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DEVELOPMENT OF A TECHNIQUE FOR TESTING OF TENSILE PROPERTIES
WITH MINIATURE SIZE SPECIMENS FOR METAL ADDITIVE
MANUFACTURING

by

SUJITKUMAR DONGARE

A THESIS

Presented to the Faculty of the Graduate School of the

MISSOURI UNIVERSITY OF SCIENCE AND TECHNOLOGY

In Partial Fulfillment of the Requirements for the Degree

MASTER OF SCIENCE IN MANUFACTURING ENGINEERING

2012

Approved by

Dr. Frank Liou, Advisor

Dr. Joseph W. Newkirk, co-advisor

Dr. Elizabeth A. Cudney

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PUBLICATION THESIS OPTION

This thesis is composed of one paper which was reformatted in the style used by the university.

The paper presented in pages 5-37 titled “DEVELOPMENT OF A TECHNIQUE FOR TESTING OF TENSILE PROPERTIES WITH MINIATURE SIZE SPECIMENS FOR ADDITIVE MANUFACTURING PROCESS” is intended for submission to SCRIPTA MATERIALIA.

ABSTRACT

The study of mechanical properties of metals provides a basis to decide on the capability of a particular metal for a task and also to make predictions about its life. The concepts of stress, strain and strength of materials are employed in practically every engineering discipline. Mechanical properties such as stiffness, yield strength, tensile strength, ductility, toughness, impact resistance, creep resistance, fatigue resistance and others, influence the design, fabrication and service life of equipment. Therefore, more than one property is considered for the material selection process for an application. For complete understanding of any material and its feasibility for a particular application, inter-related mechanical properties have to be measured. Unfortunately, these properties cannot be measured in any single test. However, the tensile test can be used to measure a number of the most commonly used mechanical properties. Extensive research has already been performed in this area. Standards have been developed and established regarding the size of test specimens, testing procedures and process parameters.

This thesis discusses the development of a testing procedure for non-standard tensile tests for evaluation of material properties. Miniature test specimens similar to the standard ASTM E8 were designed and used for testing. The tests were mainly conducted on the baseline material for aerospace industry i.e. Ti-6Al-4V.

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1. INTRODUCTION

1.1 OBJECTIVE

Research in the field of mechanical testing of metals has reached a level where specific standards have been developed that explains about the dimensions of the specimen and the testing procedure to be followed. Tensile testing being a destructive method of testing, it at the end leads to wastage of material. Thus, this research aims at developing a miniature size testing standards for tensile testing. This will not only save on material and time for testing considerably but will also help to test the effects of position and build rate in case of metal additive manufacturing process.

1.2 BACKGROUND

Conventionally, metal parts are produced by thermo-mechanical processes like casting, rolling, forging, machining and welding. These techniques require multiple steps along with heavy equipment, molds, tools and dies to produce the final part. These conventional operations often require the use of heavy equipment and molds, tools, and dies. The investment for these processes is better paid in case of large volume production. But when the part is unusual in shape or has fine internal features, the turnaround and cost will increase rapidly [2]. Additive layer manufacturing is a novel approach to the manufacturing of said components. The near net shape component is prepared by layer-by-layer material addition. It has many advantages such as short lead time and elimination or reduction of machining, thus lowering material cost over conventional manufacturing methods.

Additive layer manufacturing can be achieved through several different techniques including direct laser deposition, electron beam deposition or shaped metal deposition. The basic principal of building the part in layer-by-layer fashion remains the same. The building process is controlled according to a process plan that is generated in accordance with the computer aided design (CAD) model. In case of few materials such as Ti alloys, the process is carried out in protected atmosphere to protect it from the atmospheric conditions.

Laser Additive Manufacturing Process (LAMP) is also a type of additive manufacturing process wherein a fully dense metallic part is obtained through laser melting the metallic powder coaxially delivered and deposited on the base table. The movement of the base table and the laser beam is controlled by the planar controlling information, which is obtained from sliced part CAD model [2]. This method is used for different materials like nickel (Ni) alloys, steel and titanium (Ti) alloys. Of particular interest are Ti alloys, which are difficult to produce by conventional methods. This research concentrates mainly on Ti-6Al-4V; one of the most commonly used Ti alloys for commercial aerospace applications.

1.3 PROPOSED TECHNIQUE

Tensile properties of metallic materials are used as a measure of capability of the metal for a specific task. ASTM E8 is the commonly followed standard for tensile testing of metallic materials. As per the standard, the test specimen can either be cylindrical, or of flat cross-section. The gage length is the most significant difference between E8 and E8M test specimens. The gage lengths for most of the round specimens are required to be

at least four times the diameter. Minimum gage length is observed to be 10 mm whereas minimum length of the specimen is 100 mm.

The main intention of additive manufacturing is to create near net shape parts and reduce the scrap during machining. The purpose of this research was to test laser metal deposited parts for their strength and local variations in the strength. Study of these variations with standard full size specimens would need large size of deposits and the test sections would be fairly away from each other. Thus, this research places an emphasis on developing a technique for utilizing miniature tensile testing specimens in order to reduce consumption of material during the application of destructive testing method. This technique would be very helpful help in the examination of local variations with much better resolution as compared to standard full size specimens. Furthermore, this technique can also be used to compare the effect of different post processes, such as laser tracing or friction stir processing, on the Laser Metal Deposition (LMD) where the processed area for testing is small and concise. While designing the miniature specimen, standard architecture of the specimen was retained. Following the specimen, specific grips were also designed to conduct the tests at the universal test frames available at Missouri University of Science and Technology (Missouri S&T).

Thus, the experimental set-up was comprised of test specimens, pins, threaded grips and 10 ksi universal test frames. The newly developed technique was demonstrated using Ti-6Al-4V alloy specimens.

1.4 CONTRIBUTIONS

A technique for testing of tensile properties of metals with miniature sized test specimens having the architecture similar to ASTM E-8 standard. This technique will

save on material and time for testing. It will also facilitate the testing of positional variations and build rate dependency of tensile properties in case metal additive manufacturing process.

DEVELOPMENT OF A TECHNIQUE FOR TESTING OF TENSILE PROPERTIES WITH MINIATURE SIZE SPECIMENS FOR ADDITIVE MANUFACTURING PROCESS

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1. ABSTRACT

Research concerning the tensile testing of metallic material encompasses the methods for determination of yield strength, yield point elongation, tensile strength, elongation, and reduction of area. Testing of laser metal deposited parts and local variations in this manufacturing process would be difficult with standard full size specimens. It would need large size of deposits to generate respectable amount of data and the test sections would be fairly away from each other. The purpose of this paper is to develop a testing technique with miniature size specimens to examine local variations in laser metal deposits with better resolution. The paper covers detailed procedures for development of test specimens, actual testing set-up and the analysis of test results. From the study of cooling-rate dependency of strength, thin wall produced with slower build rate of laser deposition process was observed to be stronger. Positional variation in the strength value of laser deposited thin wall was also evident with this technique.

