

Eutectic Bonding of Boron-Aluminum Structural Components

PART II* — DEVELOPMENT AND APPLICATION OF THE PROCESS

Complex B/Al structures have been produced with strengths exceeding design ultimate loads at 600 F

BY J. T. NIEMANN AND R. A. GARRETT

ABSTRACT. This study was undertaken as part of a major program to develop a low-pressure technique for fabricating boron-aluminum structural components and in conjunction with a NASA program to evaluate boron-aluminum components for space shuttle applications. The evaluation of critical processing parameters was reported previously*¹; this Part II describes the development of production processes and their application to component fabrication.

Development of production processes was concerned first with the chemical cleaning of monolayer foil. Candidate cleaning processes were evaluated quantitatively using ellipsometry to measure residual aluminum oxide layer thickness; a technique was selected which provided good cleaning with reprocessing flexibility on the basis of metal removal rates. The effects of monolayer storage conditions — both the uncoated and copper-coated monolayer

— on the rate and buildup of oxide layers were measured by ellipsometry to define specification times and required environments. A nitrogen environment was satisfactory with permissible storage times of 80 h for the cleaned monolayer prior to vapor deposition and 800 h after copper coating. The techniques necessary to produce an adherent physical vapor deposition (PVD) copper layer on the monolayer were developed and confirmed. Ability to apply a 20 microin. coating has been demonstrated in a significant number of tests. Further, parameters for glow discharge cleaning prior to PVD were evaluated through ellipsometer studies. Finally, complex boron-aluminum structures have been fabricated with structural shapes utilizing a mechanical forming technique to simultaneously pressure-form all monolayers prior to bonding. These structures have demonstrated strengths in excess of design ultimate loads at 600 F. An integrally stiffened boron-aluminum panel has been fabricated, utilizing a simple, re-usable tooling concept which can be cost competitive with titanium machinings.

Introduction

Eutectic bonding is a diffusion brazing process developed for fabricating boron-aluminum components from composite monolayer.

This process relies on the diffusion of a thin surface film of copper into the aluminum matrix to form a liquid phase when heated above the copper-aluminum eutectic temperature of 1018 F. The first phase in the development of this process, previously reported, was concerned with defining the critical processing parameters of bonding temperature, time and copper thickness and establishing that the process could be applied to boron-aluminum fabrication. Then the processes associated with eutectic bonding were optimized and scaled-up for the production of large structural components; the development and application of these processes and component manufacture are described in this paper.

Process Development

The evolution of boron-aluminum from its receipt as monolayer foil into a laminated structural component involves several basic processing steps. After monolayers are inspected and accepted, they are chemically cleaned to remove any surface contamination. Then each foil is coated on both sides with a 20 microin. layer of copper by physical vapor deposition and laid-up to the desired shape on a combination forming/bonding tool. The part is then covered with an outer sheet which is welded directly to the tool to form an envelope which

J. T. NIEMANN is Senior Group Engineer, Materials and Processes Dept., and R. A. GARRETT is Program Manager, Composite Programs, McDonnell Douglas Astronautics Co. — East St. Louis, Mo. Paper was presented at 54th AWS Annual Meeting held in Chicago during April 2-6, 1973.

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can be evacuated. Bonding is accomplished in an autoclave with the pack evacuated and under an external pressure of about 250 psi. The processing steps to be described herein are chemical cleaning, copper coating, lay-up procedures and bonding.

Optimization Of Chemical Cleaning

Monolayer foils must be chemically cleaned to remove surface films which would prevent adherence of copper during vapor deposition or impede diffusion during the bonding process. Also, excessively thick surface films present at the time of copper coating might be trapped in the joint area and lower joint strength. The approach to selecting a cleaning process consisted of rating several standard cleaning solutions according to their ability to remove surface films and the rate at which they dissolved aluminum. An "optimum" method was selected on the basis of these ratings. Then, the rate of film build-up after cleaning was related to environment to establish storage conditions and time limits for monolayer foils after cleaning and before coating.

Selection of a Test Method. Several standard cleaning procedures were used during the initial development of eutectic bonding to prepare boron-aluminum monolayer foils for coating. Also, efforts were made to compare them and select an optimum process. However, the principal test method initially employed was peel testing of bonded samples. This approach was considered to be of limited value because the cleaning methods could not be rated quantitatively. Also, the bonding process itself may have introduced other variables,

unrelated to cleaning, and influenced peel strength. A better method of comparing the effectiveness of various cleaning methods was needed to optimize the processing of boron-aluminum foils. The method selected was to measure the residual film thickness, assumed to be an oxide, immediately after cleaning. Ellipsometry was selected as the technique for measuring these thin films and 1100-O aluminum foil was used for test specimens.

An ellipsometer is an optical instrument which has sufficient sensitivity to detect a single layer of adsorbed oxygen or oxide molecules on a metallic surface. Ellipsometry basically measures changes in the polarization of monochromatic light upon reflection at an optical boundary. The magnitude of the change can be used to calculate the thickness of surface films provided the composition and optical properties of the film and substrate are known. A photograph of the ellipsometer used is shown in Fig. 1. Its high degree of sensitivity made it an ideal instrument for comparing solutions by measuring residual film thickness.

Comparison of Cleaning Processes. A standard method of cleaning aluminum alloys for removal of surface oxides consists of solvent or vapor degreasing followed by alkaline cleaning to remove soil and finally acid pickling or deoxidizing to remove smut and oxide films. A number of commercial cleaners and deoxidizers generally are approved for aerospace application and are listed as options in applicable process specifications. Several of these materials were compared to determine if any particular combination of alkaline cleaner, deoxidizer and immersion time was superior from the standpoint of residual

film thickness. Three alkaline cleaners plus four deoxidizers were evaluated, and the immersion times were varied to provide 108 combinations of cleaning procedures. Triplicate specimens of 1100-O aluminum, 0.006 in. thick, were tested under each condition. The cleaning solutions and test conditions are listed below:

Solution	Time, min
Chromated,	
nonsilicated cleaner	5, 17, 30
Silicated cleaner	5, 17, 30
Nonsilicated cleaner	5, 17, 30
Type A,	
commercial deox.	3, 6, 10
4 Acid Type deox.	3, 6, 10
Type B,	
commercial deox.	3, 6, 10
HNO ₃ -HF-CrO ₃ deox.	1, 8, 15

Test samples were rinsed thoroughly after both alkaline cleaning and deoxidizing treatments and, after a final rinse in hot, deionized water, they were dried with gaseous nitrogen. Ellipsometer readings were made at several stages during the processing. Selected samples were checked for film thickness in the as-received condition, after vapor degreasing and after alkaline cleaning. All 324 specimens were measured after the final deoxidizing treatment. These measurements assumed that the film consisted entirely of aluminum oxide. However, atmospheric exposure probably resulted in the formation of a film of moisture less than 10 angstroms over the oxide so that the measurements did not represent the absolute thickness of the oxide film. This factor was not considered a drawback in measuring the efficiency of cleaning techniques; the error introduced by ignoring moisture was slight, and since all the test specimens were subjected to atmospheric exposure they all contained some adsorbed moisture and a similar degree of error.

