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94-1002

OUT-OF-FURNACE BRAZING TECHNOLOGY FOR ASSEMBLY OF LARGE CARBON-CARBON SPACE STRUCTURES

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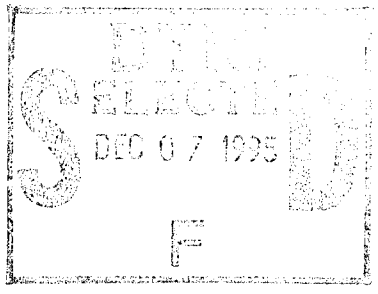

April 1994

Final Report

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13. Supplementary Notes

14. Abstract

In this Phase I SBIR program, Foster-Miller demonstrated the feasibility of an innovative joining technique for two-dimensional carbon/carbon (C/C) composite materials using active braze alloys. Traditionally, brazing of C/C composites required use of high-temperature vacuum furnaces. Vacuum brazing required expensive capital equipment with a limited working area and brazing cycles on the order of several hours to more than a day. The Foster-Miller technique utilized a controlled atmosphere induction brazing to reduce the brazing times to minutes and increased the interlaminar strengths by using of the proprietary Foster-Miller z-pin reinforcement technique. The use of the out-of-furnace brazing concept combined with z-direction reinforcements showed strengths of up to 1550 psi compared to the vacuum furnace braze joint strengths of 1153 psi. The constituents of the purging gases as well as proprietary solid oxygen getters were adjusted to eliminate all oxygen while brazing, thus minimizing problems associated with oxidation. This program analyzed the advantages of the oxygen getters in addition to the effect of braze layer thicknesses on strengths of the brazed C/C joints. The follow-on Phase II program will focus on transitioning the brazing technology to aerospace and terrestrial thermal management and structural applications of C/C composites.

15. Subject Terms Bimodal systems
Out-of-Furnace Brazing, C/C composites, joining

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FIGURES (Concluded)

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1.0 SUMMARY OF PHASE I ACHIEVEMENTS

During this Phase I program, Foster-Miller proved the feasibility of an innovative joining technique for two-dimensional (2-D) carbon/carbon (C/C) composite materials using active braze alloys. Joining of C/C composites has traditionally been confronted by the two major limitations of (1) use of vacuum furnaces and brazing cycles on the order of several hours, and (2) poor joint strengths due to low interlaminar strengths of the parent C/C material. The Foster-Miller technique utilized controlled atmosphere induction brazing to reduce the brazing times to 20 min and increased the interlaminar strengths by use of the proprietary Foster-Miller z-pin reinforcement technique. The use of the out-of-furnace brazing concept showed strengths of up to 1550 psi as compared to the vacuum furnace braze joint strengths of 1153 psi.

Foster-Miller's innovative controlled atmosphere brazing concept and the use of z-direction fibers are schematically shown in Figures 1 and 2, respectively. Traditionally, brazing of C/C composites required use of high temperature vacuum furnaces which, in addition to needing costly mechanical and diffusion vacuum pumps, also require lengthy and costly brazing cycles on the order of a few hours to more than a day. Use of out-of-furnace brazing techniques is essential to fabrication of large C/C structures. Additionally, out-of-furnace brazing can also be used for repair of the high temperature material structures and high volume production of C/C components.

The z-fibers dramatically increase the interlaminar strength of the 2-D C/C composites. This translates into increased strength for the braze joints. In addition to the increased strengths, the z-fibers could also be used to control the joint gaps and thus the thickness of the braze layers, which will aid tailoring of the thermal stresses. Also, proper choice of z-pins can result in vastly

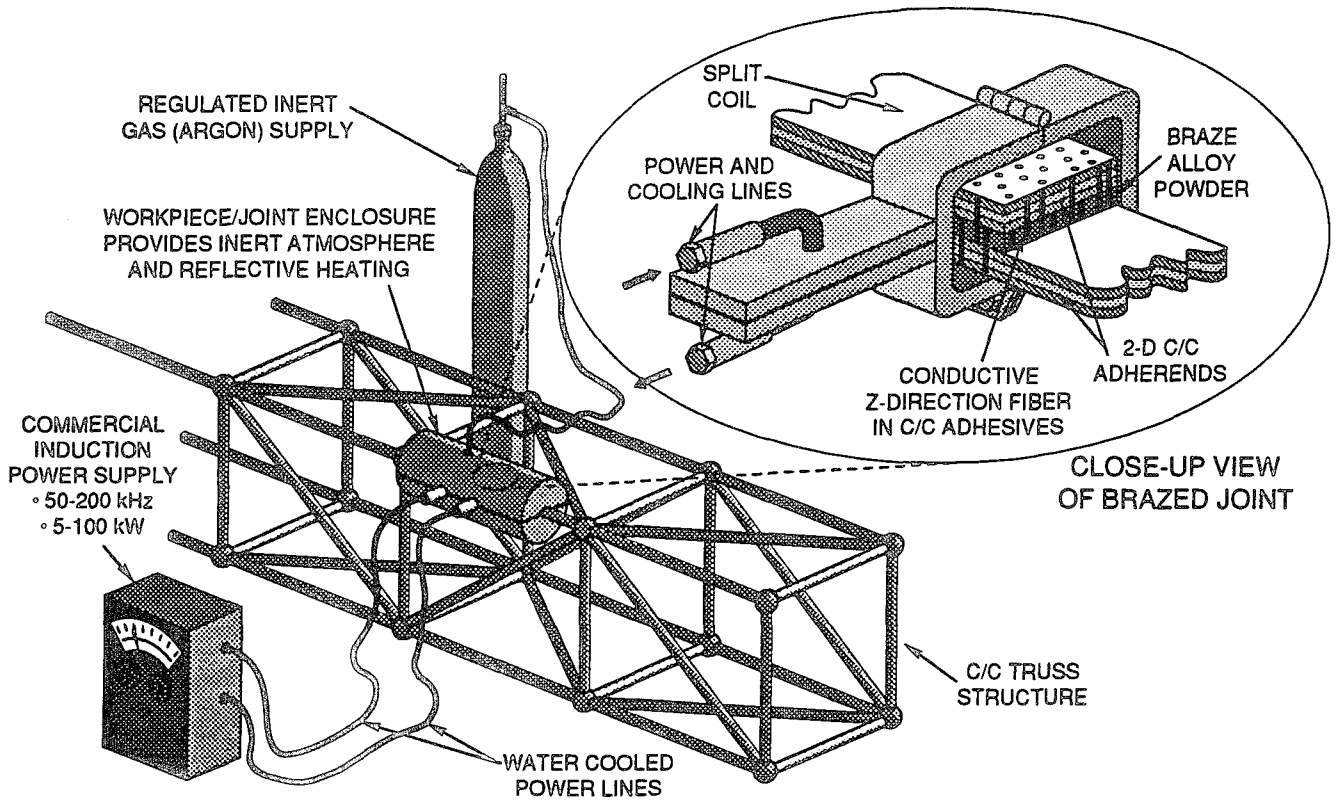


Figure 1. Out-of-furnace brazing schematic for repair of space structures.

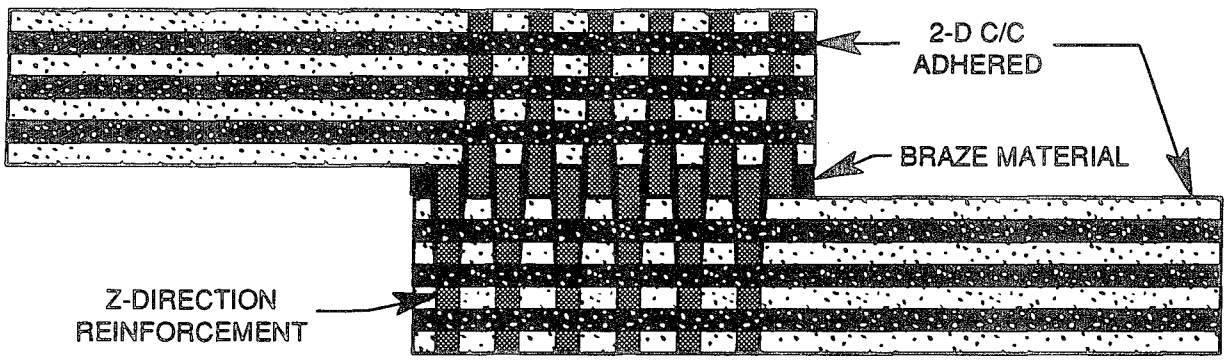


Figure 2. Superior 2-D C/C joint design based on Phase I technology.

increased through thickness thermal conductivity and controlled Coefficient of Thermal Expansion (CTEs) in the transverse direction. These are essential to application of C/C composites for thermal management in avionics and space systems.

Technical objectives and accomplishments of Phase I are presented in Table 1. The technical objectives in this program consisted of fabricating two-direction reinforced C/C composites and then developing an out-of-furnace brazing technique for these composites of strengths comparable to those of vacuum furnace brazed C/C composites. Specimens reinforced with three pin materials in addition to control specimens were successfully brazed using an active Ag-based braze alloy. The brazed specimens were characterized for shear strength and thermal conductivity. Microscopy studies showed a good interface between the braze and the C/C composite. A chemical analysis of the braze after the joining procedure showed no change in the bulk chemical composition.

Table 1. Summary of Phase I objectives and accomplishments.

Phase I Objectives	Accomplishments
1. Develop an out-of-furnace braze joining technique for C/C composites	• Demonstrated feasibility of joining C/C composites using reducing gases and O_2 gettering solids. • The unreinforced control specimen showed equivalent strengths for vacuum-furnace-brazed and out-of-furnace brazed joints
2. Fabricate z-pin reinforced C/C joints for improved strength.	• Fabricated C/C joint specimens of the following configurations - Control specimen - Ag-ABA Braze with no pins - Mo pins with Ag-ABA Braze - Stainless steel pins with Ag-ABA Braze - P100 pins with Ag-ABA Braze • Scanning electron microscopy revealed - Good C-braze bond - Good adhesion between pin and braze and pin and C/C composite
3. Improve thermal conductivities of the joints via z-direction reinforcements	• The thermal conductivities of the P100 and Mo z-direction reinforced specimens doubled compared to the control specimen without any z-direction pins • No significant reduction in thermal conductivity was observed upon brazing

2.0 INTRODUCTION

Carbon/Carbon composites have been considered the only viable option for structural applications in high temperature environments and space structures due to their excellent retention of stiffness and strengths at high temperatures and low specific gravity. Increasingly, C/C composites are also being considered for thermal management applications for satellites and avionics. For successful implementation of high performance C/C composites, it is essential to develop methods to join C/C composites to themselves as well as dissimilar materials. Extensive research has been conducted in this field and significant advances have been made in areas of designing new active braze alloys. Many of these braze alloys incorporate a carbide forming active metal, often Ti, which helps the molten braze to wet the C surface. The use of active braze alloy also eliminates costly and time consuming surface preparation and fluxing requirements. These studies, however, have almost always utilized vacuum furnace brazing techniques requiring stringent vacuum levels of 10^{-5} torr or better. The use of a vacuum eliminates formation of oxides of the C and the active metal in the braze alloy. Vacuum furnace brazing, however, requires highly capital intensive equipment, such as high pressure chambers along with vacuum pumps. Vacuum furnace brazing also requires long time cycles, sometimes over 24 hr. Additionally, it is not practical to braze large parts in vacuum furnaces. Many of the large military spacecraft structures such as Space Based Radar (SBR), Survivable Power Subsystem (SUPER), and Zenith STAR may involve C/C structures in excess of 350 ft. Figure 3 depicts the size and complexity of some of these structures.

Consequently, mechanical fastening becomes the joining method of choice for large C/C structures. Despite the weight penalty associated with mechanical joints, these methods are more readily available for assembly of large structures. For cost reasons and any large scale application of C/C composites, it is desirable to develop brazing techniques which do not require

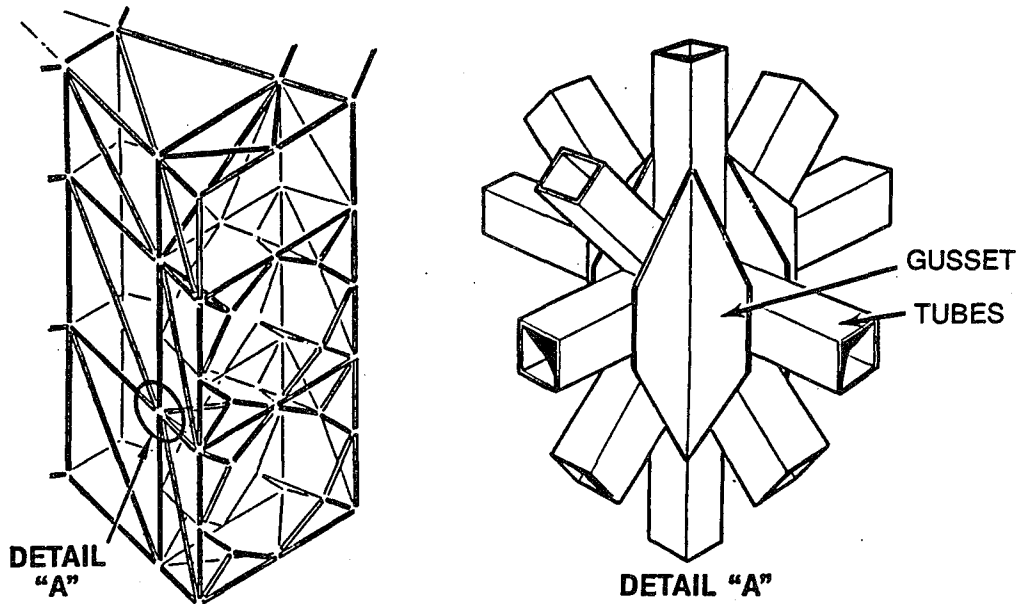


Figure 3. Generic C/C space truss structures involving a complex joint (supplied by Rockwell Internationa, city, state).

the use of a vacuum furnace. Also, there is often a need for on-site repair of these structures when in service. To meet these requirements, Foster-Miller envisions a portable brazing technique analogous to the currently used welding techniques for metallic structures. Also, vacuum furnace equipment will rarely be available for repairs, especially in space based environments.

