A Study of Low Temperature Diffusion Bonding Processing of TI-6AL-4V Alloy for Reducing Costs in SPF/DB Structures

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Abstract

Solid-state diffusion bonding (DB) process is a joining technology based on the diffusion capacity of materials. Being activated by a combination of variables, mainly temperature, pressure and time, a couple of materials can be bonded by diffusion across the surface contact. Metallurgical nature of materials to be bonded condition their DB capacity but, in the case of titanium (Ti) and Ti alloys, and specially for the most known one, Ti-6Al-4V, no problems arise if oxygen level is controlled during the bonding cycle (i.e., inert atmosphere).

DB can be successfully developed together with superplastic forming (SPF) as they share similar process parameters, although for SPF the requirements are normally more restrictive, due to the nature of the superplastic deformation phaenomenon. However, a wise introduction of DB during SPF cycle can lead to overall processing cost reduction, as DB take place in a wider range of temperature-pressure-time combinations.

SPF Ti alloys structures are considered as one of the most interesting solutions for weight reduction in military aircrafts in operative conditions that aluminium alloys can not support. Some examples of these materials can be found in *Tornado* and *EF2000* aircrafts.

DB in Ti-6Al-4V alloy devoted to SPF, has been studied and assessed for different specimen configurations, focusing on mechanical behaviour of joints produced and metallurgical effects on the material that could adverse SPF. Results show that appreciable mechanical performances can be obtained for DB conditions involving temperatures well below SPF one.

1.   Introduction

1.1.   **SPF/DB in Aeronautics**

Appreciable weight savings and operating temperatures, together with corrosion resistance, justify the need and use of Ti alloys in aerospace industry replacing steel and aluminium. Main drawbacks for Ti alloys are the costs of raw material (3 to 10 times of steel or aluminium), and machining costs (even higher, at least 10 times that to machine Al). Therefore, benefits of using Ti must overcome the added cost [1].

Superplastic forming and diffusion bonding technology (SPF/DB) of Ti-6Al-4V alloy has allowed the manufacturing of complex honeycomb-type structures, saving total processing time and number of single parts. However, and due to the just exposed cost reasons, SPF/DB applications are almost restricted to the aerospace industry [2].

Many examples of Ti alloys applications in aeronautics can be found in reference [1]. SPF/DB is mentioned there for having been used to manufacture the *Boeing 777* tail cone. Other examples of the presence of SPF and SPF/DB technologies in civil aeronautics appear in [3], where applications on *Airbus 310/320* (jack can, wing access panels), *Concorde* (engine bay panel) or *BAe 125/800* (escape hatch) are referenced.

Focusing on military aircraft, a short but good description of SPF/DB applications are found in [4], in which the main guidelines for SPF/DB manufacturing of the heat exchanger duct from *Tornado* aircraft, and the foreplane torsion box of the *EF* are exposed.

In *Tornado*, heat exchanger duct assemblies are manufactured from four flat details: two core sheets to form the outer walls, and the other two to form the splitter vanes. Once sheets 'packs' are suitably assembled, DB is driven first at 925°C, 2.07 MPa gas pressure and 90 minutes. After DB, SPF is performed at around 900°C with inert gas, at an optimum strain rate of $1.5x10^{-4}$ s^{-1}. Diffusion bonds produced by this route demonstrated to have parent metal properties and highly reproducible with rejections due to no bonds less than 1%. Saving parts by SPF/DB, in comparison with traditional manufacturing (hot forming and TIG welding combined), represents an 80% (8 to 42).

For *EF*, the foreplane torsion box has a similar configuration than the structure above mentioned. In this case, inner sheets are X-like ribs (C-like straight ribs for the former). DB parameters, and subsequent SPF, coincided. DB joint strength was that of parent material, and did not peel (peeling was of concern for this geometry) under static or cyclic loading. SPF/DB showed to be also more cost effective than alternative processing, namely carbon fibre composite moulding.

1.2. Metallurgical background

Under the technological fact of SPF/DB, two metallurgical phenomena develop simultaneously sharing similar physicochemical parameters, such as temperature, pressure and time, together with some other factors, like surface condition or microstructure [5].

