

## INVESTIGATION OF TENSILE PROPERTIES OF BULK AND SLM FABRICATED 304L STAINLESS STEEL USING VARIOUS GAGE LENGTH SPECIMENS

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### **Abstract**

The complex solidification dynamics and thermal cycling during Selective Laser Melting process is expected to result in non-equilibrium material characteristics. There is an essential need for characterization techniques which are critical towards the estimation of anisotropies. The current investigation is targeted towards establishing tensile testing methodologies and their relation to differing gage lengths. Dog-bone shaped specimen designs with gage lengths of 1", 0.3" and 0.12" were employed in this research. The characterization was performed on hot rolled-annealed 304 stainless and SLM fabricated 304L stainless. It was theorized that smaller gage length specimens would be instrumental in mapping material property anisotropy at a better spatial resolution. The ultimate tensile and yield strength data were used to identify the material property distribution and assess the anisotropy. The material property distributions were used to successfully assess the testing methodologies and material characteristics.

### **Introduction**

Selective Laser Melting (SLM) is an Additive Manufacturing (AM) technology which employs a layer by layer fabrication methodology within a powder bed setup to manufacture desired parts. The smaller size range of powders used in SLM process makes it feasible to fabricate parts with strict dimensional tolerances, while attaining good surface finish [1]. It's been identified that basic surface clean up treatments like sand blasting have been noted to improve the average surface roughness (to as low as 5 micron) of SLM components [2, 5]. With more and more research being done on the SLM process, its potential for fabricating a finished product is becoming more and more evident [1,2]. However there are still a substantial number of issues that need to be addressed [1-4]. "To achieve everything that metal AM has to offer, we have to thoroughly understand the physics of the process and exploit the unique aspects to our advantage" [2]. The procedure's range of fabrication capabilities have been well explored, however the mechanical performance of AM parts is yet to be thoroughly quantified. A variety of materials have also been studied for AM fabrications, namely, titanium alloys, steels, nickel alloys, aluminum, copper alloys etc. [6-9]. It is to be noted that while the list of materials being examined seems extensive, only commercially available alloys have been considered for examination. Researchers are also tailoring alloys (with material addition) to effectively exploit the extensive customization capability that AM technology has to offer. To guarantee the consistent production of parts though, the manufacturing technology, build strategy and the properties of the SLM constructed material need to be comprehensively studied and identified. It has been investigated and widely reported that the AM materials consist anisotropic properties

that are different from material fabricated from conventional methods [10]. The rapid solidification of material and the logistics behind the process (process variables such as rastering, overlap, scan strategy, etc.) result in non-equilibrium properties [11-13]. Also, substantial thermal gradients occur during fabrication in AM processes, whose resultant thermal stresses cause distortion and residual stresses [14]. Cooling rates as high as  $10^3$  to  $10^4$  K/s have been recorded during AM[11] in which the heat flow is directional and the layer by layer build approach causes thermal cycling. The high cooling rates, directional heat flow and thermal cycles have complex effects on the microstructure [15-17] produced using AM. These characteristics of fabrication cause various types of anisotropy and residual stresses. The scale of these effects can vary depending on the input power, build strategy employed material used etc. Material properties such as thermal conductivity, coefficient of thermal expansion, elastic modulus, yield stress etc. are all affected by the residual stresses [18-20].

Comprehensive characterization of AM material properties are therefore required to qualify the material for safe and reliable incorporation into production. One of the key advantages of the AM methodology is scalability. Different sizes of the same CAD model can be built using an AM technology such as SLM. The precision and surface roughness outcomes of the SLM process make it a definite solution for fabricating various sizes of parts [2, 5]. In such a scenario, there is a need for characterization methodologies that are capable of handling such wide range of sizes. These techniques must be robust enough to pick up the variations within the material. One of the most popular and age-old material property characterization method is tensile testing. According to current testing standards the smallest specimen that can be characterized using tensile testing would need have a minimum gage length of 1 inch and close to 3 inches in length for fixturing. To put it in terms of the SLM process, an inch size material would constitute close to 500 layers [1]. A lot of activity can happen during the fabrication of these layers and the consequent material may therefore have anisotropy. Confining testing to existing standards, can make the characterization study expensive and would limit the scope for testing stock material which is smaller than standard requirements. Miniature testing methodologies are needed to expand the capability for studying and understanding the various attributes of SLM material. Apart from using existing alloys, new kinds of alloys are always being developed for AM processes. Coming up with enough stock material for testing using existing testing standards might prove extremely difficult and cost prohibitive. The recommended course of action in this case would be the use of miniature testing methods which can be used to gather comprehensive insight from very little stock material.

