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Examination of Superplastic Forming Combined with Diffusion Bonding for Titanium: Perspective from Experience

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Superplastic forming (SPF) combined with diffusion bonding (DB) has been used successfully for the fabrication of titanium aerospace hardware. Many of these applications have been for military aircraft, whereby a complex built-up structure has been replaced with monolithic parts. Several methods for applying the two- and four-sheet titanium SPF/DB processes have been devised, including the welding of sheets prior to forming and the use of silk-screened stop-off (yttria) to prevent bonding where it is undesirable. Very little progress has been made in the past few years toward understanding and modeling the SPF/DB process using constitutive equations and data by laboratory testing. Concerns that engineers face in designing for fatigue life, acceptable design loads, and damage tolerance are currently being studied, but the database is very limited. This is a summary of past work found in the literature and forms the foundation for additional research.

Keywords aerospace, die forming, diffusion bonding (DB), hot forming, manufacturing technology, rapid prototyping, superplastic forming (SPF), SPF/DB, titanium, Ti-6Al-4V

1. Introduction

Diffusion bonding (DB) is a solid-state joining process between reactive metals under sufficient pressure and temperature to cause the coalescence of the contacting surfaces. It is a method used to join multiple pieces of titanium sheet metal, which can then be expanded into a compound countered shape using superplastic forming (SPF) techniques. Some of the early research into the DB phenomenon led to its initial description as a "welding" mechanism, since it could not be explained with any other scientific theory. However, since there is no melting of the materials being DB joined, the term "diffusion welding" is not valid and it is no longer used.

Diffusion-bonded joints are characterized by a nearly perfect bond line, with very little or no porosity. The grain structure of bonded pieces does not physically change, other than the sharing of grains between them at the joint. Figure 1 shows how the DB process evolves over time under sufficient temperature and pressure. For a Ti-6Al-4V sheet, a typical manufacturing cycle for DB of components occurs at 900 to 950 °C with a maximum applied force of 2 MPa for about 1.5 h.

Superplastic forming refers to a metal forming process that takes advantage of the metallurgical phenomenon of superplasticity to form complex and highly contoured sheet metal parts. Superplasticity is the ability of certain metal alloys and other materials to undergo very large plastic strains with minimal necking and small amounts of strain hardening. For the process to work properly, it is desirable to use a fine-grain material, an elevated temperature, and a constant strain rate. Figure 2 illustrates the degree to which the SPF process can influence the strain in an elevated-temperature tensile test.

Although currently limited to a handful of aluminum and titanium alloys for production use, other materials, such as CRES, ceramics, and aluminum-lithium alloys, have also been shown to exhibit superplastic behavior. Industry has taken advantage of SPF to design and build parts that would otherwise be impossible to produce due to the complexity of their shapes. The most commonly used material is Ti-6Al-4V (MIL-T-9046J AB-1). An excellent first review on this topic was presented by Hamilton (Ref 1). An alternative modeling approach by Pilling and Ridley is also presented (Ref 2). This review gives a brief discussion of diffusion bonding, along with personal observations from manufacturing monolithic structural aircraft components using SPF/DB of Ti-6Al-4V at the Boeing Company. Lastly, several development issues for future research are identified.

2. Results and Discussion

2.1 Superplasticity and Superplastic Forming

As with many technologies, SPF has its roots as a laboratory oddity. The metallurgical phenomenon of superplasticity was first discovered and reported in the U.K. by Bengough around 1912 (Ref 3). Later developments in the U.K. by Pearson (Ref 4) in 1934 were preceded by what appears to be independent laboratory studies in the Soviet Union by Bochvar (Ref 5).

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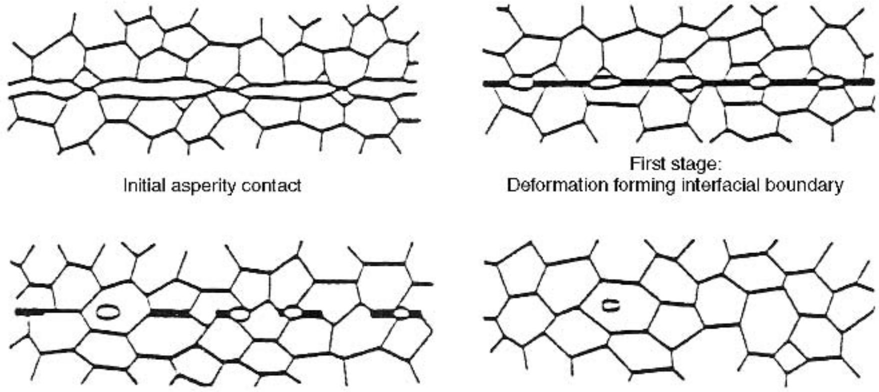


Fig. 1 Magnified idealized sketch (approximately 500× equivalent) of the DB process stages. As initial contact is made between the two sheets of Ti, at elevated temperature, the DB process begins to occur between the individual mating grains. Pressure must be applied so that the deformation of the sheets between each other causes the interfacial boundary to flatten. As time goes on and the additional contact is made, the DB bond-line grows. When DB has encompassed the entire boundary contact line, it is difficult to discern the location of the bond-line because the recrystallization of the grain structure results in random grain boundaries that cross the original interface line.