The second major limitation of the 2-D C/C composites is their inherently low interlaminar shear strength. Often, the failure of the composites is within the plies rather than the braze joints. This can be overcome by use of braided 3-D C/C composites but at a prohibitively increased cost. Thus it is essential to develop a low cost approach to making 2-D C/C composites stronger. The Foster-Miller proprietary z-direction process provides the ability to transversely reinforce a 2-D C/C composite and dramatically improve its interlaminar properties. Proper selection of the

z-direction fibers also greatly aids in controlling the through thickness thermal conductivity of the composites. This can benefit thermal management applications especially in avionics, where use of C/C composites is increasingly being considered.

The process utilizes induction heating of the C/C composites in a controlled atmosphere. The constituents of the purging gases are adjusted to minimize O_2 while brazing, thus eliminating problems associated with oxidation. The braze material is placed between the adherents and the entire assembly is inductively heated to make the braze flow and form a strong joint (Figure 2). The joint strength is provided by mechanical by anchoring the pins to the braze in and by the strength provided by the braze itself. The z-fibers also raise localized interlaminar shear strengths of the C/C material. The technology developed will be an economically feasible method to join C/C composites to themselves and to other high temperature materials using active braze alloys. This technology can be used for both on-site manufacturing of large structures as well as an easy repair technique for the already existing structures. Also, due to the short cycle times, this is a technique for rapid manufacturing of components requiring strong braze joints.

3.0 PHASE I TECHNICAL OBJECTIVES

The principal technical objective of this program was to demonstrate the feasibility of induction heating in conjunction with z-direction reinforcement to produce strong, thermally efficient C/C composite joints in a nonvacuum, controlled atmosphere. The use of z-direction pins eliminates the weak link in the 2-D C/C composites, i.e., the poor interlaminar shear strength. Induction brazing of the C/C composites helps to reduce the joining costs by eliminating the need for an expensive vacuum furnace and reducing the brazing times. The following objectives were defined for the program:

- Select 2-D C/C base materials, z-direction reinforcement materials and the braze alloy by addressing the space system mechanical and thermal properties requirements.

- Fabricate C/C composites with at least three z-pin materials in addition to the control specimens. Design the appropriate induction coils to achieve efficient heating and brazing of the C/C composites.

- Perform the induction brazing trials and study the effect of fiber geometry, z-pins, braze materials, coil spacing, inductor design and use of "susceptor" material.

- Assess the joint properties via double notch shear test, thermal conductivity and microscopy studies.

- Make recommendations for the scale-up of the process for application to rapid manufacturing of thermal management components and large space structures.

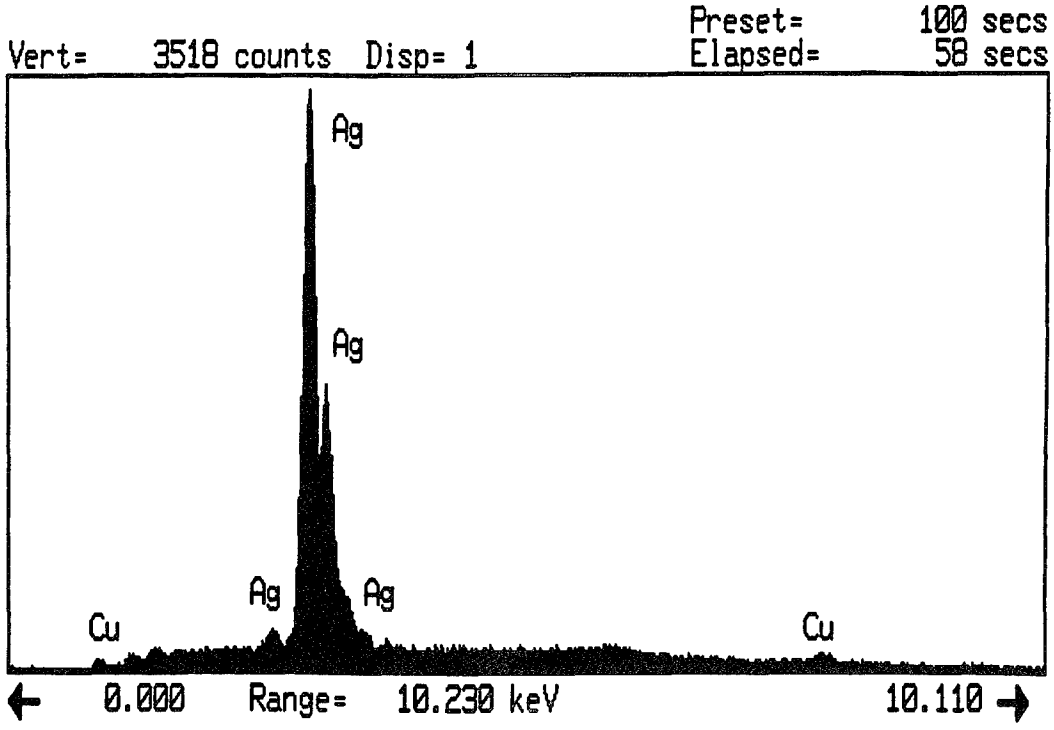
4.0 PHASE I RESEARCH AND RESULTS

During the Phase I effort, Foster-Miller demonstrated a low cost innovative process to join C/C composites out-of-furnace in a controlled atmosphere. The out-of-furnace brazing was achieved by using a reduced Ar/H_2 atmosphere and O_2 gettering Ti foils. By combining this out-of-furnace brazing technique with the proprietary Foster-Miller z-direction pins to increase the Interlaminar Shear Strength (ILSS) of the C/C composites, increased braze joint strengths were shown. The strength was increased by delaying the shear failure mode within the C/C composites by introducing transverse pins.

4.1 TASK 1 - REVIEW/SELECT MATERIALS AND JOINT DESIGN

The C/C material chosen was fabricated from a Fiberite K641 carbon/phenolic precursor. The densification of the pyrolized polymer was performed by resin infusion (per ACC-4 specifications by C/C Advanced Technologies [C-CAT] Inc., Ft. Worth, TX). Mo, SS (Alloy 304), and pultruded epoxy/P100 pins were selected for z-pins to provide through thickness reinforcement in the joint regions. All the pin materials were 0.254 mm in diam. The P100 pin material was chosen to increase the through thickness conductivity of the adherents whereas Mo and SS were selected to study their effects on induction heating of the C/C composites.

The braze material used in this study was Ag-ABA (a high thermal conductivity Cu-Ag-Ti braze alloy supplied by Wesgo Inc., Belmont, CA). Ag braze was selected due to its high thermal conductivity and was used in the form of a 0.002-in.-thick foil. Figure 4 (a and b) shows Electron Dispersive Spectroscopy (EDAX) plots of two locations in a Ag-ABA braze foil. The braze material was not uniformly alloyed as can be seen from a difference in Ti concentrations in the two EDAX profiles. This is not a major concern in vacuum furnace brazing, where relatively longer brazing cycles are utilized and the liquid state diffusion of Ti in Ag can equalize the braze



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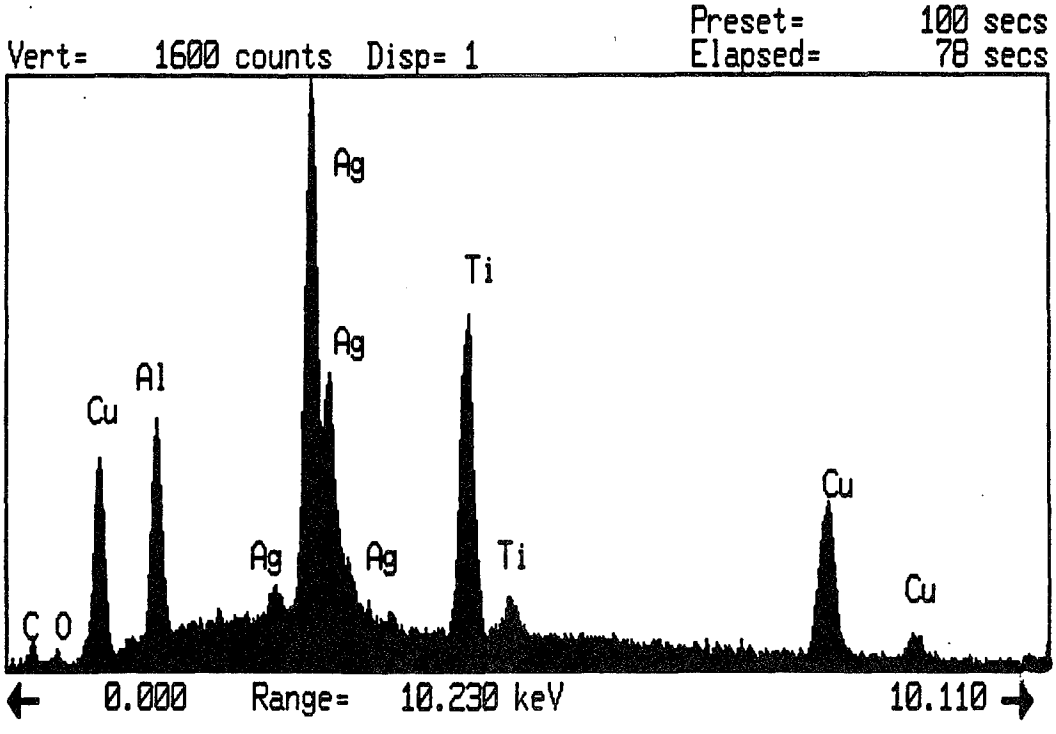


B)

Figure 4. EDAX profiles of as-received Ag-ABA braze foil showing (a) Ti-poor and (b) Ti-rich areas.

composition. However, in out-of-furnace induction brazing, where shorter brazing cycles are necessary to minimize oxidation, there may not be enough time available for Ti diffusion to the C/C composite, resulting in inadequate braze joint strength. Therefore, it is preferable to have a braze alloy with more uniform composition. In addition to Ag-ABA, a few brazing trials were made with Ti braze foil in the form of 0.1-mm-thick foil which was obtained from Johnson and Matthew, Ward Hill, MA.

The brazing trials were conducted in a quartz tube chamber flushed with an O_2 gettering Ar/H_2 atmosphere. The C/C adherents were placed in the quartz tube with the brazing foil sandwiched between the two adherents. A water-cooled Cu induction coil was wound around the quartz tube and used to inductively heat the adherents. The brazing trials are further detailed in subsection 4.4.

4.2 TASK 2 - FABRICATE ADHERENTS

This task included fabrications of z-direction reinforced C/C composites. The details of z-direction reinforcement and C/C fabrication are provided next.

4.2.1 Z-Fiber Process

Foster-Miller developed a process for inserting z-direction fibers to increase the ILSS of the C/C composites and thereby control the delamination of the composites. This simple process converts a 2-D prepreg lay-up to a 3-D structure "on-tool" with little change to the standard autoclave cycle. The z-direction process is schematically illustrated in Figure 5.

The first step in the z-process was to make a foam preform with small diameter rigid fibers. For this program, preforms with 1.5 volume percent were utilized. This preform was placed on a

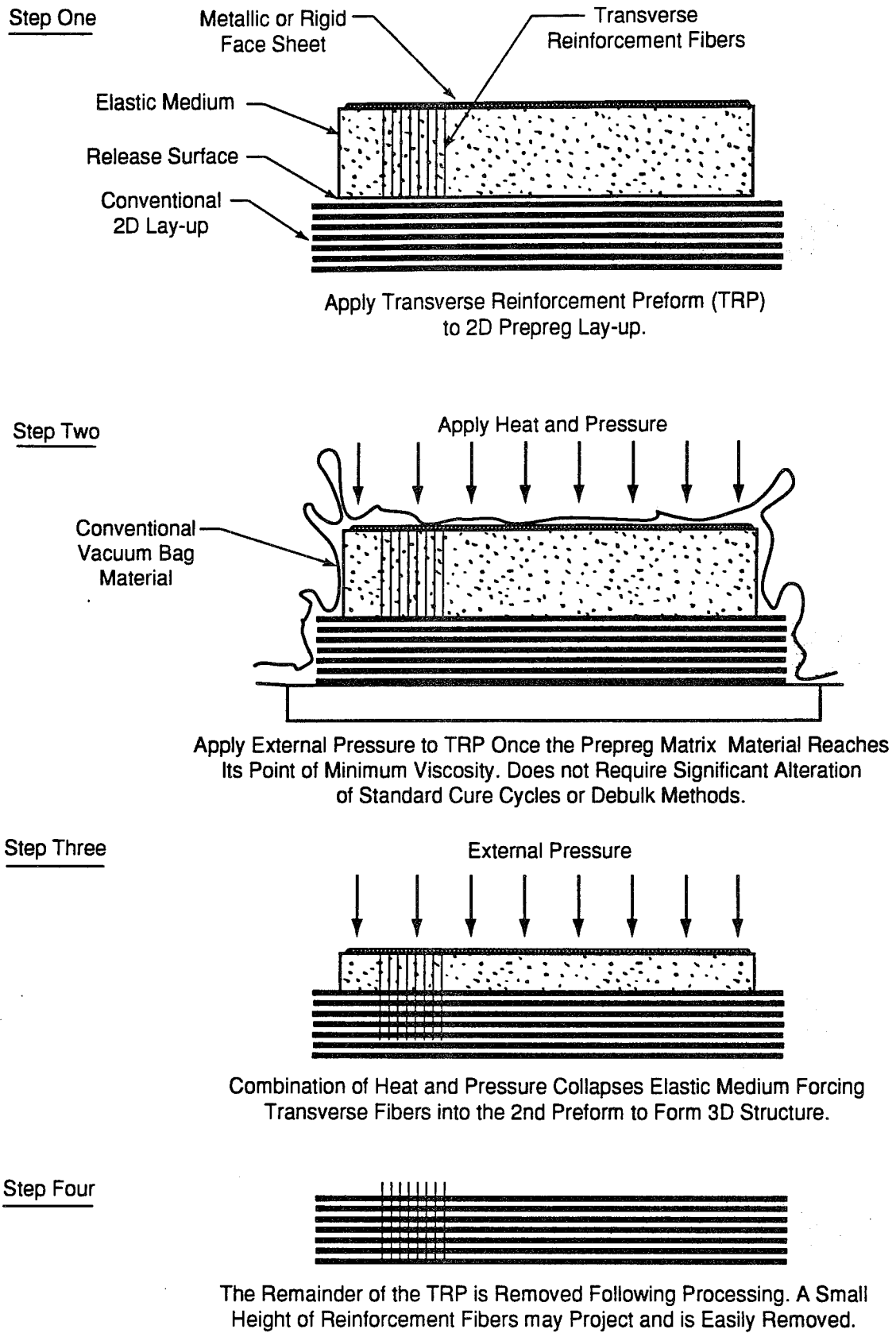


Figure 5. Z-fiber reinforcement process.

