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Post processing of SLM Ti-6Al-4V Alloy in accordance with AMS 4928 standards

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Abstract:- This Research work was done to find out the impact of AMS 4928 standard heat treatment on Selective Laser Melted (SLM) Ti-6Al-4V Grade 23 alloy. Ti-6Al-4V Grade 23 is an Extra Low Interstitial version of Ti alloy with lower impurities and is α+β type alloy at room temperature. SLM is one type of method in Additive Manufacturing based on Powder bed system. Each powder layer of few microns is coated and a laser beam is scanned to melt the metal powder according to the specification of the part and subsequently moved downwards layer by layer. The test coupons were first heat treated according to the above mentioned standard. The tensile testing and the microstructural analysis were done to compare the results with that of mentioned in the AMS 4928.The yield stress andPercentage elongation in the test coupons achieved are better than the minimum requirement by AMS 4928 standard. Coarse lamellar grain structures were obtained with no continuous network of alpha at prior beta grain boundaries.

Keywords: α+β Ti alloy, Ti-6Al-4V, ELI, Beta Transus temperature, microstructures.

I. INTRODUCTION

Titanium has almost half the density of Steels but has got strength comparable to that of steels [1]. Its excellent corrosion resistance has allowed for use in chemical industries and even in some cases for jewelry as well [2]. Also, titanium is continuously being used in medical industry as prosthetic elements due to its excellent biocompatibility [3]. At Room temperature Titanium has a hexagonal close-packed (hcp) crystal structure, which is referred to as “alpha” phase. This structure transforms to a body-centered cubic (bcc) crystal structure, called “beta” phase, at 883°C [4] and this temperature is called as Beta-transus temperature. For Ti-6Al-4V alloy, the beta-transus temperature is 995°C [5]. Titanium alloys can be basically classified into three types; Alpha alloys, Alpha-Beta alloys and Beta alloys. Ti-6Al-4V is an Alpha-Beta alloy due to presence of both alpha stabilizers such as Aluminium and beta stabilizers such as Vanadium. In Ti Alloys, the heat treatment process plays very important role in obtaining the desired mechanical properties. In Heat Treatment also, there are two processes within; heating and cooling. In heating, the residence time and the Soaking temperature are the two factors and in cooling, the rate of cooling is the only major factor which can be used to obtain different properties. Particularly for Titanium, the rate of cooling becomes very important with the increasing temperature. As per Bey Vrancken et al. [6]; it was found that in Titanium alloys, the effect of cooling rate increases with the increase in the heat treatment temperature. With faster cooling rates from the beta phase field, very hard structures are obtained with less ductility due to retention of supersaturated beta phase. The strength and the ductile properties are achieved by aging process as metastable beta phase decomposes to give fine alpha precipitates, providing high strength and good ductile properties as well.

Additive Manufacturing has evolved in the recent years with the introduction of Direct Metal Printing technologies. Metal printing technology can be classified into two parts; *powder bed system* and *powder fed system*.Both these technologies are significant in their own terms. In former, each layer of power is coated on the whole build platform and then the laser or electron beam is scanned over the layer to melt the metal powders according to the design. In the latter, there is no coating of powder layer on the whole build platform. Instead of that, only the portions where the beam needs to scan, the powder is coated in synchronization.

AMS is the Aerospace Material Specification which is one of the three standards given by Society of Automotive Engineers [7] widely followed by Aerospace Industries. Titanium alloys as we all know are widely used in aerospace industries and the industries have started to shift focus towards additive manufacturing to produce complex parts as there is no limitation on the complexity of the design in 3D Printing. The level of strength obtained by these 3D Printed parts is now days said to be comparable with that of conventionally produced ones. A lot of work is still going on to improve the properties of the parts produced by additive manufacturing.

II. MATERIALS AND METHODOLOGY

The base material used for the analysis is Extra Low Interstitial Ti-6Al-4V Grade 23. The powder has been ordered from by LPW Technologies, a major supplier of metal powders to the industries. The powder has been made from plasma atomization process.The powder size found by Sieve Tests by LPW Technologies varies from 15 to45 microns. The major percentage of the size is between 15 to 30 microns. Almost only 10% of the particles are between 30 to 45 microns. The actual composition of the powder is shown in table 1.