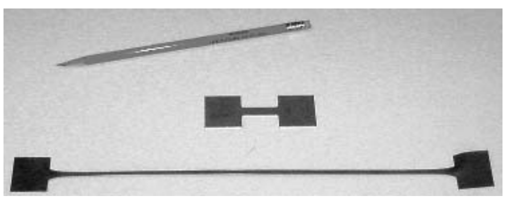


Fig. 2 Superplastic tensile coupon before testing (top) and after about 300% elongation (bottom), at which point the test had to be stopped due to the limitations of the equipment. The alloy shown is titanium SP-700 developed by NKK of Japan. This alloy exhibits excellent superplastic characteristics at 1450 °F (800 °C) and can be compared with Ti-6Al-4V that forms at 1650 °F (900 °C). The lower forming temperature of this material lengthens tool life and eliminates the need for nitric-hydrofluoric acid chemical cleaning.

Later, others such as Presnyakov (Ref 6) reported additional results around 1969. These metallurgical studies were focused on developing a simple understanding of the basic mechanics that allowed for extreme deformations of various alloys, such as cadmium-zinc, lead-tin, and bismuth-tin, which had been observed to occur under certain laboratory conditions. An investigation by Langdon (Ref 7) has shown that the term "superplasticity" originates from the Russian word "sverhplastichnost" which translates literally means "ultrahigh plasticity." Even today superplasticity seems to be a fitting description for the fundamental material science of the process.

The initial focus of the research by Rockwell International was to build monolithic integrally stiffened hardware that could be used to replace built-up fastened and welded assemblies for the B-1 Bomber airframe. Many different configurations and geometries of sandwich structure were designed, built, and tested for structural efficiency, as shown in Fig. 3. One of the team leaders of this program at Rockwell's Science Center was Hamilton. Much of the technical material relating to the development of DB included in this paper was obtained from Hamilton's lecture notes (Ref 1).

The commercial manufacturing process of SPF combined with DB (SPF/DB) for titanium sheet components was initially developed at the Rockwell Science Center in Thousand Oaks, CA, between 1971 and 1984. The testing performed at the Science Center validated the discovery made by Rockwell's scientists that DB joined titanium sheets could be inflated with high-pressure inert gas under SPF conditions to build a highly efficient and lightweight titanium sandwich structure that was cost competitive with traditional honeycomb core structures. Figure 3 shows a sample of the titanium structures fabricated by Rockwell's Science Center between 1974 and 1977 for the B-1B (Fig. 4).

Additional development of SPF/DB was performed at the Rockwell Titanium Processing plant in El Segundo, CA, where the first production parts were built. This resulted in the prototyping of a host of parts using SPF/DB (Fig. 5).

Follow-on work after the B-1B project ended included the design and fabrication of a complete aft fuselage for a fighter aircraft by Rockwell for structural tests and evaluation. The fighter program expanded to include the McDonnell Douglas Corporation, which is now a subsidiary of Boeing. McDonnell's task was to completely redesign the F-15D aft engine tunnels and deck using a SPF/DB titanium sandwich structure. McDonnell's research team investigated a new kind of sandwich structure, which used welded titanium inner cores that could be inflated using argon in a hot die so that the cores would form I-shaped stiffeners and diffusion bond to the outer skins (Fig. 6).

When the refit of the F-15D aft fuselage was implemented, the aircraft was redesignated as the F-15 Eagle (F-15E) by the U.S. Air Force, due to the dramatic changes that the SPF/DB monolithic panels made to the overall airframe. The new tunnel could accommodate a larger engine with greater thrust, which dramatically improved the performance characteristics of the aircraft. Some other benefits that resulted from using the monolithic SPF/DB structure included the elimination of 726 detail parts and 10,000 fasteners, increased load capabilities, 10 cubic