2-D phenolic prepreg lay-up. The foam was selected to degrade at a temperature when the polymer has minimum viscosity. During the autoclaving cycle, a combination of heat and pressure compacts the foam and the fibers are driven through the prepreg lay-up. Fiber buckling is prevented by elastic support from the foam. After the cure, the foam residue is removed along with the bleeder and the release ply.

The foam preform can be produced in various thicknesses, fiber densities, and fiber materials. It can also be thermoformed to conform to curved surfaces. This process has been demonstrated by Foster-Miller to be applicable to AS4/34501-6, IM7-8551, and Fiberite K641 for C/C applications. Z-fiber reinforcements of SiC, B, C/epoxy and various metallic pins such as SS, Mo, V and Ti have been demonstrated.

The ability of the foam to provide elastic stability allowed use of small diameter fibers. Foster-Miller's baseline reinforcement was 0.006 in. in diam and could be inserted at an areal density of 0.5 percent or 200 pins per square inch. The small fiber diameter and the ability to insert them at minimum viscosity prevented any significant damage to the composite.

4.2.2 Fabrication of Phase I Adherents

The z-direction insertion was used in the current research during the cure of the C/phenolic laminate. Z-fiber reinforced laminates and control panels were fabricated for evaluation of the brazed joints. As mentioned earlier, SS, Mo and P100/epoxy reinforcement pins were used. The control panels did not have any z-direction reinforcements.

Panels were fabricated from Fiberite K641 C/phenolic prepregs. This fabric is an eight-harness satin weave fabricated from Amoco T300 graphite fiber. The fabric was heat stabilized at 4000°F prior to resin impregnation to reduce any fiber shrinkage and residual stresses during subsequent densification. Panels were constructed with eight plies of the warp aligned fabric.

The z-fiber preform utilized Rohacell 31 IG polymethacrylimide foam as the transfer medium to insert the pins into the phenolic laminate. Pins were inserted using an automated machine which, based on an NC program, controlled both the insertion pattern as well as the pin length. All pin materials were degreased with solvents prior to preform fabrication. The standard vacuum bag and autoclave process utilized to insert pins and cure the laminate are schematically shown in Figure 6. The K641 cure cycle is shown in Figure 7. These panels were shipped to Beaumac Inc., Epsom, NH, for machining/grinding to prepare for carbonization.

The machined panels were properly marked and sent to C-CAT, Inc., pyrolysis and densification by the resin infusion method to the standard ACC-4 specifications. The ACC-4 densification flowchart is shown in Figure 8. Typically, the final density of the composites was 1.65 g/cc.

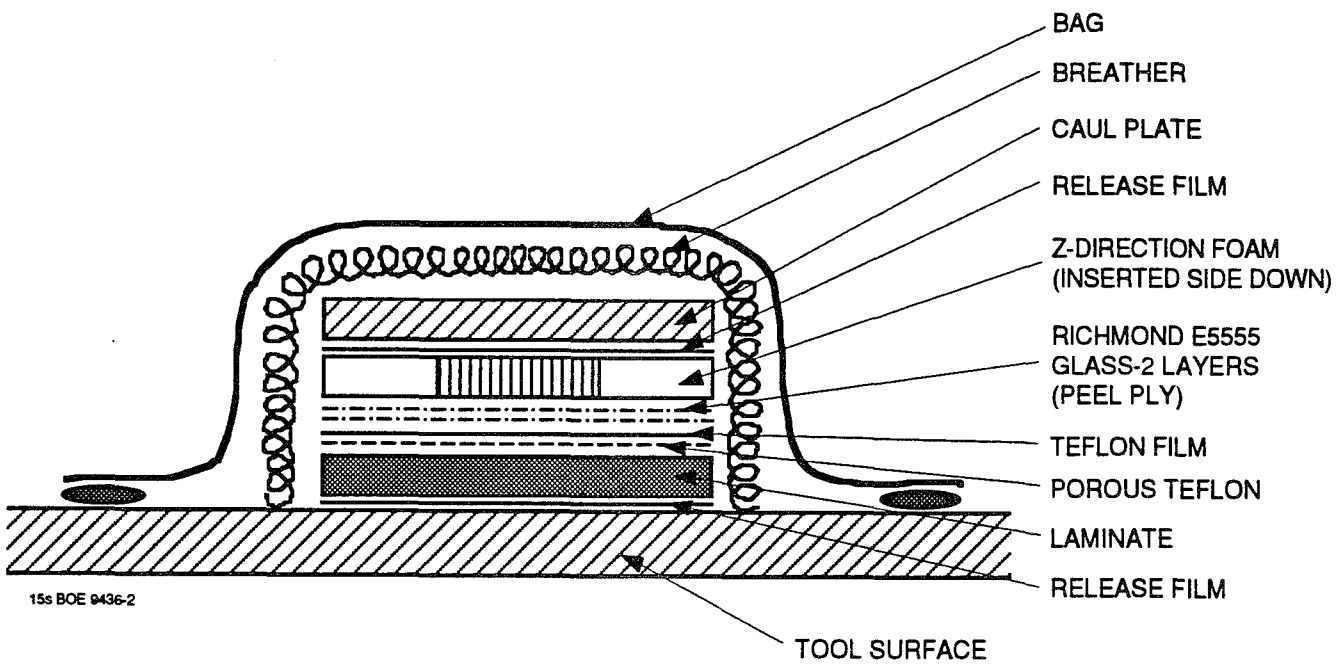


Figure 6. Schematic of the tooling for the autoclaving.

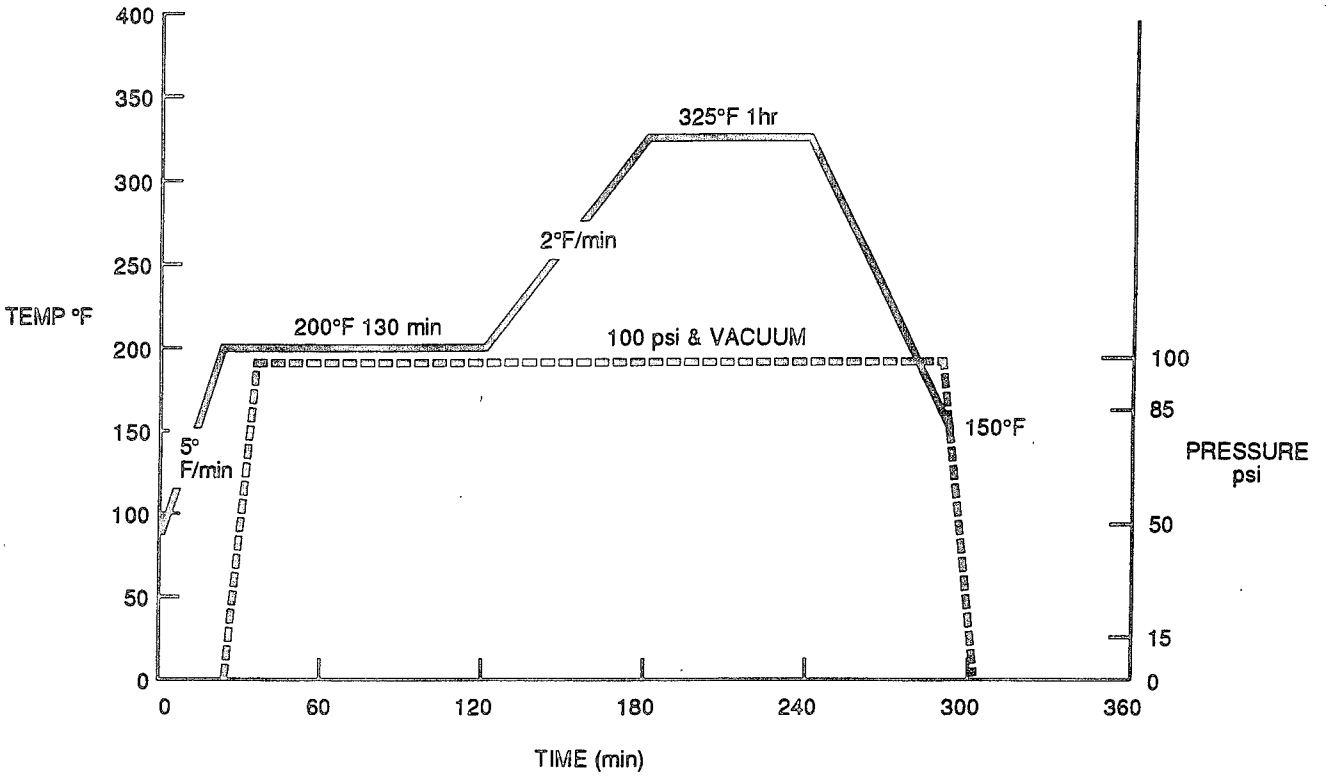


Figure 7. Cure cycle used to process Gr/phenolic precursor laminates.

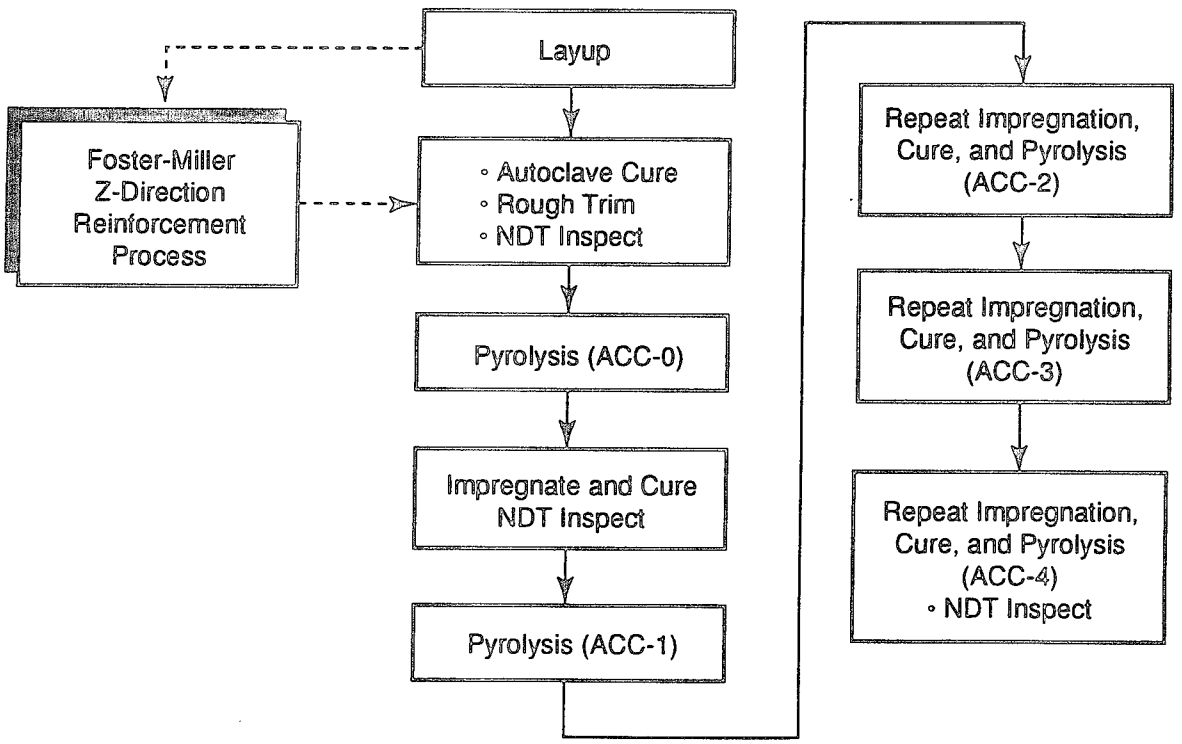


Figure 8. Process flowchart for fabrication of the z-fiber reinforced C/C composite.

4.3 TASK 3 - DESIGN AND FABRICATE SPECIMEN TOOLING AND INDUCTION COIL

A schematic illustration of the induction brazing apparatus utilized in this program is shown in Figure 9a. The brazing trials were performed at Ameritherm Inc., Rochester, NY. The induction power was supplied using an Ameritherm Model SP 25 machine, a 25-kW unit. The machine was operated at a frequency of 75 kHz. The induction brazing trials to fabricate the brazing specimens were performed in a quartz tube of 1.25-in. diam. The temperature of the specimen while being heated was monitored using an Ircon Modline Series 2000 optical pyrometer. The pyrometer had an R25 F05 sensing head and could be operated in a closed loop with the induction unit to control the temperature of the specimen. The quartz tube was sealed at both ends using commercially available rubber stoppers. The quartz tube was flushed with Ar/H_2 O_2 gettering gas. As shown in the Figure 9, the DNS test specimens were placed in the quartz tube sandwiched between two quartz rods of 0.25-in. diam. Quartz was the preferred material since it does not heat inductively. The induction coil itself was wound around the quartz tube as shown in Figure 9a.

