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Mechanical Response of Additively Manufactured Stainless Steel 304L Across a Wide Range of Loading Conditions

David P. Adams
Benjamin Reedlunn
Michael C. Maguire
Bo Song
Jay Carroll
Joe E. Bishop
Jack L. Wise
Alice Kilgo
Todd A. Palmer
Don W. Brown
Bjorn Clausen

Prepared by
Sandia National Laboratories, Mail Stop 0959
Albuquerque, New Mexico 87185 and Livermore, California 94550

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David P. Adams, Benjamin Reedlunn, Mike C. Maguire*, Bo Song, Jay Carroll, Joe E. Bishop,
Jack L. Wise, Alice Kilgo
Sandia National Laboratories
Albuquerque, New Mexico, 87185
*Livermore, California, 94551

Todd A. Palmer
Penn State University
Department of Materials Science and Engineering
University Park, Pennsylvania, 16802

Don W. Brown, Bjorn Clausen
Los Alamos National Laboratory
Los Alamos, NM, 87545

Abstract

The mechanical response of additively manufactured (AM) stainless steel 304L has been investigated across a broad range of loading conditions, covering 11 decades of strain rate, and compared with the behaviors of traditional ingot-derived (wrought) material. In general, the AM material exhibits a greater strength and reduced ductility compared with the baseline wrought form. These differences are consistently found from quasi-static and high strain rate tests. A detailed investigation of the microstructure, the defect structure, the phase, and the composition of both forms reveals differences that may contribute to the differing mechanical behaviors. Compared with the baseline wrought material, dense AM stainless steel 304L has a more complex grain structure with substantial sub-structure, a fine dispersion of ferrite, increased dislocation density, oxide dispersions and larger amounts of nitrogen. *In-situ* neutron diffraction studies conducted during quasi-static loading suggest that the increased strength of AM material is due to its initially greater dislocation density. The flow strength of both forms is correlated with dislocation density through a square root dependence akin to a Taylor-like relationship. Neutron diffraction measurements of lattice strains also correlate with a crystal plasticity finite element simulations of the tensile test. Other simulations predict a significant degree of elastic and plastic

anisotropy due to crystallographic texture. Hopkinson tests at higher strain rates ($\dot{\epsilon} = 500$ and 2500 s^{-1}) also show a greater strength for AM stainless steel 304L; although, the differences compared with wrought are reduced at higher strain rates. Gas gun impact tests, including reverse ballistic, forward ballistic and spall tests, consistently reveal a larger dynamic strength in the AM material. The Hugoniot Elastic Limit (HEL) of AM SS 304L exceeds that of wrought material although considerable variability is observed with the AM material. Forward ballistic testing demonstrates spall strengths of AM material (3.27 – 3.91 GPa) that exceed that of the wrought material (2.63 – 2.88 GPa). The Hugoniot equation-of-state for AM samples matches archived data for this metal alloy.

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NOMENCLATURE

α	Thermal Diffusivity
a	Major Radius, Lattice Parameter
a_0	Reference Lattice Parameter
A	Fully Austenitic
AF	Primary austenite followed by eutectic ferrite/austenite
Al	Aluminum
AM	Additive Manufacturing
As	Arsenic
at. %	Atomic Percent
b	Minor Radius
B	Boron
bal.	Balance
Bi	Bismuth
C	Carbon
CAD	Computer Aided Design
Cl	Chlorine
cm	Centimeter
Co	Cobalt
C_p	Heat Capacity
Cr	Chromium
Cs	Cesium
Cu	Copper
CW	Continuous Wave
ρ	Density
ρ_0	Initial Density
d	Interatomic Spacing
D	Beam Width (diameter), Deuterium
DC	Direct Current
DED	Directed Energy Deposition
DIC	Differential Interference Contrast
DICE	Dynamic Integrated Compression Experimental
DLPA	Diffraction Line Profile Analysis
DLF	Directed Light Fabrication
DMD	Direct Metal Deposition
DMLS	Direct Metal Laser Sintering
DOE	Department of Energy
DSC	Differential Scanning Calorimetry
$^{\circ}\text{C}$	Degrees Celsius
ΔH	Energy
ΔH_{fus}	Enthalpy of Fusion
ΔH_0	Heat of Formation
ΔT_m	Melting Range
E	Energy, Modulus
EBSD	Electron Backscatter Diffraction

ECR	Electrical Contact Resistance
EDX, EDS	Energy Dispersive X-ray Spectroscopy
EELS	Electron Energy Loss Spectroscopy
ϵ_i	Phase strain
E_p	Energy per Pulse
EPSC	Elasto-Plastic Self-Consistent
ESD	Estimated Standard Deviation
Er	Erbium
E_r	Reduced Modulus
eV	Electronvolt
F	Fluence
F	Primary Ferrite
FA	Primary ferrite followed by eutectic/peritectic austenite/ferrite
F_{acc}	Accumulated Fluence
fcc	Face centered cubic
Fe	Iron
FIB	Focused Ion Beam
F_p	Single Pulse Fluence
FWHM	Full Width at Half Maximum
Ga	Gallium
GIXRD	Grazing Incidence X-ray Diffraction
GPa	GigaPascal
GSAS	General Structural Analysis Software
h	Beam Overlap
H	Hydrogen
HAZ	Heat Affected Zone
HEL	Hugoniot Elastic Limit
Hr	Hour
Hz	Hertz
I	Current
ICP-MS	Inductive Coupled Plasma Mass Spectroscopy
In	Indium
IPF	Inverse Pole Figure
ipm	Inches per Minute
IR	Infrared
k	Thermal Conductivity, Dielectric Constant
K	Kelvin, Potassium
kHz	KiloHertz
km	Kilometer
κ_s	Thermal Conductivity
kV	Kilovolt
kW	Kilowatt
λ	Wavelength
L	Liter
La	Lanthanum
LANL	Los Alamos National Laboratory

LANSCCE	Lujan Center at the Los Alamos Neutron Science Center
LASL	Los Alamos Scientific Laboratory
LENS TM	Laser Engineered Net Shaping
Li	Lithium
LLC	Limited Liability Company
LMD	Laser Metal Deposition
LMF	Laser Metal Fusion
L _s	Line Spacing
m	Meter
Mg	Magnesium
Mn	Manganese
mg	Milligram
min.	Minute
mm	Millimeter
Mo	Molybdenum
MPa	Megapascal
MRD	Multiples of Random Distribution
μm	Micrometer
μs	Microsecond
ms	Millisecond
MSE	Mean Squared Error
n	Refractive Index
N	Nitrogen
Nb	Niobium
Nd	Neodymium
Ni	Nickel
NIR	Near Infrared
nm	Nanometer
N _p	Number of Pulses
ns	Nanosecond
O	Oxygen
OBJ	Objective
OES	Optical Emission Spectroscopy
ν	Repetition Rate, Poisson's ratio
ζ	Rayleigh Length
P	Power, Phosphorous
P _{avg}	Average Power
PB	Powder Bed
PBF	Powder Bed Fusion
PDV	Photon Doppler Velocimetry
PMMA	Poly Methyl Methacrylate
ppm	Part per Million
ps	Picosecond
PSD	Power Spectral Density
R	Reflectance, Reflectivity
RMS	Root Mean Square

s	Second, Speed
S	Sulfur, Peak Variance
S _a	Surface Roughness
S _q	RMS Surface Roughness
Sc	Scandium
SEM	Scanning Electron Microscopy
Si	Silicon
σ_i	Phase stress
SLM	Selective Laser Melting
SLS	Selective Laser Sintering
SMARTS	Spectrometer for MAterials Research at Temperature and Stress
SNL	Sandia National Laboratories
sr	Steradian
SS	Stainless Steel
STEM	Scanning Transmission Electron Microscope
3-D	Three-Dimensional
t	Time
T	Temperature
T	Transmittance
T _o	Initial Temperature
TEM	Transmission Electron Microscopy
T _{liquidus}	Liquidus Temperature
TOF	Time of Flight
T _{solidus}	Solidus Temperature
τ_l	Pulse Length
θ_m	Melting Temperature
UHP	Ultra High Purity
u _p	Particle Velocity
U _s	Linear Shock Velocity
UTS	Ultimate Tensile Strength
V	Volume, Volt, Vanadium
VISAR	Velocity Interferometer System for Any Reflector
W	Watt, Tungsten
WDS	Wavelength Dispersive Spectroscopy
ω_o	Beam Waist, Radius
wt. %	Weight Percent
X	Primary laser scan direction, An additively manufactured bar that has a long dimension aligned with the primary laser scan direction
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
Y	Primary process direction, An additively manufactured bar that has a long dimension aligned perpendicular to the build direction (Z) and the primary laser scan direction
Z	Primary build direction, An additively manufactured bar that has a long dimension aligned with the build direction (parallel to the downward pointing laser vector)
Zr	Zirconium

1. BRIEF INTRODUCTION TO ADDITIVE MANUFACTURING OF METALS AND ALLOYS

Additive Manufacturing (AM) refers to a variety of material fabrication techniques that synthesize a three-dimensional (3-D) object by building successive layers via computer control.[1-10] Also referred to as 3-D printing, this technology holds promise for realizing complex, near-final shape structures without the need for subtractive processes such as machining or etching. AM is currently recognized as a useful approach for rapid prototyping and is considered by many to be an emerging method for metal component production that covers the micro, miniature, and macroscopic dimensional scales. In addition to building whole objects, additive methods are being developed to repair objects in the field. Repaired objects may consist of materials made by traditional manufacturing processes or include substances made originally by AM. There is renewed interest in AM, because advances made by researchers and instrument manufacturers over the past decade have shown feasibility for increased design flexibility, reduced time to market, lower manufacturing cost, and minimal material waste.

Included in the suite of AM techniques that fabricate metal and alloy structures are several beam-assisted methods. These methods generally involve directing an intense, focused beam of coherent light or electrons onto the surface of an object (or substrate at start) for heating and fusing supplied metal. In some processes, fine metallic powder is fed continuously to the object's surface during irradiation. Different processes heat metal powder only after it has been placed on the surface of the evolving metal object. In the latter examples, powder is layered to a prescribed, uniform thickness prior to irradiation in order to promote fusion to an underlying structure when heated. Alternatively, wire may be fed to access higher build rates with improved process control. AM involves moving either the synthesized object or the laser beam under computer control to gain access to desired geometries. Thus, the scan pattern may be the same in successive layers or different depending on the object's shape. Preferred scan paths and speeds may also be chosen according to strategies associated with achieving optimal surface finish, increasing density, minimizing residual stress, or attaining other key properties.[11-15]

The two most commonly used AM processes are Directed Energy Deposition (DED) [16] and Powder Bed Fusion (PBF).[17,18] Note: other names are commonly used when referring to these methods. Directed Energy Deposition includes Laser Engineered Net Shaping (LENSTM) [19,20], Laser Metal Deposition (LMD) [21], Electron Beam Direct Manufacturing, Laser Assisted Direct Metal Deposition [22], Directed Light Fabrication (DLF) and Direct Metal Deposition (DMD) [23,24]. Powder Bed Fusion encompasses Direct Metal Laser Sintering (DMLS) [25], Selective Laser Melting (SLM) [26], Selective Laser Sintering (SLS) [27], and Laser Metal Fusion (LMF).[28]

Traditional, laser-based, directed energy deposition methods, such as LENSTM, utilize a power of ~500 W or less to fuse powder that is fed to the growth surface.[3] A survey of commercial instruments shows that Continuous Wave (CW) lasers are most commonly utilized for this approach. CW is likely preferred, because it provides a smoothly propagating melt front that avoids keyhole mode operation and point-to-point variations in structure and composition that could arise if a pulsed laser beam was utilized. More recently developed DED processes have implemented larger power, in excess of 1 kW. Presumably this trend is motivated by increased build rates and possibly higher material density. Optomec Inc. reveals the use of fiber lasers that provide access to much higher powers (up to 4 kW).[29] Interestingly, laboratory research groups have begun to utilize high peak power, femtosecond pulsed lasers in conjunction with CW lasers to process refractory metals by AM.[31]

Commercial, laser-based, DED systems process material inside a sealed chamber purged with high-purity argon. Equipped with oxygen sensors, this approach helps ensure a pristine environment for deposition that minimizes metal oxidation. Metal powder feedstock is delivered to an object's surface via feed tubes that point to a region irradiated by the laser beam as shown in Figure 1. A Computer-Aided-Design (CAD) solid model is referenced when moving the stage or the laser and feed nozzle. The part is built layer-by-layer according to the reticulated layer slices identified by the control software. More advanced systems can reprocess the powder that does not fuse to the object by recirculating this back through the powder handling network.[30]

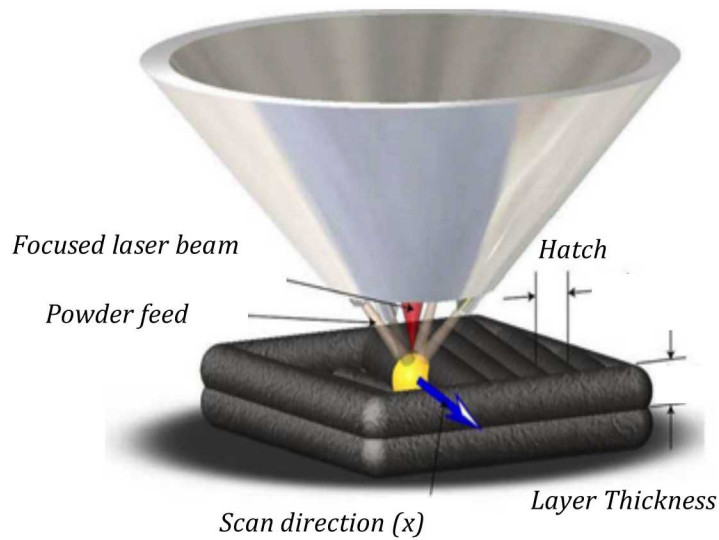


Figure 1. Depiction of Laser Engineered Net Shaping (LENS™).
Reference D. Gill, Sandia National Laboratories.

Laser based powder bed techniques are different although the general approach of utilizing an intense focused beam for heating and fusing powder feedstock is similar. Different than LENS™, powder is distributed as a ~20 to 100 μm thick layer prior to laser scanning.[32,33] The material is layered uniformly so that laser-induced heating fuses powder to the underlying object's surface. Traditional PBF systems use a single focused laser beam for melting and fusing powder. Emerging systems implement multiple laser beams for parallel processing.[34]

A review of the literature on metal additive processes demonstrates that melting of feedstock is common to laser-based DED and PBF processes, although usually differing in particle size distribution as DED processes are typically more tolerant of larger particles. The melt pool is often described as 'continuously propagating' when the beam is scanned across the growth surface. In reality, AM may involve partial melting wherein the core of a particle remains in a solid phase.[35] To this day, optimized methods for fabrication are being researched for different materials. Understanding the physical state of particles and their trajectories for different processes is a difficult task that remains an important topic of study. Other dynamic material behaviors occurring during fabrication continue to challenge the development of PBF and DED processes. For example, it was recognized more than a decade ago that flow instabilities develop in a melt pool during powder-based scanned laser processes and these can lead to balling or non-uniform layer deposition.[36,37] Additional studies reveal that melt pools

on the surface of some fabricated materials [38] may be unstable. Despite these challenges, AM processes that utilize laser-induced melting continue to improve. These processes provide access to various complex feature geometries such as solids having embedded voids/channels[39], lattice structures, and thin walled components (see examples in Figure 2).

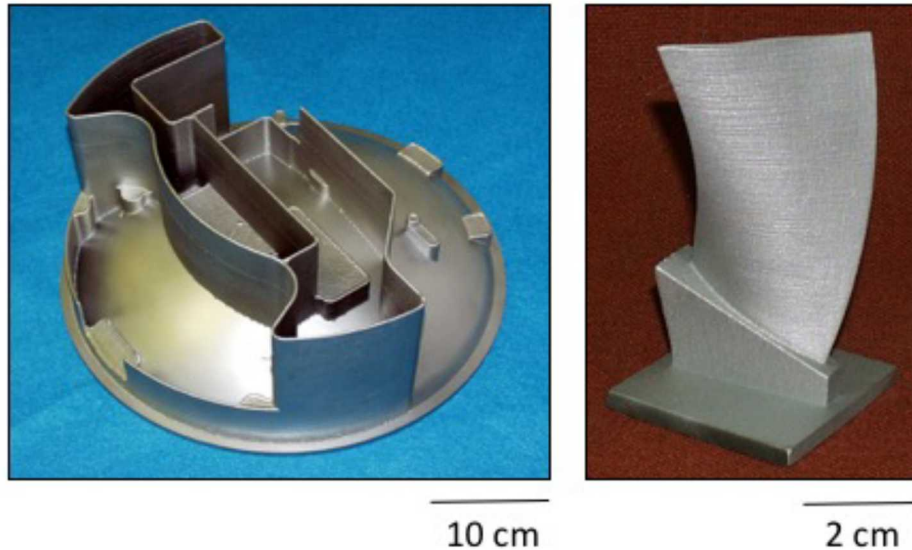


Figure 2. Example thin walled, metal alloy structures made by LENS™.

Electron beam-assisted deposition processes utilize a different form of energy to stimulate melting and fusion. Utilizing electrons, these processes require more rigorous control of the process chamber environment. Much like in an electron microscope, a vacuum chamber is required to allow the open space propagation of electrons over sizeable distance necessary to reach an object's surface. Systems operate in the high vacuum regime with base pressures of $\sim 10^{-4}$ Torr. This makes the method amenable to metals that have a strong affinity for oxygen, such as titanium.

Electron beam-assisted fabrication methods take on different forms. For example, Sciaky Co., building on their expertise in electron beam welding, has developed a technology they refer to as Electron Beam Direct Manufacturing (EBDM).[40] It uses a wire-fed process to achieve rapid build rates. Powder-based, electron beam fabrication methods have also been developed and are available with commercial systems. ARCAM irradiates pre-heated layers of powder with an intense electron beam in a vacuum to build up material layer-by-layer.[41]. It is claimed that by keeping the build chamber and powder relatively hot, between 700 and 1000°C, that the residual

stress in a fabricated object is minimized. Presumably this occurs because of the reduced range of temperature occurring during a build process. Build rates are impressive. Rates as high as 3.3 L/hr have been documented, although powder recycling of the residual powder cake is an issue.

A review of the published literature on laser- and electron-based AM processes reveals a variety of fabricated metals and alloys. This includes Ti-6Al-4V, commercially-pure (CP) Ti, nickel based alloys, tool steel, CoCr superalloy, TiAl, AlSi10Mg, AlSi9Cu3, AlMgScZr, Nb, stainless steel 316, tool steel, Invar 36, Inconel 625, Cu, and W. A summary of these and other materials is included in Table 1 along with pertinent references.

Table 1. Metals and alloys fabricated by different AM processes (includes only laser-based and electron-beam based fusion methods).

Material	Method(s)	Reference(s)
Ti-6Al-4V	DED (laser, electron), PB (laser, electron)	42,43
CP Ti	PB (laser, electron)	44,45
CoCr alloy	PB (laser, electron)	46,47
TiAl	DED (electron), PB (electron)	48,49
Stainless steel 316	DED (laser), PB (laser, electron)	50,51
Nb	DED (electron)	52,53
AlMgScZr	PB (laser)	54
AlSi9Cu3	PB (laser)	55
AlSi10Mg	PB (laser)	56,57
Al 6061	PB (laser)	58,59
Inconel 718	DED (laser), PB (laser, electron)	60,61
Invar 36	DED (laser)	62,63
Inconel 625	PB (laser)	64,65
Cu	DED (electron)	66,67
W	PB (laser)	68,69
AISI 420 steel	PB (laser)	70,71
AISI 4140 steel	PB (laser)	72
H-13 steel	DED (laser)	73,74
AISI 4340 steel	DED (laser)	75,76
Rene 142	DED (electron)	77
Stainless steel 304L	DED (laser)	78

2. PROPOSED RESEARCH

AM is currently of interest because of its potential to build unique objects that have enhanced functionality over those made by traditional manufacturing methods. In particular, AM provides access to complex geometries that are difficult to machine as well as compositionally-graded material designs. Despite recent progress with process control, a great deal of materials research is required to qualify AM materials for end use. In particular, the materials produced by these emerging methods remain poorly understood, both in terms of their structure, and properties and performance. This lack of understanding is a key barrier to their implementation into components and systems.

This LDRD proposed to investigate the mechanical response of a single AM material (stainless steel 304L) across a broad range of loading rates and test conditions in order to provide an improved understanding of how microstructure impacts mechanical response. A literature search completed prior to proposal submission revealed that no group had previously investigated the high strain rate (dynamic) response of AM metals or alloys. It is possible that industrial leaders who have invested in AM for over a decade may have knowledge of an AM material's mechanical response in this regime, but the information is not publicly available. Figure 3 includes a chart from Meyers' book *Dynamic Behavior of Materials* [79] that references the different regimes of mechanical testing. The original chart has been marked on the left with dotted, red lines to indicate specific strain rates used in our tests. The quasi-static regime is included as part of our study of AM SS 304L, and there are five rates of testing chosen in this regime. In addition, Hopkinson bar and gas gun instruments provide access to larger rates, up to $\sim 10^6 \text{ s}^{-1}$. In all, the study covers an 11-decade range of strain rates.

As described in a recent review article by Lewandowski and Seifi [1], the vast majority of work on metal AM has focused on the quasi-static mechanical response. Various research groups have attempted to correlate quasi-static mechanical response with an object's microstructure and process parameters. In advance of our study, it was understood that a resolidification microstructure, akin to that of laser-welded material, develops as a result of beam scanning and

metal fusing. This complex microstructure is often referenced when explaining the varied mechanical response of AM material. Lack of fusion defects and voids along layer interfaces must be avoided if striving to understand the intrinsic role of grain structure and composition.

Our team chose to use a high average power, laser-based directed energy deposition process for additive manufacturing. Kilowatt (kW) powers were utilized instead of the ~500 W powers used in the traditional LENSTM approach, as the former provides several advantages. First, the higher power (and increased powder feed rates) provided opportunity for high rate fabrication. More than one hundred macroscopic test samples were needed for study; thus there was a need for many cubic cm of AM material. In addition, the higher laser powers were chosen because these produce dense deposits. This helped avoid the highly variable effects of voids and lack of fusion defects that has plagued some past studies. The high power additive process used for study was developed by Professor Todd Palmer of Penn State University.

Our approach contrasts the response of AM SS 304L with that of wrought (ingot derived) stainless steel 304L. Comparisons to wrought were considered vital to a study that seeks to understand microstructural effects. The two forms were expected to have different grain structure and crystallographic texture. Different than the solidification microstructure of AM SS 304L, wrought stainless steel has an equiaxed, micrometer-sized grain structure with little preferred crystallographic texture. Thus, compared with the wrought, it was anticipated that the AM material should exhibit considerable variability in mechanical response depending on the orientation chosen for testing. Wrought SS304L was also included, because there is a wealth of material test data for this form that is the basis for comparison. Archived mechanical test data for wrought SS 304L includes high strain rate properties such as the Hugoniot Equation-of-State. In essence, our tests of wrought, ingot-derived material helped verify accurate experimental test methods and procedures.

Our LDRD combined experiments with predictive model simulations to better understand the role of microstructure on mechanical performance. A key science question for predicting the mechanical response of additively manufactured materials is the accuracy of homogenization theory for macroscale quantities of interest. Homogenization theory relies on the assumption of

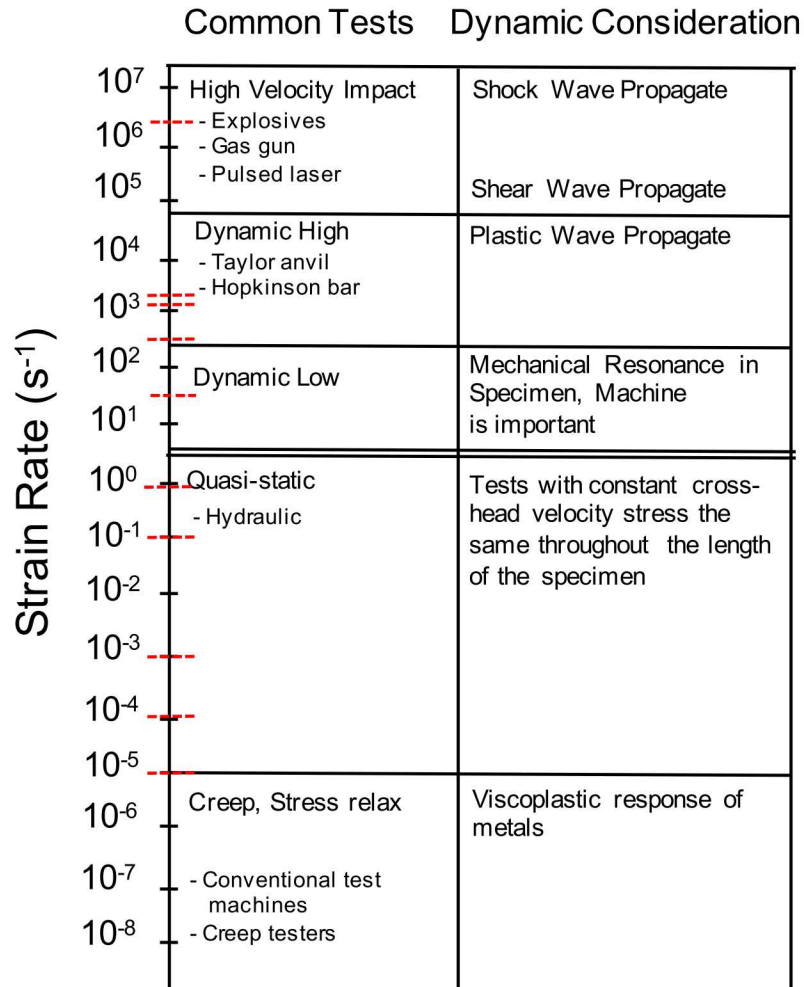


Figure 3. Chart from Dynamic Behavior of Materials summarizing strain rates accessed during experiments, methods for accessing these and dynamic considerations.

scale separation: the microstructural length scale must be much smaller than the macrostructural length scale. For AM metal alloys, the microstructure can contain relatively large, elongated grains, on the order of millimeters, that may violate the assumption of scale separation, depending upon the component size. To address whether a homogenization approach is appropriate, our team pursued a multiscale modeling paradigm consisting of several thrusts for predicting mechanical response of an additively manufactured structure.[80] The first utilized Sandia's newly-developed Direct Numerical Simulations (DNS)[81] approach that uses detailed microstructural knowledge, physics-rich crystal-plasticity models[82], and massively-parallel finite-element software (Sierra) and computer resources, to model as-manufactured

microstructures embedded within macroscale structures. Second, a conventional modeling approach utilized a macroscale viscoplastic material mode. The latter approach smears out microstructural variability and assumes that a material is isotropic. This approach is significantly less computationally intensive than either the DNS or other approaches including Computational Homogenization with Fine-Scale Recovery. Experimental tests were used to verify model predictions and determine the appropriateness of a homogenization approach.

The remainder of the report is organized as follows. Chapter 3 reviews the Additive Manufacturing processes used for the fabrication of SS 304L in our study. Following this, Chapter 4 clarifies the directional nomenclature that is referenced throughout the manuscript when describing produced bars, test directions, etc. Chapter 5 describes the microstructure, composition, phase, crystallographic texture, density, modulus and other characteristics of as-deposited material. In addition, this chapter summarizes the characteristics of wrought material used for comparison. Chapter 6 follows with a brief description of how material was machined into final shapes for mechanical testing. To clarify, our LDRD did not fabricate final test geometries using AM. Instead, AM was used to build bars from which test volumes were extracted and machined. This helped to avoid the final AM surface morphology and its effects on mechanical performance. Chapters 7, 8, 9, and 10 describe the mechanical responses of AM and wrought SS 304L at progressively higher load rates. These chapters summarize the experimental test procedures and equipment, sample geometries and discuss results. Chapter 11 introduces the modeling methods that have been used for predicting mechanical response. Predictive model simulations focus on the quasi-static response of stainless steel 304L. No attempts to model mechanical response in the dynamic regime or the high velocity impact regime were attempted as part of this study. Chapter 11 also describes model simulations of a complex, axial-torsion test geometry along with a summary of predicted results. This discussion contrasts results drawn from a homogenization approach with those derived from DNS that incorporate representative microstructures and employ crystal-plasticity models. Validation tests that confirm model predictions are included at the end of this chapter. These serve to describe, for the first time, the torsional strength of an AM metal alloy.

3. DESCRIPTION OF ADDITIVE PROCESS USED FOR THIS RESEARCH

3.1. Description of Apparatus used for Additive Manufacturing

Stainless steel 304L bars were deposited using a custom, additive manufacturing system located at Penn State University.[83] The chamber used to house samples and supplied powder was purged with ultra-high purity argon during directed energy deposition. Attempts to maintain oxygen levels below 110 ppm using this approach were successful. The chamber atmosphere was controlled by an MBraun Model MB 200G gas purifier, and the gaseous oxygen levels were measured using a General Electric CGA 351 zirconium oxide Oxygen Analyzer. CW laser light was delivered from an IPG Photonics® YLR-12000-L ytterbium fiber laser with a wavelength ranging from 1070 to 1100 nm. The light was delivered to the workpiece through a 600 μm diameter fiber into a copper-cooled reflective optics system. The collimator had a diameter of 49.5 mm and a focal length of 125 mm, while the focusing optics had the same diameter and a focal length of 600 mm. Reflective optics were utilized to avoid thermal lensing effects observed in the use of high power transmissive optics in other systems.[84] Powder was delivered using a Powder Feed Dynamics Mark XV Precision Powder Feeder to a custom-designed four nozzle powder delivery system.

Two processes were used to deposit dense bars. An incident laser power of 3.8 kW was utilized in a first process with powder flow rates of 23 g/min. This process implemented a parallel scan geometry meaning that the scan paths were identical and parallel in subsequent layers. Table speed was set at ~ 63.5 cm/minute (25 ipm), and the hatch of adjacent scan lines within a given layer was 1.925 mm. The resultant build rate was 1.27 mm/layer (0.050 inches/layer). The second process used a power of 2.0 kW and a crosshatch scan approach, which involved 90 degree rotated scan directions in alternating layers. Powder flow rates were 18 g/min, table speed was fixed at ~ 508 mm/min (20 ipm) and the hatch of adjacent scan lines was set at 1.925 mm. The latter process was characterized by a build rate of ~ 0.89 mm/layer (0.035 inches/layer). Deposition generally involved placing a substrate at a location that was approximately 10 mm

from the nozzle exits. This corresponds to the focus point of the powder flow. However, at this location, the laser beam is in a defocused position and has a measured beam diameter ($1/e^2$) of 4 mm. Previous characterization of the beam at this location using a PRIMES® Focus Monitor confirmed a Gaussian energy distribution and the aforementioned diameter.

The majority of bars made by these processes were rectangular having dimensions of 2.5 x 2.5 x 10 cm. Two additional, square bars were made for gas gun impact tests as described later in this report. These latter two had dimensions of 10 x 10 x 2.5 cm.

3.2. Details of Powder used for Fabrication of SS 304L

The powder used for AM was Micro-melt 304L (44 – 105 μm) from Carpenter Powder Products. The manufacturer’s inspection certificate provides the elemental composition which is reproduced in Table 2. Of note, the powder is nitrogen-atomized. Powder size distribution was as shown in Figure 4.

The measured compositions of as-received powder and AM SS304L bars produced by the Penn State apparatus (2.0 and 3.8 kW processes) are documented in Chapter 4 with mention of slight differences noted from study.

Table 2. Chemical composition of SS 304L powder used for additive manufacturing (as certified by Carpenter Powder Products)

Elemental Constituent	Content (wt. %)
C	0.013
Si	0.55
Mn	1.38
P	0.009
S	0.008
Cr	18.9
Ni	9.9
Fe	remainder

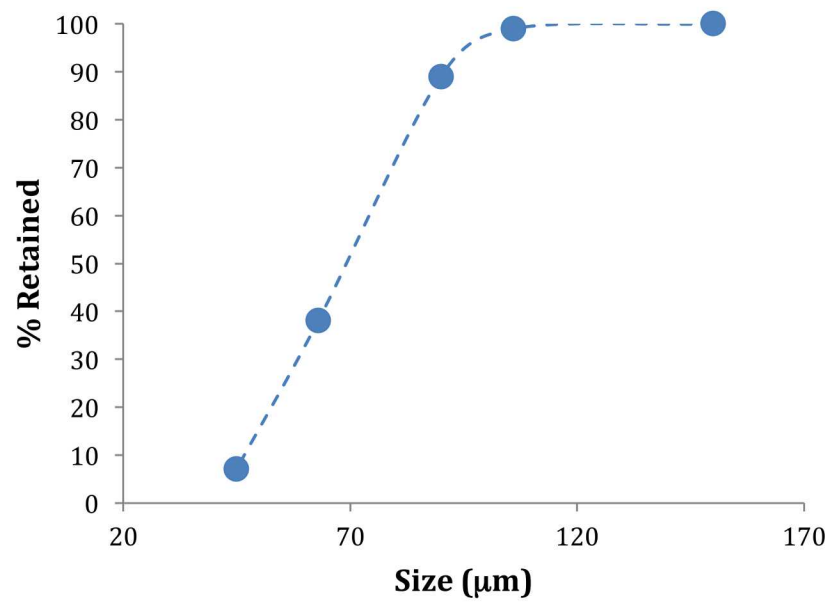


Figure 4. Cumulative powder size distribution for Carpenter 304L powder.

4. DIRECTIONAL NOMENCLATURE OF ADDITIVELY MANUFACTURED AND WROUGHT STAINLESS STEEL

4.1 Nomenclature describing additively manufactured stainless steel

The additively manufactured bars are described using an X - Y - Z notation that is largely consistent with the ASTM standard.[85] These three letters reference the key directions associated with additive processing. Specifically, Z designates the build direction which is parallel with (but opposite to) the downward-directed laser vector. X and Y are the orthogonal in-plane stage scan directions. Both lie perpendicular to Z, thus forming a Cartesian coordinate system. Our report does not generally differentiate between + and – X directions, etc.

One of the three letters is used when referring to a given bar. The specified letter designates the axis that is parallel to the longest overall bar dimension. In Figure 5, the taller block is recognized as a Z bar because it has a longer dimension aligned with the Z direction. X and Y bars are also drawn in this figure having a long dimension that is 90 degrees from Z. Our team has chosen to differentiate X versus Y bars as follows.

X bars made using the 3.8 kW AM process have a long dimension aligned with the single, in-plane scan direction utilized for fabrication. This is slightly different than the ASTM specification which describes the X direction as being aligned with the plane associated with the front face of the AM system. Conversely, Y bars made by the 3.8 kW process have a long axis that is perpendicular to both the Z direction and the laser scan direction (X). Example Y bars made using a 3.8 kW (parallel line) process are shown in Figure 6, wherein the scan lines are evident.

A somewhat similar nomenclature is used to describe the bars made using the 2.0 kW process which alternates in-plane scan direction in subsequent layers. Again, a Z bar (sometimes referred to as a tower) has a longer dimension that is aligned with the Z direction. This is depicted on the left side of Figure 7, and shown with a photograph in Figure 8. An additional bar, referred to as X, is depicted in Figure 7. This has a long dimension that is aligned with one

of the two in-plane scan directions used for this AM process. To be clear, the alternating scan direction used for 2.0 kW processes means that these bars are made by scanning in both X and Y. Thus, we may equivalently refer to these as X or Y bars. Additional pictures of these bars can be found in Appendix 13.1.

Two additional, 10 x 10 x 2.5 cm, square bars were specifically built for gas gun impact test samples and these are not described using an X, Y or Z bar notation. Both were built using a parallel scan (3.8 kW) process and the scan direction is still X (build direction is Z). However, because there is no long dimension, a letter notation is avoided for these bars. An example square bar is shown in Figure 9.

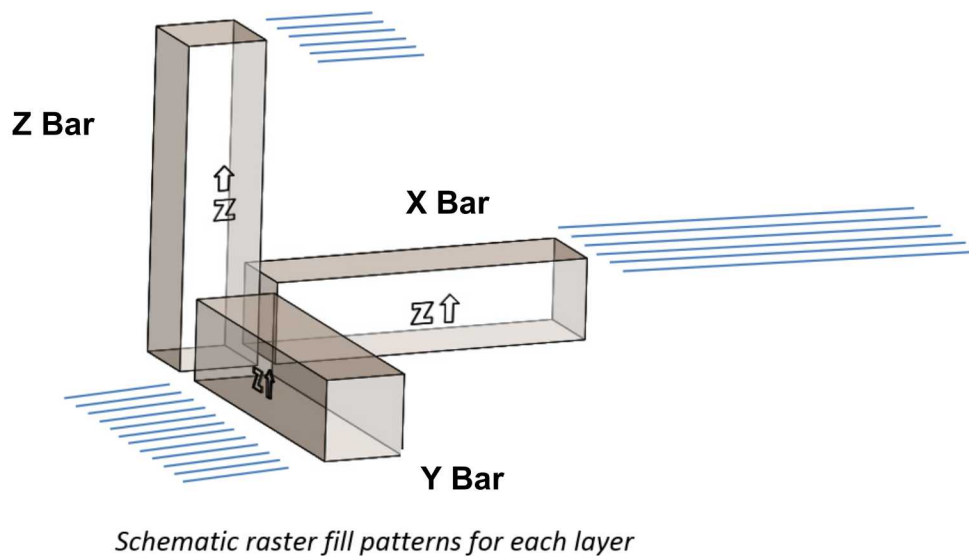


Figure 5. Depiction of X, Y and Z bars and parallel scan approach used for their fabrication. This was the procedure used in the fabrication of SS 304L when using $P_{avg} = 3.8$ kW. X is designated as the laser scan direction.

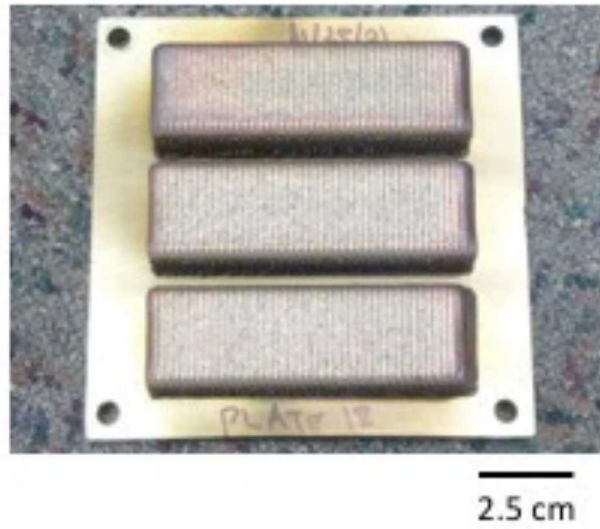
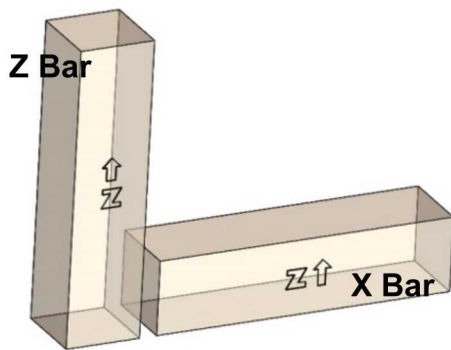
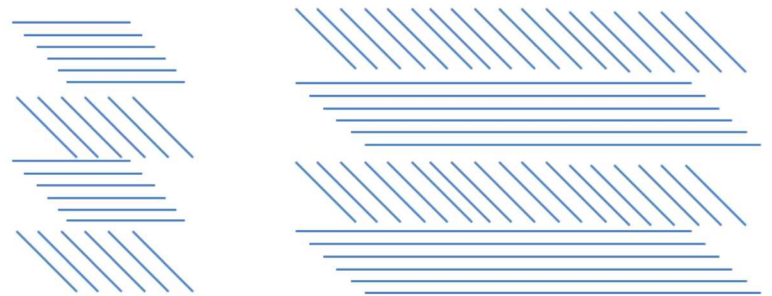


Figure 6. Photograph of Y bars produced using $P_{\text{avg}} = 3.8 \text{ kW}$.



Sample Geometry



Schematic raster fill patterns/

Figure 7. Depiction of X and Z bars and the cross-hatch scan approach used for their fabrication. This procedure used an $P_{\text{avg}} = 2.0 \text{ kW}$.

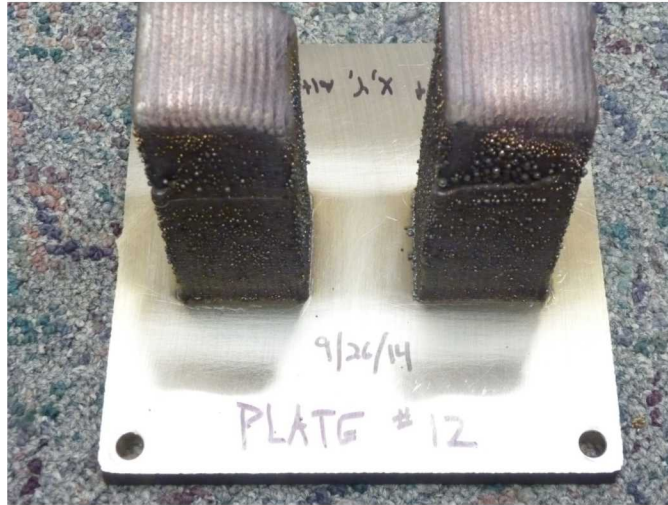


Figure 8. Photograph of Z bars produced using a $P_{avg} = 2.0$ kW process.



Figure 9. Photograph of square bar made for producing high impact gas gun test samples. The second sample is shown in Appendix 13.2.

A second letter (X, Y, or Z) is used when referring to a sectioned face of a given bar. Specifically, the second letter describes the normal direction of the created face. Thus, a given bar, say X, can be sectioned along X, Y, Z, or, in special cases, misaligned orientations. Figure 10 below shows three examples for an X bar. The semi-transparent plane cutting through each depicted X bar is used to define what is meant by a given face. The words “slice” or “cut” are used also when referring to faces. Notice, in the depictions the final laser scan direction can be

determined by the parallel hill-and-valley morphology present on the top surface of the AM object. Figures 6, 8, and 9 show said morphology on produced bars.

Looking at Figure 10 in more detail and knowing that the X bar was built up layer by layer, one can imagine the relevant information revealed when viewing different faces. A look at the X face in plan view will show the AM material through thickness. Looking from side to side of the X face one should expect to see microstructural artifacts associated with individual scan lines through multiple passes. The Y face would reveal the through-thickness structure that is associated with overlaid laser scan lines (if using a 3.8 kW, parallel scan approach). A look at a single Z face would not reveal through thickness information. Nevertheless, it would provide a top down view of structure and morphology derived from laser scanning along lines.

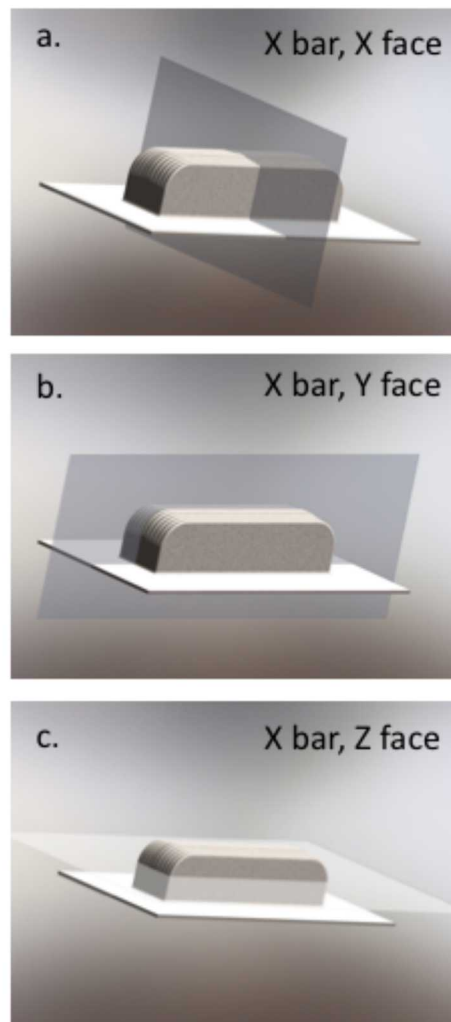


Figure 10. Depictions of X bars. Semi-transparent planes identify a.) an X face, b.) a Y face, and c.) a Z face.