Miniature testing methodology, especially tensile testing has been a topic of study for a while now. Researchers from nuclear engineering have been some of the most active in these studies. The intentions behind these studies were to limit operator's exposure to harmful radiation while characterizing radiation affected material [21-26]. Other studies have been performed to identify the impact of specimen design, aspect ratio, thickness to width ratio etc. Experimental and theoretical studies were performed to investigate the validity and also model the miniature testing strategies. The studies concluded that miniature testing methods can be valid methods to estimate performance life and characterize properties reliably [27-31]. During many of these studies the miniature specimens used for testing were fabricated by using a punch to shear them from sheet metal. This method though might prove difficult if the base material's thickness was large. Also, the punch might cause work hardening in the specimens as well. The

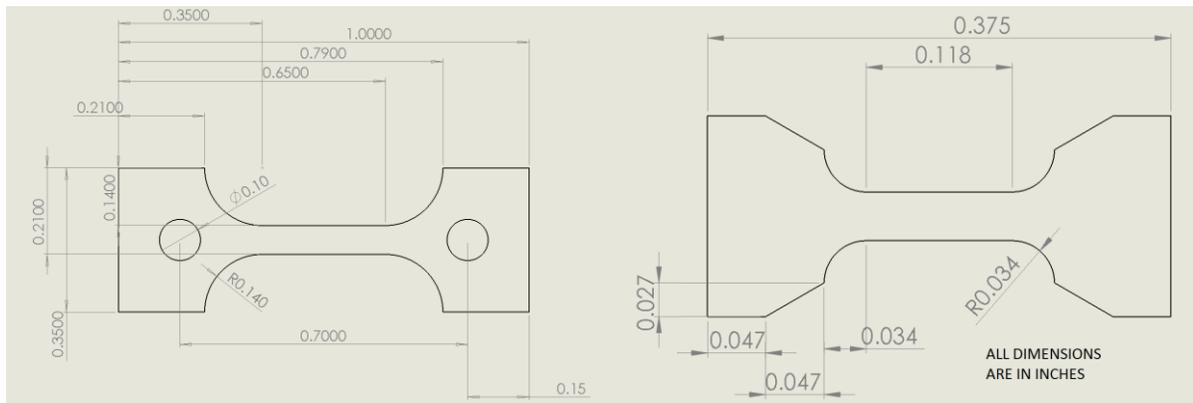
current study involves development and implementation of miniature tensile testing methodologies for testing metallic dog-bone shaped specimens. The novelty of the current study includes the specimen size, preparation methodology and the test specimen's fixturing. The comparative study on characterizing hot rolled and SLM fabricated material across various sizes and gage lengths helps gain insight into differences between materials using these miniature testing methods. Unlike existing miniature specimen designs, the smallest specimen design requires minimum material for clamping and the gage length is also comparable. Incorporation of wire based Electro-Discharge Machining process (W-EDM) gives the possibility to sample miniature specimens from material of various sizes. The near zero heat affected zone from EDM process helps preserve the stock/as-deposited structure such that the testing methodology can provide an accurate estimate of a material's properties.

### Experimental Setup

The three specimen designs considered for testing were,

- ASTM sub-size specimen ASTM-E8
- Miniature tensile specimen designed developed by ABI services LLC and,
- A mini-tensile specimen design developed at Missouri University of Science and Technology.

The ASTM sub-size specimen was a dog bone shaped tensile coupon with a gage length of 1 inch (25.4mm) and a total length of 4 (100.16mm) inches. The specimen was pulled on an INSTRON universal testing machine. The specimens were gripped on wedge grips with serrated clamping faces. The miniature tensile specimen design from ABI services LLC, had a gage length of 0.3 (7.62 mm) inches and was gripped by pin holes. A drawing of the specimen is shown in figure 1 (a). This specimen from here on is referred to as Mini-tensile 1 (MT1). The miniature specimen developed at Missouri University of Science and Technology, had a gage length of 0.118 (3mm) inches and was tested by pulling against the inclined faces on the specimen. The drawing of the specimen is shown in figure 1 (b). This specimen from here on is referred to as Mini-tensile 2 (MT2).



