CHAPTER 52

DIFFUSION WELDING

FUNDAMENTALS OF PROCESS

Although the principles of diffusion welding have been known for more than a thousand years, interest in this method of joining has grown appreciably only in the last twenty years. Diffusion welding offers solutions to several joining problems associated with newer materials: avoidance of metallurgical damage (e.g., as with beryllium, dispersion-strengthened metals and refractory metals); reduction of sensitivity to corrosion (e.g., titaniun and zirconium); improved fracture toughness (e.g., titanium); improved joints between dissimilar materials (both metal-to-metal and metal-to-nonmetal), and fabrication to intricate shapes. Successful application of diffusion welding to engineering problems requires a good understanding of the fundamentals both to make successful joints and to ensure that they perform in service as expected. Because of the nature of the process a great deal of the discussion in this chapter will necessarily be metallurgical and chemical in nature.

Historically, solid state joining processes antedate fusion joining processes which had to await the development of suitable heat producing sources. However, in most cases early solid state joining processes required large amounts of deformation, such as in the forge welding of wrought iron or steel and the early powder metallurgical methods to fashion precious metals such as silver and platinum. It is only in recent years that distinctions have been drawn between various forms of solid state joining methods such as forge welding and diffusion welding (frequently called diffusion bonding). The AWS Definitions Committee provides the following definition: "Diffusion welding is a solid state welding process wherein coalescence of the faying surfaces is produced by the application of pressure and elevated temperatures. The process does not involve macroscopic deformation or relative motion of the parts. A solid filler metal may or may not be inserted."

The distinction imposed by the absence of macroscopic deformation and relative motion of the parts introduces a less than rigid boundary between solid state joining methods which use large deformations to promote bonds. The overall characterization procedures and definitions for solid state welding processes are still in a dynamic stage. Within this chapter some continued definition by inference may occur.

(Diffusion welding)

THEORY OF SOLID STATE JOINING PROCESSES

All solid state joining processes, including diffusion welding, involve two characteristic steps. The first is to achieve mechanical intimacy of contact; the second is to induce complete metallic bonding across the area of contact. These steps must occur in proper sequence in each welded area but need not proceed at uniform rates in the entire part. Therefore, the final bonding may be completed in certain places in a joint before contact is established elsewhere. The progression of each step is a function of the conditions under which the joints are made and of the properties of the materials being joined. The necessity of these two steps to form a bond in a solid state weld is a result of the nature of a real metal surface. A schematic cross sectional view of a real surface is shown in Fig. 52.1. It illustrates several characteristics: (1) roughness (or non-smoothness); (2) an oxidized or otherwise chemically reacted and adherent layer; (3) other or randomly distributed solid or liquid products (oil, grease, dirt, etc.), and (4) adsorbed gas, moisture or both. Thus, to achieve metallic bonding both deformation of the substrate roughness and disruption and dispersal of the interfering surface contaminants must be achieved.

2 steps
1. mechanical intimacy contact
2. induce complete metallic bonding

nitrided

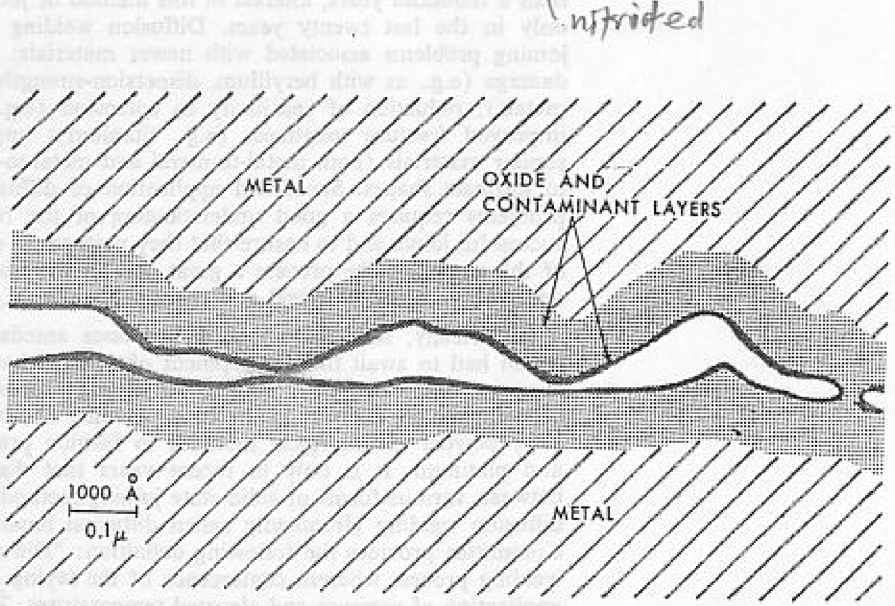


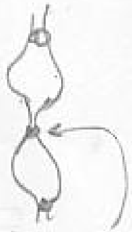
Fig. 52.1.—Schematic illustration of the character of a real metal surface showing roughness and contaminants present

DIFFUSION WELDING TECHNIQUES

1st Step

① ESTABLISHMENT OF CONTACT

A typical surface, even if perfectly clean, is rough in a microscopic sense. Surface roughness prevents full contact when parts are pressed together. The individual asperities which make up the roughness are deformed by application of increasing pressure so that the true contact area A becomes a larger fraction of the faying areas A_0. Under a force F, the fractional contact area is given by:



$$\frac{A}{A_0} = \frac{F/A_0}{Y} = \frac{\text{Pressure}}{\text{Yield Strength}}$$

↑ YS → ↓ contact area

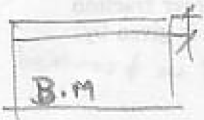
where Y is the yield strength of the metal. For parts under a constant force F, the fractional contact area is initially low because the yield strength is high at room temperature. When the parts are heated, as is more typical of diffusion welding, material yield strengths decrease and the fractional contact area increases. As the surface deforms, there is an increase in the restraint on plastic deformation due to adjacent asperities so that Herzian yielding is approached. Theoretically, full-scale plastic flow with attainment of complete contact occurs when the stress reaches three times the yield strength in compression. Thus, the necessary plastic flow is controlled principally by the yield strength of the material. This value varies with temperature, time and microstress conditions at the interface. With greater times at elevated temperatures, as occurs in most diffusion welding operations, time dependent deformation (creep) can be thought of rather than yield strength. Under such conditions the above equation must be modified and the A/A_0 may be described as a function of creep strength.

WELD FORMATION BY METALLIC BONDING

When pressure is applied, deformation begins at the highest asperities (cf. Fig. 52.1) and gradually spreads until plastic flow is general. This initial contact does not occur between metal surfaces, but rather between the barrier films trapped between the surfaces under compression. At places where the surfaces move together under shear, the films are disrupted and metal-to-metal contact begins.

Disruption of these films and formation of metallic bonds constitute the second step in diffusion welding. In contrast with the mechanical steps of establishing contact by yielding and shear, the subsequent steps in completing the bond, carried out at elevated temperature, involve thermally activated processes for both the completion of film disruption and interdiffusion to generate a bond.

This film, for further discussion, is largely an oxide film. Cleaning methods and proper procedures reduce the other components of the film to negligible levels. Two processes act to disrupt and disperse the films. The first dissolves the film in the metal; the second is spheroidization or agglomeration. Oxide films may be dissolved in titanium, tantalum, columbium, zirconium and other metals in which interstitial elements are highly soluble. If oxygen is relatively insoluble in the metal, as is the case in aluminum, the disruption process for the trapped films is spheroidization. This process leaves a few oxide particles along the weld line. However if it has been properly made these are no more detrimental than the inclusions found in all metals or alloys.



Both processes require diffusion; solution occuring by diffusion of interstitial atoms into the metal, and spheroidization by diffusion as a result of the excessive surface energy of the thin films. The time for solution of a film of thickness, x, is proportional to x^2/D where D is the diffusion coefficient. The thickness must be kept very small if diffusion welding times are to be kept short. Spheriodization also occurs more rapidly if the oxide films are thinner. Hence, control of the thickness of oxides, both the initial thickness after cleaning and the amount of growth during heating, is a critical factor in diffusion welding.

* diffusion

Once true metal-to-metal contact is established, the atoms are within the attractive force fields of each other and hence a high-strength joint is generated. * The joint at this time resembles a grain boundary because the metal lattices on each side of the line have different orientations. However, it may differ from an internal grain boundary in that it may contain more impurities, inclusions and voids which remain at a weld interface if full asperity deformation has not occured.

A planer interfacial boundary is not thermodynamically stable and tends to migrate to a more stable configuration if conditions permit. Its migration will proceed more easily if few voids or inclusions exist at the boundary. The overall process is illustrated in Fig. 52.2. This figure is a simplification since the sequences described vary a great deal with process conditions and alloys.

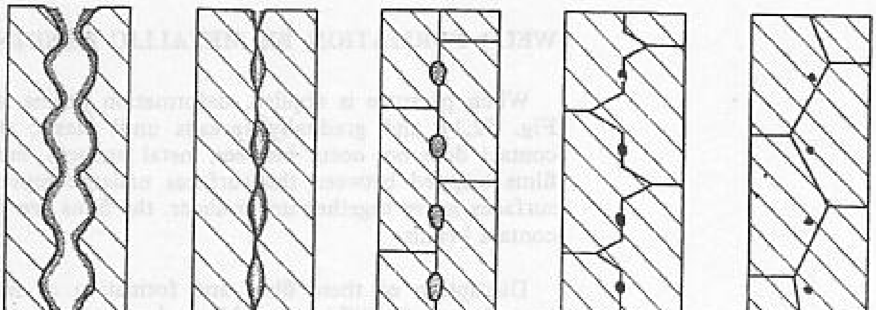


Fig. 52.2.—Overall two step diffusion welding process. (Steps expanded in sequential form for clarity.)

