to material performance after it has been processed through numerous thermal cycles. It is, therefore, recommended that a wide spectrum of materials properties test data be obtained before a decision is made to use titanium as a gore material in production.

EVALUATION

The potential advantage of using the developed diffusion bonding technology for future fabrication of large titanium structural parts is the manufacture of designs with greater efficiency weightwise and better performance characteristics. Figure 11 depicts a 12.9-m (40-ft) roll diffusion bonded titanium 8-1-1 Y-ring partial encasement and Figure 12 shows roll diffusion bonded titanium 8-1-1 skin and Y-ring segments. Further consideration of using diffusion bonded components for a welded structure such as a simulated titanium S-IC fuel tank would be premature without further research and development. In this connection, it is worthwhile to mention that the Department of Defense is sponsoring several projects to advance the state-of-the-art of roll diffusion bonding employing other titanium structural materials, notably the Ti-6Al-4V alloy.

FIGURE 11. ROLLED TITANIUM 8-1-1 Y-RING PARTIAL ENCASEMENT

This section will evaluate certain aspects of the diffusion bonded parts not previously covered and tie together salient information.

Although the cause of failure of the titanium 8Al-1Mo-1V cross beam[1] cannot be accurately defined, it appears that a stress-relief process on the structure after welding would have been helpful. Titanium Metals Corporation of America recommends[2] using temperatures in excess of 977° K (1300° F) for Ti-8-1-1 to avoid embrittlement of the welds and, in general, for Ti-8-1-1 material which contains residual stresses resulting from cold forming, warm forming, or welding.

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[1] Nichols, R. L.: Titanium 8Al-1Mo-1V Crossbeam Repair, Internal Note R-ME-IN-67-3, Manufacturing Engineering Laboratory, NASA — Marshall Space Flight Center, Huntsville, Alabama, February 1967.

[2] Letter from Russell, Harry A., Titanium Metals Corporation of America, dated May 31, 1967, to Charles Irvine, R-ME-MMP, concerning stress-relief of Ti-8Al-1Mo-1V weldments in the thicknesses contemplated for the first stage booster fuel tank (S-IC).

