# Effect of Surface Treatment and Heat Treatment on the Microstructure and Mechanical Properties of Ti6Al4V Alloy Manufactured by Selective Laser Melting

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## Abstract

One of the most attractive and widely used alloys in the industry and field of implants is titanium and titanium alloys, including Ti6Al4V. The outstanding properties and application of this with the attractive capabilities of additive manufacturing technology have increased the inclination towards additive manufacturing of titanium parts. In this research, the effect of surface treatment and heat treatment on the microstructure and mechanical properties of Ti6Al4V alloy manufactured by selective laser melting was investigated. For this purpose, Ti6Al4V alloy produced by selective laser melting was subjected to annealing heat treatment at 1050 degrees Celsius and surface treatment of surface ultrasonic mechanical stimulation. Then, the microstructure and phases of Ti6Al4V alloy, which included α and β phases, were investigated with optical microscopy and X-ray diffraction analysis. The mechanical properties of the samples were also checked by Vickers hardness, tensile and uniaxial compression tests. The results showed that annealing heat treatment and then aging decreases the strength properties but increases the flexibility and toughness. By performing surface treatment, the hardness of Ti6Al4V alloy increases. In general, it can be said that the desired properties of this alloy, produced by selective laser melting, can be obtained by performing suitable surface and thermal treatments.

***Keywords:*** Ti6Al4V, Mechanical Properties, Selective Laser Melting, Heat Treatment, Surface Treatment.

## 1. Introduction

Among the family of titanium alloys, Ti6Al4V alloy is one of the most widely used alloy, which was initially developed for aerospace applications, but now it is used in various applications, especially medical implants. This alloy is one of the two-phase α+β alloys. In the continuation of development, by removing interstitial elements, a version without interstitial elements was introduced as Ti6Al4V-ELI. Additive manufacturing of Ti6Al4V alloy has been welcomed by researchers and industries and many works have been done on it. One of the methods of additive manufacturing of Ti6Al4V alloy is selective laser melting, which is widely used for research and industrial applications of this alloy[1,2]. The Ti6Al4V alloy produced by the selective laser melting method needs modifications in terms of surface quality to be suitable for the desired applications such as implants, on the other hand, the microstructure resulting from the selective laser melting method is not the same in all directions, and as a result, the mechanical properties of different grades are different and we are also facing the remaining tensions. Therefore, it is necessary to obtain a part with desirable properties by performing heat treatment and surface treatment[2,3].

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Yan et al [1] fabricated Ti6Al4V-ELI samples by SLM method and then performed ultrasonic surface mechanical attrition treatment (SMAT) to improve a microstructural layer of Ti6Al4V-ELI to increase fatigue performance. They stated that drastic changes in the microstructure occurred due to severe plastic deformation and that the fatigue properties of the SMAT treated sample were significantly higher than that of the untreated sample. Also, Navarro et al. [2] investigated the effect of several surface treatments on the fatigue properties of Ti6Al4V alloy produced by SLM method. They observed that the laser shock peening operation resulted in the best fatigue behavior. In another study, Zhang et al [3] produced Ti6Al4V alloy by SLM method and then evaluated the effect of different heat treatments on its microstructure and compressive properties. They observed that the microstructure in the as-built state mainly consists of α′ needle martensite structure and most of β is converted to martensite, but the microstructure in all heat-treated samples contains a mixture of α and β phase and the size of α grains during the heat treatment has increased, and the α layers have become coarser with the increase of the heat treatment temperature. Liu et al. [4] consider the tensile performance of Ti6Al4V samples produced by SLM method to be highly dependent on the additional heat treatment temperature and state that as the heat treatment temperature increases, needle martensite of α′

transforms into coarser α layers and as a result, the tensile strength decreases and overall flexibility increases.

Although various works have been done on heat treatment and surface treatment of Ti6Al4V alloy manufactured by selective laser melting method, there is still a need to conduct more studies and research in this field in order to improve the surface and mechanical properties.

Therefore, in the present work, the effect of heat treatment and surface treatment on the microstructure and mechanical properties of Ti6Al4V alloy produced by selective laser melting is investigated.

