Superplastic Forming and Diffusion Bonding of Titanium Alloys

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Abstract. New and advanced fabrication methods for titanium components are emerging today to replace age-old fabrication processes and reduce component cost. Superplastic forming and diffusion bonding are two such advanced fabrication technologies which when applied individually or in combination can provide significant cost and weight benefits and a rather broad manufacturing technology base. This paper briefly reviews the state of understanding of the science and technology of superplastic forming of titanium alloys, and their diffusion bonding capability. Emphasis has been placed on the metallurgy of superplastic flow in two phase titanium alloys, the microstructural and external factors which influence this behaviour.

1. Introduction

Among the high strength materials of interest in the aerospace industry, titanium holds a position of foremost importance. It is a powerful element for construction of space vehicles, and commercial and military aircrafts. Present day aircrafts are built mostly of aluminum, with titanium being used in areas where very high temperatures are encountered and unyielding strength is required. Although aluminum has a lower density than titanium, it loses its strength rapidly at elevated temperatures, while titanium retains its strength. Titanium is virtually corrosion-free whereas aluminum is not. Since corrosion has proved to be an expensive factor in aircraft maintenance, titanium is a highly desirable material in the fabrication of key aircraft structural components.

However, the costs associated with processing and fabrication of titanium are high. The basic metal costs eight times as much as aluminum. To the man on the production floor, the low ductility and high elastic springback make this a difficult metal to work with. It adds up to cost.

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The workhorse titanium alloy for the aerospace industry is the Ti-6Al-4V alloy, for which most structural parts have been and are still made primarily by conventional fabrication techniques. For example, a modern aircraft such as Concorde supersonic transport uses frames, stringers, longerons, fittings, bulkheads and shear webs, all fastened together with mechanical fasteners, rivets and bolts and the entire structure using decades-old design concepts.

Today, aircraft manufacturers are keenly aware of a basic economic formula: as the price of labour and materials goes up, the cost of an aircraft escalates even faster as old fabrication methods are used to build more efficient modern structures. A primary factor driving the advancement to technology in the design and construction of aircraft structures is the need to decrease costs in both the design and engineering processes, as well as in their operation and maintenance. Superplastic forming is an emerging advanced technology that promises significant reductions in both procurement and life cycle costs. Ti-6Al-4V alloy, one of the high strength engineering alloys to first exhibit superplastic properties, is considered suitable for many applications in aircraft and spacecraft structures.

Superplasticity, i.e., the ability to stretch metals to extremely large (several hundred per cent) elongations before failure, can be effectively utilized for forming extremely complex shapes to functionally replace heavy multicomponent structures of an old design. Since titanium is also diffusion bonded to achieve excellent bond strength, this technology, by itself, and in combination with superplastic forming, provides unique design capabilities for advanced structures. A review of these emerging advanced technologies is presented in this paper.

2. Superplastic Forming of Ti Alloys

Superplastic forming (SPF) is receiving increasing interest as a process capable of radically extending the limitations associated with the more conventional processes. The large tensile elongations and typically low flow stresses associated with a superplastic metal permit the forming of complex parts using methods and forming pressures not previously possible. The potential of forming processes designed to take advantage of this unique capability has been widely recognized, and extensive studies have been conducted over the past 20 years to provide a better understanding of mechanisms and requirements for superplasticity, and to develop new and modified alloys and processing methods which will further extend the number of superplastic materials available.

Superplasticity, as referred to in this paper, is described as the capability of a material to undergo extensive tensile deformation. In a tensile test, elongations of more than 200-300 per cent are generally referred to as superplastic deformation. A characteristic common to all superplastic materials is a high degree of sensitivity of the flow stress to strain rate with flow stresses decreasing with decreasing strain rate.

High
strain rate sensitivity is typically associated with a fine grain microstructure. The relationship between strain rate sensitivity, \( m \), \((\varepsilon/\ln \dot{\varepsilon})\) and tensile elongation is understood well theoretically as resulting from the resistance to neck growth.\(^*\) For high values of \( m \), local increases in strain rate during the necking process generate sufficient local hardening to retard neck growth. Calculations of tensile ductility as a function of \( m \) have shown\(^*\) that Woodford ductility plot (Fig. 1) for many different superplastic metals is theoretically predictable-reasonably well.