SURFACE PREPARATION

The surfaces of parts to be diffusion welded are carefully prepared prior to assembly and welding. Surface preparation involves more than just cleanliness. It includes all the following steps: (1) generation of an acceptable finish or smoothness, (2) removal of chemically combined films, oxides, etc. and (3) cleansing of gaseous, aqueous or organic surface films.

The initial surface finish is ordinarily obtained by machining, abrading, grinding or polishing. A correctly prepared surface is flat. Flatness and smoothness are required in order to assure that the interfaces can achieve the necessary compliance without excessive deformation. Machined finishes, grinding or abrasive polishing, are usually adequate as long as proper precautions are exercised to avoid warpage and distortion. In shaped parts, where curved surfaces are to be matched, careful preparation calls for appropriate contour matching of surfaces.

A secondary effect of the initial machining or abrading, not always recognized, is the deformation introduced into the surface during machining. Cold worked surface layers ordinarily have lower recrystallization temperatures and higher diffusion rates than the bulk material. It has been suggested that a worked surface is an effective aid in diffusion welding. On the other hand surface recrystallization is not necessary or even desirable in some diffusion welding applications and in these cases surfaces are prepared to minimize surface work.

Chemical etching or pickling is commonly used as a preweld surface preparation. It has two effects. The first is the removal of non metallic surface films, mostly oxides. The second is the removal of part or all of the cold worked layer that occurs during preliminary surface preparation. The benefits of oxide removal are apparent — it is most difficult to cause two oxidized surfaces to adhere. Many chemical solvents are suitable for use with different metals systems. Manufacturers' literature may provide useful information on pickling agents.

Degreasing is a universal part of any procedure for prediffusion weld cleaning. Alcohol, trichlorethylene, acetone, detergents, etc., may be used. In some cases the recommended techniques are intricate and may include multiple rinse — wash-etch cycles in several solutions.

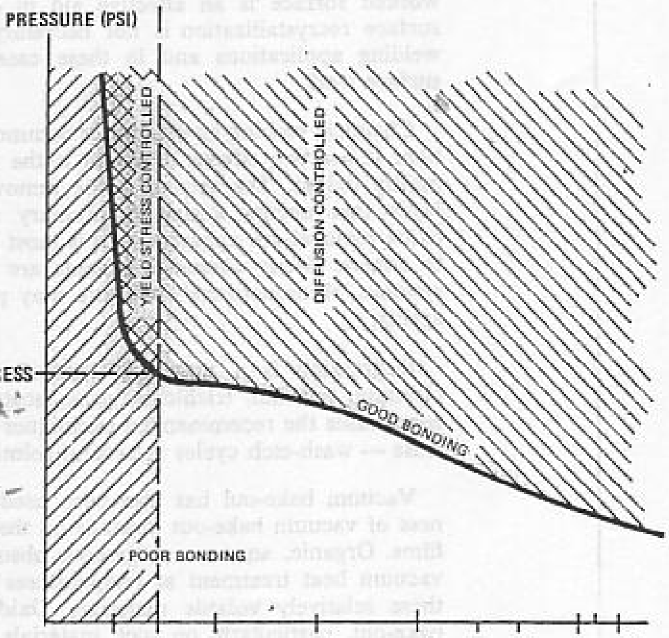
Vacuum bake-out has also been used to obtain clean surfaces. The usefulness of vacuum bake-out depends on the material and the nature of its surface films. Organic, aqueous or gaseous absorbed layers can be removed easily by vacuum heat treatment at temperatures and pressures which cause boiling of these relatively volatile materials. Oxides are rarely dissociated by vacuum bake-out, particularly on such materials as titanium, aluminum or alloys containing significant amounts of chromium. However, it is possible to dissolve oxides in some base materials (e.g., zirconium and titanium) at elevated temperatures. Samples that are baked in vacuum usually require vacuum or controlled atmosphere storage and careful handling prior to use to minimize the recurrence of surface absorbed or chemi-sorbed layers.

melting Temp.

PROCESS VARIABLES AND TECHNIQUES

Diffusion welding generally is carried out at low to modest pressure, high temperatures (> ½ Tm)* and for longer times than other solid state processes. However, much variation exists in the process parameters and it has become common to discuss diffusion welding in terms of two arbitrary subdivisions, deformation diffusion welding (or yield strength controlled) and diffusion controlled welding (or creep controlled). Fig. 52.3 distinguishes the two by illustrating the two modes in a single material diffusion welded at a given temperature. If relatively short times and high pressures are used local asperity deformation is most important and yielding dominates the formation of a bond. If longer times and lower pressures are used, creep and diffusional processes are more prevalent. Joints made with deformation controlled parameters tend toward planar joint interfaces while diffusion controlled joint conditions produce less planar joints.

Diffusion welding variables can be grouped into six categories: (1) surface preparation, (2) temperature, (3) time, (4) pressure, (5) special metallurgical effects and (6) use of interlayers.



below YP: elastic deformation

above YP: plastic deformation ∴ new contact area are got ∴ good bonding

Fig. 52.3.—Types of diffusion bond process (temperature constant)

---

* Tm is the melting point of the material involved, usually in degrees absolute.

6

Many factors enter into the selection of the total surface preparation treatment. The specific welding conditions to be used may affect the selection. With higher welding temperatures and pressures, it becomes less important to obtain extremely clean surfaces. Increased atomic mobility, surface asperity deformation and solubility of impurity elements all contribute to the self-removal of surface contaminants during welding. As a corollary, it can be stated that to lower the minimum diffusion welding temperature or pressure, it is necessary to provide better prepared, cleaner surfaces.

SURFACE PRESERVATION

It is folly to exercise extreme caution in preparing surfaces prior to diffusion welding if they are permitted to become recontaminated during subsequent handling. One solution to this potential hazard lies in the effective use of protective environments during diffusion welding. Vacuum protection during diffusion welding provides continued freedom from contamination as it does during surface preparation. Use of hydrogen as an atmosphere in diffusion welding will help to minimize the amount of oxide formed during welding and may reduce existing oxides. However, it will form hydrides in alloys of titanium, zirconium, hafnium, columbium and tantalum that may be detrimental to final service properties. Argon, helium and possibly nitrogen can be used to protect clean surfaces at elevated temperatures. When these inert gases are used, their purity must be very high. Inert gases possess none of the advantages of chemical or physical activity that hydrogen or a vacuum offer. Many of the precautions and principles applicable to brazing atmospheres can be directly applied to diffusion welding.

TEMPERATURE

Of the fundamental process parameters — time, temperature and pressure — temperature receives the most attention. The reasons for this include:

1. Temperature is readily changed and relatively easy to measure and control.
2. Due to the nature of the diffusion welding process and the effects of temperature on plasticity, diffusivity, oxide solubility, etc., temperature changes may greatly affect results.
3. Temperature changes are relatively inexpensive; by increasing temperature one can often shorten cycles and improve the economics of the operation.
4. Since other factors such as allotropic transformation, recrystallization, solution of precipitates and oxides are all temperature dependent, one must control temperature to promote or avoid these factors as desired.

Joint quality usually can be increased with increases in diffusion welding temperature. Fig 52.4 shows the variation of strength of medium carbon steels as a function of welding temperature. There is considerable disagreement of the specific temperatures at which to diffusion weld any given material or alloy. One reason is that the minimum temperature to achieve a sound diffusion weld is related to pressure, time, surface preparation and many other factors. However, most values are generally greater than 0.5 T_m and a large fraction are between 0.6 and 0.8 T_m. Some data will be given for specific alloys later.

TIME

Time is a dependent process parameter. It is related to temperature and pressure because diffusional reactions are linearly or parabolically related to time. An increase in temperature shortens the amount of time required to complete a diffusion dependent event.

In diffusion welding application, time may vary from a few seconds to several hours. Practical factors may influence the time needed for diffusion welding. In systems having thermal and mechanical (or hydrostatic) inertia, diffusion welding time is longer due to the impracticality of suddenly changing variables. If there are no inertial problems, welding times may be relatively short (seconds in some cases). For economic reasons it is desirable to reduce the time factor, thereby increasing potential production rates.

An illustration of the importance of time in obtaining a good diffusion weld in 0.45%C steel is shown in Fig. 52.5. Note also the interrelation of time and temperature.

PRESSURE

Pressure, another important variable in diffusion welding, is less easy to deal with than time or temperature. Pressure affects several of the diffusion welding mechanisms. The initial deformational phase of bond formation is directly affected by the intensity of pressure applied. For any given time-temperature value, increased pressure invariably results in better joints. Higher pressures mean greater interface deformation and asperity breakdown. Pressure also affects recrystallization behavior. Increased pressure may increase local deformation and lead to a lower localized recrystallization temperature. Conversely stated, the increased deformation accelerates the process of recrystallization at a given diffusion welding temperature.