In the as received condition, an average film thickness of 133 angstroms was measured which was reduced to 111 angstroms after vapor degreasing. After alkaline cleaning, measurements indicated that the film thickness generally increased. Readings in these three conditions do not represent true oxide thickness because of the presence of other substances such as oil films on the surface. Reduction on film thickness after vapor degreasing indicates that some of this initial film had been removed. The increase in film thickness after alkaline cleaning does not necessarily indicate that the oxide film thickness had increased but possibly indicates the buildup of reaction products which developed when the samples were immersed in the aggressive alkaline solutions.

The ellipsometry data showed no general trend for increased cleaning

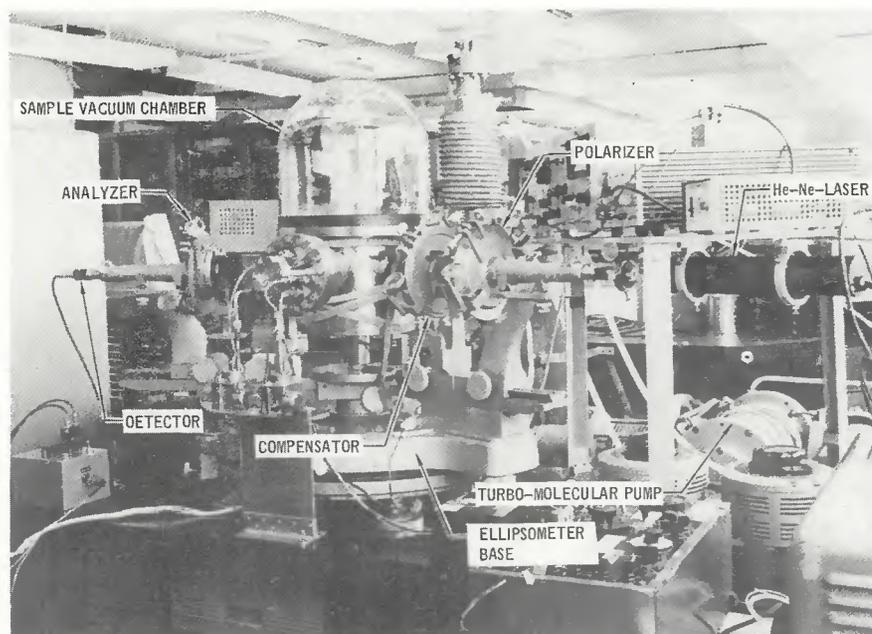


Fig. 1 — Ellipsometer and associated equipment

efficiency with increased immersion times. However, there were differences in residual film thickness attributable to variations in alkaline cleaning and deoxidizing solutions. Generally, the most efficient alkaline cleaner, regardless of the deoxidizer used, was the nonsilicated solution. Also, the most efficient deoxidizer was the $\text{HNO}_3\text{-HF-CrO}_3$ acid pickle. These general trends are shown in Fig. 2 which compares the optimum results obtained with each cleaner/-deoxidizer combination. On an overall basis, the systems can be grouped according to residual film thickness as follows:

1. *Residual Film Thickness 30 angstroms* — Nonsilicated cleaner + $\text{HNO}_3\text{-HF-CrO}_3$ acid pickle
2. *Residual Film Thickness 40 angstroms* — Nonsilicated cleaner + commercial deoxidizer A or B
3. *Residual Film Thickness 50 angstroms* — (a) Silicated cleaner + $\text{HNO}_3\text{-HF-CrO}_3$ acid pickle; (b) Silicated cleaner + commercial deoxidizer A; (c) Chromated, nonsilicated cleaner - $\text{HNO}_3\text{-HF-CrO}_3$ acid pickle

Rate of Metal Removal During Cleaning. Although the ellipsometer showed which of the cleaning methods was most efficient from the standpoint of oxide removal, the rate of metal removal also had to be considered because the layer of matrix material covering the filaments in boron-aluminum monolayer foil is very thin. The thickness of composite monolayer with 0.0056 in. diam boron is about 0.0075 in., so that the filaments are covered by about only 0.001 in. per side of aluminum. Even a small loss of surface metal could amount to an appreciable percentage of this layer. This potential problem assumes a greater proportion if re-

cleaning should be required for some reason, because significant reduction of this thin layer could reduce transverse tension properties appreciably. Therefore, it was decided that the loss of material due to cleaning should not exceed 10% of the matrix thickness over the filaments per side.

The comparison of cleaning procedures indicated significant differences existed in the aggressiveness of various alkaline cleaner-deoxidizer combinations. Therefore, a check was made of three of these combinations to determine the relative amounts of material removed and the influence of immersion times. Selection of the three systems was made to represent three degrees of severity. The conditions studied in increasing order of severity of the cleaner-deoxidizer combination were:

1. Chromated nonsilicated cleaner/10 min + commercial deoxidizer A for 3, 7 and 10 min
2. Chromated nonsilicated cleaner/10 min + $\text{HNO}_3\text{-HF-CrO}_3$ Acid Pickle for 3, 7, and 10 min
3. Nonsilicated cleaner/30 min + $\text{HNO}_3\text{-HF-CrO}_3$ Acid Pickle for 3, 7 and 10 min

The latter system had produced the thinnest residual surface film (less than 30 angstroms) in the ellipsometric evaluation of cleaning processes.

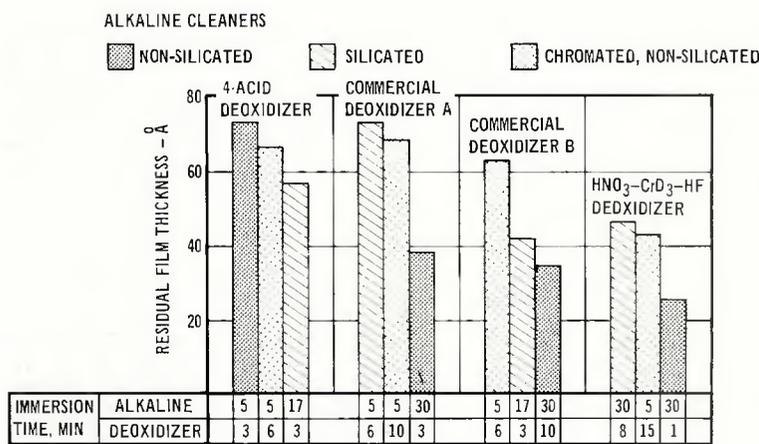
Samples of 1100-0 aluminum alloy, approximately 25.4 mm x 76.2 mm x 0.152 mm (1 x 3 x 0.006 in.) were weighed and then subjected to the candidate cleaning methods and reweighed. The weight difference was used to calculate the metal thickness removed per side. The results of the tests are shown graphically in Fig. 3. In general, the mildest cleaning method removed about 6 to 12 microin. as the deoxidizing time was increased from 3 to 10 min. A removal of 12 microin. is equivalent to a loss

of about 1% of the matrix material covering the boron filaments; therefore, monolayer foils could be re-cleaned many times in this cleaner-deoxidizer combination without removing more than 10% of the matrix thickness over the center of the filament.

On the other hand, a single cleaning in the most efficient combination (nonsilicated cleaner/30 min. + $\text{HNO}_3\text{-HF-CrO}_3$ acid pickle) for as short a time as 3 min in the deoxidizer would result in the loss of about 12% of the matrix material covering the filaments. Cleaning in this combination would have to be limited to a single cycle to avoid an excessive loss, and this limitation was considered too restrictive.

The intermediate combination of chromated nonsilicated cleaner + $\text{HNO}_3\text{-HF-CrO}_3$ deoxidizer resulted in metal removal ranging from 30 to 90 microin. as the deoxidizing time was increased from 3 to 10 min. In this case, a 3 min immersion would result in the loss of about 2.5% of the aluminum covering the filaments.