The strain rate sensitivity of metals arises from the viscous nature of the deformation process. The viscosity is a result of the resistance offered by internal obstacles within the material. In dislocation glide and climb processes, the obstacles could be a fine dispersion of second phase particles within the grain interior between which the dislocations are bent around and moved\(^4\). At high homologous temperatures, the high diffusivities around the grain boundary regions can lead to grain boundary sliding.\(^4\) This is a Newtonian \((\sigma \propto \dot{\varepsilon})\) process with a rate sensitivity index of 1. The overall rate sensitivity of a material is then a result of the rate sensitivities of the grain boundary and the grain interior, and can change from a high value of near 1 at the lower strain rates to 0.2 to 0.3 at the higher rates.\(^5\) At very slow strain rates, however, a low value (-0.2) has often been reported.\(^6\)

The superplasticity of \( Ti-6Al-4V \) has been studied fairly extensively by several investigators.\(^7-9\) Over 1000 per cent elongation has been reported in this alloy under optimum conditions. As seen from Fig. 1, extremely large elongations are possible for \( Ti-6Al-4V \) in comparison to most other superplastic metals. The optimum condi-
tions have been reported to be a forming temperature within 870° and 927°C and grain size in the range of 4-8 μm. The stress/strain rate data for superplastic materials is generally obtained by means of step strain rate (or strain rate cycling) method, although several other methods are reported in the literature. The data is often described by a sigmoidal curve in log σ vs log ε plot, having lower slopes (m) at lower (<10⁻⁵ s⁻¹) and higher (> 10⁻³ s⁻¹) strain rates, and a higher slope (-0.8) in between. Other results suggest that the slope in the intermediate ‘superplastic’ range is 0.5 and it continues to increase toward unity at lower strain rates. This latter behaviour is believed to result from diffusional creep processes while the former from power law (or grain matrix) creep. It is this intermediate regime of superplasticity which is of current interest. The measurement of strain rate sensitivity is somewhat variable from one investigator to another; however, values in the range of 0.5-0.8 have been reported which leads to the large observed elongations.

2.1 Effect of Ti Microstructure on Superplasticity

For superplastic deformation, an alloy normally requires (a) a fine grain microstructure, usually two phases, (b) a metallurgically stable microstructure at the superplastic temperature with little or no grain growth, (c) deformations at 0.5 to 0.8 Tₐₘ (the absolute melting point temperature), and (d) a controlled strain rate, usually in the 0.01 to 0.0001 in./in./s (mm/mm/s) range for maximum superplasticity.

Microstructure plays an important role in determining the superplastic properties of titanium alloys. As in all superplastic materials grain size is the single more important parameter, but because of the complexity of microstructures which can be obtained in almost all titanium alloys, grain size by itself is an inadequate description of microstructure. Because most superplastic titanium alloys are two-phase α-β alloys (at least at the forming temperature), some measure of relative volume fraction of the phases present is clearly necessary.

Several additional microstructural variables which can also be important in certain situations are: grain size, grain growth kinetics, grain aspect ratio, grain size distribution, volume fraction of phases and texture of the alpha phase. Titanium alloys, which have useful degrees of superplasticity, are generally two phase alloys at the superplastic temperature. Grain size is known to strongly influence the superplasticity of the Ti-6Al-4V alloy and an example is presented in Fig. 2 where the flow stress and strain rate sensitivity: m, are graphed as functions of strain rate for a range of grain sizes. As is typically found for most superplastic materials, increasing grain size increases the flow stress and tends to reduce the maximum m value as well as reducing the strain rate at which the maximum m is observed.

2.1.1 Grain Size Distribution: Additional complications arise when grains are not equi-axed, or vary widely in size. The importance of this issue on superplasticity of Ti alloys was studied by Paton & Hamilton by examining a series of heats of Ti-6Al-4V alloy
of similar composition but widely differing microstructures. It was shown that although a fine grain size was important in achieving good superplastic properties, grain aspect ratio, and grain size distribution could also have significant influence on superplastic parameters, particularly total elongation. In their study, the heat of material with the lowest elongation also had a small average grain size. This apparent anomaly was ascribed to the presence of a few very large \( \alpha \) grains in that material (Heat 2). The influence of a nonuniform grain size on superplasticity has been examined analytically by Ghosh & Raj\(^2\) by modeling flow in a material with a duplex grain size. This work suggests that the transition from dislocation controlled flow to diffusion controlled flow was much broader and occurs over several orders of magnitude if a distribution in grain

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**Figure 2.** Stress vs strain rate plots for a variety of grain sizes of Ti-6Al-4V, and the corresponding strain rate sensitivity, \( m \), at the superplastic temperature.
A trimodal grain size has the effect of reducing peak “m” at intermediate strain rates. The origin of this effect is believed to be the uniform strain rate constraint applied to all regions of the material regardless of grain size, thereby setting up an internal stress distribution with the large grains supporting larger stresses than the small grains. A uniformly fine grain size would, therefore, appear to be beneficial.

2.1.2 Grain Growth Kinetics: Although a fine uniform grain size is clearly desirable for optimum superplastic properties, other factors are of importance if large uniform elongations are to be obtained. Among these other factors is grain size stability. Two-phase Ti alloys have some degree of inherent stability imparted by the equilibrium volume fraction of the phases present. Grain growth as a result of both thermal and straining effects can still have a significant influence on superplastic deformation in Ti alloys, however, resulting in an apparent “strain hardening” effect. This subject will be discussed in detail in a letter section. While strain induced grain growth in Ti-6Al-4V alloy at superplastic temperature may be rapid, statically the α-γ fine grain structures reasonably resistant to grain growth. This is possibly because grain growth in this material requires long range diffusional transport to maintain the chemical equilibrium of the individual phases (α: Al-rich, γ: V-rich).

