High-throughput Stochastic Tensile Performance of Additively Manufactured Stainless Steel

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Submitted July 2016 to *Journal of Materials Processing Technology*

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Abstract

An adage within the Additive Manufacturing (AM) community is that “complexity is free”. Complicated geometric features that normally drive manufacturing cost and limit design options are not typically problematic in AM. While geometric complexity is usually viewed from the perspective of part design, this advantage of AM also opens up new options in rapid, efficient material property evaluation and qualification. In the current work, an array of 100 miniature tensile bars are produced and tested for a comparable cost and in comparable time to a few conventional tensile bars. With this technique, it is possible to evaluate the stochastic nature of mechanical behavior. The current study focuses on stochastic yield strength, ultimate strength, and ductility as measured by strain at failure (elongation). However, this method can be used to capture the statistical nature of many mechanical properties including the full stress-strain constitutive response, elastic modulus, work hardening, and fracture toughness. Moreover, the technique could extend to strain-rate and temperature dependent behavior. As a proof of concept, the technique is demonstrated on a precipitation hardened stainless steel alloy, commonly known as 17-4PH, produced by two commercial AM vendors using a laser powder bed fusion process, also commonly known as selective laser melting. Using two different commercial powder bed platforms, the vendors produced material that exhibited slightly lower strength and markedly lower ductility compared to wrought sheet. Moreover, the properties were much less repeatable in the AM materials as analyzed in the context of a Weibull distribution, and the properties did not consistently meet minimum allowable requirements for the alloy as established by AMS. The diminished, stochastic properties were examined in the context of major contributing factors such as surface roughness and internal lack-of-fusion porosity. This high-throughput capability is expected to be useful for follow-on extensive parametric studies of factors that affect the statistical reliability of AM components.

Keywords: additive manufacturing; rapid prototyping; 3D printing; deformation; tensile; statistics

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1. Introduction

One of the key benefits of Additive Manufacturing (AM) is the ability to rapidly produce customized components with complex geometry. However, the full benefit of this rapid production turnaround cannot be fully realized unless all elements of the design-production-qualification route are streamlined. In addition to the development of topological optimization algorithms for efficient requirements-driven design, such as described recently by Gardan (2014), it is necessary to develop qualification pathways that can rapidly assess material quality and performance.

To take advantage of the AM benefit of geometrically complex features, a new high-throughput tensile testing approach was developed. This new approach provides an extensive assessment of stochastic variability in mechanical properties. With this new approach, hundreds of high-fidelity tensile tests can be performed in a similar time and for similar cost to a few conventional tests. The current study has three primary objectives: (1) demonstrate the efficacy of this high-throughput testing methodology to extract statistical distributions of mechanical properties; (2) employ the high-throughput method to compare the performance of nominally-identical material produced by two independent commercial vendors; and (3) identify the defects associated with lower-tail worst-case performance and statistical outliers.

The AM process of interest in the current study is powder bed fusion, defined in ISO/ASTM 52900 (2016). Variations of powder bed fusion are also known by common names such as Selective Laser Sintering, Direct Metal Laser Sintering, Selective Laser Melting, and Electron Beam Melting. For examples of the usage of these four alternative names in the literature, see works by Beaman et al. (1990), Khaing et al. (2001), Kruth et al. (2004), and Cormier et al. (2004), respectively. Powder bed fusion is also known by proprietary names such as Direct Metal Printing (3D Systems, Inc.) or LaserCUSING® (Concept Laser GmbH). In these processes, a layer of unfused powder is placed on the build tray and locally fused through the directed application of an external heat source, typically a laser or electron beam. Sequential layers are built-up and fused together in this layer-by-layer process. The use of a bed of packed powder distinguishes this process from other common metal AM processes such as Laser Engineered Net Shaping employed by Atwood et al. (1998) or Direct Metal Deposition employed by Mazumder et al. (1997) in which the powder is dynamically injected into the focal point of the laser. Some of the most common defects that arise in metal powder bed additive manufacturing which give rise to stochastic performance include voids, channels bridging many layers and partially melted and sintered particles. For example, Bauereiß et al. (2014) observed and simulated the formation of channel-like faults that form due to insufficient heat input. For laser powder bed fusion, several researchers including Shamsaei et al. (2015) and Liu et al. (2016) have described how the type, size, and spatial distribution of defects is dependent on a number of factors, including: powder chemistry, particle size distribution, printer atmosphere, laser power, traverse rate, hatch pattern, part thickness, and build orientation. Many of these parameters can vary within a build, between builds, or from vendor to vendor, leading to a lack of consistency in material performance.