Keywords: *tensile testing, testing test procedure, miniature size, testing of metals*

2. INTRODUCTION

Research concerning methods for tension testing of metallic materials has been very extensive. ASTM E-8 comprises standards for different metals and it includes various test specimens' dimensions and various control methods for testing. Yongzhong Zhang *et al.* [2] has conducted such tests for characterization of laser direct deposited metallic parts. This work concentrated on laser deposited 663 copper alloy and 316L stainless steel samples. Bernd Baufeld *et al.* [3] also have contributed to this field by studying the tensile properties of Ti-6Al-4V components fabricated by shaped metal deposition. This work includes the testing of specimens for confirming the variation of tensile properties with respect to position, orientation, cooling rates and testing environment. Total length of the test specimen used was 10 mm. The standards and the previous work in the testing field have certain requirements for minimum dimensions of the test specimens. The gage length of 25 mm to 200 mm with overall length of 100 mm to 450 mm is set as standard for square cross section specimens.

Following the standard specimen dimensions is impossible in cases where available material for testing is insufficient. This happens in case of development of new materials or processes where the production of large specimens is either impossible or too expensive. For determining location-dependent properties, having a smaller specimen improves the spatial resolution of the investigation.

R. Kapoor *et al.* used 13.5mm long tensile specimens with 5.7 mm long gage length for study of the mechanical behavior of ultrafine grained AA5052 processed through different techniques [4]. Dog-bone-shaped mini-tensile specimens were also used

by X. L. Shi *et al.* [5] and Z. Y. Ma *et al.* [6]. X. L. Shi synthesized ultrafine-grained Al-4Y-4Ni and Al-4Y-4Ni-0.9Fe (at%) alloys and studied the mechanical behavior by performing uniaxial tension tests. Z. Y. Ma studied the effect of multiple-pass friction stir processing on microstructure and tensile properties of a cast aluminum-silicon alloy. X. L. Shi and Z. Y. Ma used the tensile specimens with gage length of 1.3mm and the width was 1mm. These tensile specimens were then polished to final thickness of ~0.5 mm.

The testing procedures discussed above were tested for maximum stress less than 500MPa. This paper aims at developing a testing procedure that would specifically be used for stronger aerospace materials, such as Ti-6Al-4V with expected UTS of approximately 900 MPa. It covers the information regarding specimen preparation, testing, and analysis of test result.

Fryer 5X - 45 machining center was used for the fabrication of test specimens and the actual tensile tests were run using a universal tester rated for 10Kpi load settings. The fractographs were obtained using Hitachi S - 4700 scanning electron microscope and the grain structure was studied via a Zeiss MC 63 optical microscope with a Canon Rebel XSI DSLR camera.

3. SPECIMEN DESIGN

ASTM-E8 provides standard test methods for tension testing of metallic materials. It provides guidelines for different types of specimens like plate-type specimens, sheet-type specimens, specimens for sheet, strip, flat wire and plate or specimens for wire, rod, and bar.

Considering these guidelines and the previous work in the field, sheet-type specimen with square cross section was designed for miniature tensile test. These specimens could either have wedge shaped shoulder ends for gripping or with pin ends. Considering the approximate size of the specimen, the grip section and the gripping mechanism in the universal test frames, a pin-loaded tension test design was selected. The ASTM E-8 standard allows for square cross section, pin-loaded specimens, but does not include a procedure for the size range dictated by these design constraints.

To design the dimensions of miniature specimen, various simulations were run. Different values of gage length, width of the specimen and the curve radius were tested. Stresses and deformation in the gage section and the grip section of the specimen were analyzed. The final dimensions were decided to confirm the elongation in the gage section leaving the grip section least affected. The designed miniature specimen thus had a gage length of 3.3 mm and width of 1 mm. The overall length of the specimen was 17.74 mm with the thickness of 1 mm. The gage area was nominally 1 mm by 1mm. The test set-up was designed for 2000N load ratings.

Grips were designed for this rating and hardened steel pins of 3 mm diameter were selected for the tests. Two 3 mm diameter holes were thus provided in the specimen

for mounting the pins. The miniature specimens follow the same architecture as ASTM E-8 standard pin loaded, square cross section test specimen, as shown in Figure 3.1 [1]. except the dimensions of the specimen. Figure 3.2 shows the schematic representation of miniature size specimen. To consider the test to be valid, tensile failure has to be in the designed gage section. Figure 3.3 shows the expected post-failure condition of tensile test specimen that would confirm the validity of the test. The allowable dimensions of standard pin loaded and miniature specimens are compared in Table 3.1.

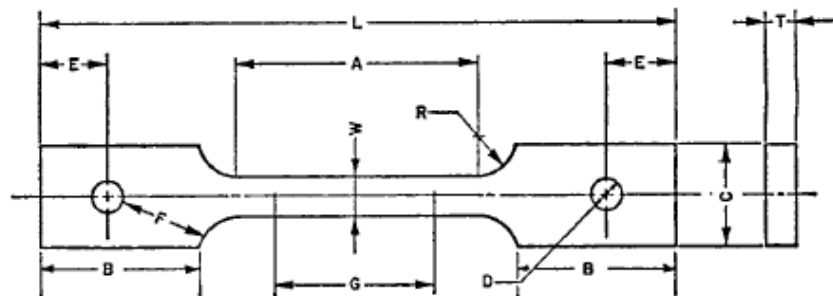


Figure 3.1: ASTM E8 – Sheet type pin-loaded tensile test specimen with 50mm gage length and minimum 200 mm of overall length

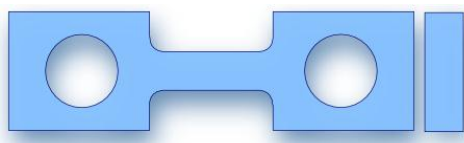


Figure 3.2: Schematic representation of designed miniature pin-loaded tensile test specimen with 3.3 mm gage length and 17.7 mm of overall length

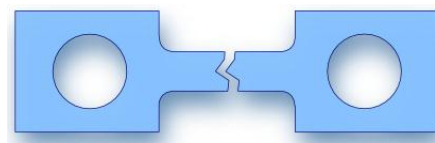


Figure 3.3: Expected post failure condition of designed miniature tensile specimen showing the failure in the gage section

Table 3.1: Comparison of dimensions of ASTM-E8 standard pin loaded specimen and designed miniature test specimen

Description	Standard pin loaded specimen dimension, mm [in.]	Miniature tensile test specimen dimension, mm
G – Gage length	50.0 ± 0.1 [2.000 ± 0.005]	3.3
W – Width	12.5 ± 0.2 [0.500 ± 0.010]	min 1
T – Thickness, max	16 [0.625]	min 1
R – Radius of fillet, min	13 [0.5]	1.25
L – Overall length	min 200 [8]	17.739
A – Length of reduced section	min 57 [2.25]	
B – Length of grip section	min 50 [2]	5.92
C – Width of grip section	approximate 50 [2]	approximate 6.05
D – Diameter of hole for pin	min 13 [0.5]	3
E – Edge distance from pin	approximate 40 [1.5]	approximate 3.02
F – Distance from hole to fillet	min 13 [0.5]	3.4

Specific grips for the miniature specimens were also designed and manufactured. The grip design consists of a 1.2 mm wide slot and a 3 mm diameter through hole for the loading pin. The test specimen placed in the slot is held together with the loading pins. Grips were machined out of 4150 steel alloy, and then heat treated to get the required hardness of approximately 42 Rockwell C. The grips were threaded for easy attachment in the universal tester. Figure 3.4 shows an exploded view of the test set-up.