2.1.3 Grain Aspect Ratio: Grain aspect ratio effects on superplasticity in Ti alloys are recognized as being important, but very little quantitative work has been accomplished. Processing of Ti alloys above the β transus leads to development of an acicular microstructure which is highly detrimental to superplasticity, resulting in both high flow stresses and low “m” value. The flow stress can be an order of magnitude higher than that for the same alloy having an equiaxed microstructure giving rise to difficulties in superplastic forming, diffusion bonding, forging and powder compaction operations. Acicular β microstructure leads to long hot pressing times to achieve full density and long diffusion bonding times in comparison to more equiaxed microstructures. The reason for this high flow stress in such microstructures is easy to visualize in terms of a grain switching mechanism in which grains exchange neighbours in corner locations as they flow past one another like grains of sand flowing through a nozzle. Such a mechanism would be difficult or impossible for plate shaped grains at least if continuity is to be maintained.

2.1.4 Phase Ratio: As already stated, the majority of Ti alloys having superplasticity properties of interest are dual phase (α + β) alloys. A major variable influencing elevated temperature flow properties is the relative volume fraction of the α and β phases. This is a variable controlled by a combination of test temperature and alloy composition, higher concentrations of β stabilizers such as H, V and Mo leading to higher volume fractions of β phase at a given temperature. Considering the anomalously high diffusivity of β Ti which is almost two orders of magnitude higher than in α Ti, and the significant contribution that diffusive flow is known to have on to the total strain rate, one might assume the superplasticity would be enhanced by a higher β volume fraction. This is in sharp contrast with experimental results employing hydrogen.
doping to increase the volume fraction of \( \beta \) phase in a \( Ti-6Al-4V \) alloy.\textsuperscript{15} The data presented in Fig. 3 for elongation in a series of \( \alpha-\beta \) \( Ti \) alloys (\( m \) value 'plots are also similar) have been compiled and plotted as a function of volume fraction \( \beta \). It can be seen that maximum superplasticity as measured by elongation (and \( m \)) occurs at around 40 per cent \( \beta \) phase and drops off significantly on either side of this value.

![Figure 3. Elevated temperature ductility as a function of P-phase content for several Ti alloys.](image)

It has been explained earlier\textsuperscript{15} that an increase in \( \beta \) phase volume per cent is associated with an increase in \( \beta \) grain size in the equilibrium temperature range, and thus the beneficial effect of higher \( \beta \) diffusivity is lost by the coarseness of \( \beta \) grains. This effect is shown in Fig. 4, where the flow stress at the lower strain rates actually increases with increasing \( \beta \) volume per cent (or hydrogen content). The flow stress at the higher strain rates, which is relatively insensitive to grain size, however, does decrease with hydrogen addition—an effect useful in forging at the higher strain rates. Unfortunately, the resulting lower \( m \) values are responsible for poorer tensile elongation.
INFLUENCE OF \( \beta \) STABILIZER

![Diagram showing a and \( \beta \) grain sizes and volume fraction relationship.]

**Figure 4.** (a) Theoretical influence of F-volume fraction on equilibrium F-grain size. (b) Experimentally determined effect of hydrogen doping on stress vs strain rate plots for Ti-6Al-4V at 927°C.

With the anticipation that higher volume fractions of \( \beta \) phase at a given temperature might enhance superplasticity, several workers have added \( \beta \) stabilizers to Ti-6Al-4V. However, the results do not show extensive superplasticity. A further method of modifying conventional alloys to enhance superplasticity has been pursued by Mahajan et al. where hydrogenation was employed to increase the volume fraction of \( \beta \) phase.
Subsequent to forming, this hydrogen was removed to recover the original mechanical properties of the base alloy. The basic problem found with these approaches is excessive grain growth effect during the superplastic deformation.

### 2.2 Effect of Temperature

Temperature is a fundamentally important parameter for superplasticity, and it is generally agreed that the temperature must be in excess of about $0.5 \, T_m$, where $T_m$ is the absolute melting point. This criterion holds for titanium alloys as well as for other alloy systems. Superplasticity is reported for temperatures as low as about $800^\circ C$\textsuperscript{18,20} to as high as $1030^\circ C$\textsuperscript{7} which are approximately 0.47 and 0.61 of the melting point. The need for the elevated temperature is based on providing the diffusion mechanisms necessary for permitting the superplastic deformation to proceed. At temperatures below roughly $0.3 \, T_m$, the diffusion kinetics are too sluggish and deformation occurs by more dislocation creep and climb mechanisms for which the strain rate sensitivity of flow stress ($m$) is less than 0.3.