The current study demonstrates the high-throughput testing methodology and ensuing analysis of stochastic mechanical properties in a precipitation hardenable stainless steel, commonly known as alloy 17-4PH produced by laser powder bed fusion. Precipitation-hardening stainless steels such as 17-4PH were developed to provide high toughness and strength while maintaining the benefit of corrosion resistance. Several ferrous metallurgy reference books describe the metallurgical principles of precipitation hardened stainless steel alloys, including the book by Krauss (1990). Precipitation-

hardening alloys are particularly amenable to AM because the mechanical properties can be controlled via post-deposition heat-treatment. While some stainless steel alloys made by additive manufacturing have been shown to have average properties that can be comparable to wrought Rafi et al. (2013) or cast Tolosa et al. (2010) product, generally these previous studies involve only a few measurements with very limited detail regarding the *variability* in mechanical properties.

2. Method

Material and Specimen Design

Tensile specimens were fabricated from precipitation-hardened 17-4PH, also known as Alloy 630 or AMS 5604 (UNS number S17400). While 17-4PH is the commonly used name for this alloy in wrought form, similar casting variations include CB7Cu-1 (UNS J92180) and AMS 5342-5344 (UNS J92200). This alloy was produced from powder feedstock directly using a powder bed fusion processes at two vendors using different commercial systems. Vendor 1 utilized a ConceptLaser Mlab printer and Vendor 2 utilized a 3DSystems ProX™ 300 printer. Both machines employ a laser-based powder bed fusion process to produce a three-dimensional metallic object. Both vendors produced net-shaped tensile bars with the tensile axis parallel to the vertical build direction. This resulted in the individual print layers arranged in a laminate structure perpendicular to the tensile axis. The layer thickness of parts manufactured by the Concept Laser Mlab printer was 20μm and by the 3DSystems ProX™ 300 printer was 40μm. Aside from the build orientation and specimen geometry, no additional constraints were placed on the process parameters such as laser power, traverse rate, powder chemistry, feedstock source, etc. Instead, each vendor utilized their proprietary expertise to select appropriate conditions that would achieve nominal 17-4PH components. In this way, the present study is not intended to be a systematic assessment of process parameter effects on resulting properties, but rather an illustration of the variation that can be observed when requesting nominally identical manufactured components. After printing, the AM tensile specimens were solution treated (1037°C/1 hr/air cool) and subsequently aged to the H900 condition (482°C/1 hour/air cool). To complement the population of AM tensile bars, geometrically-equivalent tensile specimens were also electrodischarge machined from 1 mm thick commercially-produced wrought sheet supplied in Condition A and heat treated to the same nominal H900 condition. Samples were machined with the tensile axis parallel to the rolling direction.

The nominal tensile bar geometry is shown in Figure 1. The nominal width and thickness of the specimen gauge section were both 1mm. The nominal gauge length was 4mm. The geometry deviates from ASTM (2015) E8 standard geometries to eliminate unprintable horizontal overhang features and facilitate a compact, cost-efficient test sample volume. The unusual 45-degree wedge-shaped end sections of the rectangular dog-bone tensile bars not only eliminated overhang issues, but also facilitated self-alignment in the open-face grips. A layout of 120 tensile samples was recommended to the vendors as shown in Figure 1. While Vendor 1 produced this exact arrangement (Figure 1b), Vendor 2 added protective structural sidewalls to prevent the print wiper/roller from bending the vertical tensile bars. The Vendor 1 product was able to avoid bending without the protective barriers, presumably due to differences in the print roller: a rigid roller employed in some 3D printers such as the ProX™ line is more likely to deform high-aspect ratio features.

Figure 1. a) Build Array of 120 Tensile Specimens. b) Printed array of 120 Tensile Specimens. c) Tensile specimen geometry.

The chemical composition of each material was measured by inductively coupled plasma mass spectroscopy. Light elements including carbon, nitrogen, oxygen, and sulfur were measured using LECO combustion analysis. Table 1 lists measured compositions. Industrial alloy specification limits for investment cast and wrought sheet 17-4PH are also included for reference. While the composition was generally consistent with the 17-4PH alloy, the AM parts exhibited elevated copper, oxygen, and nitrogen.

Table 1: Chemical Composition of 17-4. “NS”= None specified.