Superplasticity development in Ti-6Al-4V requires a temperature around 930°C. However, DB could be produced at lower temperatures, provided that the rest of parameters would be modified in the most suitable way. Savings in the processing of parts in which DB would be involved, could be beneficial for the SPF/DB technology as a whole.

Nevertheless, a reduction in the temperature for DB must not be planned carelessly. A temperature reduction involves a decrease in the thermally-activated processes which control DB, namely creep- and diffusion-related mechanisms [6-9]. Creep rates for Ti-6Al-4V alloy are a maximum closer to 950°C, but when temperature is reduced to 900°C, this rate decreases strongly, as Kellerer and Milacek discussed [10]. Diffusion is driven by constituent phases proportion, because β phase has a diffusivity two orders of magnitude higher than α phase in Ti-6Al-4V, and the proportion of β increases with temperature, from less than 20% at room temperature, stabilising 35-40% beyond 900°C [11]; if a decrease in temperature would be desired, it must be compensated by longer diffusion times [10].

On the other hand, the deformation of contact surfaces to be bonded must be also considered, specially during the initial stages of diffusion-bonded joint formation [12]. An increase in welding pressure could enhance local plastic deformation, increasing interface contact, and so promoting creep and diffusion mechanisms, regardless temperature lowering. However, low bulk deformation is always desirable, specially for near net shape components [13].

Another beneficial effect of temperature reduction is the material grain growth minimisation. Grain growth is of a big concern as it affects superplastic deformation in a strong manner. Main known mechanisms proposed to explain how superplastic deformation is developed, are supported by small grain sizes that allow grain movement (slidig, rotating) under thermomechanical forces [14-17].

Considering DB again, as the β transus temperature for Ti is approached, rapid grain growth occurs, the creep rate so decreases and the bonding time increase. As an example, in Ti-6Al-4V, at 2 MPa bonding pressure, the time to produce a pore-free bond increase by a factor of six when

grain size grows from 6.4 μm to 20 μm. Other product presentations, like plate or forgings, have a coarser grain size in comparison to sheet, so that different bonding parameters are required [13].

In conclusion, and independently to SPF phenomenon, cost-driven temperature reduction for DB does produce slower creep rates to form the joints that should involve increases on pressure and time. However, some considerations must be done on pressure and time increasing:

- Elevated pressures favour void collapsing on joints, but tend to produce undesirable macroscopic deformation and affect cost.
- High times promote diffusion to form DB joints, but cause grain growth and also affect cost.

A compromise between temperature reduction and how the rest of parameters can be altered to get good quality, less cost, DB joints has been the main aim of the current investigation.

2.   Experimental work

2.1.   **Plan of experimentation**

After the theoretical assessment, DB parameters shown on Table-I were selected in order to produce DB joints below 930°C.

Table-I:
Experimental Diffusion Bonding conditions

Temperature (°C)	Pressure (MPa)	Isotherm Bonding Time (min)	Surface Roughness (mm)	Vacuum (Pa)
850	4	120 90 60 30	1×10^{-3}	13.2×10^{-3}
	2	120 90 60 30		
750	6	180 120 60 {1}		
	4	180 120		

{1} DB condition only considered for impact tests.

Mechanical tests were the tools to check the quality (in terms of 'strength') of the DB joints obtained, complemented with metallographic studies. Shear (single lap joints), peel and impact tests were studied.

Shear and peel are usual mechanical tests for lapped-joints assessment, and that is why they were applied. Impact test was chosen because values obtained are more significantly related to the density of defects present at the bond interface in DB joints than any other mechanical test, as Ohsumi et al. showed [18].

2.2.   **Materials, equipment and procedure**

Different commercial presentations of mill annealed Ti-6Al-4V alloy were used for the investigation:

- 1 mm thickness sheet, used for assessing shear behaviour,
- 2.5 mm thickness plate, for assessing peel,
- 28 mm thickness block (thick plate), for assessing impact.

The chemical compositions for all presentations are given on Table-II.

