Innovative Developments in Virtual and Physical Prototyping – Bártolo et al (eds)
© 2012 Taylor & Francis Group, London, ISBN 978 0-415-68418-7

A study of mechanical and biological behavior of porous Ti6Al4V fabricated on EBM

V. Petrović, J.R. Blasco & L. Portolés
Metal Processing Research Institute AIMME, Valencia, Spain

I. Morales, V. Primo & C. Atienza
Instituto de Biomecánica de Valencia, Valencia, Spain

J.F. Moreno
Bio-Vac & Instituto Tecnológico de Materiales-UPV, Valencia, Spain

V. Belloch
ERESA, Valencia, Spain

ABSTRACT: EBM is a ALM technology capable of processing ferrous and non ferrous powders to fully-dense material using layer-by-layer principle. It is highly suitable for manufacturing of medical implants since it can fabricate designed and controlled porosity and tailored surface quality for the purpose of better bone in-growth. Due to great advantages in controlled geometry it has become important rival to other porous titanium materials available on the market, processed with different technologies such as space holder method [1], sintered microsphere porous coating, etc. This paper brings one of the most thorough analysis of mechanical testing of porous material made by EBM for the time being. So as to obtain a full picture of porous EBM Ti64, the results of testing are then compared to the properties of commercially available materials as well as to the human bone properties. In addition, some basic results of "in vivo" testing on bone in-growth of EBM specimens are also mentioned in this paper.

1 INTRODUCTION

Additive Manufacturing (AM) technologies have been available on the market for many years. Initially, these technologies were considered only for prototyping since the first technologies that appeared on the market were capable of fabricating only polymer parts of low quality and low resistance. However, in the last decade, the sector of AM has experienced an important evolution with constant growth in sales of machine systems and rapid products. Numerous advantages of 'freeform fabrication' have driven new developments in processing principles and materials. New value-added materials have been adapted for layer-by-layer processing. On the other hand, new technologies have been developed to process demanding materials for different sectors. New energy sources have been introduced in order to process high melting point metals such as Titanium, Cobalt Chromium, etc. One of the most powerful active principles is Electron Beam Melting that has been commercialized by the Swedish company Arcam®.

Electron Beam melting (EBM) is a free-form fabrication technology capable of processing ferrous and non ferrous powders to fully-dense material, using layer-by-layer principle. A 3D model is sliced electronically and the slices are "printed out" one upon each other to form a final part. This "3D printing" is done by selective melting of powder. Only the part of powder layer that corresponds to a part slice is melted; the rest of the powder remains un-melted. In the case of EBM, the energy source consists of an electric circuit that is formed between a tungsten filament placed inside of the electron gun and the building plate (Figure 1). A high voltage unit supplies 60kV to the filament which emits a beam of electric current that may vary between 0 and 50mA.

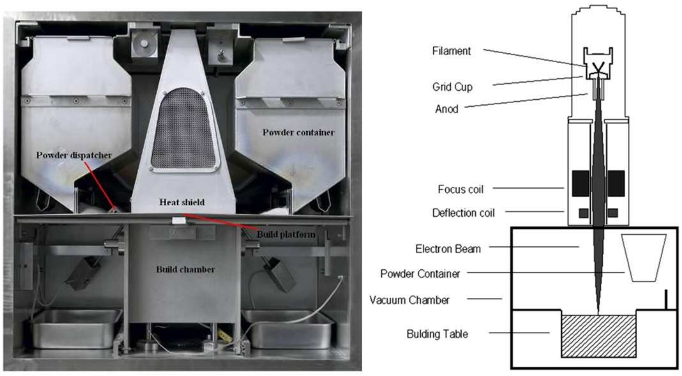


Figure 1. Fusing on EBM machine (a); scheme of an additive machine (b).