## 2. Materials and Methods

To manufacture Ti6Al4V alloy by laser selective melting method, spherical plasma atomized powder of Ti6Al4V-ELI grade 23 alloy with a size range of 20 to 53 micrometers (with the highest abundance in diameter of 37 micrometers) from AP&C company was used. The used powder has a chemical composition of Ti 90%, V 4%, Al 6% and the amount of carbon, nitrogen, hydrogen, iron and oxygen elements is less than 1% in total. A selective laser melting machine (SLM) Noura M120 model was used to manufacture the sample under a very pure and neutral argon gas atmosphere. SLM samples with the dimensions of tensile test sub-size sample and compression test sub-size sample (a cylinder with a diameter of 8 mm and a height of 12 mm) were made four of each. Annealing heat treatment was performed above β transformation temperature at 1050°C for 60 minutes, then quick cooling (quench) in water and then aging at 600°C for 2 hours were done. In this research, ultrasonic surface mechanical attrition treatment (SMAT) method was used for surface treatment. In order to evaluate the simultaneous effect of heat treatment and surface treatment on mechanical properties, SMAT method was also implemented on one of the samples after heat treatment. The samples were named as as-built (Ti6Al4V), heat treated (HT), surface treated (ST) and heat treated and surface treated (HT + ST). Microstructural studies were done with optical microscope and phasing of samples was done with X-ray diffraction (XRD) analysis. In order to check the mechanical properties, hardness, tension and compression tests were used.

## 3. Results and Discussion

X-ray diffraction analysis (XRD) was performed on the samples with the aim of investigating phase changes and detecting new phases, the results of which are shown in Fig. 1. In the samples heat treated at 1050 degrees Celsius, the intensity of peaks related to α phase has increased compared to the sample without heat treatment. Also, due to the overlap of one of the peaks related to phase β by the main peak and high intensity related to phase α, the intensity of the peaks related to this phase has also increased. Therefore, it can be seen that this β phase also became coarser at this heat treatment temperature, but α phase prevented its further growth and prevented it from becoming coarser.

Also, by examining and analyzing the X-ray diffraction results by Expert software, it is clear that in the temperature range of 1050 degrees Celsius, the thickness of the α phase has increased with the increase in the storage time at this temperature. In general, the size of the β phase is smaller, which is consistent with the optical microscope images of the microstructure resulting from the heat treatment.

From the results of Fig. 1., it is clear that ultrasonic surface mechanical attrition treatment causes distortion in the structure and broadening of the peaks, which according to Scherer and Williamson-Hall relations, this peak broadening indicates two factors including reducing the crystal grain size and increasing the strain of the internal network. By comparing the diffraction pattern of Ti6Al4V-ST with the diffraction pattern of Ti6Al4V, the result will be obtained that due to the surface work on the sample, dislocations move and lock them together, and finally the intensity of the peaks compared to the witness sample has increased.

The microstructure of Ti6Al4V alloy always has some residual β phase. The difference in the cooling rate for samples with the same composition leads to the difference in the alloy structure. Changing the cooling rate also affects the dispersion and dimensions of α-phase plates. At a high cooling rate, α phase is formed on the interface of the β phase and its size decreases with the increase of the cooling rate.

Fig. 2.A shows the optical microscope image of the microstructure of the sample produced by the selective laser melting process in the manufacturing direction. In this figure, the structure is made of α phase grains that are layered inside the β columnar grains, which were created at the beginning of the manufacturing process. The dark phase corresponds to the β phase and the light phase corresponds to α phase. α phase grows in layers and is formed in the form of a Wiedmann-Statten structure and a tissue basket in different sizes and directions. In the created microstructure, the tissue basket structure and α+β layers, which are inside the initial β phase layers, can be seen. The resulting microstructure is consistent with previous research such as the research conducted by Rafi et al. [5]. Primary β-grains are obtained as a result of a large temperature gradient, which leads to strong sedimentary growth along the construction direction [6].