The effect of temperature on the diffusion kinetics of titanium alloys is perhaps more complex than other alloy systems because of variations in microstructural phase content. Depending on the alloy and temperature, $\alpha$, $\beta$, or $\alpha + \beta$ phases may exist. Since the $\beta$ has a diffusivity approximately two orders of magnitude higher than does the $\alpha$ phase, and grain growth tendencies are also influenced by temperature, the temperature dependence of the superplastic properties are quite complex in these alloys.

The elongation changes with temperature are exemplified in Fig. 5 for fine grain $Ti-6Al-4V$ alloy\textsuperscript{?}. As shown in this figure, there is a limited temperature range over which superplastic ductility is observed, a characteristic typical of superplastic titanium alloys although the superplastic temperature range can vary with the alloy. The effect of temperature on the $Ti-6Al-4V$ and $Ti-5Al-2.5\, Sn$ alloys was first evaluated by Lee and Backofen\textsuperscript{7}. They found that the superplastic behavior for $Ti-6Al-4V$ alloy was confined within a temperature range of approximately $815^\circ$-$980^\circ C$; and for the $Ti-5Al-2.5Sn$ alloy within $1000^\circ$-$1030^\circ C$. In the case of these alloys, the upper limit of superplasticity corresponded approximately with the transformation to all $\beta$ (the $\beta$ transus temperature), a temperature above which titanium alloys are known to undergo very rapid grain growth.

The effect of temperature on superplasticity can be characterized by the activation energy for deformation. In most such materials, the activation energy is found to be close to that for grain boundary self-diffusion, supporting the concepts that relate superplastic flow to grain boundary diffusion processes. While there are only limited data reported on activation energies for titanium alloys\textsuperscript{7,11,20}, there appears to be a difference in the characteristic indicated by the $\beta$ and $\alpha/\beta$ alloys. The determination of the activation energy, $Q$, for the $Ti-8Mn$ alloy\textsuperscript{20} showed $Q$ to be about 20 kcal/mole, a value which is consistent with the expected value of grain boundary diffusion. However, for the $\alpha/\beta$ alloys, these values are consistently above the activation energy even
for bulk self-diffusion and do not, therefore, correspond to values expected for grain boundary diffusion, values which are generally about half that of bulk diffusion.

2.3 Processing for Superplasticity

The strong influence of microstructure on superplasticity would indicate that thermomechanical processing to achieve a fine equiaxed microstructure in Ti alloys would be a worthwhile endeavour. In contrast to the approach taken on many other superplastic alloy systems where processing to achieve a fine grain size has been studied in great detail, there is only a limited amount of work reported on Ti alloys. This appears to be largely due to the fact that standard mill processing generally results in acceptable superplasticity. Certain alloys do not necessarily possess microstructures favourable to superplasticity and have been found to respond to special processing. An example of such an alloy is Corona 5, and recent work reported by Froes et al. In their work a final cold rolling sequence involving approximately 40 per cent reduction followed by annealing at 845°C for 4 hours was employed to arrive at a fine (2 μm) equiaxed microstructure which provided good superplastic properties. This was in comparison to material produced by standard unidirectional warm rolling techniques which produced a nonuniform highly directional structure, with inadequate superplasticity.

Processing of Ti alloys frequently results in a strong texture in the α phase, particularly if warm rolling is employed. Kaibyshev et al. studied the effect of texture in

![Figure 5. Tensile ductility vs temperature for Ti-6Al-4 alloy (VT6)](Ref. 213.)

\[ \text{GRAIN SIZE} = 3.6 \text{ pm} \]
a Ti-6.5Al-5V alloy with two textures, one strong and one weak. In both cases the alloy microstructure was equiaxed and fine grained, with an average grain size of 3.5 pm. They found a pronounced effect of texture, particularly at lower temperatures. At 200°C, elongation of the textured samples was 3 times that of the textureless one, while at 900°C elongation was only 1.07 times greater. This greater difference in behaviour at 200°C is consistent with the implication that slip should provide a greater contribution to total elongation at lower temperatures, while grain boundary sliding and diffusion processes, presumably unaffected by slip and texture, would be the most significant contributor at higher temperature.