The thermal conductivity specimens, due to their larger size (2.5 in. x 2.5 in.), required a rectangular copper coil of 3 in. x 0.75 in. Due to difficulty in obtaining a quartz tube of rectangular or elliptical shape, it was decided to place the entire assembly of the thermal conductivity braze specimen and the coil inside a larger quartz tube as shown in Figure 9b. A similar Ameritherm Model SP 25 induction machine was used for this specimen. Subsequent to discussions with various braze alloy manufacturing companies, brazing conditions and a tooling design to hold the fixture were finalized. The thermal conductivity specimen was placed in the fixture shown in Figure 9c to eliminate any contact between the C/C composite and the coil. The fixture also had provisions to apply pressure on the C/C adherents while brazing. The tooling material was made from machinable "lava rock." The tooling design allowed for control of pressure and prevented overflow of the braze material upon melting.

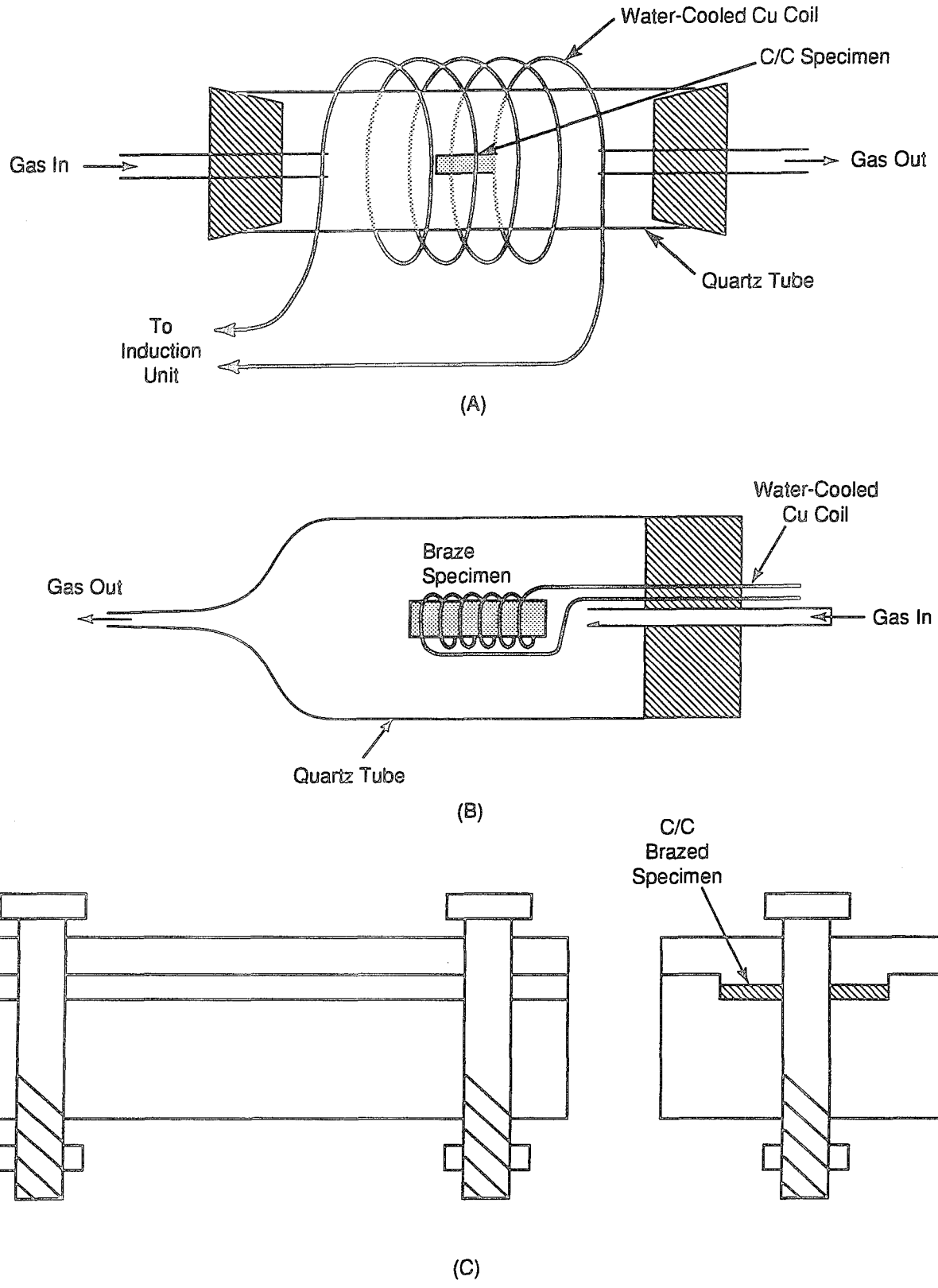


Figure 9. Apparatus used for brazing of (a) DNS specimen, (b) thermal conductivity specimen, and (c) ceramic fixture used to hold the thermal conductivity specimen for brazing.

4.4 TASK 4 - OUT-OF-FURNACE INDUCTION BRAZING TRIALS

Prior to actual fabrication of the brazed specimen for evaluation, the effect of fiber orientation and the pin material on the induction heating of the C/C composites was evaluated. This was done by simply measuring the potential (voltage) required to heat 0.125 in. x 0.5 in. x 2.75 in. coupons to the brazing temperature of 1750°F. Two different prepreg layups (0/90 and 0/±45/ 90) with Mo P100 and SS z-pin materials were studied. Power levels of about 120 to 125 V were required to heat the unreinforced 0/90 C/C coupons to 1750°F. Reinforcing the composites with the z-direction pins made no noticeable difference in the voltage required to heat the composites. The pins themselves heated up rather rapidly (they glowed at a much lower power level). However, due to their small volume fraction in the composite (about 1.5 percent), the pins did not affect the heating rate of the entire specimen. Use of a quasi-isotropic specimen (0/±45/90) required a slightly higher voltage of 130 to 135 V to heat to a brazing temperature of 1750°F. Again the use of pin materials did not alter the power requirements for heating the quasi-isotropic specimen. One factor which significantly affected the heating rate of the C/C composite was use of a susceptor such as a metal foil on the surface of the composite. Use of a 0.0025-in.-thick Ti foil reduced the potential required to heat the C/C specimen by over 40 percent to only 70 V. Titanium foil also served as an O_2 getter, as will be described later in this subsection.

As mentioned earlier, several new, active braze alloys have been developed for brazing of C/C composites. These braze alloys have a strong carbide former like Ti, Zr, or other transition elements. The braze used in this study, Ag-ABA (93 weight percent Ag 5.75 weight percent Cu, and 1.25 weight percent Ti), had about 1.25 weight percent Ti. The carbide-forming element in the braze reacted with the C/C composite and helped wet the alloy, as per the following reactions.

$$Ti + C = TiC$$

$$Zr + C = ZrC$$

In addition to being strong carbide formers, the active metals in the braze almost always are strong oxide formers. To avoid the oxidation of the active metal during the brazing cycle, the brazing of C/C composites is done in vacuum furnaces. The standard recommended vacuum level for brazing of C/C composites is 10^{-5} torr.

The oxidation of the braze alloy, and to a lesser extent oxidation of the C/C composite, were the major challenges in out-of-furnace brazing of C/C composites. To overcome the oxidation of the braze alloys, it was necessary to minimize the brazing cycle by faster heating of the specimen, thereby reducing the time available for oxidation. This was accomplished during this program by resorting to induction heating and by proper design of the water-cooled Cu coil. To ensure uniform and speedy heating of the entire material, it was necessary to have a coil of the same shape as the C/C material and to place the coil as close to the specimen as possible. The use of a susceptor metal foil around the C/C adherent further reduced the time required to heat the specimen.

The second and perhaps more important technique to reduce the oxidation was the use of inert gases such as He and Ar. Combined with oxidation getters, they could greatly reduce the O_2 amount in the brazing atmosphere. Selected specimens, which were brazed with pure Ar atmosphere, resulted in severe oxidation and showed no bonding between the C/C substrates. The Foster-Miller technique utilized an Ar/H_2 atmosphere to reduce the O_2 content in the atmosphere. Since H_2 has a higher affinity for O_2 than Ti, the reaction $H_2 + 1/2\ O_2 = H_2O$ proceeded faster than the reaction $Ti + O_2 = TiO_2$. For these brazing trials, an Ar-5 volume percent H_2 atmosphere was used. This minimized the oxidation of braze in the alloy. It must be

cautioned that the affinity of H_2 for O_2 was reduced by the amount of H_2O present in the gas. Thus it was important to use "dry" gas atmospheres during brazing. The O_2 gettering could be made more efficient by wrapping the C/C specimen with a foil of a metal which has an equal or higher affinity for O_2 than the active metal in the braze alloy. During this study, several specimens were brazed with a Ti foil wrapped around the C/C specimen. A listing of the various braze conditions evaluated in this study is given in Table 2. In addition to serving chemically as an O_2 getter, the Ti foil also acted as a susceptor and increased the heating rate. Due to the reduction in the time to heat, the C/C composite was exposed to an oxidizing atmosphere for an even shorter time.

After deciding upon the composition of the gases and the use of Ti foil as an additional O_2 getter, the brazing trials were performed using the apparatus described in subsection 4.3. As listed in Table 2, the major parameters in the brazing trials were the number of braze foils utilized (1 or 2) and the effect of the Ti foil as an O_2 getter. Initially, the adherents were lightly polished and placed in the quartz tube without the braze materials and degassed at about 1800°F for about 30 sec in an Ar/H_2 atmosphere. The specimen was cooled, removed from the quartz tube, cleaned with acetone, and assembled for brazing. The specimen was heated to the recommended brazing temperature (925°C, 1697°F) and held there for about 5 sec. Often times, due to nonuniform heating of the specimen, slightly higher temperatures were utilized for brazing (about 1750°F). A typical time-temperature profile for the brazing run is plotted in Figure 10. The specimen was heated and cooled extremely rapidly at higher temperatures to minimize the oxidation. A successful brazing operation could easily be determined by observing the liquid braze ooze from the edges of the specimen. The entire cycle of degassing and brazing took about 20 min.

In addition to the brazed specimens fabricated for the DNS tabulated evaluation, several unbrazed specimens with only z-pins were fabricated for comparison. Also, several 2.5 in.

Table 2. Summary of the braze conditions for fabrication of DNS test specimen.

Specimen	Pin Material	No. of Ag-ABA Braze Foils	Use of Ti O_2 Gettering Foil
C11	None	1	Yes
C22	None	2	Yes
C32	None	2	No
C41	None	1	No
C51	None	1	Yes
C61*	None	1	No
C71*	None	1	No
M11	Mo	1	Yes
M21	Mo	1	No
M31	Mo	1	No
G11	P100/Epoxy	1	No
G21	P100/Epoxy	1	Yes
G42	P100/Epoxy	2	Yes
S11	SS	1	No
S21	SS	2	No
S32	SS	1	Yes
SS1	SS	0	No
SS2	SS	0	No
SS3	SS	0	No
SS4	SS	0	No
SS5	SS	0	No
GR1	P100/Epoxy	0	No
GR2	P100/Epoxy	0	No
GR3	P100/Epoxy	0	No
GR4	P100/Epoxy	0	No
GR5	P100/Epoxy	0	No
MO1	Mo	0	No
MO2	Mo	0	No
MO3	Mo	0	No
MO4	Mo	0	No

*These specimens were brazed in a vacuum furnace.

x 2.5 in. brazed and unbrazed specimens were fabricated to evaluate the effect of brazing and z-direction pins on the transverse thermal conductivity of the C/C composites. In addition, control specimens were brazed in a vacuum furnace for comparative studies.

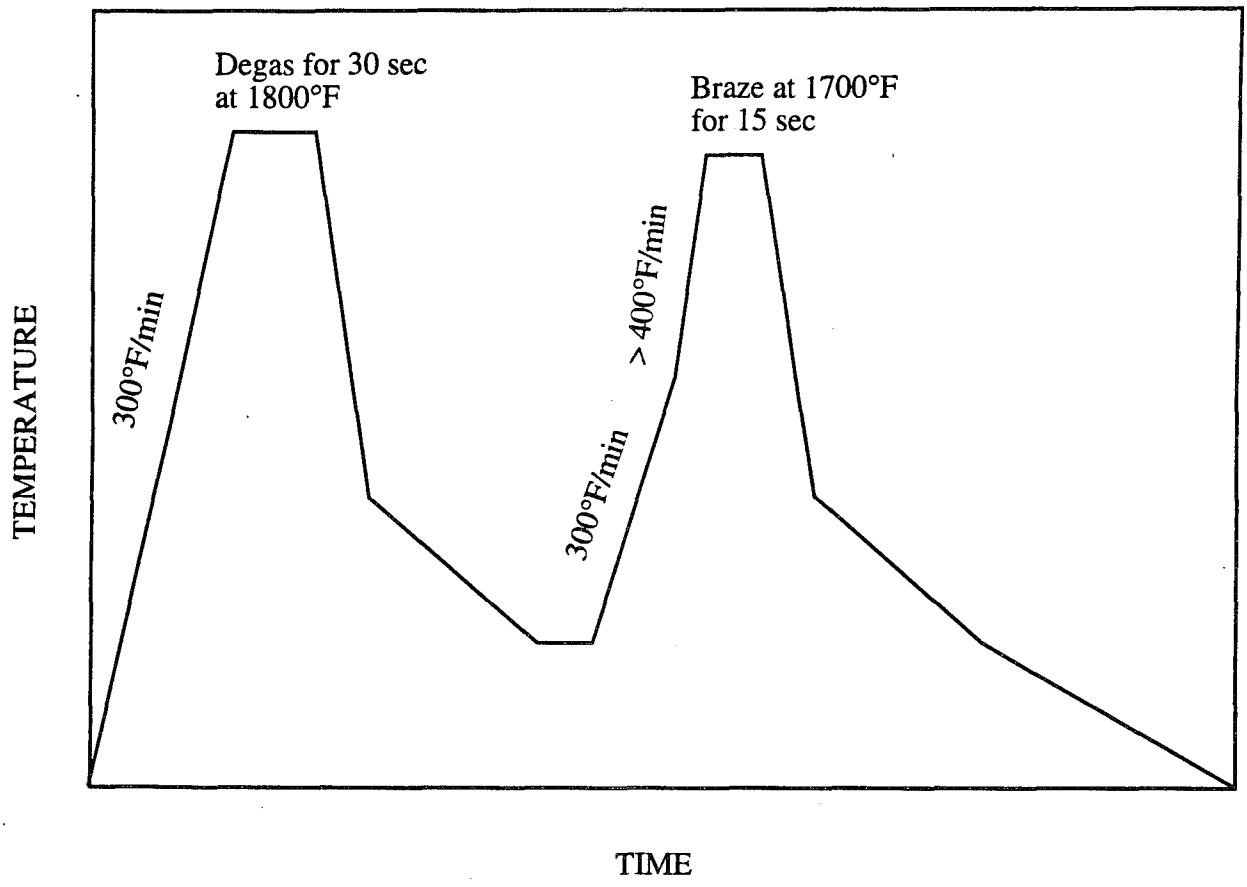


Figure 10. Typical out-of-furnace brazing time-temperature profile.