The same lettered nomenclature is used to describe the mechanical test sample orientations. One of the three letters is used when referring to the axis of loading. This includes load axes in compression and tension. For example, a tensile sample could be machined from the X bar depicted in Figure 10(a). If the loading axis is aligned with the X direction, then this axis would be designated as X. A few samples use two letters when referring to the loading direction. These particular samples were intentionally machined off the primary axes of build. An example of the latter is a test sample machined from an X bar with a load axis of XY (45 degrees from both X and Y).

The letter nomenclature is also used to describe the axis of loading for gas gun impact experiments. The square bars were machined into discs for subsequent gas gun testing. The directional nomenclature for these again references a primary build direction (X, Y, or Z) when describing the axis of loading which was a direction normal to the produced face.

Appendix 13.3. summarizes other details involved with additive manufacturing. Bars were built onto plates which are identified with a unique number. Bar types are specified using the aforementioned X/Y/Z nomenclature for each plate listed in this appendix. Additional information includes the table speed, the powder feed rate, average laser power, beam spot size (measured at the growth surface of the AM part), and the layer thicknesses per pass.

4.2 Nomenclature describing wrought stainless steel

A different nomenclature is used to describe the orientation of machined test samples or sectioned faces of wrought stainless steel. All wrought samples were derived from a cylindrical, wrought billet of stainless steel. Hence, we use the cylindrical axis of the original form when referencing sample and face orientation.

Longitudinal and transverse orientations are clarified in Figure 11. In this figure two depictions of the wrought billet show the locations of smaller cylinders that are extracted and machined to produce mechanical test samples. The depiction on the left shows longitudinal test samples prior to their removal. By longitudinal, it is meant that the load axis of these test samples is parallel to

the axis of the wrought billet. Longitudinal test samples extracted from near the edge of the billet are often differentiated from those extracted from regions near its center. The second term, transverse, is used to describe orientations that are perpendicular to the axis of the wrought billet. Specifically, transverse wrought mechanical test samples have a load axis that is perpendicular to the billet axis as shown by the second depiction in Figure 11. The wrought, transverse samples are not necessarily aligned with a radius of the billet.

The same two terms are used to describe sectioned faces of wrought material. A longitudinal face of a wrought stainless steel specimen corresponds to a plane that has its surface normal aligned with billet axis. A transverse face is oriented perpendicular to this. Occasionally, two terms are used to describe a face of a test sample. For example, a transverse face of a longitudinal test specimen may be described.

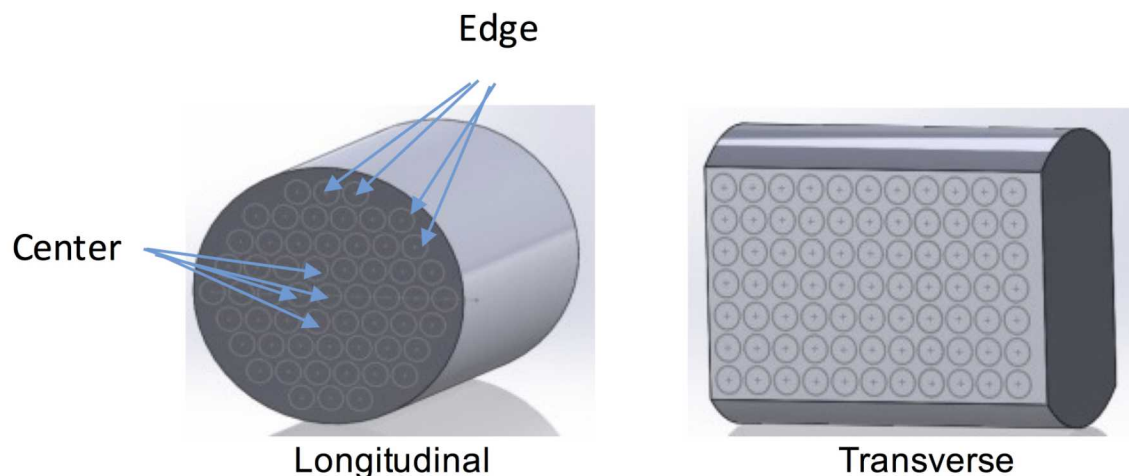


Figure 11. Depiction of wrought billet and sample orientations (longitudinal and transverse).

5. DESCRIPTION OF ADDITIVELY MANUFACTURED AND WROUGHT SS 304L STRUCTURE

5.1. Characteristics of Additively Manufactured SS 304L prior to Mechanical Tests

5.1.1. Composition

The composition of AM SS 304L was evaluated and compared with that of received powder to determine if changes occurred as a result of high power additive manufacturing. Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS), Optical Emission Spectroscopy (OES) and Leco methods were chosen for inspection.

Table 3 documents the compositions of received powder and SS 304L made using a 3.8 kW process. Compositions are listed in wt. % with Fe being the remainder. Study of this table and the bar graph in Figure 12 show that small changes have occurred as a result of processing. The plot in this figure shows the % wt. change for each elemental species wherein a negative value (in blue) corresponds to a reduction and a positive value (in green) means an increased percentage. At the top of this graph (in orange) is the measured elemental wt. % of received powder. Note, the changes due to AM are small, typically less than 1 wt. %. In particular, Cr, Mn and Ni have a reduced composition after processing at 3.8 kW. Other slight, but detectable, changes include modifications to the wt. % of C, Si, N, and O. The slightly increased O concentration is attributed to the interaction of residual oxygen gas resident in the Ar purged system with laser-heated metallic species that have an affinity for this chalcogen. This same interaction may have increased the volatility of Cr, Mn and Ni leading to their decreased concentration.

The composition of SS 304L produced by the 2.0 kW AM process was also determined by the same methods and compared that of the starting powder. Again, small changes in composition are found when using 2.0 kW as summarized in Table 4 and Figure 13.

Table 3. Measured composition of received powder and steel deposited using $P_{avg} = 3.8$ kW. Remainder as Fe.

Elemental Constituent	Powder Composition (wt. %) from ICP-MS	Powder Composition (wt. %) from Leco*	Deposit Composition (wt.%) from ICP-MS	Deposit Composition (wt.%) from Leco*	Deposit Composition (wt.%) from OES
C	n/m	0.015	n/m	0.010	n/m
Cr	19.07	n/m	18.8	n/m	19.16
Cu	0.03	n/m	0.03	n/m	0.03
Mn	1.55	n/m	1.49	n/m	1.43
Mo	0.04	n/m	0.04	n/m	0.04
N	n/m	0.089	n/m	0.075	n/m
Ni	10.38	n/m	10.28	n/m	9.97
O	n/m	0.017	n/m	0.023	n/m
P	0.006	n/m	0.007	n/m	0.011
S	0.006	0.006	n/m	0.007	n/m
Si	0.5	n/m	0.51	n/m	0.56
V	0.02	n/m	0.02	n/m	0.03

* Measurements by Leco involved gas fusion (for O, N) and combustion for (C, S).

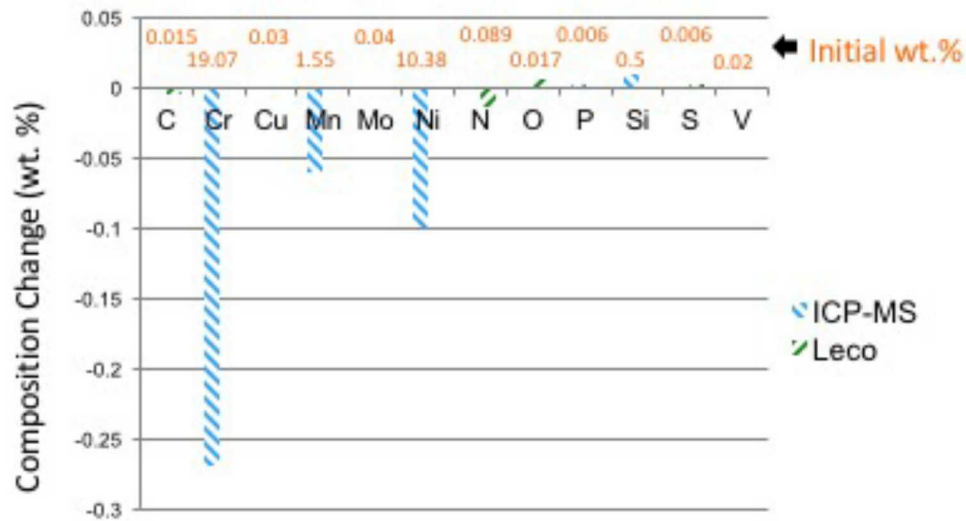


Figure 12. Compositional changes of deposited material referenced to powder used. AM used an $P_{avg} = 3.8$ kW. A reduced percentage is shown in blue and an increase in green.

Table 4. Measured composition of received powder and material deposited using $P_{avg} = 2.0$ kW. Remainder as Fe.

Elemental Constituent	Powder Composition (wt. %) from ICP-MS	Powder Composition (wt. %) from Leco*	Deposit Composition (wt.%) from Leco*	Deposit Composition (wt.%) from OES
C	n/m	0.015	0.010	n/m
Cr	19.07	n/m	n/m	19.02
Cu	0.03	n/m	n/m	0.03
Mn	1.55	n/m	n/m	1.48
Mo	0.04	n/m	n/m	0.04
N	n/m	0.089	0.081	n/m
Ni	10.38	n/m	n/m	9.97
O	n/m	0.017	0.023	n/m
P	0.006	n/m	n/m	0.012
S	0.006	0.006	0.008	n/m
Si	0.5	n/m	n/m	0.55
V	0.02	n/m	n/m	0.03

* Measurements by Leco involved gas fusion (for O, N) and combustion for (C, S).



Figure 13 Compositional changes of deposited material referenced to powder used. These are results from AM using a $P_{avg} = 2.0$ kW.

It is noted that the changes induced by the 3.8 kW and 2.0 kW processes are sufficiently small that the produced material can accurately be described as stainless steel 304L. The resultant elemental compositions are well within the ranges listed in the AISI (ASTM/ASME)[86] and UNS standards for this material.[87]

5.1.2. Phase

X-ray diffraction and Feritscope were used to determine the different phases present in both forms of AM SS 304L. X-ray Diffraction involved a Siemens model D500 theta-theta powder diffractometer (Bruker AXS, Inc. Madison, WI) and monochromatic Cu K alpha (0.15406 nm) radiation that was produced using a diffracted-beam curved graphite monochromator. Fixed 1 degree slits were used for XRD, and the instrument power settings were 40 kV and 30 mA. Datascan V4.3 (Materials Data Inc.; Livermore, CA) software was used to operate the diffractometer. Crystallite size estimates were determined using the Scherrer equation and based on the full-width-at-half-maximum (FWHM) of the individual peak profiles. XRD results were compared with the phase content determined by Feritscope. A Fischer Feritscope FMP30 uses a magnetic induction probe to measure ferrite content to an accuracy of 0.2% and is commonly used to assess ferrite content in weld metal.

These two methods consistently showed that the produced AM steel consisted of austenite and a small amount (approximately 1-3 vol. %) of retained ferrite. Example XRD scans obtained from AM stainless steel (3.8 kW) and wrought SS 304L are included in Figure 14 for comparison along with the archived patterns for austenite and martensite (at the bottom). The intense (111), (200) and (220) reflections for austenite relative to the diminished signal assigned to the single (110) reflection of martensite demonstrate austenite as a majority phase in both forms.

Furthermore, the narrow peak widths of the austenite reflections indicate large grains (> 100 nm) for this phase. Peak broadening analysis of the martensite/ferrite peaks indicate smaller grain dimensions of the order 50-100 nm for both forms. It is noted that samples evaluated by X-ray diffraction were electropolished as a final step to avoid mechanical activated, preferential growth of ferrite/ martensite. Refined lattice parameters were consistent with reported literature values

for austenite [88] and martensite.[89] Measured values were $a = 3.590 \text{ \AA}$ (for austenite) and $a = 2.870 \text{ \AA}$ (for martensite).

There appears to be a preferred crystallographic texture for austenite in the evaluated AM sample (Z face on an X bar). Comparisons of measured (hkl) intensities relative to archived, random texture patterns reveal a preference for the (220) in the Z, or build, direction. The piece extracted from the cylindrical wrought billet appears to be randomly textured.

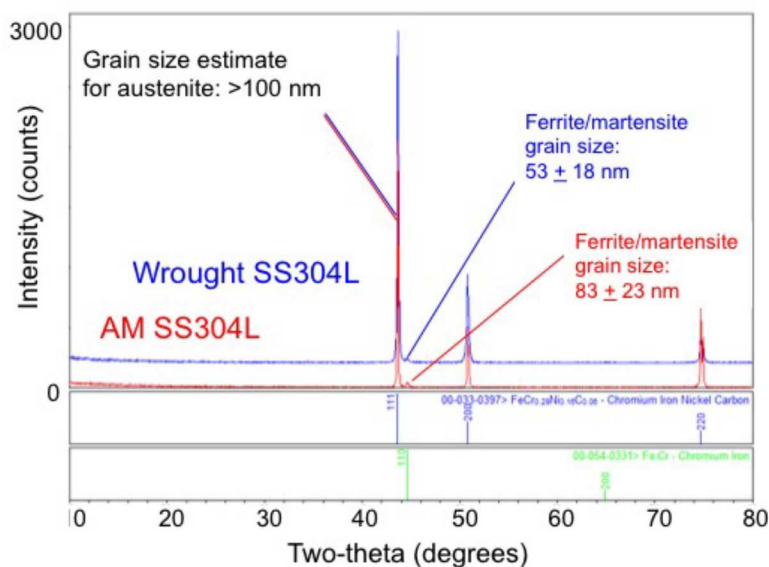


Figure 14. XRD pattern showing evidence of austenite and a small amount of retained ferrite in AM SS 304L. A pattern obtained from the wrought SS 304L is also included.

5.1.3. Microstructure

Samples were prepared for microstructural analysis by cutting along different faces of interest and polishing. Samples were first cut using an abrasive saw with water coolant and gently ground using SiC papers through 600 grit. Polishing started with a $9 \mu\text{m}$ slurry and was followed by 3 and then $1 \mu\text{m}$ slurries to remove all evidence of the grinding and provide a low deformation surface. Electropolishing was then used for final surface preparation. This involved a Struers LetroPol 5 polisher with an electrolyte solution consisting of 600 mL methanol, 360 mL ethylene glycol mono-butyl ether and 60 mL perchloric acid (60%).

Microstructural analysis involved scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD). Scanning electron microscopy utilized a Zeiss Supra 55VP field emission gun electron microscope and a K.E. Developments solid state backscattered electron detector. Channeling contrast was achieved by using an appropriate accelerating voltage and short working distance. EBSD utilized a Bruker detector and Esprit software. When referring to a particular EBSD map, a lower case letter (x, y or z) is used to describe its orientation. This letter does not refer to the bar type or the sectioned face. Instead it defines the direction of color-keyed $\langle hkl \rangle$ orientations of grains and sub-grains. For example, x means that the map shows the left-to-right directions of grains, expressed as $\langle hkl \rangle$ using color. The letter z would indicate the out of paper directions and y would be vertical as shown.

5.1.3.1. Microstructure of SS304L Built with 2.0 kW (Cross Hatch) Process

The EBSD map included in Figure 15 shows a large, electropolished area of additively manufactured stainless steel 304L. This particular bar was made using the 2.0 kW process which involved a cross hatch scan approach. The sample was moved such that the laser-induced melt pool travelled into the page (X) for one layer followed by movement left-to-right (in Y) with this process repeating until the end of a build. The map height in this figure is slightly larger 8 mm. Thus, with a layer thickness ~ 0.89 mm, a portion of 10 layers is revealed within this map.

The indexed Kikuchi diffraction patterns that are used to generate this EBSD map demonstrate that the sample was nearly all austenite – consistent with XRD and Feritscope. Ferrite appears black in this and other EBSD maps. Also important to our study which sought to produce dense material, EBSD maps show no lack-of-fusion defects, large voids or other defects near layer interfaces. It is apparent from this and other EBSD maps and SEM images that grains orient epitaxially from one layer to the next. Inverse pole figures associated with this image show evidence for preferred crystalline (100) texture in the X (stage travel) direction with (022) oriented in Y. Grains are large, up to 1 mm or perhaps even larger in some volumes. These crystallites are larger than those reported for traditional (low power) LENSTM processes.

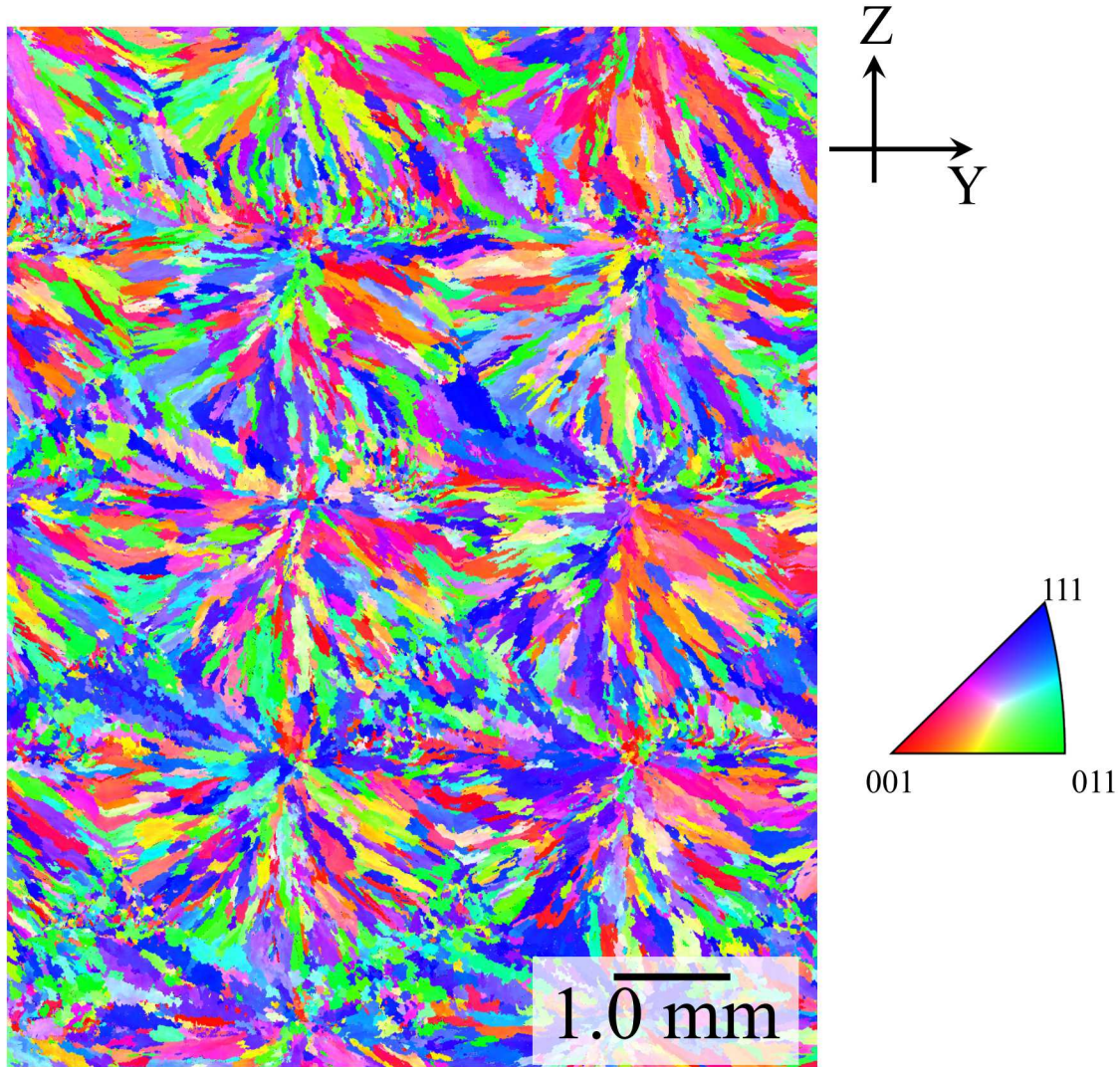


Figure 15. Large area view of microstructure within an AM SS 304L X bar made using a laser power of 2.0 kW. This particular processes implemented an alternating, 90°-rotated scan method for fabrication. The colors coordinate the (hkl) orientations of grains with their x (i.e., left-right) direction as shown.

Additional study at higher magnification shows a variety of microstructural features. Figure 16 includes three images of stainless steel 304L made using the 2.0 kW, cross-hatched process; three separate areas of a single sample are shown. As before, images show only the X face of an X bar with directions detailed within the figure. Figure 16(a) includes a portion of the EBSD map shown earlier. This particular one displays a reduced area, although presented at higher magnification. Evident in this first map, significant sub-grain structure is present in samples made with the 2.0 kW process. A higher magnification EBSD map included in Figure 16(b)

demonstrates sub-grains with detectable mis-orientation angles indicated with color differences. The sub-grain structure shown in Figure 16(c) by SEM has a characteristic dimension as small 5 μm . Also, evident in images 16(b) and 16(c) is a fine dispersion of ferrite. Ferrite appears black in Figure 16(b) yet white in Figure 16(c). In the area shown in Figure 16(c), ferrite is recognized as elongated, sub-micron wide islands which lie along sub-grain boundaries. This is consistent with its location in 16(b). Several ferrite islands are highlighted in 16(c) using red arrows.

Other microstructural features are evident in Figure 16(c). Sub-micron wide, round features are found throughout the stainless steel with no obvious preferred locations such as sub-grain boundaries. Only evident in our SEM images, these features form with a particle-to-particle spacing that is approximately several microns. These features often appear bright in the SEM; two of these are circled using a green line. In other cases, the center of the feature is black. It is expected that the latter features have been removed by etching, likely during electropolishing. There is a bright, circular feature around these which is attributed to edge enhancement artifacts in the SEM. These features are likely oxide dispersions. Small oxide particles can form during AM due to reaction of laser heated metal with oxygen contained within the feedstock (likely incorporated in the powder particles as an oxide film) or residual oxygen contained within the Ar purged vessel. Oxygen may enter the chamber as a trace impurity of the UHP Ar gas or be evolved by heating of internal components. The microstructure maps and images suggest that this form of AM material is fully dense. There is no evidence of voids.

5.1.3.2. Microstructure of SS 304L Built with 3.8 kW (Parallel Scan) Process

In some respects, the microstructure of SS 304L built with the 3.8 kW (parallel scan) process is similar to that developed when using a 2.0 kW process. For example, both have a solidification microstructure akin to laser welded material. However, differences are detected as described in this section.

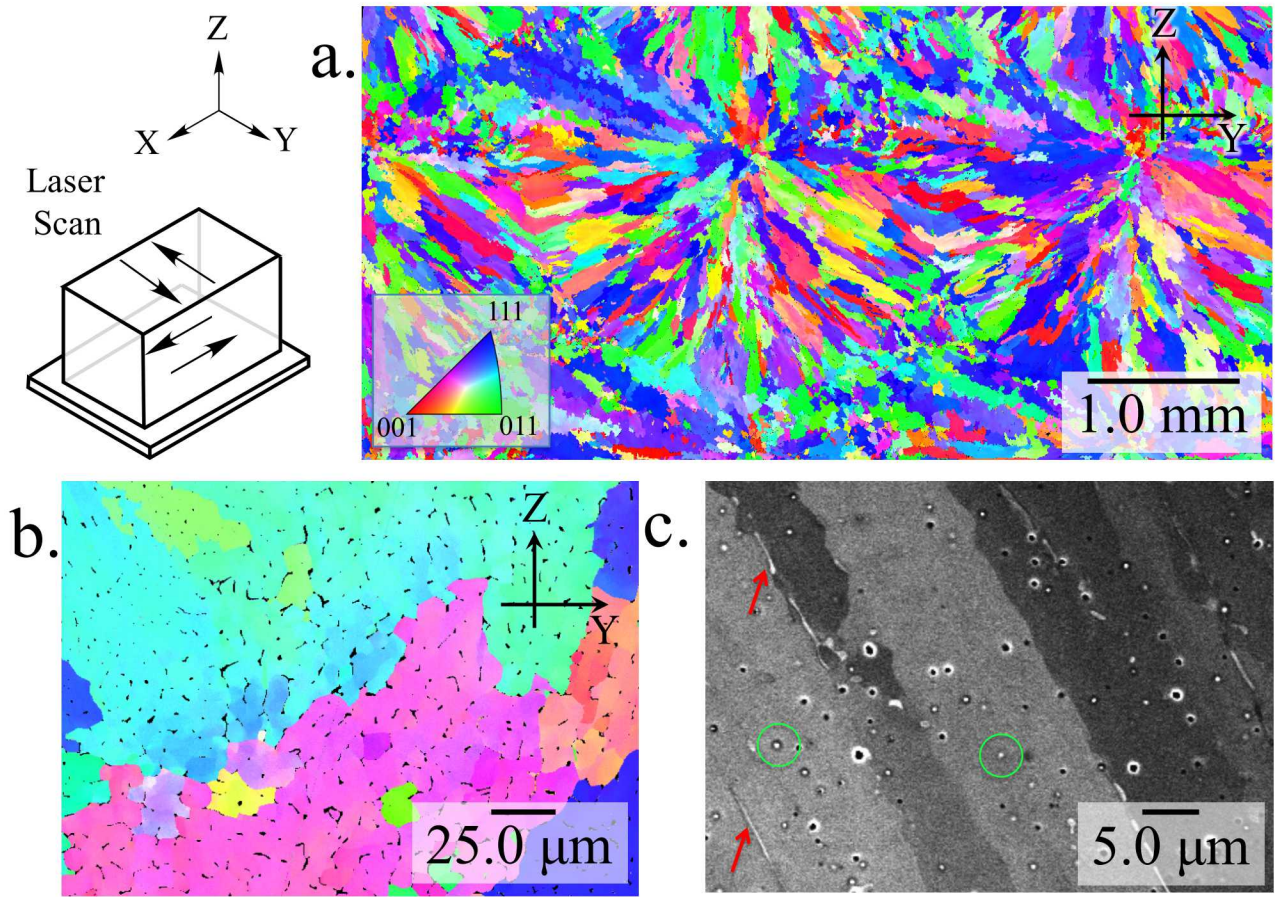


Figure 16. Microstructure of AM stainless steel 304L made using a laser power of 2.0 kW implementing an alternating, 90°-rotated scan fabrication method. Crystal orientation maps in (a) and (b) display grain structure of austenite for a polished X-face of the deposited material. Ferrite is black. The three orthogonal directions associated with the AM process are shown to the left of (a). Scanning electron micrograph in (c) shows a high magnification view of microstructure wherein a few ferrite islands are indicated with red arrows. A few oxide inclusions are circled green. The colors in (a, b) coordinate the (hkl) orientations of grains with their x (i.e., left-right) direction as shown.

A large area EBSD map of an X bar produced using $P_{avg} = 3.8$ kW is included in Figure 17. In this map, an X face is shown with the Z direction oriented up as indicated. The melt pool propagated only along X which is into and out of the page. The solidification microstructure is somewhat different than that revealed in Figure 15. There are periodically-spaced regions of vertically-oriented grains shown in Figure 17 with a spacing that can be understood in terms of the additive, direct write process. The hatch, defined as the centroid-to-centroid spacing, used for this process was 1.925 mm which corresponds to the observed spacing between the

aforementioned regions. This spacing remains constant to the top of the built bar demonstrating good control of stage motion. Individual layers are not evident in the large area EBSD map even though the area imaged contains approximately three (with layer thickness = 1.27 mm). Good layer-to-layer fusion is likely a result of significant heating from the kW beam and induced epitaxial growth.

The effects of stage motion on grain morphology can also be observed when viewing along other sectioned faces such as the one included in Figure 18. The back and forth stage motion along the X direction is apparent in this view of a Z face.

Large grains are apparent in Figure 17 although closer inspection shows considerable sub-grain structure in this form of additively manufactured stainless steel. Additional images and maps are included in Figure 19 for a detailed look at internal structure.



Figure 17. Large area view of microstructure of AM SS 304L made using a laser power of 3.8 kW implementing parallel scan method for fabrication. Crystal orientation maps show evidence of multiple layers used in building. The colors coordinate the (hkl) orientations of grains with their x (i.e., left-right) direction as shown.

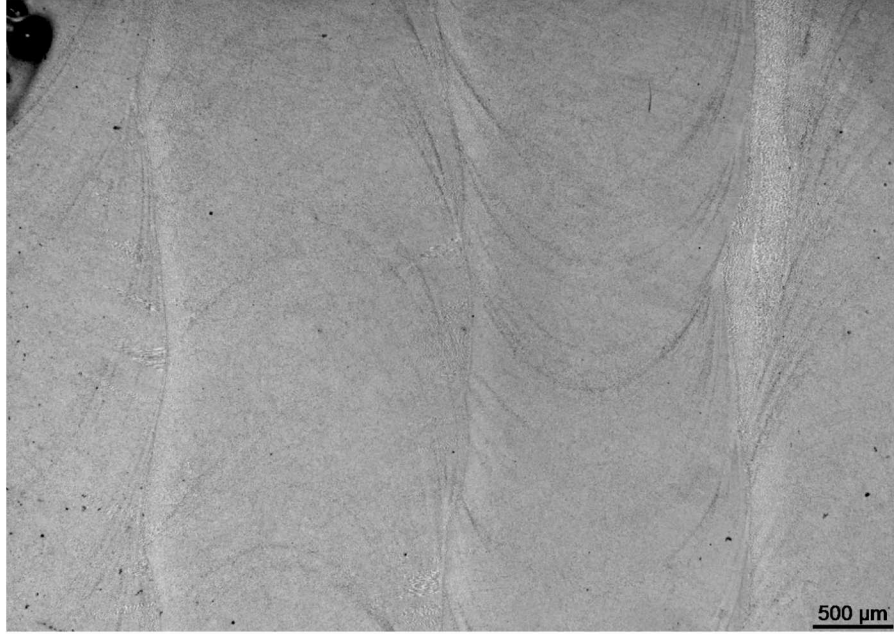


Figure 18. SEM image of a Z face sectioned from an X bar.

Figure 19 includes two EBSD maps and an SEM image. Each shows the structure of a sectioned X face. The second EBSD map (b) and the SEM image (c) show evidence for significant sub-grain structure. The characteristic spacing between sub-grain boundaries is as small as 5 μm . Ferrite is shown as black in EBSD maps. A fine dispersion of ferrite is again found as for the material built using a 2.0 kW process with evidence for ferrite forming along sub-grain boundaries in Figure 19(b). Figure 19(c) shows evidence for ferrite with secondary dendrite arms branching out from what is likely island lying along sub-grain boundaries. Oxide inclusions are again present, with a few features circled in green.

The microstructure maps and images suggest that this form of AM material is fully dense. There is no evidence of voids. Additionally, Wavelength Dispersive Spectroscopy (WDS) shows evidence of compositional variations within stainless steel made using the 3.8 kW process. Figure 20 demonstrates variations in Ni, Cr, Mn composition across and within sub-grains. The relevance of observed compositional modulations is discussed in the context of solidification theory as part of Section 5.3.

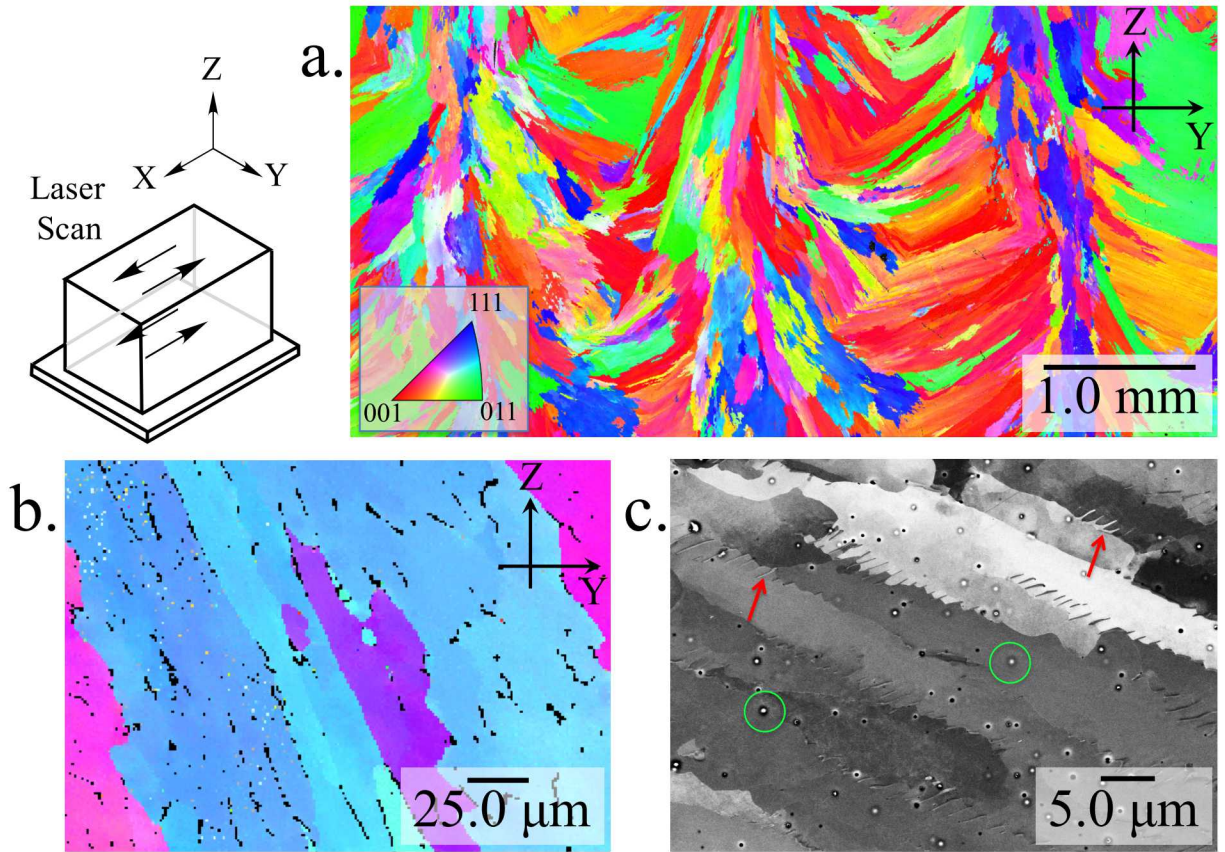


Figure 19. Microstructure of AM stainless steel 304L made using a laser power of 3.8 kW implementing a single scan direction fabrication method. Crystal orientation maps in (a) and (b) display the grain structure of austenite as seen on a polished X-face of the deposited material. Ferrite is black. The three orthogonal directions associated with the AM process are shown to the left of (a). Scanning electron micrograph in (c) shows a high magnification view of microstructure; ferrite is indicated with red arrows. A few oxide inclusions are circled. The colors in (a, b) coordinate the (hkl) orientations of grains with the x (i.e., left-right) direction as shown.

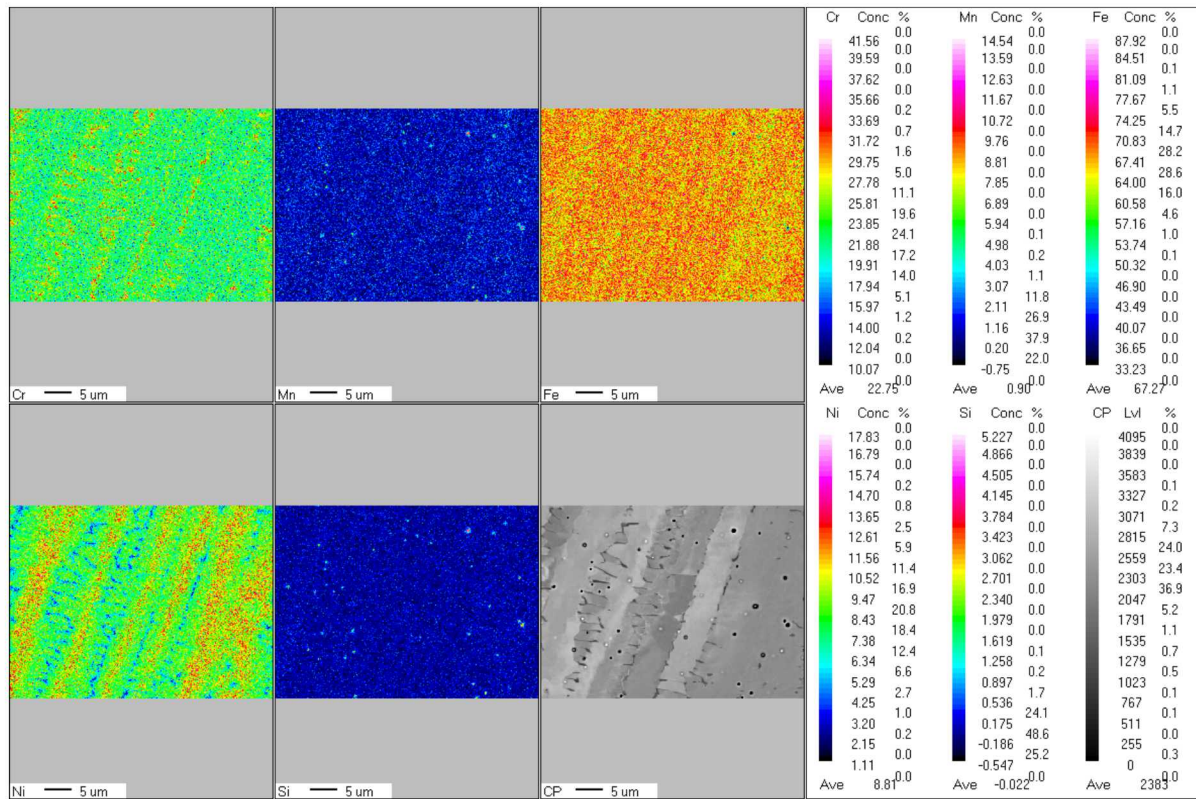


Figure 20. Compositional maps of AM SS304L (X bar, X face) made using $P_{avg} = 3.8$ kW.

5.1.4. Crystallographic Texture

The crystallographic texture was initially characterized using the EBSD data. In an attempt to assemble a statistically significant number of grain orientations, three different EBSD data sets were combined for a total area of roughly 80 mm^2 . The three data sets corresponded to the X, Y, and Z faces of our AM material. EBSD software typically returns grain orientations relative to the face being scanned, so the data sets were imported into Python and transformed into the same (X, Y, Z) coordinate system.

Rather than export the data again to a texture visualization package, pole figures and inverse pole figures were generated within Python. The Python pole figure and inverse pole figure code was written specifically for this project. It calculated pole densities on the unit sphere by counting the number of poles inside a specified bin angle, and then dividing by the area of the bin. The

bin angle was 10° for all pole figures and inverse pole figures in this report. Once binned, the densities are projected onto the stereographic plane. The plotting code was validated two different ways. First, orientation distributions without any texture were plotted to verify that the pole figures and inverse pole figures were uniform. Second, a textured orientation distribution was plotted using the newly developed Python code, MAUD [90], and MTEX [91]. The results were visually inspected, and no differences between the three codes were detected.

Pole figures generated from the EBSD data are shown in Figure 21 for the austenite phase. The density ρ_{hkl} of pole hkl is reported as multiples of uniform density $\frac{\rho_{hkl}}{\rho_u}$, where ρ_u is the density that would have been observed if all poles were uniformly distributed. Note that $\frac{\rho_{hkl}}{\rho_u}$ is plotted on a log scale in order to have the distance between $\frac{\rho_{hkl}}{\rho_u} = b$ and unity be the same as the distance between $\frac{\rho_{hkl}}{\rho_u} = \frac{1}{b}$ and unity. All three pole figures are oriented looking down the Z axis, with the X axis at 3 o'clock and the Y axis at 12 o'clock. The pole distribution is clearly non-uniform, but the data is rather noisy, despite the smoothing provided by the 10° bin angle. Fortunately, cleaner texture measurements were also collected.

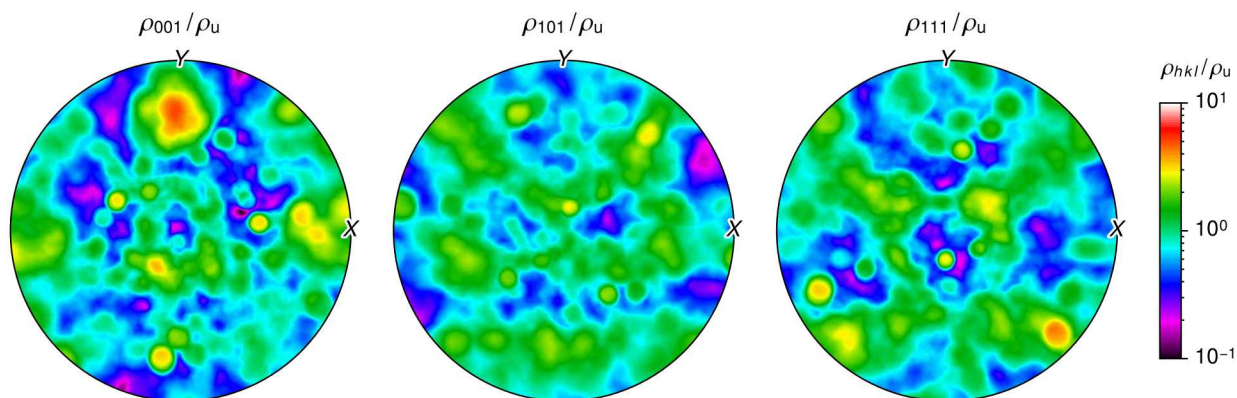


Figure 21. Pole figures derived from EBSD data, for 3.8 kW AM SS304L austenite.

In an effort to measure the texture more accurately, a $1.0 \times 1.1 \times 1.2$ cm cuboid was excised from a 3.8 kW laser power X bar and sent to LANL. At the Lujan Center, the texture was measured using a neutron time-of-flight powder diffractometer called the High-Pressure-Preferred

Orientation (HIPPO) diffractometer. The cuboid was measured using the 0° , 67.5° , and 90° orientations for a total of 20 minutes, and the orientation distribution function (ODF) was calculated by the E-WIMV algorithm in MAUD. The austenite and ferrite phases were measured at 97.5 wt. % and 2.5 wt. %, respectively. Further details of HIPPO and the general analysis procedure can be found in [92]. After calculating the ODF, it was imported into Python and the pole figures were plotted in Figure 22.