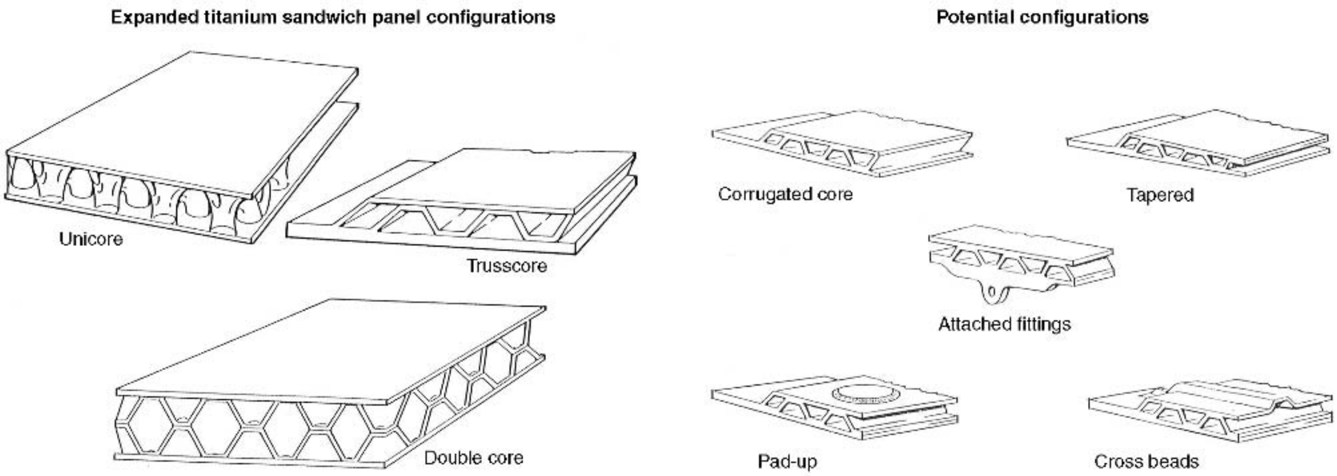


Fig. 3 The drawing at the left shows several different configurations of integrally stiffened flat panels using DB stiffeners, trusses, and webs. The drawing at the right illustrates the ability to fabricate SPF/DB panels to include bonded attach features and peripheral extensions.

Fig. 4 Many sections of the B-1B were studied for potential SPF/DB applications.

Fig. 5 Several parts were developed and prototyped using SPF/DB for the B-1B. This photograph shows many of the titanium parts that were built as demonstration and testpieces, but did not make it onto the baseline design.

feet of space for special equipment, and significantly reduced weight (Ref 8). Figure 7 shows the F-15E.

2.2 Mechanisms of Diffusion Bonding

There are several conditions that govern the DB process that must be fulfilled for DB to progress without disbonding, bond-line porosity, or other defects:

- It is generally accepted that DB of titanium occurs between 900 and 950 °C with the actual extreme upper limit being the β transus temperature for Ti-6Al-4V (990 °C).
- All surface oxides, oils, dirt, dust, hair, abrasive particles, and other debris must be completely removed or they will contaminate the titanium surface and bond-line.
- An inert atmosphere is needed around the surfaces of the parts being bonded to prevent the formation of high-temperature oxides (α case), which would inhibit or completely prevent bonding. A vacuum of 10^{-4} to 10^{-6} torr is desired to completely remove air from between the two mating surfaces.
- Titanium forms a very tough oxide that can inhibit diffusion, but it takes about 30 days for this layer to form naturally under standard atmospheric conditions. A flash pickle using nitric-hydrofluoric acid is needed at least a month prior to bonding of the sheets to ensure that a thick oxide layer is not present.
- The superplastic flow stress of the titanium plays an important roll in the deformation of the sheet and influences the amount of time required for complete bonding.
- The sheets being bonded must have some pressure or force applied to encourage intimate contact, and once the sheets touch, additional force is required to deform/flatten the high points so that they make contact over the interfacial

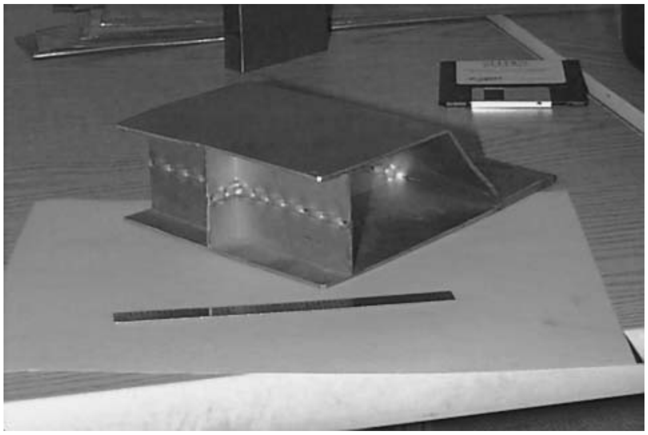


Fig. 6 McDonnell Douglas' SPF/DB process using welded core. This is a testpiece that has been formed and diffusion bonded.

Fig. 7 The Boeing F-15E, first fighter to make extensive use of SPF/DB structure

planes. Superplastic deformation using gas pressure is the preferred method, since this speeds up the process considerably compared with other methods such as squeezing of two sheets between a hot tooling plate. A bonding pressure of 2 to 8 MPa is generally accepted as the range needed for DB using modern hot presses and heated pressure chambers.

- Microporosity between the sheets will disperse only if sufficient time is given for trapped inert gas and oxides to diffuse into the grain boundaries of the titanium matrix.
- Grain boundary migration is a necessary element of diffusion, and this requires soaking time at the DB temperature to ensure a homogeneous grain structure is achieved across the bond line.
- The grain size before heating affects bonding. Smaller grains diffuse faster than large grains. As time goes by, grain growth occurs due to the effects of temperature, and excessive grain growth (or bonding time) can lead to a lack of diffusion.
- Contaminant metals or oxides attached to the titanium alloy sheet or on the surface of the sheets must be absorbed into the grain boundaries during bonding. Similarly, any trapped argon (or other inert gas) must diffuse into the grain boundaries surrounding the bond-line or there will be microscopic pores left at the bond-line. Pores that reach a percentage surface area greater than 3%, or a size larger than 0.250 mm, are generally unacceptable, although these factors can be relaxed for the design of noncritical structures.

