Testing for homogeneity and orthotropy of Ti6Al4V (ELI) parts built by Direct Metal Laser Sintering

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Abstract

Design of medical prostheses is typically based on the mechanical properties of the materials used. However, apart from non-loadbearing implants, medical prostheses in use are exposed to various forms of dynamic loading. Therefore, to eventually deliver qualified medical prostheses produced through additive manufacturing (AM), it is necessary to develop a data bank on both their static and dynamic properties. This is done here with reference to the fatigue properties of Direct Metal Laser Sintering (DMLS) of Ti6Al4V (ELI) parts, produced at the Centre for Rapid Prototyping and Manufacturing (CRPM) of the Central University of Technology. In recognition of the effect of microstructure on the mechanical properties of materials, post process heat treatment of Ti6Al4V (ELI) will also be done to produce prostheses with high fatigue strength.

Rectangular bars of Ti6Al4V (ELI) of dimensions 60 x 11 x 11 mm were built in the X, Y and Z directions in the DMLS machine. Ultrasonic testing was undertaken on each specimen to determine its heterogeneity and orthotropy.

This paper presents the results obtained from the ultrasonic tests of as-built DMLS Ti6Al4V (ELI), without post production stress relief or heat treatment.

Keywords: DMLS of Ti6Al4V (ELI), heterogeneity, orthotropy, ultrasonic testing

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1. INTRODUCTION:

Direct metal laser sintering (DMLS) is an additive manufacturing (AM) process through which objects are made from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies [1]. Its ability to produce geometrically complex designs with precision and with little or no waste makes it an ideal manufacturing method for medical prosthesis. This is particularly important due to the fact that the human anatomy consists of complex parts with geometries that differ from patient to patient. In order to facilitate the growth of bone onto a prosthesis, a porous structure is needed, which adds to the complexity of design, but which can be manufactured with ease using DMLS. AM is therefore a flexible and economical means of manufacturing prosthesis [2].

This paper discusses some physical and mechanical properties of DMLS of medical implants that are manufactured at CRPM based on Ti6Al4V (ELI).Ti6Al4V (ELI) is biocompatible, bio-adhesive, bio-functional, corrosion resistant and it has a good strength to weight ratio. This makes the material good for use in manufacturing medical prosthesis. The titanium alloy is an alpha/beta ($\alpha + \beta$) alloy with 6% aluminium and 4% vanadium α phase stabilizer and β phase stabiliser, respectively [3]. The β stabilizer makes the retention of β crystals in Ti6Al4V (ELI) possible at room temperature, thus facilitating alteration of the mechanical properties of the alloy through various heat treatment regimes. Upon heat treating, the alloy can undergo an equilibrium or a non-equilibrium phase transformation to an $\alpha + \beta$, or $\alpha' + \beta$ or full martensite (α') phase at room temperature, depending on the heating temperature and cooling rates [4]. The equilibrium phase transformation is that of $\alpha + \beta$ which exists in either lamellar, equiaxed or duplex microstructural morphology. The lamellar microstructure consists of colonies of α lamellae with retained β boundaries, and have good fatigue propagation resistance[5]. An equiaxed microstructure comprises of equiaxed primary alpha (α_p) with β boundaries, and has high fatigue nucleation resistance but low propagation resistance [4]. The duplex microstructure consist of a percentage of α_p grains and α lamellar grains, and therefore has both fatigue nucleation and propagation resistance. This makes the duplex microstructure the preferable microstructure for fatigue resistant products.

DMLS of Ti6Al4V (ELI) results in an α' microstructure distinguishable by fine needle shaped morphology.This is a super saturated α phase due to the non-equilibrium phase transformation [6]. The martensitic transformation arises from the rapid solidification of the molten layer due to the high temperature gradient of the DMLS process [7]. During the heating (melting) and cooling cycles of the DMLS, the layer undergoes non-uniform plastic deformation resulting in residual stresses within DMLS parts. The substantial shrinkage of the molten layer during cooling and solidification is constrained by the cooler underlying previously processed layers leaving the DMLS parts in tension with maximum stresses at the surface [8]. This is a drawback of the DMLS process, because the tensile residual stresses lower the fatigue strength as they promote crack nucleation and propagation. However, the amendability of Ti6Al4V (ELI) to heat treatment that relieves residual stress provides a means of resolving this drawback [7][9].