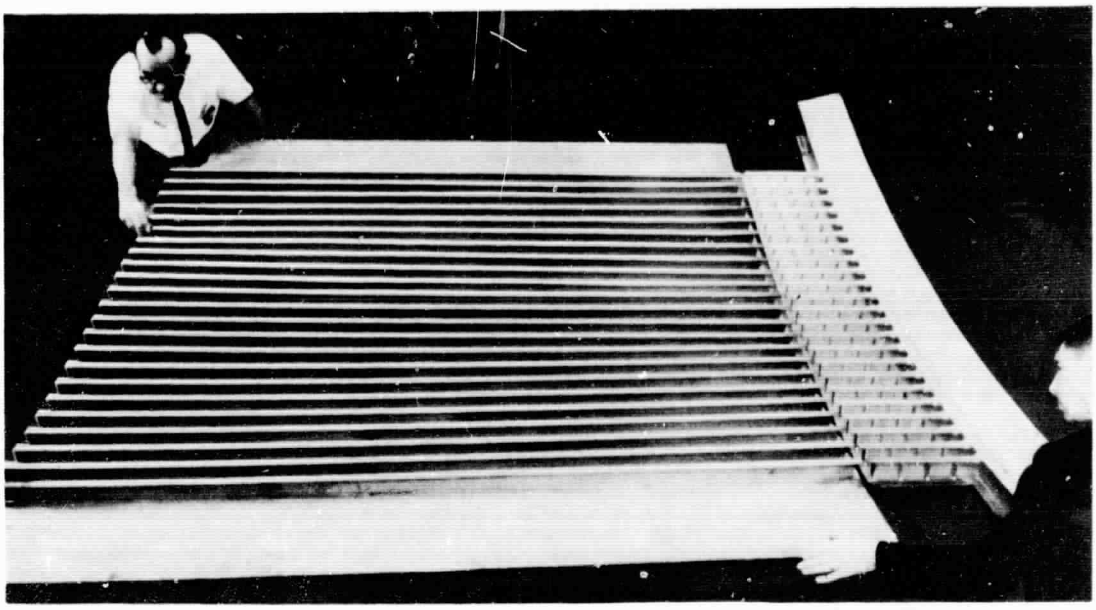

FIGURE 12. ROLL DIFFUSION BONDED SKIN AND Y-RING SEGMENTS

The Boeing Company developed Ti-8-1-1 welding techniques for subscale base gore materials prior to being hot formed but ran into difficulty when a weld repair was attempted on a diffusion bonded port embossment. Figures 13, 14, and 15 depict a metallurgical evaluation of this examination. This proves, as does the experience of North American Rockwell Corporation, that it is difficult, if not impossible practically speaking, to reliably weld repair diffusion bonded Ti-8Al-1Mo-1V parts in the area of the bonds.

In any case, additional research and development is required in this area whether it is the Ti-8-1-1 or other diffusion bonded titanium alloys. The effect thermal processing has on the weldability of the diffusion bonded joints should be investigated.

In general, it can be stated that there is no great difficulty in welding Ti-8Al-1-1 diffusion bonded parts in areas other than the bonded joints if the material is a fine grained, equiaxed structure. However, material can be ruined for welding when the beta transus is exceeded.

And the fact remains, no acceptable techniques have been developed to acceptably weld diffusion bonded Ti-8Al-1Mo-1V joints in the area of the bonds.