Since the pressure needed to achieve success is related so closely to the other parameters of temperature and time, there is a great degree of latitude in the pressure needed to make good welds. From an economic point of view, reductions in welding pressure are desirable. Increased pressures require costlier apparatus, greater need for control and more complex part-handling procedures.

Uniform pressure is most important. Diffusion welds are usually made in components with large faying surfaces that are often curvilinear or discontinuous. Some components are assembled by diffusion welding solely because of geometric factors that preclude the use of other joining methods. In such cases, pressure must be uniform to assure consistency of bond formation in all areas. This factor is a prime subject in tooling and apparatus design and will be discussed in more detail later.

METALLURGICAL FACTORS

A number of specialized metallurgical events can become important factors in determining the process parameters for diffusion welding. They can set limits on the remaining chosen parameters for a variety of reasons. The most important are allotropic transformation, recrystallization and surface behavior.

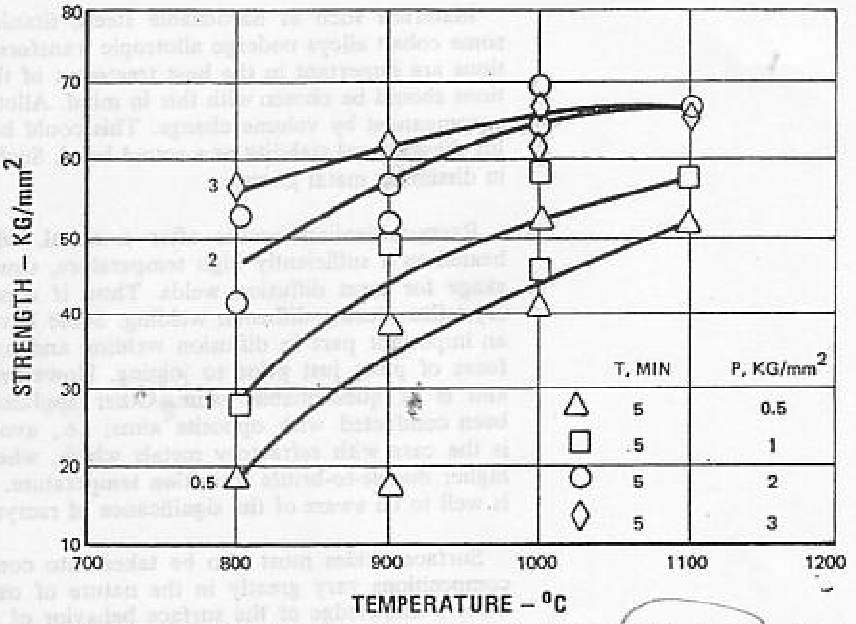


Fig. 52.4.—Effect of temperature and pressure on strength of 0.45%C steel diffusion welds

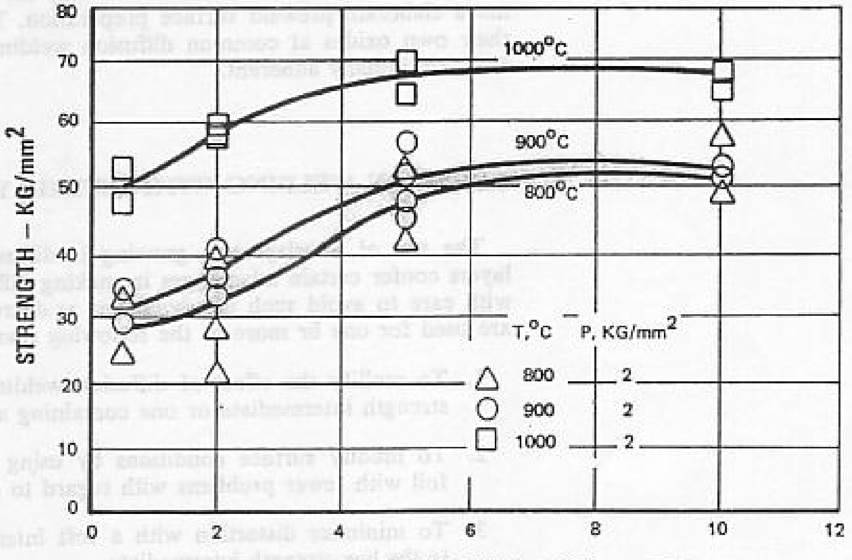


Fig. 52.5.—Effect of time and temperature on strength of 0.45% C steel diffusion welds.

Materials such as hardenable steels, titanium alloys, zirconium alloys and some cobalt alloys undergo allotropic transformation. Because these transformations are important in the heat treatment of the alloy, diffusion welding conditions should be chosen with this in mind. Allotropic transformations are usually accompanied by volume change. This could be an important factor in preserving dimensional stability or a sound bond. Such factors are even more important in dissimilar metal joints.

Recrystallization occurs after a metal, which has been cold worked, is heated to a sufficiently high temperature, usually $> 0.4\ T_m$. This is the same range for most diffusion welds. Thus, if a part has been worked it may recrystallize during diffusion welding. Some have felt that recrystallization plays an important part in diffusion welding and have attempted to cold work surfaces of parts just prior to joining. However, this has not been widely used and is of questionable value. Other applications of diffusion welding have been conducted with opposite aims; i.e., avoidance of recrystallization. This is the case with refractory metals which, when recrystallized, exhibit a much higher ductile-to-brittle transition temperature. Whichever approach is taken, it is well to be aware of the significance of recrystallization.

Surface oxides must also be taken into consideration. Alloys with different compositions vary greatly in the nature of oxides which cover their surfaces. Thus a knowledge of the surface behavior of a metal is needed to assure success. Beryllium, aluminum, chromium and other active elements form tenacious surface oxides. They and alloys containing them are more difficult to weld than those which form less stable oxide films such as copper, nickel, gold, etc. Tenacious films make diffusion welding more difficult and usually require more elaborate preweld surface preparation. Titanium and zirconium dissolve their own oxides at common diffusion welding temperatures even though the films are initially adherent.

DIFFUSION WELDING WITH INTERLAYERS

The use of interlayers is growing in diffusion welding applications. Interlayers confer certain advantages in making diffusion welds but must be chosen with care to avoid such disadvantages as decreased strength or stability. They are used for one or more of the following reasons:

1. To mollify the effect of diffusion welding parameters by using a lower strength intermediate or one containing a diffusing element.
2. To modify surface conditions by using an electroplate or intermediate foil with fewer problems with regard to oxide films.
3. To minimize distortion with a soft interlayer by confining deformation to the low-strength intermediate.
4. To solve alloying compatibility problems when joining dissimilar metals.

Intermediate layers have also been used to promote melting at the interfa in a manner very similar to brazing. Whether this technique is legitimate a part of diffusion welding has been argued without resolve. However, copp interlayers can produce thin, temporary liquid layers in joints between titaniu (or zirconium alloys) to promote joint formation. Boron will do the same wi nickel alloys. When this scheme is used resolidification and homogenization l suitable thermal treatment during or after diffusion welding is called for. Th prevents damaging effects such as in-service melting or embrittlement.

MATERIALS WELDED AND CHARACTERISTICS OF JOINTS

A number of materials in similar and dissimilar combinations have bee joined by diffusion welding, but most applications of this process have bee with titanium alloys, zirconium alloys and nickel-base alloys. As has bee shown, the ease with which properties approaching base metal in each of thes alloys can be attained depends largely on the material characteristics. Gooc joint properties can be attained readily in titanium alloys with relatively low pressure and low homologous temperature. Of help in this case is the low creep strength of titanium alloys and the ability of titanium to dissolve its oxide at the welding temperature.

On the other hand, nickel-base superalloys are most difficult to diffusion weld because their creep strength is high, requiring high interface pressure to bring about intimate contact. In addition, layers of aluminum, chromium and other very stable and insoluble oxides form on the surface to restrict metallic interface contact and further limit welding.

The ability to achieve base metal properties must incorporate the means used for strengthening the base material, i.e., cold work or heat treatment. If cold work is used, then the diffusion weld and surrounding material strength may be irreversibly lowered by the diffusion welding heat treatment. On the other hand, heat treatable alloys can generally be heat treated after welding to restore base metal properties.

Tensile strength alone is not an adequate measure of a diffusion welded joint. An incompletely welded joint may have greater than 95% of the base metal strength but fracture at the interface with almost zero ductility. A properly diffusion welded joint will tolerate significant deformation and fracture will not occur at the joint interface.

Representative diffusion weld property data and parameters are discussed according to material groups in the following section.

Interlayers are used in several ways. Some techniques are shown diagrammatically in Fig. 52.6. Interlayers can be applied in many forms — electroplated to the welded surfaces, as foil inserts, as evaporated or sputtered coatings and even as powdered fillers. They are generally kept thin to minimize the effect of heterogeneity at the weld region after the joint is made. Thicknesses of less than 0.001 in. to more than 0.010 in. are commonly used. It is frequently possible by judicious choice of interlayer to homogenize the joint area either during diffusion welding or by post-heat treatment. Such homogenization, when possible, minimizes the deleterious effects of properties or corrosion resistance that could occur due to the presence of an interlayer.