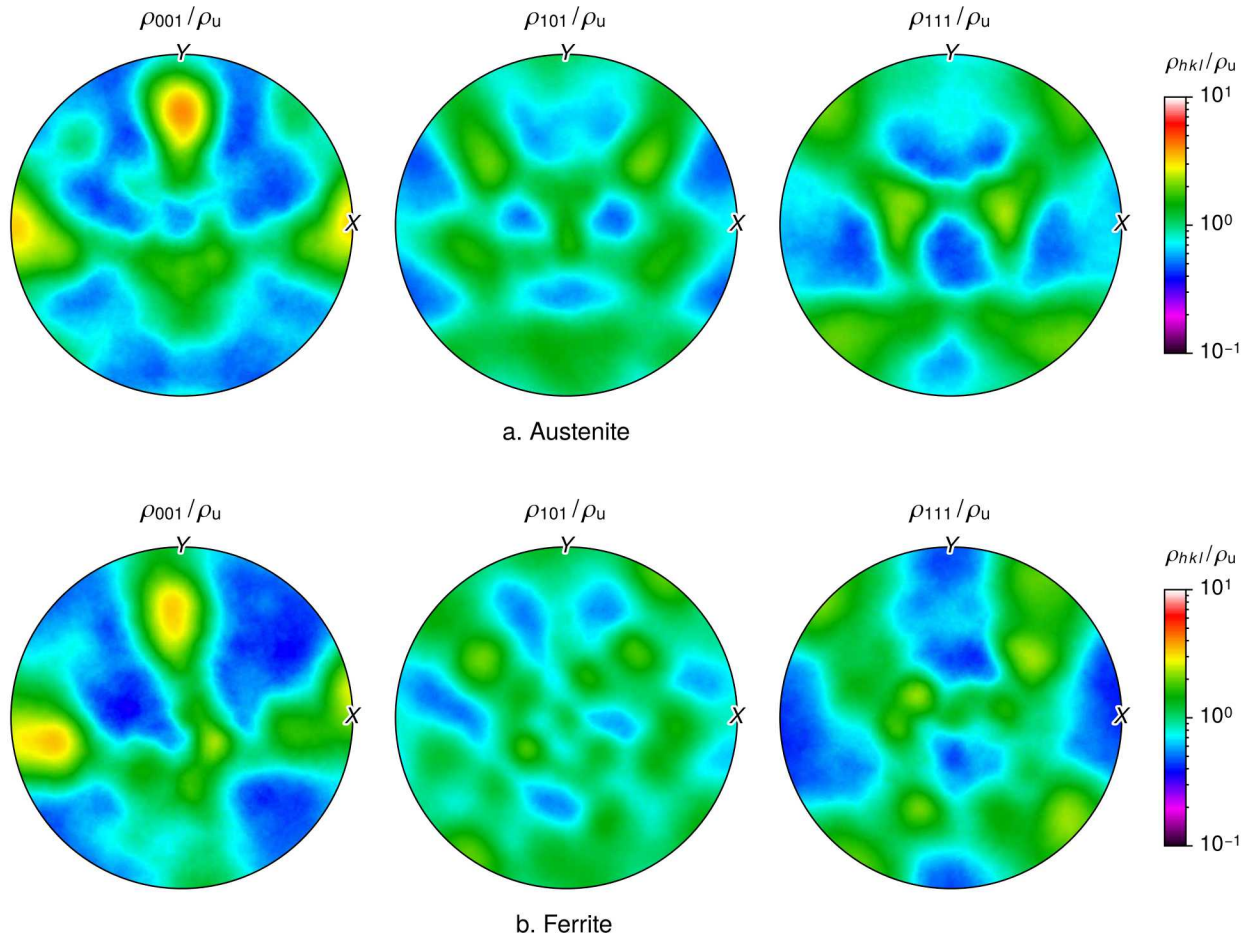


Figure 22. Pole figures derived from neutron diffraction data, for 3.8 kW AM SS304L.

Whereas the EBSD pole figures were difficult to interpret, the neutron diffraction pole figures reveal a clear structure to the texture. To first order, the austenite texture can be thought of as single cubic crystals wobbled by roughly $\pm 30^\circ$ about a primary orientation. The primary orientation's (100) pole is aligned with the X-axis, but the (001) pole is tilted about the X-axis by

about 25°. The ferrite texture is also similar to many single crystals distributed about a primary orientation. The ferrite primary orientation, however, is tilted about both the X-axis and Y-axis by 20° to 25°. Both phases have four local maxima that stand out in the {001} pole figure, but some local maxima are stronger than the others. This indicates the strongest maximum is a common direction for orientations that do not share the other three maxima.

Although the EBSD pole figures are noisier than the neutron diffraction pole figures, they are reasonably similar. Both Figure 21 and Figure 22a have the same qualitative patterns, and the quantitative values of local maxima and local minima are not significantly different. If they do not disagree, then one must ask, “Which measurement captures the true texture more accurately?” Electrons can only penetrate microns into stainless steel, whereas neutrons can reach centimeters beneath the surface. The three EBSD data sets were surface measurements that covered less than the surface area of our cuboid, while HIPPO gathered volumetric measurements of the texture. Thus, Figure 22 is believed to be a better representation of the 3.8 kW X-bar texture.

The 25° tilt of the austenite texture about the X-axis in Figure 22a was somewhat unexpected. Given that the laser beam (the Z-axis) scanned back and forth along the X-axis, one might have anticipated texture with symmetry about the X-Z plane. Sven Vogel, however, measured seven other samples from the same X-bar as the 1.0 x 1.1 x 1.2 cm cuboid, and six samples from another X-bar, and found tilt about the X-axis in all cases. In fact, the tilt about the X-axis can also be seen in the EBSD pole figures in Figure 22. Further study is needed to determine what causes this effect.

Regardless of the cause, the lack of sample symmetry about the X-Z plane can make inverse pole figures misleading. Figure 23 displays austenite inverse pole figures corresponding to the X, Y and Z directions. The (100) peak in the X-direction is clearly visible, and the Z-direction exhibits the relative dearth of (100) and (111) poles, but little else stands out. Figure 22a gives a much more holistic picture of the 3.8 kW X-bar texture.

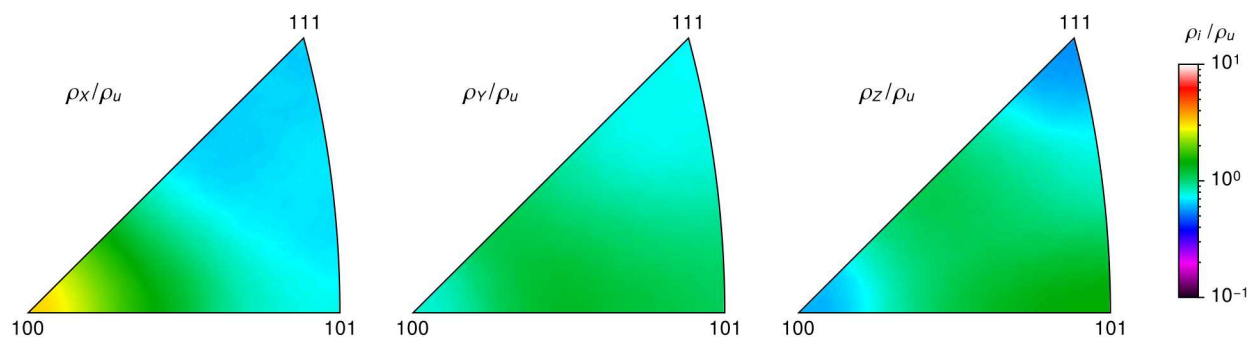


Figure 23. Inverse pole figures derived from neutron diffraction data, for 3.8 kW AM SS304L austenite.

5.1.5. Density, Moduli, Poisson Ratio, Longitudinal Velocity and Shear Velocity

Ultrasonic measurements by Sandia's X-ray/NDE (non-destructive evaluation) group, Organization 01529, characterized the longitudinal and shear wave velocities for samples of both conventional and additively manufactured 304L stainless steel by means of a pulse/echo technique. These measurements were accomplished using piezoelectric contact transducers such that the same transducer coupled to one side of the sample both transmitted and received the high frequency signal. An Olympus Model V111 transducer operating at 10 MHz detected the transmission/reflection time of flight for longitudinal waves, whereas a Model V155 transducer operating at 5 MHz measured the roundtrip timing for shear waves. These transducers were driven by an Olympus Model 5077R pulser that provided tunable square wave excitation. Elastic material constants, namely the Poisson's ratio, Young's modulus, shear modulus, and bulk modulus, were subsequently calculated for each test sample on the basis of its respective longitudinal and shear wave velocities.

Table 5 summarizes the measured properties of stainless steel 304L produced using the 3.8 kW process. Samples used for measurement were discs extracted from plate 19 (similar to those described in Chapter 10 as reverse ballistic samples). These had a diameter of 5.715 cm (2.25"), a thickness of 3.0 mm and a normal direction aligned with Z. Appendix 13.4 includes a complete listing of all measurements. Comparisons with wrought steel are included in section 5.2 with reference to this table.

Table 5. Measured characteristics of AM SS 304L deposited at $P_{avg} = 3.8$ kW. All measurements were made on machined and polished Z-cut samples.

Data Type	Measured Density (kg/m ³)	Longitudinal Velocity (mm/μsec)	Shear Velocity (mm/μsec)	Poisson Ratio	Young's Modulus (GPa)	Shear Modulus (GPa)	Bulk Modulus (GPa)
Average	7832.6	6.01	3.11	0.31	199.69	76.10	181.33
St. Dev.	10.9	0.11	0.16	0.02	17.00	7.62	14.8

5.2. Characteristics of Wrought SS 304L prior to Mechanical Tests

5.2.1. Composition

Wrought stainless steel 304L extracted from a 10.16 cm-diameter, cold-finished, cylindrical bar was used as a baseline material for mechanical tests. The vacuum arc re-melted 304L material was provided as excess material from Honeywell Federal Manufacturing and Technology (Kansas City), and had originally been acquired as a controlled sulfur weld-critical composition with the chemical analysis shown below in Table 6. The bar was tested in the as-finished state.

Table 6. Elemental composition of cold finished wrought 304L bar

Elemental Constituent	Composition (wt. %)
C	0.013
Cr	19.5
Cu	n/m
Mn	1.5
Mo	0.027
N	0.049
Ni	10.1
O	n/m
P	0.015
S	0.015
Si	0.58
V	0.02

5.2.2. Phase

The phase of wrought stainless steel 304L was also characterized using Feritscope and XRD. Both of these methods showed that there was ~1-3% ferrite in starting wrought material. This is consistent with the provided materials certification which had ferrite between 0.6 and 1.2%. Similar amounts of ferrite are present in AM stainless steel as described earlier.

5.2.3. Microstructure

EBSD and SEM were used to evaluate the microstructure of wrought stainless steel 304L. Transverse and longitudinal faces of samples were first extracted by electro-discharge machining (EDM). The machined surfaces were subsequently electropolished prior to characterization.

Figure 24 includes several views of a transverse face. The length scales used in these maps/images are identical to those used in Figures 16 and 19 for AM material. In 24(a), a large area EBSD map reveals a fine equiaxed grain structure. The colors indicate the crystallographic orientations of austenite grains specified in the x-direction of the images (or left-to-right). Comparing this large area map with the EBSD maps of the AM SS 304L, one finds a much smaller average grain size in the wrought material. A higher magnification EBSD map in Figure 24(b) demonstrates an austenite grain size ~10s of microns. Austenite grains are equiaxed. The face of this transverse section is oriented such that the billet axis is left-to-right, which is parallel to the long direction of ferrite stringers (again shown in black with EBSD). The amount of ferrite suggested by these polished faces is consistent with the amount determined by XRD and Ferritscope. However, the ferrite is distributed differently in the wrought form compared with the AM steel. The wrought material does not contain the fine dispersion of ferrite. The microstructure maps and SEM image suggest that the wrought material is fully dense. There is no evidence of voids.

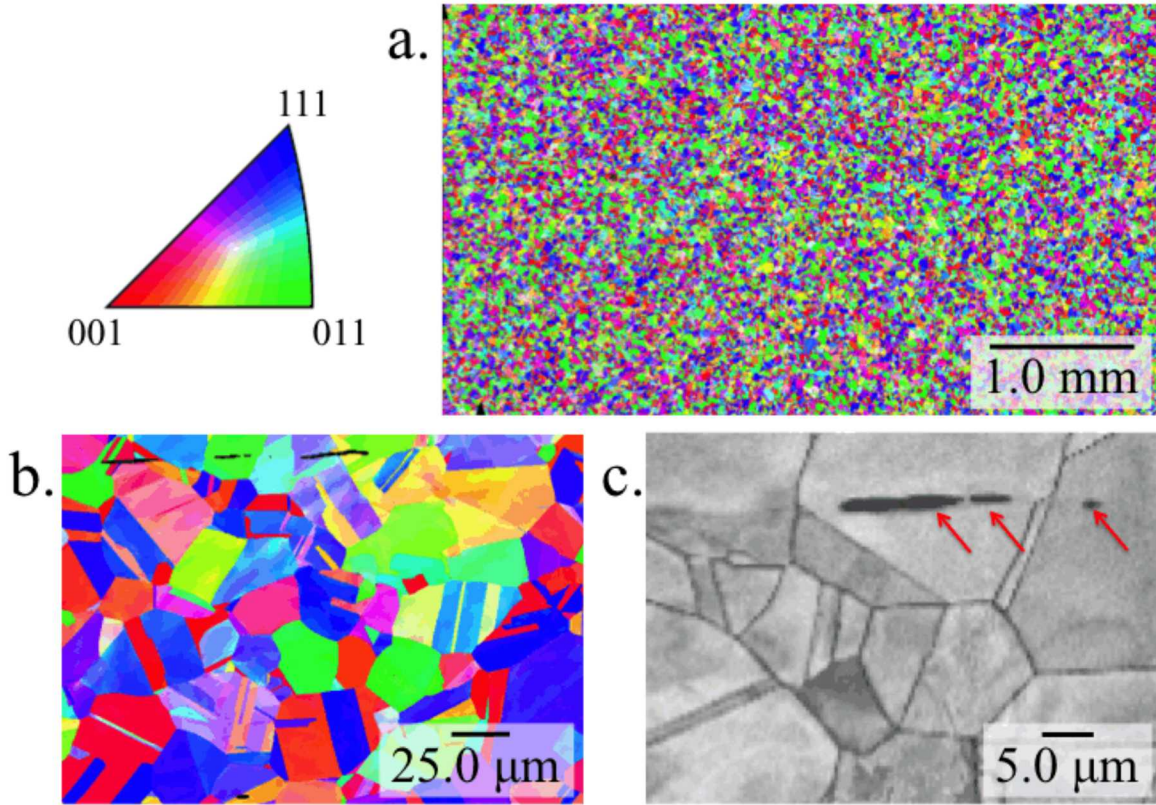


Figure 24. Microstructure of wrought stainless steel 304L used in baseline mechanical tests. Crystal orientation maps in (a) and (b) display the grain structure of austenite. Ferrite is black in these two maps. Scanning electron micrograph in (c) shows a high magnification view of microstructure; ferrite (dark) is indicated with red arrows. The colors in (a, b) coordinate the (hkl) orientations of grains with the x (i.e., left-right) direction as shown.

5.2.4. Crystallographic Texture

In contrast to the 3.8 kW AM material, the EBSD measurements sufficiently captured the wrought texture. The wrought grains were an order of magnitude smaller in diameter, meaning two orders of magnitude more grains were visible in the same size EBSD scan. Roughly 25,000 grains were analyzed within a 15 mm² area on the transverse face of the wrought bar. The resulting pole figures are shown in Figure 25. Clearly, the wrought bar exhibits uniform pole figures with virtually no texture.

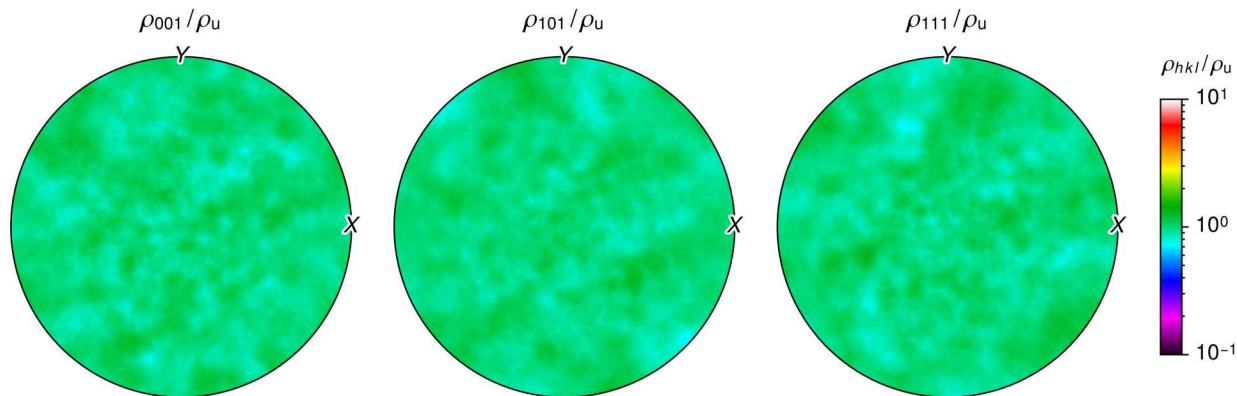


Figure 25. Pole figures for the wrought bar.

5.2.5. Density, Moduli, Poisson Ratio, Longitudinal Velocity and Shear Velocity

Table 7 lists the measured density, longitudinal velocity, shear velocity, Poisson ratio, and moduli of wrought stainless steel 304L determined by the methods described in section 5.1.4. In general, the average values listed in Table 7 are similar to those reported previously for conventional stainless steel 304L.

Comparisons of data included in Table 7 (wrought) with those listed in Table 5 (AM) show similar average values. For example, the AM material has an average density of 7832.6 kg/m^3 which is 99.4% of the average value measured for the wrought steel - consistent with our claims of minimal void volume. Other material characteristics are similar as well. AM and wrought 304L stainless steel have nearly identical longitudinal and shear velocities. The Poisson ratio, Young's modulus, shear modulus, and bulk modulus are also approximately the same.

It is expected that the larger standard deviations listed in Table 5 arise from the considerable point-to-point variability of the AM material. All samples were measured in 5 locations as depicted in Appendix 13.4 for AM and Appendix 13.5 for wrought.

Table 7. Measured characteristics of wrought SS 304L. All values are averages of multiple measurements made at different points of machined samples. Circular samples used for measurement were machined from wrought, cylindrical billet to have their normal direction aligned with the axis of the billet.

Data Type	Measured Density (kg/m ³)	Longitudinal Velocity (mm/μsec)	Shear Velocity (mm/μsec)	Poisson Ratio	Young's Modulus (GPa)	Shear Modulus (GPa)	Bulk Modulus (GPa)
Average	7880.3	5.849	3.144	0.297	202.06	77.91	165.72
Standard Deviation	4.8	0.044	0.003	0.003	0.76	0.13	3.89

5.3. Discussion of Solidification and Microstructure of AM 304L

The microstructure of the as-deposited AM material is largely dictated by solidification, and is quite similar to a multi-pass weld in that it results from solidification and subsequent reheating as additional layers are added. In comparison to the equiaxed grain microstructure encountered in the wrought material where the original ingot solidification structure has been homogenized and broken down, as-deposited AM structures are best represented as a fusion weld material and subject to the segregation, residual stresses, and multi-pass reheating effects thereof.

Solidification behavior of austenitic stainless steels is highly dependent on several factors and has been well characterized over the years.[93] Chemical composition is the foremost factor that determines which phase forms as the primary phase from the liquid as solidification commences. Figure 26 shows a calculated ternary isopleth of a composition very near the base composition of AM 304L deposits.[94] There are four solidification modes that can occur in austenitic stainless steels like 304L: fully austenitic (A), primary austenite followed by eutectic ferrite/austenite (AF), primary ferrite followed by eutectic/peritectic austenite/ferrite (FA) or primary ferrite (F). The general composition of the powder and deposits from Tables 3 and 4 indicate that the solidification mode should be FA, with the structure solidifying as primary ferrite, and then forming some amount of interdendritic (or intercellular) ferrite and austenite as a eutectic or

peritectic as the terminal solidification product. On further cooling below the solidification temperature, a solid state diffusion controlled reaction of ferrite to austenite results in a nearly fully austenitic structure (less than 3% ferrite was observed in the AM deposits). While the equilibrium ternary isopleth in Figure 26 predicts that the structure should be fully austenitic, like most diffusion controlled reactions, kinetic limitations preclude complete homogenization and some ferrite is retained in the as-solidified structure. This ferrite is usually retained as what is termed “skeletal” ferrite which refers to its appearance at the core of the dendrite (cell) arms.

Further work within the welding community has also established predictive tools for the relative amounts of ferrite retained at room temperature in stainless steel.[95] Figure 27 shows one these so-called constitution diagrams originally developed by the Welding Research Council in 1988

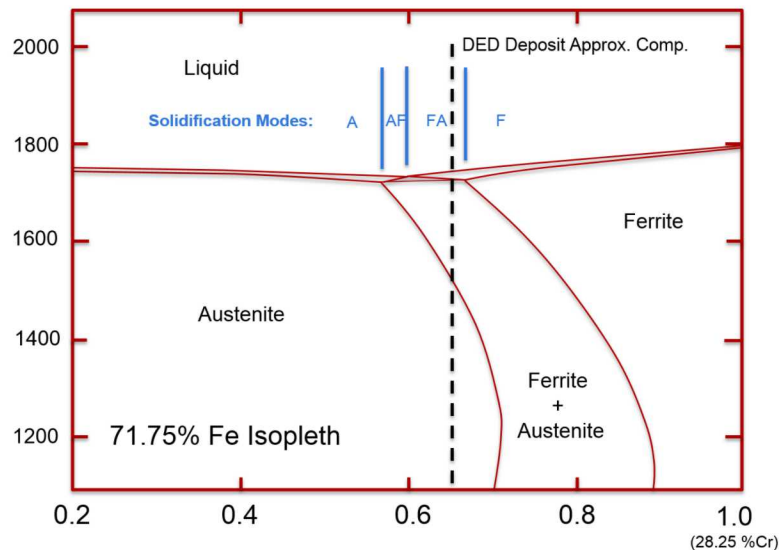


Figure 26. Isopleth at 71.75 wt.% nickel in Fe-Cr-Ni ternary phase diagram showing the approximate composition of AM 304L used in this study.

and refined in 1992 by Kotecki and Siewert.[92] It incorporates the bulk chemical composition in the form of elements that act either to stabilize austenite (nickel equivalent) or ferrite (chromium equivalent) and was developed using solidification rates encountered in single pass arc welding conditions. While the AM deposits studied here were made with laser deposition with multiple passes, the higher energy and lower power density that was used is more characteristic of arc welding heat inputs. Although peak power densities were not characterized for the 2 and 3.8 kW operating conditions, the 4 mm spot size at the deposition height, the size of

the deposition pool deposits (evident in Figure 15 and 17) and their shape as semi-circular or lens-shaped indicates that conduction mode heat transfer was the dominant solidification mode (as opposed to deeper keyhole mode welds typically encountered in laser welding). As a result, using the predictions of the WRC 1992 diagram in Figure 27 should be a useful predictor of solidification mode and residual ferrite.

As seen in Figure 27, the predicted solidification mode for the powder and the resultant chemical composition of each AM deposit after the chemistry changes during deposition should be primary ferrite solidification (FA). Predicted ferrite contents should be between 4-8%, yet all of the deposits examined had between 1-3% ferrite. For ferrite contents that low, the same constitution diagram shows that the solidification mode is expected to be primary austenite (AF). The inconsistency between the measured ferrite and the expected solidification mode was investigated as described in the remainder of this section.

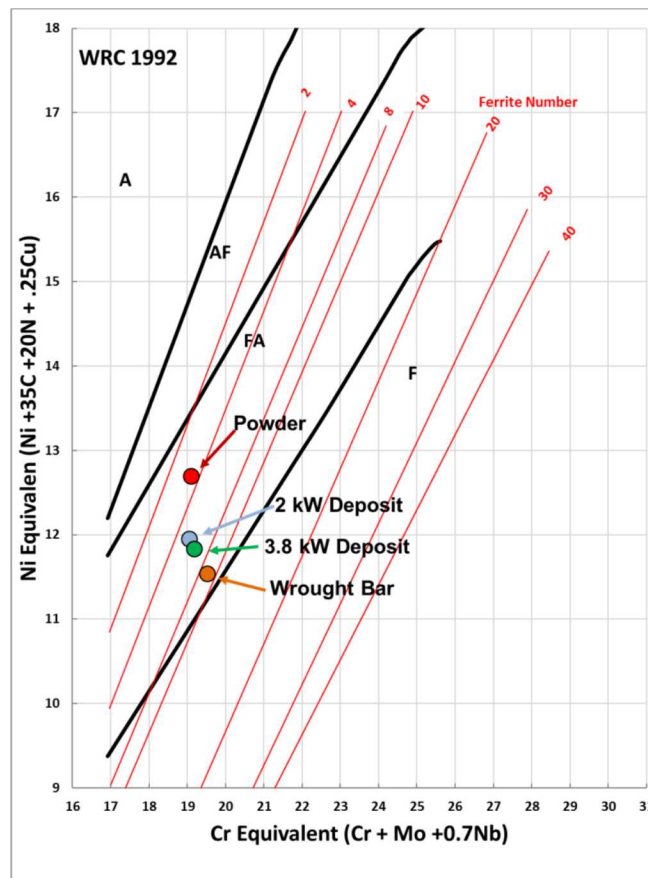


Figure 27. Constitution diagram (WRC-1992) showing the predicted solidification mode and amount of retained ferrite for compositions of 304L used in this study.

The observed microstructures of the AM deposits however were not typical of structures that solidify in the FA mode. Typically, the solid state transformation of ferrite to austenite results in a skeleton of ferrite remaining at the cell or dendrite core after transformation is complete at room temperature. The microstructures of ferrite observed here showed little or no evidence of this structure. Ferrite appears to be at intercellular or interdendritic regions only, as seen in Figure 28. The lack of so-called skeletal ferrite was a bit unexpected, but not without explanation. Firstly, solidification mode has been shown to shift from FA to AF to A as solidification rate increases with higher energy density welding. Therefore, it is possible that, while predictors of solidification behavior (like Figure 27) may have indicated FA solidification, the higher energy density of the laser heat source may have driven the solidification rate high enough to shift the mode from FA to AF. The solidification rate of the laser deposition process relative to arc welds produced an intercellular spacing on the order of 5-10 μm , as seen in Figure 28. Arc welds of similar composition built by additive methods (i.e. multipass shield metal arc welds) have been shown to have cell spacings on the order of 10-20 μm . [96] This difference in cell size is not significant enough to indicate a significant difference in solidification rate between these two heat sources (laser vs. arc). Hence, one would not expect a change in solidification mode.

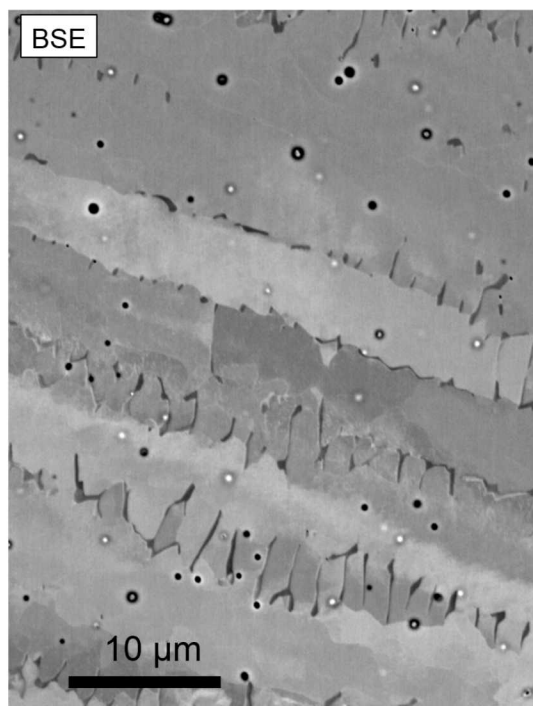


Figure 28. Backscattered electron image of a 3.8 kW deposit. Ferrite appears at interdendritic or intercellular boundaries, but no skeletal ferrite remains in the dendrite cores. Smaller round features are oxide or remnant holes of oxides during preparation.

The definitive method to determine solidification mode involves microchemical analysis of microstructures like those in Figure 28. By conducting wavelength dispersive spectroscopy (WDS) line-scans across dendrites and examining the solute partitioning during solidification, one can unambiguously identify the solidification mode as shown in Figure 29.[97,98] The resultant nickel partitioning behavior across the austenite differs markedly between primary austenite solidification (AF) and primary ferrite solidification (FA, F). In either solidification mode, both nickel and chromium partition to the interdendritic liquid (partition coefficients < 1). In primary ferrite solidification, nickel content appears high in the center of the austenite (the former interdendritic region as solidification terminates) and remains high as it is unable to completely homogenize across the dendrite width during transformation and cooling. Due to kinetic limitations in diffusion, the result is usually a small amount of ferrite remaining at the dendrite core as skeletal ferrite. Conversely, in primary austenite solidification, nickel partitions to the interdendritic region and remains high in that location after cooling. Hence, by conducting a linescan across an austenite cell at room temperature, the nickel partitioning shows that center

of that cell was either formerly an interdendritic region which is now austenite, or was the core of a primary austenite grain and remained so down to room temperature after solidification. Lower nickel at the center of the austenite cell indicates primary austenite solidification, and higher nickel at the center of the austenite indicates that, despite the absence of any skeletal ferrite, the structure solidified as primary ferrite. The remaining ferrite in Figure 30 must then be the result of a eutectic or peritectic terminal solidification product (Figure 26).

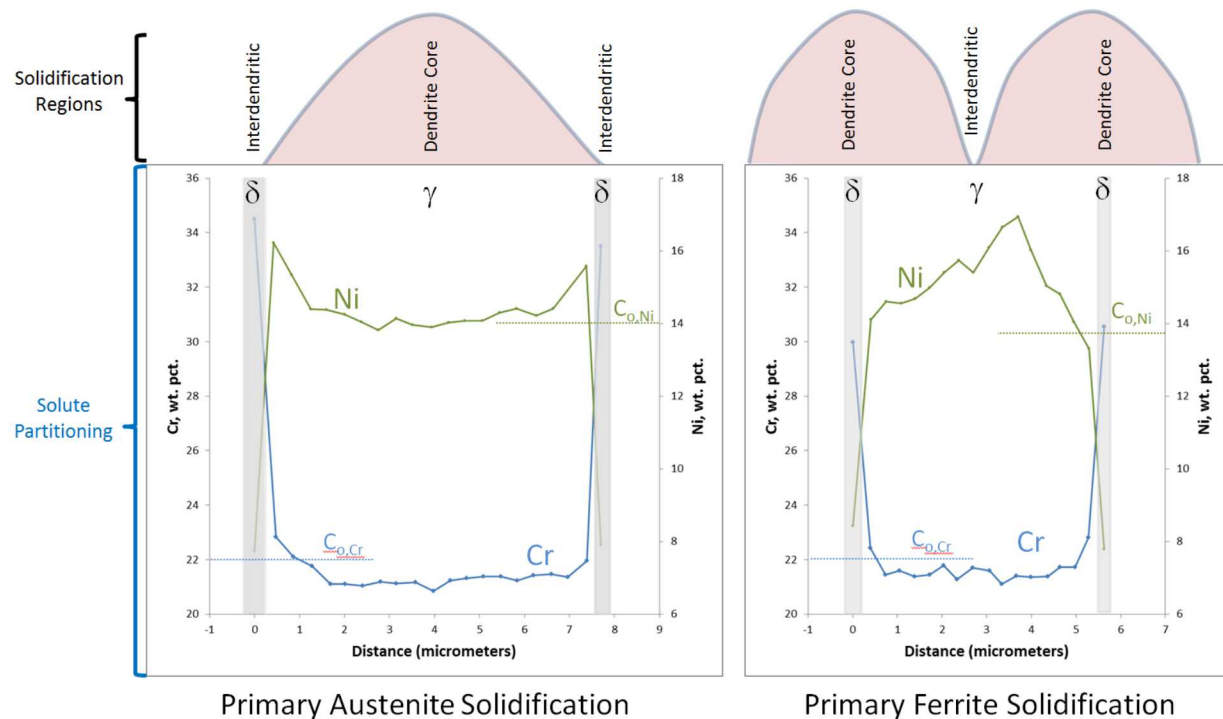


Figure 29. Solute partitioning behavior (after Brooks, 1983) showing the difference in the appearance of nickel partitioning in the solidified structure of austenitic stainless steels (γ = austenite, δ = ferrite).

The constitution diagram shown in Figure 27 indicates that the deposits should have somewhere between 4 and 8% ferrite (Ferrite Number at low levels like these are almost identical to % ferrite). The observed ferrite contents of 1-3% in these structures is thus not due to a shift in solidification mode, but likely rather the result of the multi-pass reheating allowing some amount of the ferrite to dissolve via diffusive mechanisms, resulting in a structure that may appear to be primary austenite solidification with interdendritic ferrite, but microchemical analysis has

confirmed that microstructure solidified as primary ferrite as predicted by the constitution diagram.

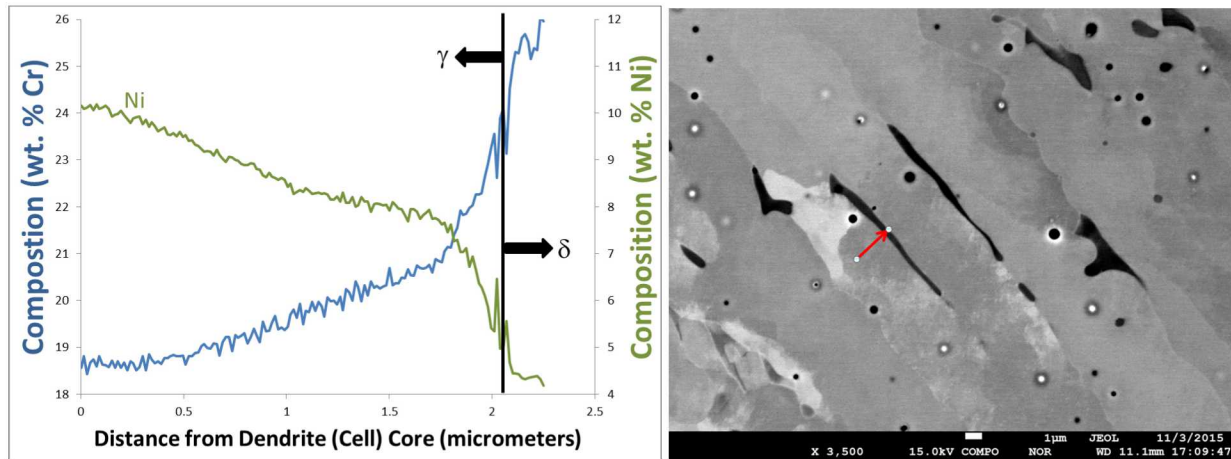


Figure 30. WDS linescan data (left) and location of a linescan (right) in 3.8 kW AM 304L deposit from a dendrite core (now austenite) to the interdendritic ferrite.

6. FABRICATION OF MECHANICAL TEST SPECIMENS

6.1. Precision Machining

The study of AM stainless steel 304L response involved the testing of material extracted from produced bars. To reiterate, there was no attempt to build test samples to final or near-final geometry. Instead, volumes of material were extracted from built bars. Blanks were removed by EDM and then lathe machined to final shape. A cumulative sample log of all tests samples made for quasi-static and Kolsky bar tests is included in Appendix 13.6.

Figure 27 depicts a bar and the final test samples that may be removed from it and their locations. Depending on their overall size, several were successfully removed from each AM bar for testing. As reference, machine drawings that show the original location of extracted cylinders (i.e., blanks) are included in Appendix 13.7.

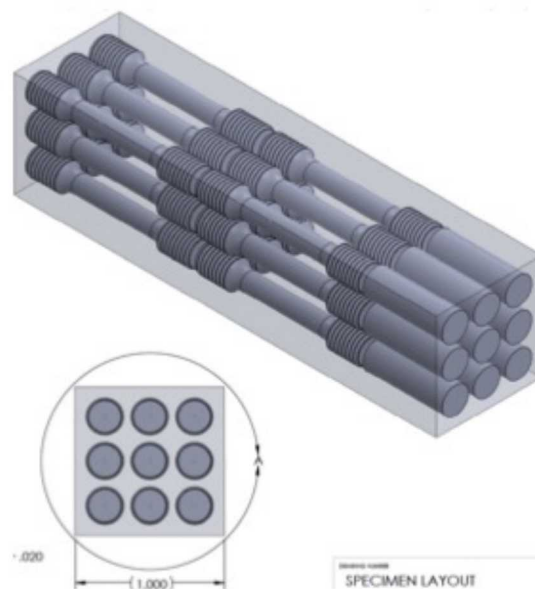


Figure 31. Depiction of tensile and compression samples showing locations within an AM bar. The test samples have axes of loading aligned with the long dimension of the modeled AM bar. Assuming this is an X bar made with the 3.8 kW process, the test samples have axes of loading aligned with the X direction.

Wire EDM was used successfully to remove blanks such as those shown in Figure 32. No obvious bending or distortion to bars was evident after EDM. This was somewhat surprising considering the high residual stress that is apparent in AM metals/alloys and the obvious bending of the build plate. It was discovered that considerable wire erosion occurred during EDM of AM stainless steel.

Final test geometry was obtained by lathe machining. Example tensile bars made for high strain rate testing (Hopkinson) are shown in Figure 33. Again, like with EDM, little or no distortion occurred and the desired final shape was achieved in almost every case. The quasi-static and high strain samples had ASTM test geometries which are specified in later chapters and associated appendices.



Figure 32. Photograph of cylinders extracted by electro-discharge machining.



Figure 33. Photograph of machined tensile samples for Hopkinson tests. Samples are 4.572 cm. (1.80 in.) long.

6.2. Heat Treatment (Portion of Samples)

A portion of AM samples were heat treated prior to machining in order to potentially relieve residual stress and examine how this affects mechanical response. Cylindrical blanks extracted from AM bars were placed in a vacuum furnace that was pre-equilibrated to 750°C. Samples achieved temperature after 0.25 hr. after which they were held at 750°C for 1 hr. Blanks were then removed from the furnace and allowed to cool in air. Heat treated blanks were lathe machined to final ASTM geometries. Similar to as-deposited material, heat treated stainless steel samples were machined without consequence. There was no evidence of distortion or deviation from the desired ASTM geometry. The remaining paragraphs in this chapter describe experimental test results that motivated the selection of temperature and time.

6.2.1. Recovery Annealed Method

After the strategy for extracting mechanical test samples from the various bars was established, hardness traverses were measured from the bottom to the top of Z towers in two locations. The hardness (Rockwell B) along the edges of bars that coincided with material adjacent to either the outer surface or near the center of a bar was measured to determine if there were any significant hardness changes dependent on location. Each bar had a flat surface ground to a width of 0.25 in. (6 mm) on which to make the measurement. Results of the hardness measurement are shown in Figure 34 where two effects of location can be seen. First, both deposits start near the baseplate at the same hardness, but the traverses taken along the edge show a rapid decrease in hardness within the first inch of deposit height. Conversely, the hardness near the center shows a gradual decline from the bottom to the top of the deposit.

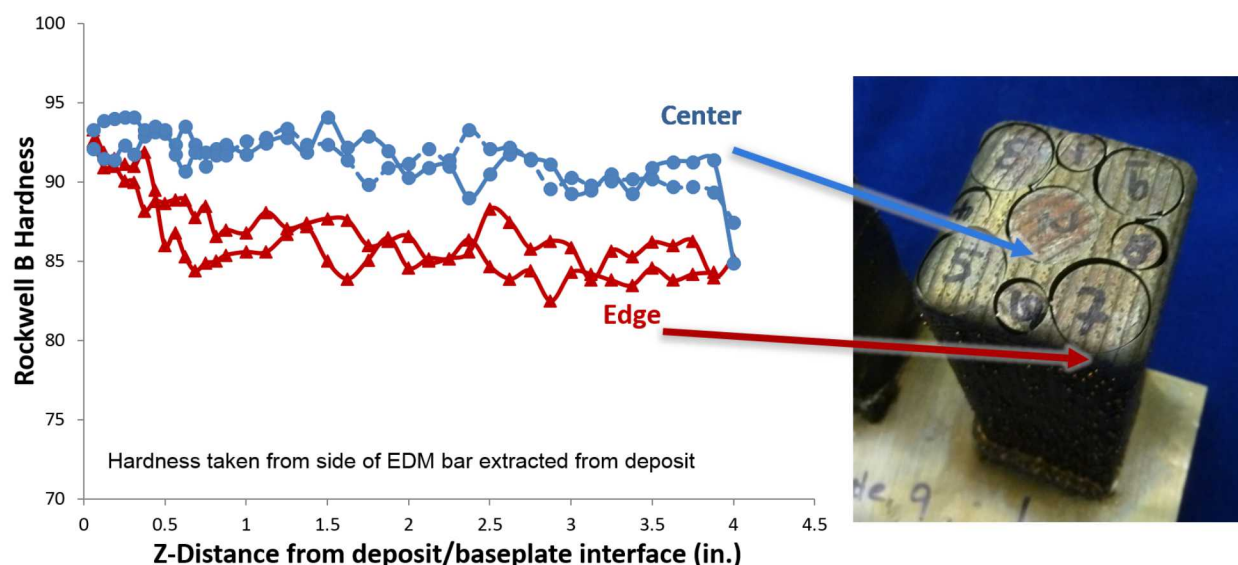


Figure 34 - Hardness traverse results showing the hardness along the height of a Z tower deposit taken immediately adjacent to the edge or near the center.

One possible source of the difference was believed to be the result of residual stress levels sufficient enough to cause plastic deformation. As a result, a subset of samples from AM deposits were subjected to a low temperature annealing study. The goal of the annealing was to attempt to reduce the difference in hardness between the center and edge of the deposits without changing the phase structure (i.e. ferrite content) with a “recovery anneal.” Temperatures between 650°C and 750°C have typically been used for recovery anneals with 304L. The result

of a 1 hr, 750°C anneal is shown in Figure 35 with dotted lines where the center hardness dropped to similar levels as the edge. Ferrite measurements and microstructures before and after the 750°C anneal are shown in Figure 36. Both the as-solidified structure and the annealed structure show the characteristics of cellular dendritic solidification with only a slight decrease in ferrite content (approx. 1% drop) after the annealing. Hence, it is likely that the resulting change in hardness of the center section was a decrease in residual stress level associated with a reduction in dislocation density.

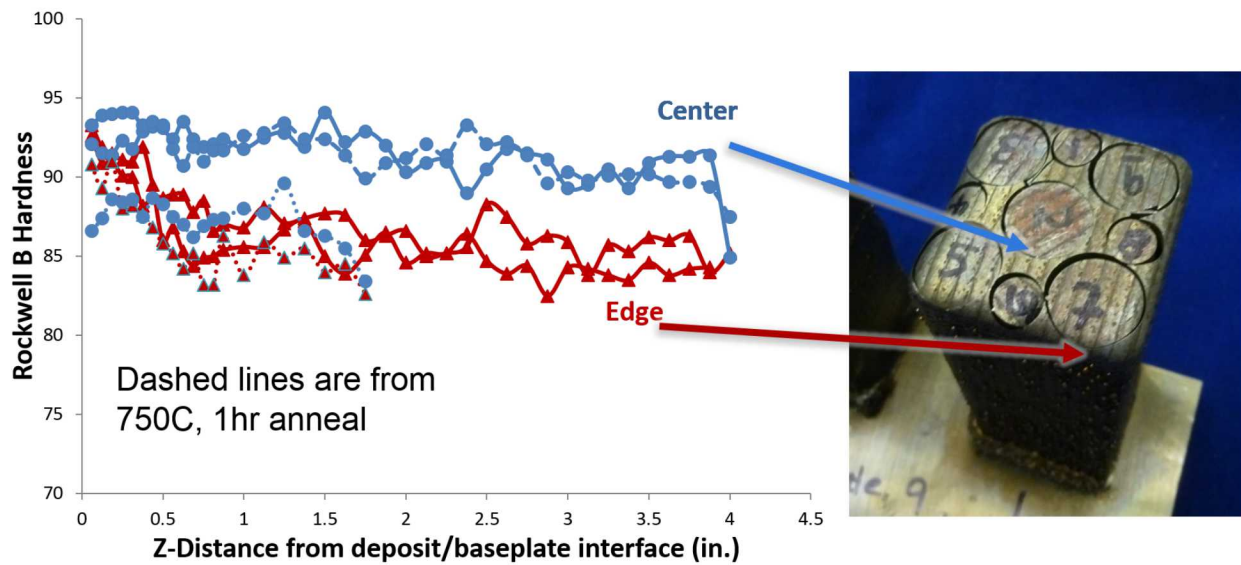


Figure 35 -Hardness traverse comparison after a 1 hr, 750°C anneal (dotted lines) on a sample from the first 2 inches of the same Z tower samples from Figure 34.