Element	Actual Weight (%)
N	0.02
C	0.01
H	0.0075
Fe	0.20
O	0. 13
Al	6.3
V	3.9
Residual-Ind	≤0.1
Residual-Tot	≤0.4
Ti	Balance

Table 1:Actual Composition of Ti-6Al-4V Grade 23 supplied by LPW Technologies. All testing undertaken at ISO 17025 & approved NADCAP labs. Sieve analysis conforms to ASTM B-214 standard.

The test coupons of Ti-6Al-4V Grade 23 were 3D Printed on EOSINT M 280 DMLS 3D Printer using SLM method. The dimensions of these test coupons are given in table 2. The Yb fibre laser is used having power of 400W. Each powder layer is 30 microns thick as specified by EOS for this material. One set of test coupons produced are shown in figure 1. The heat treatment processes specified in AMS 4928 standard are as:

- Solution Heat Treatment at temperature within the range 28 to 83°C below the beta transus temperature, hold at that temperature within ±14°C for a time commensurate with section thickness and heating equipment and procedure used and cool at a rate equivalent to air cool or faster.
- Heat to a temperature within the range 704 to 788°C, hold at the selected temperature within ±14°C for not less than 1 hour and cool as required.

Fig.1: Macroscopic Samples of Ti-6Al-4V test coupons prepared by SLM Method on EOSINT M 280 DMLS 3D Printer.

The actual heat treatments performed on them were as:

- Solution Treatment at 940°C ± 10°C for 45 minutes followed by rapid cooling.
- Heating at 748°C ± 10°C for 1 hour followed by rapid cooling.

In both processes, Rapid Cooling was done inside the furnace by Argon flow at 5 bar pressure.

After Heat treatment, tensile testing was performed on four of these test coupons. The specimen diameter printed is 4.5 mm and the gauge length is 23mm. These tests results were then compiled and their mean values are taken.

Microstructure Analysis was followed for heat treated samples. The Imaging was done using Nikon MODEL EPIPHOT 200 metallurgical microscope. Its resolution range is 50X- 1000X. Images of these two samples were taken at 100X, 200X, 500X and 1000X.

Specimens were prepared with the following steps:

- Two specimens were cut and grounded on Silicon carbide (SiC) paper.
- The samples were then polished by diamond paste by placing on a rotating buffer cloth for almost 5 minutes at an average speed of 800 rpm.
- They were then etched using Kroll's reagent. Ratio of the components used in the etchant was 50mL H_2O + 4mL HNO_3 + 2mL HF.