Interlayers are used to minimize or eliminate problems caused by specific chemical or metallurgical characteristics of the metals to be joined. This requires careful selection of interlayers for specific applications. The most common interlayer used is a less alloyed version of the base metal being joined. For example, unalloyed titanium is frequently used as an interlayer with titanium alloys. Nickel is used as an interlayer with chromium containing nickel-base superalloys. Silver, however, has been used as an interlayer with aluminum alloys.

for

· Ti → Ti alloy
· Ni – Ni super-alloys
· Ag – Al alloys

Another kind of interlayer that has been suggested is one with rapid diffusing elements. Alloys containing beryllium have been suggested for use with nickel-base alloys to increase the rate of joint formation. In such cases the rapid-diffusion atoms usually lower the melting point or form compounds. This may require additional homogenization after welding to assure minimization of harmful effects (e.g., reduced properties or lowered service melting temperature). The value of rapid element diffusion is somewhat questionable, however, due to the tendency to induce diffusion or Kirkendall porosity in welds that have substantially differing diffusion rates among the species that move across or near the joint interface.

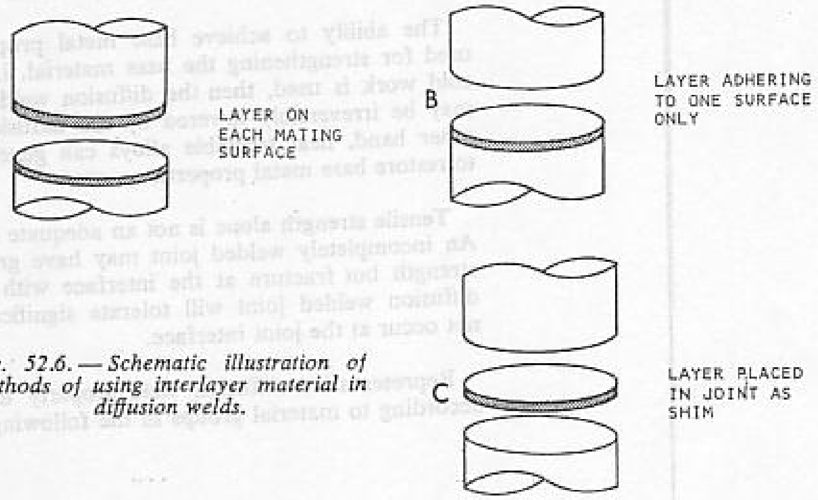


Fig. 52.6. — Schematic illustration of methods of using interlayer material in diffusion welds.

Intermediate layers have also been used to promote melting at the interfa in a manner very similar to brazing. Whether this technique is legitimate a part of diffusion welding has been argued without resolve. However, copp interlayers can produce thin, temporary liquid layers in joints between titaniu (or zirconium alloys) to promote joint formation. Boron will do the same wi nickel alloys. When this scheme is used resolidification and homogenization l suitable thermal treatment during or after diffusion welding is called for. Th prevents damaging effects such as in-service melting or embrittlement.

MATERIALS WELDED AND CHARACTERISTIC OF JOINTS

A number of materials in similar and dissimilar combinations have bee joined by diffusion welding, but most applications of this process have bee with titanium alloys, zirconium alloys and nickel-base alloys. As has bee shown, the ease with which properties approaching base metal in each of thes alloys can be attained depends largely on the material characteristics. Gooc joint properties can be attained readily in titanium alloys with relatively low pressure and low homologous temperature. Of help in this case is the low creep strength of titanium alloys and the ability of titanium to dissolve its oxide at the welding temperature.

On the other hand, nickel-base superalloys are most difficult to diffusion weld because their creep strength is high, requiring high interface pressure to bring about intimate contact. In addition, layers of aluminum, chromium and other very stable and insoluble oxides form on the surface to restrict metallic interface contact and further limit welding.

The ability to achieve base metal properties must incorporate the means used for strengthening the base material, i.e., cold work or heat treatment. If cold work is used, then the diffusion weld and surrounding material strength may be irreversibly lowered by the diffusion welding heat treatment. On the other hand, heat treatable alloys can generally be heat treated after welding to restore base metal properties.

Tensile strength alone is not an adequate measure of a diffusion welded joint. An incompletely welded joint may have greater than 95% of the base metal strength but fracture at the interface with almost zero ductility. A properly diffusion welded joint will tolerate significant deformation and fracture will not occur at the joint interface.

Representative diffusion weld property data and parameters are discussed according to material groups in the following section.

TITANIUM ALLOYS

There is much disagreement in the data presented for diffusion welding of titanium alloys over the amount of pressure to achieve bonding. Typical bonding parameters and corresponding mechanical properties for Ti-6Al-4V are given in Table 51.1. All combinations of pressure and time produce nearly equal ultimate strength, but elongation and reduction in area vary considerably. It will be noted that the higher levels of elongations and reduction in area are associated with welding times of 30 minutes or more. A specimen bonded at 640 psi for 10 minutes had poorer elongation than any of the specimens bonded for 30 to 60 minutes at lower pressures. The surfaces of all these samples were prepared by surface grinding, then hand lapping to achieve flat, smooth surfaces. Specimens that were merely ground exhibited much lower strength and ductility than specimens bonded at the same parameters as those shown in Table 52.1. In such cases, much higher bonding pressure would be required to achieve adequate bonding at the same parameters.

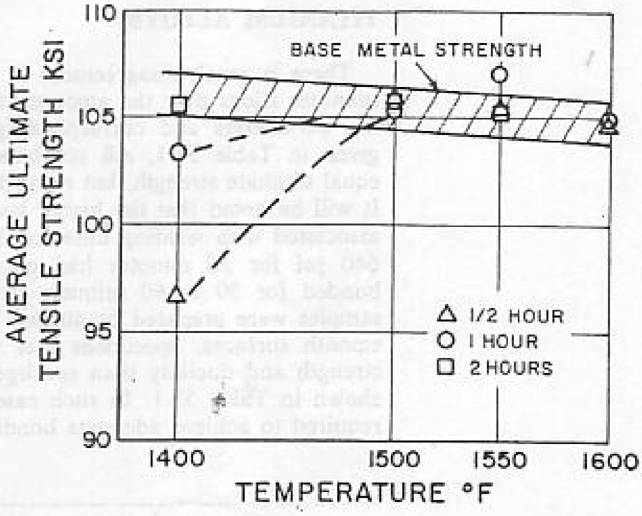
Table 52.1—Parameters and properties of diffusion welds in TI-6AL-4V

Temperature °F	Pressure psi	Time Min.	Upset ΔL/Lo %	Expansion ΔD/Do %	UTS psi	Elongation %	R.A. %
1600	63	60	—0.6	0.6	144,200	18.9	33.8
1600	127	60	—1.6	1.4	144,200	21.5	37.1
1600	253	60	—4.9	4.4	143,800	25.2	43.1
1600	382	30	—4.2	4.3	144,300	24.4	40.6
1600	127	30	—1.3	1.1	143,800	20.7	33.8
1600*	637	10	—5.1	3.2	144,500	4.4	4.2
1600	127	15	—0.5	0.6	144,600	4.7	7.2

Surface preparation consisted of machine grinding, lapping on 600 grit paper, then acid etching.
*Acetone degreased instead of acid etching.
Specimens were 1-in. diam. x 1-in. long cylinders joined end to end.

Joint properties equal to base metal properties have been attained in commercially pure titanium, AMS 4921 at 1600°F (871°C), 1000 psi pressure and 30 min. time, on surfaces prepared with 600 grit metallographic paper. It is likely that base metal properties could be attained at considerably lower pressures. However, time or temperature could not be reduced substantially without sacrificing joint ductility as shown in Fig. 52.7. This also illustrates the insensitivity of tensile strength to parameters while ductility is very sensitive to improper parameters and a good indicator of quality.

Microstructures of specimens prepared in a manner comparable to the test data included in Fig. 52.7 are shown in Fig. 52.8. Notice the relative progression of joint quality as a function of temperature. Further note the similarity of this figure to the schematic drawings of Fig. 52.2, showing progressive improvement in joints.