The electron beam is conducted by a set of different coils that guide, focus and orient it until it impacts the powder surface. During the impact, electric energy is transformed to heat which fully melts the powder. In order to prevent dispersion and deflection of the beam, the working chamber is kept under deep vacuum (order of magnitude 10^{-4} mbar). Hence, as in other layer-by-layer processes, powder is released from containers and distributed by a powder dispatcher over the build platform in fine 70-100 µm layers. The beam melts powder to a solid slice, merging it with previous slices. The build platforms descends for the value of layer thickness and a new powder layer is dispatched. The process repeats until the part is completed.

EBM has three major advantages in comparison with other AM processes:

- Due to high power (up to 3000W), the nominal speed of processing is 40-60 $^{cm^3}/_{h}$ which is substantially higher than in laser machines.
- Due to processing under vacuum (absence of oxygen), the processed material has very high purity which results in higher properties and better biocompatibility.
- The processing temperature in the build chamber is very high (in the case of Titanium alloys, around 650ºC). Hence, there is less difference between melting temperature and powder temperature and less thermal stresses, causing almost no warpage in processed material.

2 STATE OF THE ART

In numerous previous works, it was proven that freeform fabrication offers a step forward in providing additional value to the design of medical devices: prosthesis, orthosis and implants ([6], [7], [8]). However, for the time being no exhaustive study was found that demonstrates that advanced design is accompanied by good mechanical properties of porous material. In addition, there are other porous titanium materials available on the market. These materials are processed with different technologies such as the space holder method [1], sintered microsphere porous coating, etc. The present work was conceived to demonstrate that the porous material fabricated with EBM is equal or better in properties to commercially available alternatives.

In addition, for medical applications, a thorough analysis of any material is essential. There is an abundance of previous work regarding characterization of titanium foam. One of the most complete works has been published by Winkelried [1]. In porous tantalum foam, which is also commonly used in human implants, one of the most complete studies has been offered by Zardiackas et al [3]. Hence, these two works were used as a point of reference in this study. Regarding other efforts, Heinl et al [9] characterized porosity of Ti64 samples made on "selective EBM" with acid etched surface and performed bioactivity testing in SBF. Ponader et al [10] evaluate in vivo Ti64 samples made by sEBM by implanting in craneal zone of pigs. By micro X rays and hystomorphometrics study, the authors prove good interconnectivity of Ti64 scaffolds. Finally, Haslauer [12] tested biocompatibility of titanium alloy discs made using direct metal fabrication in vitro.

Regarding mechanical properties of porous titanium fabricated with EBM, only recently some relevant work has been encountered [4], [5], [11], [13]. However, the present study shows to be the most complete mechanical study of porous EBM titanium for the time being. Performed within the framework of the MEDIFUTUR project and initiated at the beginning of 2009, it was aimed to contribute with the full biomechanical characterization of EBM porous titanium.

For that purpose, the authors present and analyze the results of complete mechanical testing of porous material made by EBM. These results are then compared to the properties of commercially available materials as well as to the properties of human bone. In addition, some basic results of "in vivo" testing on bone in-growth of EBM specimens are also mentioned in this paper.

3 METHODOLOGY

3.1 *Selection of the pore type*

Porous material engineering is enabled by the use of specific software solutions for lattice structure generation. For the purpose of this study, Netfabb® by FIT was employed. This software allows definition of zones of different porosities on a model imported in STL (Figure 2b). The model is divided into cube units and a type of cell is assigned to each cube (Figure 2a). There is a huge number of cells that can be designed in Netfabb (Figure 2c). For the purpose of this project a square cell was selected (Figure 2d). This cell is determined by cube width (L), bar diameter (d) and pore diameter (D).

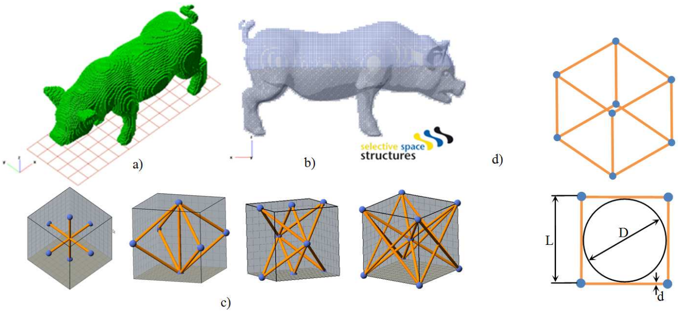


Figure 2. Modelling of porous material in Netfabb: a) model divided in cubes; b) model divided in different porous zones; c) types of cells; and, d) selected cell – frontal and isometric views.