Although it is clear that each of the mechanisms listed above has a contributing influence over the bonding process, there is insufficient research at this time to determine the extent to which each plays a role. Additional research is needed to quantify and model each of these parameters. However, there is sufficient understanding of the macro-requirements for DB and the physical laws governing atom-to-atom bonding to use the process in a manufacturing environment. It is noteworthy that hollow, wide chord, titanium SPF/DB jet engine fan blades have been built by Rolls-Royce and Pratt & Whitney for more than 20 years and that these production enterprises have been very successful (Ref 9).

The interdiffusion of atoms and the sharing of grains across the bond-line will usually commence immediately upon initial contact, as shown in Fig. 8. As the surfaces of the mating sheets are forced into closer contact, additional grains become engaged and recrystallize.

The extent of the effects of oxides, hydrocarbons, rogue gases, and other foreign material on the required time for DB and the quality of the bonded joint has not been completely quantified. The presence of these contaminants in the pores found at the DB joint of test panels and production hardware has been investigated. It is known that the effects of these materials on the quality of the bond-line and the time required for complete diffusion of grains is significant, perhaps even the greatest influencing factor for the manufacturing cycle time required for complete diffusion of the surfaces.

2.3 Surface Deformation Mechanics for Diffusion Bonding

A simple constitutive equation has been developed by Hamilton (Ref 1) to predict the bonding time and pressure needed under idealized conditions for DB to occur, with the assumption that the surface flattening that is needed to achieve intimate contact of the grains along the bond line is the most significant variable for complete diffusion. Figure 9 is a step-by-step depiction of how an idealized square pore, rotated 90° relative to the bond line, would deform using this model. Figure 10 illustrates how the deformation model has been developed.

2.3.1 Average Stress and Creep Rate. One can assume:

- Plane strain
- That the required time for diffusion to occur can be ignored
- That the following is based on experimental test data that has not been fully validated

$$P_b = \frac{\bar{\sigma}}{\sqrt{3}}$$

$$\tau_b = \frac{\bar{\varepsilon}_c}{\dot{\bar{\varepsilon}}} = \frac{2}{\sqrt{3}} \ln(0.5) \frac{1}{\dot{\bar{\varepsilon}}} = \frac{0.8}{\dot{\bar{\varepsilon}}}$$

where P_b is the pressure required for bonding to occur, τ_b is the bond time for complete bonding, $\bar{\sigma}$ is the effective stress from

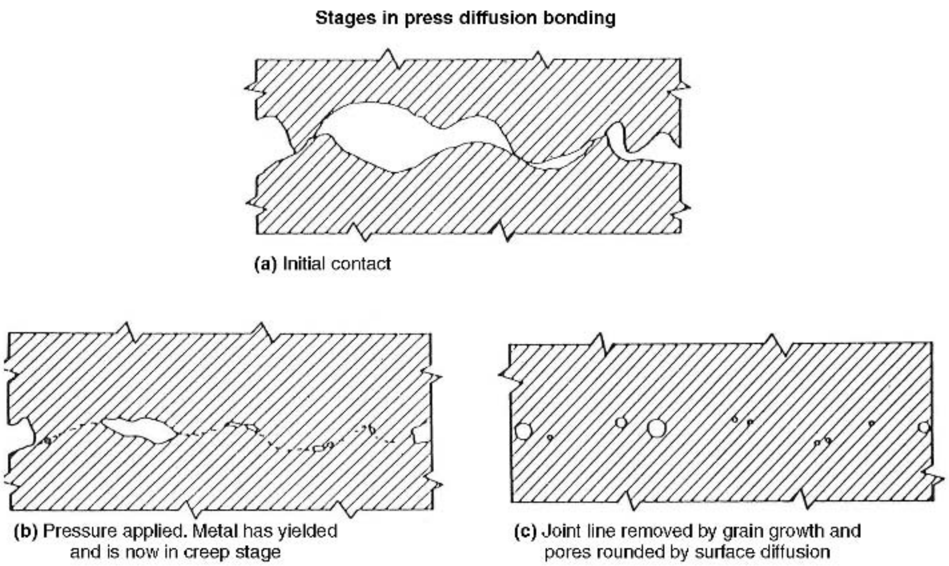


Fig. 8 The progression of DB between two separate sheets of titanium, which become joined into a homogeneous structure as time progresses. Note that the shape of the surface and its texture can have a significant effect on how quickly the process occurs. Any trapped materials or gas must diffuse out of their porous cavities through the individual grain boundaries. Oxides present between the sheets can completely inhibit diffusion bonding or result in unacceptable bond-line porosity. Micropores can become fatigue crack initiation sites if they are allowed to occur in highly stressed regions.