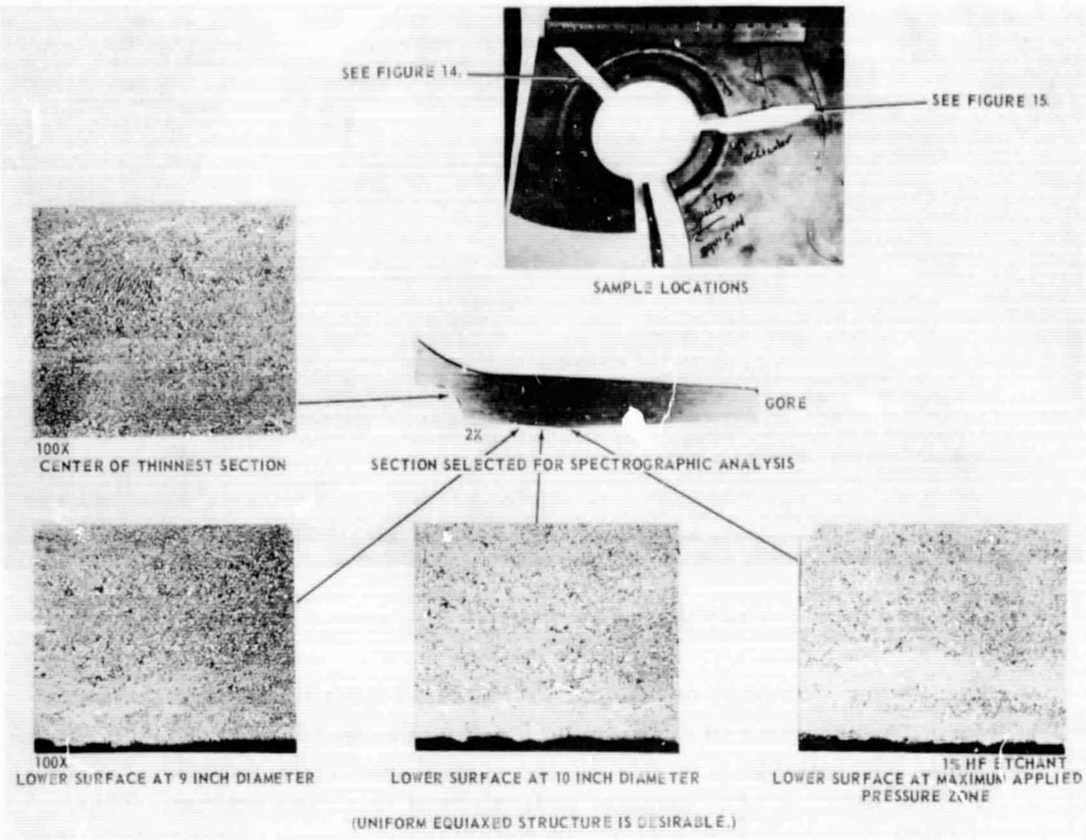

FIGURE 13. SAMPLE LOCATION AND MICROSTRUCTURE OF DIFFUSION BOND

It is of interest to note that the recently developed plasma arc welding process has been adapted for making high quality welds in titanium alloys. Preliminary manufacturing process data[1] has been documented for plasma arc welding the gore to Y-ring to skin for simulated segments of the S-IC fuel tanks.

Normally, fine cracks appear to be a result of surface contamination caused by the hot roll bonding and hot contour forming operations. The cracks or fissures would pose a serious deficiency unless removed; consequently, it was normal practice to remove from 7 to 8 mils on the titanium Y-ring by a chem-milling operation such as that illustrated by Figures 16 and 17 which show "before" and "after" chem-milling to a quality acceptable finish.

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[1] Sipe, R. C., MEL Technical Report WD-64-67, Development of Plasma Arc Welding Procedures for Manufacturing Large Titanium Structures, March 24, 1967.