Composition	Carbon	Manganese	Silicon	Phosphorus	Sulfur	Chromium	Nickel	Niobium	Copper	Oxygen	Tantalum	Aluminum	Tin	Nitrogen	Molybdenum	Iron
Wrought Specification AMS5604	0.07 Max.	1.00 Max.	1.00 Max.	0.04 Max.	0.03 Max.	15.00-17.5	3.00 - 5.00	0.45 Max.	3.00 - 5.00	NS	NS	NS	NS	NS	0.05 Max.	Remainder
Wrought Sheet	0.042	0.51	0.21	.026	<0.001	15.65	4.67	0.28	3.24	0.0036	0	0	0	0.023	0.28	
Cast Specification AMS5344	0.06 Max.	0.70 Max.	0.50 - 1.00	0.025 Max.	0.025 Max.	15.50 - 16.70	3.60 - 4.60	0.15 - 0.40	2.80 - 3.50	NS	0.05 Max.	0.05 Max.	0.02 Max.	0.05 Max.	NS	
Vendor 1	0.012	0.24	0.38	0.019	0.003	16.64	4.24	0.3	4.05	0.100	0	0	0	0.056	0.045	
Vendor 2	0.021	0.46	0.62	.023	0.006	16.51	4.53	0.29	4.08	0.083	0	0	0	0.15	0.17	

The tensile specimens in this study were produced in the final shape by the AM process with no post-process machining. Vendor 1 removed loose particles and improved the surface finish through a bead blasting process whereas Vendor 2 provided as-printed parts. The surface roughness of the tensile bars was measured using a Bruker ContourGT-I 3D Optical Microscope. The surface roughness (R_a) of the samples produced by Vendors 1 and 2 was 5.6 μm and 18 μm (± 0.1 μm) and maximum peak height (R_p) of 36 μm and 82 μm (± 0.6 μm), respectively. The wrought 17-4 samples had a surface roughness (R_a) and maximum peak height (R_p) of 0.05 μm and 6.6 μm , respectively, on the as-rolled surface (top and bottom) and R_a and R_p of 2.6 μm and 33 μm , respectively on the EDM surface (sides). The rough surfaces of the AM material obscured accurate dimensional measurements and in the case of Vendor 2, appeared to be the origin of premature tensile failures, as addressed in detail in the Results and Discussion sections.

Figure 2 shows representative backscatter electron micrographs of electropolished 17-4PH wrought sheet and AM material produced by Vendors 1 and 2. The wrought sheet microstructure (Figure 2c) heat-treated to the H900 condition shows a matrix of lath martensite with uniformly distributed equiaxed particles that appear bright in the image. Murayama et al. (1999) and Chen et al. (2012) have shown that these equiaxed particles are associated with the MC-type Nb carbides expected in heat treated 17-4PH. The as-heat-treated AM microstructures from both vendors (Figure 2a and 2b) show predominantly martensitic microstructures with similar dispersed submicron Nb-rich second phase as observed in the wrought material. The backscatter electron micrographs of both AM microstructures are largely devoid of solidification substructure due to the diffusional and martensitic transformations following solidification plus the additional phase transformations occurring during the solution treating and age heat treatment thermal cycles. The microstructure of Vendor 2 (Figure 2b), shows grains without any intragranular contrast formation associated with lath martensite—these grains are denoted with arrows. These regions were identified using electron backscatter diffraction as austenite retained after heat treatment. X-ray diffraction indicated ~30% volume fraction of retained austenite in the Vendor 2 microstructure, consistent with prior reports of retained austenite present in AM 17-4PH by Murr et al. (2012), Rafi et al. (2013), and Yadollahi et al. (2016). The austenite phase is a lower strength phase that does not gain strength during an aging heat treatment. The origin for the large fraction of retained austenite in Vendor 2 material is attributed in part to elevated nitrogen content (0.15 wt %); however, a quantitative analysis of the causality is the subject of a separate study. A prior study on AM 17-4PH by Murr et al. (2012), explored the role of the atmosphere during gas atomization process on subsequent phase formation. They showed that gas atomization in a nitrogen atmosphere promoted austenite formation.

Figure 2. Backscatter electron micrographs of electropolished metallographic cross-sections comparing the laser powder bed materials produced by (a) Vendor 1 and (b) Vendor 2 to (c) wrought sheet.

Tensile Method

A high-throughput test protocol was developed to evaluate the tensile properties of the AM structures. In lieu of hydraulic- or screw-clamped grips, open face “drop-in” grips were custom manufactured to facilitate rapid sample loading and unloading. Figure 3 shows a cutaway of the grips with a tensile sample loaded.