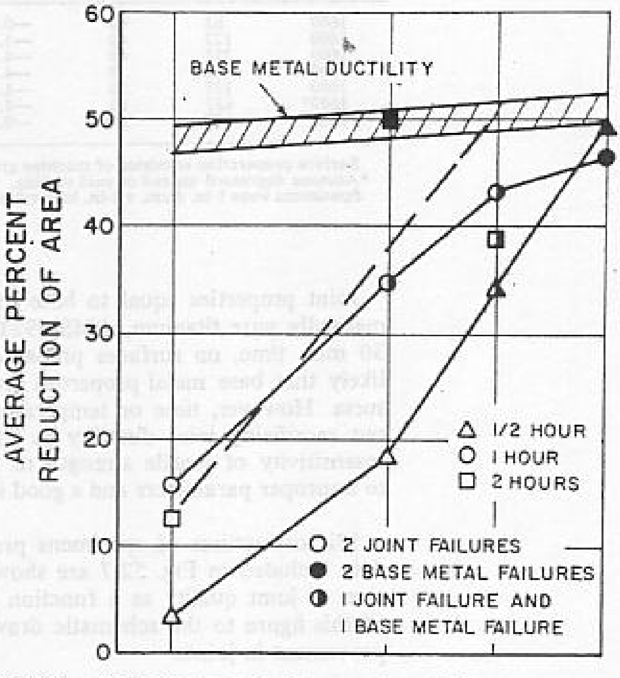
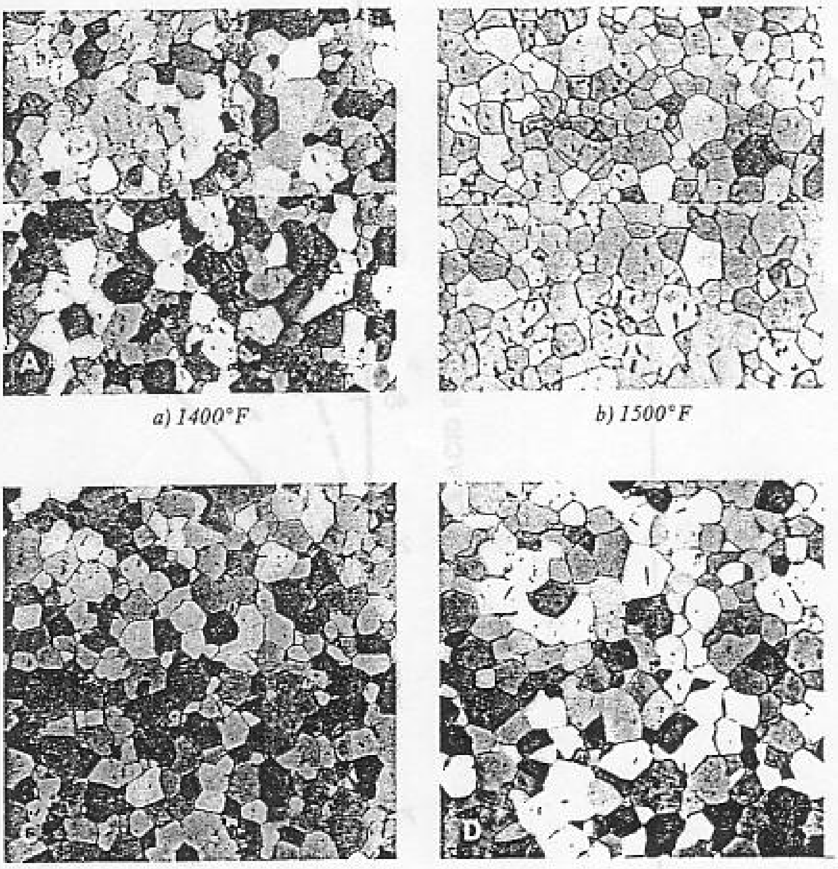


Fig. 52.7.—Plots of average joint properties with temperature for three times



a) 1400°F

b) 1500°F

c) 1550°F

d) 1600°F

Fig. 52.8.—Photomicrographs showing the influence of temperature on bond line quality of joints made at 1000 psi for 1 hour in commercial purity titanium. Note fewer bond line voids and increased grain boundary mobility with increasing temperature. 250X

Surface roughness plays an important role in diffusion welding of titanium alloys. The rougher the mating surfaces the larger the voids, necessitating greater pressure, time or temperature to eliminate them. A post-heat treatment with virtually no accompanying pressure can also be used. The effect of post welding heat treatment at 1600°F (871°C) for Ti-6Al-4V with two different initial grain sizes is shown in Fig. 52.9. The primary mechanism of void elimination is grain boundary diffusion which is more rapid for the smaller grain size. The initial welds are made at 1300° to 1400°F (704° to 760°C) and at pressures of 1750 to 4000 psi.

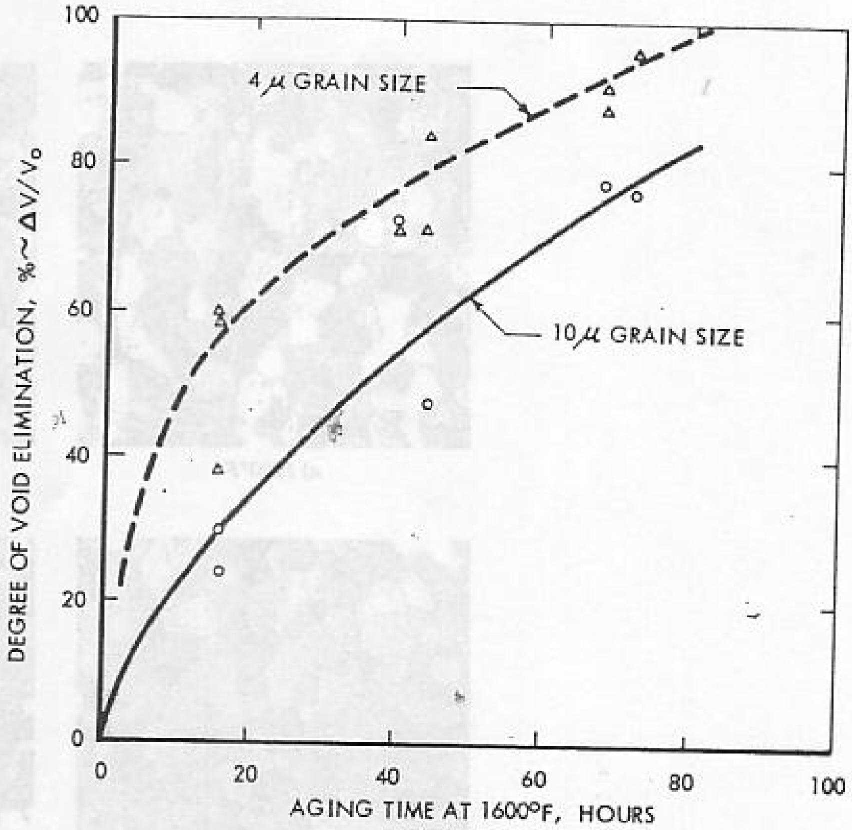


Fig. 52.9.—The effect of post welding heat treatment on void elimination.

NICKEL-BASE ALLOYS

Nickel-base superalloys require higher homologous temperatures and higher pressure to achieve diffusion welding than most other metals. In addition, extra care must be taken in preparing the surfaces to be welded to ensure mutual conformity and cleanliness. Surface oxides formed on these alloys are stable at high temperatures and do not diffuse rapidly within the alloy. During the welding operation at elevated temperature, atmosphere must be carefully controlled whether vacuum or inert gas, to prevent joint interface contamination.

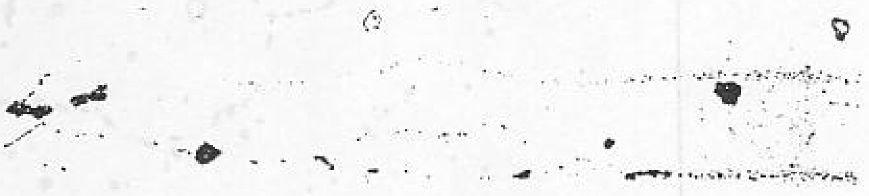
Interlayers in the form of nickel or nickel-alloy foil are frequently used with nickel-base superalloys. These foils, generally about 0.001 in. in thickness, allow surface conformity to take place at lower pressures because of their lower yield strength. They have the disadvantage of forming a low strength plane at the joint unless sufficient diffusion of elements in the base alloy occurs to homogenize the composition.

Welding conditions, surface preparation techniques and post welding treatment for some nickel-base alloys are given in Table 52.2. Fig. 52.10 shows the microstructures of welds in René 41 made with 0.0005 in. Ni-Be foil. The resulting yield strength is equal to that of similarly heat treated solid material, but ultimate strength and ductility are considerably lower. Fig. 52.11 shows the microstructure of diffusion welds in Hastelloy X with no intermediate foil.

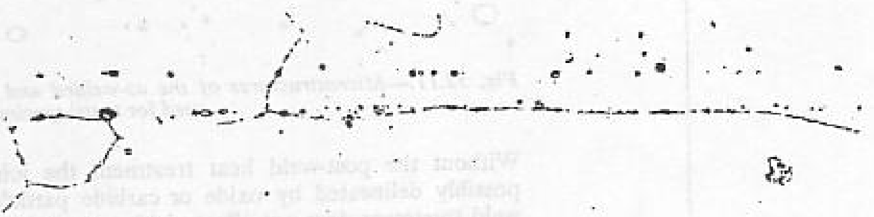
Table 52.2—Diffusion welding parameters and properties for nickel-base alloys

Alloy	Foil Material	Welding Temp. °F	Pressure psi	Post Heat	UTS Ksi	Y.S. Ksi	Elong. % lin.	R.A. %	Test Temp. °F
René 41	Base Metal	C			164	95	26	22	1200
René 41	Ni-Be	2150	*1550	A	120	99	5	6	1200
Hastelloy X	Base Metal				106	43	51	37	Room
Hastelloy X	Base Metal				72	25	50	37	1200
Hastelloy X	Ni-Be	2150	+1300	B	101	43	37	29	Room
Hastelloy X	Ni-Be	2150	+1300	B	58	24	19	20	1200
Hastelloy X	None	2150	+1300	B	106	44	45	33	Room
Hastelloy X	None	2150	+1300	B	71	27	52	40	1200

* 4400 psi initial load allowed to drop to 1550 psi upon reaching bonding temperature, then held at this level to produce a uniform compression rate.
+ 200 psi initial load increased to 1300 psi after temperature is reached.
A 2150°F — 2 hrs. + 1650°F — 4 hrs. — AC + 1400°F — 16 hrs. — AC.
B 2100°F — 3 hrs.
C 2150°F — 2 hrs. + 1650°F — 4 hrs. — AC.
Initial welding time — 2 hrs.
Ni-Be — 0.0005 Ni — 2% Be foil.