In summary, this series of tests indicated that the cleaner-deoxidizer which produced the cleanest surface would also dissolve more than 10% per side of the aluminum matrix. Therefore, using this combination for chemical cleaning of monolayer foils would prohibit recleaning, if needed. At the other extreme, the mildest combination was relatively inefficient for oxide removal, showing a residual film thickness of about 60 angstroms. The intermediate combination of chromated nonsilicated cleaner + $\text{HNO}_3\text{-HF-CrO}_3$ acid pickle for 3 min offered a good compromise. The metal removal rate (~25 microin.) was sufficiently low to permit recleaning. Also, this combination is considerably more efficient than the milder combination evaluated. Ellipsometer readings made during this series showed residual films after cleaning



NOTE: CONDITIONS SHOWN REPRESENT "OPTIMUM" RESULTS FROM 108 COMBINATIONS OF CLEANER, DEOXIDIZER AND TIME (324 SPECIMENS)

Fig. 2 — Effect of chemical treatment on 1100 aluminum residual film thickness

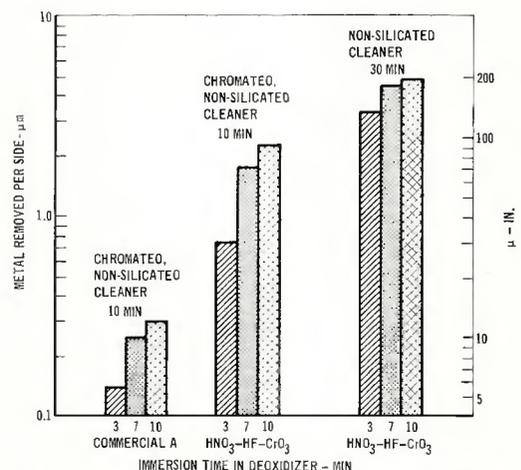


Fig. 3 — Effect of cleaning conditions on dissolution of 1100-0 aluminum foil

in the intermediate system to be about 37 angstroms, or 40% less than measured on specimens subjected to milder environment. Therefore, the chromated nonsilicated cleaner plus $\text{HNO}_3\text{-HF-CrO}_3$ acid pickle was selected as the method of cleaning boron-aluminum monolayer foils prior to copper coating.

Finally, a limited test series was made to determine if the boron filaments would be attacked in the cleaning solutions. This was accomplished by immersing duplicate samples of boron filament in commercial A deoxidizer for periods up to 20 min and in the $\text{HNO}_3\text{-HF-CrO}_3$ deoxidizer for intervals up to 30 min. There was no change in weight of the boron filaments as a result of these exposure conditions.

Effect of Storage Conditions. One of the tasks in the cleaning optimization study was determining the rate at which an oxide film forms on a freshly cleaned aluminum surface. This investigation was undertaken to establish if a time limit should be imposed on the interval between cleaning and coating and if storage in an inert environment would retard the rate of film buildup. In this evaluation, strips of 1100-0 aluminum foil were chemically cleaned in nonsilicated cleaner and $\text{HNO}_3\text{-HF-CrO}_3$ deoxidizer and then stored in small controlled atmosphere chambers and exposed to a continual uniform flow of either air, nitrogen or argon. Ellipsometer readings of oxide thickness were taken on each sample immediately after cleaning and at intervals during the test period.

Ellipsometer readings taken immediately after cleaning showed the oxide film on the test sample to be about 22 angstroms thick. At the end of 1 h exposure to the test environments, the film thickness had increased significantly. The oxide continued to increase throughout the test period of 960 h, but at a slower rate. After 960 h, the oxide film thickness had increased about 80% overall but the increase amounted to 40% during

the first hour. Also, the rate of increase was not influenced appreciably by environment. These relationships are shown in Fig. 4.

The test results showed that unless coating was begun almost immediately after the cleaning operation, an increase in oxide film thickness on monolayer foils could not be avoided even if stored in an inert gas environment. This was not considered practical for a production operation. Therefore, it was necessary to set a limit on the storage time between cleaning and coating based on the test data and practical considerations. A limit of 80 h was established to be consistent with a normal production flow cycle. Also, this extended interval would result in very little increase in oxide thickness over a one hour exposure, as shown in Fig. 4.

Vapor Deposition Studies

During the initial development of the eutectic bonding process, both electroplating and physical vapor deposition (PVD) were considered as potential methods of depositing copper on aluminum. Tests were made to compare both methods, and vapor deposition gave more consistent results. This was attributed to the better control of coating thickness available with PVD and the fact that, unlike electroplating, this process did not require an intermediate metallic layer. To ensure that a high purity copper deposit would be obtained, copper wire meeting the requirements of ASTM B 170, Class I (99.99% Cu) was used.

Initially, copper coating for eutectic bonding was accomplished in an 18 in. diam, diffusion-pumped bell jar vacuum system. Because of the limited size of this laboratory facility, a scale-up of the basic concept was needed before large size components could be manufactured. This was accomplished by modifying an electron beam welding chamber, but before undertaking a major manufacturing

program, studies were made to ensure that high quality coatings of uniform thickness could be produced.

Glow Discharge Cleaning. One area of investigation was concerned with the parameters of the glow discharge process (ion bombardment) which cleans the monolayer foil just prior to vapor deposition. The purpose of the evaluation was to determine the effect of current density and time on oxide removal. This was accomplished in the ellipsometer (Fig. 1) which was modified to accommodate an atmosphere chamber in which a sample could be glow-discharge cleaned and the surface film measured without exposure to air. Test samples were 0.006 in. thick by 1.12 in. diam 1100 aluminum alloy discs.

The test procedure consisted of evacuating the sample chamber and imposing a high voltage between the chamber wall and the sample, with the sample as the cathode. A glow discharge was then initiated by back-filling the chamber with argon until the desired argon ion current was achieved. Ellipsometer measurements were made prior to and during the discharge to determine the thickness of the oxide film. The discharge was continued until the measurements indicated that a minimum of film thickness had been achieved. All the tests were made at 3700 volts potential difference but the current density was varied for each run.

Test results showed that the oxide was removed rapidly during the early stage of glow discharge but the removal rate decreased with time and eventually approached zero. Increasing the current density reduced the time required to reach the final film thickness, but did not change this minimum value appreciably. The removal rate, defined as the ratio of total film removed to the time required to reach the minimum value, was essentially a linear function of ion current as shown in Fig. 5.

