Microstructure and micro-hardness analyses of titanium alloy Ti-6Al-4V parts manufactured by selective laser melting

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Abstract. Selective Laser Melting (SLM) is one of the powder based additive manufacturing technologies and it is, as well, the most rapidly growing technique in Rapid Prototyping. In this paper is presented a microstructure analysis using Scanning Electron Microscope (LEO 1525 SEM), of Ti6Al4V parts exposed into a corrosion environment. The corrosion environment was generated using a salt chamber with 5% and 10% NaCl concentration and an ACS-Sunrise climatic chamber. The parts were also subjected to tests in order to determine their micro-hardness, followed by a statistical processing of the obtained data. The parts, having a lattice structure, were built on a Selective Laser Melting machine.

1 Introduction

Additive Manufacturing (AM) of metal finished parts is well recognized as an interesting alternative to other conventional or unconventional processes, thanks to its capability to produce complex shapes and integrated parts of a high strength-to-weight ratio [1, 2].

In the recent years, AM has evolved from simple 3D printers used for rapid prototyping in which the most works has been built using polymer materials to sophisticated rapid manufacturing systems that can be used to create functional parts in different engineering materials directly without the use of tooling. The development of AM processes such as Selective Laser Sintering, Selective Laser Melting, Electron Beam Melting and Laser Engineered Net Shaping enabled to build parts by using metallic materials, metal matrix composites and ceramic materials. Today, Additive Manufacturing is frequently used in aerospace [3], automotive, medical fields and also in sustainable energy for building the components of small size and complex geometries [4-8].

Selective Laser Melting (SLM) is one representative of additive manufacturing processes [9, 10]. SLM is a layer-by-layer technology using a high-energy laser system. In order to manufacture a component with SLM technology the 3D CAD model is sliced into layers. The laser melts powder on a substrate plate selectively along scan tracks [11].

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Components manufactured with SLM offer a high geometrical flexibility and accuracy without almost any loss of material. It is well known that for obtaining components by SLM technology is used a narrow range of materials: cobalt-chrome, stainless steels, tool steels, aluminium alloys, gold alloys, nickel-based superalloys such as Inconel 625 and 718, and TiAl alloys [4, 5, 12]. The most popular for medical and aerospace applications, and not only, is the titanium alloy Ti-6Al-4V.

Among first studies of processing titanium and its alloys by Selective Laser Melting were focused on Ti-6Al-4V. Therefore, the properties of this material have been extensively examined. The influence of the process parameters like the scanning strategy on the microstructure of Ti-6Al-4V, for instance, was investigated by Thijs et al. in [13]. Vandenbroucke et al. [14] determined the mechanical properties in tensile load of SLM fabricated Ti-6Al-4V. Studies of Vrancken et al. [15] pointed out that conventional post treatments, which are used to enhance mechanical properties, appear different to additively processed Ti-6Al-4V. Another study by Vaithilingam et al. dealt with the remelting during the SLM process of Ti-6Al-4V [16]. In [17] Vickers hardness evolution of the as-received Ti-6Al-4V alloy as a function of the post heat treatment is investigated. In [13] is revealed that the micro-hardness of as-built SLM Ti-6Al-4V varies with the processing parameters and is normally in the range of 3.43–4.90 GPa and in [18] the micro-hardness of the struts of as-made Ti-6Al-4V TPMS (Triply periodic minimal surface) structures was measured to be 4.01 ± 0.34 GPa [19].

In this study, micro-hardness of the specimens manufactured by selective laser melted Ti-6Al-4V alloy was evaluated by using a Vickers hardness tester for establishing the influence of corrosive environments on this property. The specimens made of titanium alloy, Ti-6Al-4V, before determining the micro-hardness were subjected to corrosive environments in saline and climatic chamber.

2 Methods samples and materials

This chapter presents the equipment used for manufacturing the specimens and the equipment used in performing corrosion tests. Finally are presented the measurement instruments used to determine the micro-hardness and the SEM analysis.

2.1 Parts manufacturing through a selective laser melting process

Selective Laser Melting (SLM), sometimes referred to additive manufacturing, or rapid manufacturing, is a 2.5 axes manufacturing process used to produce complex shape parts, starting from the CAD 3D model, saved as *.stl (STereoLithography) file. During the manufacturing process, a very fine powder of metal (usually from 20 to 100 micrometres thick) is applied in successive layers and melted, layer by layer, with a laser beam, to create a 2D image of each layer.