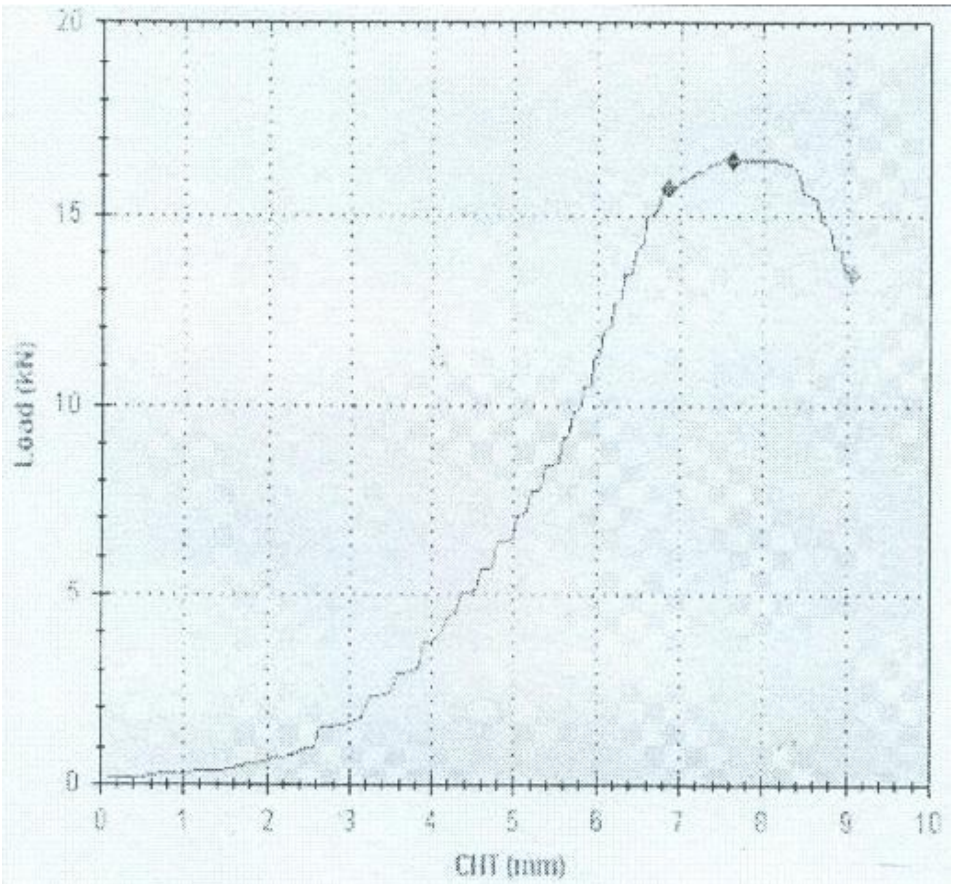

Fig.2: Load vs. Elongation curve for SLM Ti-6Al-4V test coupon after heat treatment According to AMS 4928 standard

III. RESULTS

The average Hardness of the test coupons found by Rockwell hardness testing method is 33 HRC.The hardness test results for our four samples of the test coupons are given in table 4. The Load vs. Elongation curve obtained is shown in figure 2 and it shows the exactly at which point the yield and fracture occurred.

Coarse Lamellar types of structures were obtained with the presence of elongated primary alpha and transformed beta in the microstructure analysis of the test specimens after the heat treatments, shown in figure 3. There was no continuous presence of alpha phase which is a positive result for us as the tensile properties obtained due to continuous alpha phase are not up to the standard as studied by Robert P. [5].The primary alpha were obtained in the transformed beta matrix with no continuous network of alpha to prior beta grain boundaries as can be seen in figure 3. The Ductile properties obtained in our work were better than that of minimum specifications by AMS 4928 standard. The yield stress obtained also exceeded the minimum required value quite comfortably.

In sample number 1, the ASTM gain size number, G is 6.58 and 6.72 in sample number 2. Here, we have followed ASTM E112 [9] standard test methods for obtaining average grain size. Intercept Procedure mentioned in the ASTM E112 was used to calculate the grain size number. The intercept method involves an actual count of the number of grains intercepted by a test line or the number of grain boundary intersections with a test line, per unit length of test line. The approximate results obtained are provided in table 5 after comparing value of G with the charts provided by ASTM.

INPUT DATA	
Specimen Dia	**: 4.5mm – 4.6 mm**
Gauge Length	**: 23 mm**
Specimen CS Area	**: 16.1- 16.7 mm^2**
Pre load value	**: 0 KN**

Table 2:Dimensions of the specimens used for tensile testing.
Note that these specimens are half the size of the built test coupons

OUTPUT DATA	
Load at yield	**: 15.74 KN**
Elongation at yield	**: 6.29 mm**
Yield stress	**: 956.15 N/mm^2**
Load at peak	**: 16.67 KN**
Elongation at peak	**: 7.60 mm**
Tensile strength	**: 1013.82 N/mm^2**
Load at break	**: 13.40 KN**
Elongation at break	**: 8.61 mm**
% Reduction Area	**: 41.36**
% Elongation	**: 11.71**

Table 3:The output of the tensile tests. Note that four of the test coupons were tested and only the average value of the results has been listed in the table.

Sample No.	HRC
1	32
2	33
3	33
4	33

Table 4: Hardness test results for the four samples of test coupons which were also put in tensile tests.

Result	Value
No. of grains per unit area	701.45 /mm^2
Average grain area	0.00143 mm^2
Average Diameter	0.0378 mm
Mean intercept	0.0336 mm
Average no. of intercepts per unit length of test line	29.73 /mm

Table 5: The approximate grain dimensions obtained by using Intercept Method defined in the ASTM E112 standard.

