

Manufacturing of a hybrid component in Ti6Al4V-ELI alloy by combining diffusion bonding and superplastic forming

Pasquale Guglielmi^{1,a*}, Antonio Piccininni^{1,b}, Angela Cusanno^{1,c} and Gianfranco Paumbo^{1,d}

¹Department of Mechanics, Mathematics and Management, Politecnico di Bari, via Orabona 4, 70125 Bari, Italy

^apasquale.guglielmi@poliba.it, ^bpiccininni.antonio@poliba.it, ^cangela.cusanno@poliba.it, ^dgianfranco.palumbo@poliba.it

Keywords: Bonding, Sheet Forming, Titanium Alloys

Abstract. Through Diffusion Bonding (DB) large surfaces can be joined with low distortions and localized microstructural changes. Heterogeneous Titanium (Ti) components, composed, for example, by a porous layer over a bulk one, which play a key role for biomedical applications, can be obtained. In the present work, the possibility to combine the SuperPlastic Forming (SPF) process (for creating the part's shape) and the DB process (for joining two different layers) is investigated. In particular, the porous layer was obtained by solid state foaming, made possible thanks to a heat treatment set on a slice cut from a billet produced by compacting Ti powders via Hot Isostatic Pressing (HIP). SPF was used to shape both the Ti sheet and the slice cut from the HIPed billet. Since the SPF is conducted at high temperature, a porous structure could be obtained in the HIPed material (solid state foaming occurred); setting the proper pressure and time, the two layers could be successfully joined by DB. All the investigated pressure levels revealed to be able to produce a complete solid state joint, without any discontinuity; in addition, the final hybrid component could be manufactured according to the desired geometry.

Introduction

Diffusion bonding is a solid state joining process. The joint made using such a process are affected exclusively by a microscopic deformation and characterized by high homogeneity, without secondary materials or liquid phases. During the process, two surfaces are joined at elevated temperature (between 50 and 80 percent of the melting point) by means of a pressure applied to the interface. The pressure must be sufficiently reduced so as to avoid obvious deformations of the parts to be joined; this usually determines that the characteristic times of the process are around two hours and more depending on the geometry of the part. Since diffusion bonding is caused by atomic migration across a solid-state interface, there are theoretically no microstructural discontinuities at the interface region and, consequently, the mechanical properties and microstructure in the bonded region are no different from those of the base metal. In addition, when differences exist between the two materials to be joined, the interface zone would be able to mediate their properties [1]. DB can be considered a valid solution in obtaining joints in materials, such as Ti alloys, usually difficult by conventional techniques [2]. Among others, aspects of significant importance in the adoption of such a technology are attributable to (i) reduced distortions, (ii) applicability to large areas, as well as the aforementioned (iii) limited microstructural distortions. [3]. A further interesting aspect is related to the possibility of combining the DB and SPF processes for the fabrication of highly resistant joints with complex geometries that would otherwise be hardly produced. In fact, it has been demonstrated that this strategy can make it possible to obtain high-performance and sustainable final components from an economic point of view [4]. Typical applications involve sandwich structures with cost and weight reductions of between 30 and 50 percent compared to what is possible with conventional

techniques [5]. The diffusion bonding process must be optimized according to three parameters: time, applied pressure and temperature; the choice of suitable values for such parameters is essential to promote the diffusion which is necessary for the joining. In addition, specific geometric parameters such as (i) the final shape and (ii) the number of layers shall be taken into account at the design stage [6]. Furthermore, the microstructure of the starting material should be considered [6]. Finally, two necessary conditions that must be satisfied during the process are represented by (i) the intimate contact between the two surfaces and (ii) the absence of surface contaminating substances that could interfere with the bonding.