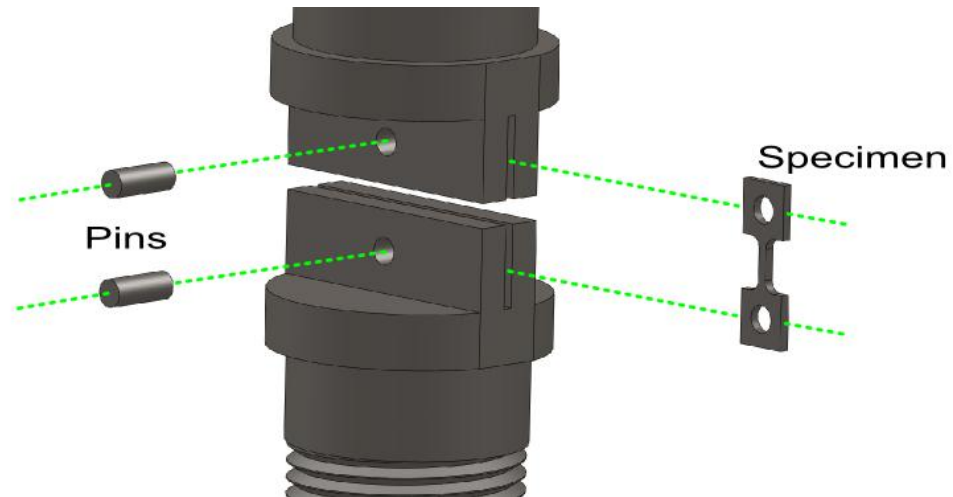


Figure 3.4: Exploded view of tensile test set up with newly designed miniature specimen, slotted mounting grips and the loading pins

The test set-up was designed for 2000 N force. Ti-6Al-4V specimens were expected to generate the strength of 850 – 900 MPa. For testing stronger material with this technique, testing set-up will have to be redesigned. Larger size pins would be required depending upon the expected strength value. Consequently, mounting grips and grip section of the test specimen will also need modifications to accommodate the newly confirmed loading pins.

4. EXPERIMENTAL PROCEDURE

The experimental setup was comprised of a universal testing machine, tensile test grips, loading pins and the designed miniature test specimen. Tests were conducted as per the crosshead speed control method defined by ASTM E-8 standard. The rate of straining was set and maintained at of 0.015 ± 0.003 mm/mm/min [in./in./min] of the original reduced section. The tests were thus conducted with constant cross head speed of 0.000835 mm/s.

Specimens from wrought Ti-6Al-4V plate were first tested with this technique. Figure 4.1 shows the positioning of test specimens in each of the small plates. The specimens were cut from a large rolled plate of Ti-6Al-4V. It was thus expected to possess uniform properties. To test the validity of the technique, specimens generated from this plate were tested and consistent results were expected in these runs.

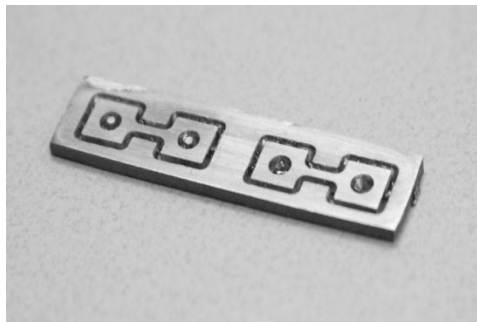


Figure 4.1: Positioning of miniature tensile test specimens in wrought Ti-6Al-4V plate

Having tested the specimens obtained from wrought Ti-6Al-4V plate, a few large sized laser deposits were tested using this technique. For this purpose, 45mm wide by 70

mm tall thin walls were deposited and specimens were then obtained as shown in the schematic representation in Figure 4.2. Next step of the experiment was to understand the ability of this technique to evaluate laser deposited structures with different cooling rates. All the deposition experiments were conducted at Laser Aided Manufacturing Processes lab (LAMP Lab) at Missouri University of Science and Technology. Ti-6Al-4V powder was supplied by Starmet Corp. and was sized at -60 +120 mesh.

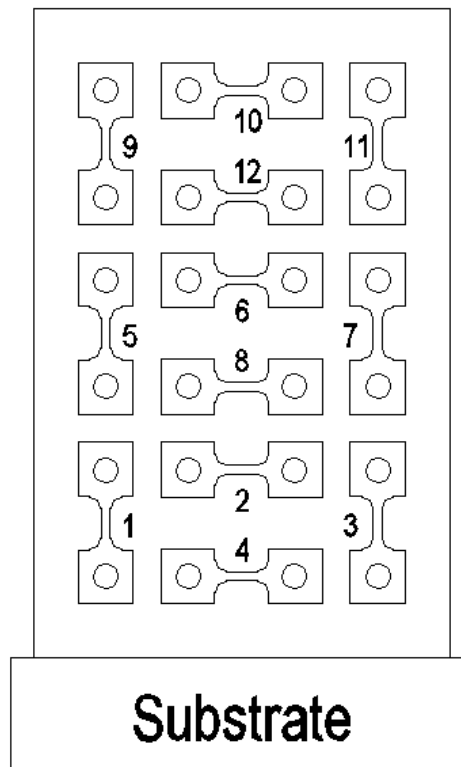


Figure 4.2: Schematic representation of 45mm wide by 70 mm tall deposits with positioning and orientation of the test specimens

Thin walls were deposited with the same amount of total energy and total material but with different build rates. For this purpose, the low build rate setting (375 MPPM) had laser power of 530 W with mass flow rate of 6 gm/min and travel speed of 375 mm/min. The high build rate setting (535 MPPM) used the laser power of 757 W with mass flow rate and table speed of 8 gm/min and 535 mm/min respectively. Both the settings had the same preheat conditions of two passes of 1000W and 169 layers of deposition with 45 mm travel to achieve 30 mm tall deposit. Figure 4.2 shows the wall deposited with 375MPPM settings and the build scheme.

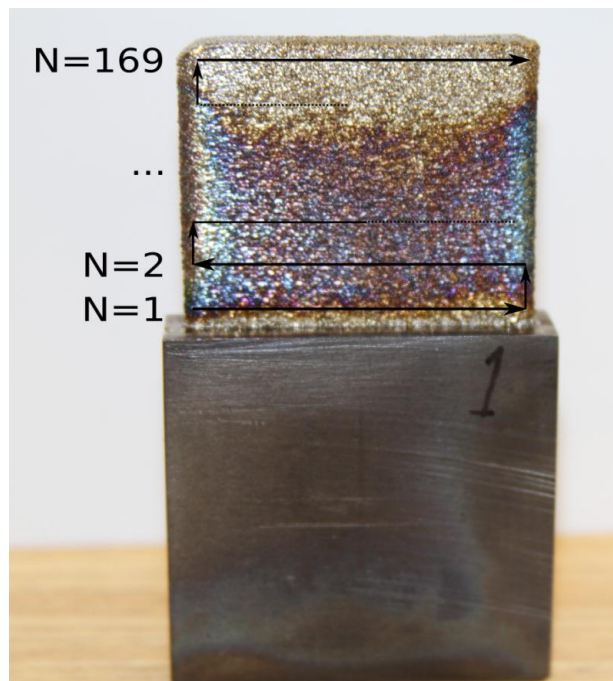


Figure 4.3: Ti-6Al-4V thin wall deposited with 375 MPPM setting of laser with zig-zag build scheme. Deposition started at the lower left corner. Five specimens were tested from each of such wall

A zig-zag pattern was followed for the deposition process. Two replicates were generated with each build rate setting and these are denoted with suffix A and B

respectively. The number of passes being odd, the location of start and the end of the deposition process were different. Five specimens positioned as shown in Figure 4.3 were tested from each of these walls. To analyze the positional variation in the deposit, specimen # 1 in all the deposits was taken from the region directly above the starting point of the deposition.