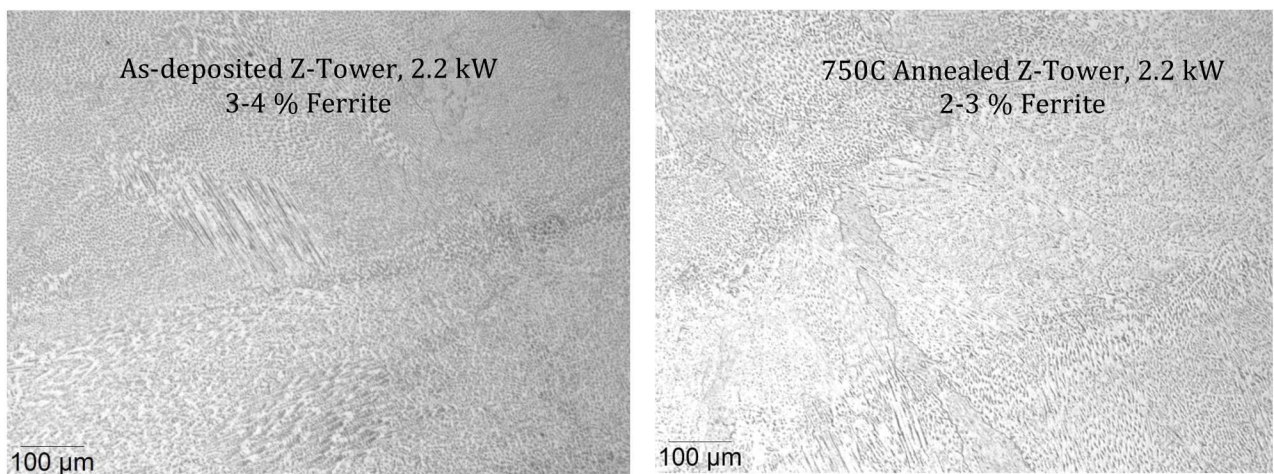


Figure 36 - Light optical micrographs of Z tower sample showing as-deposited (left) and 1 hr, 750°C anneal microstructures. Both structures show as-solidified characteristics (cellular dendritic), with only a negligible 1% change in ferrite content after the anneal showing a slight effect of any significant long range diffusion.

7. QUASI-STATIC MECHANICAL RESPONSE

The purpose of quasi-static testing was to assess the effects of AM build parameters and produced microstructure on mechanical behavior and to develop a better understanding of how response varies with strain rate. In particular, the variables considered include manufacturing method (DED vs. wrought), laser power, specimen location, specimen orientation relative to the build directions, and effects of annealing. The large number of variables combined with the inherent variability of the additive manufacturing process necessitated a large number of experiments. In total, more than one hundred samples were produced for testing.

Tests described as “quasi-static” in this report encompassed a range of strain rates from 10^{-5} to 1 sec^{-1} . One tensile specimen was also tested at a strain rate of 50 s^{-1} , to better understand the gap between conventional quasi-static and dynamic testing (Hopkinson bar).

7.1. Sandia Test Apparatus and Specimen Geometry

Quasi-static tension and compression loading was performed using servohydraulic load frames. Specimens had round cross sections with dimensions shown in Appendices 13.8 and 13.9. An image of these specimens is shown in Figure 37. The longer tensile specimens were originally designed for high temperature studies, but were reallocated for room temperature tests.

Tension tests employed threaded grips with a universal coupler on one side. A 12.5 mm (0.5 inch) conventional extensometer shown in Figure 38(a) measured strain. Compression tests were performed on the 3:1 length to diameter specimens, following ASTM standards, to minimize effects of platen friction and barreling. In compression tests, strain was measured by laser extensometer (Figure 38(b)), compliance corrected machine displacement (for high rates), and in a few cases through displacement gages attached to the platens. Each of these techniques is sufficiently accurate for the purposes of this work, i.e. identify yield stress and hardening behavior.

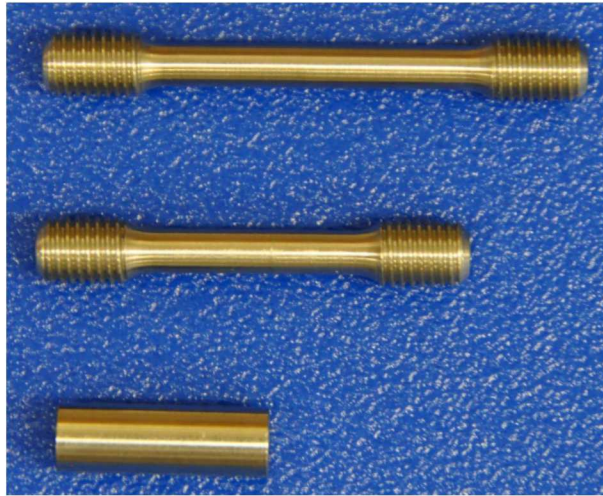
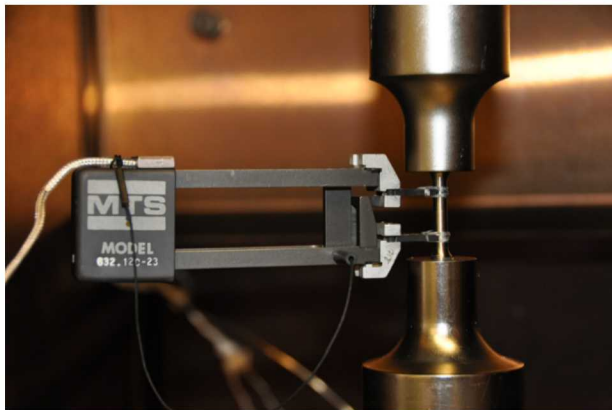
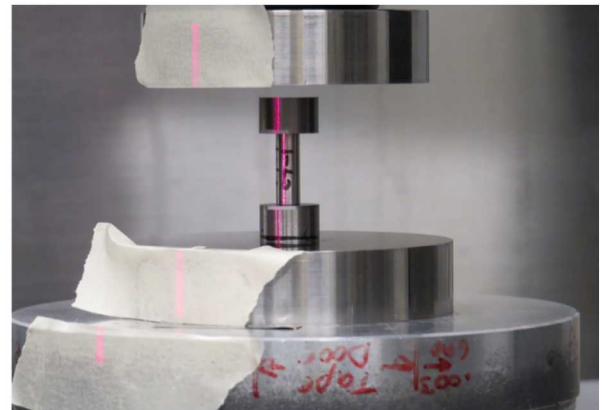


Figure 37. Image of quasi-static specimens.
 For scale, the compression sample (bottom) is 19.05 mm (0.75 inches) long.



(a)



(b)

Figure 38. Photographs of instruments used for quasi-static tests at SNL including a) a conventional extensometer and b) a laser extensometer.

7.2. Results from Mechanical Testing

Over 100 quasi-static specimens were tested as part of this LDRD. Samples had different processing parameters, different geometries, and different loading conditions. Figure 39 is included to show the varied mechanical response across all samples and quasi-static test conditions. This plot includes test results derived from AM as well as wrought stainless steel. Careful study of AM material response revealed considerable variability even for a particular build process. Some insight on this topic will be given in the following sections.

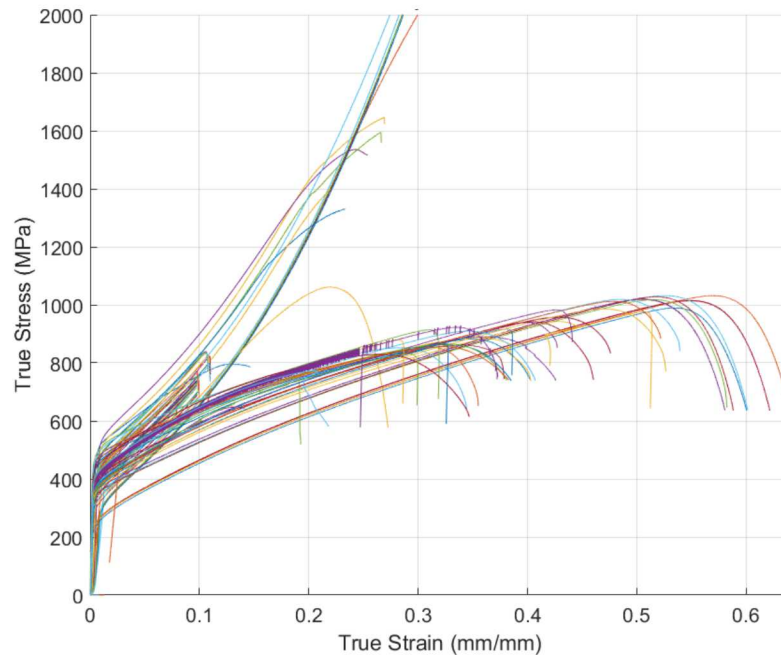


Figure 39. Plot of all quasi-static test results. This includes results of tension and compression tests of AM (including annealed bar) and wrought SS 304L. Compression data is plotted in the positive space.

7.2.1. Tension

Raw stress vs. strain curves from tension specimens are shown in Figure 40 for AM material (LENS) with a legend that indicates strain rate. Results include measurements of AM stainless steel 304L made with the 2.0 kW crosshatch process (indicated with solid lines) as well as

material made with the 3.8 kW parallel line approach (shown with dashes). Also indicated by color, the quasi-static response of as-deposited material is contrasted with annealed material.

Although there is considerable scatter in the tests, several trends are evident. The yield stress of as-deposited material (shown in red) is generally more than 450 MPa. Careful study shows specimens loaded at higher strain rates tend to exhibit higher yield stresses.

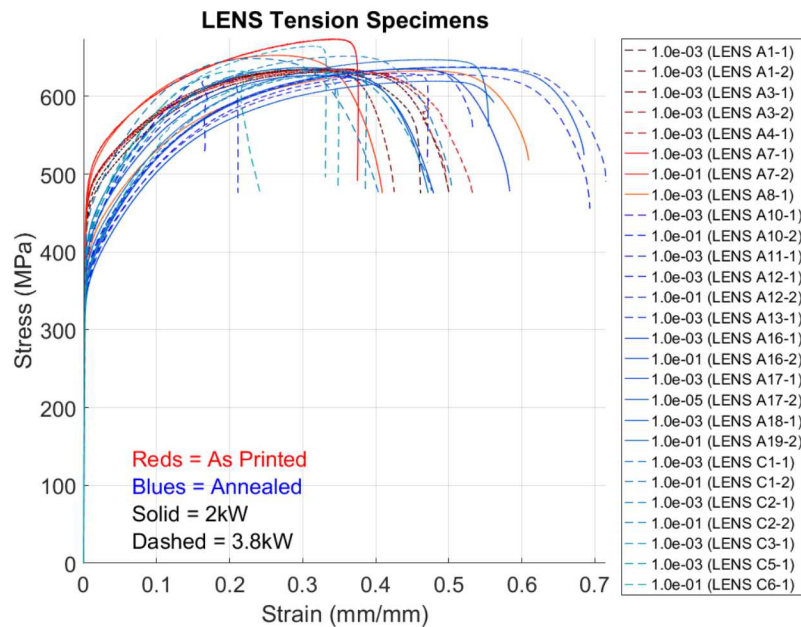


Figure 40. Stress-strain curves for AM SS 304L (in tension). 2.0 kW solid and 3.8 kW dashed. Heat treated samples in blue. As-deposited material in red. Strain rates are identified in legend.

This trend with strain rate can be more easily examined by observing the plot in Figure 41. This figure plots the yield stress for X axis (X bar) samples made with the 3.8 kW, parallel line approach. The plots also include data obtained from high strain rate, Hopkinson bar experiments described in Chapter 9. In all, approximately 9 decades in strain rate are covered. Across this range, there is a clear trend of yield strength increasing with increasing strain rate.

In addition, Figure 41 indicates a decreased yield stress after annealing. Samples tested at 10^{-3} s^{-1} show a reduction $\sim 50\text{-}100 \text{ MPa}$ after heat treatment.

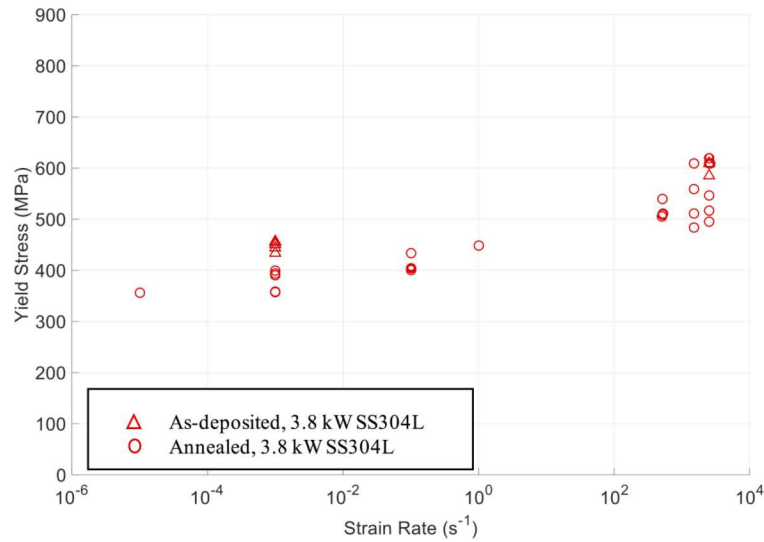


Figure 41. Yield stress vs. rate for only the X direction AM samples in tension comparing data over 9 decades of strain rate.

Figure 42 plots the ultimate tensile strength (UTS) and ductility for X axis (X bar) samples made with the 3.8 kW, parallel line approach. Similar to the yield stress, UTS increases with strain rate. There is not a large difference for quasi-static tests completed at different load rates, but the comparison of this data with measurements taken from Hopkinson bar experiments shows a definitive trend. On the other hand, there is very little difference in ductility across this same range of strain rate.

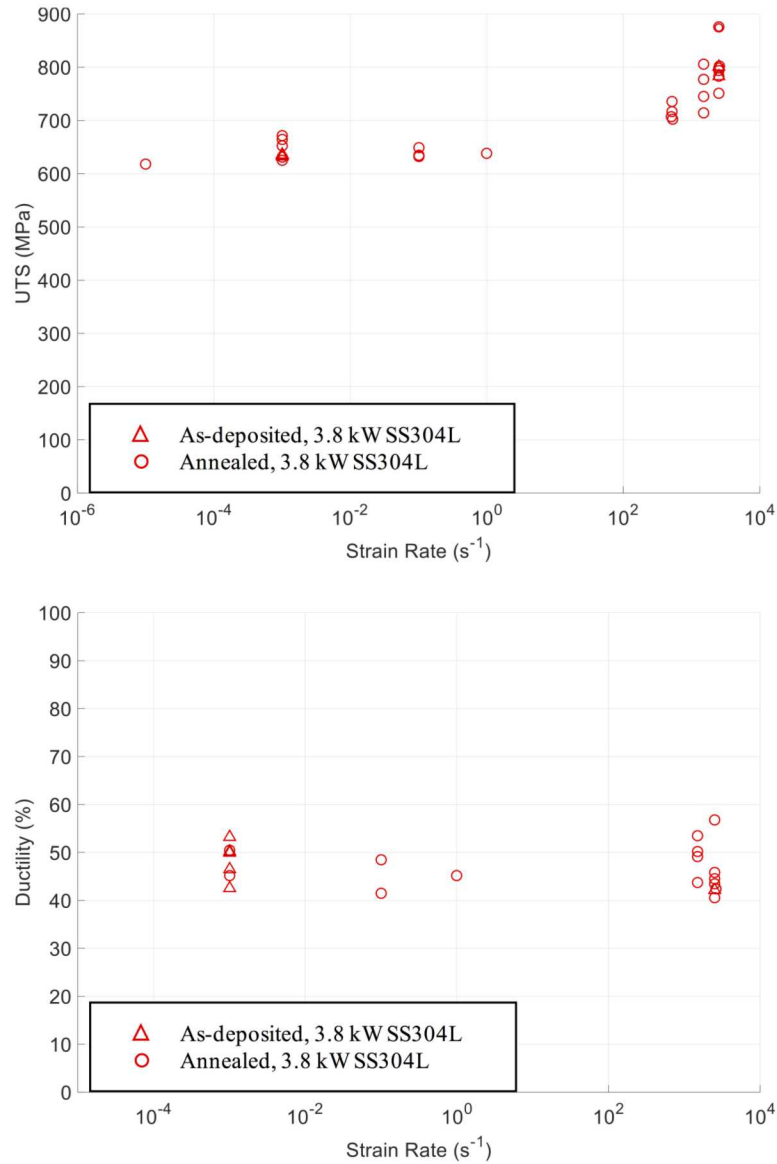


Figure 42. Plots of ultimate tensile strength (UTS) and ductility for X-direction AM SS 304L (3.8 kW) comparing data over 9 decades of strain rate. Data at high strain rates is obtained from Hopkinson bar tests.

7.2.2. Compression

Stress-strain curves from approximately 30 compression tests are shown in Figure 43. This includes tests of both forms of AM stainless steel including as-deposited and annealed forms. Careful study of the included data sets reveals a clear effect of strain rate on yield strength. It should be noted that nearly all of these compression samples were annealed.

For clarification, Figure 44 presents the yield stress of different AM stainless steel samples including those made with the two described processes. Data includes only results from quasi-static tests of as-deposited material.

The dominant effect of strain rate is clearly evident by the linear increase in yield stress with increasing strain rate on a logarithmic scale. It is also apparent from this data that specimens extracted from the centers of bars (filled symbols) tend to be stronger than those cut from near the edge (unfilled symbols in plot). It also appears that crosshatched (2.0 kW) printed specimens are weaker than specimens pulled in the X direction from parallel printed (3.8 kW) bars. Finally, pulling specimens in the Y direction from parallel printed (3.8 kW) bars appears to be slightly weaker than pulling specimens in the X direction. With this data set, no significant tension/compression asymmetry has been revealed.

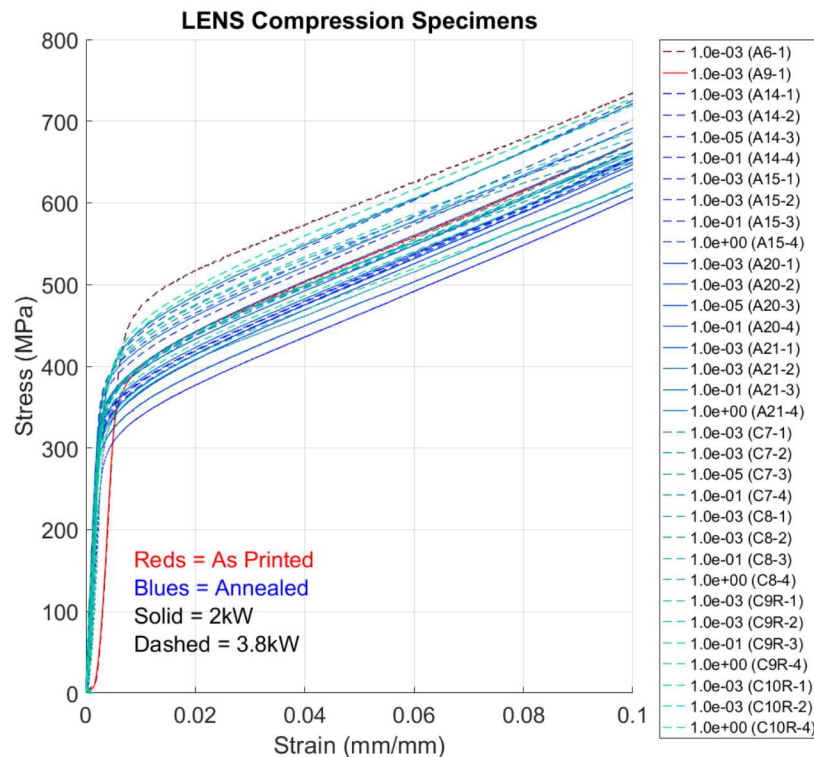


Figure 43. Stress-strain curves for AM SS 304L (in compression). 2.0 kW solid and 3.8 kW dashed. Heat treated samples in blue. As-deposited in red. Strain rates are identified in legend.

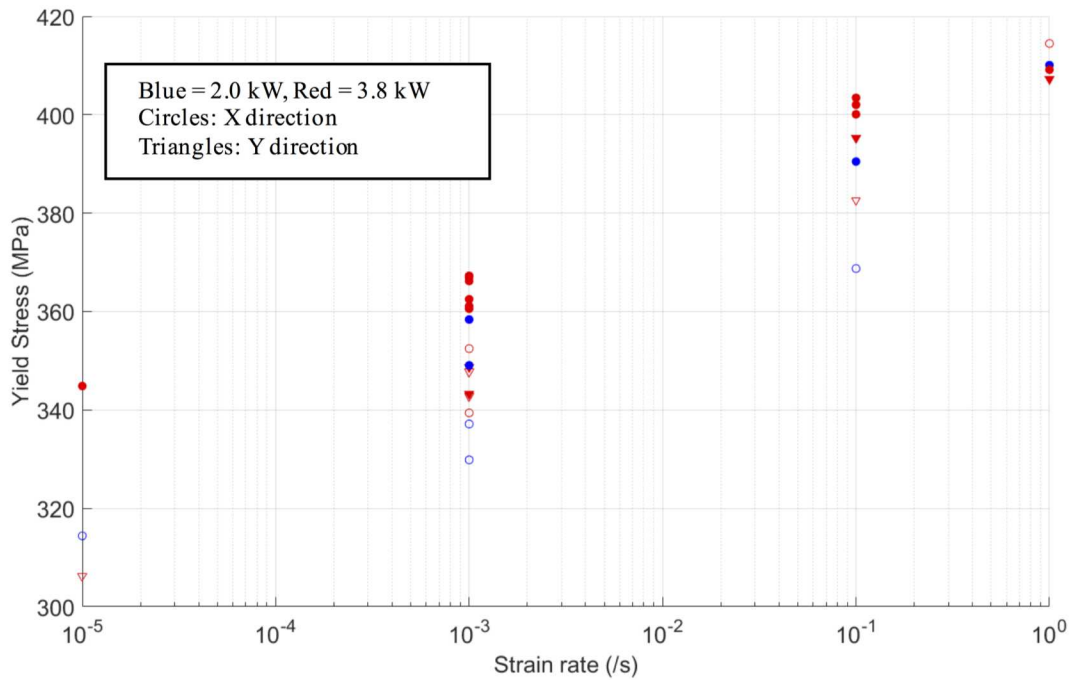


Figure 44. Yield stress vs. strain rate for compression tests. All data is obtained from as-deposited samples (not annealed).

7.2.3. Comparisons with wrought steel

Additional quasi-static tests with wrought stainless steel have allowed for meaningful comparisons with AM material. Both transverse and longitudinal wrought samples were made with the aforementioned ASTM test specimen geometries. Samples were tested with the same equipment and procedures described in section 7.1; this included nominally the same strain rates listed in Figures 40 and 43. Test samples made of wrought stainless steel 304L were partitioned according to whether the material was removed from near the center of billet or taken from close to the edge. A hardness trace in Figure 13.10 demonstrates differences in this mechanical property depending on radial location in the billet with hardness being reduced toward the center.

Results from various tension tests at a single strain rate (0.001 s^{-1}) are included in Figure 45 to compare wrought and AM stainless steel 304L. This stress-strain plot shows several trends in

tension that are similar to observations taken from compressive quasi-static tests at similar rates. First, additively manufactured stainless steel (both forms) has a greater yield stress than wrought. This difference is found for both 2 kW AM Z-bar material (tested in the Z direction) and 3.8 kW AM X-bar material (pulled in the X direction) when compared to both wrought edge and center samples. Second, the AM materials have a slightly greater UTS compared with both types of wrought samples.

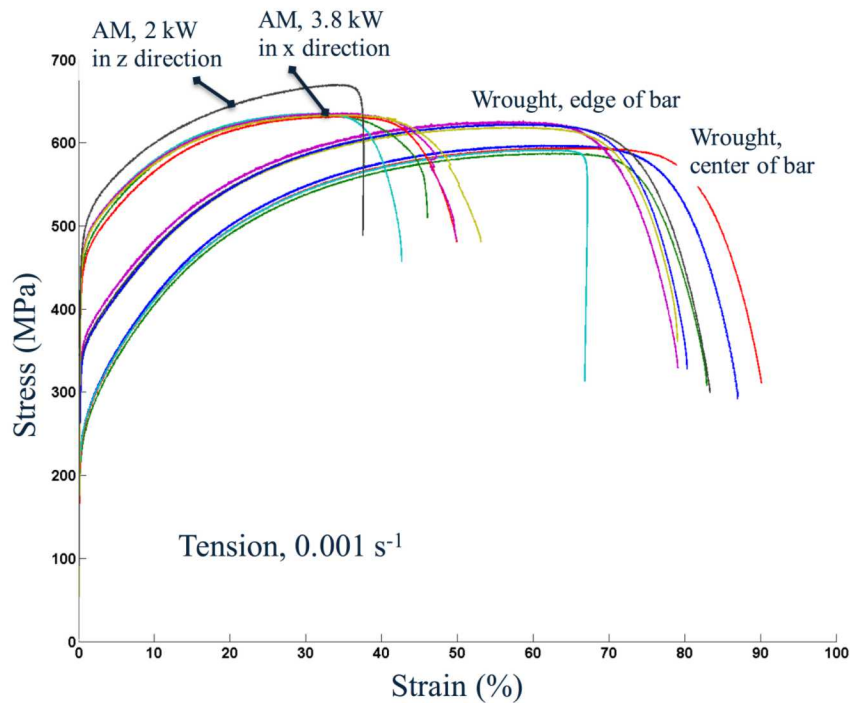


Figure 45. Stress-strain plot highlighting key differences between AM and wrought SS 304L. Results are included for AM material made with powers of 2.0 and 3.8 kW. All AM samples were as-deposited (i.e., not annealed after deposition).

Third, the AM stainless steel 304L has a reduced ductility compared with wrought samples. The ductility of AM material is about 50% that of wrought although there is considerable variation in this mechanical property for the additively manufactured samples (even when limiting comparisons to X-axis samples).

The larger yield stress of additively manufactured stainless steel shown in Figure 4 was at first surprising considering the larger grain size of this material. Traditional Hall-Petch strengthening

attributed to grain boundary effects on dislocations identifies a greater yield stress in finer grain size material (wrought). Clearly, the observed trends are opposite of this particular strengthening mechanism, demonstrating that other factors are germane.

Figures 16 and 19 demonstrate the complex microstructural elements formed within the AM steel and identify other features that could inhibit dislocation motion and strengthen this material. A fine dispersion of ferrite, oxide inclusions and significant sub-grain structure are present within AM stainless steel 304L. The spacings between ferrite islands and oxide inclusions is on the order of the grain size for the wrought material.

For detailed study of strengthening and its relationship to structure, the team turned to *in-situ* neutron diffraction. Neutron diffraction non-invasively probes the lattice parameter of bulk, macroscopic objects such as mechanical test samples. Characterization can be completed before, during and after loading in order to reveal detailed structural changes associated with deformation. The next section starts with a description of neutron diffraction methods and then turns to an examination of mechanical and microstructural response. At the end of this chapter, we discuss various possible strengthening mechanisms which includes mention of structural and compositional [99,100] effects.

7.3. Material Changes Occurring during Loading as Revealed by *In-situ* Neutron Diffraction

7.3.1. Neutron diffraction methods

7.3.1.1 Experimental setup

The *in-situ* neutron diffraction measurements of lattice parameter during compressive and tensile loading were performed on the Spectrometer for MAterials Research at Temperature and Stress (SMARTS) at the Lujan Center at the Los Alamos Neutron Science Center (LANSCE). Details of the instrument are published elsewhere [101] and only a short description is presented here.

LANSCE produces a pulsed (20 Hz) neutron beam via spallation reactions generated by collisions of 800 MeV protons with a tungsten target. The spallation neutrons are moderated by water at 283 K, creating a continuous wavelength spectrum from 0.5 - 4.0 Å, before traveling down the SMARTS incident flight path (~31 m long) to impinge on the sample. Diffracted neutrons are counted in detector banks (³He filled aluminum tubes) fixed at ±90° from the incident beam and 1.5 m from the sample position. In essence Bragg's Law is satisfied by scanning the neutron wavelength during each pulse: the highest energy, shortest wavelength, neutrons arrive at the detectors first. The fixed angles of the distinct detector banks define diffraction vectors at 2θ's of ±135° from the incident beam. The long incident and secondary flight paths make SMARTS a high resolution instrument suitable to measure the small changes in lattice spacing associated with elastic strains.

A purpose-built horizontal load frame was utilized to deform the samples in uniaxial tension and compression. A load cell measured the force (stress) applied to a sample and an extensometer was placed across the gauge section of each one to determine the macroscopic strain during tests. As shown in Figure 46, the load axis was oriented at 135° relative to the incident beam such that the straining direction was parallel to one diffraction vector, $Q_{||}$ (-90°), and the transverse (or Poisson's ratio) direction was parallel to the other, $Q_{\perp}$ (+90°).[102]

During compression tests, a high resolution extensometer was utilized enabling the test to be completed in strain control. The engineering strain was increased incrementally throughout the test to a final value of roughly -10%. After each increment, neutron diffraction data was collected for ~15 min. in order to determine the lattice parameters of both the austenite and ferrite phases in the AM material, specifically, with sufficient accuracy to calculate lattice strains. It was anticipated that higher macroscopic strains could be achieved during the tensile tests (~30%), necessitating the use of a lower resolution extensometer with greater range. This forced the tensile tests to be completed in crosshead displacement control, but otherwise the protocol was identical to compression. The difference will be evident when comparing the flow curves.

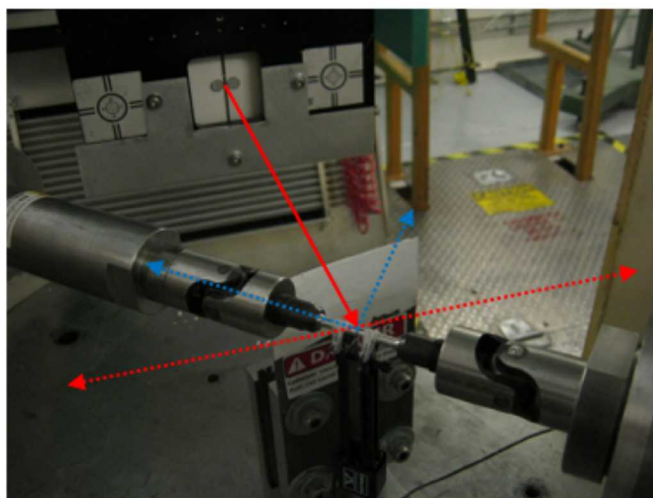
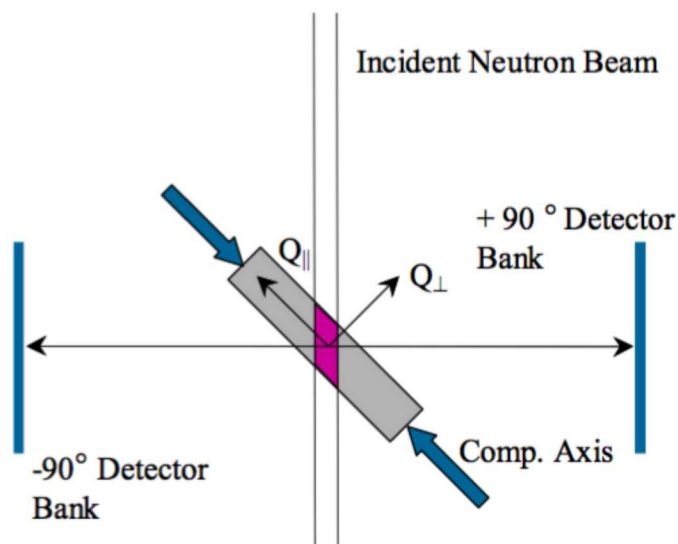


Figure 46. Depiction of test geometry and photograph of mechanical test apparatus positioned at the end of the SMARTS neutron diffraction beamline.

7.3.1.2 Data Analysis and Interpretation

The diffraction patterns were analyzed with Rietveld refinement of the full pattern using the General Structural Analysis Software (GSAS) developed at LANL [103] to determine the lattice parameter (a), phase fractions, peak variances and inverse pole figures [104,105] in both measurement directions. Individual diffraction peaks were analyzed using the rawplot subroutine in GSAS to determine the interatomic spacings (d) of specific crystallographic planes

(hkl). Uncertainties reported in this paper are the statistical estimated standard deviations (esd's) returned by GSAS, and thus ignore any potential systematic errors, for example from the sample physically moving during the deformation test.

7.3.1.3 Texture

Time-of-Flight (TOF) neutron diffraction is ideal for determining a specific projection of the orientation distribution function, that is the inverse pole figure (IPF).[104] The IPF is a plot of pole density as a function of crystal orientation (hkl) along a specific sample direction, e.g. the longitudinal direction. This is precisely what is measured on a TOF diffractometer with fixed detector banks, that is with the diffraction vector aligned with a specified sample direction, e.g. the longitudinal direction. The pole densities are determined with GSAS using a spherical harmonic expansion of the orientation distribution function.[103] The coefficients of the spherical harmonics are determined by fitting the observed peak intensities to those calculated by the microstructural model. The multiplicative correction factors for the preferred orientation in the Rietveld refinement are the Multiples of Random Distribution (MRD) plotted on the irreducible stereograph to produce IPFs.[106] However, in this case in particular, the inverse pole figures are determined from a limited number of statistically significant peaks and should be considered as semi-quantitative.

7.3.1.4 Lattice Strains

The term “lattice strain” will be used in general to refer to elastic distortions of the crystal structure. The more specific terms “phase strain” and “hkl-specific” strain are defined as follows. The phase strains are determined uniquely for each phase from the lattice parameter by

$$\varepsilon(\sigma) = \frac{a(\sigma) - a_0}{a_0} \quad 7-1$$

where a_0 represents a reference lattice parameter. Similarly, the hkl-specific strains are found from

$$\varepsilon^{hkl}(\sigma) = \frac{d^{hkl}(\sigma) - d_0^{hkl}}{d_0^{hkl}}$$

7-2

where d represents the interplanar spacing along a specific plane normal (hkl). The reference lattice parameters/d-spacings were taken to be the lattice parameters determined at a nominal holding stress of 10 MPa. Thus, the reference lattice parameter explicitly ignores residual lattice strains that may have been present following the processing of the sample.

The phase strain, calculated from the lattice parameter (a) of each phase, determined by whole pattern Rietveld refinement, effectively averages over all grain orientations and represents the average phase response.[107,108] In contrast, the hkl-specific strains are determined from the response of a individual peaks. Each diffraction peak (hkl) comprises neutrons diffracted from a unique subset of grains from within the irradiated volume with a common orientation, that is, a specific crystallographic plane normal (hkl) that is parallel to the diffraction vector defined by the instrument geometry. The effective elastic properties of that specific grain family, which are dependent on the bulk crystallographic texture through the interaction of the grain set with its average neighborhood, are then found from the evolution of the interatomic spacing (d) as a function of load in the elastic regime. The elastic strains in a subset of grains with a specific crystallographic orientation (hkl) parallel to the diffraction vector (referred to as hkl-specific strains) are necessarily proportional (in a tensoral sense) to the local stress on that subset of grains, not necessarily to the surface tractions applied to the specimen.

While the lattice strains are not directly sensitive to plastic deformation, the hkl-specific strains, in particular, are indirectly related to plastic deformation as the stresses on the distinct grains sets are redistributed based on the orientation of the relevant active slip system relative to the orientation of the stress. When plasticity initiates in a grain family with a given orientation with respect to the load axis, the rate of increase of the elastic lattice strain in that family decreases markedly. Elastic straining is replaced by plastic flow, to which the elastic lattice strains are insensitive. In the case of perfect plasticity, i.e. no hardening, the lattice strain saturates, and the stress vs. lattice strain curve becomes vertical. Once (perfect) plasticity is initiated in a grain family, no further increments of stress will be supported by said grain family and other grain

families respond by bearing a larger incremental load, and thus, increased incremental lattice strains, resulting in an apparent reduced modulus.

Phase stresses are calculated from Hooke's law:

$$\sigma_1 = \frac{E}{1 + \nu} \left\{ \varepsilon_1 + \left[\frac{\nu}{1 - 2\nu} (\varepsilon_1 + \varepsilon_2 + \varepsilon_3) \right] \right\} \quad 7-3$$

using the phase strains (ε_i) and the assumption that the two transverse strain components, ε_2 and ε_3 , are equal, that is symmetric about the straining direction. Note, that it is not advisable, in general, to calculate stress from the hkl-specific strains because there is no way to uniquely associate a strain measured from a set of grains in the straining direction with one in the transverse direction. For example, strains measured in longitudinal (111) grains and transverse (111) grains come from mutually exclusive grain sets. Thus, a stress calculated from these strain measurements does not represent a stress in any single set of grains that actually exists. To calculate stresses from polycrystalline diffraction measurements, one implicitly assumes that the lattice strains are representative of the bulk response, so that measuring in two distinct sets of grains is acceptable. This is a good assumption when the strains are determined from the lattice parameters (a) [107,108] and with specific grain orientations in cubic materials (e.g. (311) in face-centered cubic steel) and is the basis of residual stress measurements with neutron diffraction.[109,110] However, most low-order orientations, such as the (111) and (200) in fcc steel, patently do not represent the bulk response, and cannot be used to calculate any meaningful stress.

7.3.1.5 Peak Breadth

Although plastic strain does not directly change the diffracted peak position (the elastic strain), it is manifested by increases in peak breadth. TOF profile function 1 in GSAS was used to fit the diffraction data in this study. The interpretation of the profile parameters is discussed in detail in the GSAS manual [111], and only a short description relevant to the peak broadening is given

here. Profile 1 assumes a peak profile that is a convolution of two back-to-back exponentials (describing the neutron source) with a Gaussian line shape defined by

$$G(\Delta T - \tau) = \frac{1}{\sqrt{2\pi}\sigma} \exp\left[-\frac{(\Delta T - \tau)^2}{2\sigma^2}\right] \quad 7-4$$

where ΔT is the difference in time-of-flight between the reflection position and the profile point. The variance of the Gaussian profile is given by σ which is a function of d-space:

$$\sigma^2 = \sigma_0^2 + \sigma_1^2 d^2 + \sigma_2^2 d^4 \quad 7-5$$

where, for simplicity, anisotropic peak broadening has been ignored. It is important to note that σ is fit over the entire profile, and is not specific to any (hkl). To generalize, strain broadening and instrumental resolution contribute to σ , and crystallite size broadening to σ_2 . With the relatively poor peak width resolution of SMARTS, crystallite size broadening would only be observable with very small (sub-micron) size grains. Also, the crystallite size is not expected to change significantly during the deformation tests, and thus σ_2 was held fixed at zero. The peak variance ($S = \Delta d/d$) in percent is determined from

$$S = \frac{1}{C} \sqrt{8 \ln 2 (\sigma_1^2 - \sigma_{li}^2)} 100\% \quad 7-6$$

where σ_{li} represents the instrumental resolution and C is a diffractometer constant. The variance is often referred to as “microstrain” or “intragranular strain”, depending on the audience, and is due to a local distribution of interatomic spacings associated with defects, such as dislocations, or other heterogeneities.

The in-situ measurements were not carried out with the intent of completing line profile analysis. As such, a sufficient instrumental resolution, which must come from a standard sample in the exact geometry as the tensile and/or compressive sample was not collected. Moreover, the

tension and compression samples had significantly different geometries (diameters and gauge lengths). Thus, while the variance will be monitored and interpreted qualitatively to gain insight into the microstructure, quantitative microstructural analysis from the line profile was not attempted.

For the purposes of line profile analysis, high statistical quality, high resolution data was collected on the SMARTS high-angle detector bank before and after deformation. For comparison, the in-situ data collected in the $\pm 90^\circ$ banks was counted for 10 minutes, the DLPA data for 6 hours. These data sets were analyzed for dislocation density and finite crystal size using the extended Convolutional Multiple Whole Profile (eCMWP) line profile analysis method. The methodology of the employed diffraction line profile analysis procedure is the following: theoretical profile functions are calculated as the convolution of the theoretical size, strain (dislocation) and planar fault profiles and measured instrumental profiles which have as parameters the characteristics of the microstructure. These theoretical profiles are based on physical models, which describe the effect of size, dislocations and faulting on the shape of the diffraction profiles. The theoretical profile functions are fitted to the full measured pattern by a nonlinear least-squares algorithm and thus the parameters of the microstructure, for example the dislocation density, are determined. It is important to note that the DLPA analysis was done “blind”. That is, the analysis was completed only with knowledge of the diffraction pattern run number, with no knowledge of the link between run number and sample condition.

7.3.2. Results

7.3.2.1 Mechanical Response

Figure 47 shows the macroscopic compressive and tensile stress/strain curves, respectively, of wrought 304L stainless steel as well as additively manufactured 304L steel produced using 2.0 kW and 3.8 kW average laser power. Tensile data was actually collected to $\sim 32\%$ true strain, but the axis of Figure 47 (right plot) is held fixed at 12% to allow for direct comparison with the compression results. The stress drops are due to relaxation during the neutron diffraction data collection periods; the difference between the relations during holds in tension and compression

is due to the different control mode utilized. Clearly, in both cases the flow strength of the AM material is enhanced relative to traditionally wrought material. The deposition power appears to have an insignificant effect on the flow strength in this regime.

A subtle asymmetry between the compressive and tensile flow strength is apparent in the AM material. At any given strain, the flow strength of the AM material is ~ 50 MPa higher in compression than in tension. To highlight this behavior, the inserts show expansion of the elastic-plastic transition of the materials manufactured at 2 kW and 3.8 kW. The compressive stress and strain have been made positive for ease of comparison. The black line represents the linear elastic response of the material. The elastic limit of the AM material is independent of deformation direction, but the initial hardening is significantly shallower during tensile deformation, resulting in the difference in flow strength throughout the remainder of the tests. The apparent matching of stress-strain curves for wrought center samples in tension and compression shown below is verified in Appendix 13.11.