In the present work the DB process has been investigated for the fabrication of a joint characterized by two layers in Ti6Al4V-ELI alloy; specifically, the two layers were produced using two different processes: the monolithic layer was obtained through the rolling process, while the other one was obtained from through the Hot Isostatic Pressing (HIP) process [7]. The porous Ti alloys, due to their high mechanical performances and good biocompatibility, have found wide use in the biomedical sector for a wide range of bone implants [8,9]. In fact, the porous structure makes the mechanical characteristics (Young's modulus) more similar to the human bone's ones, thus reducing the stress shielding phenomena. Such a reduction of the Young's modulus is intimately linked to the level of porosity inherent in the material. In addition, a porous bone-like prosthesis topography may promote bone growth [10,11]. The present study is aimed to evaluate the feasibility of the DB process, both in the presence and absence of a superplastic deformation, focusing the attention on both the bonding capability of the two layers and the possibility of increasing the porosity of the layer produced by HIP.

Material and Methodology

Investigated material

The experimental activity discussed in the present work was conducted on Ti6Al4V-ELI alloy circular samples (disks with a diameter of 75mm). More specifically, the DB process was performed by joining one disk extracted from a rolled sheet (1mm thick) and one disk extracted by Electrical Discharge Machining from a billet produced by HIP (1.5mm thick). As concerns the HIP process, the following parameters were used: (i) Ti6Al4V-ELI powders with max diameter equal to 50µm, (ii) Argon pressure of 0.2 MPa and (iii) HIP pressure equal to 80MPa.

In order to distinguish the two types of layers involved in the process, in this paper the rolled material and that deriving from the HIP process will be called "bulk" and "HIPed" respectively. The chemical composition of the investigated material is reported in Table 1.

Table 1 Chemical composition (weight %) of the investigated Ti6Al4V-ELI alloys.

Al%	V%	Fe%	C%	N%	H%	O%	Ti
6.15	3.87	0.15	0.008	0.006	0.001	0.08	Bal.

Adopted methodology

All the tests performed were conducted in atmospheric conditions, although it is known that the presence of oxygen could hinder the diffusion bonding process. In fact, there are several testimonies in the literature that focus the attention on a possible effect of the presence of oxygen during the diffusion bonding process. However, in this regard, Lee et Al [12] focused the attention on this aspect. The evidence that emerges shows the capability of the DB process to take place effectively even in the presence of oxygen. In fact, a suitable preparation of the surfaces in contact and to be welded together coupled with the capability of self-evacuation from the interstices between the two plates in contact following the pressure applied by the gas on the entire sandwich would make the aspect relating to the presence of oxygen less pressing than that it might be thought. In the light of this, it is anticipated that the presence of oxygen during the process did not

represent an obstacle to obtaining the complete welding of the various interfaced plates. An overview of the equipment used to perform the experimental activities is reported in Figure 1.

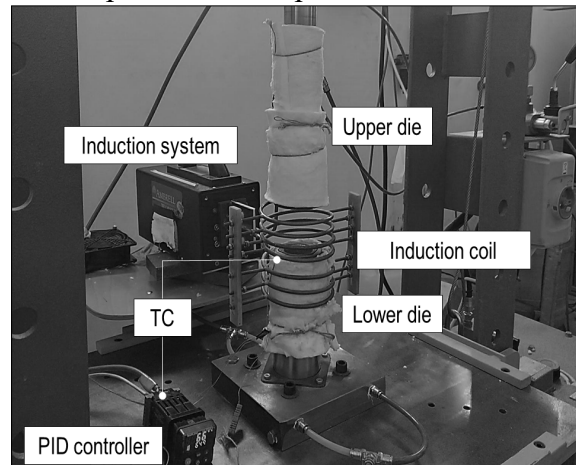


Figure 1 Overview of the experimental equipment used for DB experiments.

From a methodological point of view, the DB tests were carried out by exploiting the action of a pressurized gas (Argon) acting on a central area of the circular sample.