Apart from residual stress, DMLS parts are known to have pores, the level, shape and distribution of which can lower some mechanical properties. They arise from semi or non-melted powder particles during the DMLS process. The main effect of the pores is that they act as stress concentration sites thereby reducing the fatigue strength of DMLS parts. Achieving full theoretical densification is vital as the decrease in density from the theoretical value indicates increased porosity [10]. Previous work on the density (using Archimedes' principle) of DMLS parts produced by CRPM found the Ti6Al4V (ELI) DMLS density parts to be 99% of that specified on ASTM F136 (4.43g/cm^3) [11][12].

In manufacturing, homogeneous and isotropic properties are desired in order to achieve uniform mechanical properties. However, it was found that DMLS parts exhibit anisotropic characteristics, with the fatigue properties in the X-direction being higher than in the other two mutual mutually orthogonal directions [13]. The Young's modulus of DMLS Inconel 625 was found to be 1.5 times greater in the x- and y- direction than that in the z-direction [13]. The X-, Y- and Z-directions here refer to the recoater scan geometry; horizontally across the front, into and vertically across the front. Ultrasonic non-destructive testing may be used to test for the presence or absence of heterogeneity and anisotropy. ASTM E 494-48 gives details of the manner in which the results of longitudinal velocity (C_L) and shear velocity (C_S) ultrasonic tests can be used to determine the Poisson's ratio, shear modulus, elastic modulus and bulk modulus of a material, thus:

$$C = \frac{2d}{\Delta t} \tag{1.0}$$

$$(poison\ ratio)\ v = \frac{1-2\left(C_T^2/C_L^2\right)}{2-2\left(C_T^2/C_L^2\right)} \tag{2.0}$$

$$(shear\ modulus)\ G = \rho C_T^2 \tag{3.0}$$

$$(Elastic\ modulus)\ E = 2G(1+v) \tag{4.0}$$

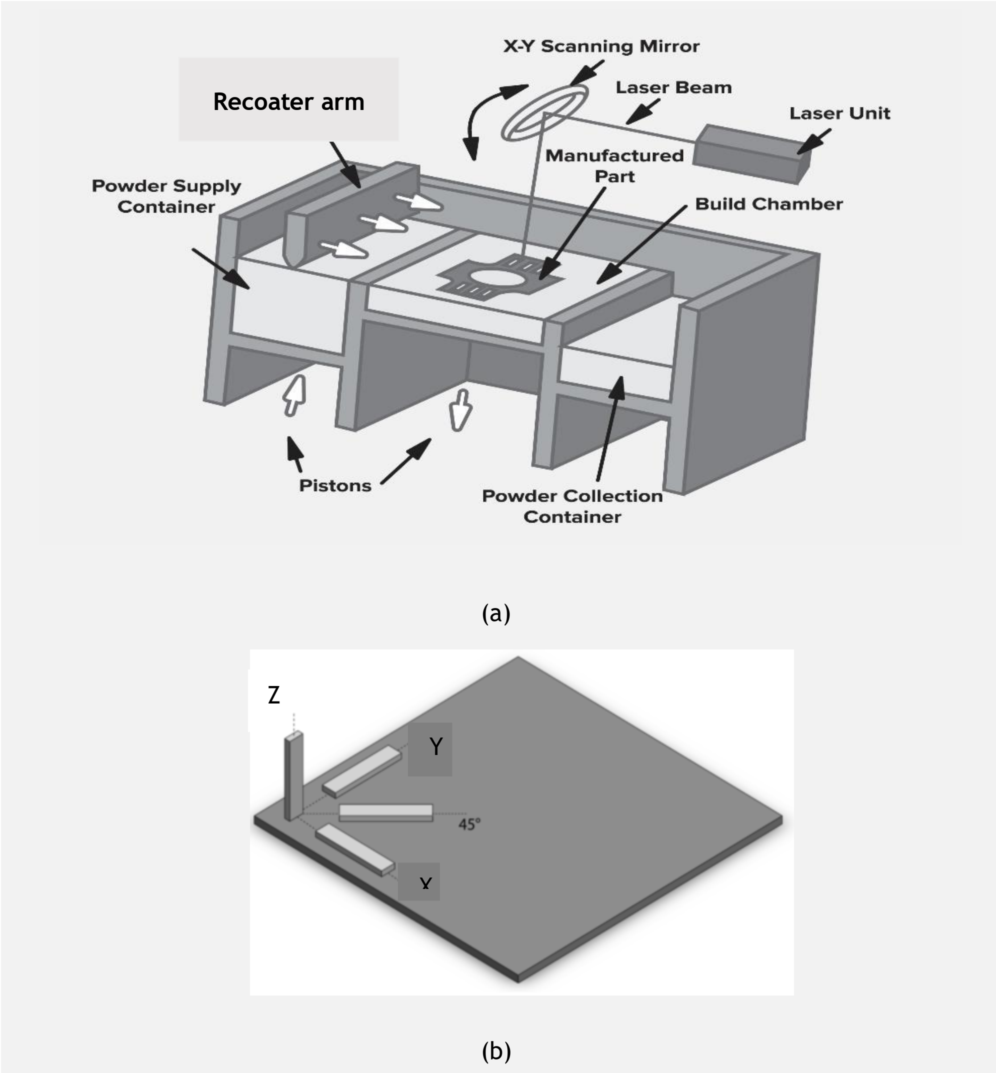
$$(Bulk\ modulus)\ K = \frac{E}{3(1+2v)} \tag{5.0}$$