Since oxide free surfaces can be achieved by glow discharge just prior to coating, it is theoretically possible to eliminate all the chemical cleaning operations with the exception of vapor degreasing. However, the rate of oxide removal from aluminum was slow even at the highest current densities evaluated. Since the oxide film on the starting material would exceed 100 angstroms, which was demonstrated in the cleaning optimization study, glow discharge times would exceed one hour even under high current densities to ensure an oxide free surface. This amount of time was considered excessive. Therefore, chemical cleaning was selected as the primary means to minimize the surface oxide on monolayers prior to copper coating. Glow discharge was relied upon primarily to remove surface layers of adsorbed gases and water

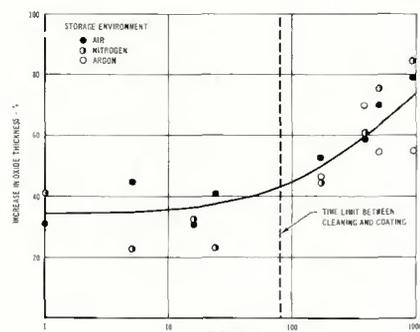


Fig. 4 — Effect of storage environment on buildup of oxide film on chemically cleaned aluminum

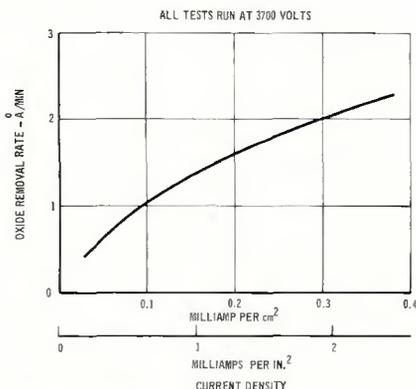


Fig. 5 — Effect of current density on rate of oxide removal during glow discharge cleaning

vapor and thereby ensure adherence of the vapor deposited layer.

The final experiment in the glow discharge study was designed to determine if an oxide free surface could be maintained under ideal conditions. This was carried out in an Auger electron spectrometer which is used to determine the chemical composition of surface deposits. The test procedure consisted of sputtering the oxide off a sample of aluminum, holding it under a partial pressure of argon (9×10^{-5} torr), and analyzing the surface periodically. The initial scan, taken 30 seconds after ion bombardment was completed, showed a single 67 eV peak characteristic of pure aluminum. As the holding time was increased, the amplitude of this peak decreased and this was accompanied by the appearance of a second peak at 57 eV. This second peak is associated with the formation of aluminum oxide and eventually became more prominent than the pure aluminum peak. The buildup was charted by the change in the aluminum peak which diminished as oxide was formed.

Results of the test, shown in Fig. 6 indicate that oxide begins to form on a clean aluminum surface almost immediately upon exposure to a high purity environment. These measurements indicate that even under ideal glow discharge conditions, an oxide film would form on the metal surface during the subsequent evacuation of the chamber to the coating pressure which required about 5 to 10 min.

Effect of Storage Environment. The possibility that freshly deposited copper would oxidize during the interval between coating and bonding was investigated using ellipsometry. Freshly coated samples of 1100-O aluminum foil were subjected to several different environments and the film thickness measured periodically. Initially, specimens were exposed for 168 h and no appreciable difference was noted in the rate of oxidation as a function of storage environment. In a second test series, samples were exposed for a total of 960 h in argon. Oxidation occurred throughout the test period, but the rate appeared to decrease with time. At the end of the 960 h exposure, the film thickness was less than 40 angstroms. This amount was not considered excessive. To ensure that oxidation would be kept within acceptable limits, the time between coating and bonding was limited to 700 h and nitrogen was specified for the storage environment.

Thickness Uniformity. During the initial development of the eutectic bonding process, the minimum copper thickness required for bonding had been defined and the physical vapor deposition process selected for production. About 12 microin. of

copper is needed to ensure that some of the coating will survive the loss due to diffusion during heating to the bonding temperature and be available to form an adequate amount of liquid phase for bonding. On the other hand, an excess amount could result in a brittle bond. A coating thickness of 20 microin. was established as a satisfactory level that would produce sufficient liquid phase without the danger of joint embrittlement. In scaling up the PVD process for production, it was necessary to refine the coating technique to ensure that desired coating thickness could be obtained consistently within narrow tolerance limits, and that the coating would be uniform over the entire surface of each monolayer foil.

Vapor deposition of copper on boron-aluminum monolayer foils for production is accomplished in the modified electron beam welding chamber shown in Fig. 7. A drum 24 in. in diameter by 90 in. long is used to hold the monolayer foils. The outer surface of the drum is electrically insulated so that the entire outer surface on which the monolayer foils are wrapped can be glow discharge cleaned. A single boat source, positioned 12 in. below the outer surface of the drum, is kept continuously filled with molten copper by filler wire additions. The molybdenum boat is surrounded by a metal shield and a cut-out area in this shield limits the deposition area. An ion rate monitor is used in the system to measure evaporation rate which is controlled by the rate at which wire is fed into the boat.

The mode of operation consists of moving the boat containing the copper from one end of the drum to the other as the drum rotates. Approximately 25 test runs were made to develop operating parameters of boat travel speed, drum rotational speed, power input to the boat and deposition rate. For these tests, 3 rows of 6 specimens each, located 120 deg apart, were coated under various conditions and the resulting coating thickness measured by weight change. The samples covered 18 in.

of drum length. These tests indicated that on any given run, coating uniformity within the 18 sample grouping did not vary more than about 3 microin. with an average uniformity variation of 2.1 microin.

The test runs demonstrated that the vapor deposition equipment was capable of depositing a uniform copper coating of closely controlled thickness. Therefore, a specification was prepared to control the process for production and establish coating thickness limits. In order to check coating thickness on each run, seven weight samples are included with each drum load. These samples are commercial aluminum foil samples, cut into 2×3 in., and randomly distributed on the coating drum. Difference in sample weight before and after coating, measured to the nearest microgram, is converted to coating thickness. Material coated for production eutectic bonding is required to meet the following conditions:

1. The average coating thickness based on all seven weight samples must fall between 17.5 and 22.5 microin.
2. The coating thickness of any one sample cannot exceed 26.5 microin.
3. The coating thickness of any one sample cannot be less than 13.5 microin.

Coating thickness requirements are met with little difficulty. This is shown in Fig. 8 which represents an analysis of coating thickness control sample results from the first 63 coating runs on monolayer foil made under specification control and which are typical of present production capability. The average of all weight specimens (441 total) was 19.57 microin. which is very close to the middle of the specified range. Sixty of the individual runs ($\sim 94\%$) met the requirements for average thickness and the remaining exceeded the established bounds by less than 2 microin. Of all the first sixty-three runs, the upper or lower limits specified for single specimens were exceeded only twice and

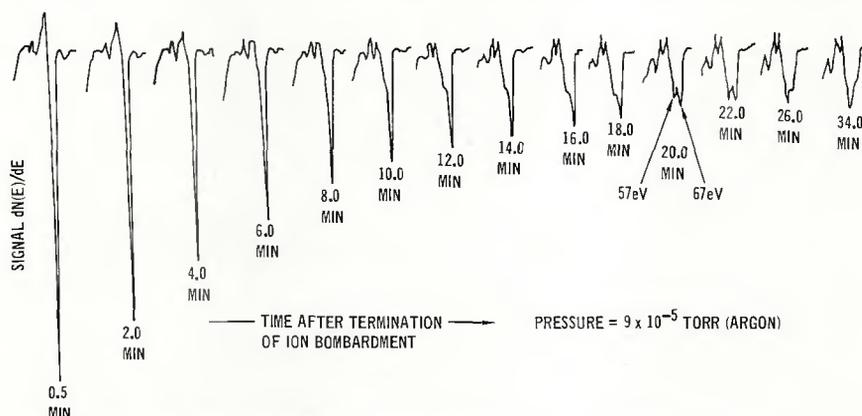


Fig. 6 — Oxidation of aluminum as studied by chemical shifts in 67eV Auger peak for aluminum