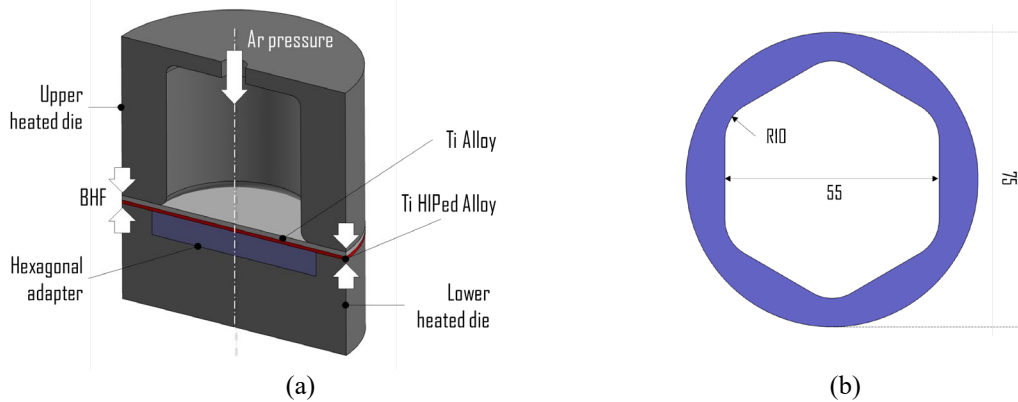


Figure 2 Scheme of the experimental DB setup (a) and the adapter for conducting tests with plastic deformation (b).

In addition, in order to avoid gas leakages during the test, a blank holder was used to apply a load on the peripheral area of the sample (see scheme in Figure 2); in the present work a constant value of the Blank Holder Force (BHF) was used (equal to 8.85MPa). All tests were conducted at a temperature of 850°C, since it is well known in literature that it is the optimal condition for the superplastic behavior of the investigated Ti alloy [13].

During each test, a constant and homogeneous temperature in the test area was ensured by the adoption of an induction heating system. The temperature was controlled during each test by a type K thermocouple (TC) welded to the interface of the two dies and directly connected to a PID system, as shown in Figure 1.

The experimental activity was initially focused on the feasibility of the DB process by investigating different Argon pressures; subsequently, for one of the previously analyzed conditions, the combined effect of the superplastic deformation was also considered. For all performed tests a fixed duration of 240 minutes was considered. Finally, regarding the test with superplastic deformation a hexagonal adapter was used (Figure 2b) with a depth of 7.5mm directly integrated into the interface between the two visible die in the experimental setup (purple block in Figure 2a). Table 2 summarizes the experimental conditions investigated in the present work. The same table shows the nomenclature used to distinguish the investigated test conditions.

Table 2 Investigated experimental conditions.

Nomenclature	Method	Argon pressure, MPa
0.75DB	DB	0.75
1.00DB	DB	1.00
1.40DB	DB	1.40
1.40DBF	DB + SPF	1.40

Before each test (3 replications), the surfaces to be joined were properly prepared by pre-polishing to eliminate residues of the previous processes and reduce the roughness (less than 1 µm). Processed samples were analysed by means of light microscopy and Vickers microhardness; finally, the porosity level was measured to evaluate the effect of both the pressure and the deformation on the foaming capability of the HIPed material. The level of porosity (quantified in terms of percentage area and average diameter) was assessed by investigating samples extracted from the component after the DB process and subjected to a proper metallographic preparation.

Metallographic analyses aimed at determining both the joined zone and changes in terms of porosities. For this purpose, the Nikon MA200 microscope and the ImageJ software for digital image processing were used. For this latter purpose each analysis was carried out considering 3 different micrographs. Finally, the mechanical properties of the hybrid component were evaluated through 10 different Vickers microhardness measurements for each zone (HIPed and bulk layers, as well as the interface) using an automatic Qness Q10 Microhardness tester with a load of 500g.

Results

The micrographs obtained for the evaluation of the hybrid structures obtained are shown from Figure 2 to Figure 5. For all tested conditions a perfect adhesion between the two different layers was recorded, supporting the effectiveness of the proposed approach. Although micrographs have indicated the two different layers of the joint (HIPed and Bulk), it is possible to distinguish them easily because of the different type of manufacturing process from which they derive: in fact, noticeable black circular areas associated with the registered porosity are visible in the layer resulting from HIP process. Red arrows indicate the interface zone: no discontinuities or clusters of porosity were found in this area. Furthermore, in accordance with the literature [12], an aspect of not negligible importance is that relating to the absence of inclusions (oxygen) in the area where the two layers are joined. This consideration is even more supported when high magnifications are considered.