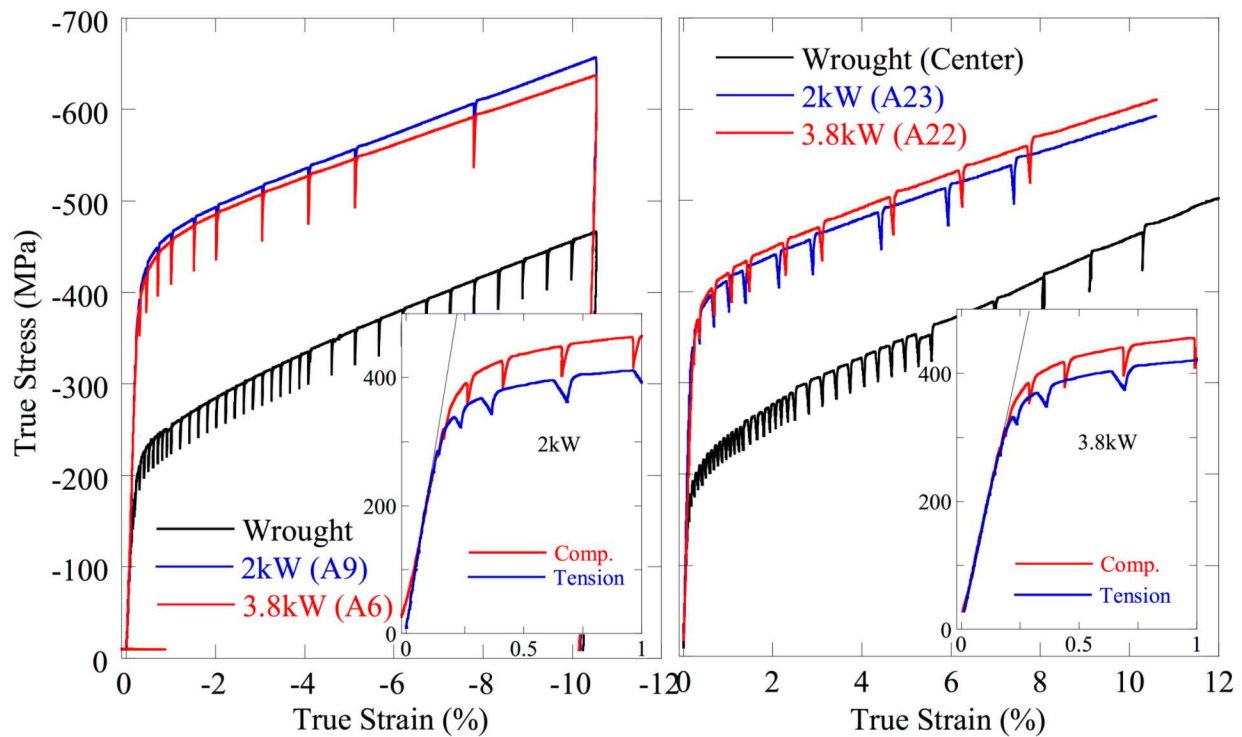


Figure 47. Compressive and tensile stress strain curves. Insets show comparison of compression and tension in elastic-plastic transition.

7.3.2.2 Phase Changes

Figure 48 shows diffraction patterns collected from the compressive samples made of the wrought material and the AM material deposited at 3.8 kW at the nominal holding stress. Only subtle differences in the pattern are apparent at this scale. The texture is slightly different, as evident in the small differences in diffracted peak intensities, and diffraction peaks from ferrite are more evident in the AM material at $2.03 \text{ \AA}$ (110) and $1.17 \text{ \AA}$ (211). In the AM material the ferrite peaks are clear, while little if any signal is present in the wrought material. Qualitative phase analysis resulted in roughly 0.023 ferrite in the AM material, independent of deposition power. Measurements with a ferrite scope also resulted in roughly 0.025 ferrite in the AM material, but indicated 0.018 ferrite in the wrought material. Based on the signal/noise ratio in Figure 48, a 0.018 ferrite fraction should be easily observable if the microstructure of the ferrite in the wrought and AM materials were the same. The lack of an apparent diffraction signal suggests that the ferrite crystals are very small ($\sim 100 \text{ nm}$) in the wrought material, producing broader peaks that would fall into the noise.

The inset of Figure 48 shows the evolution of the ferrite with compressive and tensile deformation. With 30% tensile deformation it is apparent that the ferrite fraction increases slightly, to just greater than 0.03. However, by the 12% deformation achieved in compression (and at 12% tension as well), no significant change in ferrite fraction is observed.

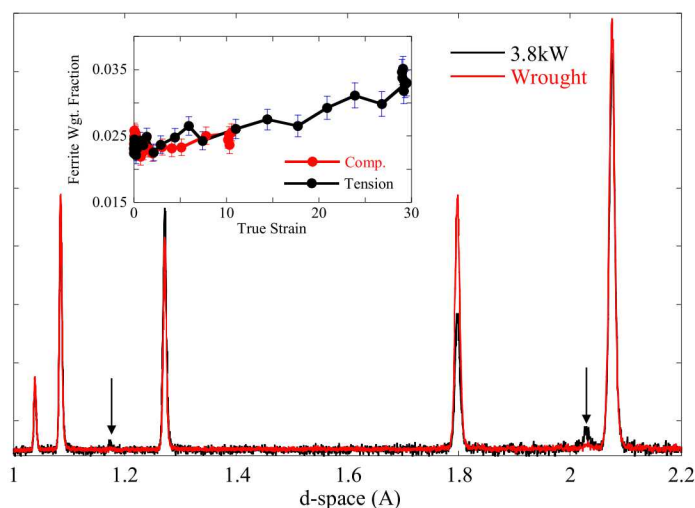


Figure 48. Representative diffraction patterns. The inset shows the evolution of the ferrite fraction.

7.3.2.3 Texture Changes

Figure 49 shows longitudinal inverse pole figures representing a projection of the texture of the wrought and AM materials before and after deformation. As described in sections 5.1.4 and 5.2.4, the initial textures are weak. The texture of the wrought material is within uncertainty of random, at no point reaching 1.1 MRD. The texture of the AM material is slightly stronger and is variable from build to build. In particular, a significant (100) fiber (2.5 MRD) is observed along the tensile loading direction in sample A22 made with 3.8 kW (X bar, X load direction).

Following deformation, the textures evolve as expected for deformation of fcc metals slipping on the (111)<110> system. A (110) texture component develops following compression, while a predominant (111) texture develops following tension, with a smaller (100) component. The different magnitudes of the deformation textures are related to the different initial textures. For instance, the stronger (100) component following tensile deformation of the 3.8 kW (A22) material is due to the initial relatively strong (100) component. The observed texture evolution during tension is consistent with experimental results and elasto-plastic self-consistent (EPSC) model calculations in a similar stainless steel.[112] Run in compression (not done as part of the previous publication), the model also produces IPF's similar to those observed here. A complete set of pole figures (before and after deformation) is included in Appendix 13.12.

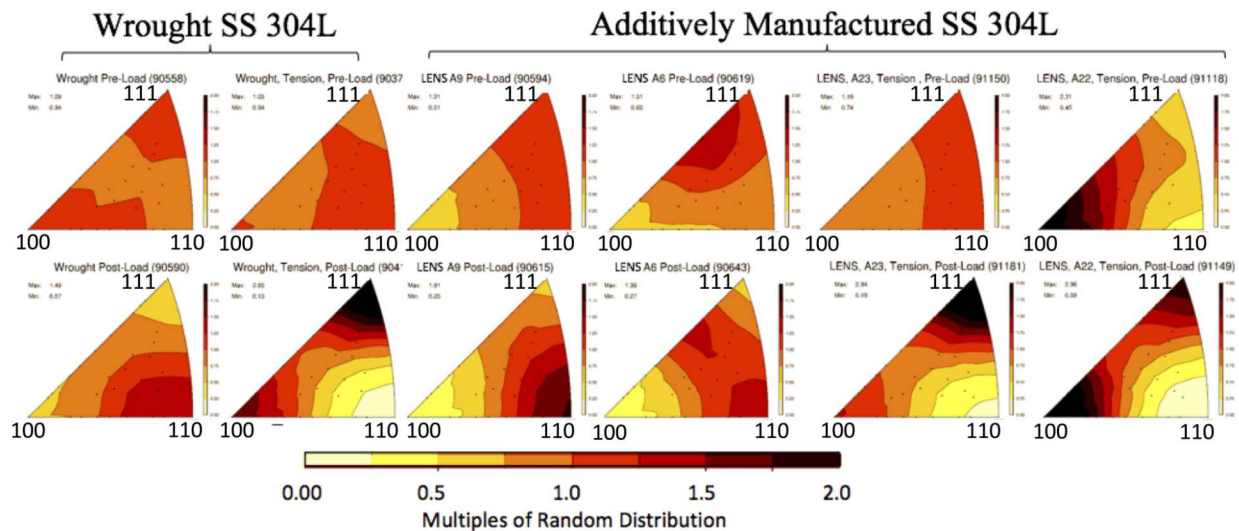


Figure 49. Inverse pole figures representing initial (top row) and final (bottom row) textures. A6 and A22 were made using the 3.8 kW process; A9 and A23 with 2.0 kW.

7.3.2.4 Peak Breadth

Figure 46 shows the evolution of the diffraction peak variance ($\Delta d/d$) as a function of applied compressive (solid symbols) and tensile (open symbols) strain, where the sign of the compressive strain has been reversed for comparison sake. Clearly, in the initial state the peaks from the AM material are broader than the wrought material. Within a given sample geometry, i.e. compression or tension, the initial peak breadths from the 2.0 kW and 3.8 kW material agree well. If we assume the peak breadth is related to dislocation density, then the conclusion is that the AM material has a much higher initial dislocation density than the wrought material. In all samples, the peak breadth increases monotonically with plastic strain, reinforcing the interpretation as related to dislocation density. The peak breadth appears to increase at a higher rate in tension than in compression but, since the geometries of the sample are different and proper instrumental resolution patterns were not collected, we hesitate to place any significance on this.

While not shown, the variance of several (5) single peak variances were determined as well and in each case the peak variance was greater in the AM material than the wrought material.

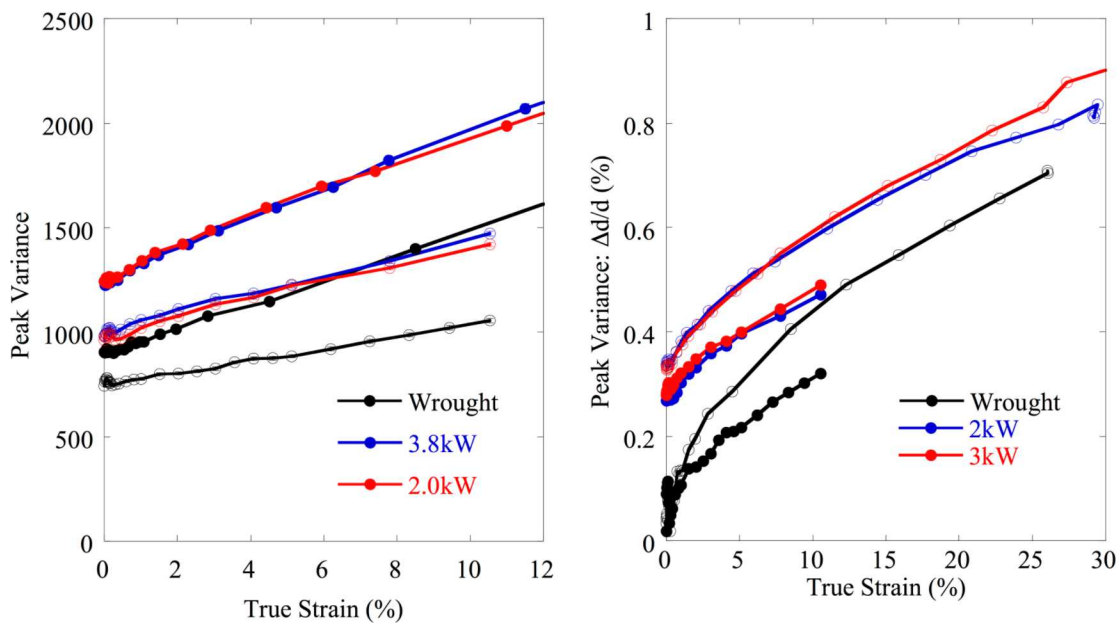


Figure 50. Evolution of diffraction peak variance as a function of applied load for the different forms of stainless steel 304L. Includes compression (solid symbols) and tension (unfilled symbols).

Additional experiments used the new scattering bank on SMARTS to obtain a series of high resolution diffraction patterns from several cylindrical samples produced by the 3.8 kW AM process and others made of wrought SS 304L. Samples were measured in the as-built condition and after deformation to 10% strain in compression or 35% strain tension. Macroscopic stress – strain curves are shown in Figure 51(a) with starting and ending dislocation densities indicated. Previously, the peak variance determined from Rietveld refinement was used as a qualitative determination of dislocation densities, and Figure 51(b) shows the peak variance versus the true strain. It is again found that the AM material exhibits a higher yield stress than the wrought reference material, both in tension and compression. The measured Diffraction Line Profile Analysis (DLPA) dislocation densities are consistent with the macroscopic stress-strain curves and the peak variances.

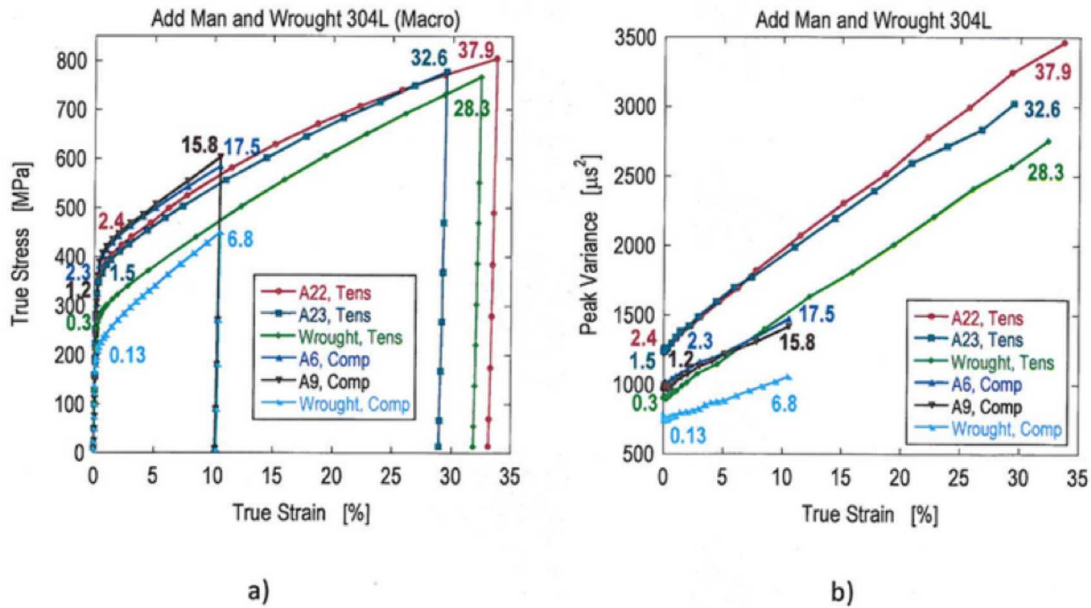


Figure 51. (a) Macroscopic stress-strain curves including measured DLPA dislocation numbers (b) Peak variance as a function of true strain including DLPA dislocation numbers. A6 and A22 were made using the 3.8 kW process; A9 and A23 with 2.0 kW.

7.3.2.5 Phase Strains

Figure 52 shows the phase strain (austenite) during compressive and tensile deformation of the wrought material. The signs of the stresses/strains have been reversed for data taken during compression to ease comparison. The dashed line at ~ 415 MPa represents the point at which the

strain in the tensile sample reached ~11%, equivalent to the total strain achieved during compression, to aid in comparison.

To that point, the phase strains are symmetric with respect to tension and compression. There is no observable non-linearity in the lattice strains near the macroscopic yield point. The slopes of the longitudinal and transverse strains in the elastic region correspond to elastic moduli of 201 GPa and Poisson's ratio of 0.28, roughly what is expected for austenitic stainless steel.[112] At large tensile strains, the phase strain begins to curve upward slightly, increasing in slope to 313 GPa over the final 4 points and unloads with a slope of 223 GPa (not shown for clarity). The increase of modulus during unload is likely due to the evolution of the (111) texture at high tensile strains [113-115], while the larger increase during loading above 600 MPa suggests that stress is being shed from the portion of the material being probed by diffraction so some other portion of material (e.g. grain or sub-grain boundaries or possibly ferrite)

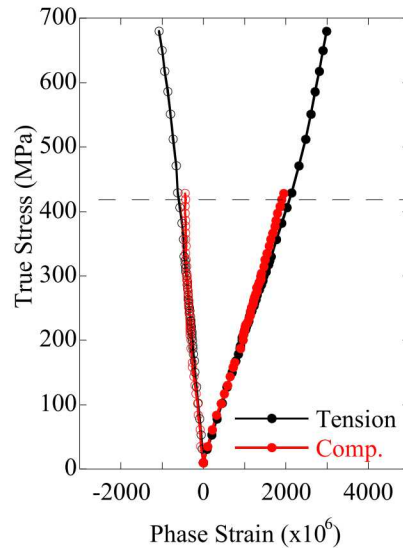


Figure 52. Austenitic true stress vs. lattice strain in wrought material in tension and compression. Filled symbol = longitudinal; unfilled symbol = transverse.

Figures 53(a-d) show the phase strains vs applied stress plots for both phases (austenite and ferrite) for material deposited with 2.0 kW (3.8 kW) laser power in compression, Figure 53(a,b) and tension, Figure 53(c,d). In the case of the compression tests, the axes have been reversed such that the longitudinal stress/lattice strain always goes up and to the right. The dotted lines represent the linearly elasticity limit. Once again, the dashed lines represent the point at which

the strain in the tensile samples exceeds that in the final strain achieved in the compression samples (11%), to aid comparison over like regimes.

In the elastic region, the elastic moduli of the 2.0 kW material in tension (sample A23) and compression (sample A9) and the 3.8 kW material in compression (sample A6) are all slightly higher than that of the wrought materials, roughly 215 MPa. This is likely due to the weak textures in these materials with higher (111) and (110) poles densities along the loading direction [113-115] compared to the wrought material. Note that the elastic modulus of the 3.8 kW material in tension (sample A22) is markedly less (~163 GPa) than the other materials. This is a result of the anomalous observed (100) texture of this build. Calculations of the elastic response with this specific texture resulted in a modulus of 165 GPa.[116] Within the significantly larger uncertainty, the ferrite elastic response is not different from the austenite below the elastic limit.

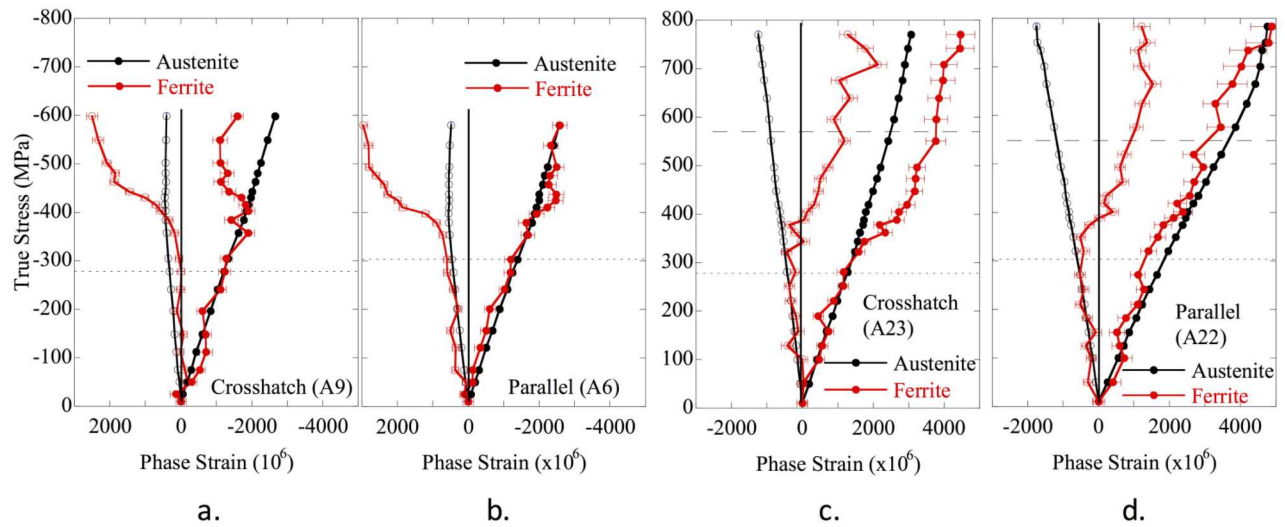


Figure 53. Austenite and ferrite true stress v. lattice strain in AM material in tension and compression. Filled symbol = longitudinal; unfilled symbol = transverse.

Once plasticity is initiated, the responses of the two phases deviate from linearity, although the deviation of the austenite is subtle on the scale of the plots. In the 3.8 kW material, the longitudinal ferrite strain tracks closely (near uncertainty) with the austenite strains. In the lower power deposited material, the longitudinal ferrite strains do deviate significantly from the austenite strains. During compression of the 2 kW material, the elastic strains in the ferrite saturate, indicating that the stress carried by the ferrite does not increase as the surface tractions

on the sample continue to increase. In contrast, during tensile deformation of the 2 kW material, the ferrite strain increases at an increase rate beyond ~ 300 MPa. Again, the austenite longitudinal strains deviate concomitantly with and in opposite direction to the ferrite strains.

The deviation from linearity of the strains in both phases is much more marked in the transverse direction. During compression, regardless of deposition power, the transverse strains in the ferrite increase rapidly (in a tensile sense) above the yield point. The transverse austenite strains saturate (become vertical) at this point. During tensile deformation beyond the yield point, the ferrite transverse strains also deviate markedly from linearity in a tensile sense and the slope of the austenite strains decreases. The asymmetry of the ferrite transverse response is notable. In particular, under both compressive and tensile uniaxial loading, the ferrite transverse strain increases markedly in a tensile sense beyond yield.

7.3.2.6 Orientation Response

Figures 54 shows the austenite hkl-dependent strains in the (a) wrought, (b) 2 kW deposition power, and (c) 3.8 kW deposition power materials as a function of applied compressive and tensile stress. Single peak fitting of the ferrite was not completed. Again, the sign of the stress/strain of the compressive tests have been reversed for ease of comparison. The macroscopic elastic limit is marked in each case by a dotted line.

In all cases, the spread in the strains below the elastic limit are due to the known elastic anisotropy of austenitic steel.[113-116] Beyond the elastic limit of each material in tension, the rate of increase of strains in grains with their (220) and (111) plane normals parallel to the straining direction increases, indicating that those grains are relaxing, i.e. deforming plastically and fail to carry their share of subsequent increases of the applied stress. The slope of the strain in the (200) oriented grains decreases, indicating that this subset of grains carries the load shed from the (220) and (111) grain sets. This situation continues to the completion of the deformation test, although slope of the strain in the (200) grains continues to decrease in the wrought material, while it remains nearly constant in the AM material beyond the elastic plastic transition. The observed load transfer amongst the different grain orientations in tension is the

signature of the $(111)\langle 110 \rangle$ slip system and the pattern is well produced by EPSC models including grain rotation.[112]

The orientation dependent strains observed in compression are markedly different, especially in the AM material. In particular, during the elastic to plastic transition at roughly 450 MPa, corresponding to roughly 0.01 true strain, the strain in the (200) oriented grains saturates, becoming nearly vertical. That is to say, the (200) grains begin deforming plastically. The (311) grain set responds similarly, if to a lesser degree. In response, the (220) and (111) oriented grains again begin accumulating elastic strain supporting subsequent increases in stress. This response is clearly distinct from that observed in tension and can only be reproduced by the EPSC model [112] by altering the elastic anisotropy, i.e. by making the (200) grains the stiffest, which is non-physical. This suggests that a relaxation mechanism is active in (200) oriented grains under compression other than the $(111)\langle 110 \rangle$ slip system, but does not yet suggest what that mechanism is, e.g. slip on a distinct system, twinning, transformation, fracture, etc. We note that a similar, but more subtle difference between tension and compression in the wrought material is also observed. Here, the increase in slope of the (200) strains is very gradual and the slope never increases beyond that of the elastic response. The response in the (220) and (111) may not be outside of uncertainty. In fact, while the (200) strains in compression and tension are different outside of uncertainty, without the result in the AM materials as guidance, that difference may well have not been remarked.

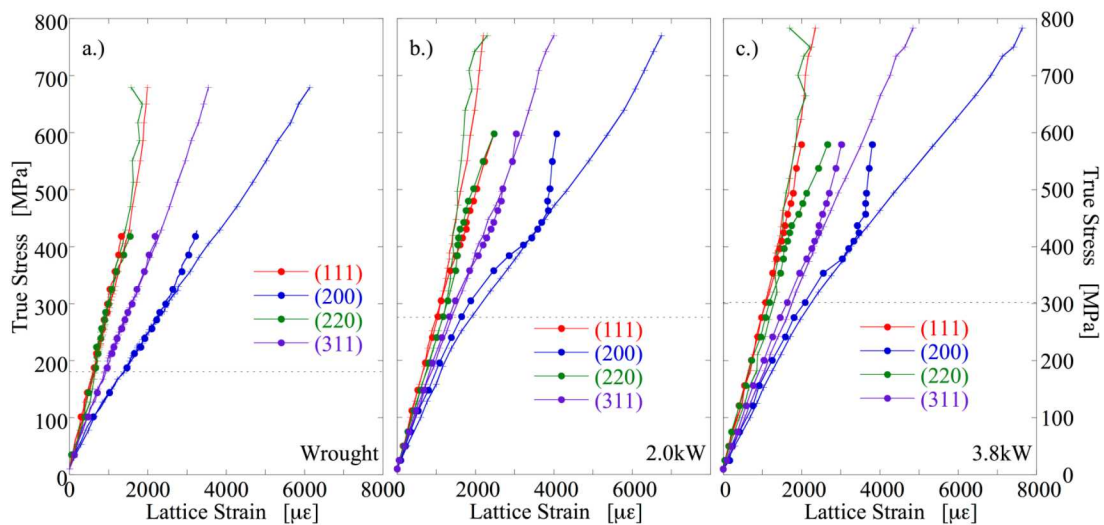


Figure 54. hkl-specific strains in a.) wrought, b.) 2.0 kW, and c.) 3.8 kW AM stainless steel 304L. Results from compressive (circles) and tensile (+) tests are included.

7.4. Discussion

There is a significant difference in flow strength between the wrought 304L steel and its additively manufactured counterpart. A more subtle difference is observed specifically in the initial hardening of the AM material between tension and compression. The results from the in-situ neutron diffraction measurements allow us to look for the microscopic origins of these macroscopic discrepancies. To aid in comparisons between the macroscopic and microscopic, the evolution of the phase stresses during deformation can be calculated from the measured phase strains. To demonstrate this, Figure 55 shows the macroscopic stress (solid line) plotted with the stress in the longitudinal (solid circles) and transverse stresses (open circles) in the austenite phase, calculated from the austenite phase strains during tension and compression of the wrought material. During the entire compression test and tension to ~20% true strain, the phase stress determined from diffraction compares very well with the stress measured by the load cell. Beyond 20% tensile strain, the phases stress begins to deviate from that measured by the load cell. This is likely related to the evolution of the predominantly (111) texture at large tensile strain, which increases the effective polycrystal elastic modulus of the aggregate. That increased modulus is not accounted for in the calculation of the stress from the phase strains results in them being low.

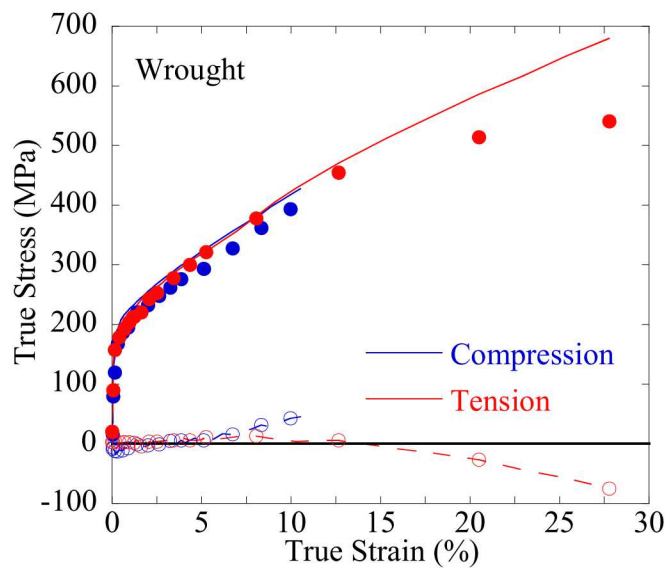


Figure 55. Longitudinal (filled circles) and transverse (unfilled circles) austenite phase stress during compressive and tensile deformation of wrought material. The solid line represents the macroscopic stress.

The transverse phase stresses should be zero as there is no applied transverse stress. However, in both tension and compression, small (< 50 MPa) but non-zero stress components are observed with the same sense as the longitudinal stress, i.e. a transverse compressive stress is observed during the compressive test. In compression, this could be rationalized as due to friction at the compression platens, but no such rationalization is obvious during the tensile test. This could just represent the limit of the accuracy of the stress calculation from diffraction data given the assumptions involved. In general, the diffraction measurement of phase stress satisfactorily reproduces the macroscopic applied stresses.

Figures 56(a-d) show the evolution of the macroscopic and phase stresses (austenite and ferrite) during deformation tests of the AM material. While not shown, in general the weighted sum of the austenitic and ferritic longitudinal stresses ($0.97 \times \sigma_a + 0.03 \times \sigma_f$) closely match the applied stresses reported by the load cell, with tension on the 2.0 kW (sample A23) material being the only exception. The weighted transverse stresses are near zero in all cases.

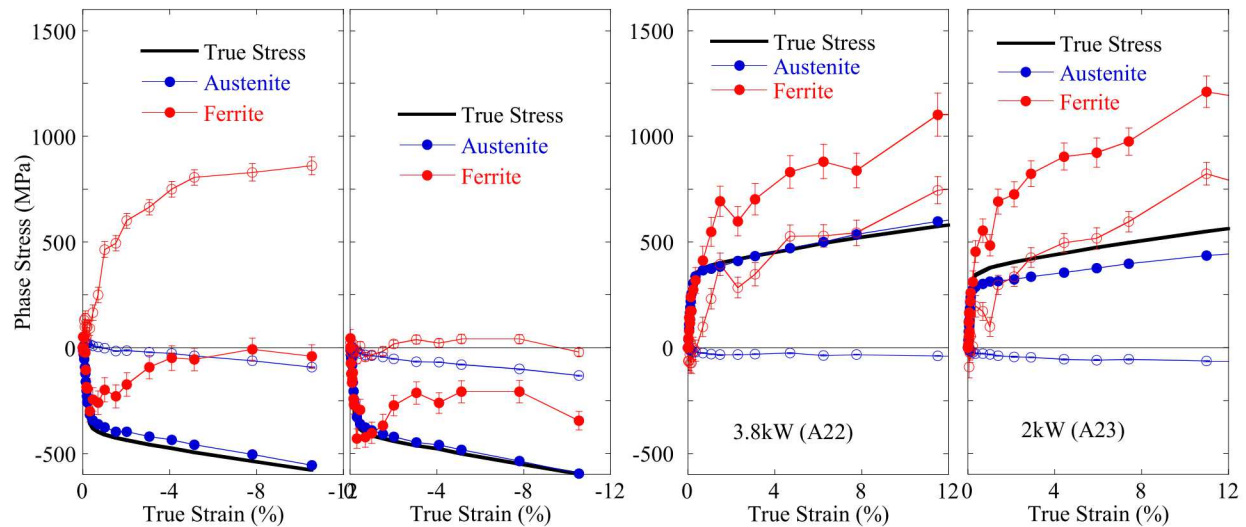


Figure 56. Longitudinal (filled circles) and transverse (unfilled circles) austenite and ferrite phase stress during compressive and tensile deformation of AM stainless steel. The black line represents the macroscopic stress.

From Figures 55 and 56, it is clear that the austenitic phase is simply stronger following additive manufacture than the traditionally wrought material, resulting in the observed increased flow strength. The AM material has a much larger apparent grain size than the wrought material, which would suggest the AM material should be weaker. The increased flow strength is likely due to the increased dislocation density in the AM material as suggested by the increase peak breadth (Figure 55). Detailed line profile measurements on the AM material at various stages of deformation are summarized below.

There are multiple tension/compression asymmetries observed in the response of the microstructure to deformation that may be related to the observed difference in the hardening in tension and compression. Clearly from Figure 56, the transfer of stress to the ferrite is very distinct during tensile and compressive deformation. During tension, the ferrite plays the role of a mild strengthening phase, that is when the austenite yields, it carries increasing longitudinal stress. It is odd however, that a large transverse tensile stress also builds up in the ferrite precipitates with tensile deformation. In contrast, during compression, the ferrite precipitates shed load beyond 300-400 MPa in the longitudinal direction, unloading to nearly zero longitudinal stress as the compressive strain increases. Especially in the case of the 2 kW material, the ferrite supports a large transverse tensile stress when being deformed in compression.

It may be necessary to consider the residual phase stresses in the ferrite which must develop during cooling following deposition. If incipient ferrite is constrained by the surrounding austenite and is in a state of significant hydrostatic compression at room temperature then this may be responsible for the asymmetries. Of course, the austenite must sense a balancing tensile stress locally, but since the volume fraction of ferrite is low, <0.03 , the residual stress in the austenite is likely insignificant when averaged over the entire microstructure such as is done by neutron diffraction.

Figure 57 shows schematics which attempt to present the situation in the ferrite at different stages. Note: this assumes that ferrite is in a hydrostatic state of compression which may come from the combined effects of cooling after resolidification, phase transformations and other

effects. We do not have a quantitative number for that stress. The middle schematic in the figure illustrates what the diffraction results could indicate following -0.1 compressive strain. Recall, the residual stress prior to deformation was explicitly ignored, so stress state shown schematically is relative to the as-deposited state. At -0.1 compressive strain, with -550 MPa on the surface of the sample essentially zero longitudinal stress is transmitted to the sample, while 800 MPa of tensile stress is perceived in the transverse direction. Similarly, at 0.1 tensile strain with a tensile stress of 500 MPa on the surface of the sample, the ferrite perceives a nearly hydrostatic stress state with 800 MPa longitudinal and 500 MPa transverse stress. While the neutron diffraction provides us with the means to observe these results, a deeper understanding likely requires coupling to a detailed finite element analysis [116] or 2-site elastic plastic self-consistent model [117] which explicitly accounts for the transformation stresses.

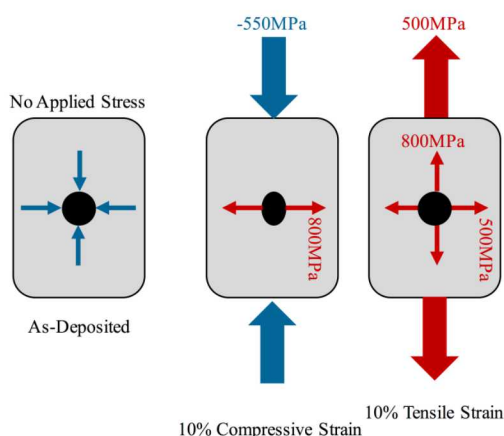


Figure 57. Envisioned state of ferrite at different stages including mechanical testing.

The hkl-dependent response of the austenite was also noted to be asymmetric with respect to tension and compression, Figure 54. Specifically, saturation of strains (200) oriented grains during compression indicates activation of a relaxation mechanism that is not activated during tensile deformation and cannot be reproduced by an EPSC model accounting for only (111)<100> slip.[112] Unfortunately, the current data does not allow for identification of the relaxation mechanism that is active during compression of (200) grains specifically. Deformation twinning is very orientation dependent and polar (acting only in tension or compression along specific directions [118]) and (111)<112> has been observed in high-nitrogen 304LN stainless steels. However, due to the nearly random initial texture and 6-fold symmetry

of the twin system, the characteristic texture signature of deformation twinning that is so easy to observe in hexagonal metals [119] is unlikely to be observed in this material. A very small increase in the ferrite phase fraction was observed with large tensile strains, suggesting that stress induced phase transformation does not play a role. It is tempting to associate the inability of the ferrite phase to carry longitudinal stress during compression with the observed relaxation of the austenitic (200) oriented grains because they both occur during the elastic-plastic transition. To link the asymmetric ferrite phase response to the orientation-dependent response of the austenite, the ferrite phase would have to grow as platelets or needles on preferred austenitic planes. Microscopy studies are currently being undertaken to look for this. Moreover, it seems likely that the differences observed in the microscopic response between tension and compression must be related to the asymmetric initial hardening exhibited by the AM material.

With regards to strengthening mechanisms, there remain two: dislocation density effects and solid solution strengthening. Returning to the data in Figure 51, we have plotted in Figure 58(a) the flow stress as a function of the measured dislocation densities. The flow stress of the un-deformed samples is taken as the yield stress as no increase in the dislocation density is expected in the elastic region.

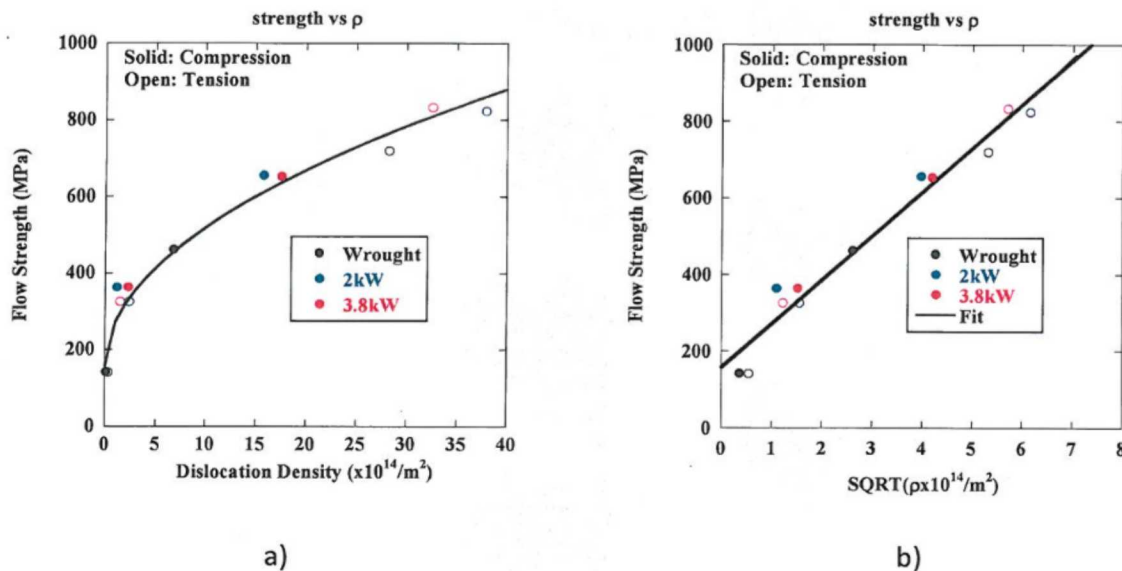


Figure 58. (a) Measured flow stress versus measured DLPA dislocation densities. (b) Measured flow stress versus square root of measured DLPA dislocation densities. The yield stress was used as the flow stress for the as-built samples.

It is obvious that the strength data for the both the AM and wrought reference material fall on the same curve. Figure 58(b) shows the same data but plotted against the square root of the dislocation density to follow a Taylor like relationship between the flow stress and the dislocation. Again it is seen that the data follows the same linear behavior. The correlation is quite good which strongly suggests that the higher flow strength of AM material is due to its higher dislocation density.

Solid solution strengthening is a second possible mechanisms that underlies the higher strength of the AM stainless steel 304L. Review of Tables 3, 4 and 6 show that the AM steel had nearly double the concentration of nitrogen compared with the wrought form, likely due to the powder being nitrogen atomized. A nitrogen concentration of ~0.08 wt.% for the AM form vs. ~0.04 wt.% in the wrought is considered significant. Nitrogen acts both as an austenite stabilizer but also as a potent solid solution strengthener.[99,100] The effect on yield strength has been shown to be linear, and follow the form of:

$$\sigma_y = \sigma_o + \frac{\mu}{K} X_n \quad 7-7$$

where σ_o is the nitrogen-free yield strength, μ the shear modulus, K a constant, and X_n is the atomic fraction of nitrogen. The effect at room temperature is quite potent. For example, increasing nitrogen content in a stable austenitic stainless steel from 0.04 wt. % N to 0.36 wt. % increases the yield strength from approx. 205 MPa to 375 MPa. A change from 0.04 wt. % to 0.08 wt. % nitrogen will likely increase the yield strength in a 304L approximately 40-50 MPa.[96] Similar results were seen when comparing fully annealed wrought 304L and the 2 kW AM deposits. When annealed at 1110°C for one hour to exclude any effect of residual stress, the hardness of the AM deposit was Rb 77.0 vs. the wrought material at 71.1 Rb. The only remaining strengthening mechanism between these materials in the fully annealed condition is the higher nitrogen content in the AM deposit.

8. DAMPING PROPERTIES

Due to the different microstructures of AM material versus wrought material, the two materials may exhibit different damping behavior. This chapter gives some preliminary results into comparing the damping properties of both forms.

Material damping is the property of a material to irreversibly convert mechanical work into heat or other forms of non-recoverable stored energy. *Structural* damping consists of both material damping and other damping mechanisms such as joint friction, air damping, thermoelastic damping, viscous energy absorbers, viscoelastic layers, etc. In mechanical design, high-damping materials and structures are generally favored for reduced vibrational response and less severe component environments. In polycrystalline materials, material damping arises from several mechanisms including point-defect relaxation, dislocation motion, and grain-boundary sliding.[120,121] Material and structural damping mechanisms are typically a function of frequency and strain amplitude. Waves propagating within a polycrystalline material also experience dispersion due to scattering at grain boundaries.[122] An additional structural damping mechanism, known as thermoelastic damping (often called elastothermodynamic damping), is also present in vibrating structures due to the thermoelastic effect. Whenever a material is stressed adiabatically, there is in general a small change in temperature, typically less than 1°C. In a structure with a heterogeneous stress field, the resulting temperature field will also be non-uniform, and heat will conduct from the high-temperature regions to the low-temperature regions. This irreversible heat conduction is manifested as a conversion of mechanical work into heat, or damping. Thermoelastic damping is frequency dependent, but is independent of the strain amplitude.

For a given structure, the measured damping can be due to several damping mechanisms simultaneously. In order to compare the intrinsic damping of two materials, it is important to remove other sources of damping that are not intrinsic to the material such as air damping and thermoelastic structural damping. Air damping can be removed by performing damping experiments in a vacuum. Frictional damping in fixturing devices can be minimized by using

certain types of optimized support structures.[123,124] A thorough damping investigation would require careful examination of these effects. Here, our goal was to get an initial assessment of the difference in damping behavior of 304L in the wrought condition and manufactured using DED.

8.1 Test Overview

A modal comparison was performed between two specimens (parts 1 and 2) of wrought 304L and three AM 304L specimens (parts 3, 4, and 5). These samples are shown in Figure 59. Each specimen has nominal dimensions 50 x 10 x 2 mm. Modal results from the wrought samples will be identified with the letter (W), and AM samples will be identified with (AM).

Figure 60 shows the experimental modal setup. The test articles were placed on soft foam. A small instrumented modal hammer was used to excite each specimen. A solenoid actuator was used to swing the hammer. The actuator was triggered using a square wave output from the data acquisition system. This provided repeatable excitation to the structure. A PSV-500 Scanning Laser Doppler Vibrometer was used to measure the response of each sample. Frequency response functions were computed from the measured excitation force and responses at 28 points on the surface of each test article (see Figure 61). Modes were fit to these frequency response functions using the SMAC algorithm.[125] Natural frequencies, critical damping ratios, and mode shapes were fit to the data. These values are reported in Table 8.