The ultrasonic longitudinal velocity of wrought Ti6Al4V is 6172 m/s [14].

2. EXPERIMENTAL PROCEDURE

2.1 Specimen preparation

Thirty rectangular bars of W=11mm, H=11mm and L=60mm were manufactured by an EOSINT M 280 DMLS machine set to the standard parameters of the supplier as shown on a typical DMLS schematic in Figure 1(a). Figure 1(b) shows the DMLS build platform with four rectangular bars representing four build directions with reference to the recoater arm movement. There is an X, Y and Z build directions as referred to in this paper, the 45 build direction is excluded here. For the X build direction, the length of the bar is built parallel to the recoater arm movement. For the Y build direction the bar length is built perpendicular to the recoater movement in the X-Y plane and for the Z direction the length is built vertically to the X-Y plane as seen in Figure 1(b).A total of 30 of the rectangular bars were build, samples 1- 10 in the X direction, samples 11-20 in the Y direction and samples 21-30 in the Z direction.



(a) **(b)**

Figure 1 : Illustration of a typical DMLS process and build directions

The as built specimens were thereafter used for the determination of density and heterogeneity as well as isotropy.

2.2 Density

The density was determined from measured values of specimen weight and specimen dimensions in accordance with the equation:

$$\rho = \frac{m}{W \times B \times L} \ \left({}^{g}/_{cm^3}\right)$$

The width and the breadth of the bars were measured using a micrometre screw gauge (accuracy ±0.005mm) and the lengths were measured using a vernier calliper (accuracy ±0.05mm). It is appreciated that this approach will lead to an underestimate of the value of density. However this will apply equally on all specimens in all directions. The bars were weighed on a WTC 200 Precision Balance (accuracy 0.0005g).

2.3 Ultrasonic Testing

Due to constraints of time and malfunction of the ultrasonic shear probe signal injection, only the longitudinal back echo ultrasonic test was carried out. Without values of the shear velocity, it was not possible to calculate the values of v, G, E, and K. However, calculated values of the longitudinal velocity (C_L) are adequate in investigating the existence or lack of heterogeneity and anisotropy of the material.

A Panametric NDT machine with a 40 MHz 0° Wave Probe with a Crystal Size of Ø10mm was used in this work. A coupling gel was used between the wave probe and specimen to ensure there was no air between the two because sound energy at the ultrasonic frequencies is not effectively transmitted through air. A total of 8 frequency transmissions were done at equally spaced distances along the length of each specimen; four through the width, four through the depth and one more through the length.

3. RESULTS AND DISCUSTIONS

3.1 Density

It was found that all of the bars had constant geometry throughout except for specimens 11 to 15 which had a step along the width. Therefore specimens 11 to 15 were left out in the calculations of density.

The average densities, their percentages of the theoretical density and their respective standard deviations for the X-, Y- and Y- DMLS built directions are given in Table 1.

Table 1: Calculated values of densities of the specimens tested for various build directions

Number of specimen	10	5	10
Build direction	X	Y	Z
Average Density (g/cm^3)	4.26 ± 0.05	4.38 ± 0.05	4.28 ± 0.05
% of the theoretical density	96	99	97
Standard deviation	0.05	0.22	0.01

The theoretical density of wrought Ti6Al4V (ELI) for surgical implants is 4.43 g/cm^3 [12]. The overall average density percentage achieved in this experiment is 97%. The calculated values compared well with the theoretical value with percentage variations of the mean values from the theoretical density of -3.84%, -1.13% and 3.39% in the X-, Y- and Z- direction, respectively. The percentage variations are all below the highest measurement percentage error of 5% arising from use of the vernier calliper and are therefore considered acceptable.