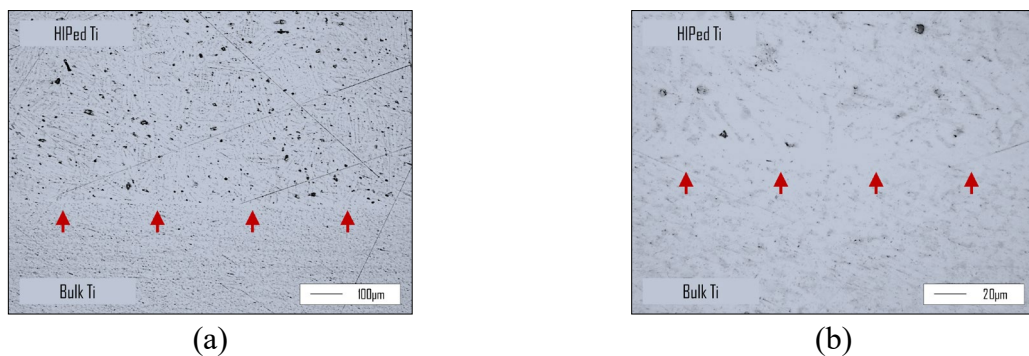


Figure 3 DB test conducted with Argon pressure of 0.75MPa at two different magnifications: (a) 70X (b) 350X.

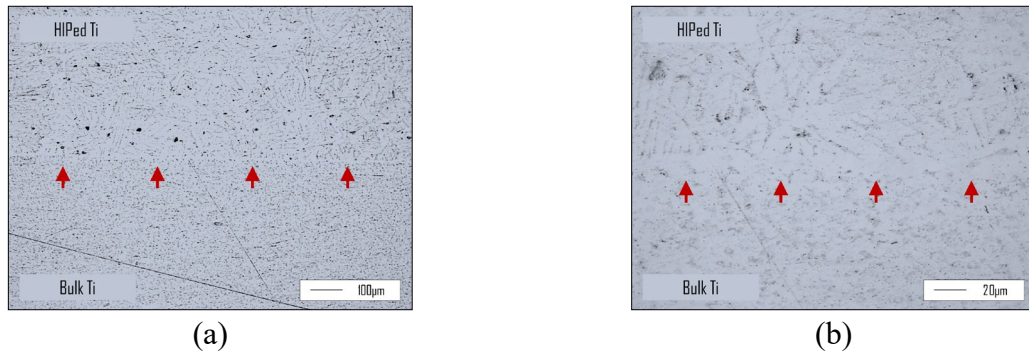


Figure 4 DB test conducted with Argon pressure of 1.00MPa at two different magnifications: (a) 70X (b) 350X.

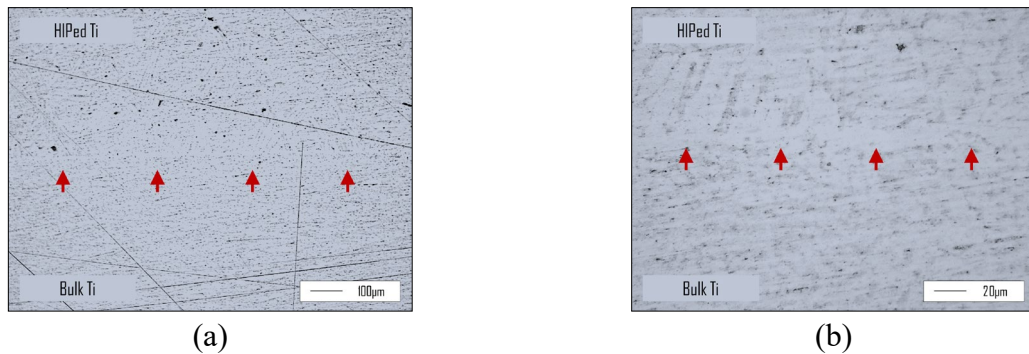


Figure 5 DB test conducted with Argon pressure of 1.40MPa at two different magnifications: (a) 70X (b) 350X.