4.5 TASK 5 - JOINT ANALYSIS AND TEST

The out-of-furnace brazed joints were analyzed for double notch shear strength and through thickness thermal conductivity. In addition, selected specimens were cross-sectioned and polished to evaluate the braze-C/C composite and braze/pin interfaces. The failure surfaces from the DNS testing were macroscopically examined. The thermal conductivity measurements were conducted at Foster-Miller using an "Axial Copper Rod" apparatus. The details of the thermal conductivity measurement technique are described in subsection 4.5.4. Control and brazed C/C specimens were evaluated for thermal conductivity.

4.5.1 Double Notch Shear Strengths

The C/C panels were machined into 1.0 in. x 2.5 in. specimens. These specimens were brazed using the out-of-furnace induction brazing technology. The brazed coupons were machined to the final specimen geometry illustrated in Figure 11. This procedure was adopted to assure parallel edges for testing. The test specimens were placed in a fixture and tested per modified ASTM D3846-79. This test technique was adopted by General Dynamics, Fort Worth, TX as a standard method to test brazed C/C composites for the National Aerospace Plane (NASP) program. The bolts were torqued to a load of 18 lb/in.^{2}. Adequate care was taken to ensure that the specimen edges were orthogonal to the applied load. The load was applied using a United (Huntington Beach, CA) testing machine at a cross-head speed of 0.02 in./sec. The United testing machine was equipped with a Sintech, (Huntington Beach, CA) computerized data acquisition system.

The brazed and control specimens tested for double notch shear strength are listed in Table 2. The varied brazing parameters were braze thickness, the pin material in the C/C adherent, brazing atmosphere (Ar and Ar/H_2), and use of Ti foil as an O_2 getter. As mentioned in the previous subsection 4.4, a few specimens were tested for DNS strength without the braze material to estimate the maximum DNS strengths possible via use of brazing as a joining process.

Figure 12 shows the DNS strengths of the unbrazed C/C specimen with 1.5 areal percent transverse P100, SS and Mo pins. Reinforcing the C/C composite with z-direction Mo pins resulted in the maximum ILSS strength of 1960.1 psi as opposed to 1538.2 psi and 1161.9 psi for SS and P100 graphite reinforcements. The pins helped to improve the ILSS of the C/C adherent and prevented or delayed the failure of the braze joint. The ILSS, as measured by DNS, estimated the maximum strengths which could be obtained from the brazed joints if the failure was induced in the braze layer.

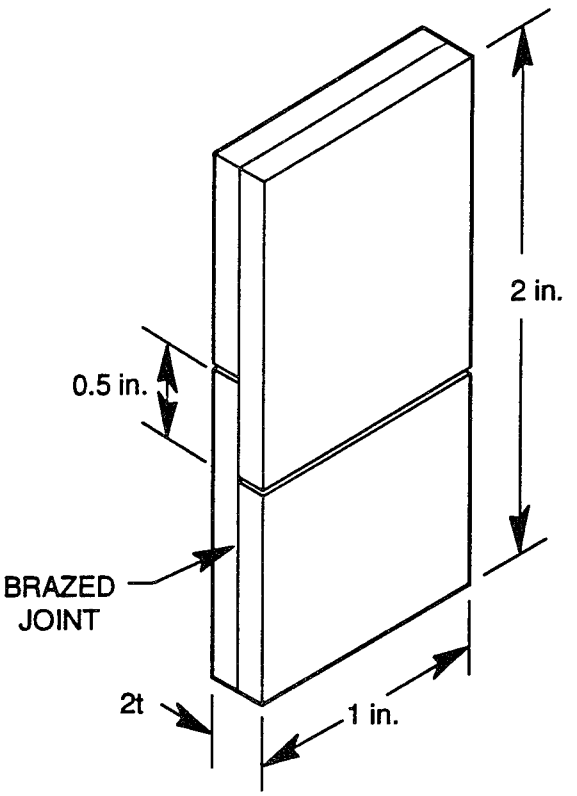


Figure 11. Double notch shear specimen geometry.

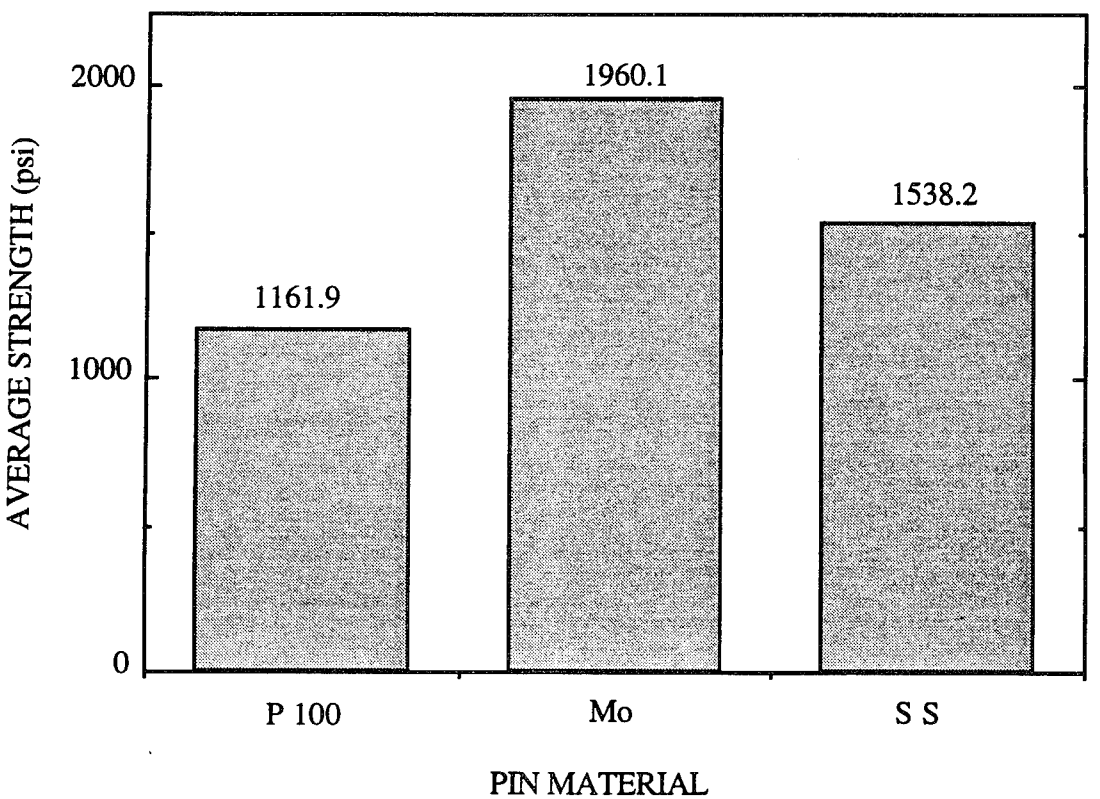


Figure 12. DNS strength of the unbrazed C/C composites with various z-direction pins.

The DNS strength results of the brazed joints are shown in Figure 13. This figure shows results of brazed C/C composites with various conditions, i.e., results of the specimens with or without the Ti O_2 getter and with one or two Ag-ABA braze foils. The results of the various brazed specimens with different conditions are discussed in subsection 4.5.1.1.

4.5.1.1 Control Specimens. Two sets of control specimens were used in this study. The first set was specimens without z-direction pins and brazed in a vacuum furnace as per Wesgo's (the manufacturer of the braze foil) recommended heating and vacuum specifications. The second set of control specimens was specimens without z-direction pins brazed by the out-of-furnace induction heating technology. The two vacuum-furnace-brazed specimens showed strengths of 718 and 1154 psi. As a comparison, the out-of-furnace brazed control specimens showed strengths as high as 1179.5 psi. This study conclusively demonstrated that out-of-furnace brazing was a technically viable process for joining of C/C composites using active braze alloys.

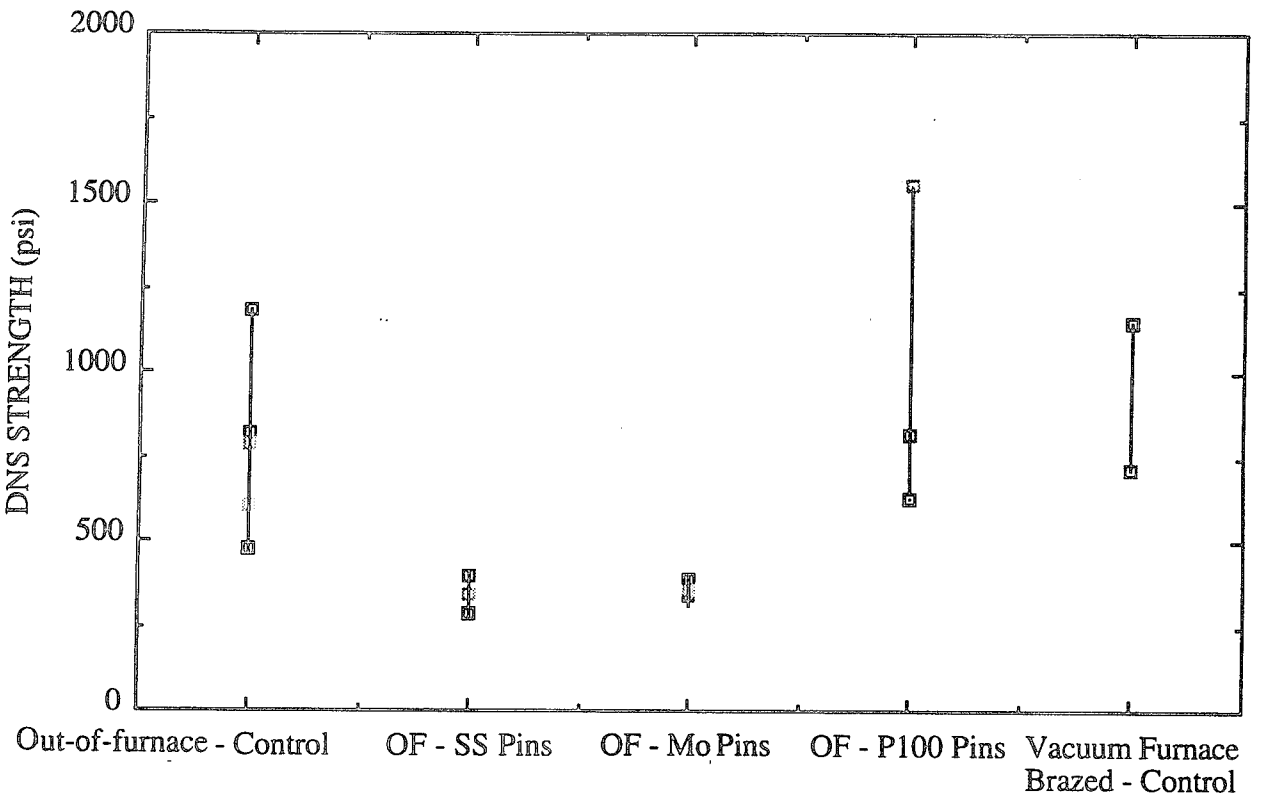


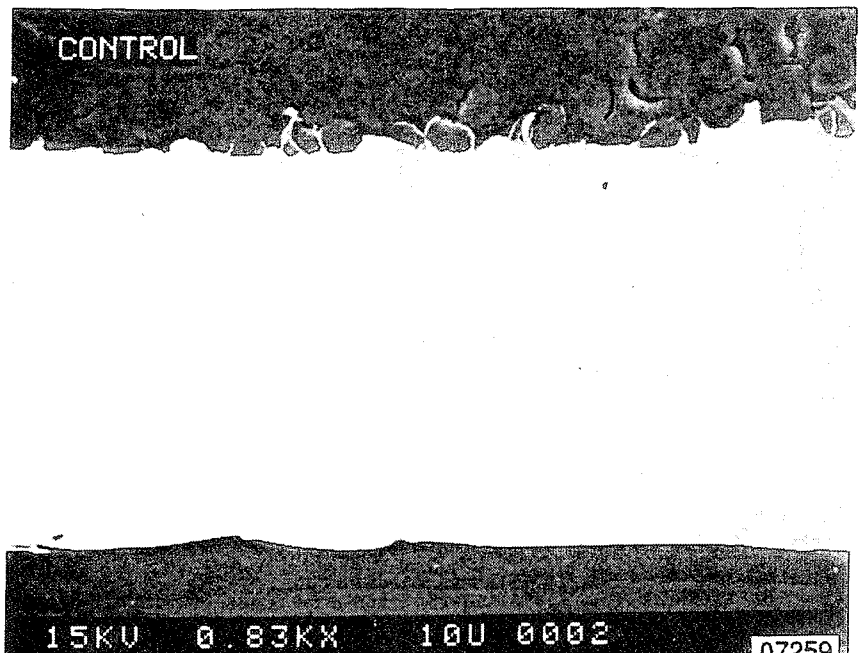
Figure 13. DNS strength results for brazed C/C joints.

the out-of-furnace brazing technique. The interface showed excellent wetting of the C/C composite by the Ag braze. At several locations, the molten braze penetrated the space between the C fibers and the C matrix. There was no degradation of the interface by formation of any reaction products. Figure 15 shows the EDAX maps of the braze layer near the interface between the braze and the C/C composite. The EDAX map shows the presence of Ag, Cu, Ti, and C. The presence of C and Ti probably indicated formation of a TiC interlayer between the C and the braze.