A. As Welded Microstructure



B. Post bond heat treated: 2150°F (1177°C)/2 hours + 1650°F (899°C)/4 hours + 1400°F (760°C)/16 hours

Fig. 52.10.—Microstructure of diffusion welded René 41 with 0.0005 in. Ni-Be foil, 2150°F (1177°C), 2 hrs., 1550 psi.

Fig. 52.11.—Microstructures of the as-welded and heat-treated Hastelloy X material used for tensil specimens

Without the post-weld heat treatment the joint interface is clearly evident, possibly delineated by oxide or carbide particles. As with René 41, the post weld treatment does not affect yield strength but enhances ultimate strength and ductility.

Shear tests for a number of iron, nickel and cobalt base alloys are shown in Table 52.3. The samples were joined in a vacuum of less than 1 x 10^{-4} torr with a weight for loading that produced a pressure of less than 1 psi. Joints made with pure nickel or Ni-0.28% beryllium did not weld. An indication of increased shear strength with increased beryllium content in the interface material is indicated for iron and nickel-base alloys.

Table 52.3—Shear strength of single lap joint specimens welded with Ni-Be intermediate alloys

Base Metal	Intermediate Alloy	Welding Temperature °F	Welding Time Minutes	Shear Strength, psi Room Temperature	1500°F
AISI 410	A	2190	2	Not welded	
AISI 410	B	2110	5	31,500	
AISI 410	C	2110	5	46,550	
AISI 347	A	2175	1	Not welded	
AISI 347	B	2110	5	33,300	
AISI 347	C	2110	5	39,600	
AISI 410	D	2075	4		7400
AISI 410	D	2010	10		8800
AISI 410	E	1960	10		8500
AISI 410	E	2100	½		7750
AISI 347	D	2080	10		15,000
AISI 347	D	2100	10		20,000
AISI 347	E	2080	10		18,800
AISI 347	E	2100	10		15,300
Inconel X	B	2110	5	35,850	
Inconel X	C	2110	5	72,900	
Haynes 25	B	2110	5	43,350	
Haynes 25	C	2100	5	31,250	
Inconel X	D	2100	10		50,950
Inconel X	D	2075	10		32,750
Inconel X	D	2010	10		44,400
Inconel X	E	2030	½		37,250
Inconel X	E	1995	10		30,000
Inconel X	E	2010	10		36,500
Haynes 25	D	1940	10		30,250
Haynes 25	D	2055	7		25,850
Haynes 25	E	2100	10		31,000

Intermediate Alloy Composition
A Ni-0.28%Be
B Ni-1.51%Be
C Ni-3.02%Be
D Ni-20%Cr-0.3%Mn-3.0%Be
E Ni-5.8%Be-13.6%Cr-0.1%C-0.3%Mn-3.9%Be

TD-NICKEL AND TD-NICKEL CHROMIUM

Diffusion welding is one good method of joining dispersion strengthened type alloys because it does not cause agglomeration of the dispersed strengthening phase as would fusion welding processes. Agglomeration of dispersed particles reduces the useful strength of the alloy both at room and elevated temperatures. It also reduces ductility. Mechanical properties and parameters of diffusion spot welds made in TD-Nickel on a resistance spot welder are presented in Table 52.4. The values for the post-weld, diffusion-treated joints are about the same as fusion spot welds in TD-Nickel. Without the post-diffusion treatment, strengths are about 20% lower.

STEEL

Although plain carbon steel is not generally diffusion welded because it is more easily welded by other processes, this material can be diffusion welded in air. Tensile properties equal to or greater than unwelded material can be attained for welds. The welds described in Table 52.5 were made in a flash welding machine modified to heat the specimens by self-resistance heating. Before welding, butting surfaces were machined to 12-15 micro-inch finish. Applied pressure was 1000 psi. 1800°F (982°C) appears to be a minimum temperature at which reliable welds can be made. At 2300°F (1260°C) and above, grain coarsening causes weld strength to drop rapidly.

Table 52.4—Parameters and properties of diffusion welds in 0.050 in. TD nickel sheet

Test Temp. °F	Spot Diam. In.	Fracture Load, Lbs.	Shear Stress, psi	Fracture Mode
Room		1980		pullout
1000	0.290	780	11.3	shear
1400	0.290	450	6.8	shear
1800	0.290	250	3.8	shear
2000	0.290	200	3.0	shear
2200	0.290	177	2.7	shear
2400	0.290	150	2.3	shear

Resistance diffusion welding parameters
Heat time — cycles — 2
Cool time — cycles — 1
Number of pulses — 20
Total cycles — 60
Weld current, amps — 17,300
Force, weld, lbs. — 700
Force, forge, lbs. — 1700
Post weld treatment — 2300°F — 1 hr.

Table 52.5—Parameters and tensile properties for diffusion welded 1020 steel

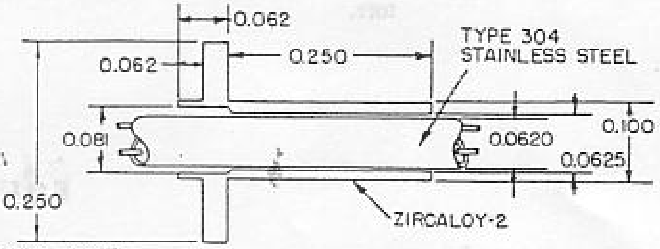
Welding Temp. °F	Welding Time, Min.	Condition a	UTS Ksi	Y.S. Ksi	Elongation %	R.A. %
Base Metal		As rolled	66.1	42.2	34.2	65.6
Base Metal		Normalized	67.4	46.2	35.0	67.8
1700	5	As welded	58.6	46.4	3.5 b	8.0
1700	10	As welded	71.4	47.2	10.3 b	14.6
1800	5	As welded	75.7	52.1	c	59.4
1900	1	As welded	76.9	51.9	c	58.5
1900	15	As welded	70.8	45.2	c	63.5
2200	1	As welded	75.5	48.6	c	60.9
2200	1	Normalized	65.0	44.4	32.0	67.6
2300	(1 sec.)	As welded	78.4	48.8	c	67.0
2300	1	As welded	74.9	50.7	c	60.3
2300	10	As welded	19.4	19.4	0 d	0
2500	(1 sec.)	As welded	80.4	52.6	c	56.9

a — normalizing at 1650°F 1 hr.
b — failure in weld
c — failed outside gage marks
d— extreme coarsening of specimen

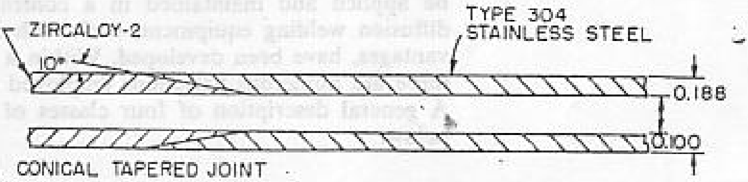
DISSIMILAR COMBINATIONS

Diffusion welding is useful for joining dissimilar metal or alloy combinations particularly where fusion welding is not applicable. For example, if brittle intermetallic phases would be formed because the melting points of the two materials differ widely or if fusion would render a material brittle or lower strength drastically, diffusion welding may be used. Joints between different refractory metals or between refractory metals and the more common metals may be diffusion welded successfully. Interface materials are sometimes used to prevent the formation of brittle intermetallic phases between combinations in which this is possible.

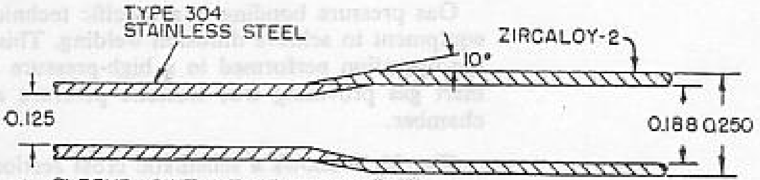
Representative shear strengths and the parameters involved for some dissimilar metal combinations are shown in Table 52.6. Many other dissimilar combinations can be formed but result in brittle intermetallic phases, and in some cases the reaction proceeds very rapidly due to the formation of a liquid phase at the welding temperature. Although these combinations are brittle, useful joints can be attained by allowing for it in the component design. A combination of Zircaloy 2 with type 304 stainless steel is a good example of the situation where a strong useful joint can be made despite the presence of brittle phases. Fig. 52.12 shows the joint designs employed for joining type 304 stainless tube to Zircaloy 2. In the conical, tapered joint pressure is provided by pressing the tubes together axially; thermal expansion of the stainless steel provides the pressure in the tapered sleeve joint.



a. SLEEVE JOINT



b. CONICAL TAPERED JOINT



c. SLEEVE JOINT WITH TAPERED EDGES

Fig. 52.12.—Joint designs used in various applications which utilize a transient liquid phase during diffusion welding

Table 52.6—Parameters and shear strength for dissimilar material diffusion welds*

Material Combination	Interface Material	Temperature °F	Time Hrs.	Test Temp. °F	Shear Strength Ksi	Fracture Location
Cb-1Zr-Cu	Cb-1Zr	1800	4	R.T.	5.8	Cu sheet
Cb-1Zr-Cu	Cb-1Zr	1800	4	—100	7.1	Cu sheet
Cu-316SS	Cu	1800	2	R.T.	14.7	Cu at joint
Cu-316SS	Cu	1800	2	—100	16.0	joint
Cb1Zr-316SS	Cb-1Zr	1800	4	R.T.	10.7	joint
Cb1Zr-316SS	Cb-1Zr	1800	4	600	11.5	joint
Cb1Zr-316SS	Cb-1Zr	1800	4	1200	12.0	joint
Cb1Zr-316SS	Cb-1Zr	1800	4	1800	4.2	joint

* joint pressure applied by tightening a plug on a cylindrical molybdenum capsule. Actual pressure not measured.