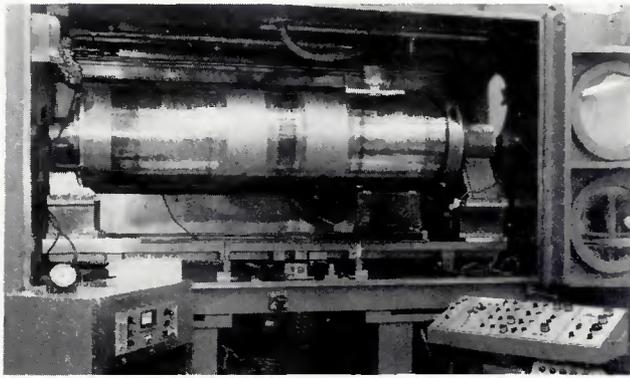


Fig. 7 — Vapor deposition facility

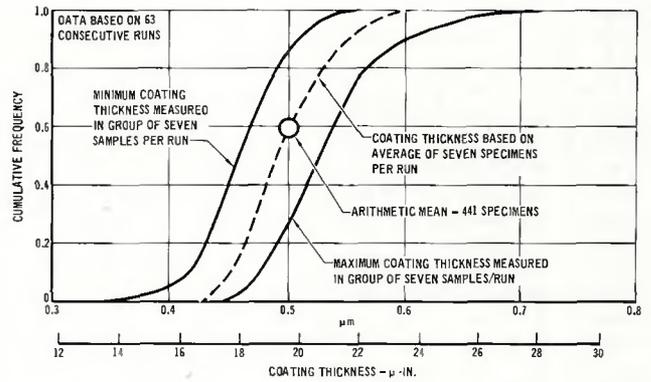
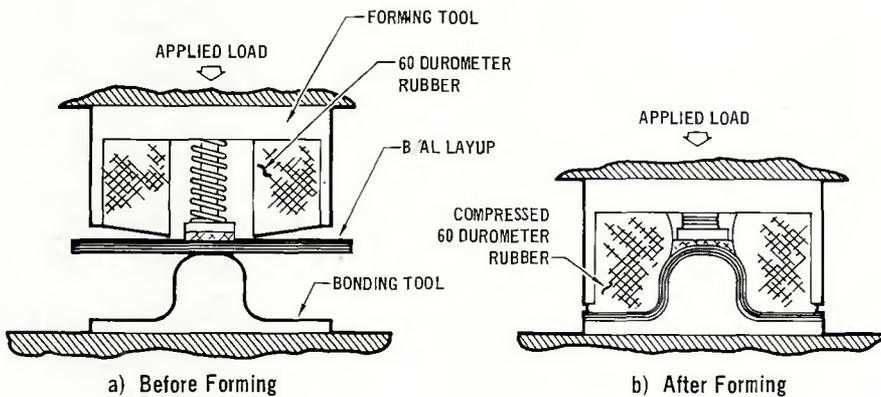


Fig. 8 — Results of weight change quality control samples showing run-to-run variation in copper coating thickness on B/A monolayer



A FLAT PACK LAY-UP IS MADE CONSISTING OF COPPER-COATED BORON ALUMINUM MONOLAYER FOILS, TITANIUM FOIL INTERLEAVES, STOP-OFF COATED SLIP SHEETS AND AN OUTER ENVELOPE SHEET. THIS LAY-UP IS THEN PLACED IN A BRAKE PRESS ON THE BONDING TOOL AS SHOWN IN (a). PRESSURE THEN IS APPLIED AS IN (b) TO FORM THE ENTIRE LAY-UP. FORMING PRESSURE IS MAINTAINED WHILE THE OUTER ENVELOPE SHEET IS WELDED ALL AROUND THE PERIPHERY OF THE BONDING TOOL. THE INTERIOR OF THE WELDED PACK IS EVACUATED AND THE FORMING PRESSURE THEN RELEASED. ATMOSPHERIC PRESSURE HOLDS THE LAY-UP TO THE TOOL AND PREVENTS SPRINGBACK OF THE B/A AND TITANIUM FOILS.

MECHANICAL FORMING OFFERS THE FOLLOWING ADVANTAGES:

1. SUBSTANTIAL REDUCTION OF LAY-UP COSTS
2. HIGHER QUALITY OF FINAL PRODUCT BY VIRTUE OF BETTER CONTROL OF RADIUS CONTOUR AND CONTACT BETWEEN PLIES
3. PERMITS INCLUSION OF TITANIUM ALLOY INTERLEAVES — SPRINGBACK OF TITANIUM CANNOT BE PREVENTED IN HAND LAY-UP OPERATIONS.

Fig. 9 — Procedures for mechanically forming B/A structural shapes prior to eutectic bonding



Fig. 10 — Mechanically formed hat section ready for bonding

in both cases by 1 micron. or less.

This analysis demonstrated that coating thickness can be closely controlled although some run-to-run variation within a narrow range can be expected. No requirements are specified to ensure a minimum variation among the seven specimens from the same coating run. However, coating parameters were selected to reduce the variation within the individual groups with the objective of holding this variation to 4 micron. or less. An analysis of weight change data show that sample-to-sample variation within individual groups of seven averaged 2.9 micron.; 86% of the runs showed a variation of less than 4 micron.

Layup Techniques

A principal advantage of fabricating structural shapes from individual monolayer foils is the versatility this approach offers for producing complex shapes of varying thickness. The layup of flat sheets is simple and straightforward, but the fabrication of structural shapes can be considerably more difficult. Initially, layups of this type were made by handforming each ply over a shaped tool. This procedure was time consuming and the quality of the finished part was variable because good contact between adjacent layers especially at corners, was not always attained. Therefore, a study was undertaken to develop layup procedures that would reduce costs and improve quality. At the same time it became necessary to further increase the versatility of the monolayer approach to permit the inclusion of local reinforcement in the form of titanium interleaves. The use of individual monolayers was ideally suited to interleaving, but hand layup techniques are not suitable because of the springback characteristics of titanium.

The need for an improved layup process for shapes led to the development of a mechanical forming procedure. This concept, shown sche-

matically in Fig. 9, utilizes a flat layup consisting of a cover sheet, slip and spacer sheet as well as the boron-aluminum interleaves with titanium. This entire pack is formed as a single unit and the forming pressure maintained until the cover sheet is welded to the bonding tool and the pack evacuated. A welded pack ready for bonding is shown in Fig. 10.

Several small hat-section test assemblies were fabricated with prototype tooling to develop tooling shapes, materials, etc. These parts were complex in that thickness was varied by terminating plies externally and titanium alloy interleaves were included internally. Some of the titanium plies were continuous while others were terminated internally at an abutting boron-aluminum ply. Metallographic examination of these test assemblies, which were bonded in the conventional manner after forming, showed them to be of high quality with good contact between adjacent plies. On the basis of the good results obtained on the subsized assemblies, mechanical forming was selected for fabricating structural shapes. A typical stringer cross-section is shown in Fig. 11. The development of mechanical forming represents a significant advance in boron-aluminum fabrication. Mechanical forming, followed by eutectic bonding or any other low pressure, liquid phase bonding system, permits the fabrication of thick, complex structural shapes which vary in thickness and incorporate titanium interleaves. Assemblies of such shapes cannot be easily fabricated by other manufacturing methods with the degree of design flexibility afforded by this system.

Bonding Procedure

Eutectic bonding is carried out in an autoclave under a nominal pressure of 250 psi. Heating is accomplished in an auxiliary furnace capable of handling parts as large as 5 x 6 ft. This furnace has six heating zones, each of which is individually controlled and programmed to provide the same thermal cycle of heating rate, maximum temperature and time and temperature. The autoclave and furnace are shown in Fig. 12.