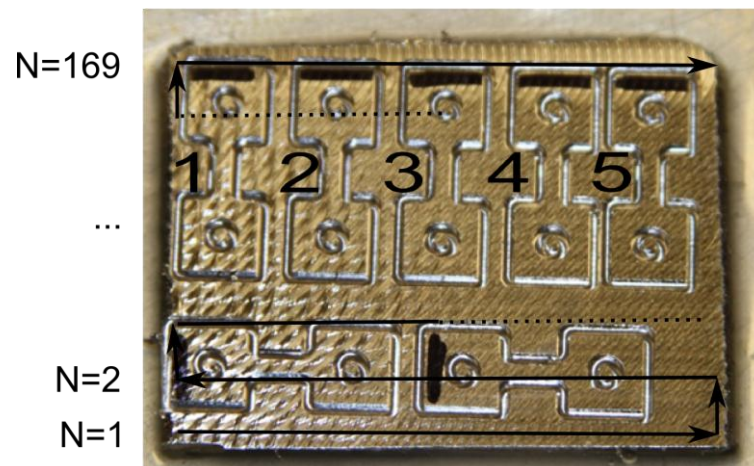


Figure 4.4: Positioning of miniature tensile test specimens in thin wall to test positional variation and build rate dependency. Specimen # 1 is located above the start point of deposition

5. RESULTS AND DISCUSSIONS

5.1 DATA PROCESSING FOR UTS AND YS VALUES

Force-displacement data was acquired from the test frames. Considering the original gage area, the data was plotted as stress—displacement and yield strength was further obtained. Figure 5.1 explains these calculations to obtain the yield strength value.

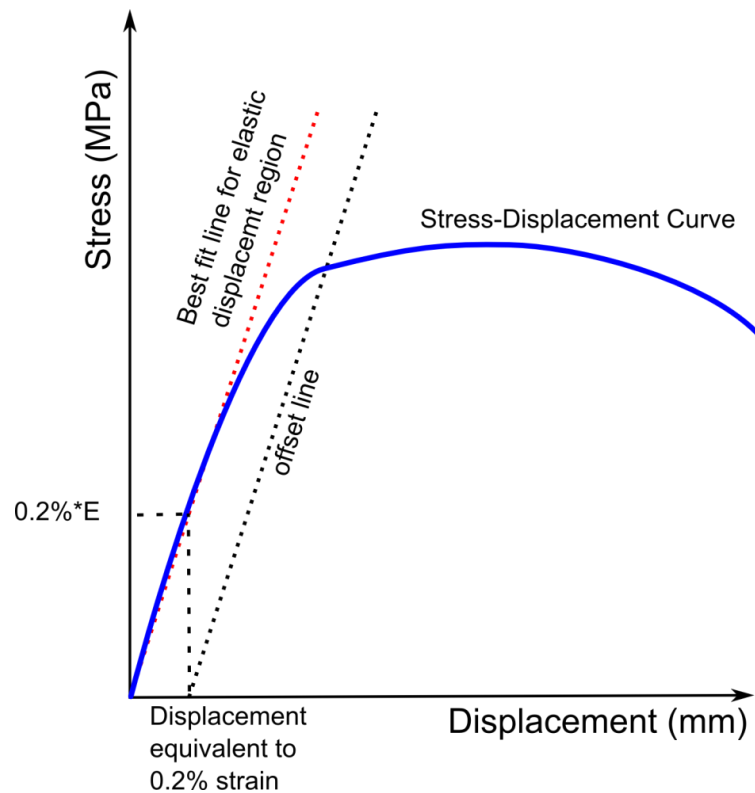


Figure 5.1: Schematic representation for yield strength calculation procedure using the Young's modulus value

To approximately calculate strain, the material was assumed to have constant Young's modulus value equal to 113 GPa which is the published value for annealed Ti-6Al-4V. To obtain the displacement equivalent to 0.2% strain, the 226 MPa ($113\text{GPa} * 0.002$) stress line was drawn to intersect with the stress—displacement curve. An offset line for yield strength measurement was then plotted from the x-intercept of the intersection point and parallel to the elastic portion of the curve. The point of intersection of the offset line with the actual curve Stress-Displacement curve thus provides the yield strength value. Figure 5.2 shows an example of the stress-displacement plot and respective values for ultimate tensile strength (UTS) and yield strength (YS).

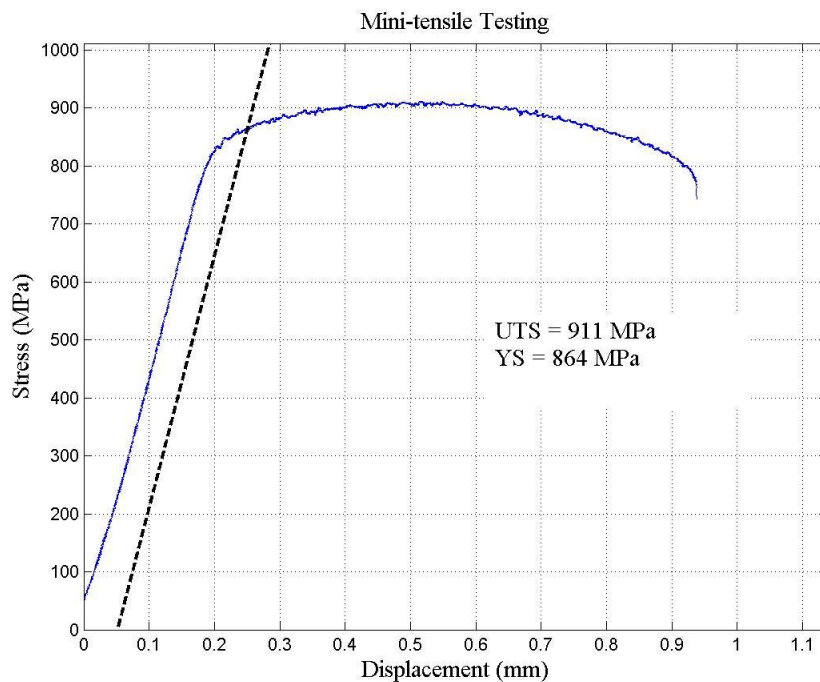


Figure 5.2: Example of Stress vs. Displacement plot with 0.2% offset and UTS and YS values

5.2 TESTING OF WROUGHT Ti-6Al-4V PLATE

Wrought Ti-6Al-4V plate was used to understand reproducibility of the technique. Eight specimens were tested from this plate and the results were studied for mean values and variations. The detailed test results are as mentioned in Table 5.1. One specimen showed values out of the order with others. The results of the test were more reliable if the lowest reading was excluded. Chauvenet's criterion [7] provided a means to test the data and to determine whether a particular measurement could be removed from the data set. It was noted that this procedure allows only one measurement to be removed.