3.2 Ultrasonic Longitudinal Velocity of Sound

The calculated values of ultrasonic longitudinal velocities for specimens built in the x-, y- and z-directions are presented in the ensuing three tables, and discussed after each table of results.

3.2.1 Orthotropy in the X-, Y- and Z-directions built Ti6Al4Vspecimens

X-direction built DMLS Ti6Al4V specimens

Table 2 shows the average ultrasonic longitudinal velocities (C_L) recorded and standard deviations of the X-direction built specimens in the x-, y- and z-directions.

Table2: x-, y- and z-axis average values of C_L for X-direction built specimens

Echo transmission direction	x-axis	y-axis	z-axis
Thickness of transmission (mm)	60	11	12
Average (m/s)	6084	6154	6230
Standard deviation	71.25	59.2	197.56

In the X-direction built specimens, the average ultrasonic velocities are seen in the foregoing table to increase in the order, x-, y- and z-axis. This would imply orthotropy. However the curve plots shown in Figure 2 indicate very small differences in the values measured in the three directions. It is reasonable therefore, to assume isotropy, within the limits of experimental error. The determined values of velocity are a percentage of the theoretical velocity of 98.6%, 99.7% and 100.9% in these three directions, respectively. The standard deviations of the mean velocities in the three mutually orthogonal directions are small compared to the mean values being 1.17%, 0.96% and 3.17% of the means in the x-, y- and z-axis directions, respectively. This implies a small scatter of data from the mean, from one specimen to the other in the same direction. It is reasonable to conclude therefore that the X-direction built DMLS Ti6Al4V specimens are homogeneous.

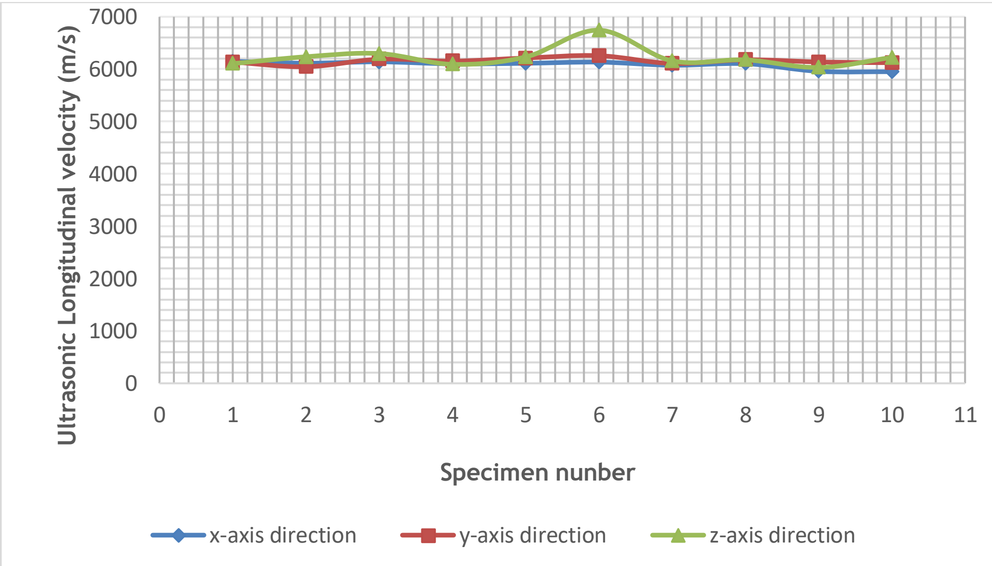


Figure 2: Average values of C_L in the x-, y- and z-axis directions for the X-direction built specimens.

The curves in Figure 2 are clustered together with an outlier for specimen 6 in the z-axis direction. It is evident from the clustering of the data that the directional variation of the ultrasonic velocity is small. The X-direction built DMLS Ti6Al4V specimens may therefore be taken to be isotropic.

Y-direction built DMLS Ti6Al4V specimens

In the Y build directions, the average velocities increase in the order of y, x and z axis with percentages of 98.6%, 98.9% and 99.4%. Table 3 show the measured average ultrasonic longitudinal velocities and standard deviations of the Y-direction built specimens in the x-, y- and z-axis directions.