Mechanical tests were performed using an MTS servo-hydraulic load frame and an Interface 445N load cell. For all tests, the actuator displacement rate was 0.05 mm/s, resulting in a gauge section strain rate of $\sim 4 \times 10^{-4} \text{ s}^{-1}$. Strain measurement was performed by real-time non-contact Digital Image Correlation (DIC) using the commercial software VIC-Gauge™, by Correlated Solutions, Inc. Unlike conventional DIC analysis where strain values are computed by post-processing of raw images, the VIC-Gauge™ approach produces real-time strain measurements at camera refresh rates. In the current study, these strain measurements were acquired at 30 Hz or faster. The strain values were transferred from the VIC-Gauge™ software into the MTS data acquisition software using A/D and D/A converters. The native surface roughness of the test samples provided sufficient speckle contrast for DIC analysis. The subset size in DIC is known to affect spatial resolution and strain resolution. For these experiments, a subset size of $0.52 \times 0.52 \text{ mm}$ (181×181 pixels, $\frac{1}{2}$ of the gauge width) was chosen, resulting in a measured strain noise floor of 7.22×10^{-4} (1 SD). An MTS automation procedure was written to streamline sample engagement, preloading, testing, and sample recovery. A Matlab code was developed to process all of the raw data and calculate stress/strain response curves as well as 0.2% offset yield

strength, ultimate tensile strength, uniform elongation, and strain to failure. Minitab 17 was used to generate statistical distribution graphs and JMP statistical analysis software was used to estimate distribution parameters using a maximum log-likelihood methodology.

Figure 3. a) Model of Additive Manufactured Tensile Specimen in grips (cut-away). b) Mechanical Test Set-up

3. Results

High-Throughput Tensile Testing

Figure 4 compares the tensile stress-strain behavior of additively manufactured 17-4PH from two different vendors to that of conventional wrought sheet. In all three cases, the tensile sample size/geometry was nominally identical and a similar statistical population of size $N=97-104$ was sampled. The stress values in Figure 4 were calculated by dividing the applied force by an average cross-sectional area, as measured using contact-based micrometers. The bias caused by these contact-based measurements on the rough AM surfaces is further analyzed in Section 4. Both of the AM vendors produced 17-4PH material that exhibited lower strength and ductility than wrought sheet in the same specimen form factor. However, since AM involves melting and re-solidification, a more fair assessment may be to compare against the properties of castings. While the tensile behavior of casting-equivalent 17-4 alloys was not measured in the current study, there are minimum allowable properties established in standards such as AMS 5344. The minimum allowable values are meant to set lower bound expectations for reliable engineering design, and are not typical properties. Dashed lines in Figure 4 indicate these AMS 5344 minimum allowable properties for both cast and wrought material. Vendor 1 produced samples with higher ultimate strength and on average, larger strain to failure than that of Vendor 2. Roughly half of the samples produced by Vendor 1 satisfied the minimum allowable values for the ultimate strength and

elongation for castings. None of the tensile bars produced by Vendor 2 satisfied the minimum property requirements for cast or wrought product.

Previously, Rafi et al. (2014) measured the tensile behavior of 17-4PH H900 produced by the laser powder bed fusion process (described as selective laser melting in that manuscript) using a commercial EOS M270 machine, notably distinct from the two commercial systems used in the present study. They reported a yield strength, ultimate tensile strength, and elongation for the H900 condition as 1126 MPa, 1457 MPa, and 12% based on 3 replicate tests. That reported yield strength is quite comparable to the characteristic value for the yield strength measured in the current study for material produced by Vendor 1 (1117 MPa), although the characteristic values for ultimate tensile strength and elongation of Vendor 1 material were somewhat lower (1239 MPa and 7%, respectively). Yadollahi et al. (2015) also measured the tensile behavior of 17-4PH H900 produced by the laser powder bed process, using a ProX™ 100 printer similar to the printer used by Vendor 2. The yield strength, ultimate strength and elongation values reported for the H900 condition were reported as 700 MPa, 950 MPa, and 3% respectively. This previously reported elongation value was comparable to those measured for the Vendor 2 material; however the strength values for Vendor 2 material were considerably higher than the previous Yadollahi study.

Figure 4. A comparison of the stress-strain response of tensile tests from commercially available wrought sheet (97 nominally identical tests) and two different commercial additive manufacturing vendors (104 nominally identical tests each). Indicated minimum properties are for castings and sheet in H900 condition.