(a)

(b)

Figure 1: (a) & (b) are drawings of MT1 and MT2 specimen designs respectively (Not to scale)

Custom grips were needed to be manufactured to facilitate the testing of MT1 and MT2 on the INSTRON testing machine. The grips were designed to be robust and rigid enough to handle these tests. In order to exclude any torsion or bending during testing of the miniature specimens, self-aligning grips were designed and fabricated. The self-aligning grips contain two joints, which have perpendicular axis of rotation that emulate a ball and socket joint within a small solid angle. These degrees of freedom were expected to sort out any miss-alignment issues and thereby ensure a uniaxial tensile testing for both the miniature tensile designs. The self-aligning grips on the INSTRON machine are shown in figure 2.

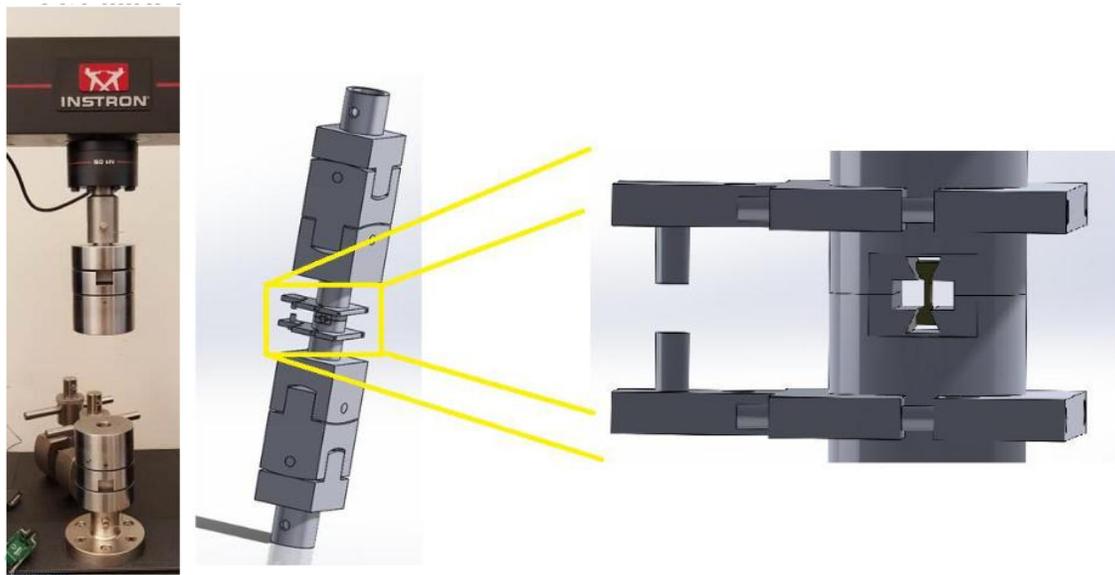


Figure 2. Setup showing the self-aligning grips on the INSTRON testing machine. Also, a CAD representation of assembly of the self-aligning grips and expanded view of grips of the MT2 specimens. The extensions on the MT2 grips represent the extensometer.

The tensile tests were run as a series of closed loop strain controlled tests. Extensometers of 1 inch (25.4 mm) and 0.6 inch (15.24 mm) were used to run these tests. The 1 inch gage length extensometer was attached directly to the ASTM sub-size specimens. However, setting the extensometer directly on the smaller specimens was not feasible. The extensometers were attached directly to the grips of the miniature specimens and it was assumed that all the deformation noted by the extensometer came from elongation of the specimen. A strain rate of 0.015mm/mm/min was used up to a strain of 0.05 and then the extensometer was removed and a strain rate of 0.5mm/mm/min was used to pull the specimen to fracture. The same strain rate schema was used for all types of specimens.

For the comparative study, specimens were machined from hot-rolled and annealed steel while the SLM specimens were built to size using the SLM process. A Renishaw AM250 machine was used to build these SLM specimens. The bulk material was machined using the W-EDM to obtain ASTM, MT1 and MT2 specimens. The specimens were machined to sizes within 0.1% of the required dimensions. The bulk specimens were pulled with smooth EDM finish,

whereas the SLM specimens were pulled in as built condition. The number of specimens per type are shown in table 1. The chemistry of the bulk material and powder used for SLM fabrication are listed in table 2. By chemistry the bulk material can be categorized as 304 stainless steel and the powder is 304 L stainless steel (due to its low content of interstitials, C, N, O etc.). The close chemistries of both materials was intended to aid in better estimating the potential of the miniature testing methods.