3.2 *Selection of the pore size*

The power of electron beam is huge. Previous experience with EBM porous material indicates that, although designed to certain value, pore sizes are somewhat smaller when fabricated. It is supposed that this fact is due to excessive heat that cannot be evacuated instantly. More material is melted, bar cross-section is increased and consequently the pore size is smaller. In order to predict the deviation between designed pore and real pore, previous test fabrications have been performed. Table 1 shows some of the results of preliminary testing.

Table 1. Preliminary pore size measurement

Dimension	D	L	d	$\overline{D}_{real}$
	μm	μm	μm	μm
1	1067	2500	700	985
2	1067	2500	700	987
3	814	2000	600	645
4	814	2000	600	658
5	614	2000	800	521

A thorough revision of existing porous materials and living tissues brought us to the optimal range of pore size for bone in-growth is from 300 to 700 μm. This was the target range for this study. After the analysis of preliminary results, 3 different pore size samples were chosen in the above mentioned range. Accordingly, specimens with three nominal pore sizes: 600, 700 and 800 μm (P-600, P-700 and P-800) were preselected and tested again. As shown in Table 2, the measured values of pore size (Column 4) were inside the recommended range and the obtained porosity (Column 5) was relatively high (~ 50-65%).

Table 2. Preselected pore size measurement

d	D	$\overline{d}_{real}$	$\overline{D}_{real}$	% p
μm	μm	μm	μm	μm
450	600	666	376	49.7
450	700	647	504	57.5
450	800	577	681	66.5

3.3 *Testing*

The purpose of this study was the complete testing of porous titanium fabricated on EBM for biomedical use. On one side, this entails testing of porous material to all types of loads that implants are commonly exposed in the human body: tensile test, bending test, compression test and fatigue test. However, the biological behaviour of porous material is crucial for medical use. As mentioned before, this aspect was very important for choosing the testing samples' porosity. Hence, the samples were used for in vivo testing of bone in-growth and compared to commercial materials. All details on this are shown in the Results section.

4 RESULTS & DISCUSSION

4.1 *Test specimens*

Due to the absence of standards related to lattice structure testing, the decision was made to emulate the shape and size of the specimens used in previous work. Among relevant works used for the definition of mechanical tests are those of Imwinkelried et al. [1], Hong et al. [2] and Zardiackas et al. [3]. However, in contrast to other commercial materials (foams, porous coating, etc.), the porous material made by EBM is a composition of predefined cells and, as such, it has a regular and controlled porosity.

For each of the preselected porosities (P-600, P700 and P-800), the cell size is different (1.05, 1.15 and 1.25 mm, respectively). Therefore, it is not possible to define unique dimensions of specimen which would contain a round number of cells in all cases. Consequently, the specimens were designed as a function of the number of cells. Figure 3 shows different test specimens.

Table 3. List of test specimens and cell dimensions

Test type	Test specimen description	Nº of t.s.
Tensile	74x13x6.5 mm load zone: 8x4x20 cells	5
Compression	load zone: 10x10x10 cells	5
Bending	load zone: 8x4x20 cells	5
Fatigue	load zone: 10x10x10 cells	1

Cell type	L [μm]	d [μm]	D [μm]
P-600	1050	450	600
P-700	1150	450	700
P-800	1250	450	800

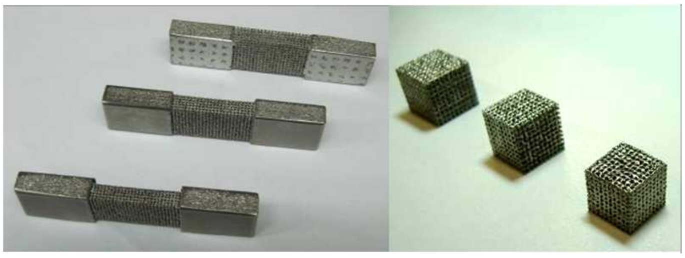


Figure 3. Test specimens for tensile and compression test.