Empirical cumulative probability distributions for the yield strength, ultimate tensile strength, and elongation were determined using a 3-parameter Weibull distribution, as shown in Figure 5. The American Society for Testing and Materials, ASTM (1995) standard C1683, defines how the Weibull distribution can be used to describe the stochastic tensile strength of brittle materials. The Weibull (1951) distribution has also been used to describe statistical variation in properties of structural metals including the ultimate tensile strength as described by Han et al. (2009); fracture toughness as described by Wallin (1984), and Williams et al. (2001); and elongation as described by Zahedi et al. (2007). While a 2-parameter variation of the Weibull distribution is most commonly used, the original distribution is a 3-parameter distribution. The accurate determination of the 3rd parameter, a threshold value below which there is zero probability of occurrence, typically requires large datasets as described by Boyce (2010); in the absence of sufficient data, this 3rd parameter is typically set to zero. For the current dataset, the Weibull distribution yielded a superior goodness-of-fit compared to a Gaussian distribution based on an Anderson-Darling metric. The 3-parameter Weibull cumulative distribution function can be described in Equation 1 as follows:

$$P = 1 - e^{\left[-\left(\frac{\alpha - \gamma}{\eta - \gamma} \right)^\beta \right]} \quad (1)$$

where the cumulative probability of failure P is a function of the variable α and three parameters: the Weibull modulus β is the shape parameter that generally describes the breadth, η is the scale parameter or characteristic value, and γ is the location parameter or threshold below which the probability is zero. The parameter estimates for each of the datasets are shown in Table 2. From the graphs and this accompanying table, several general trends are clear. Most importantly, while the wrought material demonstrated better characteristic performance in yield strength, ultimate strength, and elongation, it also benefited from substantially less scatter. Moreover, both AM vendors produced material with significantly lower *threshold* values (yield strength, ultimate strength, and elongation) compared to the wrought material.

Figure 5. Cumulative probability distributions for the (a) yield strength, (b) ultimate strength, and (c) strain at failure (elongation). The maximum likelihood estimate for a 3-parameter Weibull distribution and corresponding 95% confidence bounds are shown for each dataset.

Table 2. Weibull Distribution Parameters for tested 17-4PH H900

Property	Source	Number of Tests	Shape Parameter (Weibull Modulus)	Scale Parameter (Characteristic Strength)	Location Parameter (Threshold Value)
		N	β	$\eta + \gamma$	γ
Yield Strength, MPa	Vendor 1	104	4.4	1117	1005
	Vendor 2	104	1.9	932	884
	Wrought	97	2.5	1393	1359
Ultimate Tensile Strength, MPa	Vendor 1	104	2.2	1239	1198
	Vendor 2	104	63*	1034*	0*
	Wrought	97	1.7	1454	1443
Elongation, %	Vendor 1	104	2.4	7.0	2.0
	Vendor 2	104	2.8	2.6	0.6
	Wrought	97	78*	24.9*	0*

*These values were obtained from a 2-parameter fit because the location parameter could not be uniquely determined, as discussed in Section 4.

Fractographic Observations

Large statistical datasets offer the ability to identify atypical failure modes. Through the use of fractography, it is often possible to diagnose sources of poor fracture resistance. Figure 6 compares the fracture surface of the lowest ductility test (2% elongation) from Vendor 1 to the highest ductility test (12% elongation) from the same vendor. The 2% elongation in Figure 6a and b shows extensive lack-of-fusion voids and unmelted/partially melted particles present within some of the larger voids in the center of the fracture surface. The largest voids were $>200\text{ }\mu\text{m}$ in width and the combined voids occupied $\sim 20\%$ of the reduced cross-section. The outer edges of the fracture surface contain a pronounced shear lip. A large interlayer cliff indicated a link-up of mode-I crack planes from one print layer to another. By comparison, the fracture surface in Figure 6c and d from the highest elongation test shows only very small voids, with the largest void $\sim 25\text{ }\mu\text{m}$ and occupying $<5\%$ of the reduced cross-section.