Joints between stainless steel and Zircaloy 2 tubing of ⅞ in. diameter and ⅛ in. wall can withstand from 12,000 to 17,000 psi internal pressure when tested hydraulically. The fracture initiates by longitudinal splitting of the Zircaloy tube. Similar joints have withstood 100 pressure cycles between 100 and 3500 psi at 500°F (260°C) and 200 temperature cycles between 100 and 600°F (37.8 to 316°C) without failure.

Parameters for joining Zircaloy to stainless steel range from 1870°F (1021°C) for 30 min. to 1900°F (1038°C) for 30 sec. in a vacuum of less than 5 x 10^{-5} torr.

EQUIPMENT

A wide variety of equipment and tooling is employed in diffusion welding activities. The only basic requirements are that pressure and temperature must be applied and maintained in a controlled environment. Various classes of diffusion welding equipment, each with its own special advantages and disadvantages, have been developed. Within a given class of equipment or approach there are numerous variations employed depending upon the specific situation. A general description of four classes of diffusion welding equipment is given below.

HIGH-PRESSURE ISOSTATIC EQUIPMENT

Gas pressure bonding is a specific technique utilizing high-pressure isostatic equipment to achieve diffusion welding. This technique is basically a hot pressing operation performed in a high-pressure autoclave. The working fluid is an inert gas providing true isostatic pressure application to any part within the chamber.

Fig. 52.13 shows a schematic cross section of a typical gas-pressure-bonding unit. The primary component is a cold-wall autoclave which permits pressures up to 150,000 psi to be employed even though specimen temperature in excess of 3000°F (1649°C) might be employed. Internal cooling is usually provided to aid in maintaining a low wall temperature. Closures in each end provide access to the vessel cavity. Utilities and instrumentation are brought into the vessel through high-pressure fittings located in the end closures. The high temperatures are possible because the heater is located inside the autoclave. Resistance-wound furnaces of varying designs are employed. Alumina or silica insulation is used to reduce heat losses to the wall. Temperature is monitored and controlled by thermocouples located throughout the furnace and vessel. Pressurization is achieved by pumping the inert gas from its storage area through a multistage piston-type compressor. Control of temperature and pressure are independent and any combination of heating and pressurizing rates can be programmed.

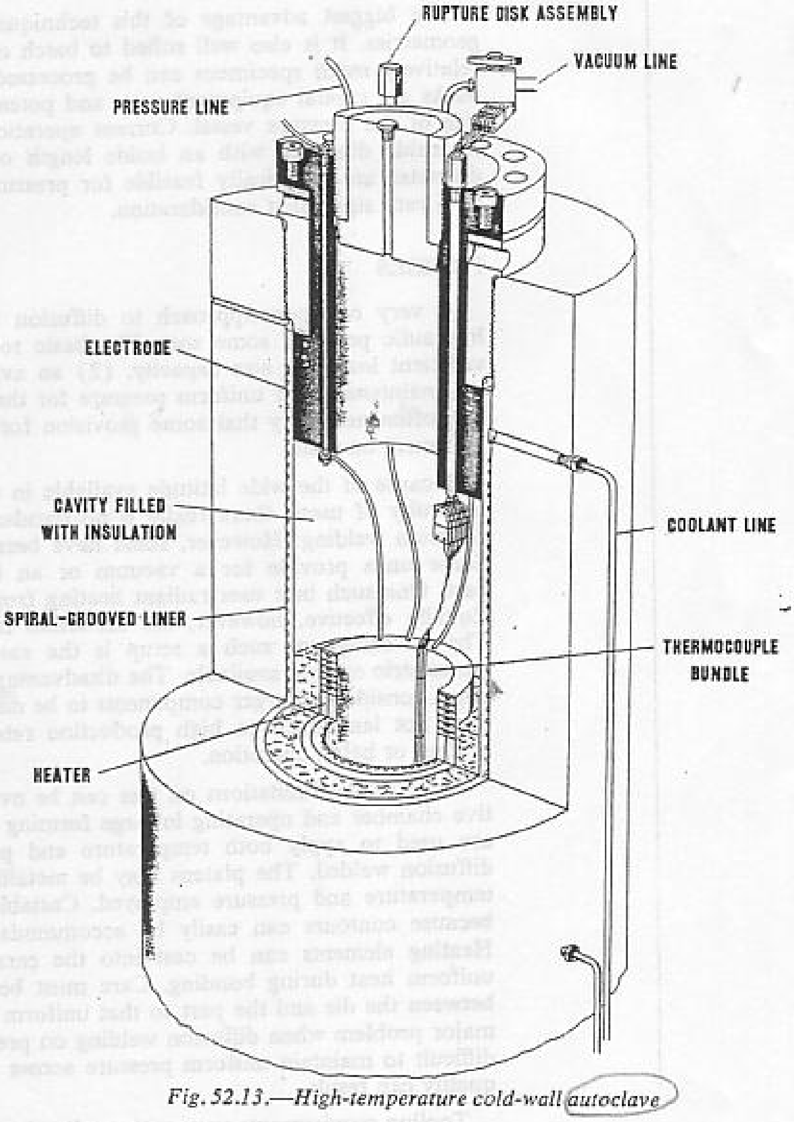


Fig. 52.13.—High-temperature cold-wall autoclave

The tooling required is minimal. The most important consideration is the gas-tight envelope, or can, in which the specimen must be contained. If a leak develops in the can, no pressure differential can be maintained and no useful work can be accomplished. Internal tooling may also be required if cavities exist in the geometry to be diffusion welded. Usually sufficient pressure is applied so that plastic flow will occur until all void space is filled. If proper tooling is not provided, the structure might collapse. With proper conditions essentially no deformation occurs and no change in dimensions will occur during the operation.

The biggest advantage of this technique is the ability to handle complex geometries. It is also well suited to batch operations where large quantities of relatively small specimens can be processed simultaneously. The major drawbacks are capital equipment costs and potential size limitations imposed by the size of the pressure vessel. Current operational equipment ranges up to 36 in. in inside diameter with an inside length of nine feet. Units up to 10 ft. in diameter are technically feasible for pressures up to 10,000 psi but cost may be a very significant consideration.

PRESSES

A very common approach to diffusion welding employs a mechanical or hydraulic press of some sort. The basic requirements for the press are: (1) sufficient load and size capacity, (2) an available means for heating and (3) the maintenance of uniform pressure for the required time. It is also desirable and often necessary that some provision for protective atmosphere around the weldment be made.

Because of the wide latitude available in using presses, and the far-reaching ingenuity of users, there really is no standard equipment established for press diffusion welding. However, some have been produced for sale commercially. Some units provide for a vacuum or an inert atmosphere to surround the part. One such unit uses radiant heating from tungsten-mesh heating elements. Equally effective, however, are induction heating and self-resistance heating. The advantage of such a setup is the ease of operation and the excellent parametric control available. The disadvantage is the practical limitation of size when considering larger components to be diffusion welded. Also, this approach does not lend itself to high production rates; it is not suited to rapid turnaround or batch operation.

Some of the limitations on size can be overcome by eliminating the protective chamber and operating in large forming or forging presses. Heated platens are used to apply both temperature and pressure to the components to be diffusion welded. The platens may be metallic or ceramic depending upon the temperature and pressure employed. Castable ceramics are particularly useful because contours can easily be accommodated without extensive machining. Heating elements can be cast into the ceramic die to provide the required uniform heat during bonding. Care must be taken to ensure close tolerances between the die and the part so that uniform pressure will be applied. This is a major problem when diffusion welding on press type equipment. It is extremely difficult to maintain uniform pressure across the section and variations in weld quality can result.

Tooling requirements vary with application. If no lateral restraint is provided upsetting may occur during processing. In such cases, lower pressures are usually required. The process is quite similar to closed-die press forging except that lower pressures and longer times are employed. Because of the time factor, heated dies are required and die materials become a problem. The die must be able to withstand both the temperature and pressure as well as be compatible with the material to be diffusion welded. Interaction between the part and the die can be controlled by stopoff agents in many instances. Atmosphere protection is often achieved by sealing parts in evacuated metal cans which are flexible and conform to die shapes.

Equipment that can be adapted to diffusion welding applications is frequently available. Nearly every sizeable manufacturing or development organization has a press of some type that can be modified to perform diffusion welding. Also, because no requirement for closed containment exists, quite large structures can be handled. A major limitation is geometry since only uniaxial pressure application is practical. This approach is also quite slow and may not be scaled readily to high production rates.

The further development of diffusion welding in the next decade will undoubtedly lead to its adaptation for use in higher production runs. An example of this type of development can be found in current interest in hollow gas turbine components. Such an application could lead to manufacturing capabilities of thousands of parts per month that would lend itself to more rigorous economic analysis. Until such productivity is achieved, however, it will not be possible to state meaningful cost comparisons of diffusion welding to other joining methods and the factors of quality, reliability, and ability to make otherwise impossible parts will dominate.