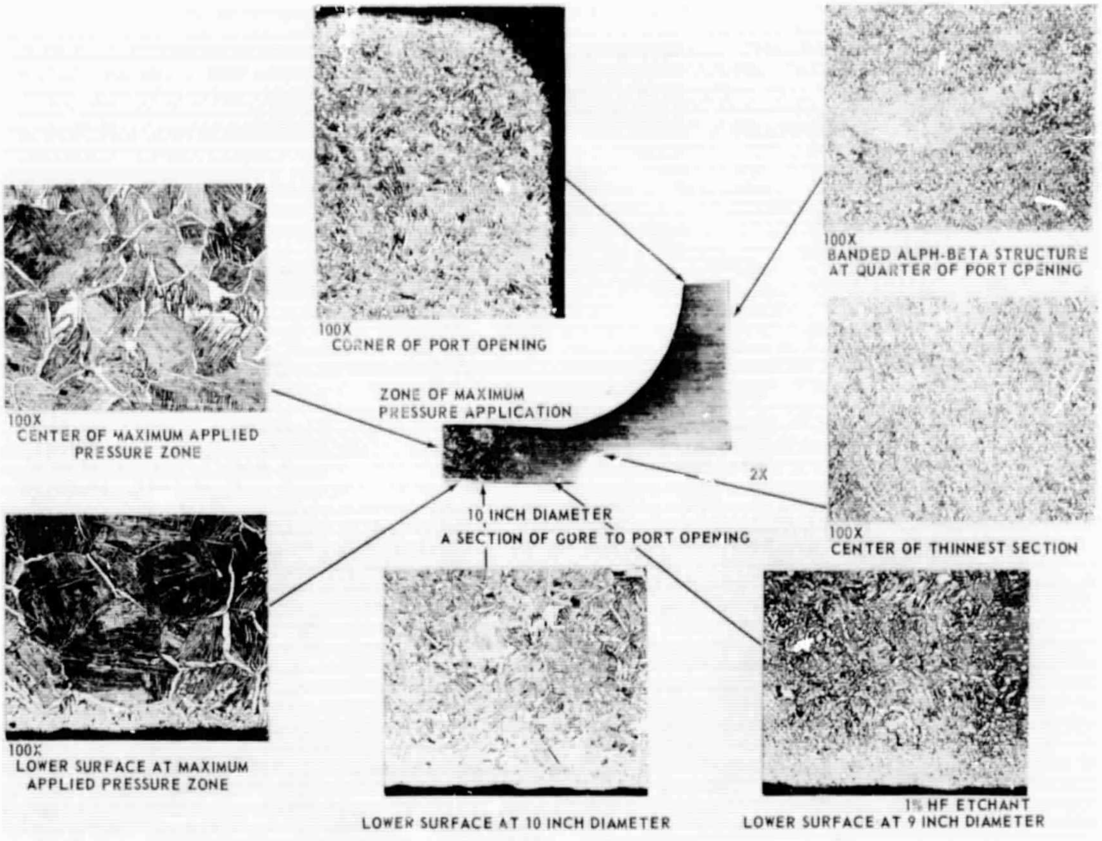

FIGURE 14. HETEROGENEOUS STRUCTURE OF PART OPENING FUSION BONDED TO GORE SEGMENT

However, the fact remains that fine cracks or fissures sometimes occur without logical reason such as the delayed cracking of the titanium 8Al-1Mo-1V S-IC skin section when given additional thermal treatments. Delayed cracking has been attributed to stress corrosion cracking and may be the logical explanation but sufficient evidence has not been established to verify these findings. It has been determined that a Widmanstatten structure has shown a tendency toward surface embrittlement after exposure to temperatures above 727° K (850° F), with or without stress.

Metallographic specimens of roll-diffusion bonded Ti-8Al-1Mo-1V parts were examined microscopically by Battelle Memorial Institute. In their letter[1], Battelle stated they have no knowledge of porosity problems in titanium alloys

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[1] Letter from Buchheit, R. D., Battelle Memorial Institute, dated November 8, 1967 to Earl Hasemeyer, R-ME-MW, concerning examination of roll diffusion bonded Ti-8Al-1Mo-1V parts.