In addition to comparing two different specimens from the same population, fractography also is valuable in comparing the failure modes between the three sources of material. Macro fractographs shown in Figure 7 compare the fracture surfaces of the Vendor 1, Vendor 2, and wrought sheet material. The fractographs in Figure 7 are presented at the same magnification. While all three samples started out with similar cross-sectional areas, the final size of the fracture surface for the wrought material (Figure 7c) is much smaller than the wrought samples due to the extensive necking ductility. Vendor 1 and 2 samples exhibited, on average, 14% and 10% reduction in area, respectively, while the wrought samples exhibited, on average, 70% reduction in area. The wrought fracture surface had a characteristic flat ‘fibrous’ central zone with large ($5\text{ }\mu\text{m}$) equiaxed dimples and numerous secondary cracks running perpendicular to the fracture surface. A symmetric shear lip was present around the entire perimeter. Example fracture surfaces from prior studies, such as those provided in the fractographic handbook from the IIT Research Institute, IITRI (1979) confirm that these features are commonly observed in 17-4PH and other similar precipitation hardened stainless steels. In contrast, the Vendor 1 tensile samples (Figure 7a and Figure 6a) exhibited large $>100\text{ }\mu\text{m}$ zones of lack-of-fusion voids in the central ‘flat fracture’ zone, and large peripheral shear lips. The Vendor 2 sample (Figure 7b) also had voids, although they were considerably smaller $<100\text{ }\mu\text{m}$ and appeared to occupy a smaller fraction ($<5\%$) of the reduced cross section. The Vendor 2 sample had a shear lip that was notably smaller than the Vendor 1 shear lip, indicating a higher strength/ lower ductility response. However, the stress-strain curves in Figure 4 show Vendor 2 to have a *lower* strength than Vendor 1, as is further discussed in Section 4. Unlike Vendor 1 where the exterior surfaces were reasonably smooth, there was extensive penetrating voids from the rough exterior surfaces in the Vendor 2 image. These notch-like penetrating voids appeared to provide the stress concentrating feature that drove premature failure.

a) Failure at 2% elongation. Sample E18

b) Close-up of lack-of-fusion porosity in sample E18

c) Failure at 12% elongation. Sample B7

d) Close-up of porosity in sample B7

Figure 6. Secondary electron SEM macrograph comparison of the fracture surface of two nominally identical Vendor 1 samples that resulted in (a and b) the lowest ductility failure at 2% strain and (c and d) the highest ductility failure at 12% strain.

Figure 7. Fractography comparing Vendor 1, Vendor 2, and Wrought material at the same magnification.

4. Discussion

This brief study on the stochastic tensile performance of additively manufactured 17-4PH raises a number of interesting questions. This Section focuses on addressing topics which are central to the theme of the high-throughput modality. Firstly, while a detailed microstructural analysis of the causes of property difference is beyond the scope of this work, we emphasize the first-order effect of properly accounting for the load-bearing cross-sectional area in tensile measurements. This awareness of the effect of surface roughness on measured properties leads to a short discussion of the expected size effects and the realization that with AM processes it is difficult to deconvolute the intrinsic material properties from the extrinsic features caused by the manufacturing process. Finally, a brief analysis and commentary is made regarding the relative immaturity of the AM process and the need to adopt efficient

stochastic methods to avoid non-conservative interpretations that could arise from limited datasets. The end of the Discussion section offers a partial listing of the potential follow-up studies that could be pursued as a result of this initial effort to unravel the stochastic reliability issues in more detail.

Effect of True Cross-Sectional Area on Strength

The engineering stress values presented in the Results section employ the standard engineering practice of measuring tensile bar dimensions with contact-based instruments such as calipers or micrometers. Specifically, for each tensile test the engineering stress σ_{eng} was calculated as a function of the applied force F , divided by the product of average width $\bar{w}$ and thickness $\bar{t}$ as measured by contact gauges:

$$\sigma_{eng} = \frac{F}{\bar{w} * \bar{t}} \quad (2)$$

While these measurement techniques work well for most engineering materials, in the current additively manufactured materials the excessive roughness relative to the sample dimensions causes a substantial discrepancy between this *apparent* stress area and the net area that carries mechanical load.

To approximately correct for the reduced cross-section due to roughness, the width and thickness were both reduced by the average peak roughness $\bar{R}_p$:

$$\sigma_{corrected} = \frac{F}{(\bar{w} - 2\bar{R}_p) * (\bar{t} - 2\bar{R}_p)} \quad (3)$$

An illustration of the difference between the apparent stress area and the net/corrected stress area is shown in Figure 8.

Figure 8. Cross sections of tensile samples from a) Vendor 1, b) Vendor 2, and c) Wrought material. The red area shows the maximum cross sectional area and the blue area shows a corrected cross sectional area using the R_p roughness values.

Figure 9 compares the tensile stress-strain behavior of the three sources of material, this time using the corrected stress area. With this corrected stress area, both AM material sources satisfy the minimum UTS requirements for cast product. However, even with the correction for net area, the strength properties were still lower than that of wrought sheet. The observation of depressed yield strength of Vendor 2 material relative to Vendor 1 and wrought sheet is expected to be at least partially attributed to the presence of retained austenite, as described in Section 2. A secondary factor is the likely possibility that the notch-like stress concentrations associated with the surface roughness triggers premature plasticity. While this first-order analysis accounted for the reduced cross-sectional area associated with surface roughness, a more detailed notch-effect study is necessary to understand the role of these stress-concentrating features on promoting early plasticity and premature failure.