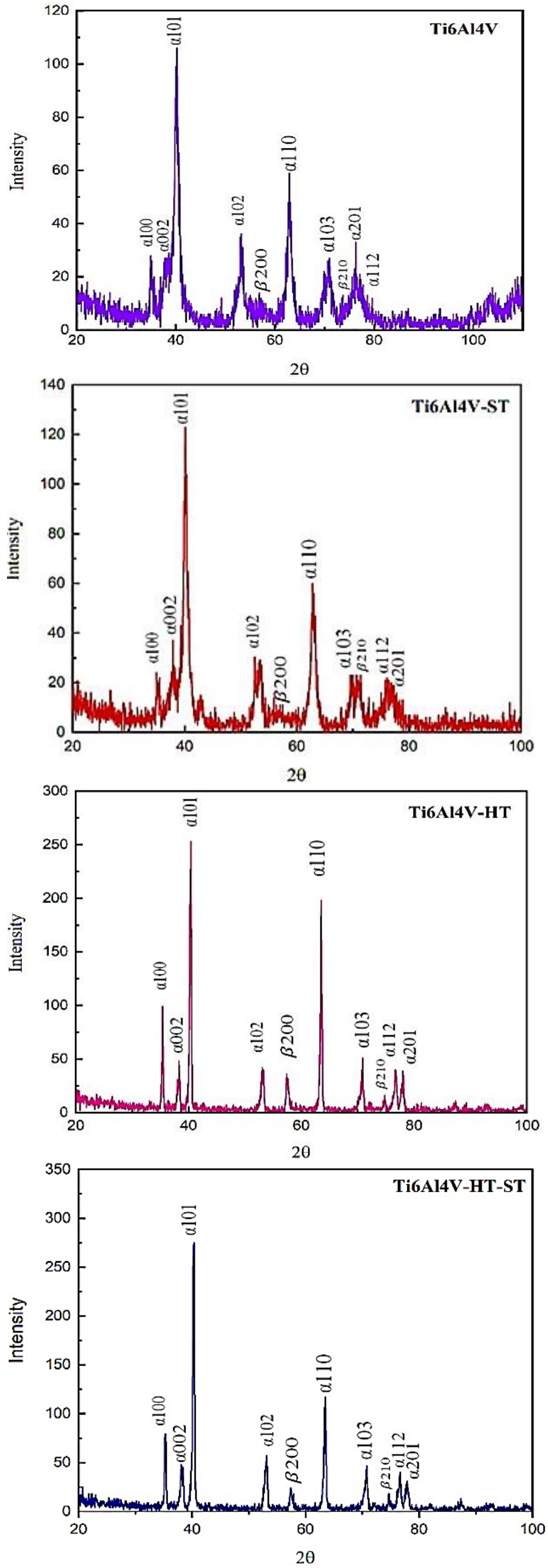


**Fig. 1. X-ray diffraction test results of different samples.**

According to Al-Bermani et al.'s research [7], it was reported that the <001> direction of growth during solidification is known in cubic crystals. Therefore, this direction tends to align with the direction of the maximum thermal gradient in the material, that is, perpendicular to the surface of the melt pool.

As a result, β grains preferably grow parallel to the construction direction. In this figure, the martensitic phase can be seen as blades. Melting of the powder and its rapid freezing with a high cooling rate leads to the formation of supersaturated phase of martensite of α′ and its immediate decomposition into α and β phases at high temperature (587°C) in the manufacturing chamber. Due to the high cooling rate, the formed β phase is very fine [8].

In the resulting microstructure after annealing heat treatment at 1050°C (Fig. 2.b), the grains have changed from columnar to co-axial and β+α microstructure can be seen. In this heat treatment, the α phase is completely dissolved during the heating caused by this heat treatment temperature, and the final microstructure and the secondary α phase after cooling, which depends on the cooling rate, remain constant.

According to Fig. 2.b, the resulting microstructure of the Ti6Al4V-HT sample is still Widmanstatten in some places, and due to the short time (60 minutes) the phase α is not completely dissolved at first, so the initial phase α is still observed in the form of a fine Wiedmann-statten microstructure and in some places the co-axial layered microstructure of α is also seen.

This coaxial structure increases in longer times. At temperatures above the phase transformation temperature, there are no longer barriers to β-grain growth.

Therefore, in the case of heat treatment above the phase transformation temperature, rapid growth of β-grains occurs, which leads to the formation of coaxial β-grains, which can be up to one millimeter as measured from optical microscope images. According to Fig. 2.b for the heat treated Ti6Al4V-HT sample, after cooling down to room temperature, β-α transformation has led to the formation of fine needle layers in some places. But with the increase of the storage time at this temperature when cooling is done, α phase first preferentially nucleates in the boundaries of the β grain and as a result, a continuous $\alpha_{GB}$layer is formed; which can be seen in microscopic images. After that, α sheets nucleate from this $\alpha_{GB}$layer and grow between β grains which are parallel sheets.

These layers grow in the β grain until they reach other α colonies germinated in other border areas of the grain. in addition, at heat-treated Ti6Al4V-HT sample, according to the microstructure observed in the optical microscopic images, in some of the parent β grains, the selection of a specific cluster leads to the existence of about three α layers, all of which have the same direction [9].