Table-II:
Chemical Composition of Ti-6Al-4V alloys used for experimentation

Material	Composition (%)							
	Al	V	Fe	O	C	N	H	Ti
Sheet and Plate	6.20	4.30	0.08	-	0.05	0.009	0.005	Balance
Block	6.15	4.40	0.09	-	0.05	0.010	0.005	Balance

Microstructure revealed by scanning electron microscopy (SEM) show α grain, with stabilised β phase in grain boundary. For sheet and plate, grains are quasi-equiaxial or equiaxial (see figure-1), but in big plate, or 'block', α grains appear as bands, not equiaxial (see figure-2), and even some 'clusters' of β phase are present where α bands are distorted, most probably due to the hot rolling manufacturing process (figure-3, optical microscopy image).
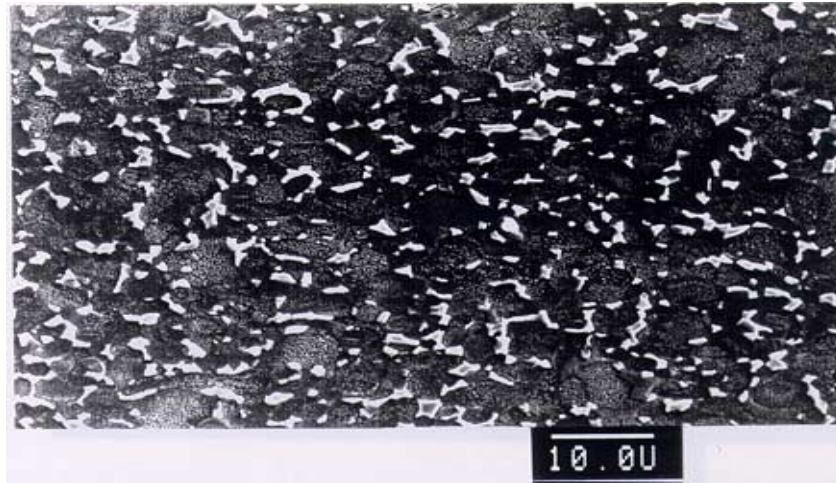

Figure-1:
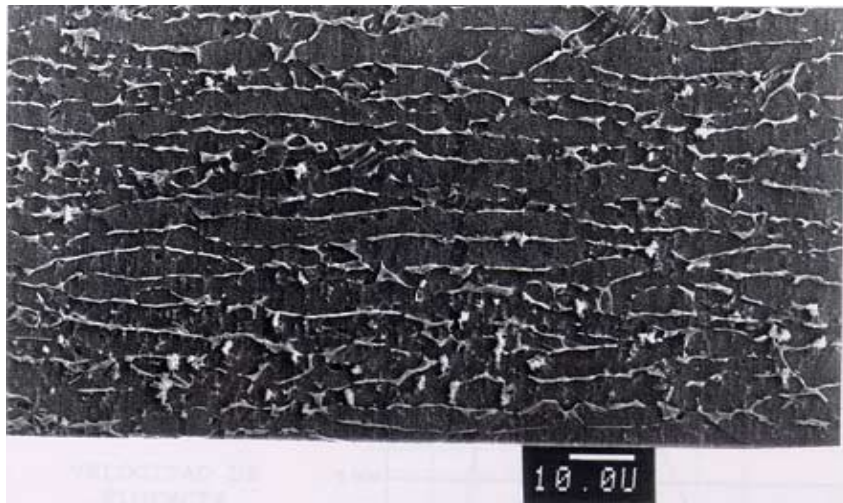**Ti-6Al-4V parent alloy microstructure for sheet and thin plate presentations**