Table 1. Total number of specimens prepared and pulled per specimen design

Specimen Design	Bulk	SLM
ASTM	35	36
MT1	42	39
MT2	55	38

Table 2. Chemistries of the bulk material and powder (for SLM) used for the current study

Material	C	Mn	Si	S	P	Cr	Ni	Cu	Mo	Co	N	O
Bulk	0.023	1.69	0.43	0.020	0.034	18.10	8.02	0.63	0.24	0.15	0.0840	-
Powder	0.015	1.4	0.63	0.004	0.012	18.5	9.9	<0.1	-	-	0.09	0.02

The SLM specimens were all built in a single run cycle. The specimens were laid in a grid fashion with a separation of 15 mm on all sides. A total of 40 specimens each per category of specimen design were built. Each specimen had a 3 mm tall support structure. The layout of specimens on the build plate is shown in figure 3. Upon preliminary inspection, it was noted that the build was distortion free. All the specimens had consistent surface finish and all the features of the specimens were built as designed. The pin holes on the MT1 specimens were slightly undersized. The holes had to be reamed to right size to accommodate the pins. This is one of the drawback of the MT1 specimen design, drilling/ reaming the holes might create issues with respect to fixturing. Improper and careless handling during drilling can induce work hardening or distortion within the reduced section length.

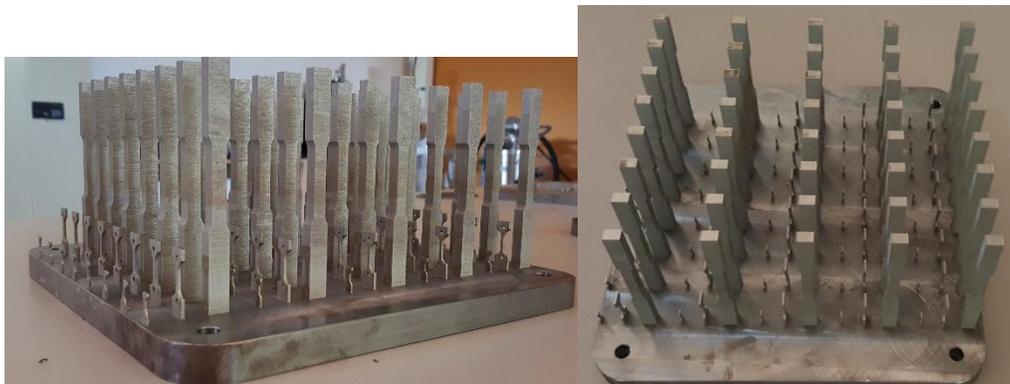


Figure 3. Side and top view of SLM specimens on the build plate. A total of 120 specimens, 40 each of ASTM, MT1 and MT2 specimens were built

## Results & Discussions

A total of 245 specimens from all the three types of specimen designs were successfully prepared and tested. The INSTRON testing machine was certified to be valid for testing specimens in accordance with existing testing standards. The miniature specimens were expected to require careful attention in terms of setup and machine alignment. Even a slight misalignment could induce torsion or bending on the specimen and, make the test invalid. Figure 4, shows the MT2 specimen (smallest of all the designs) at various stages of testing. The images show signs of perfect uniaxial testing, the specimen goes through elastic region, plastic region, necking region and failure.