2.4 Beta Titanium Alloys

The β-Ti alloys deserve special comment since their high temperature deformation characteristics are different from the α/β alloys. These alloys, because of their low β transus temperature, are single phase in the temperature range normally considered to be the superplastic temperature range for Ti alloys. As such, they undergo very rapid grain coarsening at these temperatures, and therefore much of the “superplastic” data reported are for grain size well in excess of that considered necessary for superplasticity. The deformation mechanisms corresponding to these large grain sizes are therefore different from those for the fine-grained two-phase materials.*

The β and near β alloys have been studied by several investigators2324 and the results of these studies are generally consistent in terms of the superplastic properties, regardless of the specific alloy. The resulting ductility is low in comparison to the α/β alloys, and is indicated in the Table 1. In some of the earliest studies on the superplasticity in β alloys, Griffiths and Hammond25 observed that some extended ductility was

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Test Temp (°C)</th>
<th>Strain Rate (sec⁻¹)</th>
<th>m</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6Al-4V</td>
<td>840-870</td>
<td>1.3 x 10⁻⁴ to 10⁻³</td>
<td>0.75</td>
<td>150-1 110</td>
</tr>
<tr>
<td>Ti-6Al-5V</td>
<td>850</td>
<td>8 x 10⁻⁴</td>
<td>0.70</td>
<td>700-1 100</td>
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<tr>
<td>Ti-6Al-2Sn-4Zr-2Mo</td>
<td>900</td>
<td>2 x 10⁻⁷</td>
<td>0.67</td>
<td>538</td>
</tr>
<tr>
<td>Ti-4.5Al-5 Mo-1.50</td>
<td>871</td>
<td>2 x 10⁻⁴</td>
<td>0.63-0.81</td>
<td>&gt;510</td>
</tr>
<tr>
<td>Ti-6Al-4V-2Ni</td>
<td>815</td>
<td>2 x 10⁻⁴</td>
<td>0.53</td>
<td>670</td>
</tr>
<tr>
<td>Ti-6Al-4V-2Co</td>
<td>815</td>
<td>2 x 10⁻⁴</td>
<td>0.54</td>
<td>650</td>
</tr>
<tr>
<td>Ti-5Al-2.5Sn</td>
<td>1000</td>
<td>2 x 10⁻⁴</td>
<td>0.49</td>
<td>430</td>
</tr>
<tr>
<td>Ti-15V-3Cr-3Sn-3Al</td>
<td>815</td>
<td>2 x 10⁻⁴</td>
<td>0.5</td>
<td>299</td>
</tr>
<tr>
<td>Ti-13Cr-11V-3Al</td>
<td>800</td>
<td>2.7 x 10⁻⁴</td>
<td>0.43</td>
<td>150</td>
</tr>
<tr>
<td>Ti-8Mn</td>
<td>150</td>
<td></td>
<td>0.60</td>
<td>100</td>
</tr>
<tr>
<td>Ti-15Mo</td>
<td>800</td>
<td></td>
<td>0.60</td>
<td>100</td>
</tr>
<tr>
<td>C.P. Ti</td>
<td>850</td>
<td>1.7 x 10⁻⁴</td>
<td></td>
<td>115</td>
</tr>
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</table>
found for the $\beta$ alloys even though the grain size was large. Tensile elongations of 100-150 per cent were measured for sheet specimens of several compositions, including $\text{Ti-15Mo}$, $\text{Ti-8Mn}$, and $\text{Ti-13Cr-1V-3Al}$ alloys. Furushiro and Hori\textsuperscript{23} also report about 140 per cent elongation for the $\text{Ti-8Mn}$ alloy.

The alloy $\text{Ti-15V-3Cr-3Sn3Al}$ has also been evaluated\textsuperscript{24} at temperatures of 850°C and 760°C in order to determine its flow characteristics and potential superplastic formability. The material as received was evaluated as such and had a grain size of about 80 $\mu$m. It is found that this alloy exhibits deformation behaviour quite different from the two-phase alloys in that it tends to flow soften with about 2-4 per cent strain as shown in Fig. 6. The softening is reflected in the stress vs strain characteristics where the pre-strain is observed to reduce the flow stresses and increase the strain-rate sensitivity observed over a wide range of strain rates. The microstructural changes that occur during the softening include the development of sub-grain structure. Other studies conducted on $\text{Ti-10V-2Fe-3Al}$ and $\text{Ti-8Mo-8V-2Fe-3Al}$\textsuperscript{25} support the general behaviour of $\beta$ Ti alloys as noted above, but did not show a promising degree of superplasticity at strain rates of practical interest, except in isothermal forging operations.

![Figure 6](image-url)

**Figure 6.** Flow stress vs strain at constant strain rate for $\text{Ti-15V-3Cr-3Sn3Al}$ at 815°C.

### 2.5 Microstructural Changes during Superplastic Forming

As with most metal deformation processes, microstructural changes are found to take place during superplastic deformation\textsuperscript{410}. However, under conditions of what is
generally referred to as optimal superplastic conditions, the microstructural changes in
titanium alloys are not large in comparison to other deformation processes. For alloys
with a fine, equiaxed microstructure, superplastic deformation often causes grain
coarsening but the equiaxed grain structure is retained, even to quite large
strains. Also, there appears to be no significant internal structure development under
these conditions. The reason for this is that the primary deformation mechanisms are
grain boundary sliding and diffusion, and the temperatures as well strain rates are such
that grain boundary migration can also occur. There is no doubt, however, that some
deformation occurs by dislocation motion. However, evidence for this is difficult to
obtain because at these high temperatures dislocations sweep completely across a grain
and remain in the structure only as grain boundary dislocations.