Figure-2:
Ti-6Al-4V parent alloy microstructure for thick plate presentation
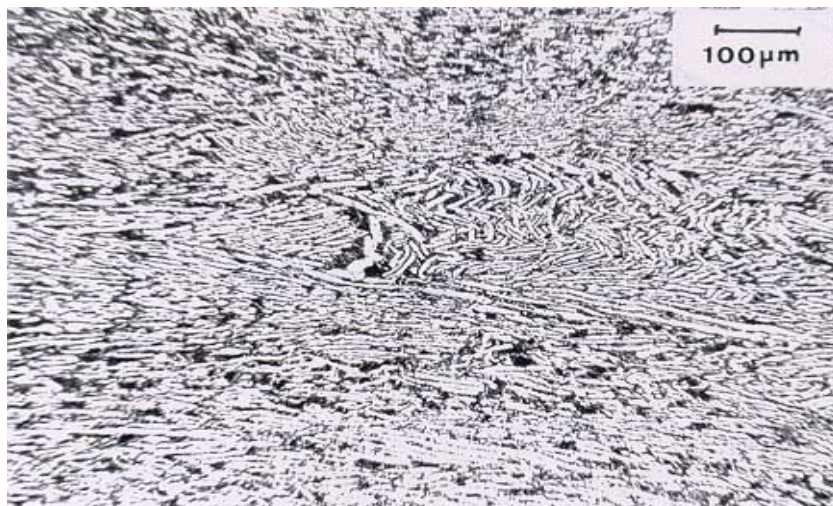
Figure-3:
α phase banded grains distortion in Ti-6Al-4V parent alloy thick plate presentation

DB processes were performed inside a vacuum furnace, specially designed to admit external mechanical pressure, and where the bonding parameters were programmed. The diameter of inner chamber (65 mm) limited the size and geometry of suitable bonded specimens.

For all tests, welding surfaces were ground with SiC emery paper down to 600 grade to get a common roughness (0.1 μm), and then cleaned with acetone in an ultrasonic bath. Finally, each pair of specimens were assembled by local liquid adhesion, and placed on corresponding tooling. Details on specimen's geometry will be dealt in following paragraphs.

2.3. Shear tests

The results obtained by single lap shear testing of Ti-6Al-4V DB joints were strongly dependant on test specimen configuration. Two test specimens were used as it is discussed as follows, but in both cases, it is worth mentioning that test configuration was produced by machining original DB specimens after bonding them only in a small area (see figure-4).
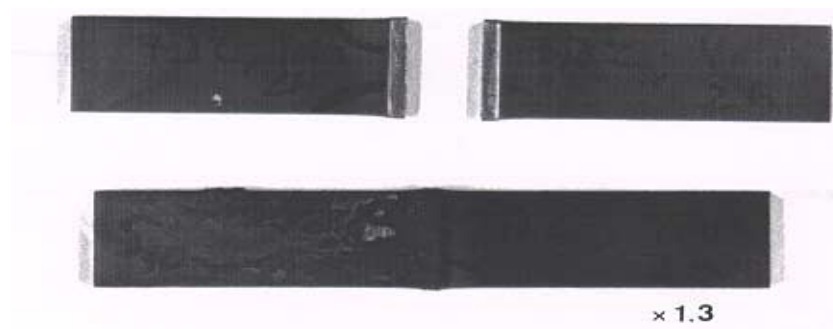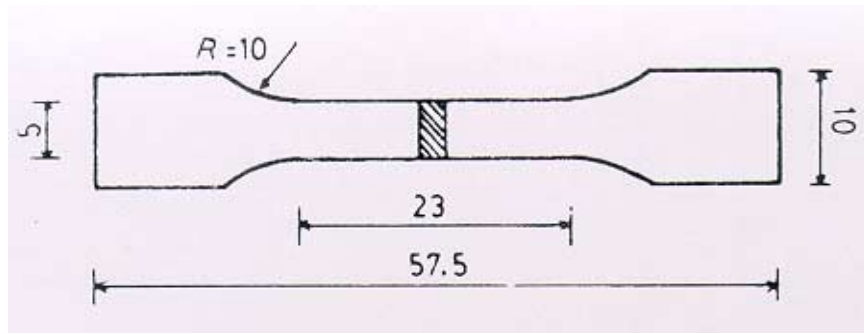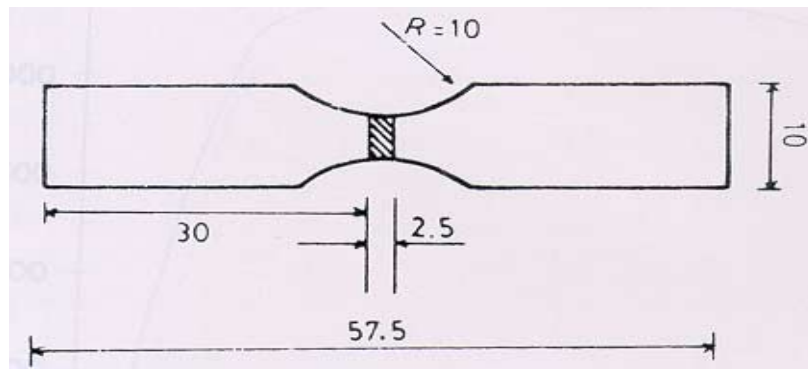