Table 3: x-, y- and z-direction average values of C_L for Y-direction built specimens

Echo transmission direction	x-axis	y-axis	z-axis
Thickness of transmission (mm)	11	60	12
Average (m/s)	6105	6087	6135
Standard deviation	164.36	58.44	117.64

The determined values of velocity are a percentage of the theoretical velocity of 98.6%, 99.7% and 100.9% in these three directions, respectively. The standard deviations of the mean velocities in the three mutually orthogonal directions are small compared to the mean values being 2.69%, 0.96% and 1.92% of the means in the x-, y- and z-axis directions, respectively. This implies a small scatter of data from the mean, from one specimen to the other in the same direction. As was observed for the X-direction built specimens, it is reasonable to conclude therefore that the Y-direction built DMLS Ti6Al4V specimens are homogeneous. In the Y- direction built specimens, the average ultrasonic velocities are seen in the foregoing table to increase in the order, x-, y- and z-axis. This would imply orthotropy. However the curve plots shown in Figure 3 indicate very small differences in the values measured in the three directions. It is reasonable therefore, to assume isotropy, within the limits of experimental error.

Figure 3 shows the ultrasonic longitudinal velocities of the 10 Y-direction built specimen in the x-, y-, and z-axis directions.

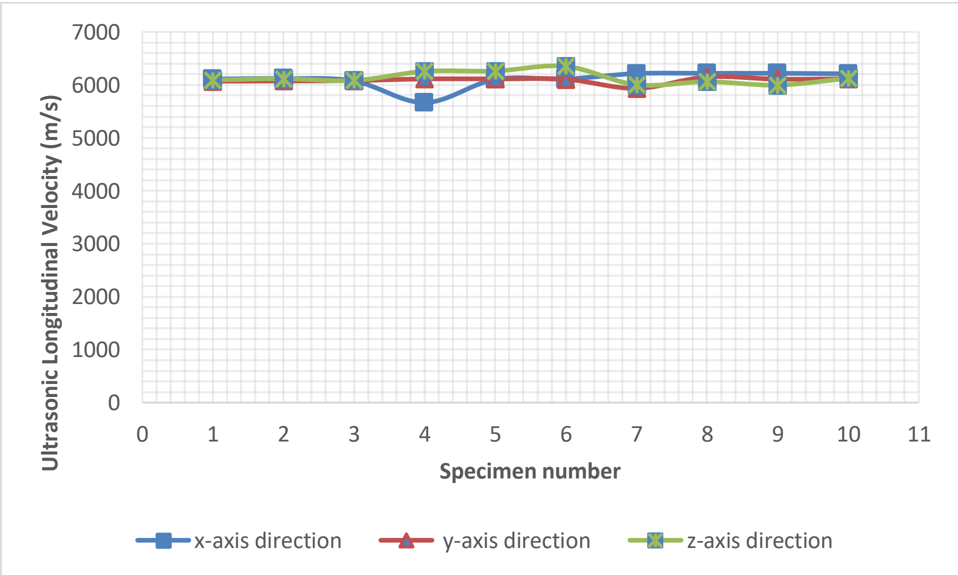


Figure 3: Average values of C_L in the x-, y- and z-axis directions for the Y-direction built specimens.

The curves in Figure 3 are clustered together with an outlier for specimen 4 in the x-axis direction. The Y-direction built DMLS Ti6Al4V specimens may therefore be taken to be isotropic.

Z-direction built DMLS Ti6Al4V specimens

Table 4 show the measured average ultrasonic longitudinal velocities and standard deviations of the Z-direction built specimens in the x-, y- and z-axis directions, respectively.

Table 4: x-, y- and z-direction average values of C_L for Z-direction built specimens

Echo transmission direction	x-axis	y-axis	z-axis
Thickness of transmission (mm)	11 mm	11 mm	60 mm
Average (m/s)	6166	6199	6058
Standard deviation	100.33	154.13	192.23

In the Z-direction built specimens, the average ultrasonic velocities also portray an orthotropic characteristic seen in the foregoing table to increase in the order, z-, x- and y-axis. The percentages of the theoretical velocity are 98.2%, 99.9% and 100.4% in these three directions, respectively. The differences in the ultrasonic velocities are small and the results can be regarded as isotropic within the limits of experimental errors. The standard deviations of the mean velocities in the three mutually orthogonal directions are small compared to the mean values being 1.63%, 2.49% and 3.17% of the means in the x-, y- and z-axis directions, respectively. This implies a small scatter of data from the mean, from one specimen to the other in the same direction. As was observed for the two mutual orthogonal direction built specimens. It is thus reasonable to conclude that the Z-direction built DMLS Ti6Al4V specimens are homogeneous, as evident from the curves of velocity shown in Figure 4.

Figure 4 shows the ultrasonic longitudinal velocities of the 10 Z-direction built specimen in the x-, y-, and z-axis directions.