Grain growth during superplastic flow is the most apparent microstructural change
normally observed for the fine grained \( \alpha' \beta \) Ti alloys. Fig. 7 shows microstructural
changes in specimens tested under superplastic conditions (927°C). The strain-induced
grain growth is significantly greater than static grain growth for \( Ti-6Al-4V \) (Fig. 8), the
effect showing an increase with increasing applied strain rate. This effect is believed
to be related to stress enhanced grain boundary mobility. Similar effect has been reported
in other alloy systems as well. This strain-induced grain growth leads to concurrent
hardening of \( Ti-6Al-4V \) during superplastic deformation. This characteristic is revealed
by constant strain rate tests as shown in Fig. 9 for the \( Ti-6Al-4V \) alloy at 927°C.
This hardening can more than double the flow stress at the strain of 1.0, and therefore
can be significant in the determination of pressure schedules for forming. Also, the
strain rate sensitivity, \( m \), tends to decrease with strain as the flow stress increases, and
can therefore influence the total elongation and determination of optimized forming
parameters. Under conditions of lower strain rate, grain coarsening becomes more
noticeable because the time of exposure to achieve a given level of strain elevated temp-
eratures is longer than at the higher strain rates.
Figure 8. Static and dynamic grain growth kinetics for Ti-6Al-4V.

Figure 9. True stress vs true strain curves at constant strain rates for Ti-6Al-4V alloy with three different initial grain sizes.
At the higher strain rates than optimal, grain refinement can occur as shown first by Lee and Backofen\textsuperscript{7} for Ti-6Al-4V. Such grain refinement is due to dynamic recrystallization processes. Furushiro et al\textsuperscript{19} have shown that the temperature of deformation can have a pronounced effect on the observed changes in microstructure and $m$ with strain for Ti-6Al-4V as shown in Fig. 10. For a strain rate of $4.1 \times 10^{-4}$/s and $760^\circ C$, $m$ was shown to increase by about 100 per cent, after which it remained nearly unchanged. The corresponding microstructure showed some grain refinement as strain progressed and $m$ increased, followed by very slight coarsening. In contrast, $m$ was shown to decrease continuously with strain at $860^\circ C$, accompanied by significant growth of the $\alpha$ grain size and total average grain size (although some refinement of the $\beta$ phase was reported). Thus, the grain size changes, which have been shown to contribute significantly to the flow hardening, are accompanied by corresponding

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure10.png}
\caption{Changes in grain sizes of $\alpha$ and $\beta$ phase, and corresponding changes of $m$ with superplastic deformation of Ti-6Al-4V (Ref. 19).}
\end{figure}
variations in the strain rate sensitivity of the flow stress, and therefore the superplasticity.

The superplastic deformation of titanium alloys has been shown to accelerate the development of equilibrium structures, and the corresponding microstructural changes have been observed and reported by Kaibyshev for the VT9 alloy (Ti-6.7Al-3-Mo-1.73). For this alloy deformed under isothermal conditions, it was shown that the phase ratio changed during deformation approaching the equilibrium phase ratio more rapidly than for thermal exposure without deformation. For example, at the strain rate of $2.6 \times 10^{-3}$ s$^{-1}$ the equilibrium phase ratio was achieved after 3 minutes whereas 30 minutes were required to achieve the same phase ratio under static conditions. It has also been shown that a lamellar structure progressively refined into an equilibrium, equiaxed structure under superplastic conditions. For the Ti-6.7Al-3Mo-1.7Zr alloy (VT9), this refinement was less effective at high deformation rates, apparently due in part to deformation rates exceeding the recrystallization kinetics. Such refinement of microstructure during deformation may be accompanied by an increase in the strain-rate sensitivity, $m$.

2.6 Gas Pressure Forming of Superplastic Ti Alloys

Gas pressure forming is by far the most common forming method for superplastic processing. While the thinning characteristics can be strongly influenced by the forming method employed and associated die interactions, the material properties, such as $m$, can also have a pronounced effect on the thinning profiles. The influence of this material parameter on the thinning characteristics in the bulge forming of spherical domes has been illustrated both experimentally and analytically by a number of investigators. Thomsen, Holt and Backofen evaluated such thinning characteristics of a function of $m$, with the finding that the lower the $m$, the greater is the thinning.

The source of this thinning has been explained through analytical developments of Cornfield and Johnson and Holt and is shown to be based on the stress gradient developing from the base to the pole of the dome. A corresponding strain rate gradient develops which depends, therefore, on the strain-rate sensitivity to flow stress as indicated by $m$. Under such a stress gradient, the uniformity of thinning will increase with increasing $m$, as shown in Fig. 11, since the corresponding strain rate gradient will decrease.