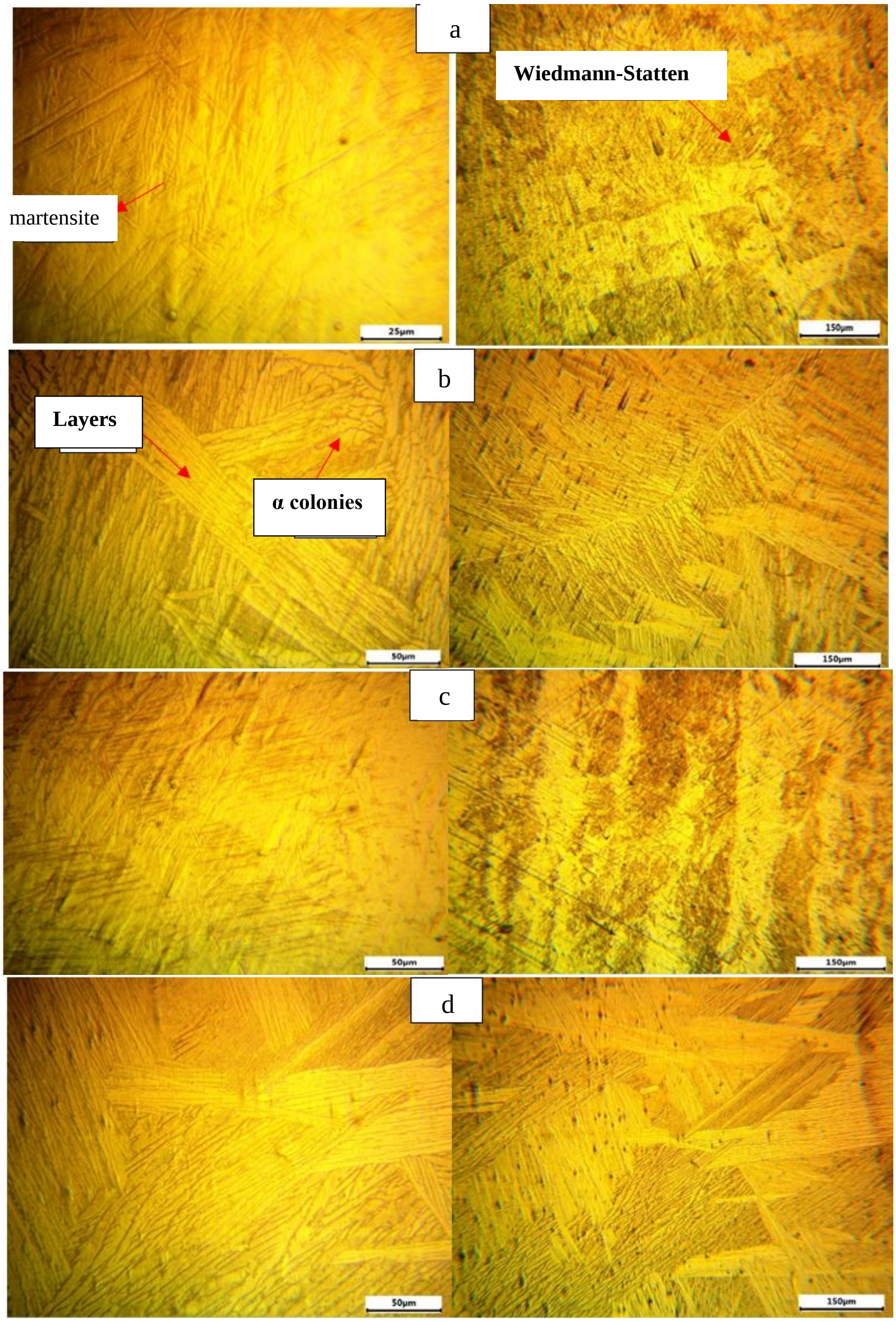


**Fig. 2. Optical microscopic images of the microstructure of samples a) Ti6AL4V, b) Ti6AL4V-HT, c) Ti6AL4V-ST and d) Ti6AL4V-HT-ST produced by SLM method in different magnifications.**

**Table. 1. Results extracted from tensile test charts for different samples.**

| **Characteristic / Sample** | **yield strength (MPa)** | **Young's modulus** | **Ultimate strength (MPa)** |
|---|---|---|---|
| Ti6Al4V | 1080 | 168.69 | **1155** |
| Ti6Al4V-HT | 860 | 178.73 | **879** |
| Ti6Al4V-ST | 1500 | 174.79 | **1207** |
| Ti6Al4V-HT-ST | 840 | 177.57 | **853** |

Table. 1. given the results of the tensile test for different samples. An easier comparison of the results is possible by Fig. 3. According to the information in the table, it can be seen that the choice of type of heat treatment cycle and surface treatment has a great effect on the mechanical properties of the alloy. The mechanical properties reported for Ti6Al4V alloy samples produced by selective laser melting are different among different sources.