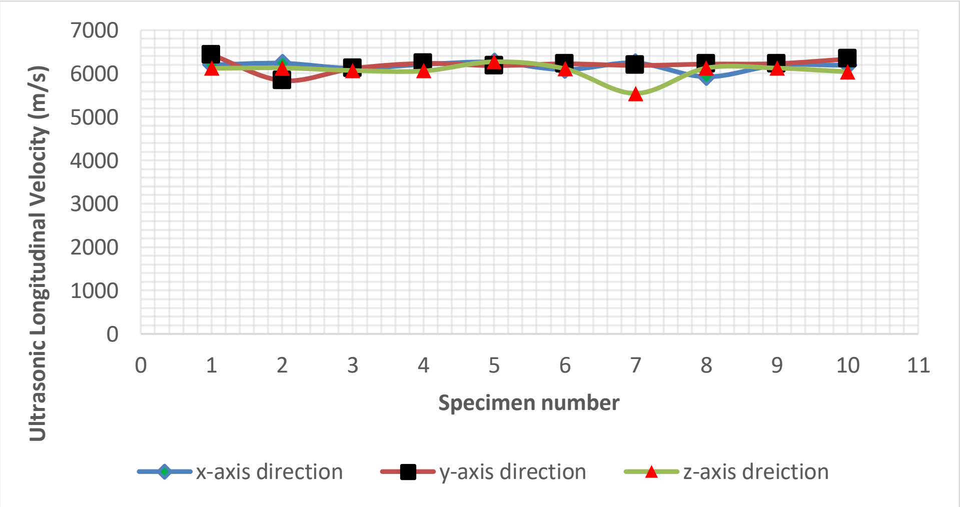


Figure 4: Average values of C_L in the x-, y- and z-axis directions for the Z-direction built specimens.

As was the case for the X- and Y-direction built specimens, the results of longitudinal velocity in the three axes directions; x-, y- and z-directions are seen in Figure 4 to be clustered, with an exception of the values for specimens 2 and 7 for the y-axis and z-axis directions, respectively. This denotes isotropy of the DMLS Ti6Al4V material.

Comparing the mean values for the three cases discussed so far; the X-, Y- and Z-direction built specimens show that the ultrasonic longitudinal velocities increase from the x-axis direction through to the z-axis direction for the X-direction built specimens, from the y-axis direction through to the z- axis direction for the Y-direction as built specimens, and from the z-axis direction through to the x-axis direction for the Z-direction built specimens. The fact that the lowest velocities in each case are all in the built direction of each respective set of specimens, implies higher densities of the DMLS Ti6Al4V material in the built direction. There is however, no apparent order in the relative magnitudes of the ultrasonic longitudinal velocities with reference to the remaining two mutually orthogonal directions in each set of specimens, despite the built strategy being the same in all build directions.

3.2.2 Homogeneity in the X-, Y- and Z-direction built DMLS Ti6Al4V specimens

The ultrasonic through thickness test was used to test the presence or lack of homogeneity only along the lengths of the test bars as this provided opportunity to obtain a number of readings along one surface. For the X-direction built specimens, the ultrasonic signal was injected in both the x- and y-axis directions separately at different positions along the length of the bars. Similarly for the Y-and Z-direction built specimens, the ultrasonic signal was injected in both the z – and x-axis directions and the x- and y-axis directions, respectively.

X-direction built DMLS Ti6Al4V specimens

Table 5 shows the average ultrasonic longitudinal velocities for ten specimens, measured at intervals of 15 mm along the length of each specimen. The average velocities given in the table are for transmission of an ultrasonic wave in the y- and z-axis directions.

Table 5: Average values of longitudinal velocity (C_L) in the y- and z-axis directions of the X-direction built specimens

Position	1	2	3	4	Average	Standard Deviation
Number of specimens	10	10	10	10		
y-axis direction ultrasonic longitudinal velocity C_L (m/s)	619.62	6162.52	6112.97	6145.6	6153.93	34.042
Standard deviation	156.37	79.08	112.04	90.74		
z-axis direction ultrasonic longitudinal velocity C_L (m/s)	6375.14	6196.15	6143.95	6182.95	6224.55	102.81
Standard deviation	596.81	225.5	161.95	96.42		

The standard deviations of the y-axis direction and z-axis direction transmission of an ultrasonic signal taken along the length, X-direction built are from the last column of Table 5 are 0.55% and 1.65% of the calculated mean value, respectively. These are very small deviations as is evident by inspection of the curves shown in Figure 5.