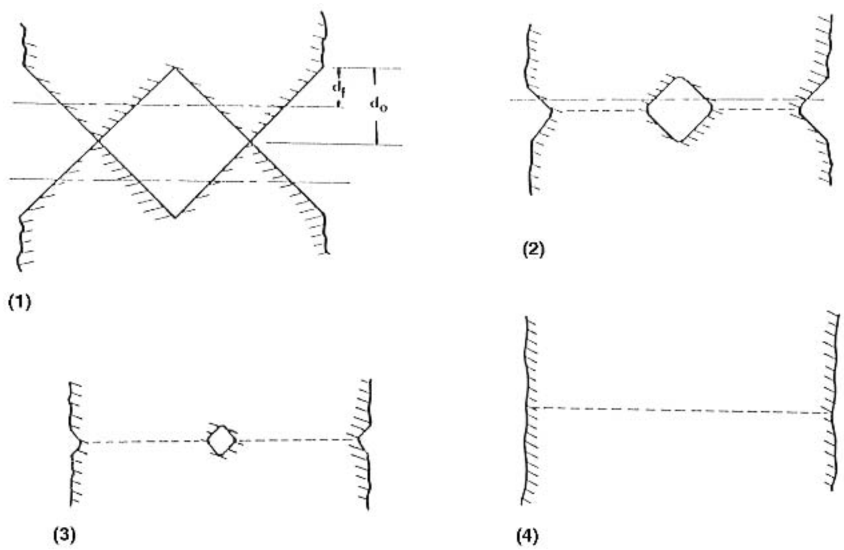


Fig. 9 An idealized model of how a bond-line might evolve after (1) initial surface contact and (2) and (3) as time goes on the closing of the pores as the surface is flattened out due to deformation, and (4) a completed bond line

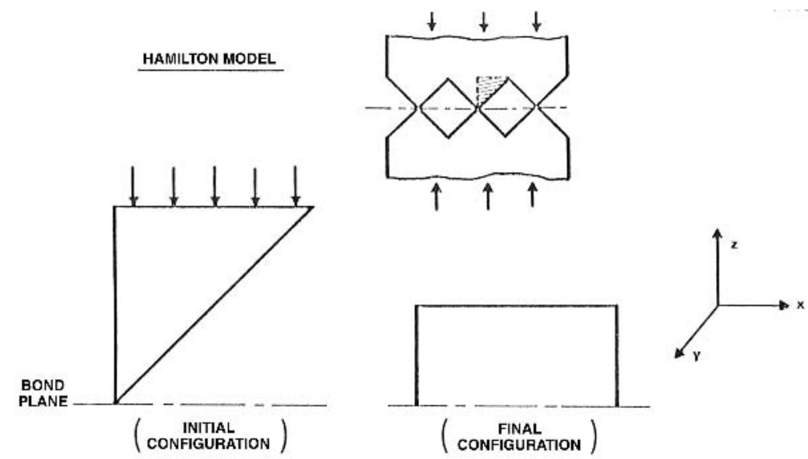
uniaxial test data, $\bar{\dot{\varepsilon}}$ is the effective strain rate from uniaxial test data; and $\bar{\varepsilon}_c$ is the effective strain for closure of the surface (i.e., flattening of asperities).

The effect of temperature on DB was studied extensively through empirical testing that has been verified by many research organizations around the world. It is clear that the processing temperature for a titanium workpiece is one of the primary factors influencing the deformation of the surface and for diffusion to occur between individual grains. This is shown in some of the early test data collected by Rockwell, which has been graphed in Fig. 11.

The combined effects of surface asperity creep (flattening of the surface over time) and the diffusion annealing of voids can be modeled into a single constitutive equation. This formula can be used to approximate the time needed under idealized conditions for micropores to diffuse.

2.3.2 Combined Void Shrinkage Rate. One can assume:

- Plane strain
- That long and short wavelength asperities are present
- That the following is based on experimental test data that has not been fully validated



$$\epsilon_z = \ln(1/2) = -0.69$$

$$\bar{\epsilon} = 0.80 \qquad \text{WHERE} \quad \dot{\bar{\epsilon}} = f(\sigma)$$

$$t = \frac{\bar{\epsilon}}{\dot{\bar{\epsilon}}} \qquad \text{AND} \quad \bar{\sigma} = p\sqrt{3}$$

Fig. 10 The simplified deformation model used to develop the average stress and creep rate formula

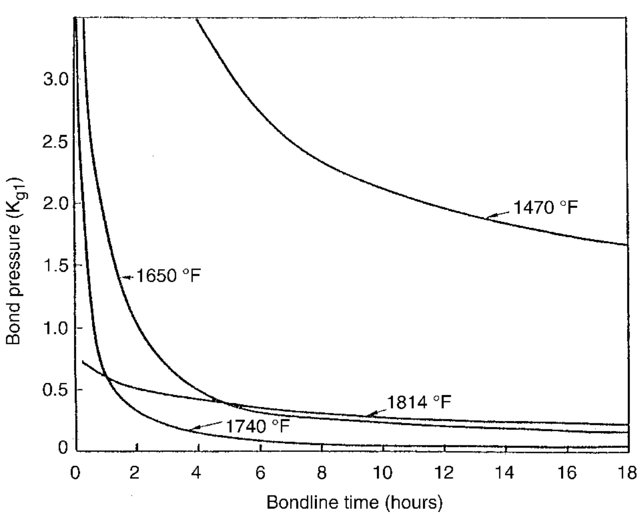


Fig. 11 A summary of test data for DB of Ti-6Al-4V at various temperatures. The Y-axis is bonding pressure (ksi), and the X-axis is the observed time required for complete diffusion to occur (h).