Figure-4:
DB specimens for single overlap shear testing of joints before machining

First test configuration (a 'classical approach', based on single lap shear test in adhesive bonds) is that shown in figure-5. To not inducing out-of-plane bending (peel stresses) on the base material next to the bonded area, causing premature failure and not throughout the DB joint, special jigs were designed and clamped to the specimens to keep them aligned during the tests. Results were not satisfactory as they were too much related to the relation overlap length/single sheet thickness, for which advisable values should be between 0.6 and 2 [19-22]. As it was not possible to control this factor appropriately, a change in test specimen configuration was decided.

Figure-5:
First configuration of specimens for single overlap shear testing of Ti-6Al-4V DB joints

Second test configuration appears in figure-6. In this case, a specimen with variable width was designed in order to focus strain forces on DB area. These configuration induced failure by shear through the bonding area (bonding interface), and only when the thickness reduction of the parent sheet by plastic deformation was higher than 10%, the failure occurred away from the DB area.

Figure-6:
Second configuration of specimens for single overlap shear testing of Ti-6Al-4V DB joints

Unfortunately, mechanical results did no show good correlation with diffusion bonding conditions, although shear stresses were reasonably close to those got for parent material in the same heat treatment conditions (see Table-III).

Table-III:
Summary of single lap shear tests on Ti-6Al-4V DB joints.
Comparison with parent material shear stress.

BONDING CONDITIONS			DB JOINTS {1,2}	PARENT MATERIAL {1,3,4}
Temperature (°C)	Pressure (MPa)	Isotherm Bonding Time (min)	Shear Stress (MPa)	
850	4	120	No failure	582
		90	428	576
		60	451	606
		30	436	582
	2	120	428	582
		90	355	576
		60	390	606
		30	433	582
750	6	180	453	561
		120	415	577
		60	No bond	Not available
	4	180	304	561
		120	474	577

{1} Mean value of three specimens.
{2} Normalised values for overlap length = 2 mm, and overlap width = 4,5 mm.
{3} Approximation from 0,6 times tensile strength, tested under ASTM A370.
{4} Parent material conditions include temperature-time heat treatment;
     no pressure influence has been considered.

As a conclusion of these test series, it was found that specimen configuration influenced attainable mechanical values much more than DB parameters.

2.4.   **Peel tests**

Peel tests were undertaken as they are very useful to simulate stress behaviour in inner skins for X-type SPF/DB sandwiched structures. Very little work was developed on that matter when the investigation was running, at least on Ti-6Al-4V alloy [23, 24].

Specimen used is shown on figure-7. It consisted on a couple of sheets 90° bent, yielding a 1 cm^2 bondable area (70 mm total length of sheets). Bending was driven by mechanical forging at around 450°C. No annealing for stress relieving was performed after forging, as DB cycles would undergo subsequently.
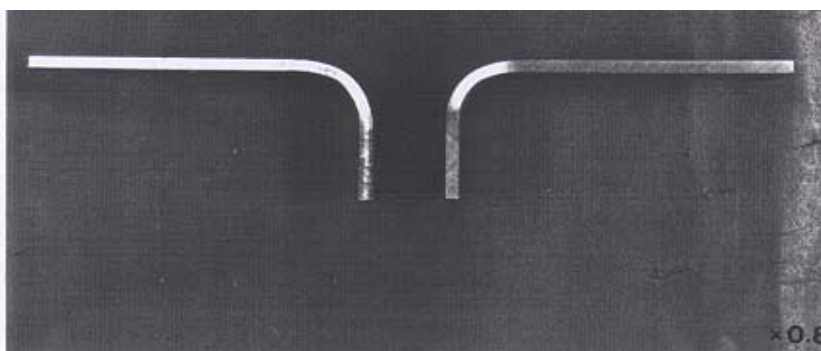

Figure-7:
Configuration of peel testing specimens of Ti-6Al-4V DB joints

The DB specimen is directly the specimen to be tested by pulling from both bond-free sides. Results are shown on Table-IV, for which no comparison with parent material is applicable.