After parts have been laid up and placed into weld sealed packs as shown in Fig. 10, they are instrumented with 24 thermocouples. Six of these are used to control the heating cycle and the remainder used to "map" the temperature profile of the overall part. The instrumented packs are placed in the furnace which in turn is moved into the autoclave. The packs are then evacuated to a pressure of less than 1×10^{-2} torr, back-filled with argon, and re-evacuated. This procedure is repeated three times. Then, the autoclave is pressur-

ized to about 250 psi and the heating cycle started. Parts are heated at a rate of about 10 F/min to the bonding temperature range from 1030 to 1060 F. When the thermocouples indicate that the entire part has reached this range, the power is shut off and the pack allowed to cool to

300 F under pressure.

The studies conducted to establish a suitable thermal cycle for eutectic bonding showed that boron filament degradation could be expected as a result of bonding but that the degree could be minimized by controlling the thermal cycle. On the basis of these

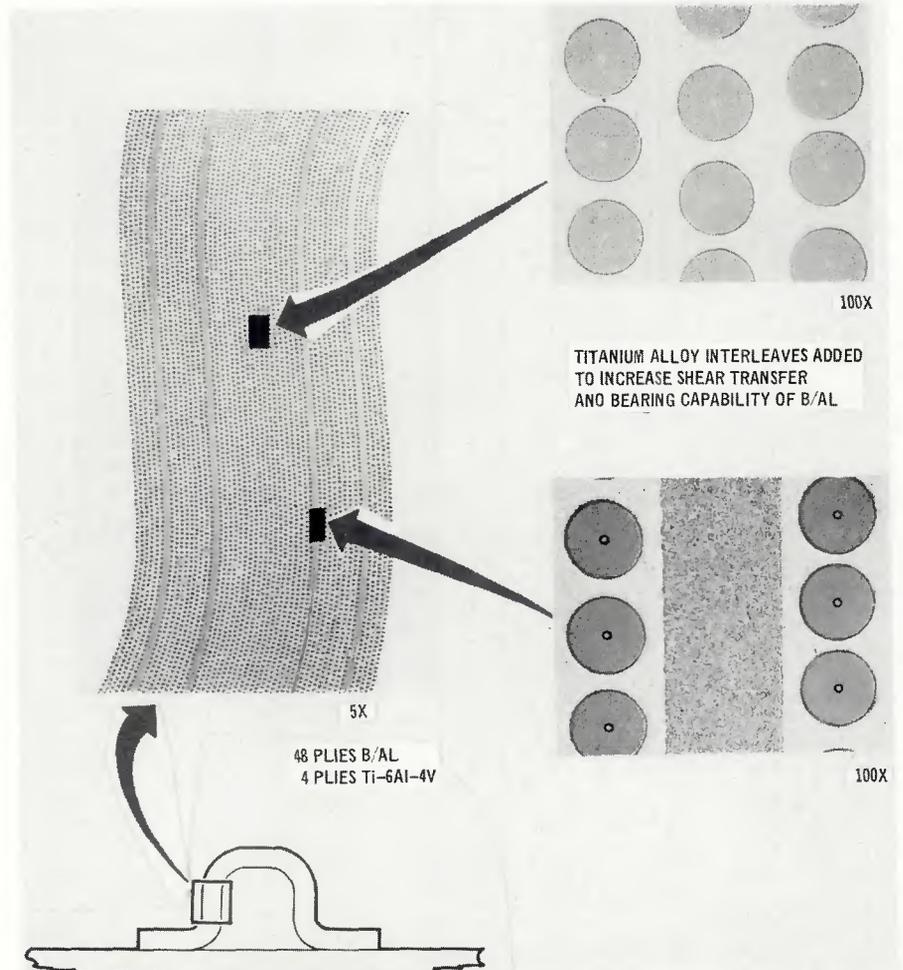


Fig. 11 — Structural component fabricated by mechanical forming and eutectic bonding

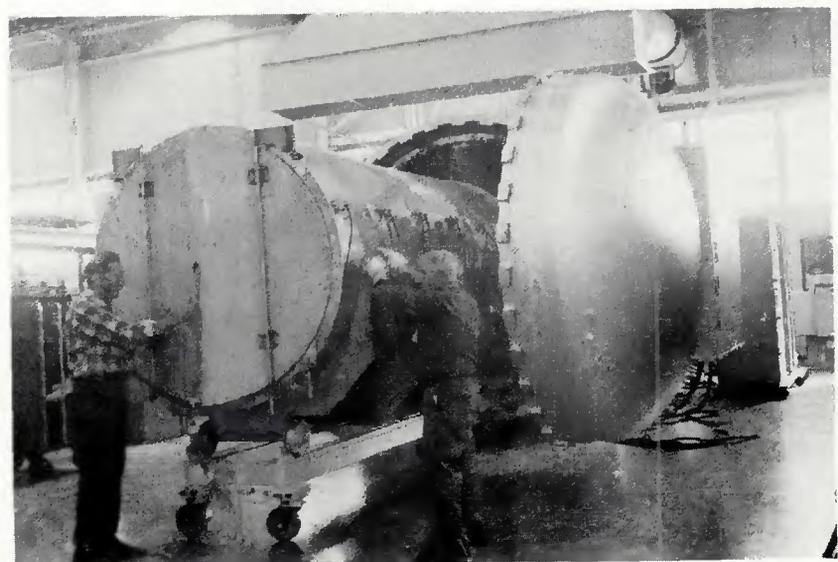


Fig. 12 — Bonding furnace and autoclave

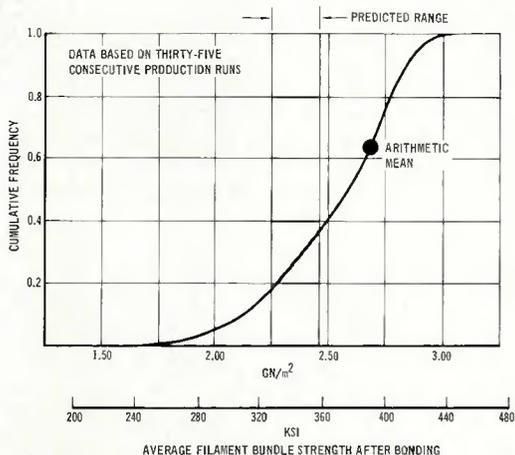


Fig. 13 — Strength of boron filaments after eutectic bonding based on tensile specimens bonded with production parts