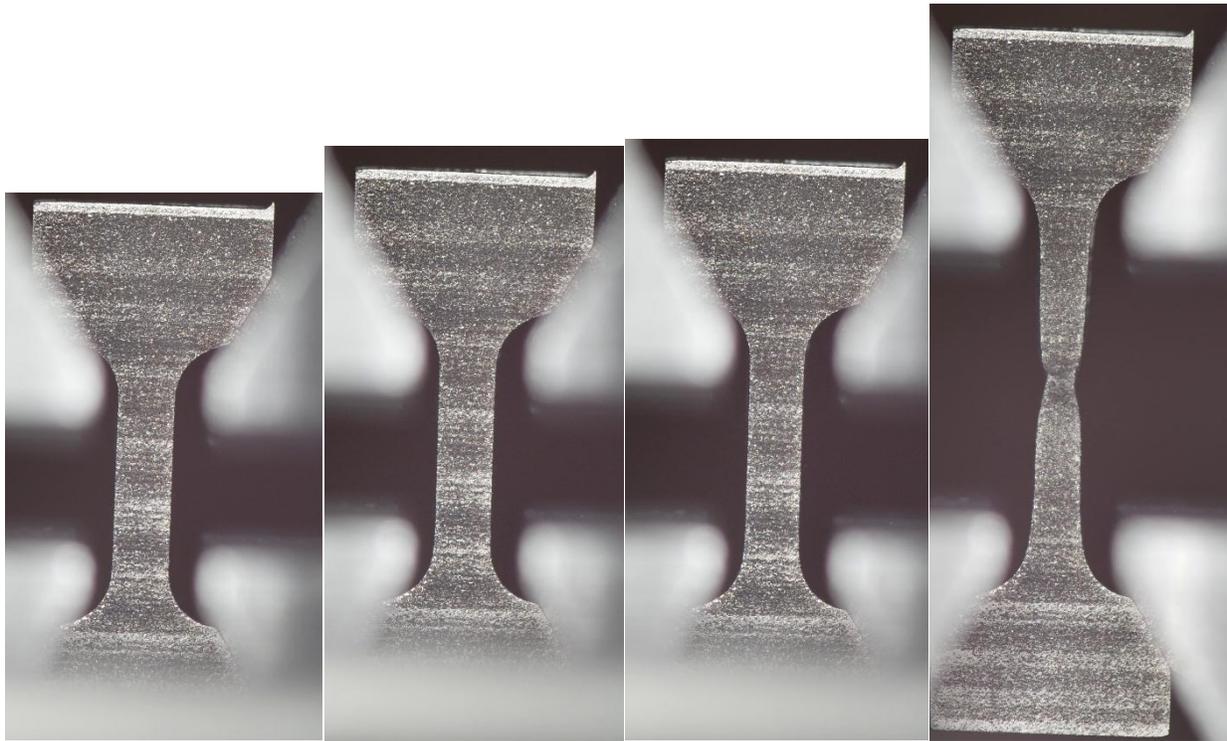


Figure 4. Images of a bulk MT2 specimen in various stages of deformation during testing. Left to right, the specimen at start of the test, during the elastic region, during the elastic-plastic region and, necking right before failure (Images taken across unequal spans of time)

Specimens of all three designs that were built using the SLM were also successfully tested. The fractured specimens from all specimen designs are shown in figure 5. The tensile data gathered from these test includes 0.2% Offset Yield Strength (YS02), Yield Strength at 0.5% Strain (YS05), Ultimate Tensile Strength. The average and standard deviation values obtained are listed in table 3. The YS02, YS05 and UTS data was then fitted with multi-modal Weibull distribution to identify the distribution in material properties. The curve fitting was done using MATLAB's Statistics toolbox. The fits were verified using JMP statistical analysis software. A lack of fit analysis revealed all the fits to be good within a significance level of 0.05.

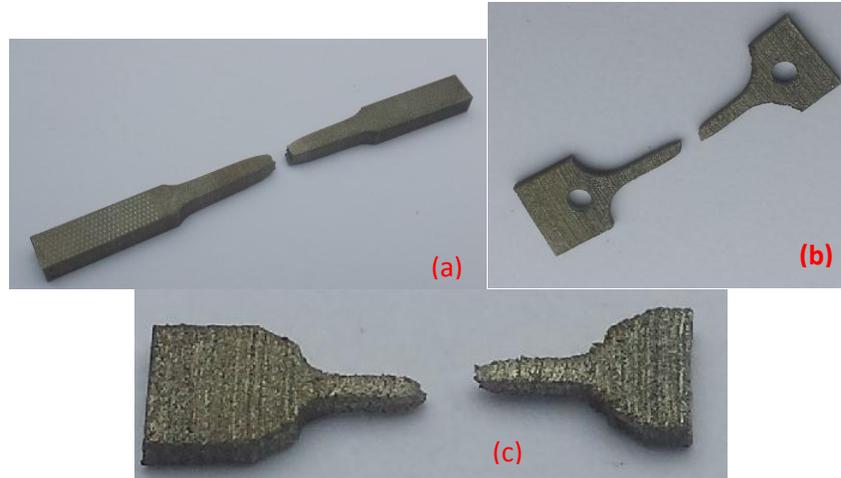


Figure 5 (a), (b) and, (c). Fractured SLM built tensile specimens of ASTM subsize (a), MT1(b) and, MT2(c) (Images not to scale)