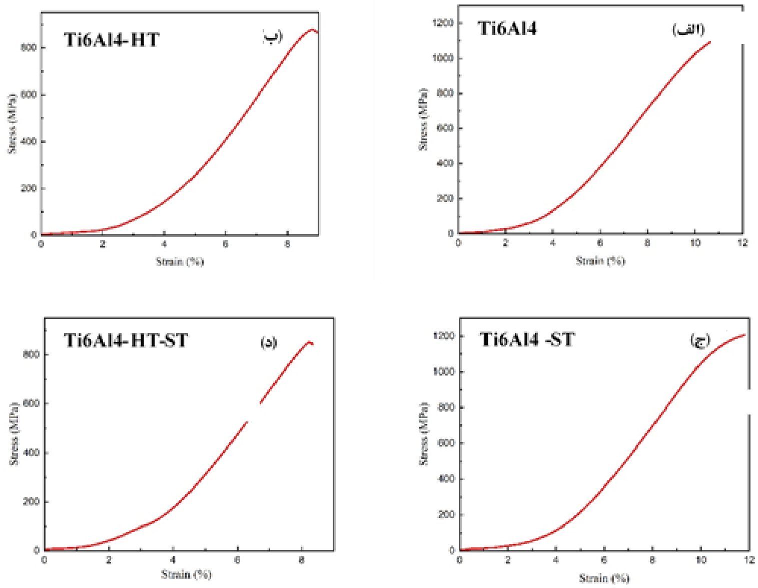


**Fig. 3. Stress-strain diagrams obtained from tensile test for different samples.**

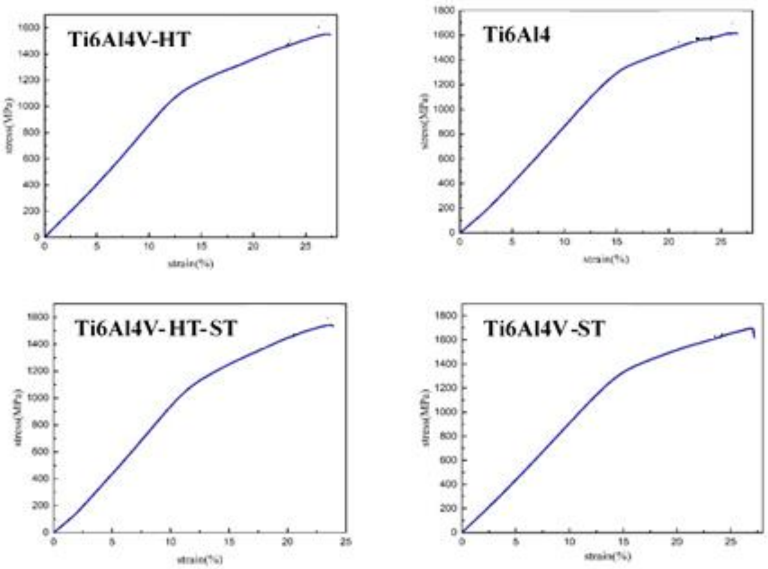


**Fig. 4. Stress-strain diagrams obtained from the pressure test for different samples**

**Table. 2. Results extracted from compression test graphs for different samples.**

| Characteristic Sample | Elastic Modulus. | Compressive strength (MPA) | (MPa)Flow stress |
|---|---|---|---|
| Ti6Al4V | 92.21 | 1617 | **1275** |
| Ti6Al4V-HT | 90.83 | 1552 | **1080** |
| Ti6Al4V-ST | 94.35 | 1695 | **1235** |
| Ti6Al4V-HT-ST | 100.28 | 1541 | **1070** |

**Table. 3. Hardness measurement results of alloys before and after heat treatment and surface treatment.**

| Sample | Ti6AL4V -HT-ST | Ti6AL4V -ST | Ti6AL4V -HT | Ti6AL4V |
|---|---|---|---|---|
| Hardness (HV) | 387.2 | 388.6 | 385.1 | 386.8 |

Facchini et al. [10] found that the ultimate tensile strength (UTS) of the samples made by SLM process is higher than the samples produced by the traditional and annealed method. While Koike et al. [11] have said that the ultimate elasticity and flexibility of the samples produced by the traditional method and casting of Ti6Al4V alloy is higher than the selective laser melting process.