All test specimens were fabricated on the Arcam A2 machine. The material used for fabrication is Ti6Al4V in powder form with 52-108 μm granulometry. The chemical composition of this material corresponds to the Ti6Al4V defined in Ti6Al4V-ISO 5832-3 "Implants for surgery - Metallic materials - Part 3: Wrought titanium 6-aluminium 4-vanadium alloy".

4.2 *Test results*

Tensile test

Table 4 represents the summary of tensile test results for porous titanium while the Figure 4 shows the comparison in achieved porosity and tensile strength to Ti foam.

Table 4. Results of tensile tests and comparison to titanium foam.

Cell type	Force [N]	Section [mm^2]	Rm [MPa]
P-600	7483	79.38	94.27
P-700	4082	95.22	42.87
P-800	5949	114.40	61.75

	Ti64 (EBM)		Ti64 (foam) [1]	
	Rm [MPa]	%p	Rm [MPa]	%p
P-600	94.27	49.7		
P-700	42.87	57.5	70	62.5+0.5
P-800	61.75	66.5		

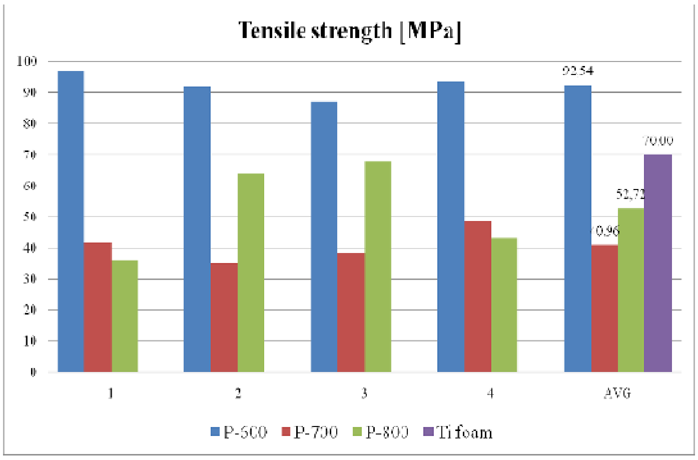


Figure 4. Comparative view of tensile test results.

The values shown in the table represent the average value of four test samples per each porosity. Unlike other tests where mechanical properties decrease with the increase of porosity, the lowest average tensile strength was detected in P-700. Nevertheless, the order of magnitude of tensile strength of EBM samples is similar to that of titanium foam.

Compression test

The following table shows the summary of the compression test results. In addition, a graph is shown to compare Ti64 made by EBM with other commonly used materials as well as trabecular and cortical bone. It may be perceived that the elastic modulus of EBM porous titanium is similar to that of porous tantalum, while the compressive strength is superior to that of tantalum, and to cortical and trabecular bone.

Table 5. Results of compression tests and comparison to commonly used materials.

Cell type	Section [μm]	Rm [μm]	E [MPa]
P-600	166.0	159.2	2615
P-700	139.4	183.5	2927
P-800	114.9	230.2	3288

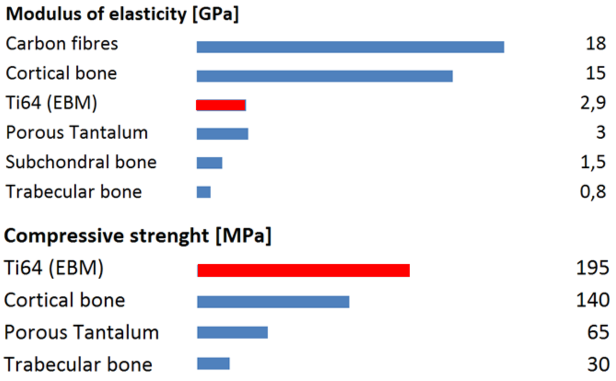


Figure 5. Comparative view of compressive strength.