Figure 9. A comparison of the stress-strain response of tensile tests from commercially available wrought sheet (97 nominally identical tests) and two different commercial additive manufacturing vendors (104 nominally identical tests each using the corrected stress area. Indicated minimum properties are for castings and sheet in H900 condition.

Size-Dependent Mechanical Behavior

The current study only explored tensile bars of a single size (4mm x 1mm x 1mm) commensurate with typical usage for component fabrication. However, both the average mechanical properties and the property distributions are expected to be size dependent – an effect that was not explored experimentally in the current study. An implicit assumption embedded in standardized tensile testing is the notion of

scale separation: the stress values and corresponding material strength values are considered to be *material properties* independent of the sample size, and therefore scalable. For this assumption to hold true, it is necessary that the tensile test interrogates a *representative volume* of material. However, in the present study, the tensile behavior is a convolution of intrinsic material properties and the superposed contributions from flaws (e.g. roughness, voids) that are of a somewhat comparable scale. One effect has already been described: the effective net section stress (corrected stress) differs substantially from the apparent stress. Moreover, the roughness and internal voids create notch-like stress concentrations that impart stress gradients over a similar dimension.

Sharon et al. (2013) and Zhao et al. (2008) have shown how ductility as measured by elongation is known to be size dependent, and hence not an intrinsic material property. First, elongation depends on the initial gauge length due to the localization of necking. Second, the onset of necking can also depend on the thickness and the size of geometric perturbations. Furthermore, Tsuchiya et al. (1998) showed that if the elongation is determined by the eventual failure at a weakest microstructural link such as a void or notch, then the elongation is also affected by the spatial distribution of these flaws, resulting in the Weibull size effect. The current study utilized a non-ASTM tensile geometry to comply with the limitations of the AM processes, e.g. to avoid large overhangs off the build plane and to allow a large number of samples in a relatively small build volume. The gauge length:width:thickness aspect ratio of the high-throughput specimen is 4:1:1- a relatively stout aspect ratio that prevents premature necking instability and encourages extensive elongation to failure. By comparison, a standard ASTM E8 compliant tensile bar made from 1-mm thick wrought sheet stock would have an aspect ratio of 50:12.5:1. The relative thinness of that geometry promotes premature necking and results in lower reported elongation to failure. Specifically, ASTM-compliant wrought sheet from this alloy/condition would be expected to have a tensile elongation of ~10% whereas the stout subsized wrought specimens in the current study exhibited a tensile elongation of ~24%. Sharon et al. (2013) described the issues regarding the size-dependent ductility and how guidelines for the ratios of length:width:thickness can minimize local necking.

Importance of Large Data Sets for a Maturing AM Process

The AM material showed marked reduction in uniaxial tensile properties compared to the wrought sheet. However, not only were average properties inferior, but the AM material exhibited substantially more variability for yield strength, ultimate strength, and elongation to failure compared to the wrought sheet. The substantial defect population and associated effect in increasing variability (e.g. 80% variability in elongation among 104 tests) in performance highlights an opportunity for further process and feedstock improvement. Moreover, it is currently ill-advised to use specification minimum values of non-AM material as the basis for component design that will be fabricated using AM. Instead it is necessary to perform a detailed stochastic property evaluation for the particular vendor and process parameters to establish representative property minimums.

The key differentiating feature of the current study was the ability to collect a relatively large statistical sample with a comparable level of effort as a few conventional tests. Small populations may be reasonable for mature, comparatively more homogenous materials that result from established thermomechanical processing routes, e.g., wrought sheet, in which defect-dominated behavior is not expected. However, small test populations may be inappropriate for materials produced by less mature processes such as laser powder bed fusion that result in less controlled defect populations. When working with small data sets, a statistical analysis would typically employ the Normal (Gaussian) distribution. For

comparison, in Figure 10 the first 5 tests are plotted using a Gaussian distribution (fits show in dashed lines) against the population of 104 tests using the Weibull 3-parameter distribution (fits show in solid lines). Only the elongation distribution is shown because it has the most obvious shift in shape, scale and threshold for the AM parts, however, this clear shift can also be seen in the yield strength and ultimate strength. No obvious shift was observed in the probability distribution for the wrought material when analyzing 5 tests versus the population. This shows that 5 tests are truly insufficient to calibrate a Weibull 3-parameter model for AM materials.