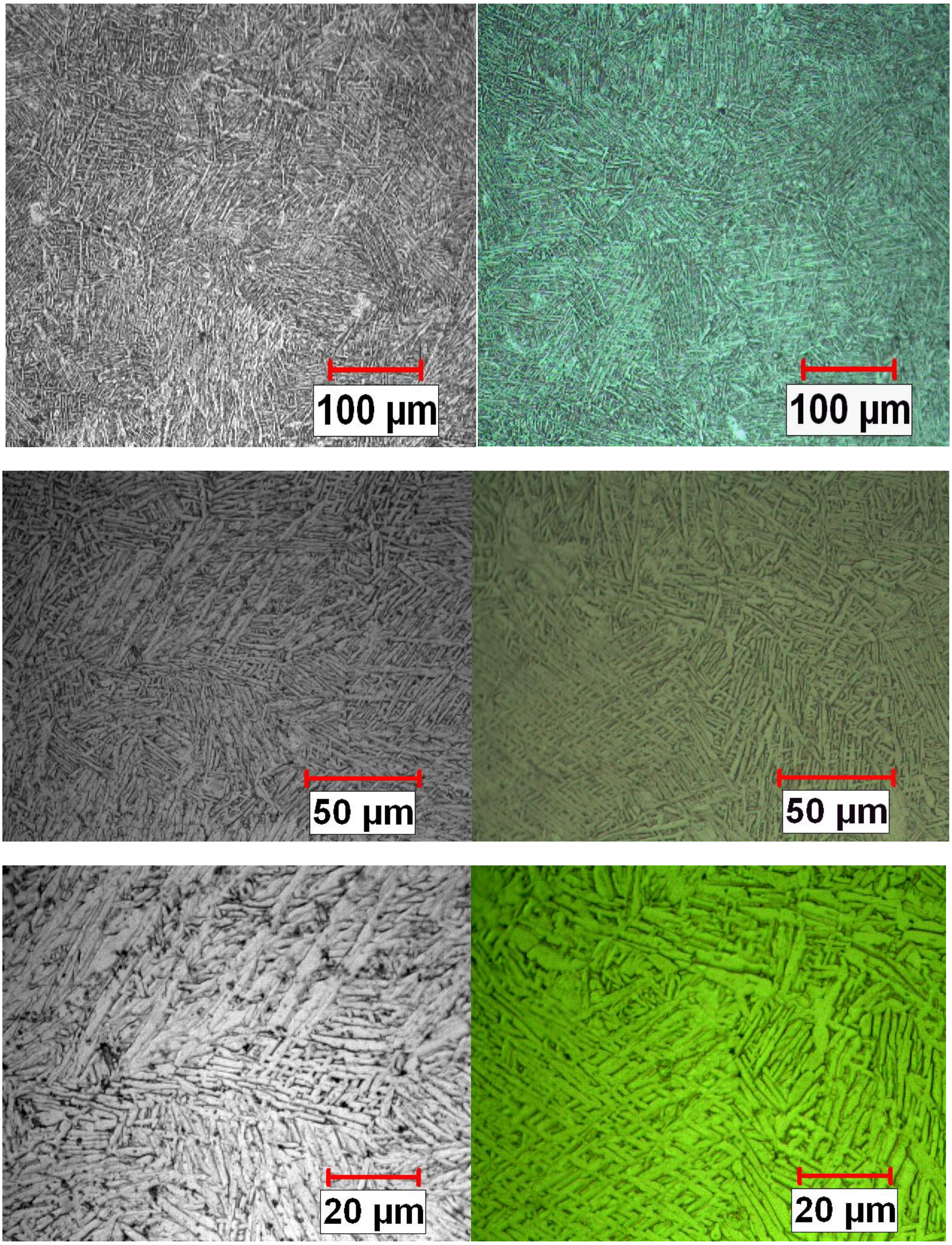

Fig.3: Microstructure images of Ti-6Al-4V at 200X, 500X and 1000X respectively after heat treatment with AMS 4928 guidelines. Left side B/W for specimen 1 and right side colored for specimen 2.