$$\left(\frac{da}{dt}\right)_{\text{Total}} = \left(\frac{da}{dt}\right)_{\text{Creep}} + \left(\frac{da}{dt}\right)_{\text{Diffusion}}$$

$$\left(\frac{da}{dt}\right)_{\text{Total}} = -\left\{\left(\frac{D_v\Omega}{KT}\right)\frac{1}{a}\left(\frac{2\gamma}{a} + P_{\text{ext}}\right)\right\} + \frac{3K}{4}a\left\{2\sigma_o \ln\left(\frac{b}{a}\right) + \left(\frac{2\lambda}{a} - P_{\text{in}} + P_{\text{ext}}\right)\right\}$$

where a is the intersurface distance (or void radius), b is the vacancy sink radius, D_v is the bulk diffusivity, K is a constant from material constitutive equation: $\dot{\varepsilon} = K(\sigma - \sigma_o)$, P_{ext} is the externally applied pressure, P_{int} is the internal void pressure, T is the temperature, Ω is the atomic volume, and γ is the surface energy.

Although the formula above has been used to create several finite element analysis computational models, it has not been shown to be accurate in terms of predicting the cycle time needed for DB in either laboratory or under industrial conditions. However, it does give a reasonably close approximation that can be taken into account when using empirical data, and it is frequently used by manufacturing engineers for calculating a "low" time estimate. This estimate can then be multiplied by some factor to increase the bonding time to a more realistic value. The formula does take into account two of the three most significant process variables: temperature and time. The other manufacturing variables, such as cleanliness of the surface (oxides), contaminants, smoothness of the titanium surfaces, the time from initial pickle (nitric hydrofluoric acid etch), argon purity, and/or vacuum level, weld quality, stop-off (yttrium) purity, and thermal distortion, have not yet been factored in.

An alternative and possibly more accurate modeling mechanism was developed by Pilling (Ref 2), which utilizes cylindrical-shaped voids within the initial diffusion contact surface rather than the triangular voids suggested by Hamilton (Ref 1). The new mathematical model, which describes the collapse of cylindrical voids under hydrostatic pressure, as diffusion overcomes them, was developed and tested by Pilling against experimental results for titanium Super Alpha-2 (Ref 10) in 1996. The results of this work showed an encouraging improvement in accuracy, but still left a substantial margin of error that could be attributed to the DB process variations during the fabrication of test coupons, the superplastic material properties of titanium Super Alpha-2, or the cylindrical model itself. It is clear that more verification testing of actual DB parts to compare against

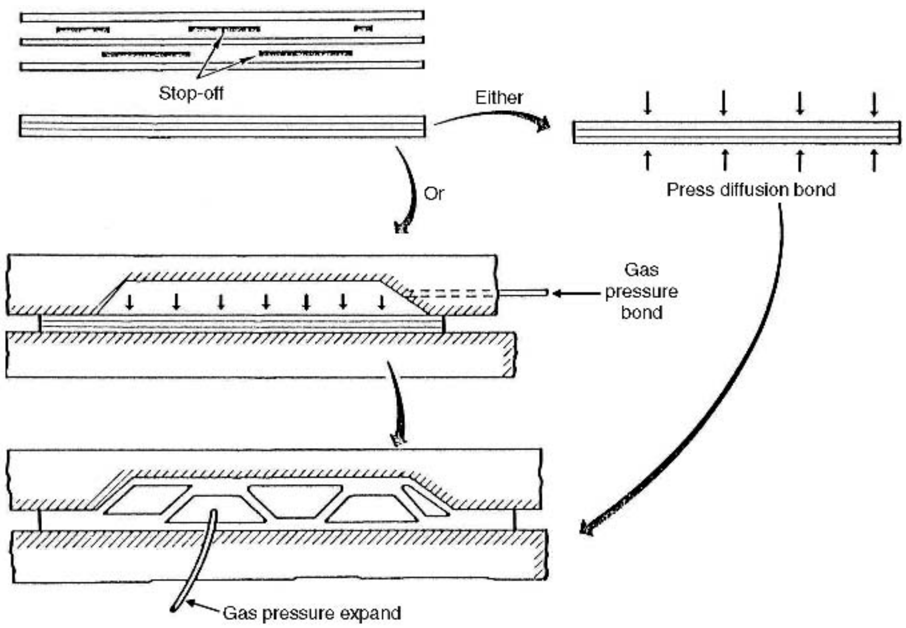


Fig. 12 The Rockwell SPF/DB methods of fabricating SPF/DB panels using gas pressure or mechanical press bonding