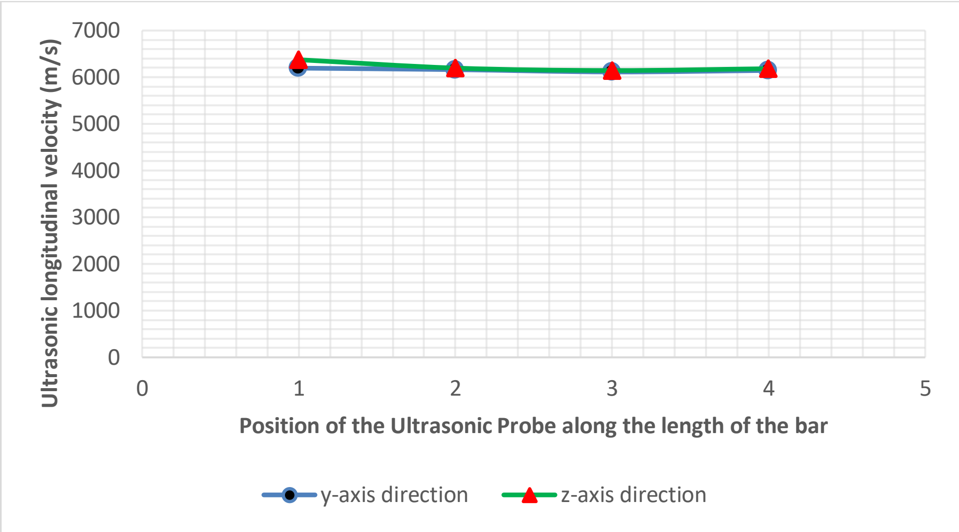


Figure 5 Ultrasonic longitudinal average velocities in the y- and z-axis directions the X-direction built bars

It can be concluded from the data in Table 5 and from the two curves in Figure 5 that the DMLS T06Al4V material is homogeneous in the X-direction of build.

Y-direction built DMLS Ti6Al4V specimens

Table 6 shows the average ultrasonic longitudinal velocities for ten specimens, measured at intervals of 15 mm along the length of each specimen. The average velocities given in the table are for transmission of an ultrasonic wave in the z- and x-axis directions.

Table 6: Average values of longitudinal velocity (C_L) in the x- and z-axis directions of the Y-direction built specimens

Position	1	2	3	4	Mean	Standard deviation
Number of specimen	10	10	10	10		
x-axis direction ultrasonic longitudinal velocity C_L (m/s)	6085.65	6069.75	6147.21	6118.21	6105.21	34.51
Standard deviation	206.40	239.97	135.85	137.33		
z-axis direction ultrasonic longitudinal velocity C_L (m/s)	6140.67	6069.23	6124.14	6205.07	6134.78	55.93
Standard deviation	269.60	115.15	199.13	115.47		

The standard deviations of the x-axis direction and z-axis direction transmission of an ultrasonic signal taken along the length, Y-direction built are from the last column of Table 6 are 0.57% and 0.91% of the calculated mean value, respectively. These are very small deviations as is evident by inspection of the curves shown in Figure 6.

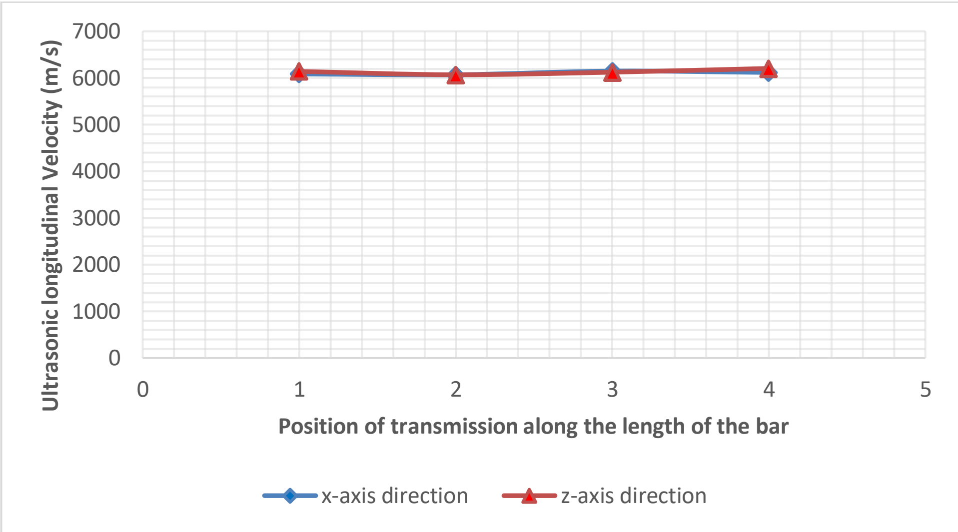


Figure 6: Ultrasonic longitudinal average velocities in the x- and z-axis directions the Y-direction built bars

As was the case for the X-built specimens, the data in Table 6 and the two curves in Figure 6 leads to the conclusion that the DMLS T06Al4V material is homogeneous in the Y-direction of build.