A cantilever test configuration was also investigated as shown in Figures 62 and 63. Test data were not as clean for this test configuration for several reasons. Primarily, the impact hammer almost exclusively delivered multiple hits to the test article rather than one single hit in the test window. With the test article clamped, it could not move away from the hammer when it was struck, so the part tended to recoil back into the hammer. The spectrum of a multiple hit impact looks like hills with deep valleys between them at a number of frequency lines, and at these valleys there is very little excitation energy which also results in a low signal to noise ratio. For this reason, damping results are not reported for this test configuration.

8.2. Modal Results

Modal parameters were extracted from test data up to 50 kHz. These parameters are shown in Table 8. For each mode, the natural frequency and damping ratio are listed. Consistently less damping was observed in the AM material compared to the wrought steel, 1-10% in the first bending mode. For the bending modes, the AM material tended to be less stiff (lower natural frequencies), but for the torsion modes the AM material tended to be more stiff (higher natural frequencies). Additionally, the mode ordering changed near 50 kHz, with the fifth bending mode appearing before the fourth torsion mode in the AM material but not in the wrought material. Data was fairly consistent between parts of the same type which gives confidence in the results.

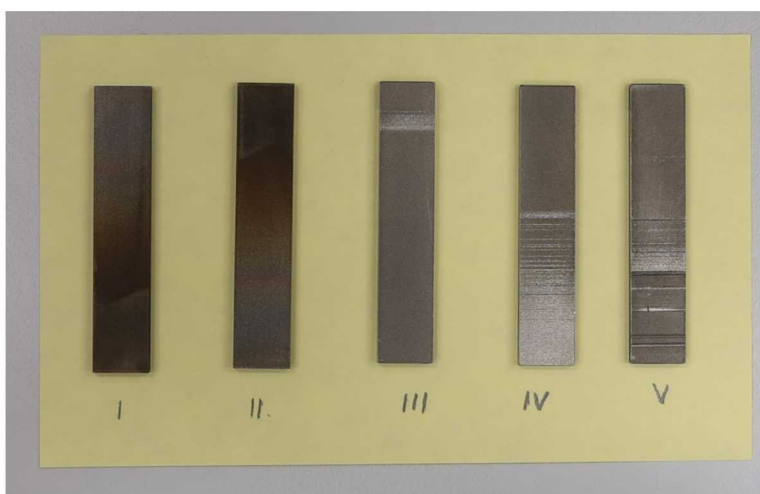


Figure 59. Five specimens used to compare the wrought material (parts 1 and 2) to the AM SS 304L (parts 3, 4, and 5). Nominal specimen dimensions are 50x10x2 mm.



Figure 60. Test setup showing (a) the laser vibrometer and (b) impact hammer setup.

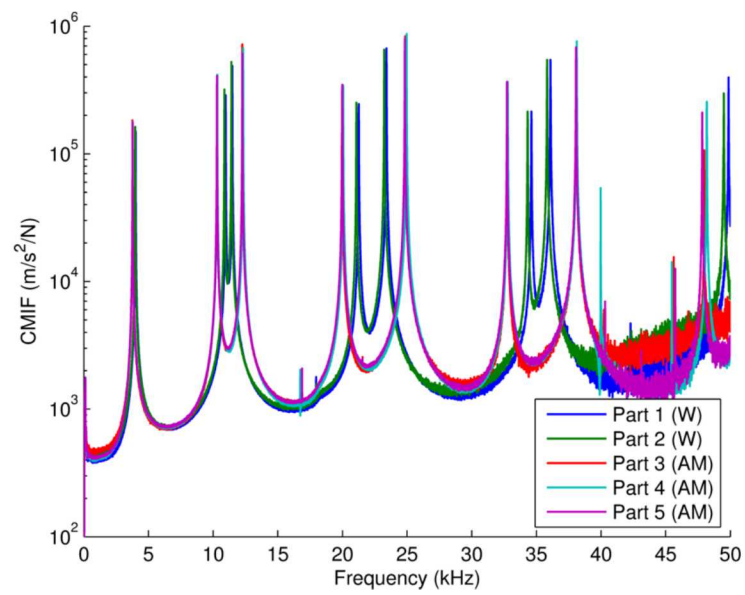


Figure 61. Complex modal indicator function vs. frequency (foam support configuration).

Table 8. Extracted modal parameters including frequency (Hz) and damping ratio. W = wrought. AM = Additively manufactured.

part number	first bending		second bending		first torsion		third bending		second torsion		fourth bending		third torsion	
part 1 (W)	4006	0.10	10970	0.046	11490	0.034	21270	0.038	23410	0.027	34620	0.027	36090	0.027
part 2 (W)	3974	0.10	10870	0.040	11420	0.035	21090	0.036	23240	0.027	34320	0.025	35830	0.027
part 3 (AM)	3763	0.092	10300	0.035	12260	0.026	19980	0.031	24860	0.021	32740	0.021	38080	0.017
part 4 (AM)	3769	0.098	10320	0.034	12330	0.024	20060	0.032	24970	0.021	32790	0.021	38130	0.017
part 5 (AM)	3778	0.10	10300	0.036	12260	0.029	19990	0.032	24830	0.024	32730	0.021	38070	0.021

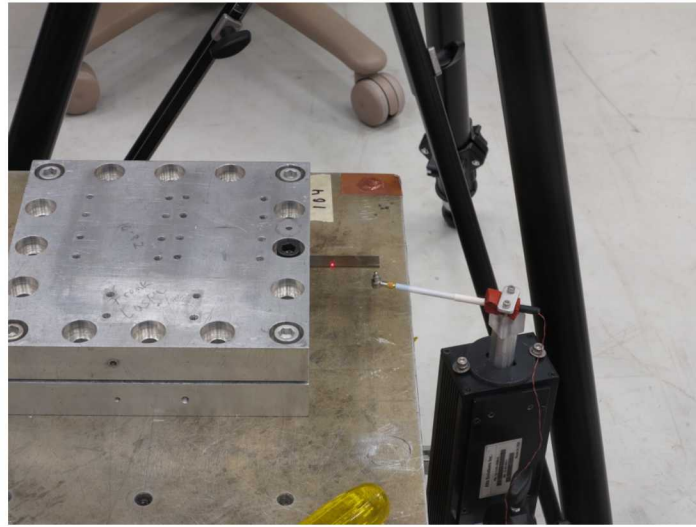


Figure 62. Test setup showing cantilever configuration and impact hammer.

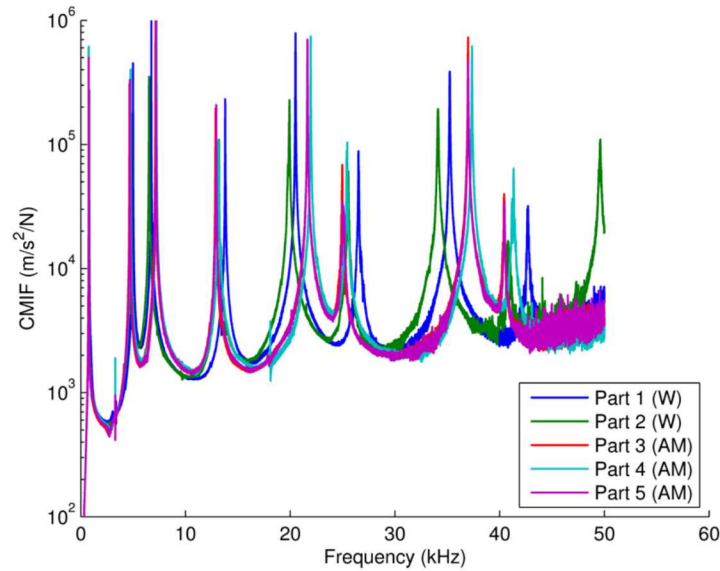


Figure 63. Complex modal indicator function vs. frequency (cantilever configuration).

9. DYNAMIC MECHANICAL RESPONSE

9.1. Experimental Methods

9.1.1. Test Apparatus

Dynamic compressive and tensile experiments were performed with Kolsky compression and tension bars, respectively. Figure 64 shows a schematic of the Kolsky compression bar apparatus. In this study, the Kolsky compression bar system was made of Maraging C350 steel and had a common diameter of 19.05 mm. The striker, incident, and transmission bars were 0.61, 3.66, and 1.83 m long, respectively. The striker was launched with a gas gun to impact the incident bar, generating a compressive stress wave (incident wave) propagating in the incident bar until it arrived at the specimen. Due to the lower mechanical impedance of the specimen than the pressure bars, part of the incident wave was reflected back into the incident bar and the rest transmitted through the specimen into the transmission bar while the specimen was being compressed. As indicated in Figure 64, a double pulse shaping technique [126], where an annealed C11000 copper disk was stacked on a larger-diameter work-hardened steel disk on the impact end of the incident bar, was applied to generate a desired shape of incident pulse to achieve early dynamic stress equilibrium and nearly constant strain-rate deformation in a 304L stainless steel specimen.

Figure 65 shows a typical oscilloscope record of incident, reflected, and transmitted signals generated in such a double-pulse-shaped Kolsky compression bar test of a wrought 304L stainless steel longitudinal specimen. Through utilization of the double pulse shaping technique, the incident pulse was generated as a typical initial elasticity followed by a work hardening behaviour, which is similar to the transmitted pulse, leading to a reflected pulse with a plateau. Figure 66 shows a comparison of stresses at both ends of the specimen, which were calculated with the following equations, respectively, where ε_i , ε_r , and ε_t are incident, reflected, and

transmitted bar strains, respectively; E_0 is Young's modulus of the bar material; A_0 and A_s are cross-sectional areas of the bars and the specimen, respectively.

$$\sigma_1 = \frac{A_0}{A_s} E_0 (\varepsilon_i + \varepsilon_r) \quad (9-1)$$

$$\sigma_2 = \frac{A_0}{A_s} E_0 \varepsilon_t \quad (9-2)$$

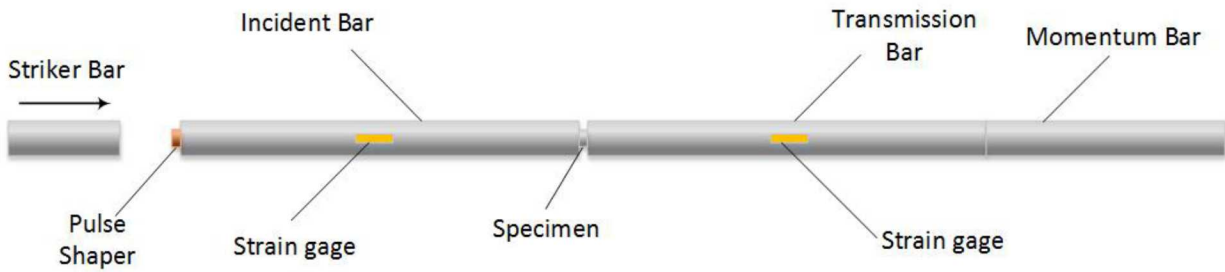


Figure 64. Schematic of Kolsky compression bar.

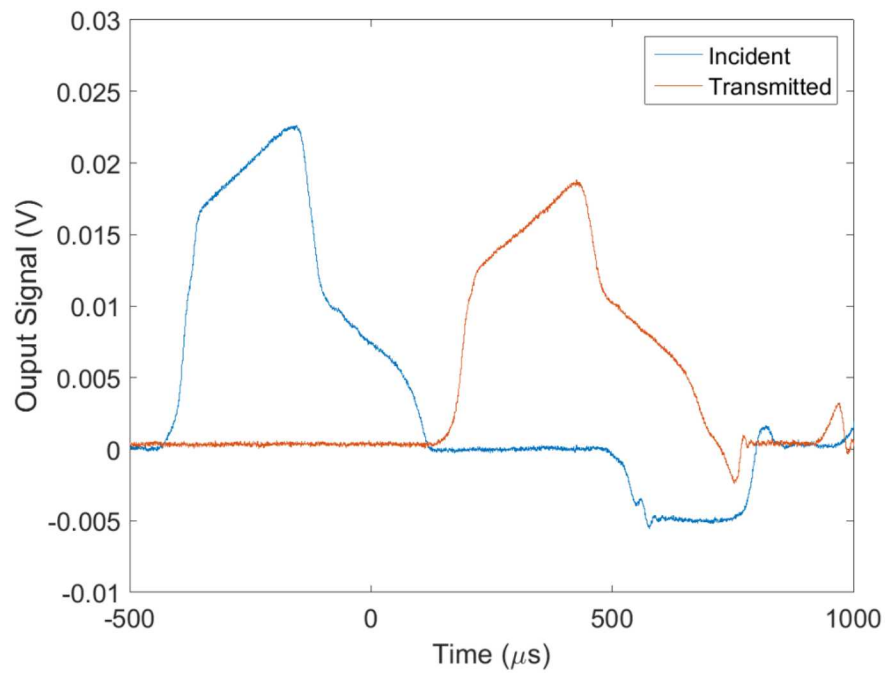


Figure 65. A typical set of incident, reflected and transmitted signals.

The nearly overlapped stress histories at both ends of the specimen (Figure 66) indicate that the specimen was equilibrated in stress over the nearly entire duration of dynamic compressive loading. The specimen stress can thus be calculated with either Eq. (9-1) or Eq. (9-2). Then the strain rate and strain in the specimen are calculated as,

$$\dot{\varepsilon} = -\frac{2C_0}{L_s} \varepsilon_r \quad (9-3)$$

$$\varepsilon = -\frac{2C_0}{L_s} \int_0^t \varepsilon_r dt \quad (9-4)$$

where C_0 is the elastic stress wave speed in the bar material; L_s is the gage length of the specimen. Equation (9-3) indicates that a plateau in the reflected pulse represents a constant strain rate in the specimen, due to the application of appropriate double pulse shaping technique.

Figure 67 shows typical stress and strain histories in the specimen calculated with Eqs. (9-2) and (9-4), which were used to calculate stress-strain response by eliminating the term of time. In addition, the linear strain history in the specimen indicates a constant strain rate of 500 s^{-1} .

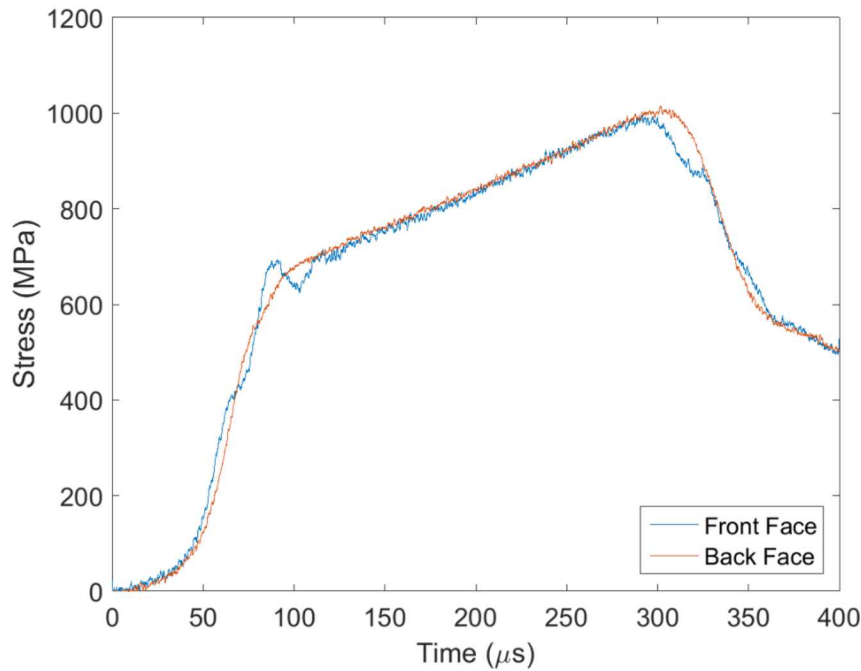


Figure 66. Dynamic stress equilibrium and strain rate history.

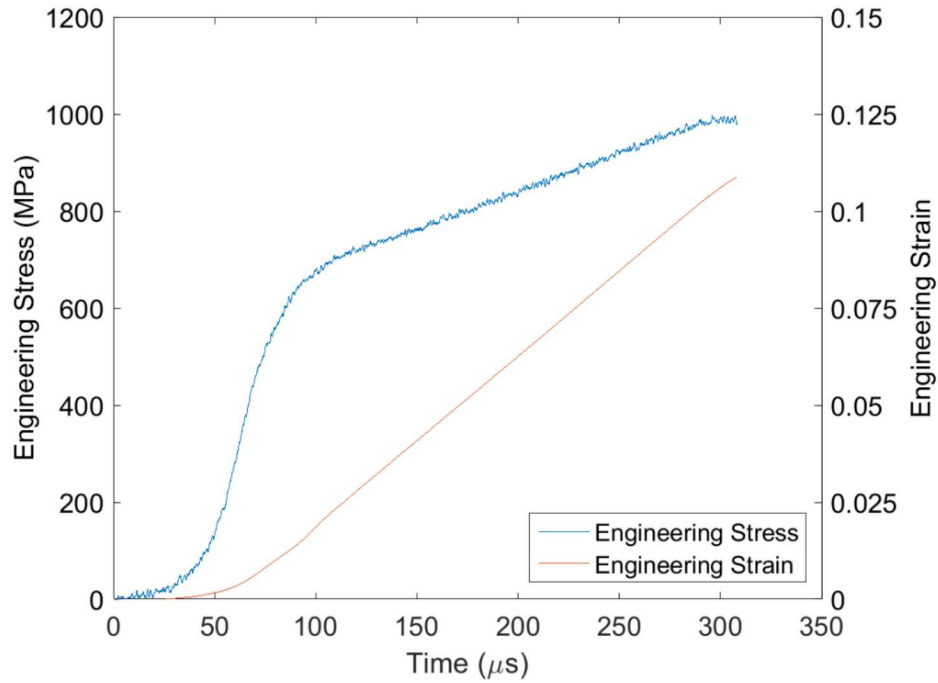


Figure 67. Stress and strain histories in the specimen.

Elastic indentation in a Kolsky compression bar test has been recognized to significantly affect the measurement of stress-strain response at small strains, i.e., Young's modulus. When the specimen is dynamically loaded in compression by the pressure bars, the smaller specimen may generate an indentation, even though in elasticity, to the bar ends.[127] This elastic indentation makes the stress wave at the bar/specimen interface no longer planar, which makes the specimen strain over estimated through the conventional data process (Eq. 9-4). The over-estimated specimen strain results in an underestimated Young's modulus of the specimen material. Use of the pulse shaping technique minimizes stress wave dispersion and ensures early dynamic stress equilibrium, but does not correct the elastic indentation. Most recent research indicates that a combination of a pulse-shaped experiment and a numerical indentation correction significantly increases the accuracy of Kolsky compression bar experiments on small-strain compressive stress-strain response.[128] In this study, the indentation correction method developed by Safa and Gary [127] was applied to correct the indentation effect on the compressive stress-strain response of the 304L stainless steels.

The Kolsky tension bar system used in this study was made of Maraging C300 steel. The 25.4-mm-diameter incident and transmission bars are 3.62 and 2.13 m long, respectively. As shown in Figure 68, a striker was launched to impact the end cap of the gun barrel, generating a tensile stress wave in the gun barrel. The tensile stress then transmits into the incident bar that was connected to the gun barrel via a coupler. Similar to the Kolsky compression bar test, a small annealed C11000 copper disk was placed on the end cap to generate a specific incident pulse to achieve stress equilibrium and constant strain rate deformation in the specimen. However, differing from the Kolsky compression bar test, the tensile specimen needs to be threaded into the bar ends, which generates numerous interfaces to disturb the stress wave propagation. In this study, we followed previous Kolsky tension bar experimental procedures to 1) apply lock nuts to the specimen to minimize the pseudo stress peak in the resultant stress-strain response [129]; 2) apply a custom-made laser extensometer to directly measure the specimen displacement [130]; and 3) numerically correct the displacement over the gage section of the tensile specimen.[131,132] All detailed procedures have been presented in Refs. 131 and 132.

Figure 69 shows a typical set of oscilloscope records of strain gage and laser extensometer signals obtained from a Kolsky tension bar test of a wrought material along its longitudinal direction. The tensile stress history was calculated with the same equation (Eq. 9-2); whereas the strain history was calculated with the laser measurements by using the following equation [131,132],

$$\varepsilon = \begin{cases} c' \cdot \frac{L_1 - L_2}{L_s} & (\sigma \leq \sigma_y) \\ \frac{L_1 - L_2 - (1 - c')(L_1 - L_2)_{yield}}{L_s} & (\sigma > \sigma_y) \end{cases} \quad 9-5$$

where L_1 and L_2 are displacements at the front and back ends of the specimen, respectively; σ_y is yield strength of the tested materials; c' is the correction factor for specimen strain calculation at gage section. According to the design of the tensile specimen, the correction factor (c') has been determined as 0.62 in this study.[132] Figure 70 shows the calculated stress and strain

histories in the tensile specimen during dynamic tensile load. Similar to the compression test, the nearly linear strain rate in the specimen indicates a nearly constant strain rate of 3000 s^{-1} during dynamic tensile loading.

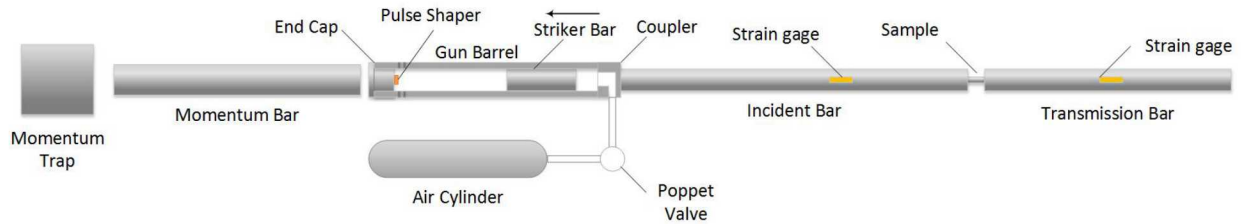


Figure 68. A schematic of Kolsky tension bar.

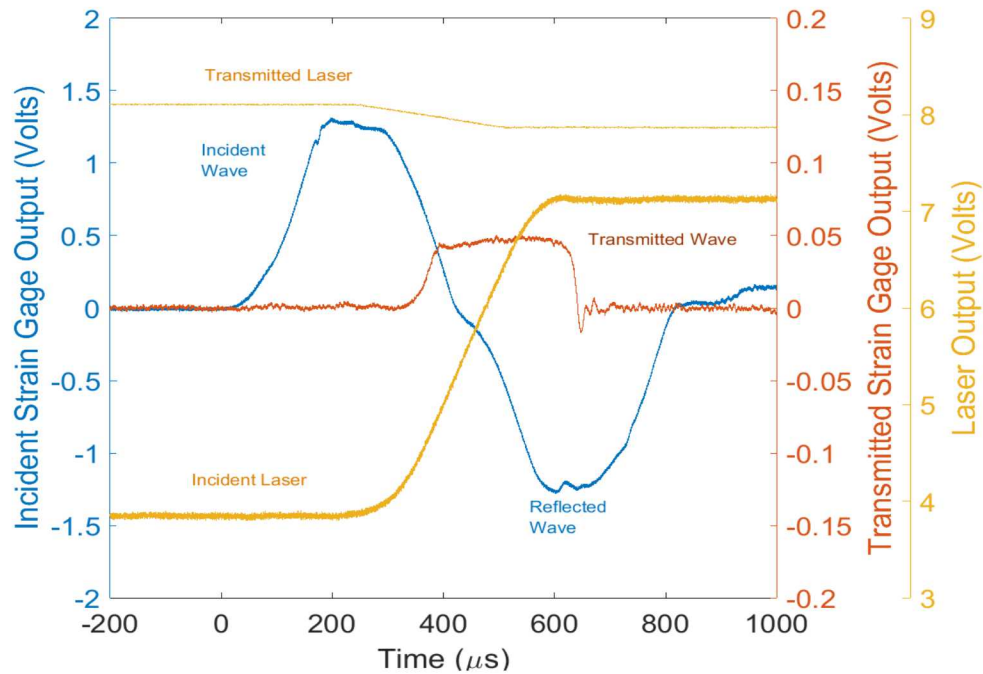


Figure 69. A typical set of strain gage and laser extensometer signals from a Kolsky tension bar test.

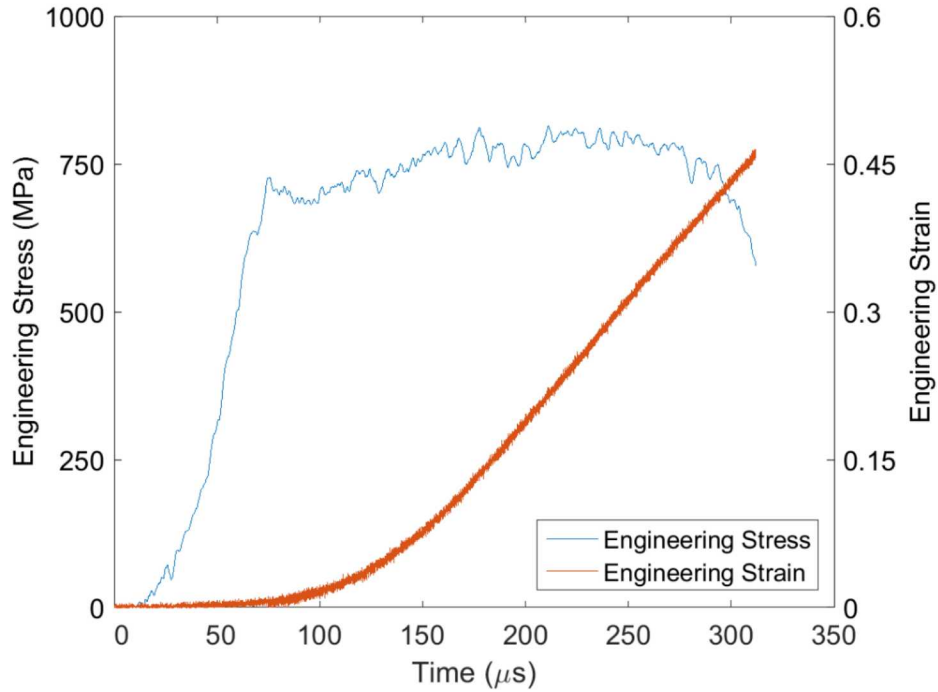


Figure 70. Tensile stress and strain histories in the specimen.

9.1.2. Specimen Geometry

Table 9 summarizes the detailed information of material processing and testing conditions. To reiterate, the orientation shown in Table 9 can be interpreted as: X-direction means that the loading (either compression or tension) direction is along the laser travel direction; Y-direction means that the loading direction is in-plane perpendicular to the laser travel direction; and Z-direction means the loading direction is along the layer built-up direction. It is noted that the orientations shown in Table 9 indicate the directions to which the dynamic loading is applied. In order to compare the dynamic response between the AM material to the wrought material, the latter was also dynamically characterized in compression and tension, along longitudinal and transverse directions.

Both AM and wrought 304L stainless steels were made into cylindrical specimens with a diameter of 6.35 mm and a thickness of 3.18 mm for dynamic compression tests. The specimens for dynamic tensile tests were made into a cylindrical dog bone shape with a gage section of 3.18 mm in diameter and 6.35 mm in gage length. Appendices 13.13 and 13.14 show the schematic

drawings for both compression and tensile specimens. The compression specimen was sandwiched between the incident and transmission bars for dynamic compression tests. The tensile specimen was directly threaded into the ends of the incident and transmission bars for dynamic tensile tests.

Table 9. Description of materials tested in compression and tension at high strain rates.

Material	Laser Power, Process	Orientation	Post-processing	Testing Condition (s ⁻¹)		Specimen Location
AM 304L	2.0 kW Cross-hatched	X	Annealed	Compression	~500	N/A
					~1500	
					~3000	
				Tension	~3000	
		Z	As-deposited	Compression	~500	Center
					~1500	Edge
					~3000	Center
				Tension	~3000	Edge
						Center
						Edge
	3.8 kW Parallel-hatched	X	Annealed	Compression	~500	N/A
					~1500	
					~3000	
				Tension	~500	
			~1500			
			~3000			
			As-deposited	Tension	~3000	
				Y	Annealed	
		~1500				
		~3000				
Tension	~3000					
Wrought 304L	N/A	Longitudinal	As-deposited	Compression	~500	N/A
					~1500	
					~3000	
				Tension	~500	
					~1500	
					~3000	
		Transverse		Compression	~500	
					~1500	
					~3000	
				Tension	~500	
~1500						
~3000						

9.2. Results

9.2.1. Compression Tests

Following the same procedure, the AM 304L stainless steel and wrought material listed in Table 9 were dynamically characterized in compression and tension. Three different strain rates (~ 500 , 1500 , and 3000 s^{-1}) were conducted in compression; whereas, dynamic tensile tests were performed only at the strain rate of $\sim 3000 \text{ s}^{-1}$ except for the 3.8 kW , parallel hatched, X-direction, annealed specimens, and the wrought specimens that were conducted in tension at three strain rates: ~ 500 , 1500 , and 3000 s^{-1} . At each loading condition, 3 to 5 experiments were repeated for consistency check. Figure 71 shows an example of compressive stress-strain curves from 5 experiments on wrought 304L stainless steel along transverse direction at the same strain rates. The compressive stress-strain curves show high consistency and repeatability. A mean curve was thus calculated as the representative curve at this testing condition, also shown in Figure 71. In this study, the mean curves were used for wrought 304L stainless steel, however, the individual stress-strain curves for each AM 304L stainless steel were used for consistency/repeatability check due to possible scattered microstructures generated in the AM process.

Figure 72 shows the dynamic compressive stress-strain curves of wrought 304L stainless steels along longitudinal and transverse directions at various strain rates. All compressive stress-strain curves exhibit very similar elastic-plastic characteristics with significant sensitivities to strain rates. The dynamic stress-strain curves showed very slight difference for the specimens along longitudinal and transverse directions at the same strain rates. At the same strain and strain rate, the flow stress in the specimen along longitudinal direction was a little higher than that along transverse direction. At the similar strain rates, the wrought 304L stainless steel shows little difference in stress-strain response along longitudinal and transverse directions. Figure 73 shows typical pictures of wrought specimens after dynamic compression tests. The specimens after dynamic compression tests still maintained a circular shape with reasonably smooth end and side surfaces.

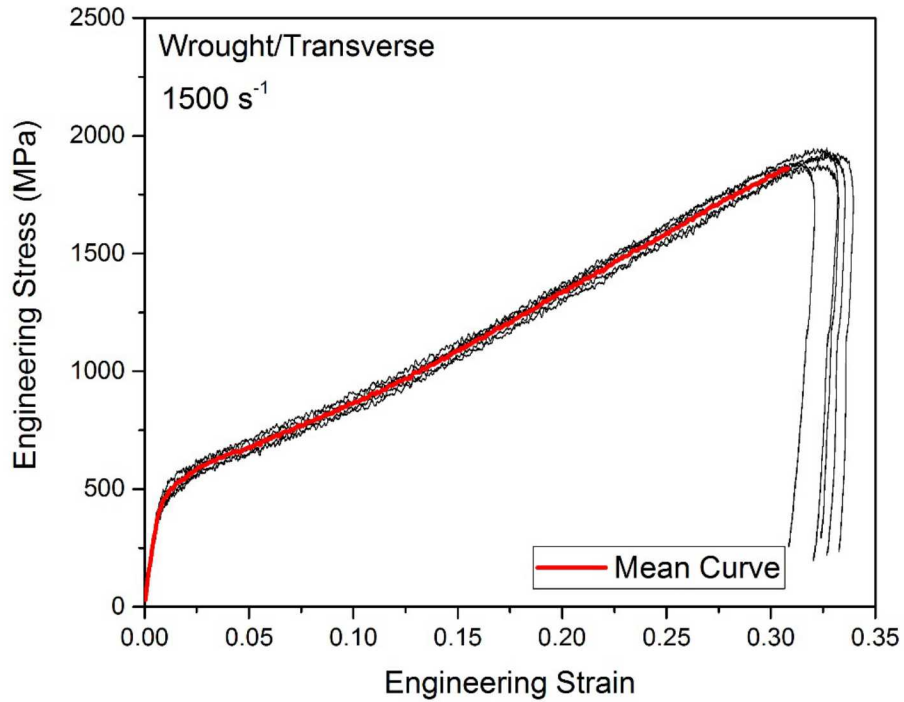


Figure 71. Dynamic compressive stress-strain curves of wrought 304L stainless steel along transverse direction at 1500 s⁻¹.

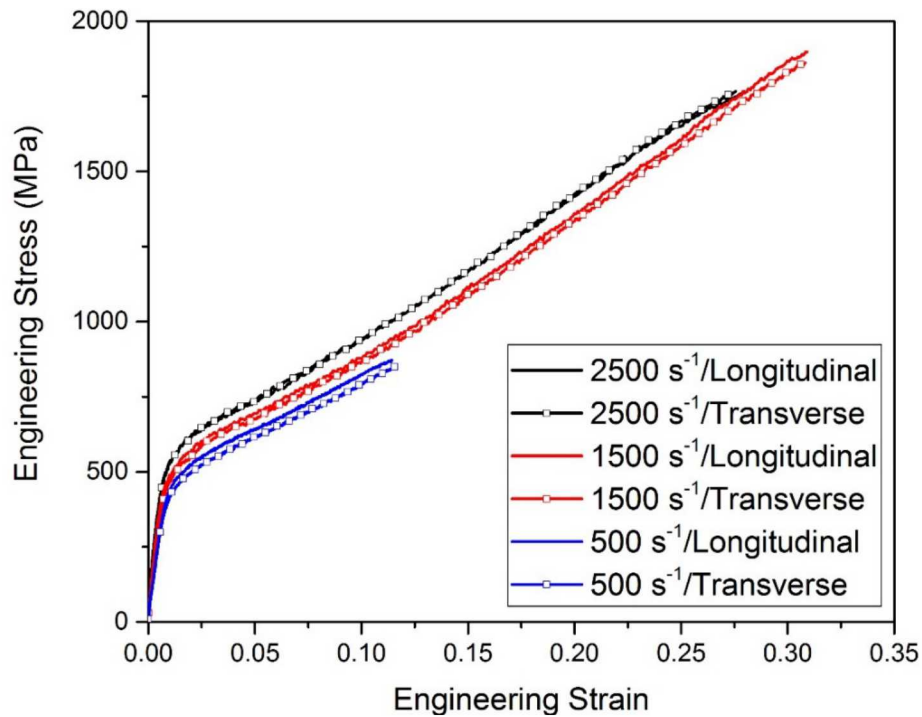


Figure 72. Dynamic compressive stress-strain curves of wrought 304L stainless steel along longitudinal and transverse directions at various strain rates.



Figure 73. Pictures of the wrought specimens after dynamic compression tests. (a) end surfaces; (b) side surfaces.

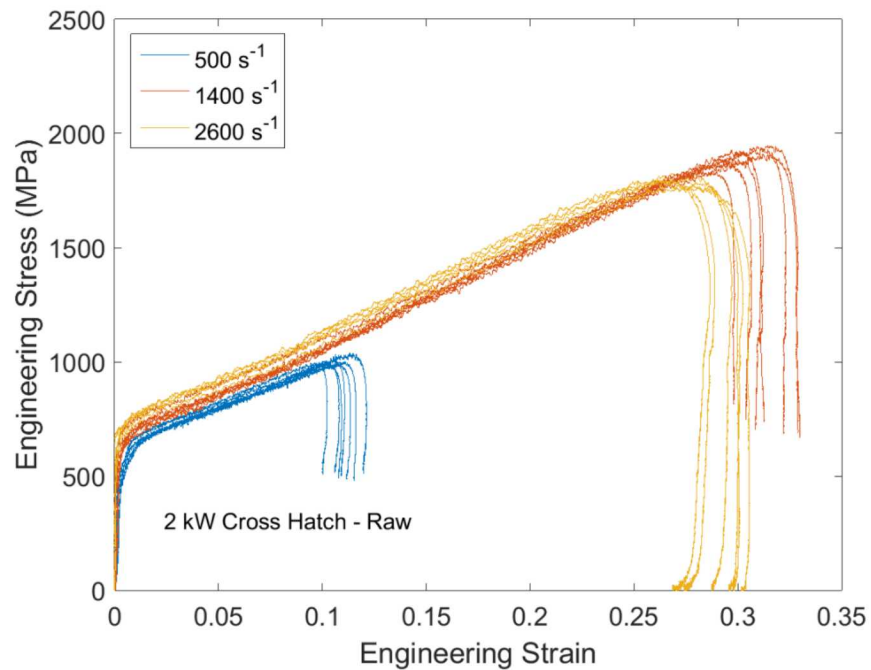


Figure 74. Dynamic compressive stress-strain curves of AM 304L stainless steel built with the 2.0 kW, cross hatched process. Strain rates are listed in the legend.

Figures 74 and 75 show the dynamic compressive stress-strain curves of 2.0 kW AM cross-hatched as-deposited 304L stainless steel along Z direction (Figure 74) and annealed material along X direction with the same laser power (Figure 75). It is noted that Figure 75 also shows the stress-strain curves of the materials made using the same AM process but removed from different locations (edge vs center). However, no difference in the dynamic compressive stress-strain curves was observed for the specimens extracted from different locations. The compressive stress-strain curves do not seem to be significantly different between the annealed X-direction specimens and the as-deposited Z-direction specimens. At the same condition, all stress-strain curves show very similar highly repeatable elastic-plastic compressive response to the wrought materials.

Figure 76 shows the pictures of 2.0 kW cross-hatched, as-deposited AM 304L stainless steel specimens along Z direction after dynamic compression tests. Differing from the wrought specimens shown in Figure 73, the AM specimens did not maintain a perfect circular shape after dynamic compression tests. Instead, deposit puddles seemed to appear on both end and side surfaces after dynamic compression tests. The side surfaces are even rougher than the end surfaces, as shown in Figure 76.

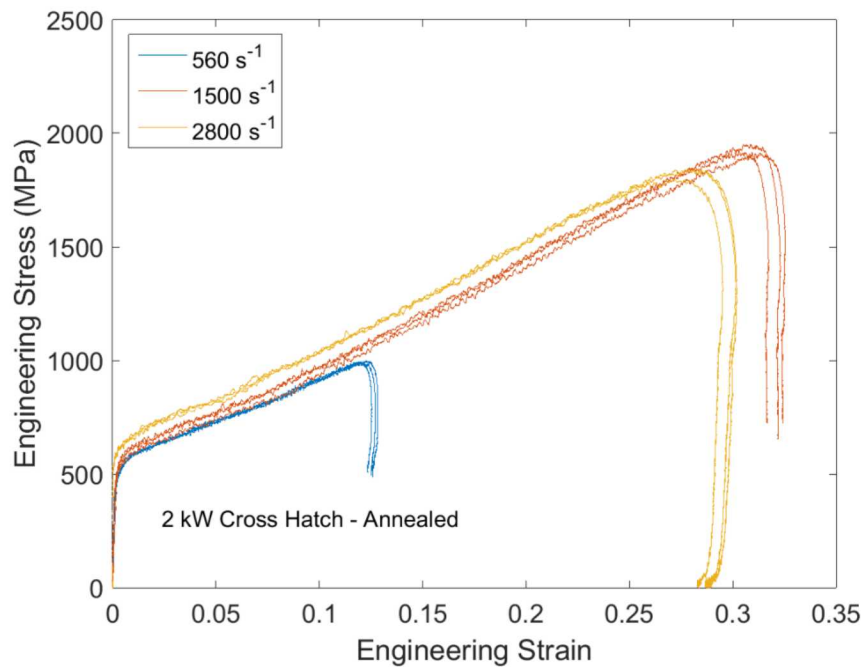


Figure 75. Dynamic compressive stress-strain curves of AM cross hatched (2.0 kW) SS 304L that has been annealed. Strain rates are listed in the legend.



Figure 76. Pictures of the 2.0 kW, cross-hatched AM steel samples (not heated after deposition) after compression tests. (a) end surfaces; (b) side surfaces.

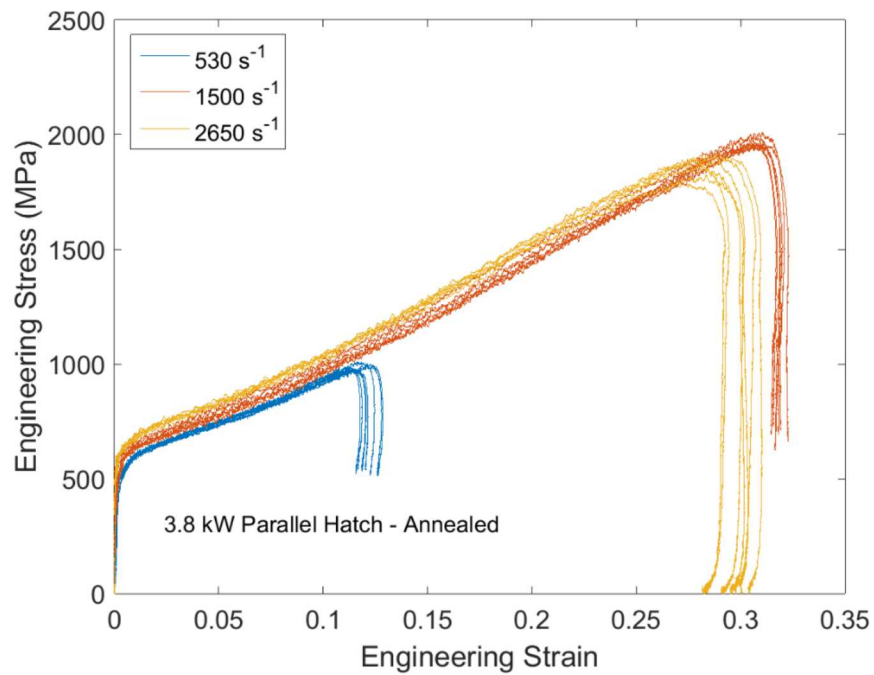


Figure 77. Dynamic compressive stress-strain curves of AM 304L stainless steel made with the 3.8 kW process. Strain rates are listed in the legend.

Figure 77 shows the dynamic compressive stress-strain curves of 3.8 kW AM parallel-hatched annealed 304L stainless steel along X and Y directions. There is no difference in dynamic compressive stress-strain response between the X and Y directions. All stress-strain curves show highly repeatable and consistent with very similar strain-rate sensitivity to the wrought materials and 2.0 kW AM materials. The 3.8 kW AM specimens also showed very similar appearance of deposit puddles on the specimen surfaces after dynamic compression tests. Detailed comparison between the AM and wrought materials will be presented in section 9.3.

9.2.2. *Tension Tests*

Figure 78 shows a typical set of dynamic tensile stress-strain curves from 4 repeated experiments on wrought 304L stainless steel along longitudinal direction at the same strain rate of 1500 s^{-1} . The dynamic tensile stress-strain curves show the similar highly repeatability, prior to necking at the ultimate strength, to the compressive stress-strain response shown earlier. It is noted that the post-necking stress-strain response in the tensile tests became unreliable due to highly localized deformation and damage that eventually caused failure. Therefore, the mean curve was calculated only prior to necking to represent the dynamic tensile stress-strain response before necking occurs.

The dynamic tensile stress-strain curves of wrought 304L stainless steel along longitudinal and transverse directions at three different strain rates: 500, 1500, and 2900 s^{-1} are shown in Figure 79. The wrought material exhibits very similar stress-strain response along longitudinal and transverse directions. The tensile stress-strain curves show a typical linear elasticity followed by plastic flow behaviour until necking occurred at the ultimate strength. Then the specimen fell into an instable state until failure. Same as the compressive stress-strain response shown in Figure 74, the flow stresses in the specimens along longitudinal direction are slightly higher than those along transverse direction. The flow stress along longitudinal direction is observed to be a little ($\sim 4\%$) higher than that along the transverse direction for the wrought material. Both longitudinal- and transverse-direction specimens exhibit a moderate strain-rate effect on the tensile stress-strain response and high ductility with maximum failure strain of ~ 0.6 . Figure 80 shows typical pictures

of a wrought specimen after a dynamic tensile test, showing the fracture surfaces, necking portion, and the outside-necking section still with reasonably smooth surfaces.