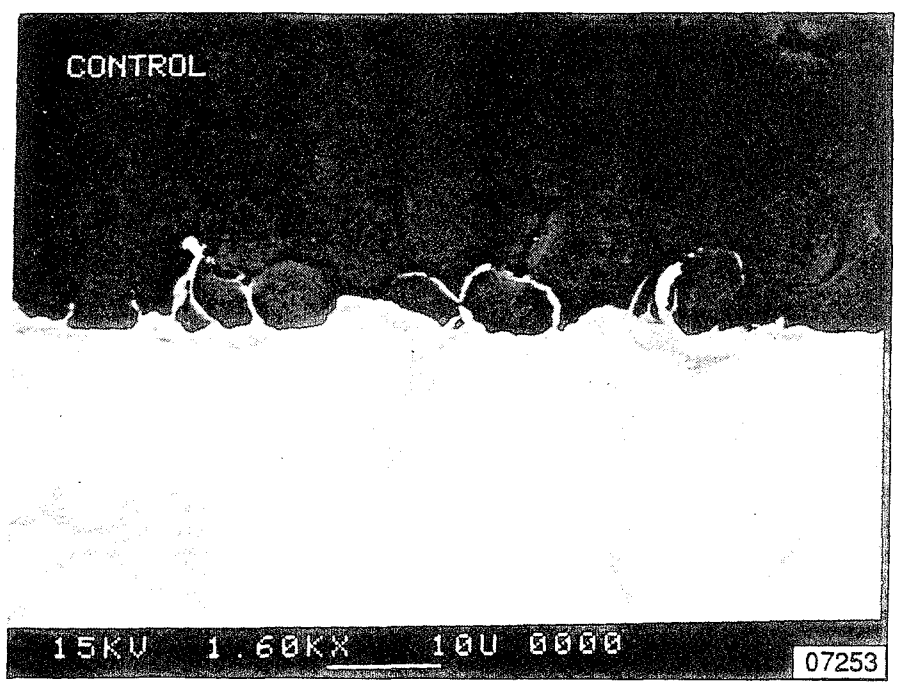
The fracture surfaces of the out-of-furnace brazed specimen are seen in Figure 16. The specimen failures are in the C/C adherent, similar to failures normally seen in vacuum brazed 2-D C/C composites.

4.5.1.2 P100 Pins. The P100 transversely reinforced pins showed strengths ranging from 630 630 psi to 1554 psi. The P100 reinforced specimen showed the highest strengths of all the brazed specimens, higher even than those of the control specimen. This indicated that the z-pins are helpful in increasing the braze joint strength of the composite. The highest strength obtained from the P100 reinforced specimen was 34.6 percent greater than the highest strength of the vacuum-furnace-brazed specimen. Like the control specimen, the P100 reinforced specimen also showed failures in the C/C adherent and not the braze material (Figure 17).

4.5.1.3 Stainless Steel Pins. The DNS strength of the C/C composites transversely reinforced with SS pins showed strengths ranging from 286 psi to 399.4 psi. The lower strength of the SS-pin-reinforced composites was due to oxidation of the braze as seen from the failure surface in Figure 18. The surfaces were heavily oxidized and the failure was always in the braze layer rather than the C/C adherent. The increased oxidation of the C/C adherents was due to entrapment of O_2 in the spaces between the pins and C/C composite and also due to the conduit



A)



B)

Figure 14. Scanning electron micrographs showing good adhesion between the braze layer and the C/C composite joined using out-of-furnace brazing.

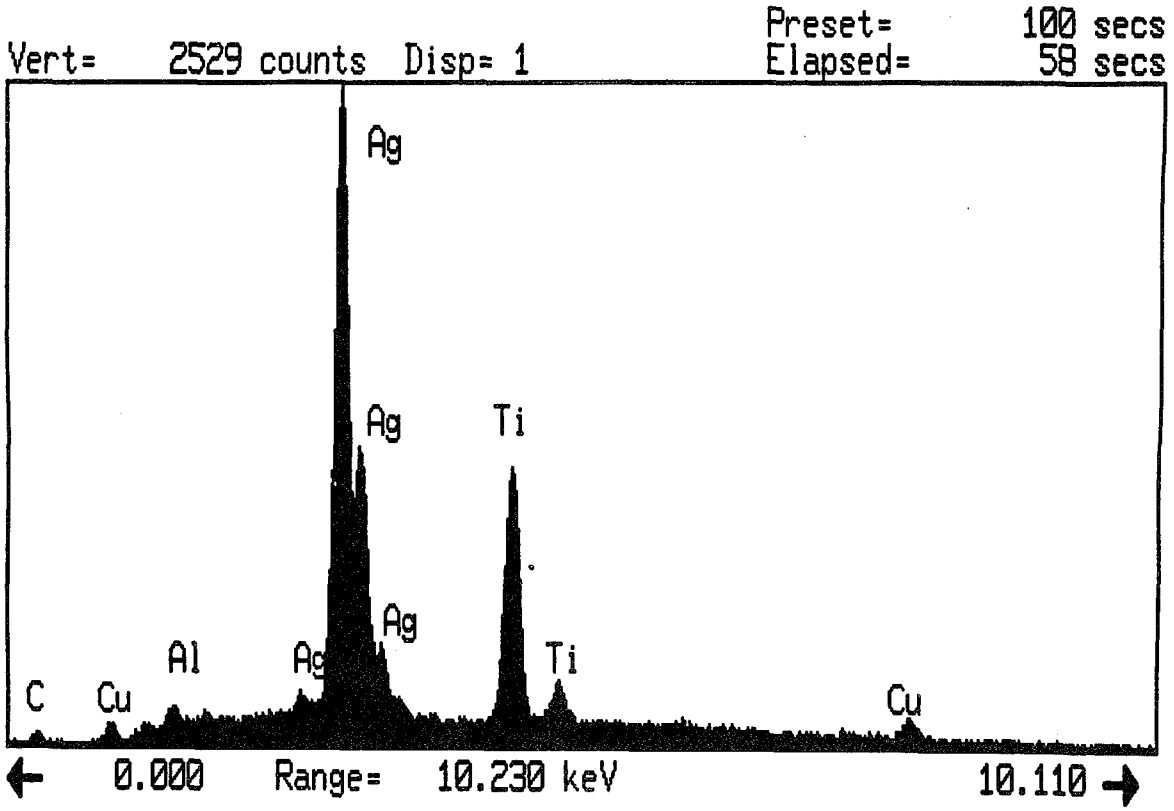


Figure 15. EDAX profile near the braze layer-C/C composite interface showing presence of Ti and C.

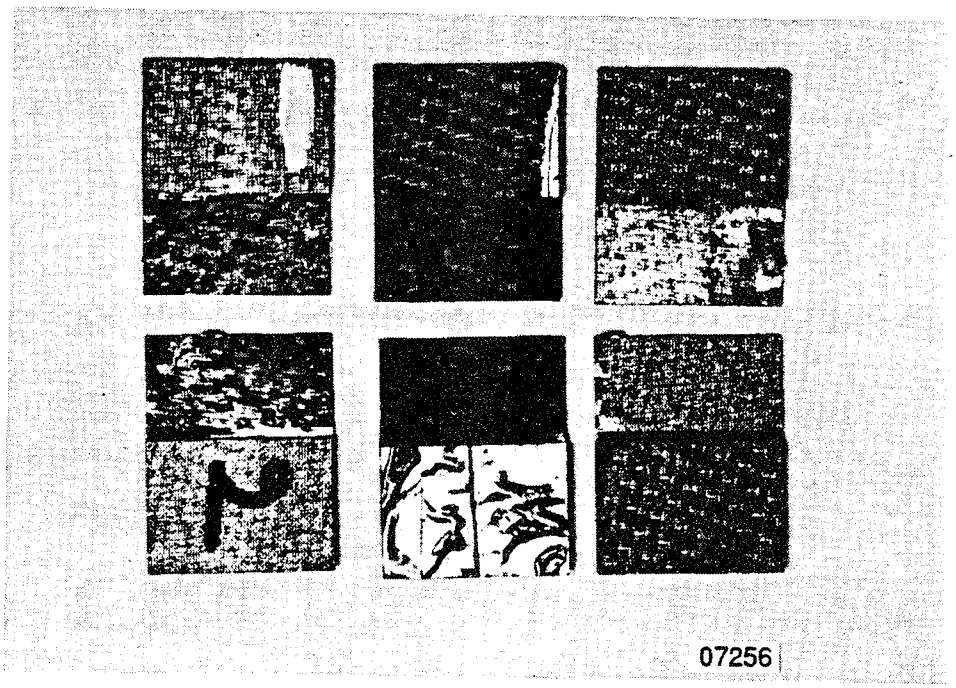


Figure 16. Failure of control C/C out-of-furnace brazed joints showing failure in the C/C adherents.

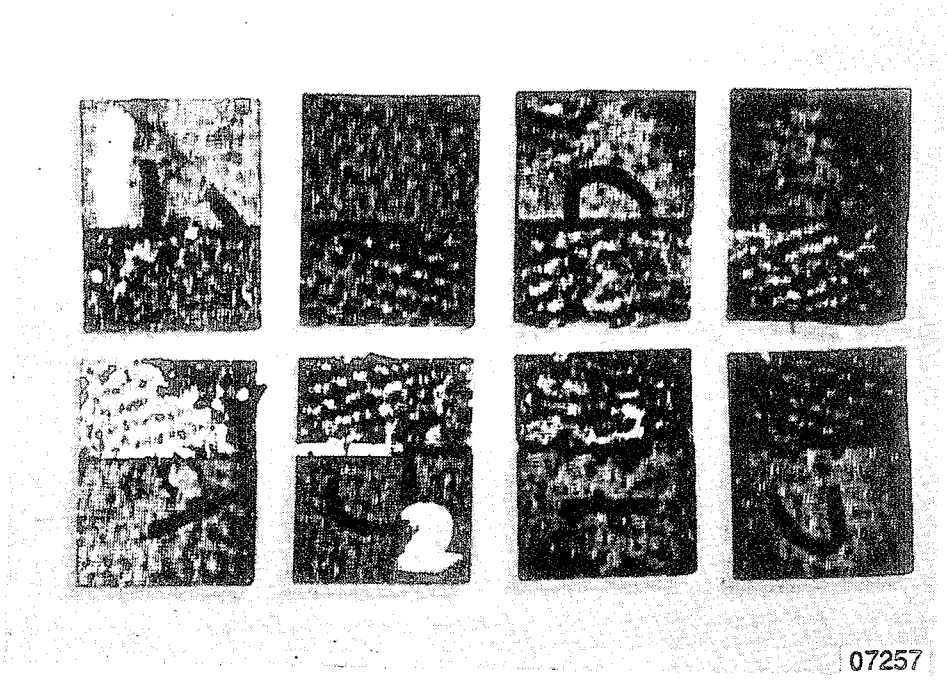


Figure 17. Failure of P100 z-direction reinforced C/C out-of-furnace brazed joints showing failure in the C/C adherents.

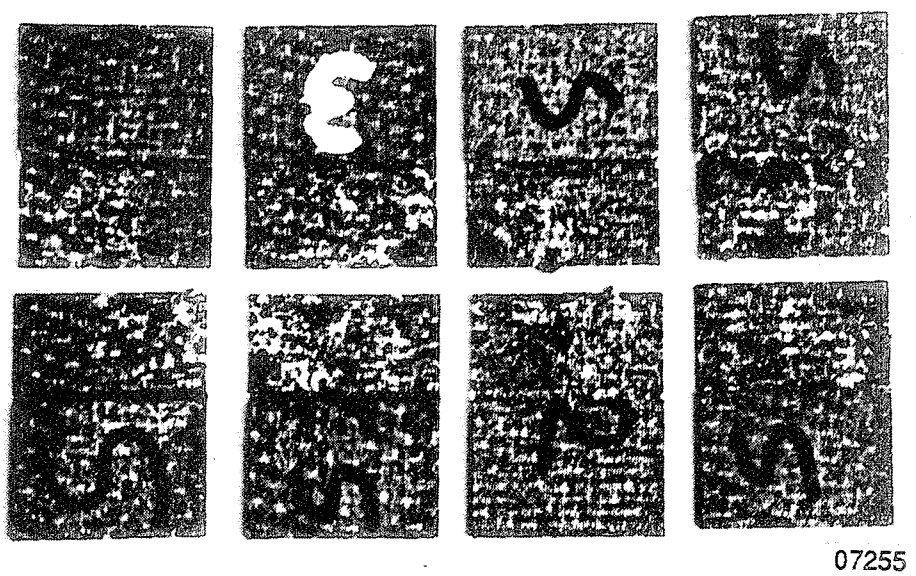


Figure 18. Failure of SS z-direction reinforced C/C out-of-furnace brazed joints showing failure in the braze layer. Note the extensive oxidation of the braze.

provided by the SS pins for the O_2 to diffuse through the C/C adherent to the braze. The gaps between the pins and C/C were because of their differential CTEs.

4.5.1.4 Molybdenum Pins. The DNS test results of the C/C composites transversely reinforced with Mo pins showed strengths ranging from 340 psi to 385 psi. Similarly to the SS-pin-reinforced specimen, the lower strengths of the Mo-pin-reinforced composites were due to oxidation of the braze similar to that in the SS-reinforced C/C adherents as seen in Figure 18. The surfaces were heavily oxidized and the failure was always in the C/C-braze interface rather than the C/C adherent as in the control and P100 reinforced specimens. The increased oxidation of the C/C adherents was again due to the conduit provided by the Mo pin (similar to the SS-pin-reinforced specimen) for the O_2 to diffuse through the C/C adherent to the braze. Even though the specimen showed extensive oxidation of the braze materials, good wetting of the C/C composite and the Mo pin by the Ag-ABA was seen (Figure 19). No reaction interlayer was seen between the pin material and the braze. This was expected because Mo and Ag/Cu alloys do not form any solid solution.