Bending test

Table 6. Results of bending tests and comparison to titanium foam.

Cell type	Force [N]	Section [mm^2]	Rm [MPa]
P-600	651	33.6	149.67
P-700	611	36.8	101.98
P-800	401	40	64.23

	Ti64 (EBM)		Ti64 (foam) [1]	
	Rm [MPa]	%p	Rm [MPa]	%p
P-600	149.67	49.7		
P-700	101.98	57.5	105	62.5+0.5
P-800	64.23	66.5		

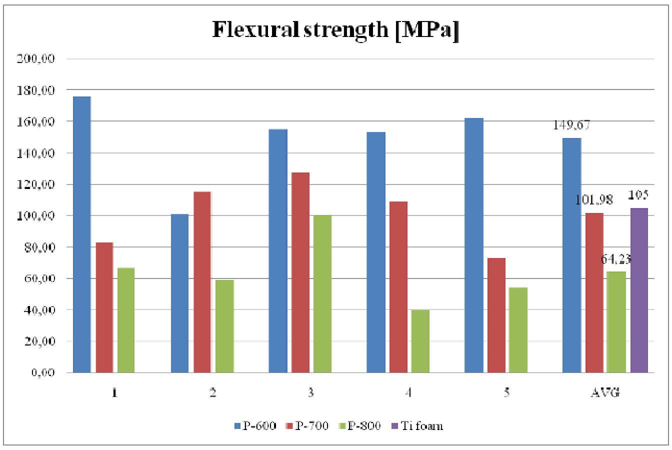


Figure 6. Comparative view of flexural strength.

As presented in Table 6 and Figure 6, porous EBM titanium shows very good flexural properties which decrease with the increase of porosity. Compared to Ti foam, it shows a similar order of magnitude.

Fatigue test


Figure 7. Compression fatigue test equipment.

Compression fatigue test was performed according to the Haversine sine cycle with the maximum force of F_m = 3820N and a stress ratio of R=0.1. The equipment is shown on Figure 7. The working frequency was 10 Hz. A limit of 5.000.000 cycles was established as the condition to end the test. All specimens passed the limit without any problem, showing reasonably good fatigue properties.

4.3 *Bone ingrowth testing*

The experimental model used in this study was condilar femoral medial defect in rabbits. Due to the elevated costs of in vivo testing and after the analysis of the mechanical results, the P-700 sample was selected for implantation. Five samples (shown at Figure 8) were fabricated to be implanted during 8 weeks, as this period was sufficient to show correct osseointegration. The samples were designed for pull-out testing: one part of the sample is implanted and the other has a hole to introduce the wire to perform the test. Porous titanium samples made on EBM were implanted together with two commercially available materials: samples with chemically etched surface and samples with sintered microsphere surface coating.