Figure 81 shows dynamic stress-strain curves, at the same strain rate of $\sim 3000 \text{ s}^{-1}$, of the 2.0 kW cross-hatched AM 304L stainless steel along different directions (annealed X direction, and as-deposited Z direction) and locations (edge versus center) along the same Z direction. Interestingly, the 2.0 kW AM 304L stainless steel did not show repeatability in dynamic tensile stress-strain response as good as either the compressive response for the same AM material or the tensile stress-strain response for the wrought materials. The dynamic stress-strain curves for the specimens along the same Z direction but at different locations fall into the same scattering envelop. So it is reasonable to conclude that the effect of specimen locations is negligible. However, the dynamic tensile stress-strain curve of the annealed specimen along X direction is slightly lower than those of the as-deposited specimen along Z direction. It is difficult to conclude if this is caused by specimen orientation (X or Z direction) or post process (annealed or as-deposited). Most important is, regardless of the specimen orientation, location, and post process, the 2.0 kW cross-hatched AM 304L stainless steel generally possessed higher flow stresses but lower ductility than the wrought material. The flow stress in the 2.0 kW AM material is approximately 15% higher than the wrought material but the failure strain decreases from 60% for the wrought material to $\sim 40\%$ for the 2.0 kW AM material. In addition, the flow stress-strain curves in the 2.0 kW AM material showed an up-and-down feature while strain increases, rather than smooth stress-strain response of wrought materials. This may indicate microstructural change or slip mechanism in the material subjected to high-strain-rate tensile load. However, this requires in-depth microstructural investigation, which has been planned and will be presented in the future. After dynamic tension tests, the failed AM 304L stainless steel specimens (examples in Figure 82), showed very similar surface features to the compression specimens shown in Figure 76(b), but significantly different from the failed wrought specimen shown in Figure 80.

Dynamic tensile stress-strain curves of 3.8 kW, parallel-hatched, annealed AM 304L stainless steels along X direction, obtained at three different strain rates - 500, 1500, and 2900 s^{-1} , are shown in Figure 83. The material exhibited a slight strain-rate effect on the tensile stress-strain response.

At the same strain rate, the dynamic tensile stress-strain curves are scattered, but showed consistent less ductility with approximately the same failure strain of 0.4 as the 2.0 kW AM material.

Figure 84 shows a comparison of dynamic tensile stress-strain curves, obtained at the same strain rate of 2900 s^{-1} , of the 3.8 kW parallel-hatched 304L stainless steel but along different directions (X and Y directions) and post processes (as-deposited and annealed) along the same X direction.

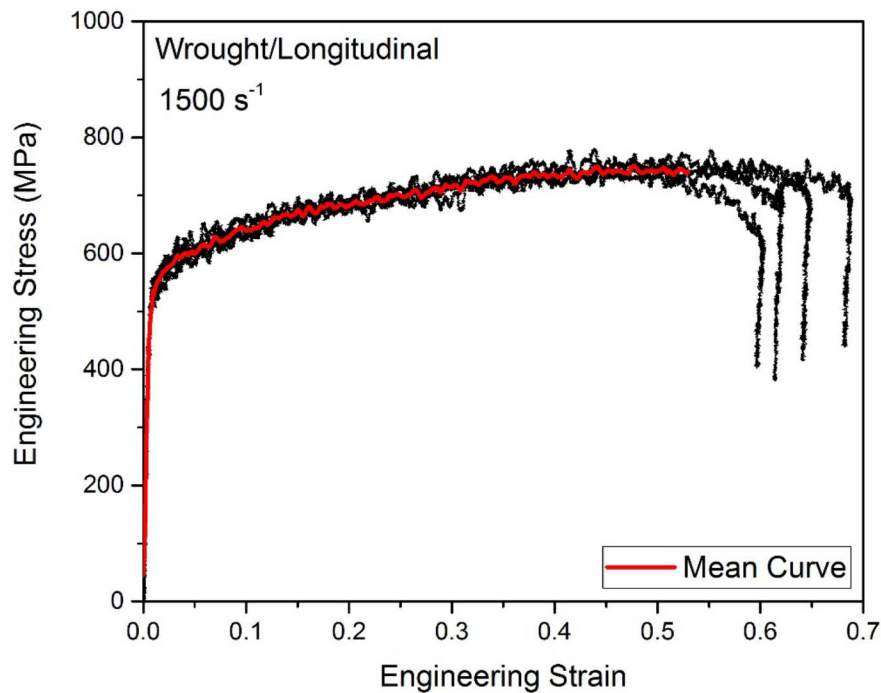


Figure 78. Dynamic tensile stress-strain curves of wrought 304L stainless steel along the longitudinal direction at 1500 s^{-1} .

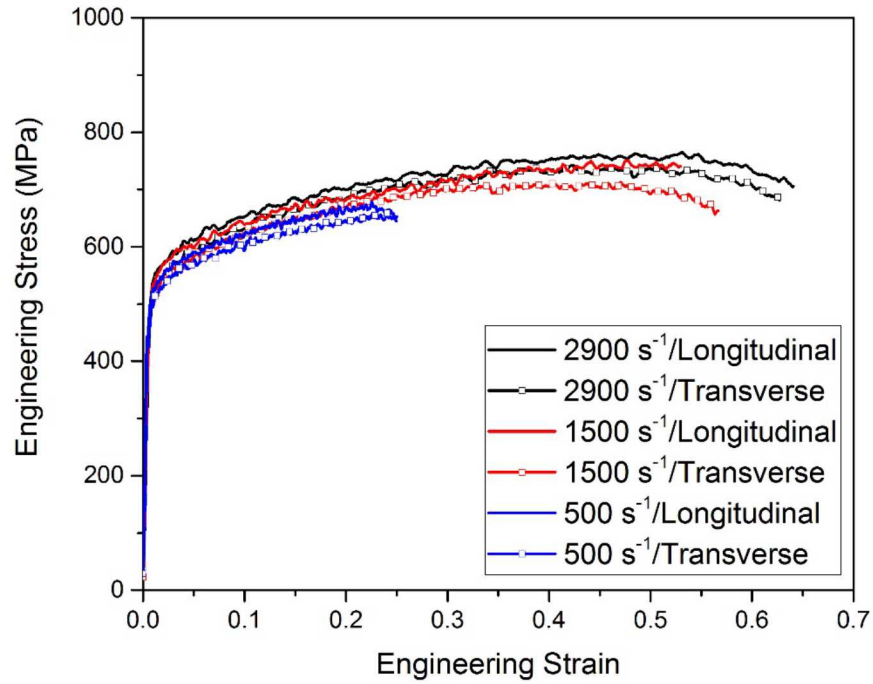


Figure 79. Dynamic tensile stress-strain curves of wrought 304L stainless steel along longitudinal and transverse directions at various strain rates.

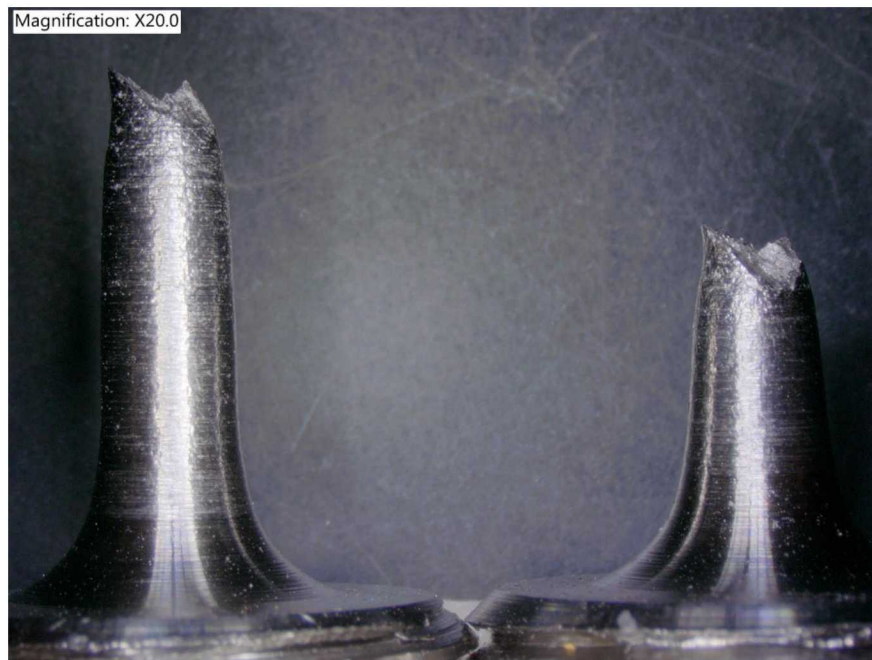


Figure 80. Picture of wrought 304L stainless steel specimen after dynamic tensile test.

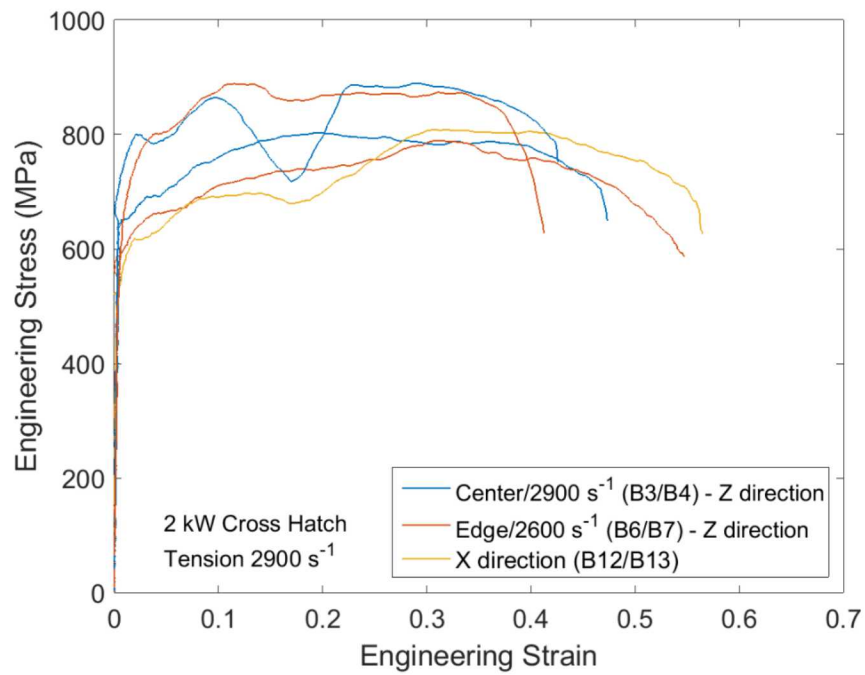


Figure 81. Dynamic tensile stress-strain curves of AM 304L stainless steel built using the 2.0 kW, cross hatched process. Results are shown for $\sim 3000 \text{ s}^{-1}$.

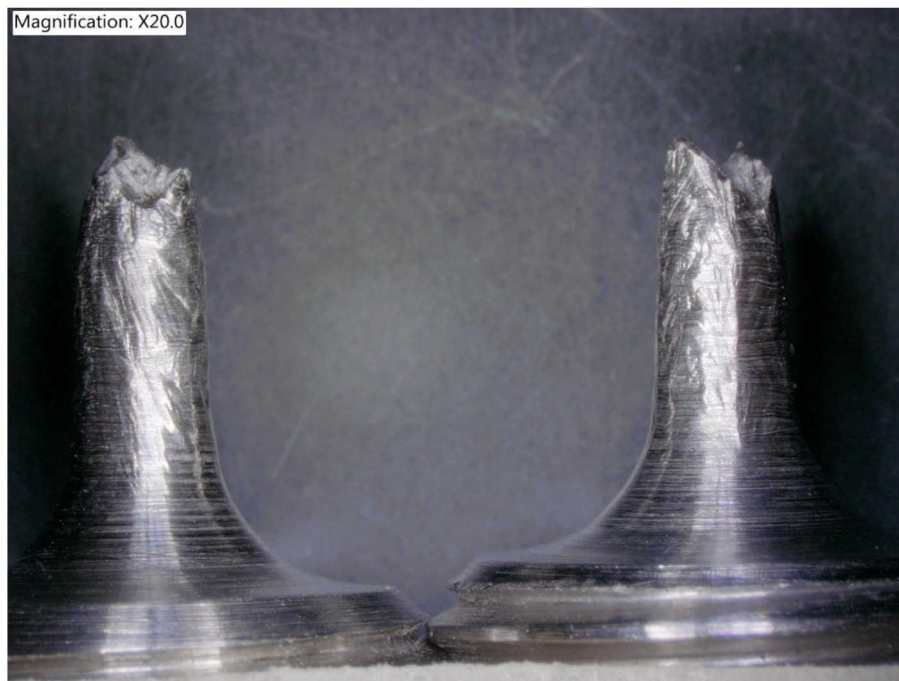


Figure 82. Picture of AM 304 stainless steel (made with 2.0 kW process) Z direction specimen after dynamic tensile test.

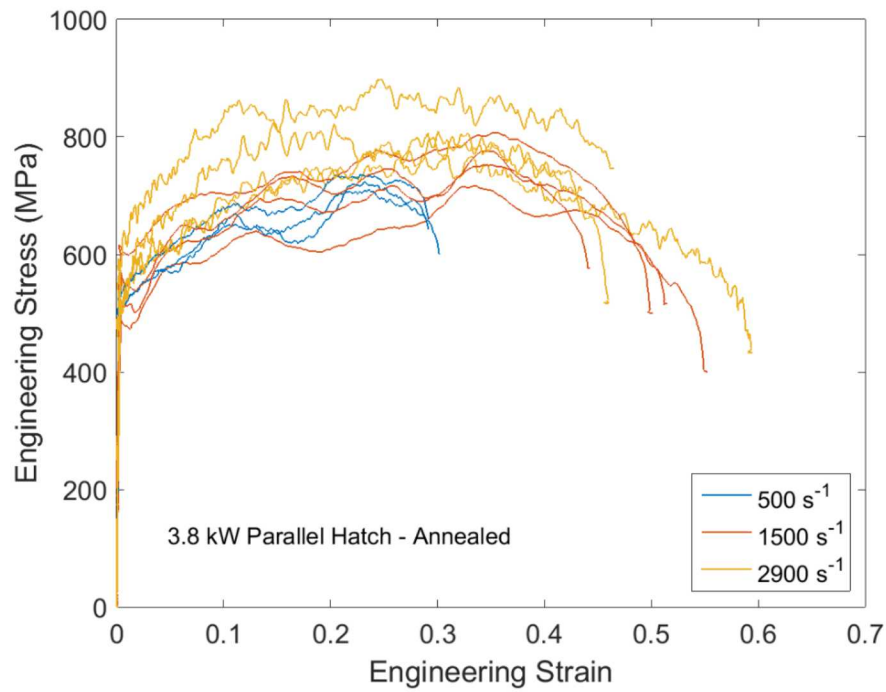


Figure 83. Dynamic tensile stress-strain curves of AM 304L stainless steel (made with 3.8 kW process and annealed). Strain rates are included in the legend.

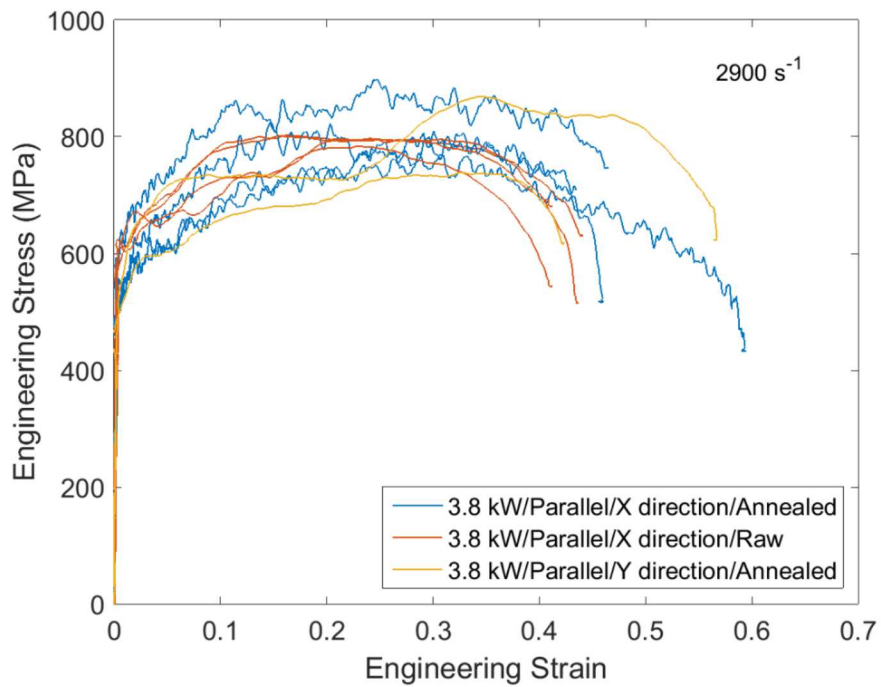


Figure 84. Dynamic tensile stress-strain curves of AM 304L stainless steel (made with 3.8 kW process and annealed) along X (as deposited and annealed) and Y directions at the same strain rate of $\sim 2900 \text{ s}^{-1}$.

9.3. Discussion

Since there are many processing variables (laser power and traveling speed, orientation, locations, heat treatment, etc.) during AM, it becomes difficult to directly determine the effect of each variable on their dynamic properties. Here, we used 2.0 kW, cross-hatched AM materials as an example to compare the dynamic compressive responses with wrought 304L stainless steel material. Figures 85-87 show the comparison of dynamic compressive stress-strain curves of the AM and wrought materials at three different strain rates: ~ 500 , 1500 , and 2500 s^{-1} , respectively. All three figures show the same trend: at a particular strain rate, the as-deposited samples (B5 and B8) along Z direction had the highest yield and flow stress; the yield and flow stresses for the annealed samples (sample B14) along X direction are lower than the samples along Z direction but still significantly higher than the wrought samples. Along the same direction (Z), the samples extracted from the center location are slighter than those from edge location in yield strength and flow stress. As discussed earlier, the wrought samples along the longitudinal direction are slightly stronger than those along transverse direction at ~ 500 and 1500 s^{-1} , but become nearly the same at 2500 s^{-1} . Due to the limit of duration of loading, the specimens were compressed up to 30% but one can still imagine from Figures 71 and 72 that the wrought 304L stainless steel may possess higher flow stress than the AM material at the strains larger than 30%. In addition, as presented earlier, the AM 304L stainless steel specimens show significant different surface features from the wrought specimens after dynamic loading. Even though both materials were compressed to large deformation, the wrought stainless steel specimen still shows fairly smooth surfaces. However, the AM specimen shows rough surfaces with irregular shape after dynamic loading, which may be attributed to significant differences in microstructure between the wrought and AM materials.

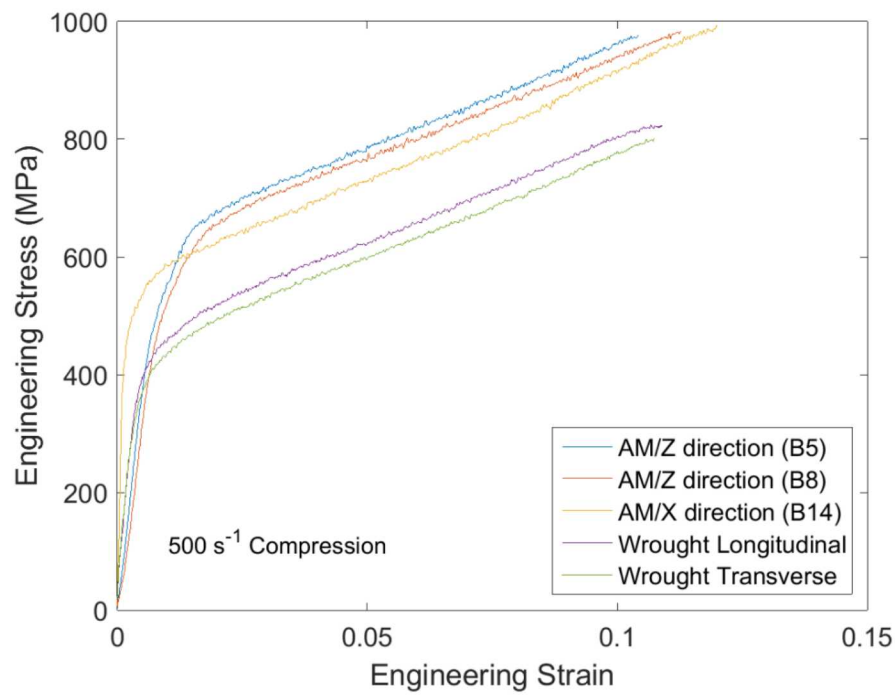


Figure 85. Dynamic compressive stress-strain curves of wrought and AM SS at 500 s^{-1} .

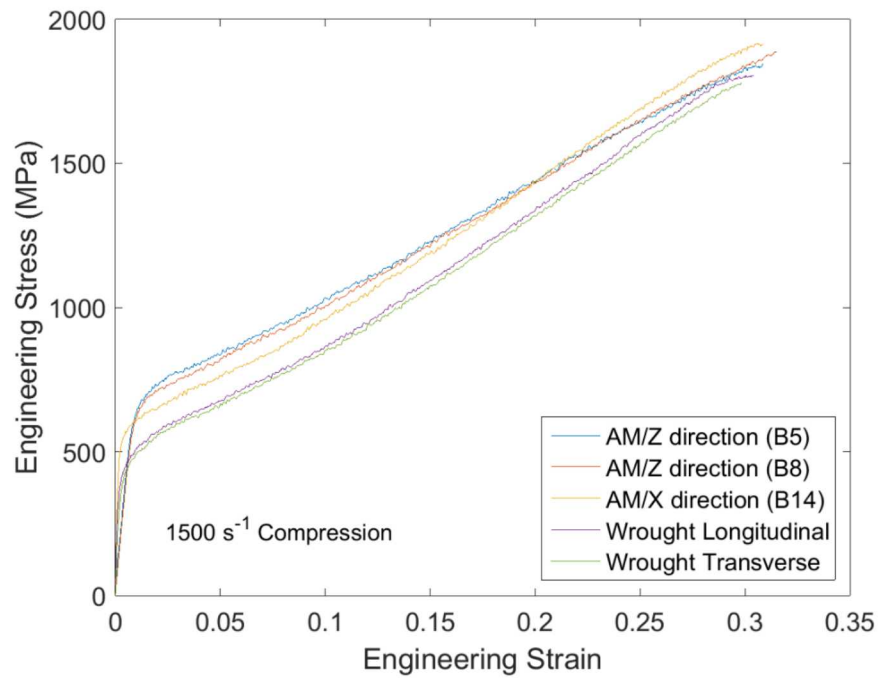


Figure 86. Dynamic compressive stress-strain curves of wrought and AM SS at $\sim 1500 \text{ s}^{-1}$.

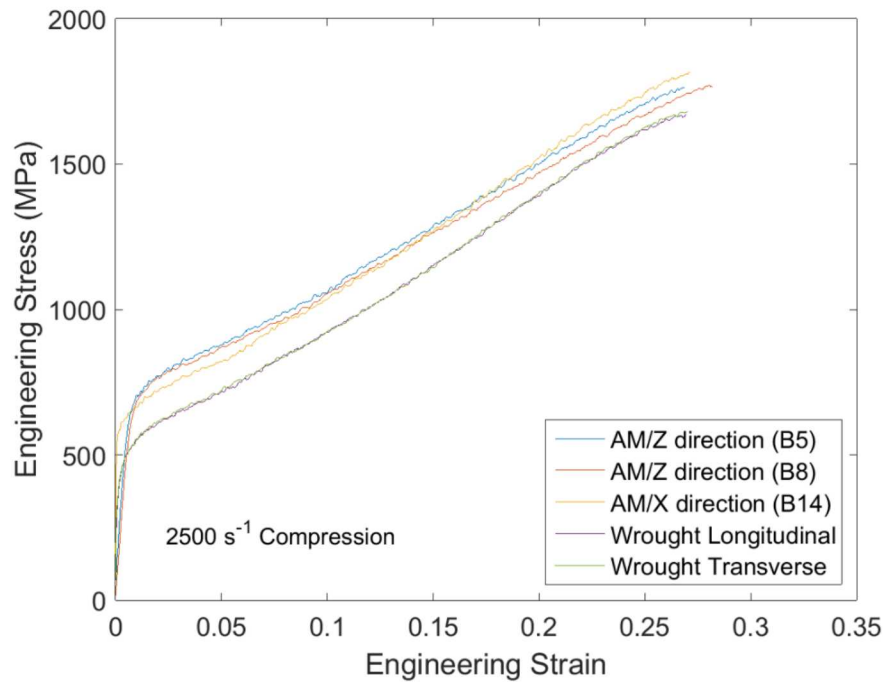


Figure 87. Dynamic compressive stress-strain curves of wrought and AM SS at $\sim 3000 \text{ s}^{-1}$.

Figures 88-90 show another example of 3.8 kW, parallel hatched, annealed AM stainless steel tested along the X direction compared to the wrought materials in dynamic tensile stress-strain responses at three different strain rates: ~ 500 , 1500 , and 2900 s^{-1} . Generally, the annealed AM material had slightly higher but more scattered dynamic flow stress than the wrought materials. The dynamic stress-strain curves of the AM material are less smooth than those of the wrought materials, which may be related to a spatially-varying microstructure in the AM material. It is more important that the AM material exhibited significantly less ductility than the wrought materials. Again, as presented earlier, the AM samples showed a feature of deposit puddles on the specimen surface after dynamic tensile tests.

Figure 91 shows a comparison of dynamic stress-strain curves of 3.8 kW, parallel hatched AM materials but with different directions and post-processing at the same strain rate of $\sim 2900 \text{ s}^{-1}$. Even though all stress-strain curves are scattered, no significant difference in dynamic tensile stress-strain response was observed.

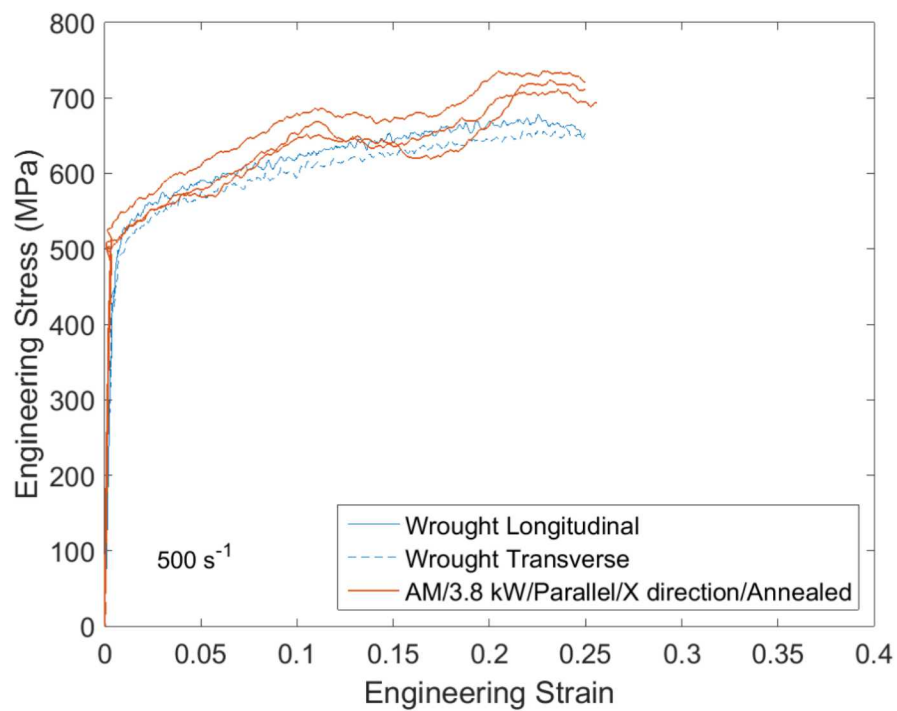


Figure 88. Dynamic tensile stress-strain curves of wrought and AM 304L SS at $\sim 500 \text{ s}^{-1}$.

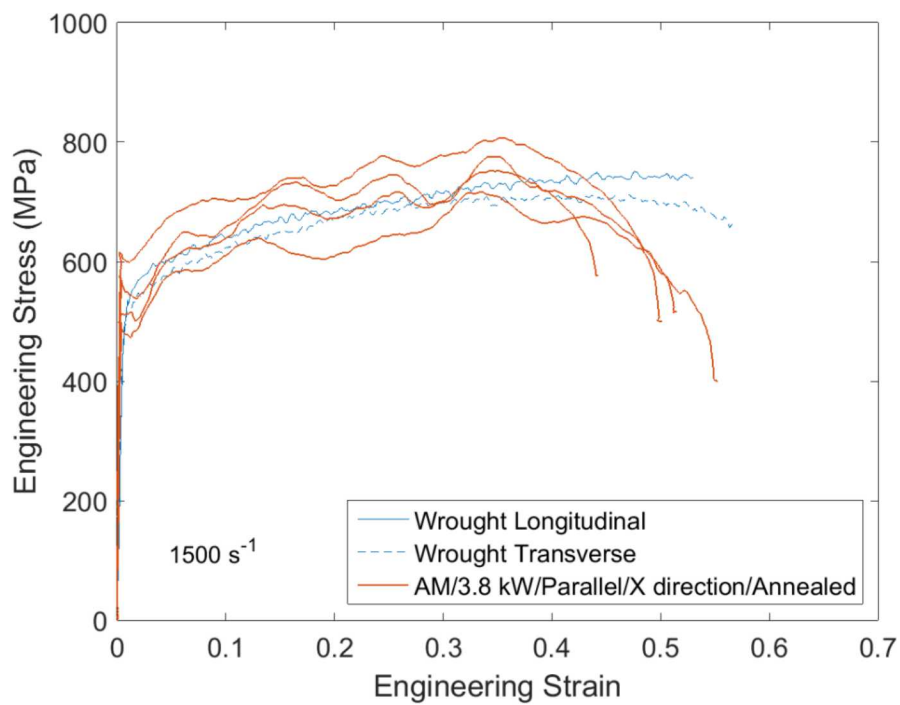


Figure 89. Dynamic tensile stress-strain curves of wrought and AM 304L SS at $\sim 1500 \text{ s}^{-1}$.

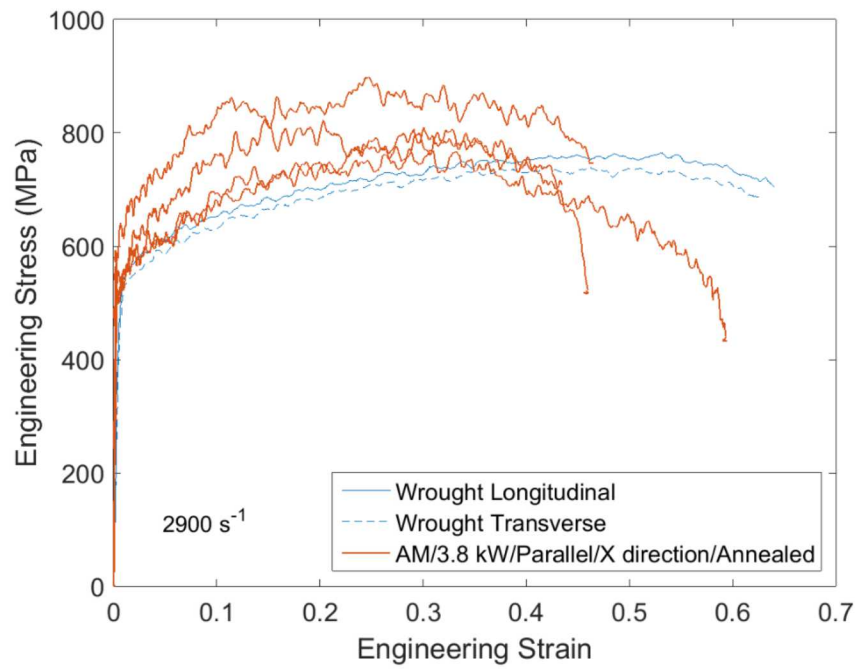


Figure 90. Dynamic tensile stress-strain curves of wrought and AM 304L SS at $\sim 3000 \text{ s}^{-1}$.

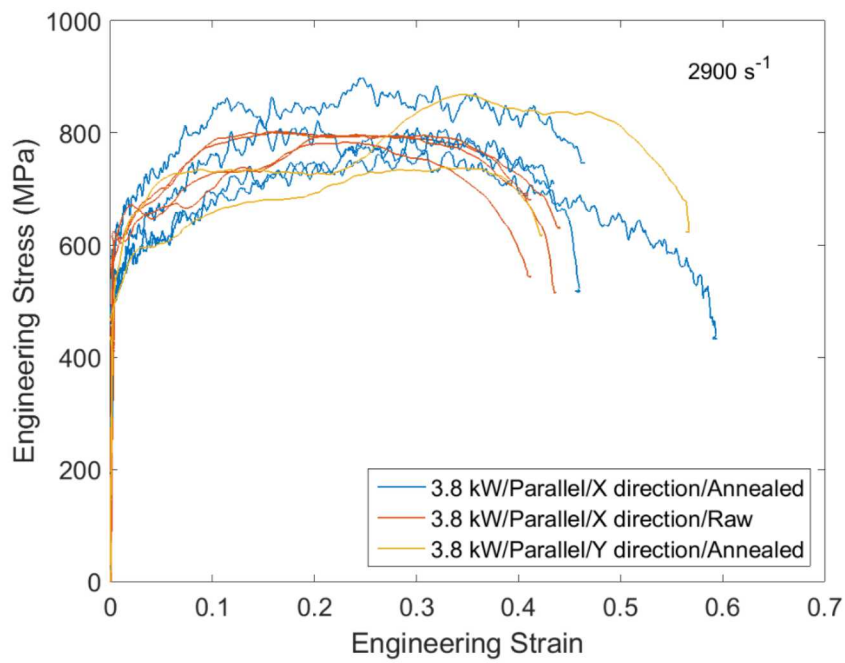


Figure 91. Dynamic tensile stress-strain curves of wrought and AM 304L SS at $\sim 3000 \text{ s}^{-1}$.

10. HIGH VELOCITY IMPACT MECHANICAL RESPONSE

10.1. Experimental Methods

10.1.1. Test Apparatus and Configurations

Impact loading experiments for this study were performed at Sandia's Dynamic Integrated Compression Experimental (DICE) facility, which is operated by Organization 01647. This facility features a 3.5 MA pulsed power machine and a 76-mm bore, single-stage gas gun that are designed for high-strain-rate material testing. The gas gun, pictured in Figure 92, was used to produce projectile velocities ranging from 76 to 323 m/s and corresponding peak stresses in the steel test samples between 0.25 and 6.7 GPa. Accurate projectile velocity and tilt (typically on the order of one milliradian) measurements were derived from relative closure times for charged electrical pins of known length that were mounted around the target plate periphery. The in-house velocity interferometer system, or VISAR, routinely fielded by DICE facility personnel yielded time-resolved records of sample motion induced by stress wave generation and propagation. These records were subsequently analyzed to extract information regarding the dynamic yield strength (i.e., Hugoniot Elastic Limit, or HEL), Hugoniot equation of state, high-pressure strength, and spall strength of the test samples. A catcher system mounted behind the target assembly allowed the recovery of intact samples for post-mortem metallurgical investigations.



Figure 92. DICE facility 3-inch compressed gas gun.

10.2. Results

Three different test configurations produced the desired dynamic response data; in all, a total of 12 gas-gun shots were completed. Hugoniot information for both the AM and baseline 304L materials were obtained with a reverse-ballistic arrangement employing a projectile-mounted stainless steel impactor (nominally 3-mm thick) that simultaneously struck three side-by-side witness windows fabricated, respectively, from polymethyl methacrylate (PMMA), lithium fluoride (LiF), and Z-cut sapphire. With this technique (depicted in Figure 93), a single shot yielded Hugoniot data for the stainless steel at three distinct shock stresses dictated by the impact velocity and respective shock impedances of the window materials. A velocity interferometer system (VISAR[133]) provided time-resolved measurements of the motion at the impactor/window interfaces. Interferometer data were interpreted on the basis of published mechanical and optical calibrations of PMMA[134], LiF[135], and sapphire[136]. Test sample drawings are included in Appendix 13.15.

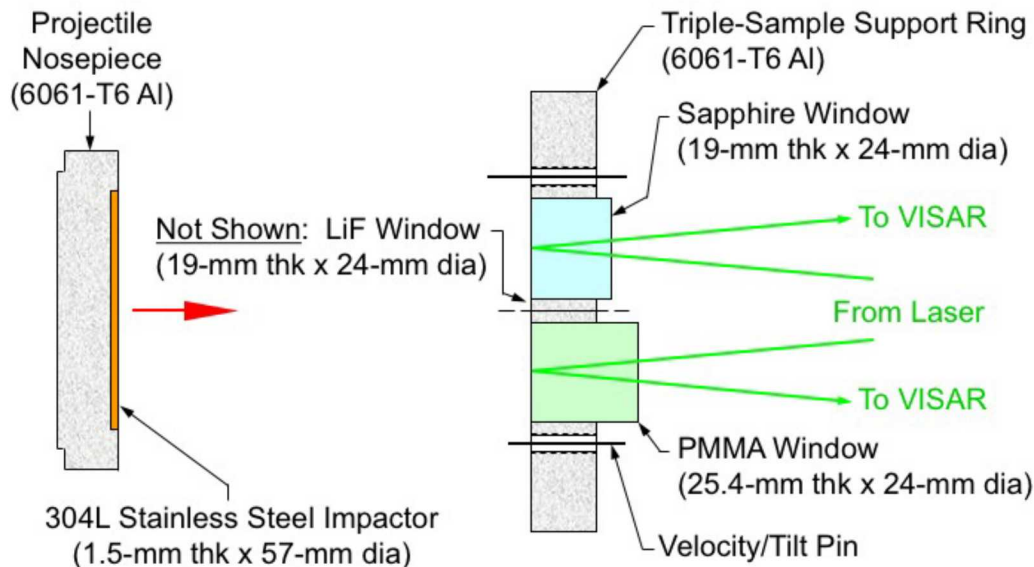


Figure 93. Depiction of geometry utilized for reverse ballistic testing.

Three reverse-ballistic experiments were performed with Z-cut, 3.8 kW AM material, followed by three with baseline stainless steel. Measured launch velocities were 0.0818, 0.200, and 0.320 km/s for the AM flyers, and 0.0809, 0.198, and 0.323 km/s for the conventional 304L impactors.

For each window, the Hugoniot particle velocity jump in the stainless steel corresponded to the difference between the initial flyer velocity and the amplitude of the impact-induced leading velocity plateau at the flyer/window interface. The corresponding shock stress in the stainless steel was calculated on the basis of this velocity plateau amplitude and the published window Hugoniot. A representative overlay of interface velocity histories is shown in Figure 94 for a matching pair of reverse-ballistic shots that, respectively, involved an AM flyer (0.200 km/s) and a conventional 304L impactor (0.198 km/s). The overall set of acquired Hugoniot data for AM and baseline materials is plotted in Figure 95, along with a fit to archival Los Alamos data [137] for 304L stainless steel. The present results for AM and baseline materials closely match one another as well as the Los Alamos fit.

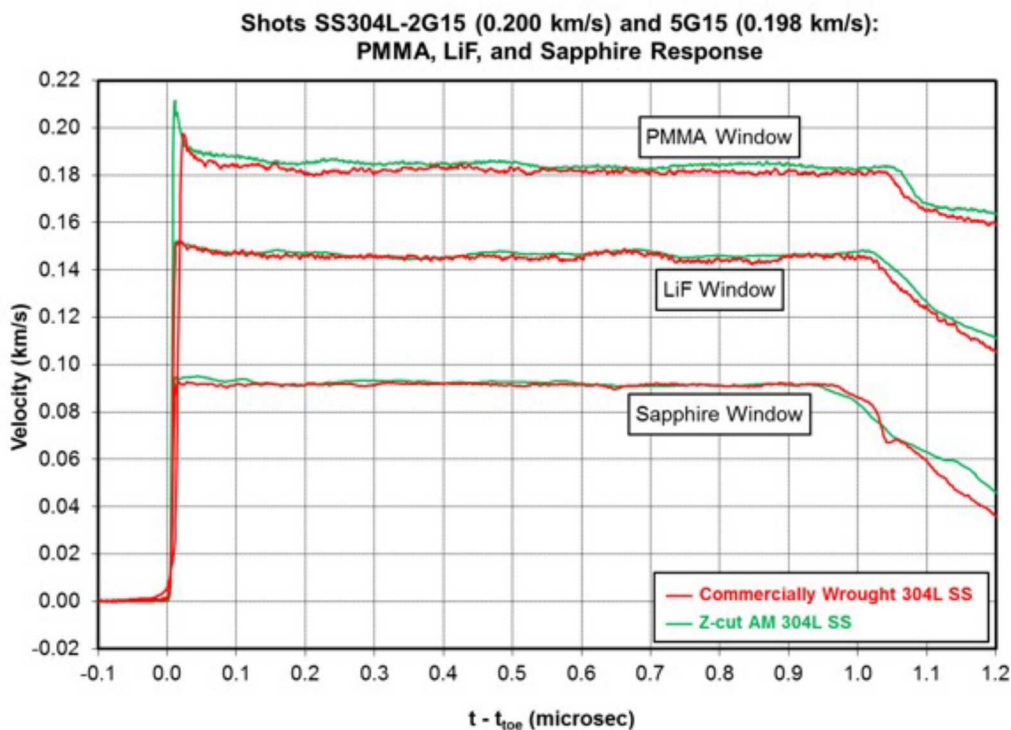


Figure 94. Reverse-ballistic test results: (a) SS 304L impactor/window interface velocity histories for Z-cut AM material and wrought SS 304L flyer velocities of 0.200 and 0.198 km/s, respectively.

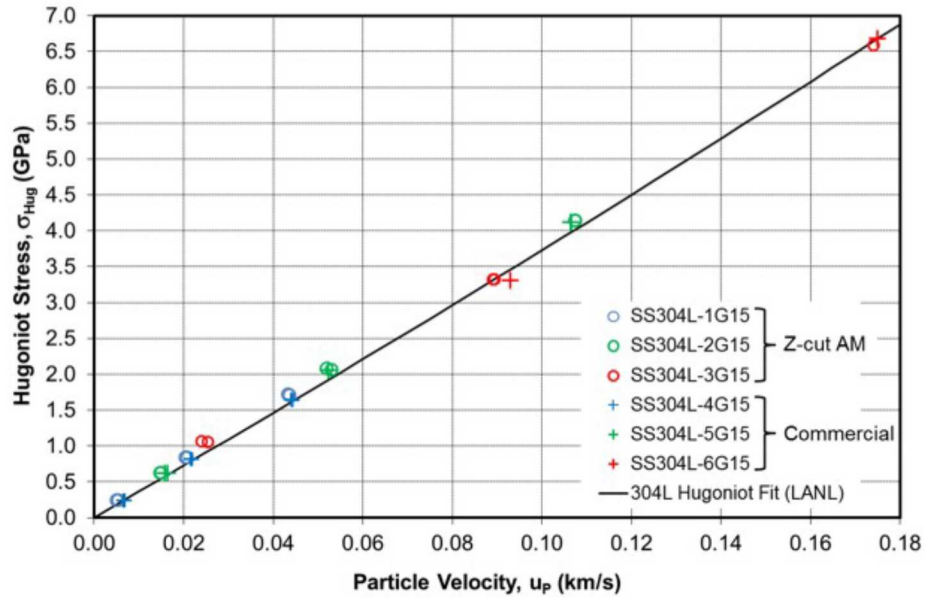


Figure 95. Reverse-ballistic test results: z-cut AM and wrought SS 304L Hugoniot measurements overlaid with fit to the Los Alamos data.