Summarizing, preventing oxidation is the key to successful brazing of the C/C composites out-of-furnace. The measures taken to prevent oxidation of the braze in this study were very successful in control specimens and composites reinforced transversely with P100 fiber. The P100 fiber reinforced composites showed strengths higher than the vacuum brazed specimens, thus proving the viability of the use of z-pins to obtain higher strength. Due to oxidation of the specimens, the SS and Mo transversely reinforced specimens showed lower strengths.

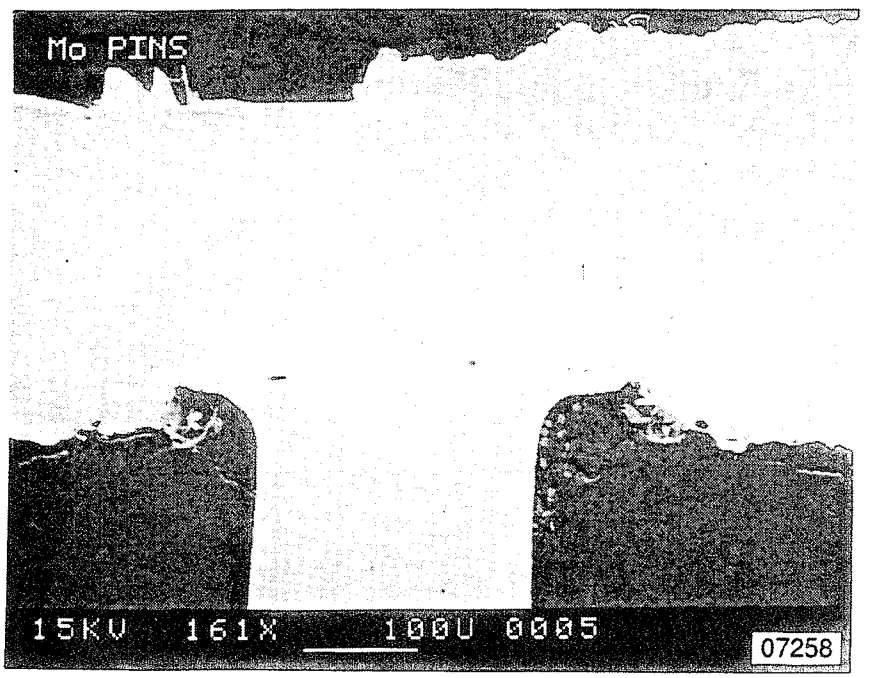


Figure 19. Scanning electron micrographs showing good adhesion between the braze layer and the C/C composite and transverse Mo pins joined using out-of-furnace brazing.

4.5.2 Effect of Titanium Foil as an O_2 Getter

The effect of using Ti foil as an O_2 getter on the DNS strengths of the brazed joints is seen in Figure 20. The figure shows the strengths of the joints in the specimens with and without the foil. In both the control specimen and the z-fiber reinforced specimen, the use of foil resulted in stronger bonds. The increased strength was very significant for the unreinforced control specimen and the P100, z-direction reinforced specimen. Although not as effective in minimizing oxidation in the SS and Mo reinforced C/C adherents, increased DNS strengths were observed in these specimens.

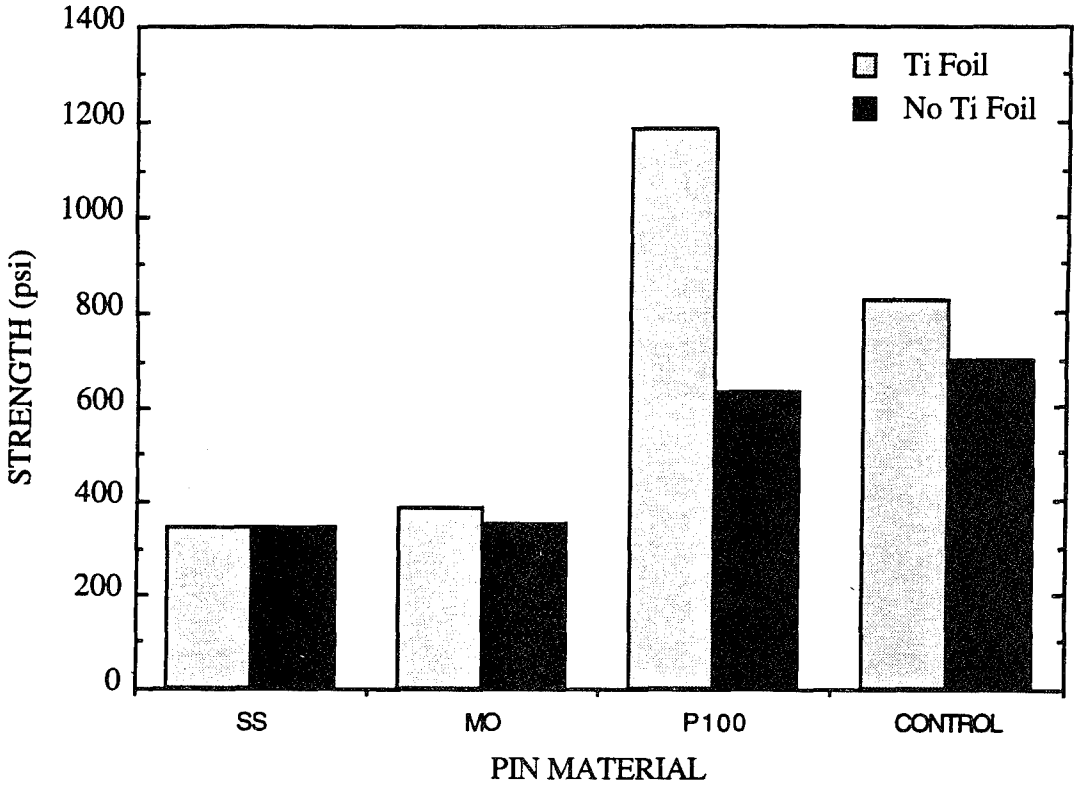


Figure 20. Effect of use of Ti foil wrap as an O_2 getter while brazing on the DNS strength of the joint for control and transversely reinforced C/C composites

4.5.3 Effect of Braze Thickness

The braze layer thickness affected the magnitude of the processing thermal stresses in the C/C adherents and the braze layer. The thermal stresses in the braze material and the C/C composite due to difference in their CTEs are given by:

$$\sigma_{thec/c} = F_{the} / A_{C/C}$$

where

$$F_{the} = \Delta e \{ E_{br}\, t / (1 + t\, E_{br} / E_{c/c}) \}$$

$$\Delta e = (\alpha_{br} - \alpha_{c/c})\,\Delta T$$

where

$\sigma_{thec/c}$ = Thermal stress in the C/C composite

F_{the} = Thermal force in the C/C composite

$A_{C/C}$ = Cross-sectional area of the C/C composite

Δe = Difference in contraction of C/C and braze during joining process per unit length

E_{br} = Modulus of the braze material

t = Ratio of thickness of braze layer to that of C/C composite

$E_{c/c}$ = Modulus of the C/C composite material

α_{br} ,$\alpha_{c/c=}$ = CTEs of the braze and the C/C composite materials

ΔT = Difference between brazing temperature and use temperature

Figure 21 plots the thermal shear stress at the braze-C/C interface versus "t", the ratio of braze layer thickness to the C/C composite thickness for a Ag-ABA braze used to join C/C composite. Increasing the braze thickness increased the thermal stresses in the composite and, thus was harmful to the properties of the composite. The normal stress in the C/C adherent was compressive and tensile in the braze layer. In the DNS test used to evaluate the C/C adherents, the compressive stresses were applied. Therefore if the failure is anticipated in the C/C in the DNS testing, a thinner braze layer may be preferable. Conversely, if failure is anticipated in the braze layer, a thicker braze layer is preferable.

The effect of braze thickness was evaluated by comparing the DNS strengths of the specimens fabricated with either one (0.002-in.-thick) or two (0.004-in.-thick) braze foils. Figure 22 shows the difference in the DNS strengths of the brazed specimen control and z-reinforced specimens

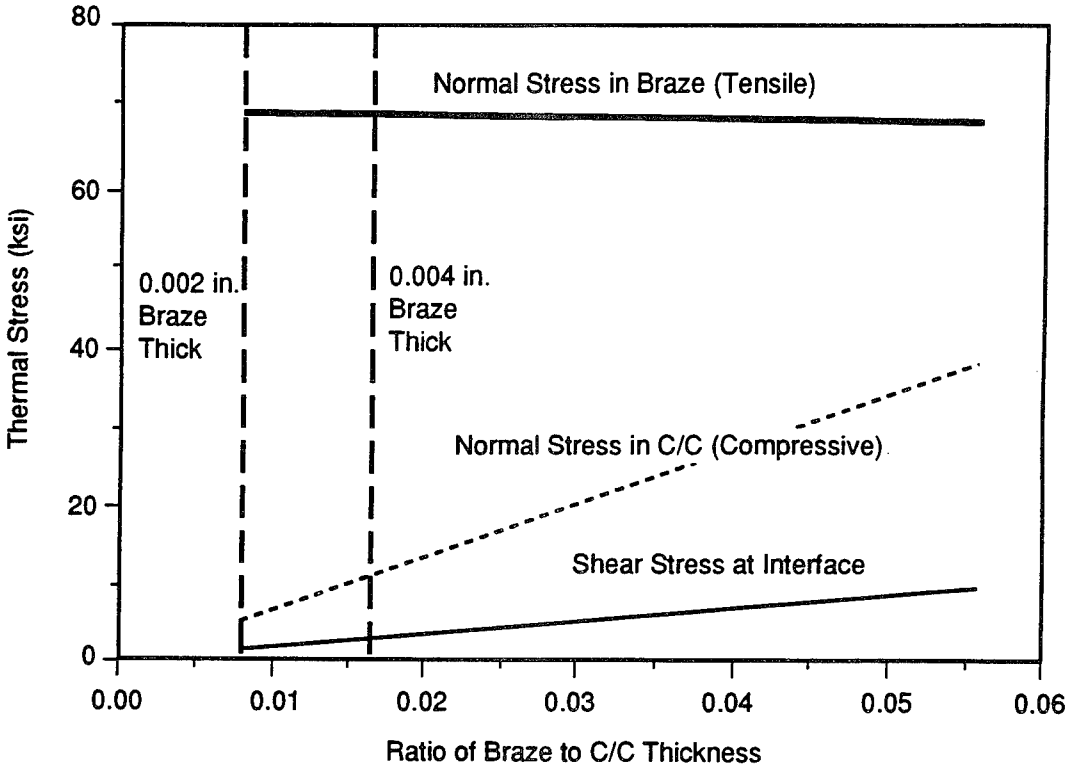


Figure 21. Predicted thermal stresses in the braze and the C/C composite.

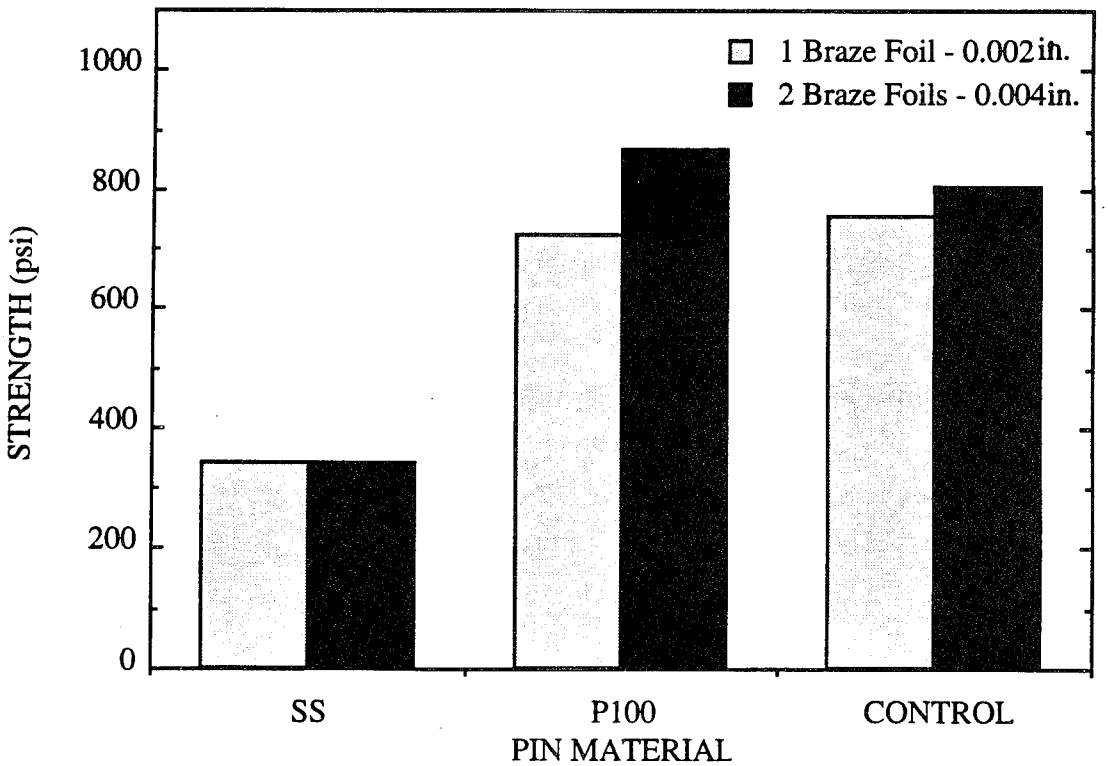


Figure 22. Effect of braze layer thickness on the DNS strength of the joint for control and transversely reinforced C/C composites.

joined with either one or two braze foils. Increases in the joint strengths were observed in the specimen brazed with two foils. However, these increases were not significant enough to make definite conclusions about the effect of braze thickness on the joint strength.