Table 5.1: Test results for wrought Ti-6Al-4V specimens

Sr. No.	Specimen #	UTS (MPa)	YS (MPa)	Comments
1	Specimen # 01	892	852	
2	Specimen # 02	902	870	
3	Specimen # 03	894	857	
4	Specimen # 04	911	864	
5	Specimen # 05	914	867	
6	Specimen # 06	912	869	
7	Specimen # 07	842	805	Outlier
8	Specimen # 08	923	869	

To apply Chauvenet's criterion, the arithmetic mean and standard deviation were calculated for the data set. In addition, the ratio of deviation, d_i to the standard deviation, σ was also calculated for each measurement using eq. (1) and these results are shown in Table 5.2 for yield strength of specimens from the wrought Ti-6Al-4V plate.

$$\frac{d_i}{\sigma} = \frac{|x_i - \bar{x}|}{\sigma} \quad (1)$$

Table 5.2: Yield strength data for miniature tensile test specimens from wrought Ti-6Al-4V plates to check for Chauvenet's criterion for rejecting a measurement

Specimen #	$\frac{ x_i - \bar{x} }{\sigma}$
1	0.21
2	0.61
3	0.08
4	0.34
5	0.48
6	0.57
7	2.36
8	0.57

The arithmetic mean = 856 MPa and $s = 21.8$ MPa.

Chauvenet's criterion requires that the ratio obtained from eq (1) must exceed a specified value before the measurement can be excluded and this value depends upon the number of tests, N . (Table 5.3)

According to the Table 5.4, the maximum deviation for the group of 8 measurements is between 1.8 and 1.96. The largest deviation in the data in Table 3 is 2.36. Chauvenet's criterion is met in case of specimen # 7 and this measurement thus can be rejected. The data was again checked for Chauvenet's criterion and the results are as shown in Table 5.4.

Table 5.3: Chauvenet's criterion for rejecting a measurement

Number of measurements, N	Ratio of maximum deviation to standard deviation, d_{\max} / σ
3	1.38
4	1.54
5	1.65
6	1.73
7	1.80
10	1.96
15	2.13
25	2.33
50	2.57
100	2.81
300	3.14

Table 5.4: Yield strength data for miniature tensile test specimens after rejecting a measurement

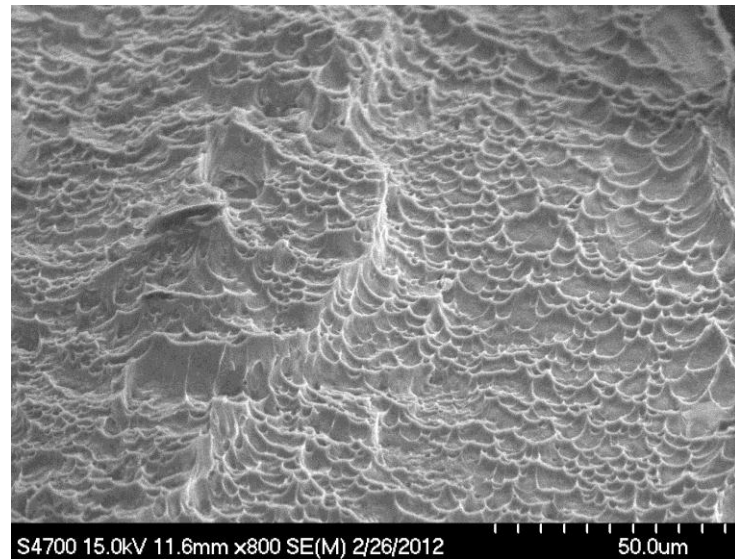
Specimen #	$\frac{ x_i - \bar{x} }{\sigma}$
1	1.73
2	0.87
3	1.01
4	0.00
5	0.43
6	0.72
8	0.72

The arithmetic mean = 864 MPa and $s = 6.9$ MPa.

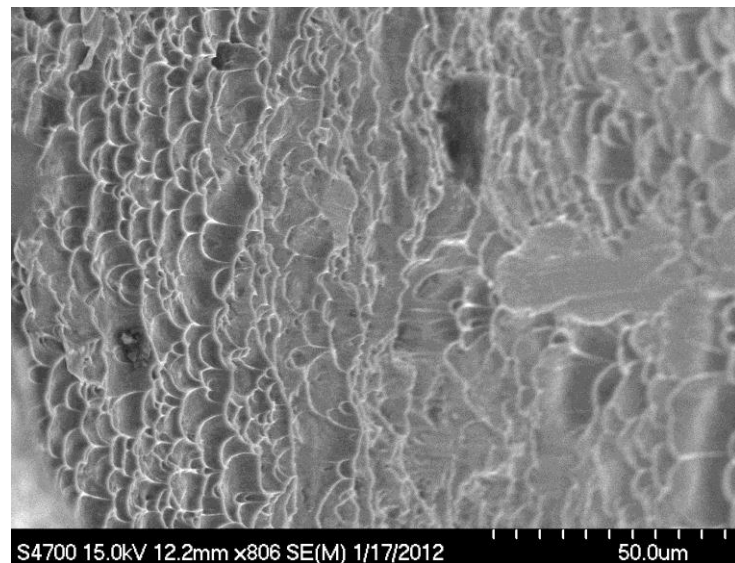
After rejecting a measurement in accordance with the Chauvenet's criterion, only seven measurements were considered for further analysis. For this data, UTS and YS was observed to be 906 ± 11 MPa, 864 ± 7 MPa respectively. These numbers are comparable

with published values [9-12] for annealed Ti-6Al-4V which is 850 – 900 MPa for UTS and 800 – 850 MPa for YS. Yield strength values obtained from miniature size specimens were also compared with the values of full size specimens obtained from different laser deposits of the same material. These specimens were horizontally oriented and were machined out of a thicker laser deposited built with different settings and conditions. Yield strengths of these specimens were 910 ± 2 MPa. Yield strength values of miniature specimens were observed to be lower but comparable with that of full size specimens. The difference in these values could be because of different build rate settings or specimen orientation. These readings help to confirm the reproducibility of the testing technique.

To investigate about the mode of fracture, fractured surfaces of test specimens were studied. Fractographs as shown in Figure 5.3 were obtained using a Hitachi S-4700 FESEM and were analyzed. The fractographs show dimple fracture appearance and failure was observed in the gage area which is typically a characteristic of ductile fracture. Strength numbers comparable with published values and fractographs that are evident of ductile failure confirm that this testing procedure can be considered to be reliable.