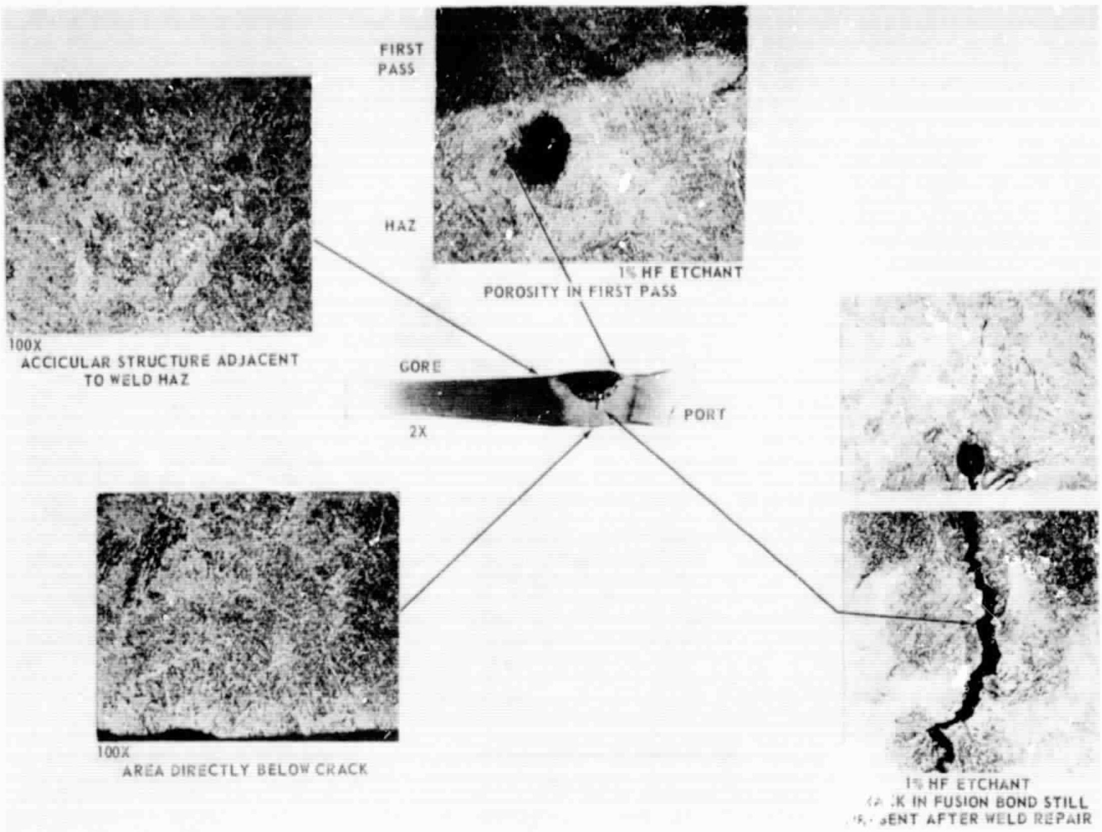
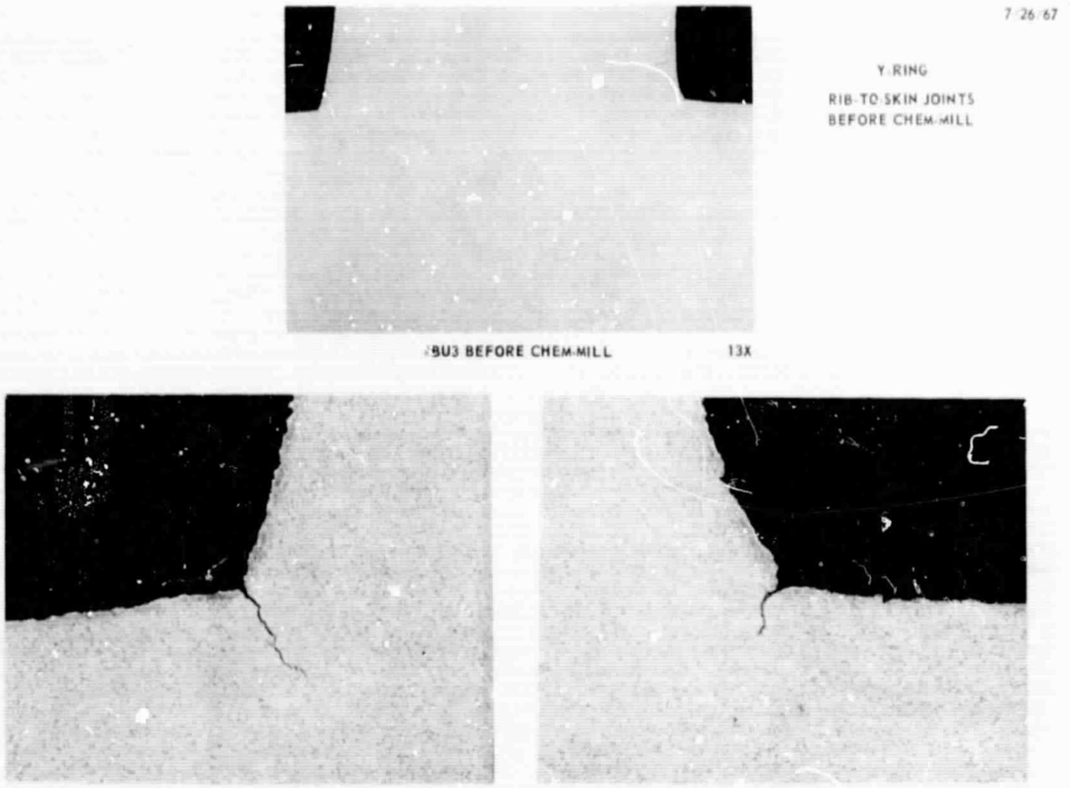


FIGURE 15. UNSUCCESSFUL REPAIR WELD TO A CRACK IN FUSION BOND OF GORE SEGMENT TO PORT OPENING

arising from roll diffusion bonding and have no explanation at this time for the presence of porosity in the MSFC Ti-8Al-1Mo-1V parts.

It has already been recommended that a temperature above 947° K (1300° F) be used to stress relieve the parts to preclude embrittlement of the welds. If the data generated by North American Rockwell Corporation is accepted at face value, it appears that a temperature of 1060° K (1450° F) followed by cool down to 755° K (900° F) within one hour is optimum and that properly duplex annealed Ti-8Al-1Mo-1V parts can even be welded without a subsequent annealing or stress relieval operation. It is apparent that at temperatures above 810° K (1000° F), surface contamination effects become more severe the greater the temperature. Protective coatings or inert atmospheres usually must be employed followed by at least a pickling operation (30 percent HNO_3 +3 percent HF) when protective coatings are used. With alloys such as