4.5.4 Through Thickness Thermal Conductivity Measurements

Carbon/Carbon composites are increasingly considered for thermal management applications due to their high specific thermal conductivity. These applications include thermal planes, radiators, and heat sinks for electronic packaging applications, especially in avionics. Efforts are being made to further increase the thermal conductivity of the C/C composites by using high thermal conductivity fibers and matrices. In this study use of transverse z-direction fibers was evaluated for increased thermal conductivity in the z-direction.

Measurements of through thickness thermal conductivity of the specimens were conducted in a vacuum chamber to eliminate the convective heat transfer from the specimen. The temperature of the chamber was controlled via a water cooled metallic jar. This also minimized the radiant heat losses from the specimens. The chamber was equipped with a water cooled sink and full instrumentation and data acquisition. A schematic of the thermal conductivity measurement apparatus is shown in Figure 23.

The test specimen was placed between two Cu rods calibrated to measure the heat flux passing through the rods. The accuracy of the instrument was further increased by using a flux meter with a narrower bottom, thereby increasing the temperature differential across the two points as indicated in Figure 23. The control temperature of the apparatus T_0 was maintained at 105°C through a feedback control circuit. Temperatures T1 and T2 in the upper Cu rod were used to extrapolate the temperature to the upper surface of the specimen. Similarly, temperatures T3 and T4 were measured in the bottom Cu rod to extrapolate the temperature to the lower surface of the

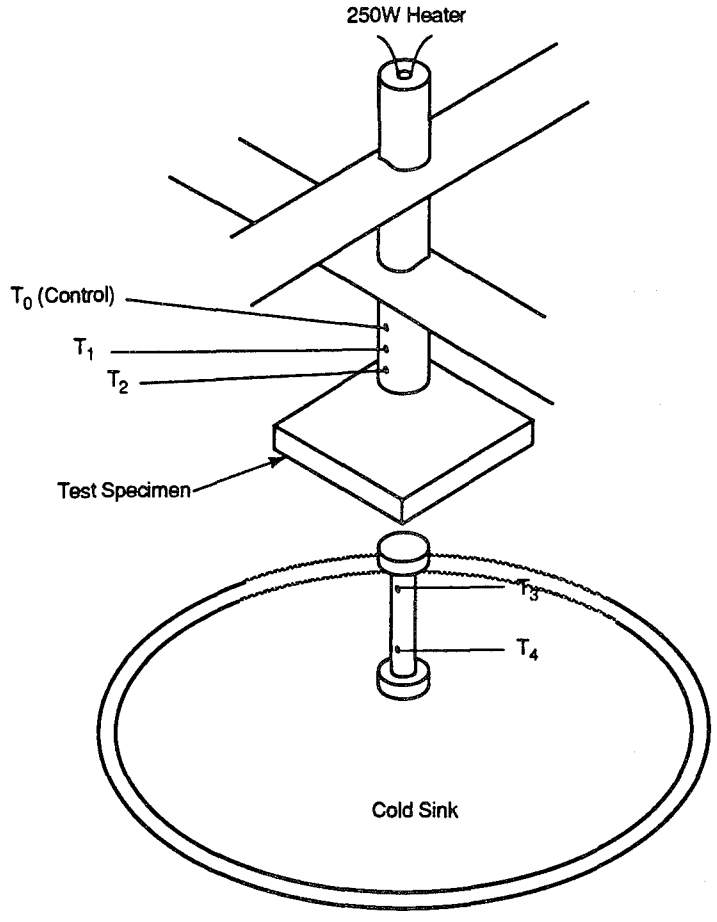


Figure 23. Schematic of the apparatus used for measuring thermal conductivity of the brazed and control C/C specimen.

specimen. Prior to testing of the samples, the Cu rods were placed in contact with the Dow 34 Heat Sink compound (thermally conductive grease, K = 0.42 W/m/K) to determine the temperature drop across the interface and to calculate the thickness of the grease. This thickness was used to calculate the temperature drop across the interface between the Cu rod and the specimen.

The temperature difference across the bottom Cu rod was determined from the embedded thermocouples. The heat flux through the bottom Cu rod was assumed to be the heat flux through the sample. In this fashion, very accurate and repeatable values for the thermal conductivity were measured.

Table 3 lists the results of the thermal conductivity evaluations for the various composites. Thermal conductivity measurements were conducted for control and z-direction reinforced brazed and unbrazed C/C composites. The thermal conductivity of the control, unbrazed specimen was 4.45 W/m/K. The introduction of 1.5 areal percent transverse fibers doubled the transverse thermal conductivity. The thermal conductivity increased to 8.87 W/m/K and 9.31 W/m/K respectively, using Mo and P100 C transverse fibers. The brazed specimens did not show any significant decrease in thermal conductivity due to formation of any low conductivity interfacial Ti carbide. It was postulated that due to low Ti concentration in the braze, the resultant interface was very thin and thermally transparent. The thermal conductivity of the control brazed specimen was 6.41 W/m/K, an increase of almost 44 percent over the unbrazed specimen. The thermal conductivity of the brazed z-direction reinforced C/C adherents showed thermal conductivities of 8.61 and 8.17 W/m/K for Mo and P100 reinforcements, a slight decrease compared to the unbrazed specimen.

Table 3. Results of thermal conductivity evaluations.

Z-Fibers	T_0 (°C)	T_1 (°C)	T_2 (°C)	T_3 (°C)	T_4 (°C)	Conductivity (W/m/K)	% Increase
None (Unbrazed)	105	103.3	98.2	50.5	27.9	4.45	-
Mo (Unbrazed)	105	103.4	98.4	53.7	32.1	8.87	99.3
P100 (Unbrazed)	105	103.9	97.9	52.7	30.3	9.31	106.6
None (Brazed)	105	103.8	99.1	44.4	24.2	6.41	44.0
Mo (Brazed)	105	103.2	97.9	47.7	24.7	8.61	93.5
P100 (Brazed)	105	103.2	97.9	47.9	26.9	8.17	83.6

5.0 RESULTS

The goal of the Phase I program was to develop a rapid, controlled atmosphere, out-of-furnace brazing technology for joining of C/C adherents using active braze alloys. This was combined with the Foster-Miller z-direction process to improve the joint strength of the brazed 2-D C/C composites. The anticipated applications of the brazed C/C composites were in the areas of thermal management requiring high thermal conductivity. Hence it was decided to use a high thermal conductivity Ag-ABA, an Ag-Cu-Ti active braze alloy, and P-100 z-direction pin material. The use of z-direction pins was expected to provide higher strength in the parent C/C composite and thus increase the joint strength, since failure of the brazed 2-D C/C composites was often in the C/C adherent material rather than the braze joint. Due to use of an active braze alloy, special cleaning and fluxing treatments for the adherents were not required.

Due to the high affinity of Ti on the braze alloy for O_2, and low oxidation resistance of the C/C composites, special care was taken in fabricating the brazing apparatus to minimize O_2. This was done by using O_2 getters and exposing the C/C to high temperatures for very short times and thereby further reducing oxidation. Commercially available quartz tubes were utilized as chambers to conduct brazing trials. Oxidation was minimized by using a reducing Ar/H_2 atmosphere and a Ti foil to wrap around the C/C adherents as O_2 getters. The brazing cycle times were reduced by using induction coils to heat the C/C specimen. Use of Ti foils as O_2 getters further reduced the brazing cycles as Ti inductively coupled with the coil and served as a susceptor. The entire cycle of degassing and brazing took less than 20 min.

Phase I DNS test results conclusively proved the viability of a controlled atmosphere, out-of-furnace brazing process for joining of C/C composites. There was a variation in the strengths of the brazed joints, both with and without z-direction reinforcements. The strengths of the control specimens without z-direction pins were similar for both vacuum furnace and out-of-furnace

brazed joints. Introduction of the z-direction P100 C pins resulted in strengths which were over 44 percent greater than the control specimen strengths. In some cases, especially with SS and Mo reinforcements, the joint strength was reduced. This was a direct result of braze alloy oxidation. Reinforcement with the metallic z-pins provided a transverse path for the "ungettered" and trapped O_2 to reach the braze and cause oxidation. The oxidation in these specimens can perhaps be reduced by having only partial z-direction reinforcement rather than through the entire thickness of the specimen.

The use of Ti foil as the O_2 getter was very effective. A comparison of the strengths of the brazed specimens consistently showed higher strengths for C/C adherents brazed with the Ti foil. A Ti wool or powder paste as an O_2 getter was recommended. Due to higher surface areas available, these may be more reactive and serve more efficiently as O_2 getters. Unlike the use of Ti foil as an O_2 getter, conclusive deductions could not be made about the effect of braze layer thickness. Although the strengths obtained with thicker braze layers were higher than those with lower thicknesses, the differences were not statistically significant and were less than the scatter in the experimental data. Further studies on the effect of braze thickness are required to conclusively determine its effect on braze joint strength.

The thermal conductivities of the brazed specimens do not show significant change from those of the parent material. Use of z-direction pins improved the thermal conductivity over the unreinforced pins by over 100 percent, indicating that in addition to increasing the strength of the parent C/C composites, the pins can also be useful in thermal management applications in avionics.

6.0 CONCLUSIONS

The out-of-furnace joining method of Phase I demonstrated a process to produce C/C joints stronger than those obtained by conventional vacuum furnace brazing. This technique relied on minimizing the oxidation of the braze by using a reducing Ar/H_2 atmosphere and an O_2 gettering Ti foil. The process reduced the brazing cycles for C/C adherents from several hours (8-40) to only 20 min. In addition, the process did not require use of an expensive vacuum furnace and the accompanying mechanical and diffusion pumps. Further, the z-direction transverse pins, in addition to providing stronger parent C/C material, aided in doubling the thermal conductivity of the material. Table 4 summarizes the advantages of the Foster-Miller process for brazing of C/C adherents.

Table 4. Advantages of the Foster-Miller process.

Foster-Miller Process Feature	Advantages
Induction furnace brazing	• Rapid brazing cycles (20 min) • Inexpensive equipment • On-site brazing possible - Fabrication of large structures and repair of existing structures
Use of active braze materials	• Compatible with C/C as well as other high temperature materials such as ceramics and refractory metals • Carbide formers create CTE gradients between C/C and the braze, thus mitigating the thermal stresses. • Eliminates the need to use expensive joint cleaning and fluxing processes
Z-direction reinforcement	• Increases ILT and ILS of 2-D C/C adherents • Increases the overall bond area and the strength of the brazed joints • More than double the through thickness conductivity of the C/C composites. • Can be effectively used to control the joint gap, i.e., the braze thickness

7.0 PHASE II PROGRAM PLAN AND RECOMMENDATIONS FOR SCALE-UP

The Phase II program will seek to optimize out-of-furnace brazing technology for three different applications of C/C composites with selective use of Z-fiber reinforcement to provide enhanced thermal and/or mechanical performance. The three target applications are (1) battery sleeve-to-radiator plate joints for management of battery waste heat (Ni-H and Na-S batteries) in commercial and military satellites, (2) tube-to-tube or tube-to-plate joints for a spacecraft truss (e.g., optical bench, antenna frame) and flap/heat shield structures and,
(3) W plate-to-C/C substrate for commercial X-ray targets. After selecting appropriate materials for these applications, the program will begin with a systematic development and optimization of the basic elements of out-of-furnace brazing technology including induction coil design, induction heating parameters, joint design and, perhaps the most crucial issue, O_2 gettering techniques. To guide and validate the approach, microscopy and mechanical/thermal property data will be generated. The best results will be adapted to representative subscale mockups of the three target applications. A shop-floor demonstration of the battery-sleeve application is planned at MMAG.

Often during the course of manufacturing, parts already joined must be disassembled on the shop floor. During the course of the Phase II program, the application of induction heating to disassemble brazed joints will be demonstrated. These specimens will be rejoined using induction brazing and evaluated for any loss of properties.

APPENDIX

Table A-1. DNS strengths of the brazed and control C/C specimens.

Specimen	Pin Material	Load	Width	Length	Strength (psi)
C11	None	237.5	0.996	0.501	475.95
C22	None	410.3	0.996	0.502	820.61
C32	None	392	0.998	0.499	787.14
C41	None	303.7	0.999	0.501	606.79
C51	None	591.5	0.999	0.502	1179.46
C61*	None	579.5	1	0.502	1154.38
C71*	None	356.5	0.997	0.498	718.01
M11	Mo	192.9	0.998	0.502	385.03
M21	Mo	169	0.998	0.498	340.03
M31	Mo	180	0.996	0.501	360.72
G11	P100/Epoxy	315	0.999	0.5	630.63
G21	P100/Epoxy	404.5	0.998	0.498	813.87
G42	P100/Epoxy	775	0.999	0.499	1554.66
S11	SS	143.4	0.996	0.502	286.80
S21	SS	170.6	0.996	0.502	341.20
S32	SS	199.9	0.997	0.502	399.40
SS1	SS	258.4	1.002	0.501	514.73
SS2	SS	984.8	1.003	0.504	1948.12
SS3	SS	997.8	1.007	0.504	1965.99
SS4	SS	999.8	1.006	0.51	1948.69
SS5	SS	661.9	1.002	0.503	1313.27
GR1	P100/Epoxy	474.3	1.002	0.499	948.60
GR2	P100/Epoxy	569.2	0.999	0.498	1144.11
GR3	P100/Epoxy	657.3	0.998	0.499	1319.87
GR4	P100/Epoxy	656.6	0.999	0.5	1314.51
GR5	P100/Epoxy	545.6	1.008	0.5	1082.53
MO1	Mo	774.5	0.998	0.506	1533.69
MO2	Mo	1220	1	0.507	2406.31
MO3	Mo	1166.9	1.004	0.495	2347.98
MO4	Mo	774.5	1.008	0.495	1552.22

*These specimens were brazed in a vacuum furnace

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