Z-direction built DMLS Ti6Al4V specimens

Table 7 shows the ultrasonic longitudinal average velocities for ten specimens, measured at intervals of 15 mm along the length of each specimen. The average velocities given in the table are for transmission of an ultrasonic wave in the x- and y-axis directions.

Table 7: Average values of longitudinal velocity (C_L) in the x- and y-axis directions of the Z-direction built specimens

Position	1	2	3	4	Mean	Standard deviation
Number of specimen	10	10	10	10		
x-axis direction ultrasonic longitudinal velocity C_L (m/s)	6221.11	6154.48	6213.89	6076.17	6166.41	67.16
Standard deviation	43.74	144.08	23.63	342.48		
y-axis direction ultrasonic longitudinal velocity C_L (m/s)	6103.23	6389.11	6191.75	6111.23	6198.83	133.00
Standard deviation	415.82	673.99	107.71	290.35		

The standard deviations of the x-axis direction and y-axis direction transmission of an ultrasonic signal taken along the length, Z-direction built are 1.09% and 2.15% of the calculated mean values, respectively. These are very small deviations as is evident by inspection of the curves shown in Figure 6.

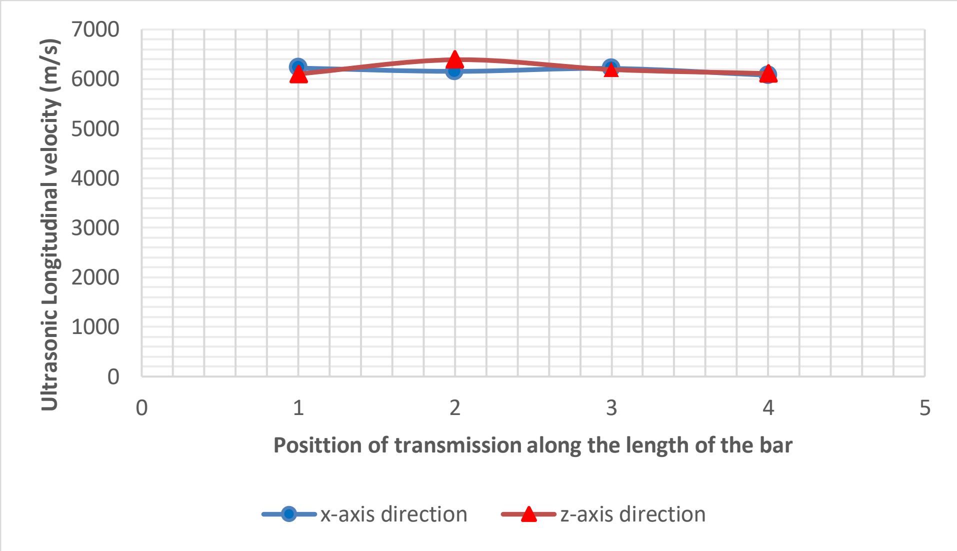


Figure 7: Ultrasonic longitudinal average velocities $_{in}$ the x- and y-axis directions the Z-direction built bars

As in the previous two cases the data in Table 7 and the two curves in Figure 7 leads to the conclusion that the DMLS T06Al4V material is homogeneous in the Z-direction of build.

4. CONCLUSION

Very high densification was achieved with the DMLS Ti6Al4V specimens prepared attaining an overall percentage of 97% of the theoretical value. More accurate density measurements can be obtained using X-Ray Tomography.

All three sets of specimens showed the lowest values of ultrasonic longitudinal velocity in the respective directions of built. It is not possible however form this observation to make an inference about stiffness, Poisson's ratio and density in each of these built directions as the ultrasonic longitudinal velocity is not a simple function of either one of them but is rather a function of all three together.

The data obtained however does show the DMLS Ti6Al4V specimens to be both homogeneous and isotropic. More data is necessary however coupled with the introduction of acceptable confidence limits within which the statements of homogeneity and isotropy can be tested.

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