Fig. 14 — Boron-aluminum demonstration panel

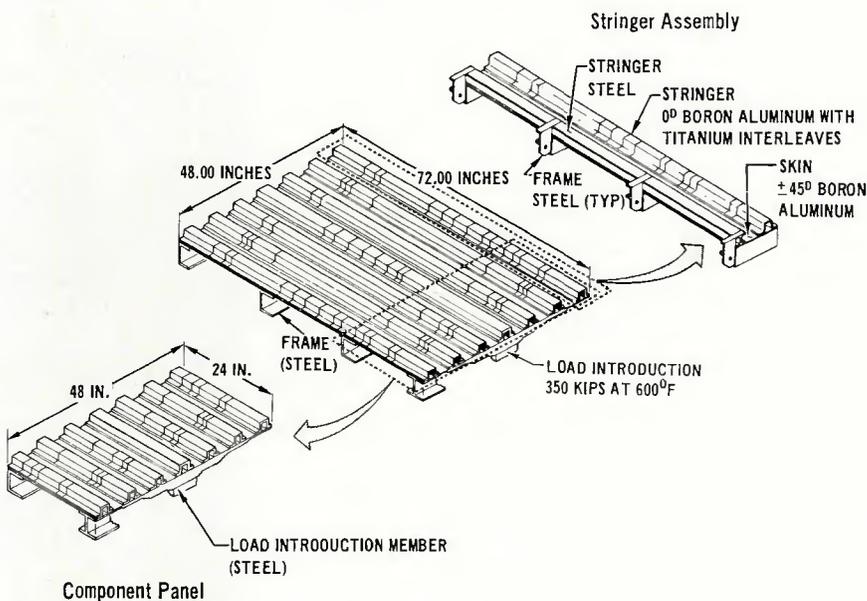


Fig. 15 — Load redistribution panel and test components

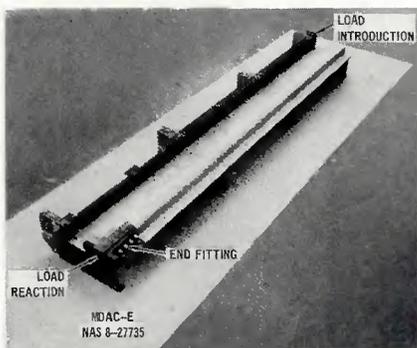


Fig. 16 — Stringer test component of load redistribution compression panel

studies, thermal cycle parameters were established for the bonding cycle to limit the degradation to an acceptable amount. The lower temperature limit was established at 1030 F with the time at this temperature not to exceed 15 min. An upper of 1060 F was selected with the time of

this temperature not to exceed 7 min. Generally, the amount of degradation which occurs during production runs is less than would be predicted on the basis of the fiber degradation studies. This has been demonstrated by quality control specimen test results. Each bonding run contains tensile test samples which are eutectically bonded along with the components in the pack. These tensile specimens are made from monolayer material for which as-received properties are known. A direct comparison of tensile strengths before and after bonding is made of each run. The results of the test specimens from 35 runs shown in Fig. 13. These data show that the average bundle strength of all of the runs was well above the predicted value based on the filament degradation studies. Also, the minimum predicted strength was exceeded in more than 60% of the runs. These results demonstrate that the eutectic

bonding cycle can be controlled to minimize degradation of the boron filaments caused by interaction of the filament and matrix during high temperature exposure.

Component Fabrication

The scale-up of the eutectic bonding process for the production of large size boron-aluminum structures culminated in the fabrication of several complex components. The first of these, shown in Fig. 14, was a 4 x 5 ft. skin-stringer panel intended to demonstrate the feasibility of producing large size boron-aluminum structures. The panel skin consists of 8 plies of boron-aluminum cross-plyed at ±45 deg orientation. The eleven stringers, 1.4 in. high by 24 plies (0 deg orientation) thick, were bonded as hat sections and then cut longitudinally to provide zee shapes. The stringers were mechanically fastened to the skin. All of the details were of uniform thickness and did not require interleaving of local reinforcement. This panel was designed to carry a uniformly distributed compression load of 7200 lb/in. combined with shear load of 1000 lb/in. at 500 F.

Successful completion of the demonstration panel led to a subsequent development program (Contract NAS 8-27735) for the "Design, Process Development, Manufacture, Test and Evaluation of Boron-Aluminum for Space Shuttle Components". The ultimate objective of this multi-phase program was to design and fabricate a 4 x 6 ft. compression panel for 600 F service. The panel was to be capable of withstanding a concentrated load of 350 kips introduced at one end and redistributing it into a uniform running load at the opposite end. The structural integrity of the panel was to be demonstrated by testing two component assemblies. Figure 15 is a schematic representation of the panel and test components.

Several unique features were incor-

porated in the panel design to provide load carrying capability while minimizing weight. The skin thickness varied across both its length and width from a maximum of 62 plies to a minimum of 10. Construction of the hat section stringers varied with their lateral distance from the load introduction point. The centerline stringer varied from 52 plies in thickness at the load introduction end to 21 plies at the reaction end. The other stringers were thin at the load introduction end and increased in thickness as the individual stringer load was introduced and increased by shear transfer along the panel length. For example, the outboard stringers varied from 5 plies at the load introduction end to 23 plies at the reaction end of the panel. In addition, both the skin and stringers had titanium alloy foil interleaved with the boron-aluminum to improve shear transfer and bearing capabilities. The number of interleaves and their location and length varied according to loading conditions. Some interleaves were continuous while others were terminated internally at an abutting boron-aluminum ply. To accommodate internal ply termination, titanium alloy interleaves were approximately the same thickness as the boron-aluminum monolayers (~0.008 in.).

Varying the thickness of detail parts and incorporating titanium alloy interleaves both represented "firsts" in the fabrication of boron-aluminum structural components. The suitability of these innovations and the ability to analyze the load distribution and deflection of a boron-aluminum struc-

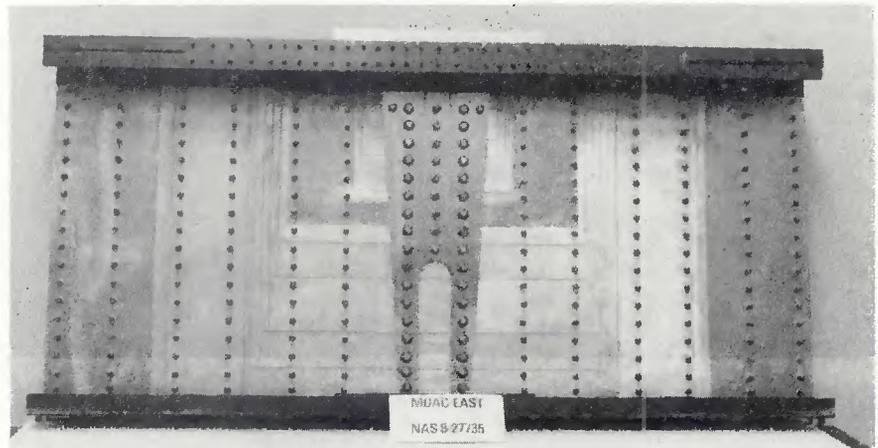
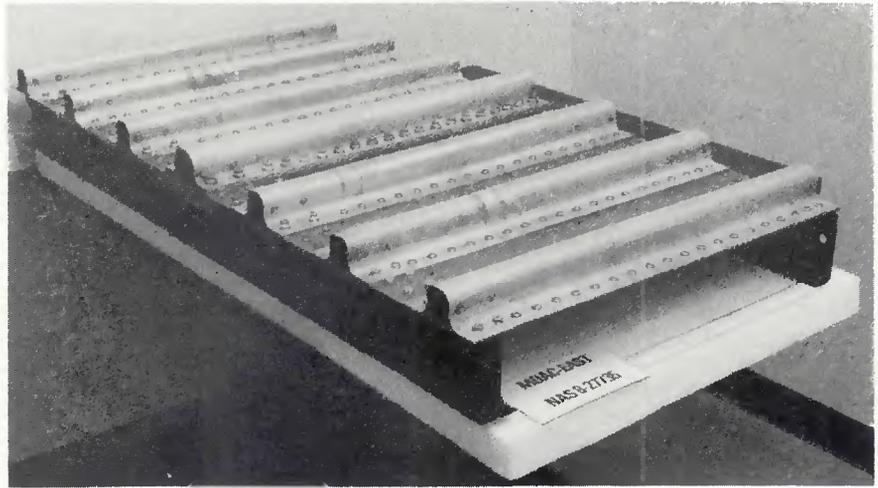


Fig. 17 — Load redistribution component panel. (Top) Stringer side; (Bottom) Skin side.