In contrast to the spherical dome, no such stress gradient develops in the forming of a cylindrical section of semi-infinite length. As a result, no significant thickness variations are observed in such a cylindrical part during forming, provided that die entry radius is not too small or no die contact has been made during forming. The thickness in a half-cylindrical section is typically 64 pct of the initial sheet thickness ($\varepsilon_t = -0.46$) for plane strain. On subsequent forming into a die configuration, the die walls can act to restrict deformation, and a thickness gradient will then result.”
This is illustrated for a reatangular section part in Fig. 12 for the case in which sticking between the die and forming black occurs, a condition observed in several forming studies. As illustrated, once die contact has been made, the areas of the part that come into contact with the die have their thickness virtually frozen to that value at the instant of contact, and the deformation then concentrates in those area where die contact has not yet been made. Thus, a thickness profile develops as shown in which a crown results in the bottom centre and at the upper sides, and minimum thicknesses result at the edge radius where die contact is made last. Nonuniformity of thinning is minimized in Ti SPF by using effective lubricants such as fine boron nitride powder.

With the continued developments in superplastic forming technology, there is greater need for accuracy of process control. Mathematical models of the forming operation have been developed for simple shapes to predict the pressure-time curves for the peak strain location in the part forming at a constant effective strain rate. Typically this location is controlled to form at the optimum strain rate for superplastic forming as suggested by maximum \( m \) in \( m \) vs \( \log \epsilon \) plot. A predicted pressure time curve is electronically followed to activate needle valves to form complex geometries. Fig. 13 shows a typical pressure vs time curve needed to form Ti-6Al-4V alloy into a rectangular geometry. The rapid initial pressurization, as shown in Fig. 13, is required when sheet curvature is relatively gentle. However, as curvature increases, this pressurization rate must be reduced with eventually a decrease in pressure, as the rate of thinning exceeds the rate of curvature increase. Once the die bottom is contacted and forming must progress in the die edge...
Figure 12. Schematic of the thinning process during blow, forming a sheet in a rectangular die.

Figure 13. Analytically calculated pressure vs time plot for gas pressure forming of Ti-6Al-4V sheet.
and corner areas, pressure must be increased to a fairly high value again. The size of these various stages in the pressure profile are influenced by die configuration, forming rate, material flow properties, lubrication, etc. It has been found that the use of a calculated pressure profile can improve the performance and productivity of forming shop significantly, by producing parts without failure. This is very important in working with an expensive material like titanium.

Fig. 14 shows a flat titanium blank being placed in a typical die arrangement for superplastic forming. Since forming temperature is excessively high, an oxidation and creep-resistant steel \((22Cr-4Ni-9Mn)\) is typically used as a die. The metal forming dies are located between ceramic heaters placed in a hydraulic press which provides the clamping action at the sheet periphery during forming. Inert gas (e.g., argon) is used for both pressurizing the sheet and maintaining a purge gas blanket. Formed parts are generally hot unloaded, although sometimes the entire die assembly is removed at the end of a forming run and replaced by a new die assembly loaded with sheet blank.

An early demonstration item for superplastic titanium was nacelle beam frame built by Rockwell International for the B-1 aircraft (Fig. 15). Using superplastic forming

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**Figure 14.** Die, blank and tooling arrangement for superplastic forming of Ti sheet.
techniques, cost and weight were reduced by 50 per cent and 30 per cent respectively in comparison to an existing conventionally fabricated structure made out of sheet and machined plate titanium. As against eight detail parts and 96 fasteners for the conventionally fabricated component, the superplastic formed part was a single part with no fasteners. Because no additional pieces needed to be joined to this part, purchase cost of joining and assembly tools and labour incurred in those operations could be avoided. A key criterion and guideline for the use of superplastic forming process is the redesign (or new design) of the part to take advantage of the extensive available formability of titanium. This allows a variety of stiffening ribs, etc., to be built into the part from.

3. Diffusion Bonding of Titanium

The diffusion bonding of α/β titanium alloys, particularly Ti-6Al-4V, has been the subject of considerable interest over the past decades. It is well established that these alloys are among the most readily bonded materials known, and virtually parent metal microstructure and mechanical properties are developed at the bond plane if the process is properly conducted. Diffusion bonding of Ti-6Al-4V primary structures was committed to the B-1 aircraft, and all four such aircraft flying today contain wing carry-through structures of this construction.