The reason for the significant difference between the apparently similar studies can be attributed to the variation in the manufacturing parameters, which leads to the change of the various characteristics of the material such as composition, microstructure, the pore size and porosity distribution. Other parameters such as sampling or its location on the tension arms can also affect the microstructure of the parts produced by the selective laser melting process.

Rafi et al. [5] have investigated and compared the mechanical properties of Ti6Al4V alloy produced by selective laser melting and electron beam melting. There was a fundamental difference in the tensile properties of the samples compared to the ASTM standard, and according to the tensile results, the tensile strength of the laser melting samples was higher due to the higher cooling rate that led to the creation of a martensitic structure compared to the layered structure in the process of electron beam melting. Also, another report showed that the yield strength of the samples of the laser melting process was higher than the yield strength of the samples made by the electron beam melting method due to the higher martensitic phase, but the electron beam melting samples had more flexibility. In the present study, the stress-strain curves have almost the same slope, which indicates that Young's modulus is close to each other. The ultimate tensile strength of the Ti6Al4V-HT sample is lower than that of the Ti6Al4V sample, which can be explained by the fact that the α martensitic phase has become a softer α phase, which has a lower tensile strength than martensite. The latent tensile strength of the Ti6Al4V-ST sample has increased compared to the Ti6Al4V sample. This is because the surface treatment causes locking of dislocations and strain in the network, which increases the ultimate tensile strength. In the last sample, Ti6Al4V-HT-ST, the ultimate tensile strength is the same as that of Ti6Al4V-HT and Ti6Al4V sample, which is caused by α phase in the structure. Compression test was performed at ambient temperature with AGX machine with 100kN load cell and constant displacement of 0.04 mm/s.

Theuniaxial compression test was performed according to the ISRM standard for Ti6Al4V alloy samples. A stress-strain diagram was drawn for each sample to check the behavior of the sample. Fig. 4. is related to the stress-strain diagrams of the tested samples. Table. 2. given the results of the Compression test for different samples. Examining the stress-strain behavior of samples under pressure loading is opposite to the tensile test in terms of the direction of force application, but the principles and arguments used in this test are the same as the tensile test. The stress-strain curves in Fig. 4. have almost the same slope, which indicates that the elastic modulus of the samples is close to the same. The results show that the compressive strength of the Ti6Al4V sample increased with surface treatment and decreased with heat treatment. In the Ti6Al4V-HT-ST sample, because the heat treatment was performed first, the compressive strength of the sample decreased and became even lower than the Ti6Al4V-HT sample. Also, to compare the mechanical properties of the samples, their hardness was calculated using three points in the cross section of the sample. Hardness calculation has been done under the condition of 30 kg force and 15 seconds’ time. By averaging three values for each sample, the hardness number has been calculated, ( Table. 3.) According to the results, the sample produced by the selective laser melting method was transformed into α-coaxial layers due to the heat treatment of the martensitic hard phase, which caused a decrease in the hardness of the heat-treated sample By performing surface treatment on Ti6Al4V alloy, its hardness number has increased from 8.386 Vickers to 6.388 Vickers, which can be justified as that by performing surface treatment on Ti6Al4V alloy, the dislocations locked together and the internal strain of the network increased, which increased the stiffness. Finally, by performing heat treatment and surface treatment, the hardness of the Ti6AL4V-HT-ST sample increased compared to the Ti6AL4V sample.

## 4. Conclusion

1. The structure of the Ti6Al4V alloy sample includes α phase and the initial β phase, which was shown as a column in the manufacturing direction.
2. In the heat-treated sample above the β to α phase transformation temperature (1050°C), the microstructure changed from a columnar state to a co-axial state and a layered β+α microstructure was formed, which when converted to the microstructure $\alpha_{GB}$, the orientation of the layers changed.
3. The highest tensile and compressive strength was observed in surface treated samples.
4. The greatest flexibility was obtained in the heat-treated samples due to the coaxial structure.
5. The hardness of the surface treatment sample increased compared to the raw sample.

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