Table 3. Average and standard deviation values of YS02, YS05 and UTS values

SLM_MT1			
	YS05	YS02	UTS
Mean	360.0	400.4	547.9
Std Dev	44.6	18.2	27.3
SLM_MT2			
Mean	217.2	368.4	536.7
Std Dev	28.3	19.1	29.8
SLM_ASTM			
Mean	380.3	378.9	556.0
Std Dev	95.2	66.3	6.6
Bulk_ASTM			
Mean	477.8	475.1	685.3
Std Dev	33.5	27.9	18.0
Bulk_MT1			
Mean	444.58	526.92	742.38
Std Dev	94.09	31.73	17.53
Bulk_MT2			
Mean	527.4	582.5	780.9
Std Dev	105.7	42.6	25.2

Figure 6 details the histogram and its weibull fit for UTS data of bulk and SLM material gathered using the ASTM specimen. It can be noted that the distribution of properties could be bi-modal fit. The number of specimens tested might not be sufficient enough to estimate the distribution accurately. However there is enough evidence to suggest the bi-modality in material properties and estimate the modes of the property distribution. It needs to be noted that the SLM specimens were built to size, whereas the bulk specimens were all cut from the same stock material. It can be expected that the bulk specimens would all have similar flaw

distribution where as the SLM specimens would have different flaw distributions and, thereby different property distributions.

The authors believe the higher material property estimates from MT1 and MT2 specimens of bulk material could be explained from a reliability stand point. The MT1 and MT2 specimens sample smaller volume of stock material and therefore contain smaller number of flaws in comparison to ASTM size specimens. This lower flaw volume could result in higher performance of the material, therefore higher values of material property estimates. The same stays true when the property estimates of MT1 and MT2 are compared. The estimates of MT1 are lower than MT2 with certain statistical significance.

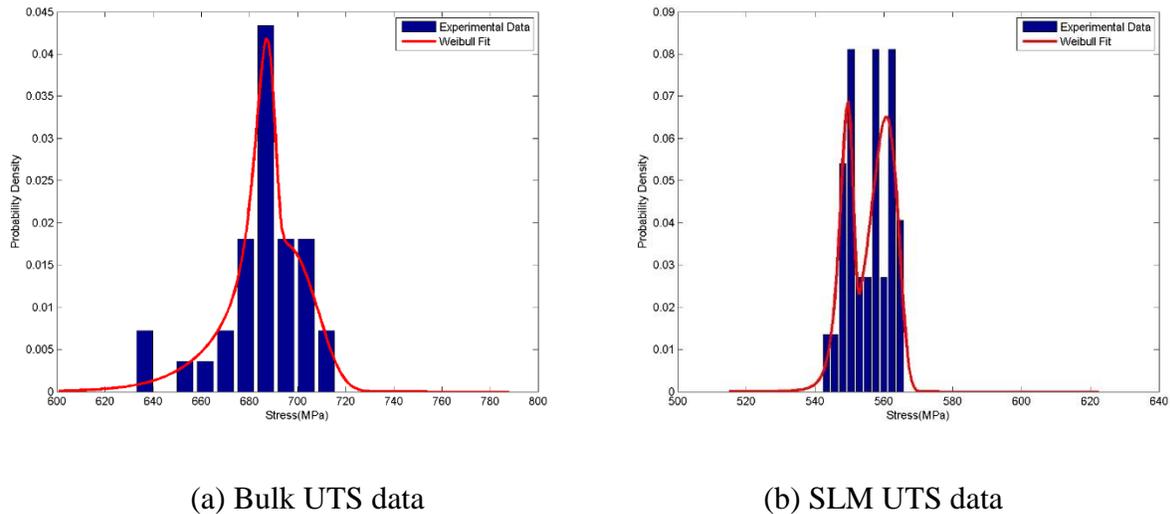


Figure 6 (a)&(b). Histograms of UTS data gathered from testing bulk and SLM material. The histogram data was fit with bi-modal Weibull distribution to model the property distribution

The distribution fits from YS02, YS05 and UTS of bulk data were all overlaid to investigate the drift in material property estimates. The overlaid fits are shown in figure 7. The distributions show a right shift indicating increase in material properties however the distribution of properties is still very similar. This further strengthens the argument that was made previously. The estimates are higher due to smaller flaw volume but the flaw distribution is still the same, hence the property distribution should also be similar.