Diffusion bonding of titanium is carried out basically at the superplastic temperature (870-927°C) where diffusional transport of atoms is substantially enhanced. A typical die pack arrangement for diffusion bonding of plates is shown in Fig. 16. An entire assembly like this is placed in a retort inside a press with ceramic heaters and bonding is conducted in vacuum after uniform temperature has been achieved throughout. Pressure is applied from all three directions to provide tool and die registry.
Superplastic Forming of Titanium Alloys

Figure 16 Die, tooling and part details for diffusion bonding of titanium plate structures.

from all sides. Subsequent to bonding, the entire vacuum retort assembly is removed from press and allowed to cool. Very large monolithic components of the thrust frame for the Space Shuttle have been fabricated by diffusion bonding.

With the long history of diffusion bonding of titanium and its corresponding importance, this process is extending the state-of-the-art of titanium technology. A substantial understanding of Ti diffusion bonding has also been established. The fundamentally important stages of diffusion bonding are: (1) Development of intimate physical contact across the bond plane via creep of surface asperities, (2) Inter-diffusion and shrinkage of cavities, (3) Recrystallization and/or grain growth across the interface, and (4) Microvoid elimination.

For titanium α/β alloys, it has been observed that the achievement of intimate contact (stage 1) is the rate determining requirement in terms of pressure/time parameters for complete bonding. The interface asperities, which are surface characteristic of normally processed material, are overcome by a creep-like plastic deformation under the influence of sustained pressure at elevated temperatures as illustrated in Fig. 17. While most of the interface deformation is achieved by pressure, there is a final stage of the bond sequence, during which the microvoids are eliminated by a mechanism of diffusion. This final sequence can occur in the absence of pressure if the void size is sufficiently small, and is accelerated by the superimposed pressure. The modeling of these aspects of diffusion bonding has been accomplished, and supporting experimental test data has validated the concepts for Ti-6Al-4V and titanium aluminides.

It is important to note that the pressure/time requirements are dependent on the strain-rate sensitive flow stress of the material being bonded; the higher the material flow stress, the higher is the required bond pressure (for fixed time and temperature).
Figure 17. (a) Sequence of diffusion bonding in Ti-6Al-4V under a pressure gradient (first stage).

Figure 17 (b). Sequence of diffusion bonding in Ti-6Al-4V under pressure gradient (second stage).
Therefore, any processing condition or treatment which would alter the microstructure ‘(especially grain size) would also alter bond pressure requirements. For example, if superplastic forming precedes diffusion bonding in an SPF/DB process (discussed below), higher bonding pressures may be required than if the sequence is reversed. The reason for this is, that grain growth occurring during the forming cycle would result in higher flow stresses for the alloy which in turn dictates the need for higher bonding pressure.

The SPF/DB process has evolved as a natural combination of processes utilized for titanium alloys: i.e., the SPF and the diffusion bonding (DB) processes. The requirements for both processes are similar, in that the temperature, inert environment, and fine grained microstructure are identical requirements for both. With the relatively low flow stresses in the superplastic sheet materials, it has been found that gas pressures (about 300 psi) are sufficient to develop quality diffusion bonds. The superplastic forming method used in the SPF/DB process at the present time is the blow forming method.

The resulting SPF/DB processes consist of the following methods (Fig. 18): (I) forming of a single sheet onto preplaced details followed by diffusion bonding under gas pressure, (2) diffusion bonding of two sheets at selected locations followed by forming of one or both into a die (the reverse sequence can also be used), and (3) diffusion bonding of three or more sheets at selected locations under gas pressure followed by expansion under internal gas pressure which forms the outer sheets into a die and pulls the center sheet(s) into a core configuration. During DB part of the cycle, a stop-off material such as boron nitride powder is used to create selected locations of unbonded material, where gas pressure is subsequently brought into for

![Figure 18. Schematic sectional views of the several types of SPF/DB processes.](image-url)
expansion of the peak (SPF cycle). The SPF/DB process has the potential of fabricating a wide range of complex parts in which the joining is conducted in the solid state, and does not require the use of dissimilar materials (e.g., braze alloys) nor additional processing steps. The resulting joints can therefore withstand higher operating temperatures.

4. Summary

Titanium alloys, particularly $\alpha/\beta$ alloys containing some small amount of $\beta$ stabilizer, were among the first materials to be recognized as being superplastic. This recognition led to the early application of superplastic forming of titanium. The complex metallurgy of titanium alloys, compounded by the requirement for high temperatures and protective atmospheres, provide significant difficulties in conducting fundamental experimental work. Recently, however, there has been a considerable increase in the amount of work done on superplasticity in titanium alloys and the level of understanding has improved significantly. The influence of the relative volume fractions of the $\alpha$ and $\beta$ phases on superplasticity, together with the $\beta$ stabilizing addition and the effects of diffusivity on superplasticity have been discussed here for the two phase alloys. The effect of microstructure, including grain size, grain shape and processing history have been reviewed briefly. The importance of concurrent microstructural change and superplastic flow hardening behaviour have been discussed in some detail. The mechanisms and conditions necessary for diffusion bonding of titanium have been outlined, and the concept of concurrent SPF/DB structures has been reviewed.

References

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