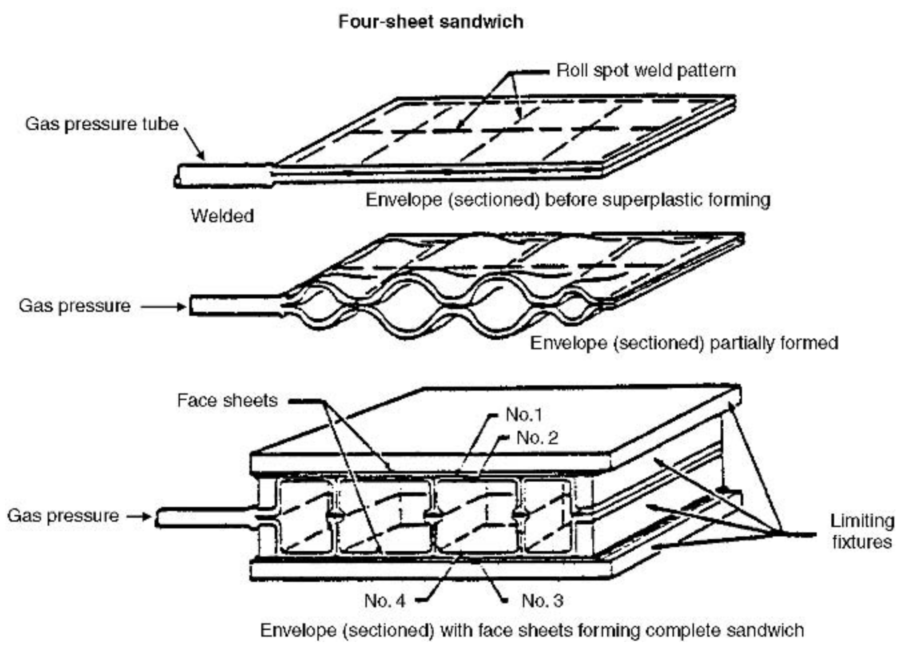


Fig. 13 The Rockwell SPF/DB expansion method for fabricating four sheet SPF/DB truss and I-beam stiffened core

the cylindrical model would help bridge the link between theory and a stable production process.

2.4 Manufacturing Processes and Tooling

Many of the Rockwell B-1 B parts that were built using two- or four-sheet titanium DB processes as a tooling methodology required bonding of individual parts one at a time. For this process, the individual sheets were silk screened with a pattern of ytrria powder. Yttria is a compound that can prevent bonding between the titanium sheets in the areas of the part that are later expanded into stiffeners. Once cleaned and silk screened, the titanium sheets were assembled into part packs and tungsten inert gas (TIG) welded around the periphery to make an airtight retort. Gas tubes were placed strategically so that a vacuum could be pulled during the DB cycle and high pressure argon could be introduced between the sheets during the SPF forming phase. Although thousands of production parts have been built using this method, the cycle time for each part is between 6 and 12 h. That means that a hot press must be