Table-IV:
Summary of peel tests on Ti-6Al-4V DB joints

BONDING CONDITIONS			DB JOINTS {1,2}
Temperature (°C)	Pressure (MPa)	Isotherm Bonding Time (min)	Peel Strength (N/mm)
850	4	120	278
		90	239
		60	216
		30	126
	2	120	238
		90	207
		60	176
		30	112
750	6	180	124
		120	103
		60	No bond
	4	180	115
		120	66

{1} Mean value of three specimens.
{2} Normalised values for 1 cm^2 bonded area

From Table-IV results, it can be deduced that DB parameters seem to influence the attainable mechanical strength: the higher the temperature and pressure are (the enhanced ideal conditions for DB development), the better strength is obtained. As it can also be seen, for 850°C processes, long times DB at 2 MPa cycles, yield better peel strength results than short time, high pressure ones. 750°C, high pressure cycles, drive to poor results in comparison to 850°C cycles, and only seem to be better than short time, low pressure ones. Macroscopic deformations are comparable in that conditions.

Defects on DB area seem favour peel development. For those DB conditions for which void collapsing processes, responsible of joint formation [25-27], are not completed, peel forces tend to connect the line of voids in the bonded interface, so promoting peeling through bonding line. Images of void-rich and void-poor interfaces can be seen on figures 8 to 10 for these tests.
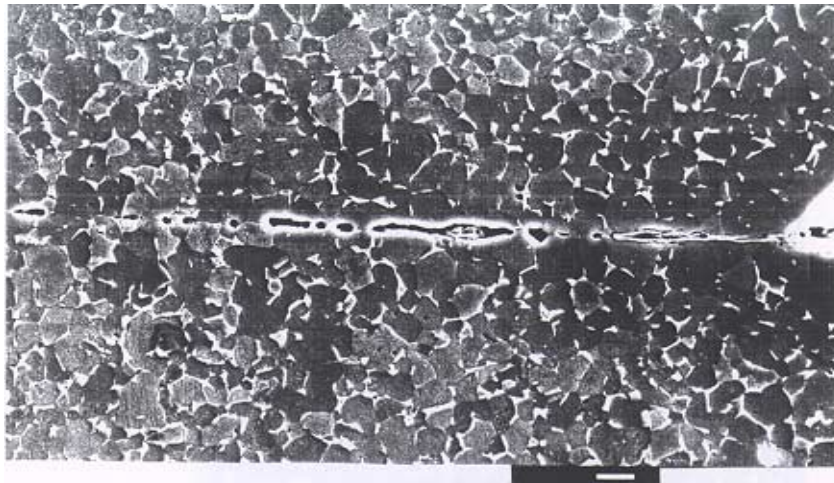

Figure-8:
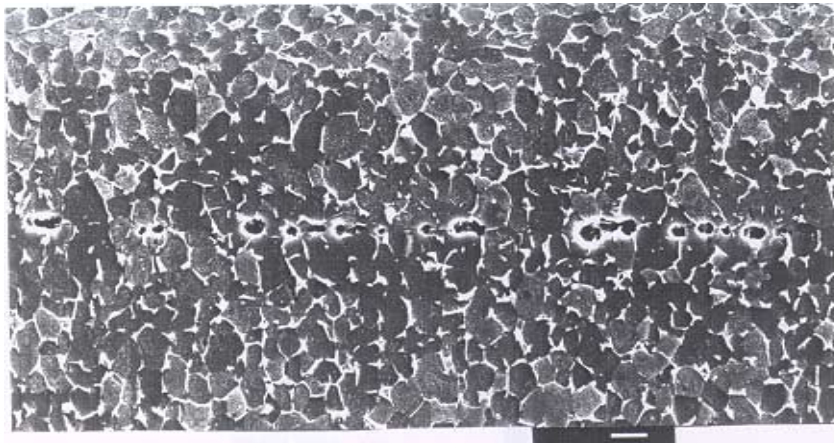**Voids on joint for 750°C/6 MPa/180 minutes DB conditions (unit = 10 μm)**