RESISTANCE-DIFFUSION-WELDING EQUIPMENT

Excellent application of resistance welding equipment has been made to diffusion welding. In general, no modification of standard equipment is necessary to achieve successful resistance diffusion bonds. Both spot- and seam-diffusion welding have been accomplished, although seam-diffusion welding is not so well developed. Closed-loop control systems are being developed that provide fast and reproducible results.

As in standard resistance welding, selection of electrode materials is important. The electrodes must be electrical conductors, possess high strength at bonding temperatures, be thermal shock resistant and resist sticking to the materials to be bonded. There is no universal electrode material because of potential interaction with the workpiece. Each system must be carefully evaluated from a metallurgical compatibility standpoint to ensure success.

One modification often employed is the addition of an atmospheric control device. This usually takes the form of a small chamber surrounding the electrodes to provide an inert atmosphere or vacuum during the diffusion welding process. The seam welder is less sensitive to potential weld contamination by the atmosphere because of the nature of the applied force. This tends to force the contamination by the atmosphere out of the joint as the rollers move forward.

The biggest advantage in using this type of equipment for diffusion welding is the speed at which joints can be made. Cycle times are measured in seconds rather than hours (as with other diffusion welding approaches). However, it must be recognized that only a small area is welded at a time and the preparation of large weld areas becomes time consuming and requires numerous overlaps to achieve welding over an area larger than the electrodes.

SPECIALIZED EQUIPMENT APPROACHES

In addition to the major equipment discussed above, engineers have devised numerous other approaches to diffusion welding. An incomplete listing of apparatus would include retorts, fixtures, dead weights and differential thermal expansion fixtures. In all cases, the apparatus used provides the pressure required and is used in conjunction with a separate heating source.

Of those listed, retorts have been the most important to date, especially in the fabrication of honeycomb-type geometries. The components to be joined are placed in the cavity of a sheet-metal stainless steel retort. The retort is welded leak-tight except for purge lines that can be used for atmosphere control and/or evacuation. The differential between the atmospheric pressure outside and a reduced pressure inside the retort is often sufficient to permit satisfactory diffusion welding. Additional loading can be provided by an overpressure of gas or by a press. The heat may be supplied by furnace, heating blanket or heating lamps. The simplicity of tooling and equipment is a major advantage of this approach, especially if no supplemental loading is required for the geometries involved.

INSPECTION AND TESTING OF WELDS

Establishing the quality of a diffusion weld is difficult with current nondestructive testing procedures. This is due to the nature of the diffusion weld. Usually little or no porosity exists if the weld is made by properly developed procedures. The main defect is lack of grain growth across the original interface. Efforts to distinguish complete intimate contact, but with no grain growth from a perfect bond, have not been very successful.

Radiography, eddy current and thermal methods have proven relatively unsatisfactory inspection methods for most diffusion welding applications. Dye penetrant methods are relatively successful for edge inspections but, of course, they are of no value for internal inspection.

Ultrasonics have proven the most useful for internal weld inspection especially if an actual hairline separation exists. The sensitivity varies with the material being tested, the frequency used, the skill of the operator, and the degree of sophistication of the equipment. In general, defects less than 0.1 in. in diameter are difficult to locate and a practical limit of about 0.04 in. exists. With specialized methods and very sophisticated equipment it has been reported that defects down to an equivalent diameter of 0.005 in. can be detected in some materials. These approaches cannot be considered routine, however, and only work under special conditions.

Ultrasonics are still limited in differentiating between complete intimate contact and true diffusion welding. The only method available to assure complete welding is metallographic examination. Since this is a destructive test, it cannot always be performed on the part in question. Fortunately, the diffusion welding process is reproducible if process control is exercised. Random destructive sampling coupled with ultrasonic inspection will provide a high confidence level in all parts produced. This approach has been used in production with proven yields in excess of 95%, including parts deliberately cut up for evaluation.

APPLICATIONS

Diffusion welding to date has found most of its application in the atomic energy and aerospace industries. Wide application in general industry has not yet been achieved. This is understandable because the approach produces very high performance joints but generally at a premium in cost. Additional research and the education of process engineers in diffusion welding should mean increased application in the next few years.

In the atomic energy field, diffusion welding is being used in the fabrication of reactor components. One of the first production uses for diffusion welding was in the fabrication of fuel elements for the first commercial pressurized water reactor at Shippingport, Pennsylvania. The unique requirements of cladding a ceramic with a metal with 100% joint efficiency, no changes in dimensions or corrosion resistance and high reliability made this an ideal application of diffusion welding by the gas-pressure-bonding method.

Applications in the aerospace industry have developed in recent years. Diffusion welding is being used for fabrication of honeycomb, rocket engines, turbine components, structural members, composites and laminates. These applications are still in advanced development, but they are finding actual production applications.

A particularly attractive application of diffusion welding is illustrated in Figure 52.14. The helicopter rotor hub shown was built up from titanium alloy sheets and press diffusion bonded to form a solid mass. The resulting rotor hub possesses fine grain size and excellent mechanical properties even in the very center of the part. This approach might replace large forgings for high-performance applications since forging operations usually result in poorer properties in the center of the forging.

COMPOSITES

One of the most promising fields in which diffusion welding seems destined to play a significant role is that of composite materials. Composites are materials which contain high strength or high modulus filamentary materials to reinforce common matrices in a manner which resembles the principles used in reinforced concrete. By this technique higher strength, higher modulus materials can be made. Reinforcing materials commonly used are carbon, boron, alumina, silicon carbide and others. The aviation industry has applied impetus to development of these materials because of their light weight and high specific modulus and strength.

In metal matrix composites, i.e., where the material which is reinforced is a metal, diffusion welding is one of the most common means for producing the shapes which contain fiber and matrix.

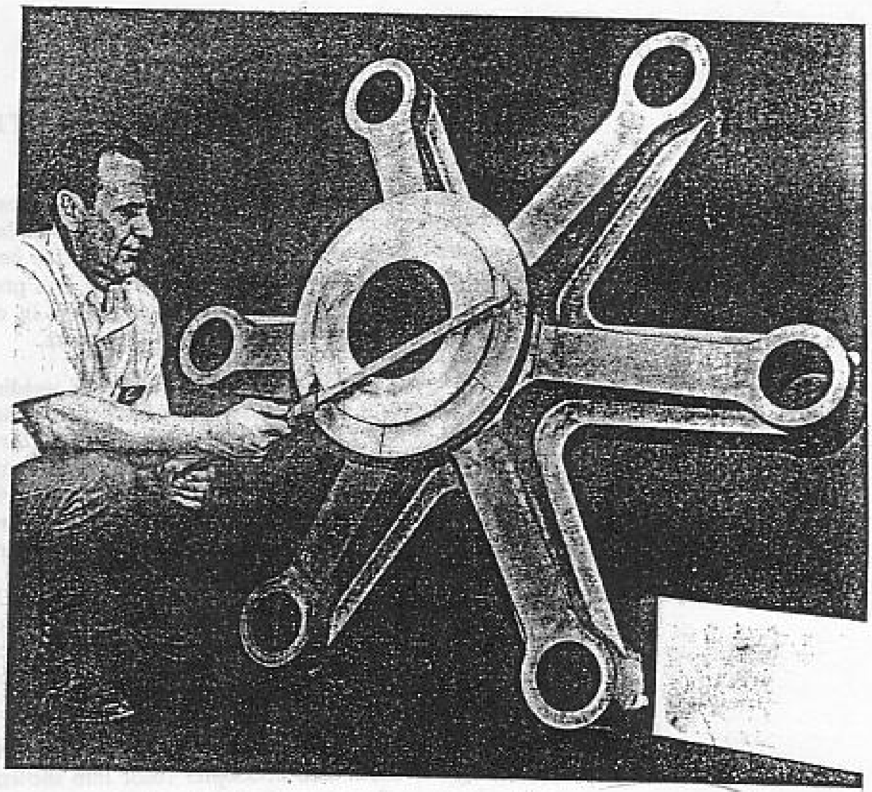


Fig. 52.14.—Press diffusion bonded helicopter rotor hub

Al

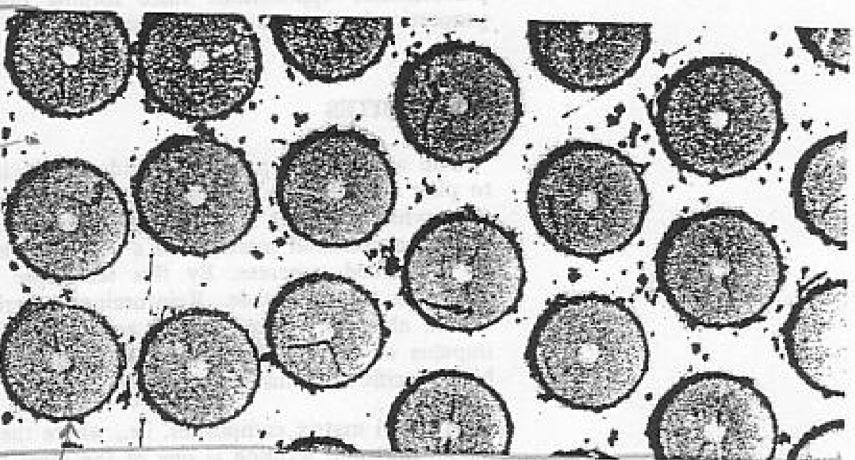


Fig. 52.15.—Diffusion-welded composite

carbon fiber

carbide coated by silicon carbide