(a)



(b)

Figure 5.3: Fractographs of miniature tensile test specimen showing dimple fracture appearance that confirm ductile failure

5.3 TEST RESULTS FROM LARGE SIZE LASER DEPOSITED THIN WALLS

Having confirmed about the reproducibility of the testing technique and its results, three thin walls of the size 45mm wide by 70 mm tall were tested. 12 specimens were machined from each of these walls. Test results were as mentioned in Table 5.5.

Specimens numbered as 2, 3, 4 and 7 from wall # 1 showed values higher than others. They will be discussed in detail later in section 5.6. The rest of the specimens were observed to have mean values for UTS and YS as 912 MPa and 877 MPa respectively whereas the standard deviations for both of these were 58 MPa and 47 MPa. To investigate more about this higher standard deviation the laser deposition process was studied. This study showed that the laser deposition process was not followed as a continuous process. This had an effect on the cooling rate which ultimately affected the microstructure and the strength values. This thus confirmed the ability of the developed technique to investigate the quality of laser deposits.

Table 5.5: Test results for specimens from three large size walls

Specimen #	Wall # 1		Wall # 2		Wall # 3	
	UTS	YS	UTS	YS	UTS	YS
1	940	889	949	905	872	843
2	1053	1038	827	822	1000	967
3	1131	1079	908	882	882	854
4	1035	1029	893	851	904	882
5	993	944	929	905	835	814
6	834	830	900	864	937	895
7	1006	937	1073	996	957	917
8	891	891	858	851	941	894
9	909	865	921	884	969	943
10	866	828	912	856	979	919
11	949	900	820	787	961	930
12	801	719	914	883	887	875

5.4 POSITIONAL VARIATIONS IN LASER DEPOSITED THIN WALLS

The ability of the testing technique to provide information regarding the positional variation in the laser deposit was studied by testing specimens from specific positions in two replicated 375 MMPM walls. UTS and YS values are tabulated in Table 5.6 and Figure 5.4 shows the distribution of YS values. Specimen # 1 denotes the area above the starting point of deposition and specimen # 5 was taken from the region closer to the end of deposition. The distribution shows that the strength values decrease from start point to the end point of deposition. In 375 MMPM A wall, lowest YS value was approximately 96 % of the highest value. This number was 91 % in case of 375 MMPM B wall. Thus the technique confirmed to investigate positional variations.

Table 5.6: Ultimate Tensile Strength (UTS) and Yield Strength (YS) values obtained from miniature tensile test results of two replicates of 375 MMPM walls to show the positional variation

Specimen #	373 MMPM A		373 MMPM B	
	UTS (MPa)	YS (MPa)	UTS (MPa)	YS (MPa)
1	856	821	914	884
2	862	832	871	845
3	842	821	851	822
4	883	827	824	801
5	832	796	833	805

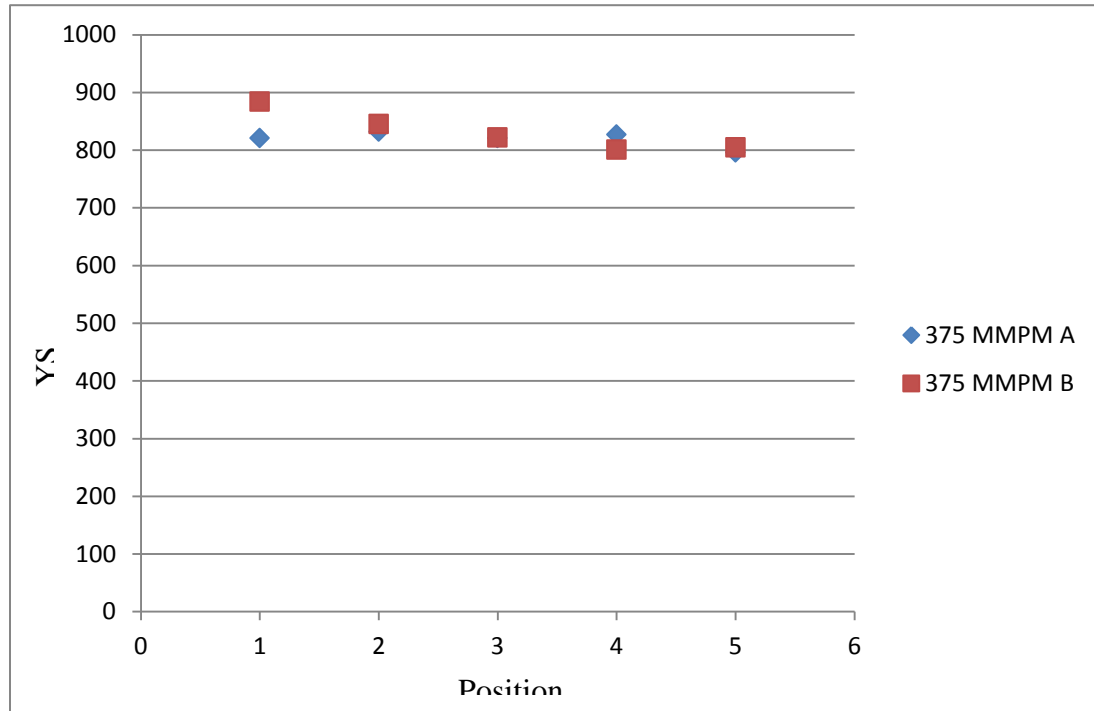


Figure 5.4: Distribution of miniature tensile test results from two replicates of 373 MPPM walls that shows decrease in strength values from specimen # 1 to specimen # 5. Position # 1 corresponds to the region above the start point of the build

5.5 BUILD RATE DEPENDENCY IN LASER DEPOSITED THIN WALLS

The thin walls generated with different cooling-rates also showed interesting results. Individual readings for yield strength (YS) of specimens from 375MPPM A and 535 MPPM A walls are summarized in Table 5.7. The strength values were observed to have a distribution as shown in Figure 5.5. From the distribution it is clear that the YS value is more in case of 375 MPPM settings. The comparison of mean values of YS also has confirmed that slower build rate has produced stronger thin wall deposit.

Table 5.7: Yield strength values in MPa obtained from miniature tensile test results 375MMPM A and 575 MMPM A walls

Specimen #	375 MMPM A	535 MMPM A
1	821	789
2	832	820
3	821	779
4	827	795
5	796	751