Figure-9:
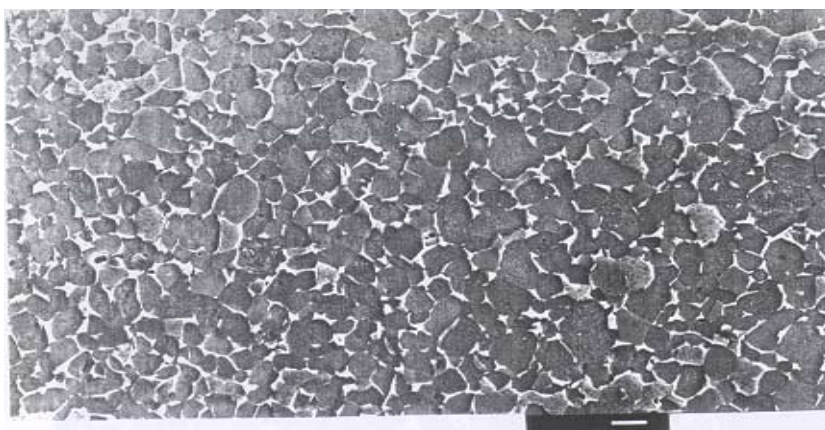**Voids on joint for 850°C/4 MPa/30 minutes DB conditions (unit = 10 μm)**

Figure-10:
Voids on joint for 850°C/4 MPa/90 minutes DB conditions (unit = 10 μm)

2.5. Charpy impact tests

Impact tests were performed on prismatic shape samples machined from 28 mm-thick plate. After welding, samples were machined into Charpy impact test specimens (ASTM E23). In order to place the V-notch, metallographic etching of the contact area with Keller's reagent was needed..

The results obtained from Charpy test appear in Table-V. Impact energies increase when bonding temperature and pressure increase too. However, for bonding at 850°C, the energy values reach a maximum for 60 minutes isothermic bonding time, decreasing for longer times.

Table-V:
Summary of Charpy tests on Ti-6Al-4V DB joints

BONDING CONDITIONS			DB JOINTS {1,2}
Temperature (°C)	Pressure (MPa)	Isotherm Bonding Time (min)	Impact Energy (J)
850	4	120	5.9
		90	8.3
		60	10.8
		30	3.9
	2	120	4.9
		90	7.9
		60	10.2
		30	2
750	6	180	9.8
		120	No tested
		60	1
	4	180	3.9
		120	1

{1} Mean value of three specimens.
{2} Energy of parent material (RT, no annealing) = 22.5 J

This behaviour may be explained by the combined effect of two factors:
a) development of the contact area between the two bonding surfaces,
b) phase transformations induced in the material by the bonding thermal cycle.

As it was exposed in 2.4., DB can be regarded as a process in which interfacial defects (voids) between two surfaces met in contact tend to collapse as a result of diffusion mechanisms which are accelerated by temperature, pressure and time. Consequently, it is assumed that any increase of any of these parameters must drive to contact area increases, and therefore, to higher impact energy values.

On the other hand, bonding thermal cycles modify the original Ti-6Al-4V plate microstructure (see figure-3), transforming the banded structure into an equiaxial structure, due to isotherm annealing below β-transus temperature.

When samples are bonded at 850°C, microstructure transformation follows two patterns. First, transformation of β phase grains into Widmanstätten-like structure α-phase grains (see figure-11). The presence of a Widmanstätten structure is of particular importance, because this kind of structure gives the highest toughness to Ti alloys, followed by the equiaxed and the martensitic [28-30]. With longer isotherm bonding times, Widmanstätten structure transforms slowly to the equiaxed. A second pattern to be considered is that α pre-existing bands transform into equiaxed grains by recrystallization annealing (see figure-12).
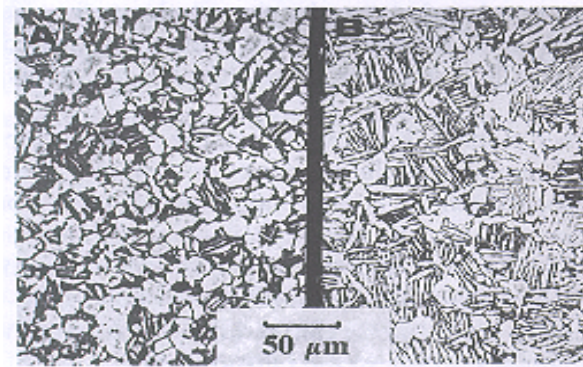