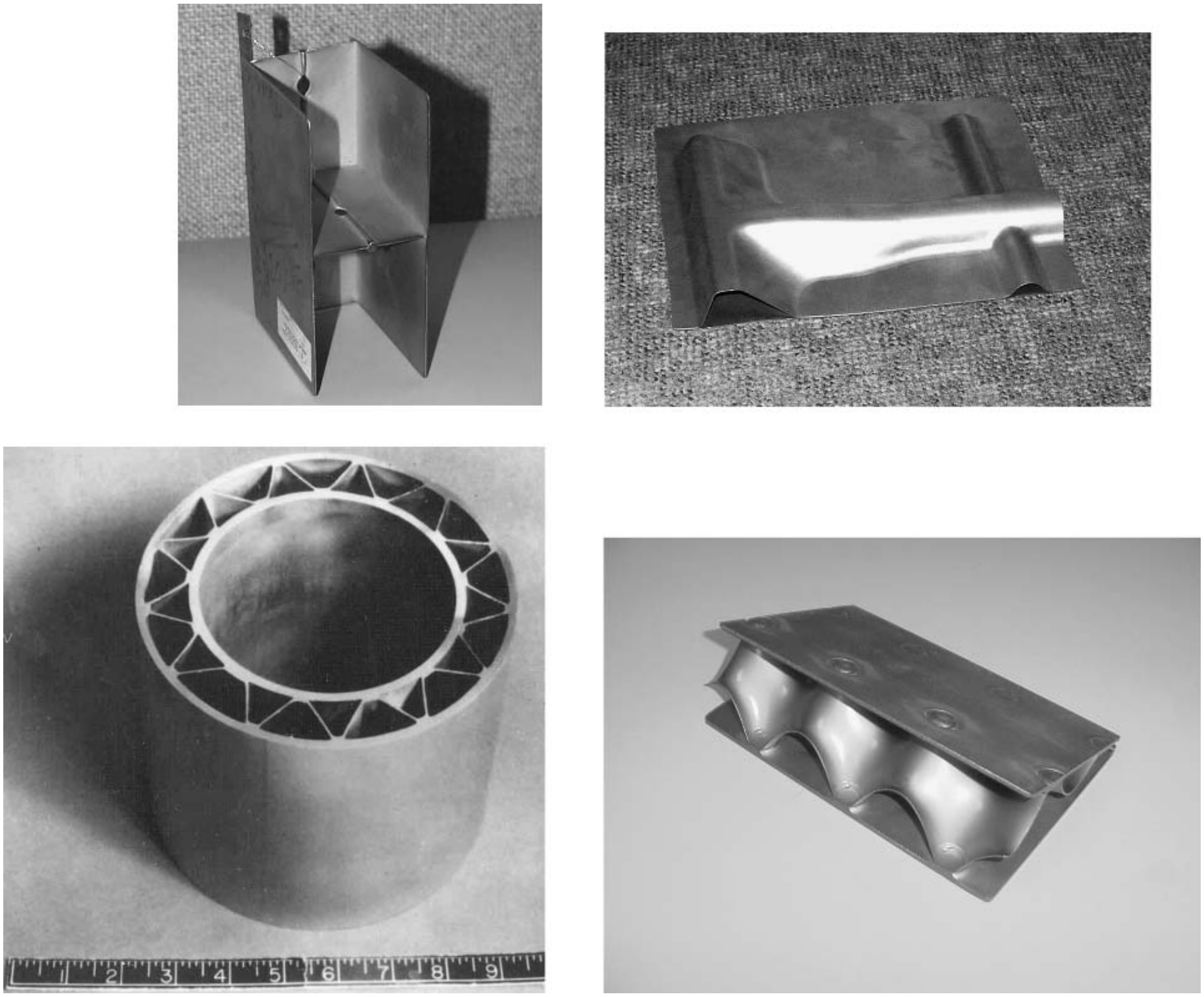


Fig. 14 Sample sections of multisheet SPF/DB truss core built by Boeing and its subsidiaries. Upper left, four-sheet welded core panel. Upper right, two-sheet stiffened panel built using yttria stop-off and gas pressure bonding in a hot isostatically pressed vessel. Lower left, circular SPF/DB using three sheets of titanium and yttria stop-off. Lower right, "dot core" built using partial penetration laser weld

used to compress the die halves together and maintain the temperature of the die for an average of 9 h per part. Since hot presses are very expensive, in the range of $2 to 5 million each, the summation of recurring and nonrecurring costs is extremely high. This process has now been refined so that many parts (up to 50) can be batch diffusion bonded in a heated pressure vessel as a separate step from the forming operation. This means that the heated die press is used only during the forming of the pack, which requires less than an hour. Figure 12 shows the basic processing steps originally used by Rockwell for the yttria stop-off and SPF/DB method, using either mechanical bonding or gas pressure bonding, depending on the part geometry. Mechanical bonding was eventually abandoned due to the high-temperature creep that often "mushroomed" or warped the die after only a few cycles to the point where parts could no longer be bonded. Figure 13 depicts a core pack that is inflated with argon.

The four-sheet SPF/DB process requires that the inner two sheets, that is, the "core," be either prewelded or DB joined with a pattern that allows argon to disperse between the cells as the core is inflated. The pressure between the core and the outer skin sheets must be maintained at a lower pressure than the gas pressure within the core, such that the outer sheets have sufficient pressure to be pushed to the inner moldlines of the forming die, but still have less pressure than the core so that the core can form outward to eventually contact and diffusion bond with the skin sheets. The result is a formed and bonded structure that can be made into many shapes of titanium sandwich tailored around the required part design envelope and structural loading characteristics. Figure 14 shows a typical four-sheet SPF/DB panel during the initial fabrication of the core, assembly with the outer skin sheets into a pack, and as-formed structure in a die.

2.5 Aerospace Applications

The aerospace industry is the primary manufacturing segment engaged in building SPF/DB components. Most of the airframe assemblies and subcomponents that have been built are in areas of the aircraft subjected to very high temperatures and very large structural loads. Some of the primary applications for titanium SPF/DB are landing gear doors, engine fan blades, engine nacelles, auxiliary power unit thermal protection, environmental control system ducting, leading edges of airfoils for bird strike and hail damage protection, erosion sheaths, foreign object damage deflectors, engine access doors, wing access panels, high-temperature bleed gas ducting, cargo bay floors, and fuselage tunnel covers.

3. Summary

The combined process of SPF and DB for titanium has been investigated for roughly 30 years by researchers and engineering scientists in the aerospace industry. Initial research focused on the fabrication of parts for the B-1B and F-15E military aircraft. Methods have been devised to bond and form sandwich panels using several novel techniques. Manufacturing and tooling approaches for forming of parts have been developed that allow complex compound surfaces to be formed in shapes that have never been possible using other technologies, especially for structures that are subjected to extreme temperatures and high loading conditions.

Although the commercialization of the SPF/DB process has matured over time, the development of constitutive equations and mathematical models for the mechanics of the process is still not complete. A set of highly idealized equations have been developed to quantify the diffusion bonding phenomenon considering creep and diffusion of titanium at the interfacial surfaces; however, important factors have been omitted—variables such as the diffusion of oxide contaminants, surface roughness of the raw material, diffusion of trapped inert gas (if present), the effects of contour shapes (die geometry), process variations (pressure, temperature, part loading), strain hardening, grain growth, and variations in material such as the yield strength, grain size, percent elongation, and composition. Additional research and development is needed to explore the characteristics of SPF/DB.

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