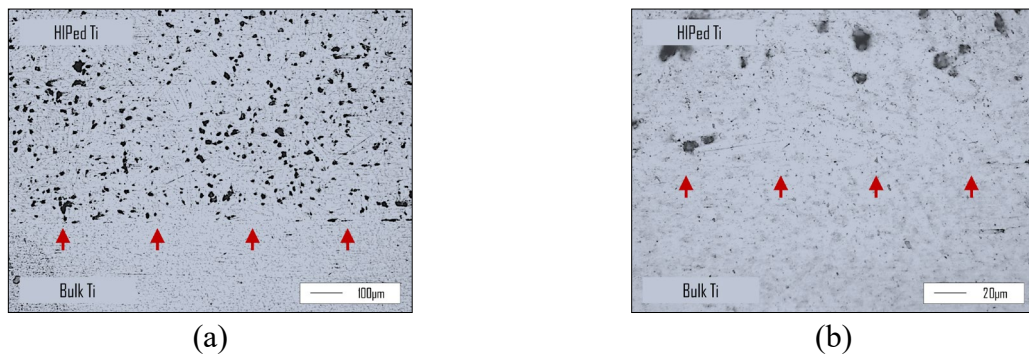


Figure 6 DB test coupled with SPF conducted with Argon pressure of 1.40MPa at two different magnifications: (a) 70X (b) 350X.

In addition, although after the diffusion bonding process there are no particular propensities of the material to foam (this is due to the low process temperature, i.e. 850°C), the contribution of the deformation seems to stimulate the porosity growth mechanism. In this regard, analyses of the porosities, both in terms of percentage area and average size are reported respectively in Figure 6a and Figure 6b. In the same figures the values referred to the “As Received” (AR) condition, deriving from the HIP process and not yet subjected to any DB process, are reported to better understand how the Argon pressure can influence the foaming behavior of the Ti alloy. For both output variables considered, the DB process allows to increase the typical values that can be associated with the AR condition. Regarding the percentage of porous areas, a certain effect of the Argon pressure applied during the DB process was recorded; on the other hand, when no deformations are provided during the process, the average diameter of these porosities does not undergo statistically significant variations. Finally, by combining the DB process with the SPF process, in addition to obtaining a further increase in the percentage of porosity (Figure 6b), there

is an increase - this time significant and equal to about 50% - in terms of the average diameter of the porosities (Figure 6b).

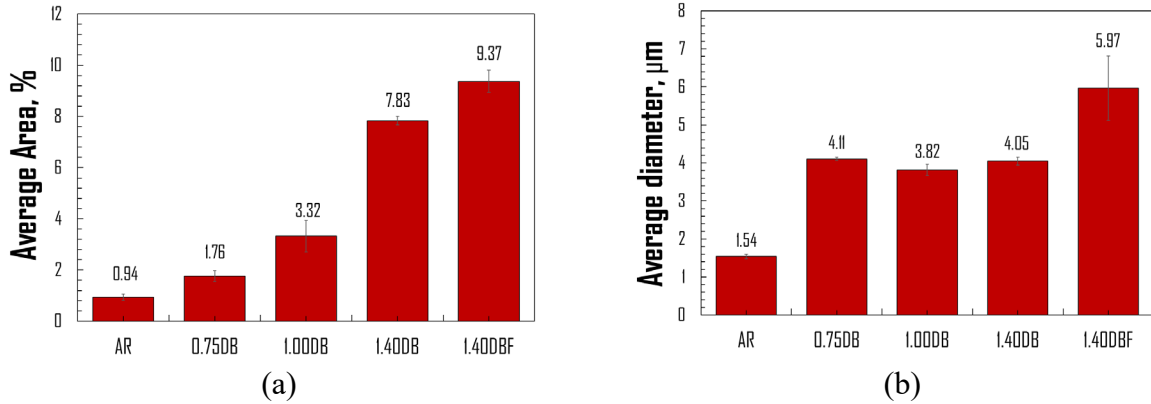


Figure 7 Analysis of the porosities for the different conditions used for the diffusion bonding process: (a) Average percentage area and (b) Average diameter of the porosities.