Figure-11:
Microstructure at 850°C/4 Mpa;
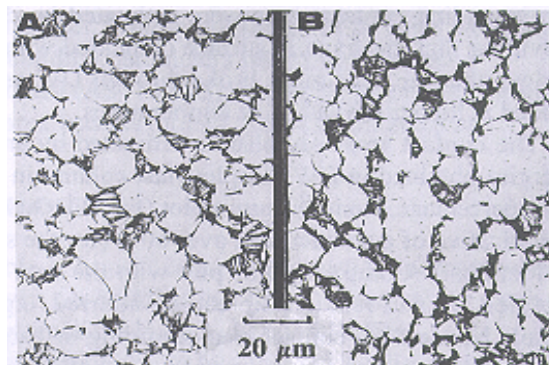**A) 30 minutes ; B) 60 minutes**

Figure-12:
Microstructure at 750°C/6 Mpa;
A) 60 minutes ; B) 180 minutes

However, when samples are bonded at 750°C, the material is too far from β-transus temperature, and the transformation to a Widmanstätten structure is neglectable (see also figure-12). Thus, only the recrystallization of the parent microstructure can be considered as a microstructural transformation caused by the bonding cycles. Consequently, the impact behaviour of the bonded joints must be only influenced by the interface void collapsing mechanisms.

If both microstructure transformations and void collapsing processes are considered together, the behaviour of the experimented DB joints can be described as follows.

Regarding specimens bonded at 850°C, when DB time is the shortest (30 minutes), the contact between surfaces is poor, and the impact energy absorbed is low. If pressure is involved, it is clear that for the higher bonding pressure (4 MPa), impact energy values are higher than that obtained at the lower pressure (2 MPa), because plastic collapse of voids is clearly favoured. When samples are bonded for 60 minutes, the presence of the Widmanstätten structure affects impact energy, so increasing impact values. As the Widmanstätten structure anneals with time (90 and 120 minutes

DB conditions), impact values are reduced, although surface contact improvement would have been favoured.

For samples bonded at 750°C, only contact area evolution (void collapsing) can be considered, and impact energies increase with bonding pressure and time.

What has had been described suggests the possibility to adjust temperature-pressure conditions for reaching good quality joints avoiding the need of long time processing DB cycles in SPF/DB technology. However, the presence of a Widmanstätten structure complicates the suitability of the proposed optimal DB cycles in respect to superplasticity development.

3.   Conclusions

A study on the phaenomenology and properties of DB joints in Ti-6Al-4V alloy at temperatures below superplastic deformation has been undertaken aiming to reduce costs for SPF/DB processing. Shear and peel tests, over sheet material, and Charpy impact test over thick plate, have been performed, and the concluding remarks for the investigation are:

1. Shear testing is strongly dependant on specimen configuration, and results suggest that the test itself seems not to be very sensitive in respect of DB parameters influence.
2. Peel test shows a relationship between mechanical results and DB parameters. Comparing the same temperature, peel strength for low pressure-long time conditions are higher than high pressure-short time ones.
3. Microstructural transformations in Ti-6Al-4V influence the behaviour of low temperature DB joints as it has been observed in Charpy V-notch test specimens. Under DB conditions that favour Widmanstätten structure development (850°C welding cycles), impact energy values are better for intermediate welding times than for longer times. When the material is of equiaxic structure (long time 850°C, and 750°C welding cycles), impact energy increases with time, and only classical void collapsing mechanisms for DB joint formation have to be considered. Although the use of Charpy test is recommended because its well known sensitivity to the quality of the DB joint, it requires thick specimens, that there are no suitable for SPF process. In the same way, Widmanstätten structures are not adequate for superplasticity development.

Acknowledgement

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