The principle of the method consists in sectioning the piece with a beam of parallel planes and its construction layer by layer on a SLM machine as follows: the machine table is covered with the first layer of powder; the first layer of the physical model is created by melting the metal powder along its section geometry; the machine table is then lowered with the layer thickness, and the process is repeated until completion of the model.

The system uses atomized metal powders to produce dense metal parts of aluminium alloys, titanium, stainless steel, cobalt-chromium or tool steel but the number of compatible materials increases year by year.

Selective Laser Melting (SLM) technology is used to manufacture metal parts that can be used in different fields as: engineering, aerospace, electronics, orthopaedics, dental care etc. The SLM principle is presented in figure 1.

Fig. 1. Schematic of a typical SLM process and the SLM machine.

2.2 The geometry of samples

The samples are obtained from a Titanium alloy Ti6Al4V through selective laser melting on a SLM250HL machine. The samples have a lattice structure with the shape and dimensions presented in figure 2.

Fig. 2. The samples shape and dimensions.

2.3 Corrosive environments description

After obtaining the samples they were grouped in four sets. Each set, without the first, was placed into a corrosive environment as follows:

- the first set of specimens - without any tests;
- the second set of specimens were hold into a saline chamber with 5% NaCl concentration with an immersion time of 72h;
- the third set of specimens were hold into a saline chamber with 10% NaCl concentration with an immersion time of 144h;

- the fourth set of specimens were hold into a climatic chamber with a combined test: temperature, humidity and ultraviolet radiation (Fig 3).

Fig. 3. The ACS-Sunrise climatic chamber and stress profile diagram (humidity, temperature, UV).

2.4 Microstructure analysis equipment

These conclusions were elaborated after analysing the micro-structural characteristics, at a scanning electron microscope (figure. 4), of all the specimens, before and after passing the testing procedures.

Fig. 4. The scanning electron microscope (LEO 1525 SEM).

A scanning electron microscope (SEM) is used for morphological, chemical and micro-structural characterizations of materials.

A high resolution, field emission LEO 1525 SEM is used. The nominal resolution of this microscope, obtained with a working distance of 2 mm, is 1.5nm at 20kV and 3.5nm at 1kV. Magnification ranges from 20x up to 500kx.

The SEM is equipped with an Energy Dispersive X-ray (EDX) microanalysis which provides both qualitative and quantitative information about the chemical composition of the sample, and with an Electron BackScattered Diffraction (EBSD) system that allows resolving local crystallographic orientations in the range of 100nm.

2.5 Micro-hardness testing equipment

The samples were cut transversally, were embedded in epoxide resin and prepared metallographic for the structural analysis used to determine the hardness values. For polishing the samples is used a Buehler Phoenix Beta Grinder apparatus (Fig. 5).

Fig. 5. BUEHLER Phoenix Beta Grinder Polisher.

When determining the micro-hardness values for the specimens of Ti6Al4V alloy is used the Vickers method which is based on the field size diagonal measurement obtained after a light load pressing of a diamond pyramid into the researched material grains. For the micro-hardness tests of titanium alloy Ti6Al4V specimens, they were transversely sliced, embedded in an epoxy resin and then was performed a grinding operation by using sandpapers with a gradual granulation change (1500, 2000 și 2500) using Buehler-Phoenix-Beta-Grinder-polisher.

It is very important for the sliced parts to present a smooth surface without cutting signs, after the cutting operation, because these imperfections can influence the accuracy of the micro-hardness tests. The micro-hardness measurements were performed on the Micro Hardness Tester FM-700 (Fig. 6), the penetrator was applied for 15 seconds using a 100 gf load.

Fig. 6. Micro-hardness Tester FM 700.

3 Results and discussions

3.1 Microstructure analysis results

When finishing the corrosion tests, important information about the corrosive resistances of our specimens was obtained by using the scanning electron microscope for morphological,

chemical and micro-structural characterizations of materials. The following figures (Fig. 7 Fig. 8 and Fig. 9) presents the micro-structural characteristics changes of the analysed material, before and after the immersion into the chamber with saline atmosphere, for 72 hours, using saline solution of NaCl (sodium chloride) of 5% concentration and for 144 hours, using saline solution of NaCl of 10% concentration, at different magnifying scales. The results are presented below:

Fig. 7. Samples before and after the immersion into the saline chamber at 10000x magnifying scale.