7/26/67

Y-RING
RIB-TO-SKIN JOINTS
BEFORE CHEM-MILL

BU3 BEFORE CHEM-MILL 13X

BU3 BEFORE CHEM-MILL 150X

BU3 BEFORE CHEM-MILL 150X

1 OF 2

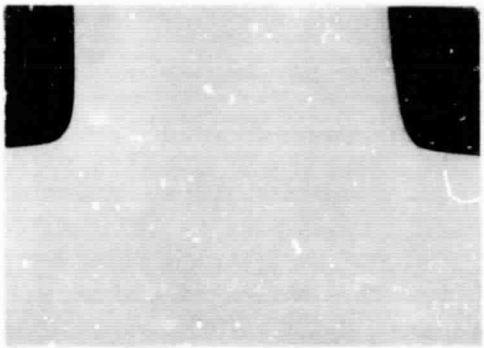
FIGURE 16. Y-RING RIB-TO-SKIN ROLL DIFFUSION JOINTS BEFORE CHEM-MILLING

Ti-6Al-4V, contamination or scale effects are not as severe because Ti-6Al-4V in the solution heat treated condition can be aged at 810° K (1000° F) for four hours. This offers the potential of employing age-forming for Ti-6Al-4V and stress relieval temperatures at least 422° K to 506° K (300° F to 450° F) lower than for Ti-8Al-1Mo-1V. Furthermore, the Ti-6Al-4V alloy is relatively immune to hydrogen embrittlement when pickled in 30 percent HNO_3 -3 percent HF. Even when exposed to times as long as 30 minutes, hydrogen concentration was less than 200 parts per million based on R-P&VE-MMC's analysis of intercostal parts used for a titanium box beam.

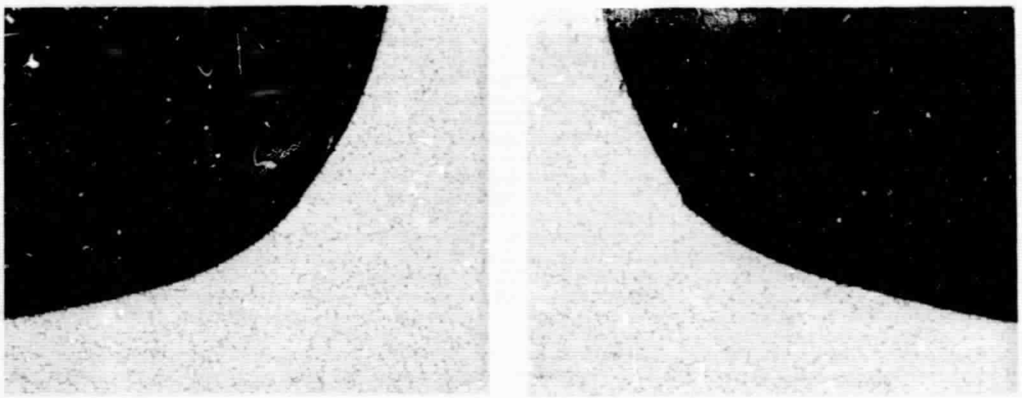
In the fabrication of roll diffusion bonded skin panels, the titanium alloy 6Al-4V wire was used as a filler material to impart integral fillet radii. While mismatch of the wire was observed on some of the skin panel tees, careful manufacturing practice can probably correct this deficiency. Every one of these

7/26/67

Y-RING
RIB-TO-SKIN JOINTS
AFTER CHEM-MILL



#BU3 AFTER .008" CHEM-MILL 13X



#BU3 AFTER .008" CHEM-MILL 150X

#BU3 AFTER .008" CHEM-MILL 150X

2 OF 2

FIGURE 17. Y-RING RIB-TO-SKIN ROLL DIFFUSION
JOINTS AFTER CHEM-MILLING

fillets showed a course, basket-like weave, or Widmanstatten structure. This structure is typically characteristic of working or heating above the beta transus and would be expected for the Ti-6Al-4V wire because the beta transus was definitely exceeded in all cases. The question, not answered here, is: due to the considerable difference in microstructure at the fillet area, is the fatigue life acceptable?