For the test performed by combining DB and SPF processes the attention was focused on the three different areas shown in Figure 7 (the wall, the corner and the flat area). The correspondent micrographs are shown in Figure 8.

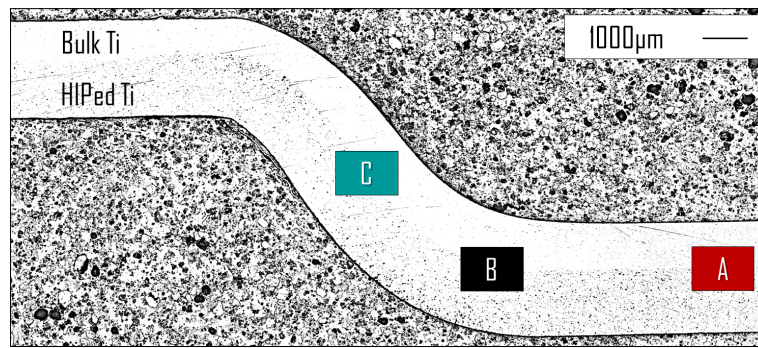


Figure 8 Macrograph of the three investigated areas: (A) Flat, (B) Corner and (C) Wall.

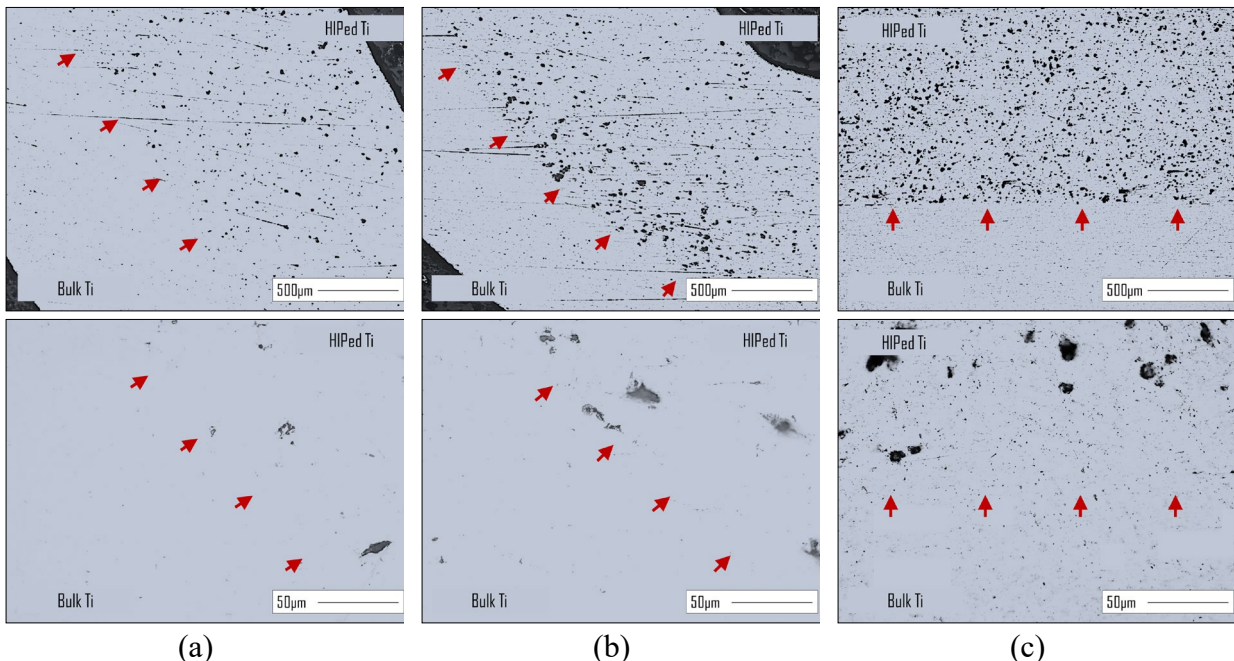


Figure 9 metallographic analysis at different magnifications referred to (a) the wall, (b) the corner and (c) the flat area of the hexagonal component.

For all the investigated areas it is possible to note that a complete adhesion of the two layers occurred; furthermore, the flat (Figure 8c) area and the one in the corner (Figure 8b) are more porous than the wall (Figure 8a), since they are the most deformed ones. Therefore, the energy supplied to the system during the combined process (DB and SPF) allowed not only to ensure the bonding of the two layers, but also to increase the level of porosity in the HIPed layer.

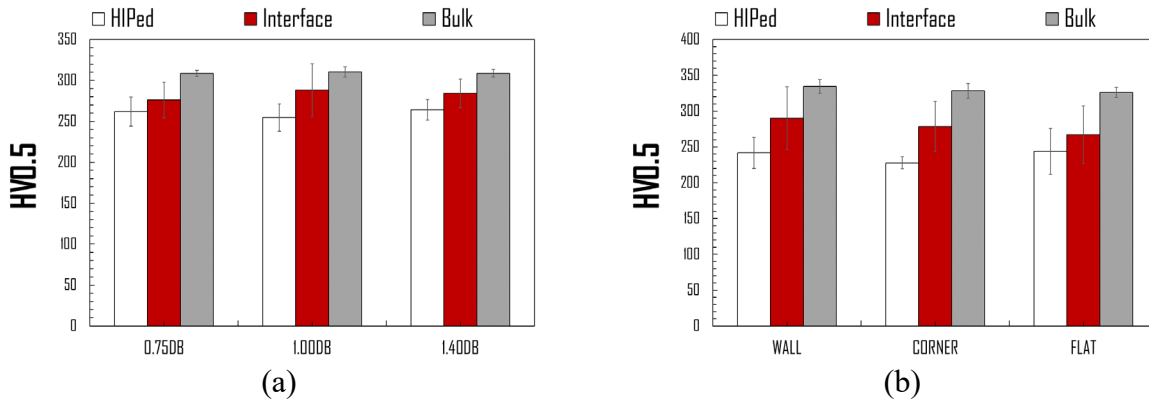


Figure 10 Microhardness analysis: (a) comparison between the different DB conditions and (b) comparison between the different areas referring to the combined process of DB and SPF.

From a mechanical point of view, Vickers microhardness analyses (Figure 9) confirm the effectiveness of the processes investigated since for all the cases analyzed (for DB tests) and the areas investigated (for DBF tests) the microhardness at the interface is intermediate between the one of the bulk layer and the one of the HIPed layer, guaranteeing a perfect merge of the mechanical properties of the two materials.

Conclusions

In the present work the diffusion bonding process aimed at the fabrication of a hybrid component characterized by two Titanium layers (one produced by rolling, one obtained through the HIP process) was investigated. In particular, the pressure levels applied during the solid state diffusion process (0.75, 1.00 and 1.40 MPa) for 240 minutes were able to produce the complete adhesion of the surfaces. With reference to the highest Argon pressure level (1.40 MPa) and by combining the DB process with the SPF process, it was possible to obtain a perfectly sound and continuous component at the interface. Furthermore, the effect of both temperature and pressure during the DB allowed to emphasize the foaming behavior of the HIPed Ti alloy; this is particularly evident when the deformation occurs (the growth of the porosity level is statistically significant). This aspect could be crucial in the design of hybrid structures for which specific mechanical properties influenced by the density of one of the two layers involved in the process are required. Finally, in order to fully investigate the obtained joints, future developments include in-depth analysis of the interface layer and any imperfections by means of more targeted analyses such as SEM and/or XRD.