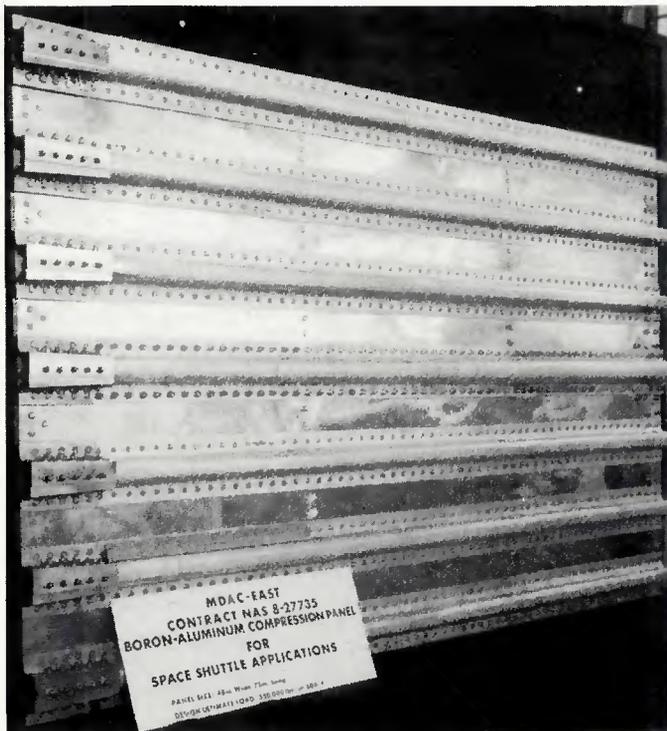


Fig. 18 — Load redistribution panel assembly

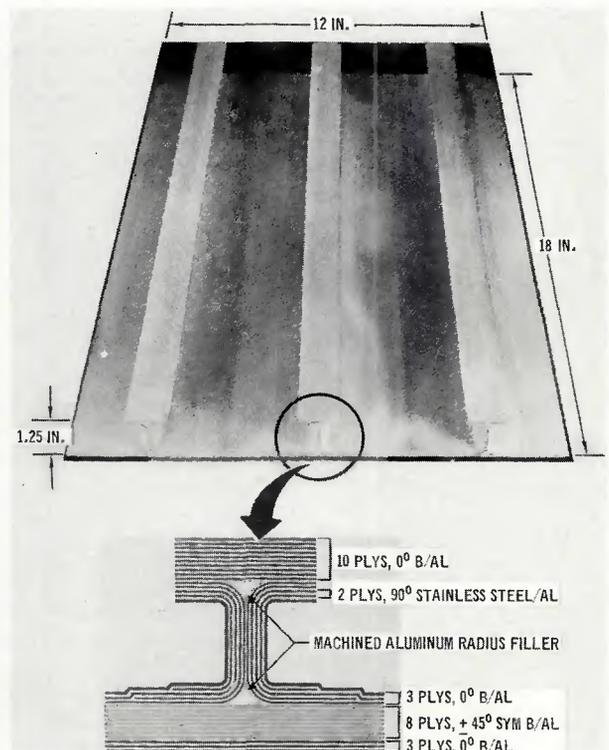


Fig. 19 — Integrally stiffened boron-aluminum skin panel

ture under complex loading was demonstrated by means of the test components. The first of these was the stringer element which was to demonstrate that column buckling and crippling behavior of the stringers, under the influence of a varying applied load, could be predicted analytically. A photograph of this component which duplicated the outboard stringer is shown in Fig. 16. This stringer was the most critical from the standpoint of compression and bending loads. The test assembly consisted of a boron-aluminum stringer and skin and a steel load introduction member. A load of 100 kips was introduced into the steel member at room temperature and reacted at the other end of the assembly solely by the boron-aluminum through a titanium end fitting. Stringer deflections were measured at several locations and good correlations were obtained when compared with predicted values.

The second component, 24 x 48 in., (Fig. 17) duplicated the load-introduction end of the full size panel and was tested to permit comparison of actual and predicted values of load distribution and panel deflection. This test was conducted at 600 F; the panel was successfully loaded to 400 kips-50 kips above its design ultimate load. Again, good correlation was obtained between measured and predicted values for internal and external load distribution and panel deflections.

Upon completion of the component tests and verification of the structural analysis, the full size panel was fabri-

cated. The completed assembly, shown in Fig. 18, is ready for NASA testing as specified under NAS 8-27735.

The unique fabrication steps required for the manufacture of boron-aluminum structural elements can also be used to advantage in producing low cost composite structures. This was demonstrated by producing an integrally stiffened panel representative of the type of structure commonly used in airframe and spacecraft construction (Fig. 19). This panel features a combined cross-plyed and unidirectional skin at 0 and ± 45 deg and unidirectional stringers with local radius reinforcement. In this case, the stiffeners were reinforced with a composite material consisting of high strength, cold worked stainless steel filaments in an aluminum matrix. This assembly was laid-up in several subassemblies for design flexibility and manufacturing ease but bonded in a single thermal cycle as an integral unit.

This type of fabrication offers design flexibility in that skin thickness may be varied, the stiffness geometry and orientation can be varied and local reinforcement can be added. In addition, this type of boron-aluminum structure offers high efficiency. The panel shown in Fig. 19 is 31% stronger than Ti-6Al-4V alloy on an equal weight basis or 17% lighter if compared on the basis of equal strength. Also, this method of construction can be cost-comparative with complex titanium shapes; simple tooling permits easy layup, bonding is

a onestep operation and little finish machining is required.

Conclusions

The results of development studies have shown that eutectic bonding is a viable process for fabricating complex boron-aluminum structures. Critical processing parameters have been identified and evaluated so that bonding can be accomplished with minimal degradation of filament properties. Finally, production capability has been developed and demonstrated to the extent that boron-aluminum is now ready for selected application to aerospace structures. Such applications can result in substantial weight savings without corresponding cost penalties if the unique fabrication characteristics of boron-aluminum are utilized fully.

Acknowledgements

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WRC Bulletin No. 184 June 1973

"Submerged Arc Weld Hardness and Cracking in Wet Sulfide Service"

by D. J. Kotecki and D. G. Howden

This study was undertaken to determine:

- (1) The causes of higher-than-normal hardness in submerged-arc welds in plain-carbon steels
- (2) The levels of strength or hardness which will not be susceptible to sulfide-corrosion cracking
- (3) Welding procedures which will assure that nonsusceptible welds will be produced.

Concentration is primarily on weld metal, though some consideration to the weld heat-affected zone is given. The study covered a two-year period. The first year was concerned with a macroscopic view of the weldments. In that first-year study, some inhomogeneities were observed in weldments which are not obvious in a macroscopic view of the weldment. It appeared likely that these inhomogeneities could affect the behavior of the weldment in aqueous hydrogen-sulfide service. Accordingly, their presence and effects were investigated during the second year.

The price of WRC Bulletin 184 is \$3.50 per copy. Orders should be sent to the Welding Research Council, 345 East 47th Street, New York, N.Y. 10017.