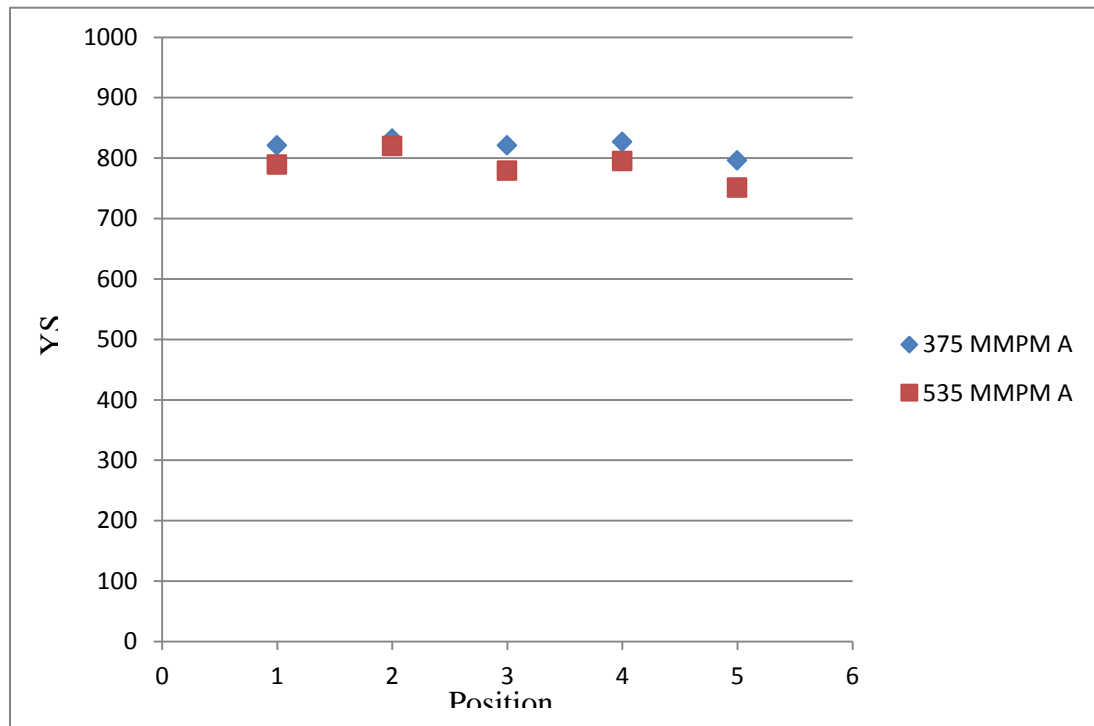


Figure 5.5: Distribution of miniature tensile test confirming the build rate dependency of strength values. Position # 1 corresponds to the region above the start point of the build

6. CONCLUSION

Tensile testing procedure with miniature sized specimens was developed and tested for Ti-6Al-4V produced with different processes and settings. The specimen design is a modified version of ASTM E-8 specifications.

- The technique of testing has proven to be reliable and reproducible using wrought Ti-6Al-4V plate.
- Newly developed test set-up is capable of 2000 N force and has been successfully tested up to 1500 N.
- Technique can also be used for stronger materials following the modifications discussed in section 3.
- Yield strength values of miniature size specimens are comparable with published values and also with previously tested full size specimens.
- The procedure is also capable of confirming positional variation in strength values in a laser deposited thin wall.
- Variation induced by virtue of different build rates during laser metal deposition can also be studied using this technique. Slower build rates were observed to generate a stronger deposit.
- Tensile testing of metallic material is thus possible with saving of substantial amount of time and material with this new technique.
- The technique whereas may produce anomalous results if large grain or colonies happen to be present in the gage region.

APPENDIX A
MACHINING PROCEDURE

MACHINING OF MINIATURE TENSILE SPECIMENS FROM WROUGHT Ti-6Al-4V PLATE

The control samples were cut from wrought Ti-6Al-4V plate. A substrate plate that was 2" X 2" X 0.5" was used for this purpose. A thin plate of around 2 mm thickness was first cut from this substrate via a band saw, abrasive saw, or a vertical milling machine. Using a milling machine ensures flatness of the surface. However, cutting thin plates out of titanium was difficult using a slicing tool because the metal tends to pull the tool towards itself which eventually resulted in tool breakage. Use of a band saw did not show any damage to the tool but it was a slow process and the surface finish was compromised. The pieces thus had to be thicker to ensure a flat surface after face milling.

Thin plates, obtained from procedures mentioned above, then had to be milled using a Fryer 45 - 5X high speed machining center. Since the plate was thin, gripping and clamping it while machining was difficult. Conventional methods of mechanical clamping could not be followed. Modified grips with a step or a groove to hold and align the plate in the vice also did not work.

It was thus decided to use non-conventional means that would withstand machining load to hold the plate. A commercially available adhesive called mighty-grip was tried. It is a heat-activated wax-based compound embedded in precision paper, coated on nylon mesh. It allows machining on five sides of a work piece without using a clamp. It served the purpose but the results were dependent upon the available area for gripping and the surface finish of material.

Hot glue was then tried and successful results were observed. Hot glue conforms to the shape of the specimen, thereby increasing the total holding surface area. It also holds the plate from the sides in addition to the bottom.

After holding the plate on aluminum block using hot glue, it was then mounted in the high speed machining center. This can be achieved using the T-slots in the machine table or by using a pneumatic fixture. The Ti-6Al-4V plate was then face milled to get rid of the irregularities generated during the initial slicing of the surface. This may require removal of a substantial amount of material from the surface and thus needed to be completed in several steps.

Specific machine parameters are thus set to ensure the surface finish of the machined surface. A Hy-Pro 0.5" diameter, carbide, flat end mill coated with TiAlN was used for this operation. All the machining was performed as up-milling with compressed cold air used as coolant. Table 1 shows the machining parameters for face milling operation and Figure 1 diagrams the face milling procedure.

Table 1: Machining parameters for face milling operation

Sr. No	Parameter	Value
1	Tool Diameter	0.5"
2	Spindle RPM	1500
3	Feed rate	24 IPM
4	Engagement	0.05"
5	Depth of cut	< 0.008"

After face-milling, actual tensile specimen profile was cut with a 1 mm diameter tool with parameters listed in Table 2. The profile was cut at a depth of 0.043". After this profile cut, the plate was taken off the aluminum plate and was then remounted but with inverted orientation. The only activity now remaining was to remove the extra material from the plate such that the specimen profile would remain sticking to the plate.

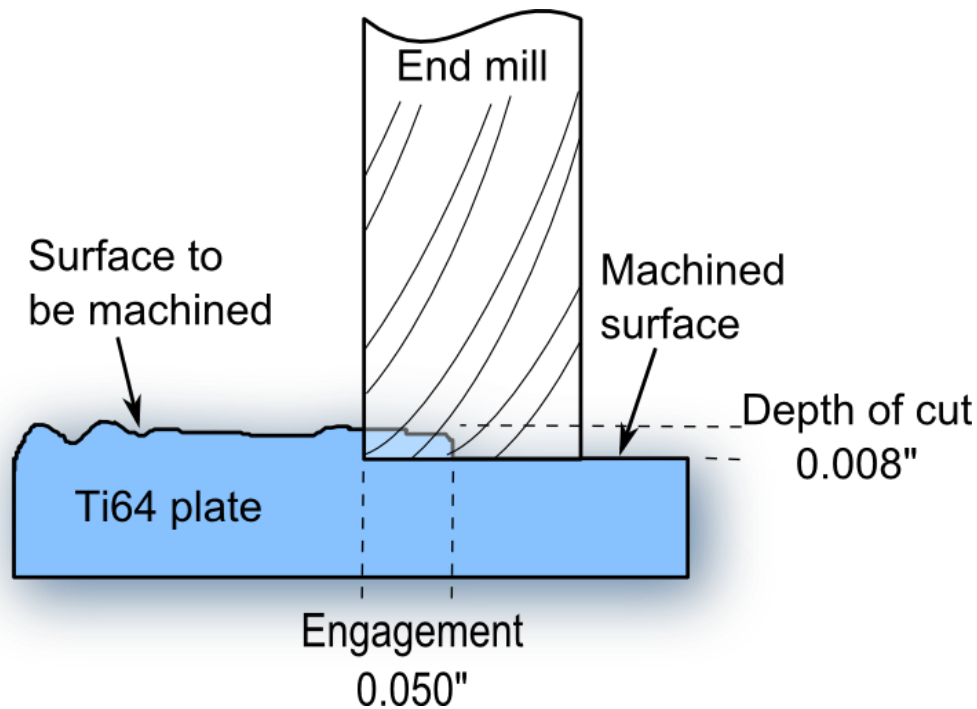


Figure 1: Details about face milling operation

The next step was to clean the specimen and get rid of the burrs and hot glue stuck to it. In order to accomplish this, the specimens were allowed to sit in an acetone bath for around 2 hrs. This aided greatly in removing the hot glue. The thickness of the generated specimen varied between 0.04" to 0.043" with gage widths of 1 mm to 1.1 mm. Thus, before testing, the exact thickness and width of the gage sections were noted. Detailed procedures for sample creation are included in the Table 3.