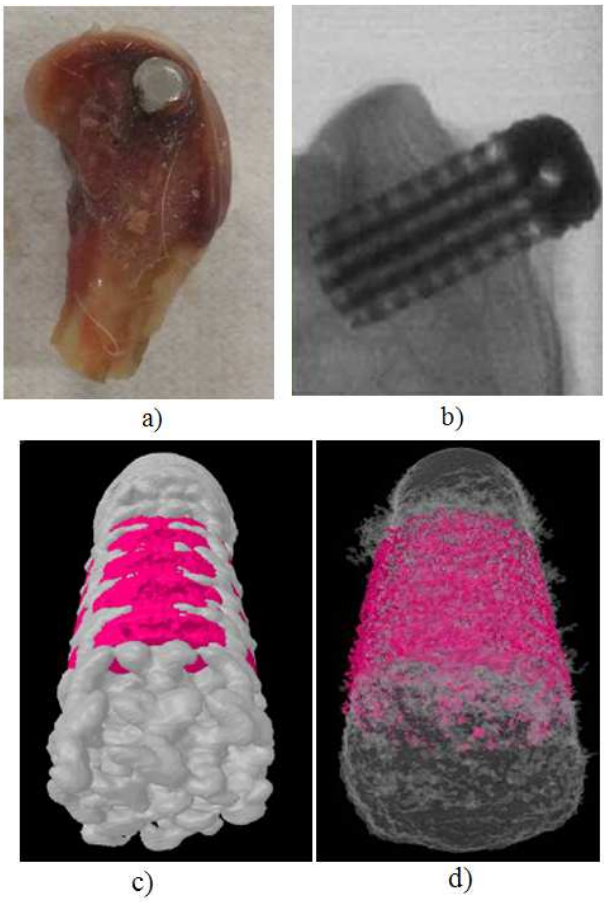

Figure 8. Test specimens for in vivo testing.

For evaluation of bone in-growth, EBM control samples were compared to samples provided by two medical device manufacturers, Biovac and Eckermann. Five samples of each type were implanted in the femur of rabbits (Figure 9 a and 9 b). The control period was 8 weeks. To compare the results of osseointegration between samples, a Region of Interest (ROI) was selected as a part of the sample completely introduced to the bone (delimited by fuchsia color on figure 9c and 9d). In the Table 7, V_{ROI} corresponds to a total volume of ROI, $V_{implant}$ is the volume taken by the implant and V_{bone} is the volume taken by bone cells. Finally, the last two columns show the percentage of remaining (void) volume taken by bone cells and the pull-out force necessary to extract the implant.

Table 7. Preliminary pore size measurement

Specimen	V_{ROI}	V_{impl}	V_{bone}	$\% \frac{V_{bone}}{V_{void}}$	$F_{pullout}$
	mm^3	mm^3	mm^3	%	N
Ecker 1	-	-	-	-	$212^{\pm65}$
Ecker 2	-	-	-	-	$212^{\pm65}$
Biovac 1	101.81	96.20	4.93	87.88	$967^{\pm323}$
Biovac 2	110.04	108.71	1.14	85.71	$967^{\pm323}$
P-700-1	107.66	35.35	45.96	63.56	$687^{\pm231}$
P-700-2	103.53	41.15	53.86	86.34	$687^{\pm231}$

Figure 9. a) Part of a rabbit femur after extraction; b) TAC of in vivo sample; micro TAC reconstruction of EBM (c) and Biovac



sample

The results show that the bone occupied a very high percentage of void space. Bearing in mind that the void volume in the case of EBM samples is much bigger than in the case of Bio-Vac, it has reached a surprisingly high level of in-growth (up to 86% of the void space was filled). Finally, no adverse biological effects, such as tissue inflammation or implant rejection, were noticed in these 8 weeks.

5 CONCLUSIONS

In this paper, a full biological and mechanical characterization of porous titanium made with EBM is offered. The results of this study show that the porous titanium made with EBM has mechanical properties similar to those of commercially available materials and can compete with them on an equal basis. Also, the biological behaviour of the studied material showed it to be very good with outstanding osseointegration and admirable pull-out results and, of utmost importance, without any adverse biological effects. These results back up the previous work already published in the literature [5].

On the other hand, engineered porous material built with EBM offers the possibility to have a designed and completely controlled, well-interconnected porosity with constant pore size. Also, freeform fabrication offers the possibility to build implants and prosthesis fully adapted to a patient's data with minimum post-processing.

In general terms, EBM represents a very good alternative to conventional processes used in medical device manufacturing. The results of this study show that, apart from being superior to alternative porous material regarding control and design of interconnected porosity, the porous titanium made on EBM is on the same level of mechanical properties as its commercial alternatives. In addition, the porous titanium made on EBM has shown to be bone ingrowth friendly. Hence, it is expected that in the forthcoming years EBM is converted into one of the references in the medical device manufacturing sector.

6 ACKNOWLEDGMENTS

The development of the MEDIFUTUR project, which results are presented in this paper, has been possible thanks to the financial support of the Government of the Valencian Community and its Council of Health through the framework of "Industrial R&D Projects of Special Relevance for the Valencian Autonomous Region".

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