References

- [1] B. Hamilton, S. Oppenheimer, D.C. Dunand, D. Lewis, Diffusion Bonding of Ti-6Al-4V Sheet with Ti-6Al-4V Foam for Biomedical Implant Applications, *Metall. Mater. Trans. B.* 44 (2013) 1554–1559. <https://doi.org/10.1007/s11663-013-9942-5>
- [2] B. Derby, E.R. Wallach, Theoretical model for diffusion bonding, *Met. Sci.* 16 (1982) 49–56. <https://doi.org/10.1179/030634582790427028>
- [3] O.D. Sherby, J. Wadsworth, *Superplasticity-Recent advances and future directions*, 1989. [https://doi.org/10.1016/0079-6425\(89\)90004-2](https://doi.org/10.1016/0079-6425(89)90004-2)

- [4] Y.H. Kim, J.M. Lee, S.S. Hong, Optimal design of superplastic forming processes, *J. Mater. Process. Technol.* 112 (2001) 166–173. [https://doi.org/10.1016/S0924-0136\(00\)00880-3](https://doi.org/10.1016/S0924-0136(00)00880-3)
- [5] W. Han, K. Zhang, G. Wang, Superplastic forming and diffusion bonding for honeycomb structure of Ti-6Al-4V alloy, *J. Mater. Process. Technol.* 183 (2007) 450–454. <https://doi.org/10.1016/j.jmatprotec.2006.10.041>
- [6] M. Dewidar, Mechanical and Microstructure Properties of High Porosity Sintered Ti- 6Al-4V Powder for Biomedical Applications, *JES. J. Eng. Sci.* 34 (2006) 1929–1940. <https://doi.org/10.21608/jesaun.2006.111343>
- [7] F.X. Zimmerman, Hot isostatic pressing: today and tomorrow, *J. Mater. Sci. Technol.* 1 (2008) 1–11.
- [8] A.T. Sidambe, Biocompatibility of advanced manufactured titanium implants-A review, *Materials (Basel)*. 7 (2014) 8168–8188. <https://doi.org/10.3390/ma7128168>
- [9] M. Sarraf, E. Rezvani Ghomi, S. Alipour, S. Ramakrishna, N. Liana Sukiman, A state-of-the-art review of the fabrication and characteristics of titanium and its alloys for biomedical applications, *Bio-Design Manuf.* 5 (2022) 371–395. <https://doi.org/10.1007/s42242-021-00170-3>
- [10] F. Li, J. Li, H. Kou, L. Zhou, Porous Ti6Al4V alloys with enhanced normalized fatigue strength for biomedical applications, *Mater. Sci. Eng. C.* 60 (2016) 485–488. <https://doi.org/10.1016/j.msec.2015.11.074>
- [11] P. Guglielmi, A. Piccininni, A. Cusanno, A.A. Kaya, G. Palumbo, Mechanical and microstructural evaluation of solid-state foamed Ti6Al4V-ELI alloy, *Procedia CIRP.* 110 (2022) 105–110. <https://doi.org/10.1016/j.procir.2022.06.021>
- [12] H.S. Lee, J.H. Yoon, C.H. Park, Y.G. Ko, D.H. Shin, C.S. Lee, A study on diffusion bonding of superplastic Ti-6Al-4V ELI grade, *J. Mater. Process. Technol.* 187–188 (2007) 526–529. <https://doi.org/10.1016/j.jmatprotec.2006.11.215>
- [13] D. Sorgente, G. Palumbo, A. Piccininni, P. Guglielmi, L. Tricarico, Modelling the superplastic behaviour of the Ti6Al4V-ELI by means of a numerical/experimental approach, *Int. J. Adv. Manuf. Technol.* 90 (2017) 1–10. <https://doi.org/10.1007/s00170-016-9235-7>