Table 2: Machining parameters for specimen profiling operation

Sr. No	Parameter	Value
1	Tool Diameter	0.04"
2	Spindle RPM	7500
3	Feed rate	6 IPM
4	Engagement	0.04"
5	Depth of cut	0.01"

Table 3: Sample preparation procedure from wrought T-6Al-4V plate

Sr. No.	Procedure	Hardware requirement	Time (min)
1	From the substrate plate cut thin plates of around 0.08 – 0.1 inch thickness	Saw blade / milling machine with parting tool	5
2	Mount the thin plate onto the Aluminum fixture plate with the help of hot glue	Hot plate, Hot glue, safety gear	45 – 60
3	Mill the top face of plate to get complete flat surface	Milling machine, 0.5” mill tool	12 – 15
4	Machining specimen profile to the depth of 0.043 inch	Milling machine, 0.04” mill tool	6
5	Flip the plate over and mount again on Aluminum fixture plate	Hot plate, Hot glue, safety gear	45 – 60
6	Mill the other face until the plate reached required thickness	Milling machine, 0.5” mill tool	15-Dec
7	Un-mount the specimens and polish	Polishing machine with supplies	30
	Total		155 – 190

APPENDIX B
TESTING PROCEDURE

EXPERIMENTAL SET UP FOR TENSILE TESTING WITH MINIATURE SIZE SPECIMENS

The experimental set up comprises a universal test frame, mounting grips, loading pins and the test specimen as shown in Figure 2. Tests are conducted as per crosshead speed control method of ASTM standards. In this method, the testing machine shall be set to a crosshead speed equal to 0.015 ± 0.003 mm/mm/min [in./in./min] of the original reduced section. The tests are thus conducted with constant cross head speed of 0.000835 mm/s.



Figure 2: Testing set-up for tensile test for miniature size specimens

Newly designed grips are mounted in universal test frame and are aligned using a flat plate so that the slot holding the specimen is in the same plane. Specimen then slides in the slot using a pair of forceps. By matching the holes in the specimen to those in one

of the grip a pin would be inserted. To align the second set of holes, the movable arm of test frame would be moved and second pin would then be inserted. The setup is now ready for testing. In the controlling software, constant cross-head speed of 0.000835 mm/s is specified as discussed above and the test is started. The software records data in the form of displacement and force applied by the arms.

Finally, when the specimen breaks, both the parts should be gently removed from the grips and stored carefully. These might be required to analyze fracture surface structure and microstructure. Table 4 below shows detailed procedure for actual tensile testing.

Table 4: Tensile testing procedure for mini-tensile specimens

Sr. No.	Procedure	Hardware requirement	Time (min)
1	Mount the grips designed for mini-tensile specimens	Grips	15 – 18
2	Align the grips to avoid torsion in the specimen while testing	Specimen plate	2
3	Mount the specimen in the grips	Specimen, forceps, pins	3
4	Set various parameters for MTS and start the test	MTS	2
5	Data collection as the test is running	MTS	15 – 18
6	Take fractured pieces out of the grips and mount new specimen	Forceps and pins	3
	Total		40 – 50

REFERENCES

- [1] ASTM, Standard E-8, “Standard Test Methods for Tension Testing of Metallic Materials”.
- [2] Yongzhong Zhang, Mingzhe Xi, Shiyou Gao, Likai Shi, “Characterization of laser direct deposited metallic parts” *Journal of Materials Processing Technology* 142 (2003) 582–585.
- [3] Bernd Baufeld and Omer van der Biest, “Mechanical properties of Ti-6Al-4V specimens produced by shaped metal deposition” *Sci. Technol. Adv. Mater.* 10 (2009) 015008 (10pp).
- [4] R. Kapoor, N. Kumar, R.S. Mishra, C. s. Huskamp, K. K. Sankaran, “Influence of fraction of high angle boundaries on the mechanical behavior of an ultrafine grained Al-Mg alloy”, *Materials Science and Engineering A*.
- [5] X.L. Shi, R.S. Mishra, T.J. Watson, “Effect of temperature and strain rate on tensile behavior of ultrafine-grained aluminum alloys”, *Materials Science and Engineering A*.
- [6] Z.Y. Maa, S.R. Sharmab, R.S. Mishra, “Effect of multiple-pass friction stir processing on microstructure and tensile properties of a cast aluminum-silicon alloy”, *Scripta Materialia* 54 (2006) 1623-1626.
- [7] J. P. Holman, *Experimental Methods for Engineers*, McGraw-Hill Book co. (5th ed.), p.63 (1989).
- [8] Tiley Jaimie, “Modeling of microstructure property relationship in Ti-6Al-4V”, Ohio State University.
- [9] Maryland Metrics: Titanium Ti-6Al-4V Grade 5 (R56400) Specifications, Maryland Metrics, URL: <http://mdmetric.com/tech/ti6Al-4V.htm>, date accessed: 06/08/2012.
- [10] Technical Data Ti-6Al-4V (Ti-6-4), North American Alloys, URL: <http://www.northamericanalloys.com/Ti-6-4%20props.htm>, date accessed: 06/08/2012.
- [11] Titanium Ti-6Al-4V (Grade 5), STA, ASM, Aerospace Specification Metals, Inc., URL: <http://www.titaniumdistributor.com/titanium-6ai-4v-grade5-eli-annealed.html>, date accessed: 08/14/2012.

- [12] Alloy Data: Titanium Alloy Ti 6Al-4V, Veridiam, URL: <http://www.veridiam.com/pdf/DataSheetTitaniumAlloy.pdf>, date accessed: 06/08/2012.
- [13] S. Naka, L. P. Kubin, C. Perrier, “The plasticity of Titanium at low and medium temperatures”, *Philosophical Magazine A* - Volume 63.
- [14] Mechanical properties and microstructure study for direct metal deposition of Titanium alloy and tool steel, Yaxin Bao, University of Missouri, Rolla.
- [15] Yongzhong Zhang, Mingzhe Xi, Shiyu Gao, Likai Shi, “Characterization of laser direct deposited metallic parts”, *Journal of Materials Processing Technology* 142 (2003) 582–585.

VITA

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