When Ti-8Al-1Mo-1V material is properly diffusion bonded, mechanical properties are equal to or greater than the parent material properties. There is, however, a great deal more research and development required on Ti-8Al-1Mo-1V structures related to such items as metallurgical instability, stress corrosion and surface cracking related to interstitial diffusion. It is known that air is catastrophic during roll or static diffusion bonding. This happened in the rolling of one of the large "J Pack Material" skin panel packs. The diffusion bonded areas, where air entered the ruptured pack, could be kicked apart. In assessing the interstitial limits North American Rockwell Corporation set up for Ti-8Al-1Mo-1V, the present allowable magnitudes may be set too high. Fissures appearing on the surface are a type of instability definitely known for the titanium alloy 6Al-4V. For example, hydrogen embrittlement can be expected when the parts per million exceeds 200. The titanium alloy 8Al-1Mo -1V is an area still being studied by the titanium producers.

In hot forming of the full scale base gores, a buckled area appeared in the same general area of the part. This is not considered a major problem since with redesign of the close ribbed configuration, this could be eliminated.

In the roll diffusion bonding of skin and Y-ring segment material, dimensional checks were surprisingly close to the design dimensions. It can be stated with confidence that roll diffusion bonding is a very useful production tool for the Y-ring segments. However, in the production of the skin segments, a regular machining process may be less expensive in the long run because tolerances are associated with machining of the steel filler bars, contour of the rolls, etc.

CONCLUSIONS AND RECOMMENDATIONS

The objective to establish design and manufacturing process parameters for simulated S-IC titanium fuel tank components was accomplished primarily through diffusion bonding technology. Considering the large structural sizes of these parts, this accomplishment was indeed commendable. The major conclusion deduced is that the aforementioned diffusion bonding technology established the capability to produce large structural parts from titanium alloy materials.

Relative to availability of materials, the titanium producers should be cognizant of NASA's potential future requirements. With the titanium market expected to expand, further advances in the state-of-the-art in rolling larger sizes of sheets and plates are a foregone conclusion. However, as was witnessed in the rolling of the largest size Ti-8-1-1 plate for the base gore part of a titanium fuel tank, the results were marginal. Fortunately, this situation is expected to be improved through the selection of another alloy. Reportedly, this is the case as Ti-6-4 alloy is for all practical purposes that specified for supersonic titanium structures whereas two years ago, Ti-8-1-1 was one of the prime titanium material candidates.

It is recommended that a welding investigation be conducted on the diffusion bonded joints to determine: (1) the feasibility of welding such joints and (2) the practicality of welding "T" stiffeners on the Y-ring and skin segment. Existing manufacturing plans call for the "T" stiffeners on the Y-ring and skin segment to be welded together. Because there is already evidence that repair welding of Ti-8-1-1 material in the area of the diffusion bonds is not feasible, it might not be feasible to weld such material. Consequently, more research and development is required in the area of diffusion bonded titanium alloys since existing optimum designs are based on the premise that the above is feasible. Also, the practicality of welding "T" stiffeners on Y-ring and skin segments has not been proven. From a manufacturing veiwpoint, it was difficult enough to develop reliable straight fusion weldment techniques of the current aluminum skin sections to the Y-ring and the gore segments to the Y-ring along the convex surface of these components.

Future titanium work should be concentrated on those alloys which show none of the ordering embrittlement reaction characteristics that Ti-8-1-1 possesses. It is fairly unrealistic to stress relieve large welded structures such as a simulated titanium S-IC fuel tank by thermal methods. Accordingly, the investigation of non-thermal techniques such as the use of low frequency vibrations for stress relief should be continued. The work being conducted by MSFC's Propulsion and Vehicle Engineering Laboratory to measure non-destructively residual stresses is an important adjunction to a titanium manufacturing program in the future.

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APPROVAL

NASA TM X-53747

DIFFUSION BONDING OF TITANIUM TO SIMULATE S-IC FUEL TANKAGE COMPONENTS

By C. N. Irvine

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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