The distribution fits from YS02, YS05 and UTS of SLM data were also overlaid (shown in figure 8). There was no drift like in the case of bulk data. Since specimens were all built to size, they contain different flaw distributions unlike the case of bulk specimens. Its interesting to note that UTS estimates from all specimen designs are very close but, the yield data consists of very wide distribution. Also the higher mode of property estimates is seen to be absent in data gathered from MT2 specimens. However the bi-modality shows up in YS05 data. This proves that the build material contains different flaw distributions with different sizes. The miniature testing methodologies are capable of estimating and pointing out the differences between material properties based on the differences in flaw distributions.

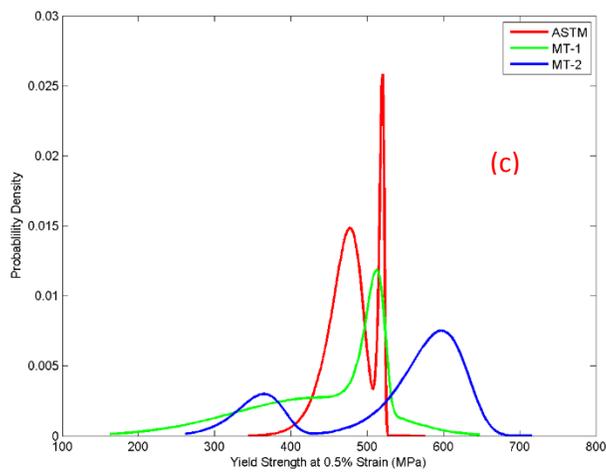
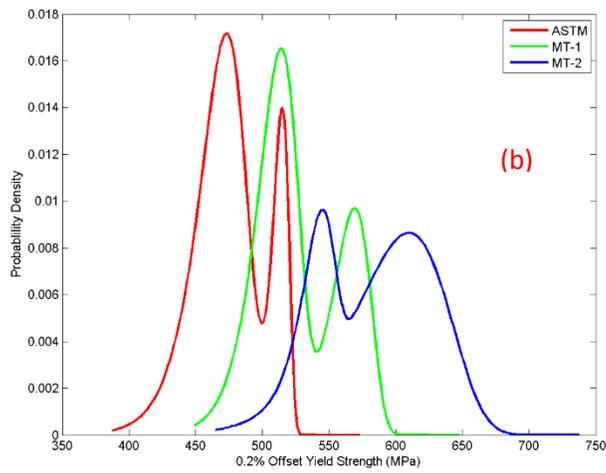
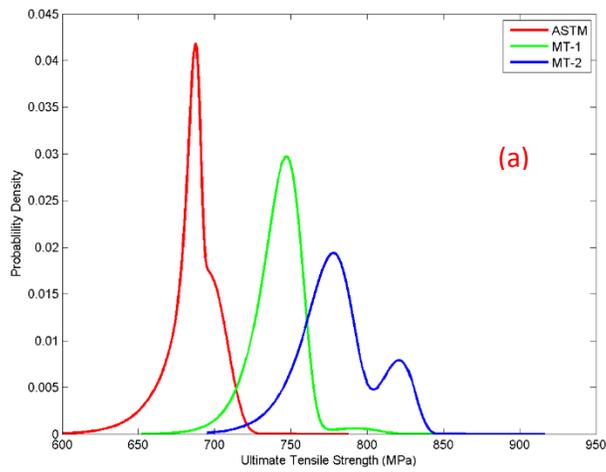


Figure 7. (a) Overlay of UTS distributions estimated from histograms obtained from all specimen designs (b) YS02 distribution overlay (c) YS05 distribution overlay