IV. DISCUSSION

The results obtained after the tensile testing and the microstructural analysis confirm that there was not much difference in the values shown by these test coupons during their tensile testing. It, thereby, also confirms that there is not much significant role of the position of the part inside the build chamber of the 3D Printer. But, this cannot be claimed for every printer as it can also depend upon printer to printer. More work is needed in this field so that 100% claim could be made.The AMS standards are set for the conventionally produced parts like from forgings and drawings. Exceeding the minimum required value of this standard for titanium is an excellent result. The results obtained by Bay Vrancken et al. [6] showed better yield stress value but at the cost of the fracture strain. The tensile results obtained in this work gave a proper balance of both yield stress and the percentage elongation. The minimum required values of the AMS 4928 standard after the application of the above mentioned heat treatment process were as: Tensile strength- 931 MPa; % elongation-10%; %reduction in area-25%. The tensile results shown in the table 3 has already been defined to exceed the minimum required value. With the advancement in the SLM method, equivalent or may be even better mechanical properties can be achieved in the materials as compared to the forged ones. The reason behind this is that there is perfect bonding between the melted layers of the powdered material.This is a significant result because five years back, we couldn't even have imagined that the strength of the 3D Printed parts will be comparable to the conventionally manufactured or forged ones. However, there are a lot of thermal stresses developed in the as built part. So, to remove all these thermal stresses, we have to go for stress relieving and heat treatment. The major limitation still is that we can only make parts one by one while repeating all those steps again and again for every part to be built. In Ref. [4], the results obtained are also more or less similar to this work within same treatment levels but a little bit of improvement in the ductile properties have been achieved in this work.

The hardness achieved after heat treatment was 33 HRC which when compared to a normal forged Ti-6Al-4V which has around 34 HRC for same set of Heat Treatment applied is acceptable within the specified limits [3].

The microstructure of the test specimens was in accordance with the heat treatment applied. The microstructure images are obtained only after both sets of heat treatment were done. As found in some previous studies, [4], the microstructure of untreated as built Ti-6Al-4V parts is fully acicular α' martensitic. The influence of temperature, cooling rates and the residence time were analyzed. It is assumed that the microstructure after first treatment is in accordance with the results obtained by Bey Vrancken et al. [6] in which presence of globularized structures was reported. Our results also verified the presence of globularization at some portions of the structure. To have a larger percentage of globularization, the residence time has to be increased more. In a previous research, it was suggested that to achieve 50% globularization of the a phase at 955°C, a residence time of approximately 8 h is required atleast [10]. It can be inferred that these globular structures were present after the first treatment that started to disintegrate after the annealing process.The complete disintegration could not have occurred due to less soaking time given in the annealing process.The effect of residence time becomes even more important when the temperature reaches close to or above betatransus temperature. Previous study [11] showed that the elongated alpha grains were obtained with prolonged residence times. The bigger alpha colony size is directly responsible for obtaining better mechanical properties. The rate of cooling likewise is more effective with the increasing temperatures as also been defined earlier. The lower cooling rates allows more time for grain growth but higher cooling rates at or above beta transus temperature allows precipitation of in form of transformed beta matrix to alpha precipitates.The level of tensile strength obtained can be verified with the presence of large colonies of primary alpha as well as the transformed beta. The percentage elongation before fracture achieved is within the acceptable limits but the presence of more equi-axed grains would have ensured an even better ductility.

V. CONCLUSIONS

Coarse lamellar alpha structures have been obtained in the transformed beta matrix with no continuous network of alpha at prior beta grain boundaries. All the tensile properties are according or we can say even better than to the minimum specified in AMS 4928. There is always scope for improvement. There is little less uniformity in the orientations of the alpha grains in the beta phase matrix. Either the residence time given was not sufficient or the cooling rate needs to be analyzed. If the cooling rate after the Annealing process would have been kept somewhat lower, the probability of obtaining even better ductile property gets higher as there'll be a little more uniformity in the grains. The slow cooling gives more time for grain growth. But still, the tensile properties obtained are more than acceptable and are verified by the microstructures.

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