As noted in Table 2, there are two datasets that have a zero value threshold and used a 2-parameter Weibull distribution. For these two datasets, there is insufficient data to uniquely determine all three parameters. The threshold and scale parameter are strongly correlated for these datasets, and therefore it is not possible to determine them independently. For this reason, statistical goodness-of-fit metrics do not clearly show a benefit to the use of the 3-parameter Weibull over the 2-parameter Weibull. While the 2-parameter distribution is statistically defensible, the notion of a zero-valued threshold is physically unrealistic. Instead, these two datasets appear to require additional tests to uniquely determine all three parameters (or to select a better alternative distribution model altogether).

The probability distributions in Figure 5 suggest that the Weibull distribution reasonably captures the observed property distributions. However, in some cases the lower tail of experimental data seems to trail off from the expected distribution. These outlying data points in elongation and ultimate tensile strength could be caused by a bimodal distribution of properties, however even more tests would be necessary to differentiate the overlapping distributions.

Figure 10. A comparison of the statistical inference from only 5 tests compared to a larger dataset from an additional 99 tests on the same material condition. Dashed lines show fits from only 5 datapoints that result in non-conservative estimates of the lower tail of the distribution.

Future Considerations

This brief study on the stochastic tensile performance of 17-4PH opens the door to a wide range of potential questions that warrant further investigation. A few of the open questions that could be addressed in further studies are described here with respect to the AM process and with respect to a high-throughput qualification methodology.

With regard to the powder-bed fusion process, there is a need for a basic understanding of how the process variables affect the heterogeneous microstructure/defects and ultimately control the stochastic

reliability. Within the scope of the current study, the issues of lack-of-fusion porosity, surface roughness, and unexpected phase formation due to impurity uptake provide exemplars of the stochastic process-structure-property connections that might be developed with more in-depth studies. This first comparison between the nominal products produced by two independent vendors raises questions with regard to the optimal process settings for a given combination of material and AM platform. Perhaps there is also an optimization tradeoff between dimensional tolerances and material properties (e.g. lower heat input mitigates thermal distortions but causes more internal porosity). There are also questions regarding the effect of build orientation and the repeatability of the process within a build (location-to-location) or between builds. Many of these questions have been addressed in recent literature but without the efficiency benefit of high-throughput methodologies nor the statistical insight gained from large datasets. For example, in the present study the individual print layers were perpendicular to the tensile axis. While numerous prior studies have explored the difference in average properties as a function of build orientation using a small quantity of tensile samples (e.g. Thijs et al. (2013), Frazier (2014), Luecke et al. (2014), Simonelli et al. (2014), and Yadollahi et al. (2016)), less is known regarding the shift in property *variability* as a function of build orientation.

With regard to the high-throughput testing methodology, there are several avenues for further development. Firstly, the current process could be further streamlined from its current state of ~2 minutes per test. Automated robotic sample loading offers the clearest path to mitigating costly technician time. There are also important opportunities to more carefully deconvolve the contributions of intrinsic material properties from extrinsic features such as surface roughness. Secondly, it would be beneficial to extend the current tensile method to other modalities such as fracture toughness, bending, etc. It is possible to develop high-throughput methods that incorporate temperature and strain-rate dependent property measurements. Automated data analysis techniques and data mining methodologies could be developed to correlate the high-throughput mechanical property measurements with datasets from other forms of characterization such as density measurements, computed tomography scans of porosity content, x-ray analysis of phase and residual stress, etc. Finally, reduced order homogenization methods must be further developed to translate the stochastic outcomes measured in these repeated mechanical tests into stochastic predictions for component reliability.

5. Summary and Conclusions

Additive manufacturing offers the opportunity to produce geometrically complex arrays of material property coupons that are amenable to rapid, cost-effective high-throughput testing. With only modest efforts to streamline sample handling and measurement, we have demonstrated an efficient tensile method capable of many 100's of tensile tests per day. The high-throughput method was used to evaluate the full stochastic stress-strain response of a laser powder bed AM 17-4PH alloy produced by two commercial vendors, and compare the performance to commercially-available wrought sheet. With this method it is possible to not only characterize trends in *average* behavior, but also evaluate the stochastic reliability of the material. This approach provides a viable pathway to efficiently determine minimum allowable property values for AM-produced materials, which can be below the established minimum values for other processing routes such as casting. The approach highlights how differences in surface roughness, porosity, and microstructure give rise to changes in property distributions. This new technique paves the way for future extensive, but cost-efficient, parametric studies on the metallurgical and manufacturing origins of stochastic reliability.

Acknowledgements

The authors would like to thank John Laing for assistance in the mechanical testing laboratory, Bonnie McKenzie and Amy Allen for scanning electron microscopy support, and Alice Kilgo for metallographic preparation. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

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