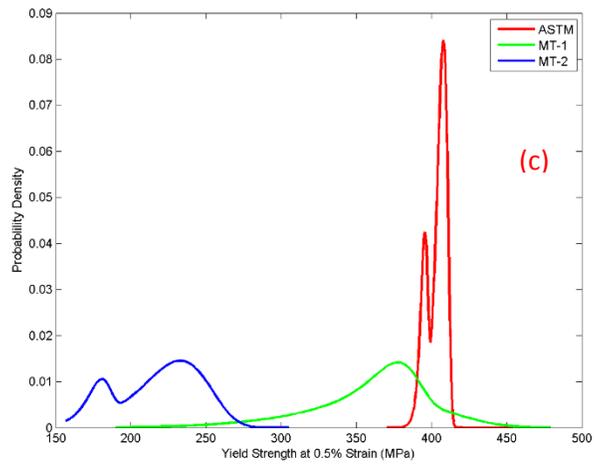
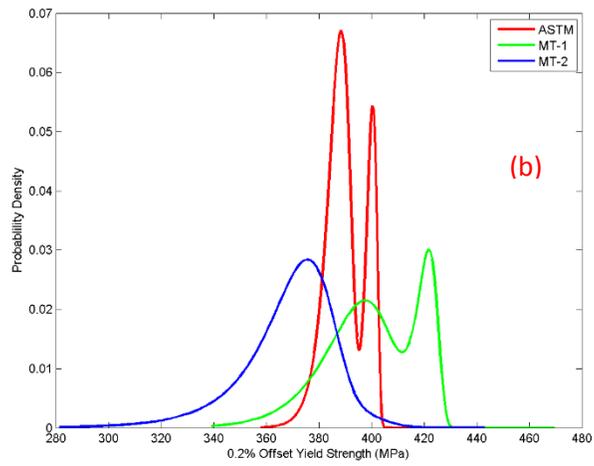
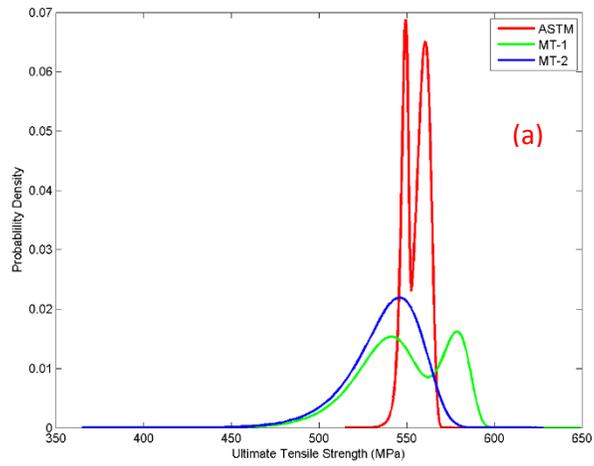


Figure 7. (a) Overlay of UTS distributions estimated from histograms obtained from all specimen designs (b) YS02 distribution overlay (c) YS05 distribution overlay

The wide variation in yield strength data gathered from the MT1 and MT2 SLM specimens could be attributed to their build. Even though both the specimens have been built with the same parameters, the MT2 specimens being smaller than the MT1 specimens would have undergone different solidification dynamics in comparison to MT1. The reheat cycles within the MT1 specimen are more than the MT2 specimens, and the sizes are also very different. These attributes could result in different microstructures.

### **Conclusions**

Three types of dog-bone shaped specimen designs were used for the current study. A robust setup for testing miniature specimens was designed and built for an INSTRON universal testing machine. Hot rolled –annealed bar stock material was used to prepare the specimens for all 3 types of specimen designs. A Renishaw AM250 SLM was used to build the same 3 types of specimens via SLM process.

The testing of these specimens was successfully carried out and material property estimates were gathered. The data was used to fit multi-modal Weibull distribution to obtain distribution of material properties. The distribution fits were overlaid to compare the different specimen design outcomes. A rightward shift (higher strength) was observed in distribution plots of bulk data. This was attributed to higher performance of material caused by sampling of smaller volume of material in the miniature specimens.

The SLM distributions showed that the UTS's modes were very similar among all specimen designs. However, the higher mode of properties was absent in the MT2 property estimates. This indicates a distinctly different property distribution. This variation is expected to be caused from being different in size and undergoing different solidification dynamics.

The miniature specimens prove to be instrumental in characterizing material properties reliably. The study on bulk material confirms consistency in results gathered from all specimen designs. The SLM study highlights the material property variation that could occur from difference in sizes.

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