Fig. 8. Samples before and after the immersion into the saline chamber at 1000x magnifying scale.

Fig. 9. Samples before and after the immersion into the saline chamber at 500x magnifying scale.

3.2 Micro-hardness test

Within the Vickers test, using penetrators with identical angles at the top, identical geometrical traces are obtained regardless of the loading force and, as a result, it can be said that the hardness is independent by the load size.

Due to the small depth of the penetrator ingress into the material, the method can be used also to determine the hardness of small parts, thin parts or for heat treated layers. For the micro-hardness determination of thin layers are performed several tests and only the results with closed values will be retained.

All the specimens were manufactured on a SLM machine with the objective to determine the characteristics of their micro-hardness (Table 1). The SLM machine has the following building parameters [20]:

- laser power 100 [W];

- laser scanning speed 150 [mm/s];
- hatch offset 0.175 [mm];
- layer thickness 0.05 [mm];

Table 1. The hardness values of specimens.

Micro-hardness for the first [HV]	Micro-hardness for the second specimen 5%NaCl/72h [HV]	Micro-hardness for the third specimen 10%NaCl/144h [HV]	Micro-hardness for the fourth specimen climatic chamber [HV]
380.7	383.1	445.2	400.1
390.9	388.4	350.2	430.7
420.5	386.6	384.9	472.1
411.6	413.4	386.6	420.3
414.6	407.2	389.8	416.5
393.1	362.4	347.2	407.6
449.6	357.0	389.1	457.6
426.2	436.6	315.3	443.0
440.8	397.5	434.9	462.7

3.3 The statistical analysis of the micro-hardness tests data

The statistical analysis of the micro-hardness tests data for all the specimens was accomplished using the SPSS Statistics 23 software. The obtained results were described by using the distributions of the occurrence frequencies (Fig. 10). Because the average and median values shows relatively equal for all the four tested samples, we can say that the distribution is normal, with a slight positive or negative asymmetry [21].

Fig. 10. Histograms of the three analysed samples at the micro-hardness tests.

After determining the micro-hardness of the titanium alloy Ti6Al4V samples, manufactured with SLM technologies, making the micro-hardness average values (Fig. 11) of the workpieces surface, was obtained an hardness average value of 405.94 HV0.1. In Fig. 10 it can be observed that a large proportion of the micro-hardness values, for the analyzed samples, are summarized in the rectangle described by the 25th percentile and the 75th percentile.

Fig. 11. Boxtype diagram representing the micro-hardness values for the tested samples.

Table 2 describes the main statistical indicators of the micro-hardness values for all the tested specimens. Each statistical indicators value transmits a certain amount of information concerning the data for which they were calculated. For the micro-hardness values of all the specimens, the statistical indicators provide information about the central tendency of the series values (mean, median and module), others give us information about scattering values (dispersion, standard deviation and coefficient of variation) and others give us an indication of symmetry values in the series (median and quartiles).

Table 2. Statistical indicators of the micro-hardness values.

Specimen		1	2	3	4
Mean		414.22	392.46	382.57	434.51
Std. Error of Mean		7.69	8.27	13.74	8.54
Median		414.60	388.40	386.60	430.70
Mode		380.70	357.00	315.30	400.10
Std. Deviation		23.08	24.82	41.23	25.64
Variance		532.709	616.11	1700.20	657.73
Range		68.90	79.60	129.90	72.00
Minimum		380.70	357.00	315.30	400.10
Maximum		449.60	436.60	445.20	472.10
Sum		3728.00	3532.20	3443.20	3910.60
Percentiles	25	392.00	372.75	348.70	412.05
	50	414.60	388.40	386.60	430.70
	75	433.50	410.30	412.35	460.15

4 Conclusions

After completing the tests and analysing the obtained results, the following conclusions can be drawn:

- the specimens tested in the Sunrise climatic chamber didn't present significant changes within their micro-structural characteristics;
- the specimens tested in the chamber with saline atmosphere presents significant changes within their micro-structural characteristics with attached salt particles on their structure, due to the corrosion phenomena;

- the micro-hardness decreases for the specimens which were placed into the saline chamber, due to the corrosion phenomena
- the micro-hardness increases for the specimens exposed to the climate chamber, where the main stress factors were temperature and humidity, due to the heat treating of the surface.

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