Transmitted wave profiles were derived from forward-ballistic experiments depicted in Figure 96. These utilized a Z-cut sapphire impactor (4.16-mm thick) to shock compress three side-by-side stainless steel samples (nominal thickness = 1.5 mm) that were machined, respectively, from X-cut 3.8 kW AM, Z-cut 3.8 kW AM, and baseline wrought material. Test sample drawings are included in Appendix 13.16. Each of these samples was backed by a Z-cut sapphire window, and a VISAR tracked the sample/window interface motion. Since the shock impedance of sapphire is similar to that for stainless steel, the recorded wave profiles approximate *in situ* data for motion within the steel. The three experiments using this approach featured impact velocities of 0.0756, 0.203, and 0.319 km/s, respectively, and produced the resultant velocity histories plotted in Figure 97 where the longitudinal stress at the 304L/window interface is displayed along the right-hand vertical axis. From these records, each material manifests two-wave, elastic-plastic loading for peak stresses of at least 7.0 GPa. Consistent with their virtually identical Hugoniot results, the X-cut and Z-cut AM samples, as well as the conventionally wrought specimen, all reached essentially the same peak velocity plateau for transmitted waves generated by a particular sapphire impactor velocity. Elastic (for $u_{\text{impact}} = 0.0756$ km/s) or elastic-plastic (for $u_{\text{impact}} = 0.203$ and 0.319 km/s) unloading began at $t - t_{\text{toe}} \sim 0.7 \mu\text{s}$ for all

specimens. The high- pressure yield strength, which is proportional to the velocity difference between the profile peak and the level at which the release-wave slope changes due to the elastic-plastic transition, may be estimated using a simplified analysis method.[138]

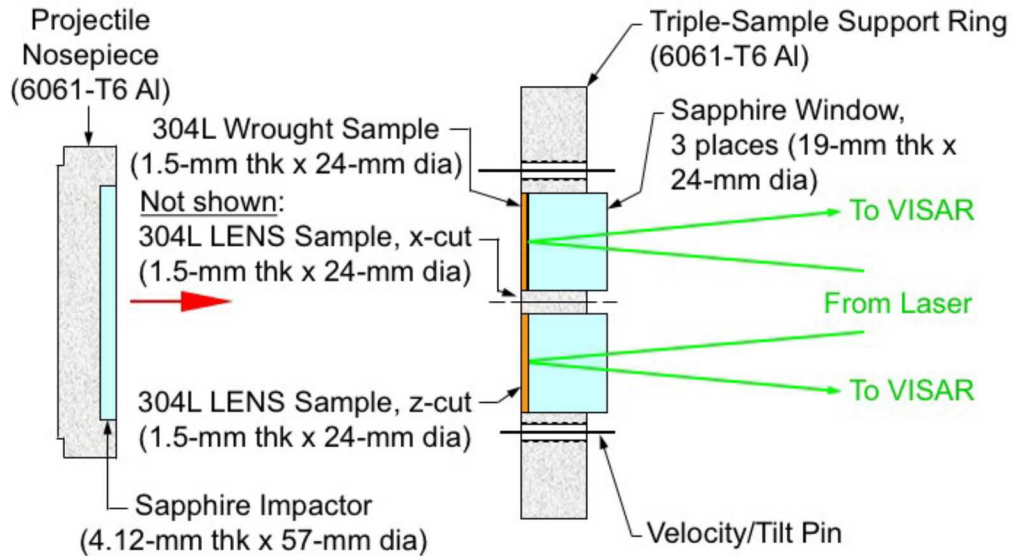


Figure 96. Depiction of geometry utilized for forward ballistic testing.

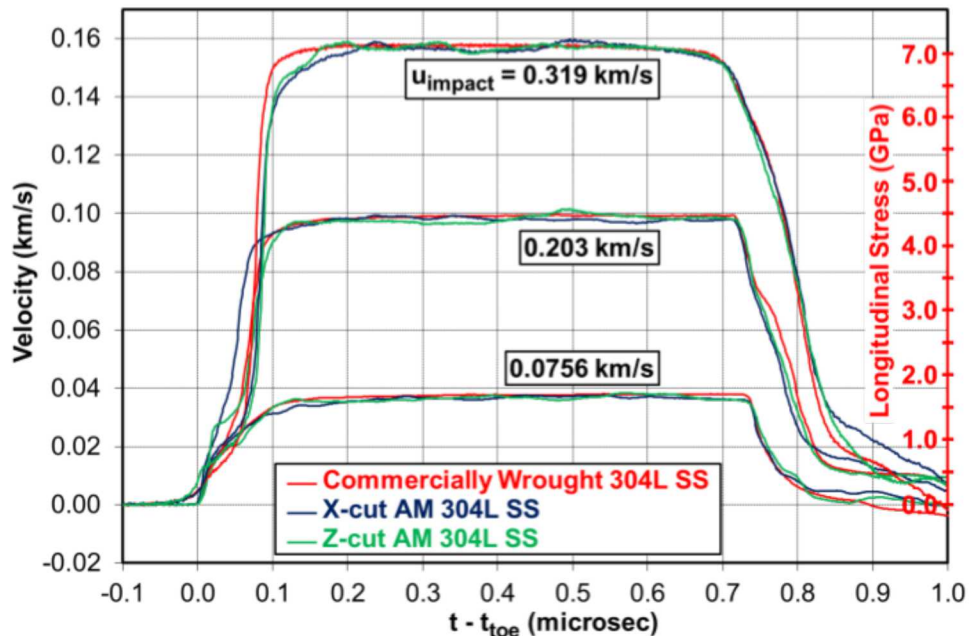


Figure 97. Forward-ballistic results: 304L target / window interface velocity histories for triple-sample experiments involving sapphire flyer velocities of 0.0756, 0.203, and 0.319 km/s.

To assess the relative spall strengths of X-cut AM, Z-cut AM, and baseline 304L stainless steel, three additional forward-ballistic experiments were conducted on side-by-side specimens (nominally 2.0-mm thick) without backing windows. In these tests (depicted in Figure 98), the free-surface motion of the steel samples was monitored for impacts by sapphire flyers (2.45-mm thick) launched at velocities of 0.154, 0.247, and 0.317 km/s, respectively. The measured velocity histories appear in Figure 99. For the lowest impact velocity (0.154 km/s), none of the samples spalled. However, the free-surface data obtained for the higher impact velocities (0.247 and 0.317 km/s) show local velocity minima at $t - t_{toe} \sim 0.5 - 0.6 \mu s$ that signal spallation. The spall strength of a given sample is proportional to the difference in velocity between the profile peak and the subsequent local minimum. Test sample geometries are included in Appendix 13.17.

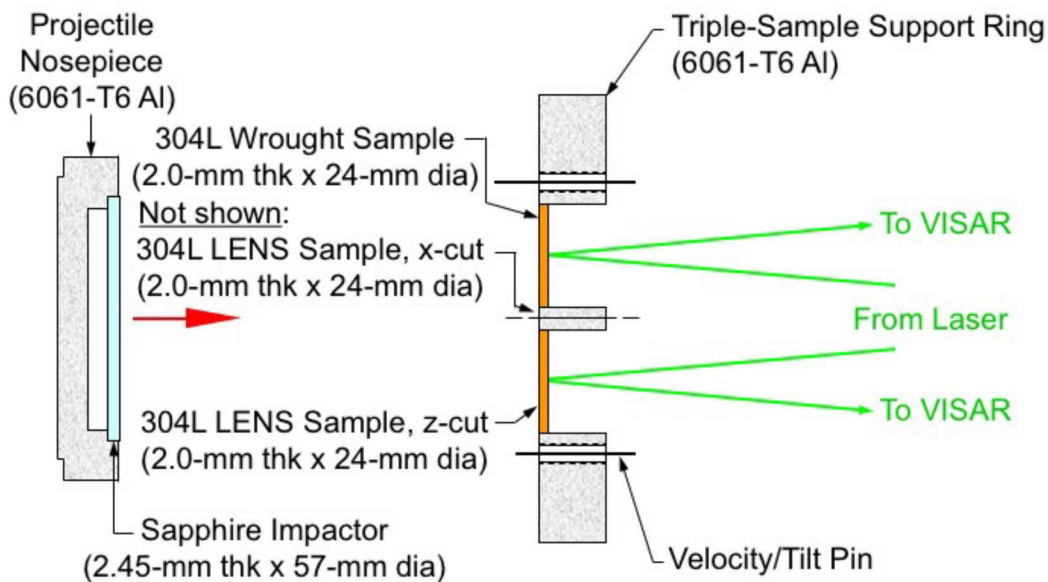


Figure 98. Depiction of geometry utilized for spall tests.

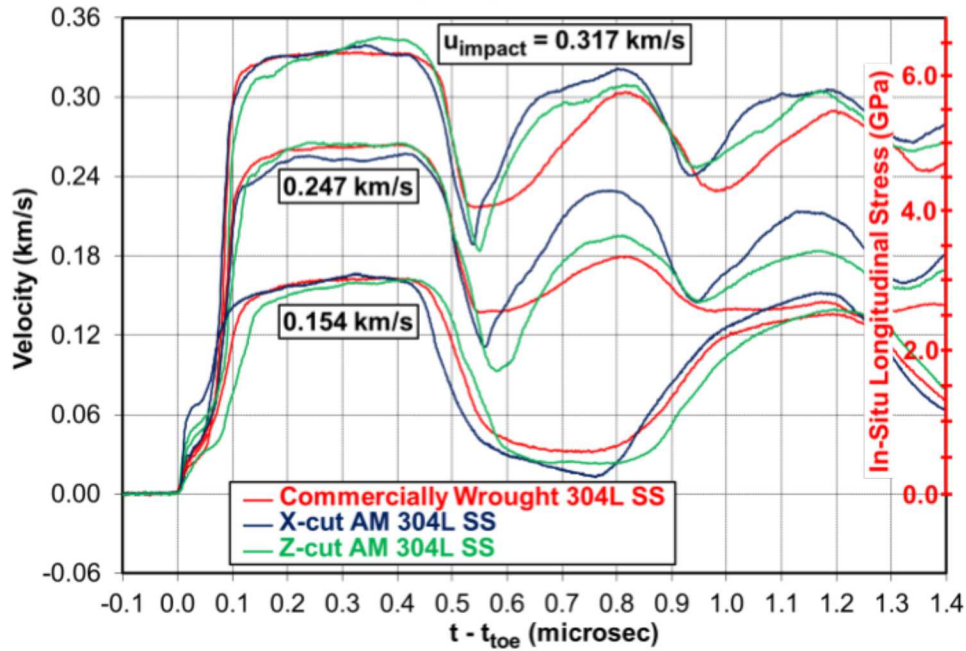


Figure 99. Forward-ballistic test results: 304L free-surface velocity histories for triple-sample tests with sapphire flyer velocities of 0.154, 0.247, and 0.317 km/s.

Spallation was confirmed by inspecting recovered samples. Figure 100 shows three tested samples; each is an AM SS 304L, X-cut sample. Recovered samples were wire cut, potted and polished to produce a view of faces perpendicular to the loading direction (X) and inspect for internal fracture. As shown left to right in this figure, the three samples were impacted at increasing flyer velocities. The lowest velocity impact did not result in spallation, consistent with data in Figure 99. The other two samples are spalled internally with increased amounts of separation occurring at the highest velocity. Similar results were observed in Z-cut and wrought samples, after sectioning. When spallation does occur, material is internally separated along the approximate mid-plane of the specimen. Spallation occurs to within 1-2 mm of outer edges.

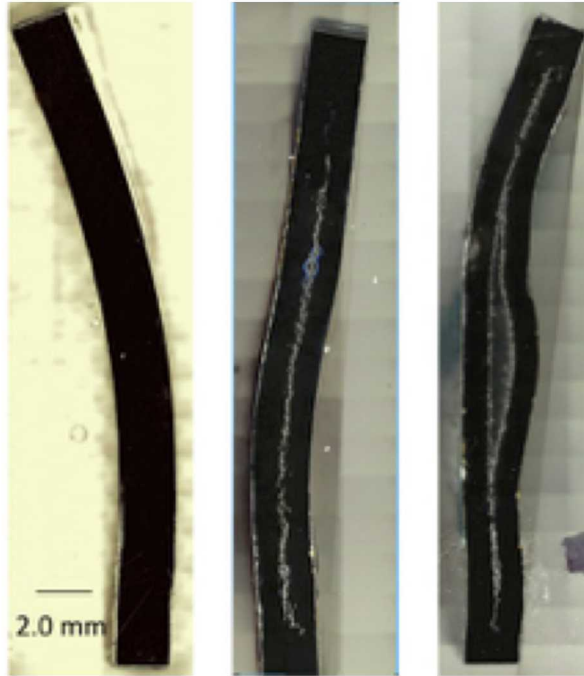


Figure 100. Cross sectional optical images of three recovered AM SS 304L samples (X-cut) after impact testing. Each was tested at a different impact velocity listed in Figure 73 (increasing velocity from left to right). Impact face is oriented to left.

EBSD and SEM were used to characterize grain structure, defect structure and phase content after impact. The top set of images in Figure 101 shows the microstructure of wrought stainless steel 304L after impact at 0.317 km/s. The lower set includes images of AM 304L impacted under identical conditions. Spallation has occurred in these samples, and the EBSD and SEM images show the full thickness of the tested sample (when viewed left-to-right). A portion of each spalled surface is included in these images, identified with red arrows. From these images it is evident that some changes have occurred to microstructure aside from spallation in the mid-plane of some. Overall, the grain structure remains relatively unchanged with small, equiaxed grains present in wrought and larger, elongated grains (with substantial intra-grain structure) present in the additively manufactured form. All grains colored in the EBSD maps are austenite. There is no obvious increase in the amount of ferrite after impact loading. Follow on analysis with Feritscope showed about the same amount of ferrite after gas gun testing, ~ 2-3 %. In addition, both samples show deformation twins after testing. No adiabatic shear bands are detected in these and other sections of wrought and AM material.

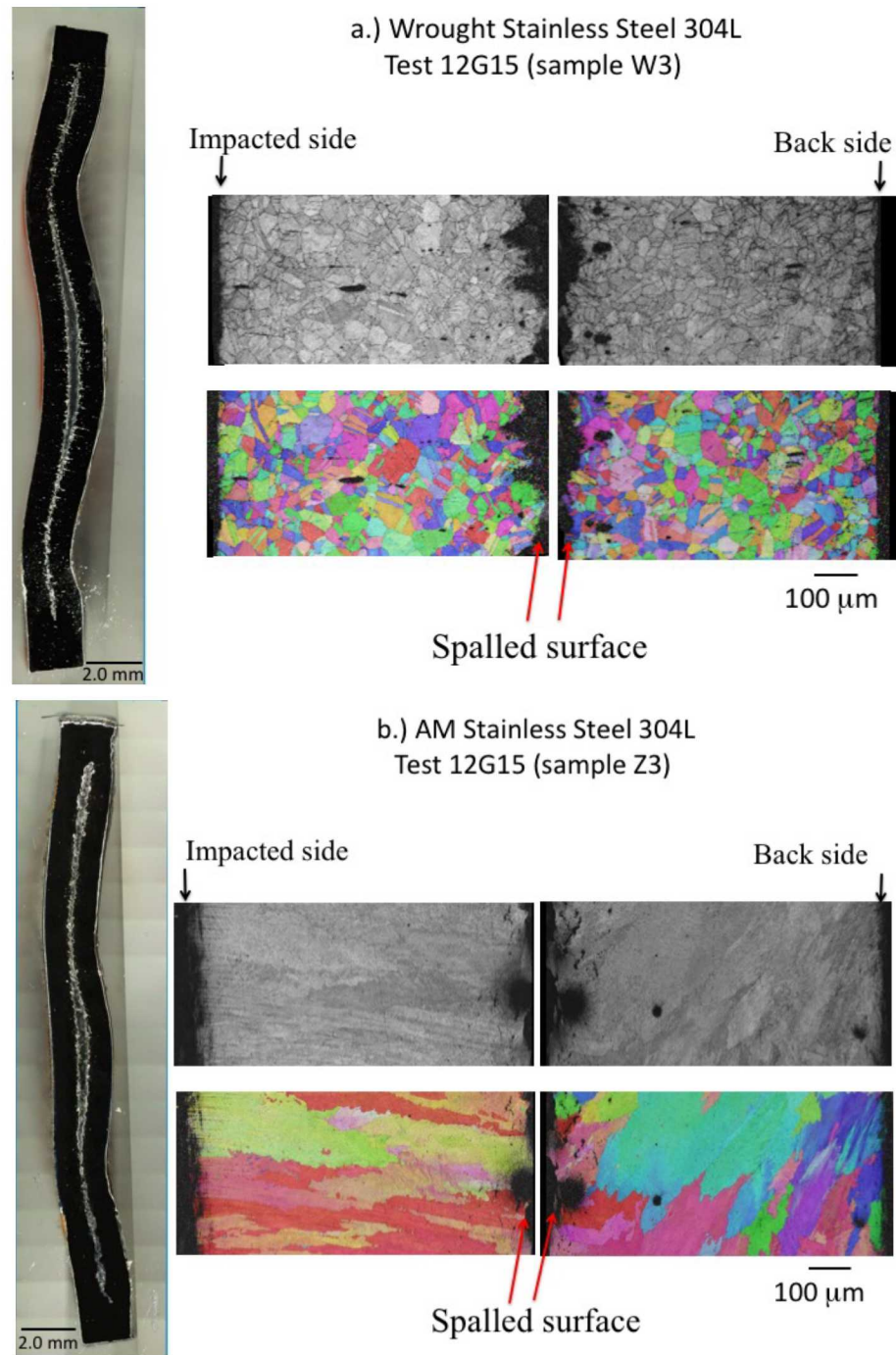


Figure 101. Images and EBSD maps of (a.) wrought and (b.) AM SS304L Z-cut specimen recovered from impact spall test ($v = 0.317$ km/s, shot 12G15). The Z-direction is left-to-right in the images.

For reference, Table 10 summarizes all experiments involving gas gun impact. Appendices 13.18 and 13.19 show the approximate location of extracted samples used for testing with respect to the original square AM bar.

Table 10. Experiments involving gas gun impact tests.

Shot Number	Test Configuration	Flyer Velocity (km/s)	Impactor Material	Impactor Thickness (mm)	Impactor Diameter (mm)	Target Sample 1	Target Sample 2	Target Sample 3
SS304L-1G15	Reverse Ballistic	0.0818	AM SS304L	3.0	57	Sapphire	LiF	PMMA
SS304L-2G15	Reverse Ballistic	0.200	AM SS304L	3.0	57	Sapphire	LiF	PMMA
SS304L-3G15	Reverse Ballistic	0.320	AM SS304L	3.0	57	Sapphire	LiF	PMMA
SS304L-4G15	Reverse Ballistic	0.0809	Wrought SS304L	3.0	57	Sapphire	LiF	PMMA
SS304L-5G15	Reverse Ballistic	0.198	Wrought SS304L	3.0	57	Sapphire	LiF	PMMA
SS304L-6G15	Reverse Ballistic	0.323	Wrought SS304L	3.0	57	Sapphire	LiF	PMMA
SS304L-7G15	Forward Ballistic	0.0756	Sapphire	3.0	57	AM SS304L x-axis*	AM SS304L z-axis*	Wrought SS304L*
SS304L-8G15	Forward Ballistic	0.203	Sapphire	4.12	57	AM SS304L x-axis*	AM SS304L z-axis*	Wrought SS304L*
SS304L-9G15	Forward Ballistic	0.319	Sapphire	4.12	57	AM SS304L x-axis*	AM SS304L z-axis*	Wrought SS304L*
SS304L-10G15	Forward Ballistic-Spall	0.154	Sapphire	4.12	57	AM SS304L x-axis^	AM SS304L z-axis^	Wrought SS304L^
SS304L-11G15	Forward Ballistic-Spall	0.247	Sapphire	2.45	57	AM SS304L x-axis^	AM SS304L z-axis^	Wrought SS304L^
SS304L-12G15	Forward Ballistic-Spall	0.317	Sapphire	2.45	57	AM SS304L x-axis^	AM SS304L z-axis^	Wrought SS304L^

*Target sample geometry was 1.5 mm thick by 24 mm diameter.

[^]Target sample geometry was 2.0 mm thick by 24 mm diameter.

10.3. Discussion

The present Hugoniot measurements for both AM and conventional 304L stainless steel shocked to peak stresses in the 0.2 – 7.0 GPa range are mutually consistent, and are closely approximated by a downward extrapolation of the Hugoniot locus computed using the linear shock velocity (U_S) versus particle velocity (u_p) fit, $U_S = 4.57 + 1.48 u_p$, that was reported for historical data acquired in the 9.0 – 184 GPa range by researchers at the Los Alamos Scientific Laboratory (LASL).[137] Comparable low-amplitude fluctuations (~1%) in Hugoniot particle velocity for AM and conventional 304L samples appear in wave profiles measured during the reverse-ballistic testing. In general, grain size and orientation have minimal apparent influence on the mean Hugoniot response of 304L stainless steel produced either conventionally or by an AM process.

From the forward-ballistic testing, low-amplitude (~1–5%) variations in peak particle velocity are exhibited by the AM 304L material, but not by the conventionally manufactured stainless steel, for transmitted wave measurements involving both windowed and non-windowed samples. This difference may be attributed to the inherent anisotropy of the coarse-grained AM 304L versus the fine-grained bar stock.

Based on transmitted-wave precursor amplitudes for the windowed samples, the Hugoniot Elastic Limit (HEL) estimates show test-to-test/sample-to-sample variability, ranging from 0.6 – 0.8 GPa for X-cut and 0.6 – 1.2 GPa for Z-cut AM 304L, as compared to a relatively repeatable figure of 0.5 – 0.6 GPa for conventionally wrought samples. Clearly, the dynamic strength of the AM samples equals or exceeds that of the baseline material. Considering the small sample thickness of 1.5 mm for the forward-ballistic experiments, only several grains at most are traversed by the compression/release waveform propagating through an AM sample as compared to ~100 grains for a specimen of conventionally wrought 304L; hence, the inequity in dynamic yield strength may be another consequence of grain size and/or orientation. However, other strengthening mechanisms could be involved in the case of AM 304L, including higher residual stresses, an elevated density of small ferrite islands (measured average size ~0.5 μm ; spacing ~5 μm), the presence of sub-grains, and an increased dislocation density.

Analyses of the unloading waveforms acquired for windowed samples that were impacted at velocities of 0.203 and 0.319 km/s, which respectively generated peak stresses of 4.12 and 6.56 GPa, indicate high-pressure yield strengths of 0.43 – 0.45 GPa for X-cut and 0.40 – 0.46 GPa for Z-cut AM 304L, as compared to 0.30 – 0.36 GPa for conventional 304L. For each material variant, a greater yield strength value is manifested upon release from a higher peak stress. In agreement with the loading response results, the high-pressure yield strength observed upon unloading of the AM samples exceeds that of the baseline stainless steel.

Free-surface measurements of transmitted wave profiles for the non-windowed samples indicate approximate spall strengths of 3.28, 3.84, and 2.86 GPa, respectively, for X-cut AM, Z-cut AM, and baseline, wrought 304L stainless steel samples shocked to a peak stress of 5.05 GPa, and somewhat *lower* spall strengths (namely 3.21, 3.58, and 2.60 GPa, respectively) for a shock amplitude of 6.50 GPa. For both of these impact conditions, the spall strength of the AM samples is substantially higher than that of the baseline 304L, with the Z-cut AM material exhibiting the greatest overall value. This result is consistent with the above-noted estimates of dynamic yield strength.

11. MODELS FOR THE MECHANICAL BEHAVIOR

As shown in Chapter 5, our additively manufactured microstructures differ substantially from typical wrought microstructures. For example, the grains sizes are an order of magnitude larger, the grain morphology is quite exotic, and the distribution of grain orientations has a significant texture. Chapters 7-10 discuss how these differences affect the mechanical behavior in experiments, but modeling can also shed light on the importance of microstructural features. This chapter discusses efforts to assess the impact of the grain morphology and texture by simulating tensile tests, compression tests, and axial-torsion tests using the crystal plasticity finite element method. The axial-torsion tests are compared against a crystal plasticity model and a macroscopic J_2 flow model to assess whether the increased computational cost of the crystal plasticity model yields greater accuracy. For simplicity, the modeling focuses on the additively manufactured 3.8 kW X-bar material. The wrought and 2 kW additively manufactured materials are left for future work. Appendices 14.20 and 14.21 include machine drawings and pictures of machined wrought and AM stainless steel axial-torsion test samples.

11.1. Model Construction

11.1.1. Crystal Plasticity Model

The crystal plasticity model used here is known at Sandia as the RPI model, since it originated at the Rensselaer Polytechnic Institute. The model formulation can be found in [139], but a short description will be stated here for convenience.

The three configurations shown in Figure 102 are the reference (typically undeformed) configuration, the intermediate (only plastically deformed) configuration, and the current (elastically and plastically deformed) configuration. A multiplicative split of the deformation gradient $\mathbf{F}$ into an elastic part $\mathbf{F}^e$ and a plastic part $\mathbf{F}^p$,

$$\mathbf{F} = \mathbf{F}^e \cdot \mathbf{F}^p, \tag{11-1}$$

relates the three configurations. The unit normal to the slip plane $\mathbf{n}_c^\alpha$ and unit slip direction $\mathbf{m}_c^\alpha$ on slip system α in the current configuration are not known *a-priori*. In the reference configuration, however, the unit normal to the slip plane $\mathbf{n}_0^\alpha$ and unit slip direction $\mathbf{m}_0^\alpha$ are known. Consequently, the model lumps the rotation $\mathbf{R}$ into $\mathbf{F}^e$, such that $\mathbf{F}^p$ is a pure deformation that leaves the lattice structure unaffected. This causes the slip plane unit normal $\mathbf{n}^\alpha$ and unit slip direction $\mathbf{m}^\alpha$ in the intermediate configuration, to respectively coincide with $\mathbf{n}_0^\alpha$ and $\mathbf{m}_0^\alpha$ in the reference configuration.

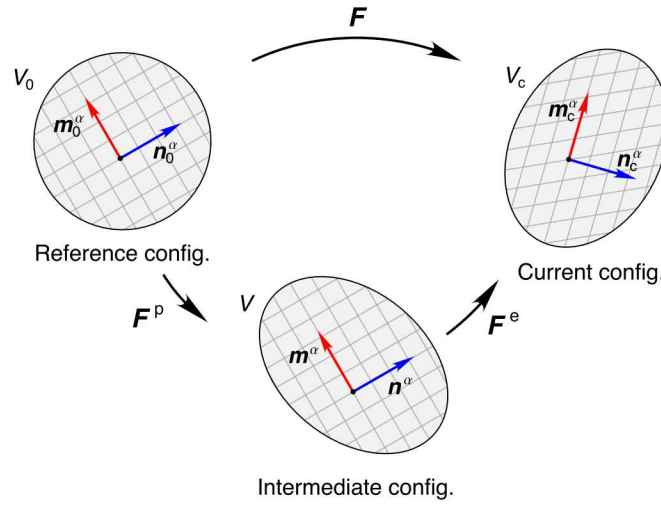


Figure 102. The three configurations due to the multiplicative split of the deformation gradient.

The second Piola-Kirchhoff stress $\mathbf{T}$, in the intermediate configuration, is defined as

$$\mathbf{T} = J^e \mathbf{F}^{e-1} \cdot \mathbf{T}_c \cdot \mathbf{F}^{e-T}, \quad (11-2)$$

where $J^e = \det(\mathbf{F}^e)$, and $\mathbf{T}_c$ is the Cauchy stress in the current configuration. The elastic Green-Lagrange strain, in the intermediate configuration, is defined as

$$\mathbf{E} = \frac{1}{2} (\mathbf{C}^e - \mathbf{I}), \quad (11-2)$$

where $\mathbf{C}^e = \mathbf{F}^{eT} \cdot \mathbf{F}^e$ and $\mathbf{I}$ is the second order identity tensor. In the RPI model, $\mathbf{T}$ is linear with respect to $\mathbf{E}^e$, such that

$$\mathbf{T} = \mathbf{C} : \mathbf{E}^e, \quad (11-3)$$

where $\mathbf{C}$ is the fourth order stiffness tensor.

The central concept behind any crystal plasticity model is slip occurs when the shear stress, resolved on slip system, exceeds the critical resolved shear stress. The resolved shear stress, per unit current area, on slip system α is

$$\tau_c^\alpha = \mathbf{n}_c^\alpha \cdot \mathbf{T}_c \cdot \mathbf{m}_c^\alpha. \quad (11-4)$$

After several standard continuum mechanics operations (see the appendix in [140], a corresponding expression for the resolved shear stress in the intermediate configuration can be defined as

$$\tau^\alpha = \mathbf{C}^e \cdot \mathbf{T} : \mathbf{m}^\alpha \otimes \mathbf{n}^\alpha. \quad (11-5)$$

The conjugate to τ^α is $\dot{\gamma}^\alpha$, the slip rate on slip system α , in the intermediate configuration. The flow rule for the slip rate is

$$\dot{\gamma}^\alpha = \dot{\gamma}_0 \left(\frac{\tau^\alpha}{g^\alpha} \right)^{\frac{1}{b}} \text{sign}(\tau^\alpha), \quad (11-6)$$

where $\dot{\gamma}_0$ is the reference shearing rate, b is the strain rate sensitivity parameter, and g^α is the critical resolved shear stress on slip system α . The slip rate $\dot{\gamma}^\alpha$ relates back to the plastic

deformation gradient $\mathbf{F}^p$ through the plastic velocity gradient on the intermediate configuration $\mathbf{L}^p = \dot{\mathbf{F}}^p \cdot \mathbf{F}^{p-1}$ as

$$\mathbf{L}^p = \sum_{\alpha=1}^{N_s} \dot{\gamma}^\alpha \mathbf{m}^\alpha \otimes \mathbf{n}^\alpha, \quad (11-7)$$

where N_s is the total number of slip systems.

Many different hardening models for g^α exist. The Voce-Kocks model, adopted here, assumes that all slip systems start with the same hardness and harden together at the same rate. Thus, $g^\alpha = g$ and the total shearing rate on all slip systems can be defined as

$$\dot{\gamma} = \sum_{\alpha=1}^{N_s} |\dot{\gamma}^\alpha|. \quad (11-8)$$

The evolution equation for the critical resolved shear stress is

$$\dot{g} = G_0 \left(\frac{g_s - g}{g_s - g_0} \right) \dot{\gamma}, \quad (9)$$

where G_0 and g_0 are material parameters and g_s is the saturation value of the hardness at a given slip rate. The quantity g_s is given by

$$g_s = g_{s0} \left(\frac{\dot{\gamma}}{\dot{\gamma}_s} \right)^\omega, \quad (11-10)$$

where g_{s0} , $\dot{\gamma}_s$, and ω are material parameters. If we set $\omega = 0$ and integrate Eq. 11-10 with respect to time we can obtain a simple expression for g in terms of γ :

$$g = g_0 + (g_{s0} - g_0) \left[1 - \exp \left(- \frac{G_0 \gamma}{g_{s0} - g_0} \right) \right]. \quad (11-11)$$

Now, it is more apparent that g_0 is the initial critical resolved shear strength, g_{s0} is the rate-independent saturation hardness, and G_0 is the initial hardening rate.

11.1.2. Synthetic Microstructures

Two methods were used to generate synthetic microstructures. One method decomposed the domain into Voronoi cells or grains, while the other method involved Potts model simulations with a moving heat source.

The Voronoi tessellations were generated and meshed according the general procedure described in [141]. A summary, which includes a few small deviations from [141], is provided here.

1. The component at hand was meshed with hexahedral finite elements using Cubit.[142]
2. The bounding box that enclosed the mesh geometry was calculated and expanded by a small distance $D=0.4$ mm on all six sides.
3. This bounding box was fed into a Python implementation of the Maximal Poisson Sampling (MPS) algorithm.[143] The MPS algorithm generated Voronoi seed points within the bounding box and ensured that each point was disk-free, i.e. at least a minimum distance D from any previous point. Thus, one can think of D as the minimum grain size. Note that the MPS algorithm also ensured that as many points as possible were added to the bounding box (maximal sampling), and that every open location had a uniform chance of being selected next (bias-free sampling).
4. Each grain (Voronoi cell) was assigned a lattice orientation by randomly sampling from the distribution of orientations measured by HIPPO (see Section 5.1.4).
5. The Voronoi tessellation was overlaid on the finite element mesh in Python. Each element was assigned to a grain by finding the nearest Voronoi seed point to the element's average nodal coordinates.

6. The orientation of the crystal lattice at each element was saved to the finite element mesh file, so that the orientation could be read into the crystal plasticity model during a simulation.

The resulting finite element meshed Voronoi tessellation is compared against the EBSD results in Figure 103. The average Voronoi grain size appears to be close to the upper end of the grain size in the EBSD maps. Reducing the Voronoi grain size was considered, but it would have required prohibitively fine meshes, because a certain number of elements are needed per grain to resolve the strain fields. Even with a fine element size of $H = D/8$ in Figure 103, the grain boundaries are visibly stair-stepped instead of smooth. In addition, the distribution of Voronoi grain sizes is sharp and spatially uniform compared to the periodic array of sizes laid down by the laser deposition process. Finally, the Voronoi grains are equiaxed rather than elongated in various directions.

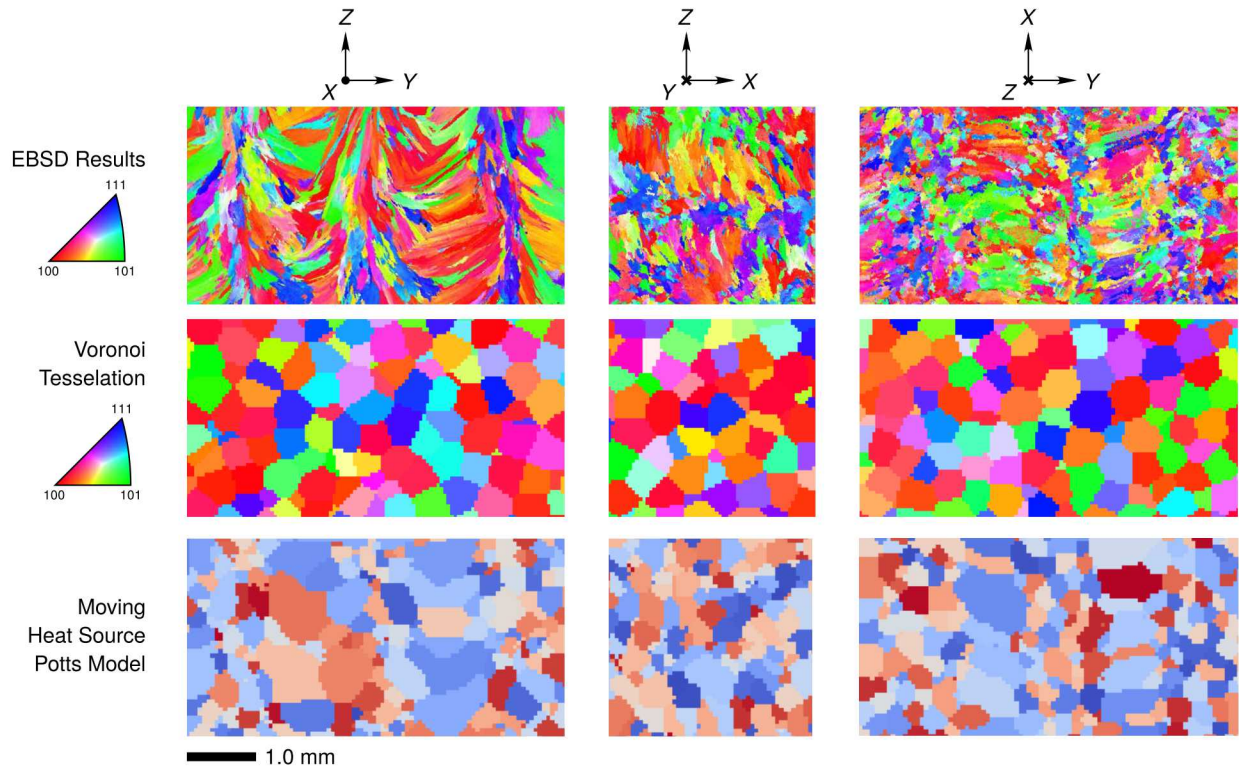


Figure 103. EBSD results compared against Voronoi tessellation results, and Potts model results for the X, Y, and Z planes. EBSD and Voronoi tessellation results have inverse pole figure coloring with respect to the X direction, while colors in the Potts model results are only used to distinguish between grains.

In an attempt to improve the match between the synthetic microstructures and the EBSD data, a secondary approach was also pursued. In a classical Potts model grain growth simulation, one starts with a fine equiaxed grain structure. The simulation randomly selects a lattice site and attempts to change its grain identification number to that of a randomly selected neighbor with a different identification number. The total energy of the system is recalculated, and the probability of accepting the change is determined by the Metropolis function. This process causes grains to grow until the simulation is terminated. Typically, the temperature field is spatially and temporally uniform, but Sandia researchers have also modeled solidification and grain growth around a weld using a moving heat source.[144] Rodgers recently extended this capability to the multi-pass, three dimensional, arbitrary weld paths used in additive manufacturing. Although the simulations have some physical basis, they are not predictive. For example, Rodgers used actual processing parameters, such as the laser scan rate and hatch spacing, in his simulations, but he tuned other simulation inputs, such as melt pool size and melt pool temperature, to get a synthetic microstructure that resembled the EBSD results.

As shown in Figure 103, the moving heat source Potts model results qualitatively agree with the EBSD measurements. Note that grain colors in the Potts model results do not correspond to lattice orientation. Colors are only used to distinguish between different grains. In the X and Z planes, the grains sizes are small along the laser scans and large in-between the laser scans, which matches the EBSD results. The Potts model grains also tend to be elongated in the direction of the heat source, similar to the EBSD observations. Future effort could focus on reducing the Potts model grain sizes slightly and capturing some of the finer features of the actual grains, but the improvement over the Voronoi tessellation is undeniable.

Unfortunately, efforts to robustly mesh the Potts model results were unsuccessful. Lattice sites in the Monte Carlo simulations are voxels, which results in stair-stepped grain boundaries. One could simply use a hexahedral element at each voxel, but this approach does not allow non-Cartesian component geometries or easy mesh refinement. One could run a Monte Carlo simulation with smaller voxels to refine the mesh, but the stochastic nature of Monte Carlo would lead to a different microstructure with each mesh refinement. Instead Sculpt [142,145] was used in attempts to

smooth the grain boundaries and mesh the resulting microstructure. For anything over roughly 100 grains, Sculpt would either fail to mesh some part of the microstructure or create meshes where elements inverted immediately upon running solid mechanics simulations. Progress was made after heavy troubleshooting with Steven Owen, the creator of Sculpt, but the complex microstructures in Figure 103 are still problematic at this time. Consequently, all crystal plasticity simulations discussed herein used Voronoi tessellation microstructures similar to those in Figure 103.

11.1.2. General Simulation Settings

All solid mechanics simulations were run in Sierra/Solid Mechanics.[146] Time steps corresponded to no larger than 0.1 % global (average) strain on a specimen. The implicit solver iterated until the relative residual norm was less than $R_{\text{lim}} = 10^{-6}$. The relative residual norm was defined as the L2 norm of the total residual divided by the L2 norm of the reaction forces at the boundaries. Unless otherwise noted, the selective deviatoric element was used in all simulations.

11.1.3. Mesh Convergence

The stair-stepped grain boundaries in the Voronoi tessellation microstructures cause stress concentrations that one would expect to retard mesh convergence rates. Pyle *et al.* [147] studied mesh convergence of a representative volume element with 107 grains, voxelated grain boundaries, and almost 200 thousand elements in their finest mesh. They found only a 0.15 % change in von Mises stress between their finest and intermediate mesh, but the difference in the number of elements between the finest and intermediate meshes was a factor of 1.3 instead of the usual factor of 8 after cutting all element lengths in half. They presumably did not have the computational horsepower to refine their meshes to such levels.

An independent mesh convergence study was performed herein to determine the number of elements needed per grain. Figure 104a depicts a cuboid compression specimen with length $L = 14.0$ mm, width $W = 6.6$ mm, and thickness $T = 6.3$ mm. Lower case (x, y, z) coordinates denote

directions with respect to the specimen, while upper case (X, Y, Z) coordinates continue to denote directions with respect to the additive manufacturing build process. For this particular specimen, the z -axis happened to correspond to the Z -axis. The specimen was compressed along the z -axis, causing a displacement δ , at an engineering strain rate of $\frac{\delta}{L} = -1 \times 10^{-3} \text{ s}^{-1}$. Sliding was not allowed between the specimen and the platens. The displacement δ_e across a virtual extensometer $L_e = 7.0 \text{ mm}$ was monitored, in order to calculate the axial log strain as $\epsilon_{zz} = \ln\left(\frac{\delta_e}{L_e} + 1\right)$. The axial force P and undeformed cross-sectional area $A = TW$ were used to calculate the typical approximation to the axial Cauchy stress, given by $\sigma_{zz} = \frac{P}{A}\left(\frac{\delta_e}{L_e} + 1\right)$. The crystal plasticity model and material parameters were the same as those described in the following section, which describes the material model calibration process. A Voronoi tessellation microstructure with 7570 grains was overlaid on four meshes with element sizes $H = D, \frac{D}{2}, \frac{D}{4}$, and $\frac{D}{8}$. The element sizes relative to the grains and specimen geometry are shown in Figure 105a.

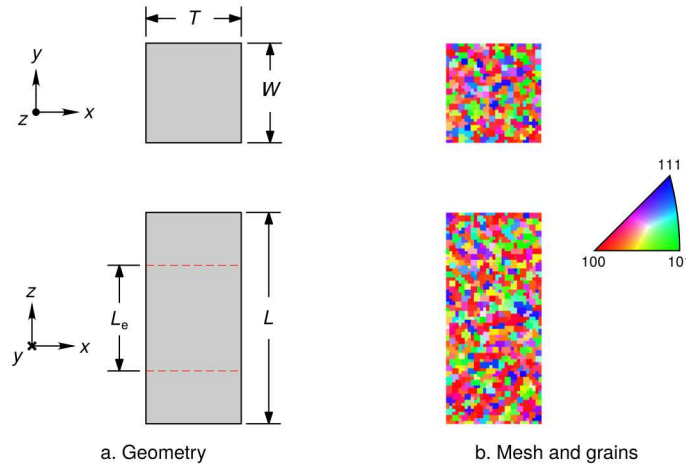


Figure 104. Cuboid compression specimen. Grains have inverse pole figure coloring with respect to the X direction.

As might have been expected, the stress-strain curves converge slowly with mesh refinement. Cutting the element lengths in half does not cause large changes in σ_{zz} for a given ϵ_{zz} in Figure 105b. For example, the $H = \frac{D}{8}$ simulation differs from the $H = \frac{D}{4}$ predictions by only 1.4 % at $\epsilon_{zz} = 2 \%$. On the other hand, a far finer mesh is probably required to be within 1 % of the

numerically exact solution, because the rate of convergence is quite slow. The $H = \frac{D}{8}$ simulation included 4.6 million elements and only reached $\varepsilon_{zz} = 2.9\%$ after 72 hours on 3000 cores, therefore a $H = \frac{D}{16}$ simulation is intractable with today's computational resources.

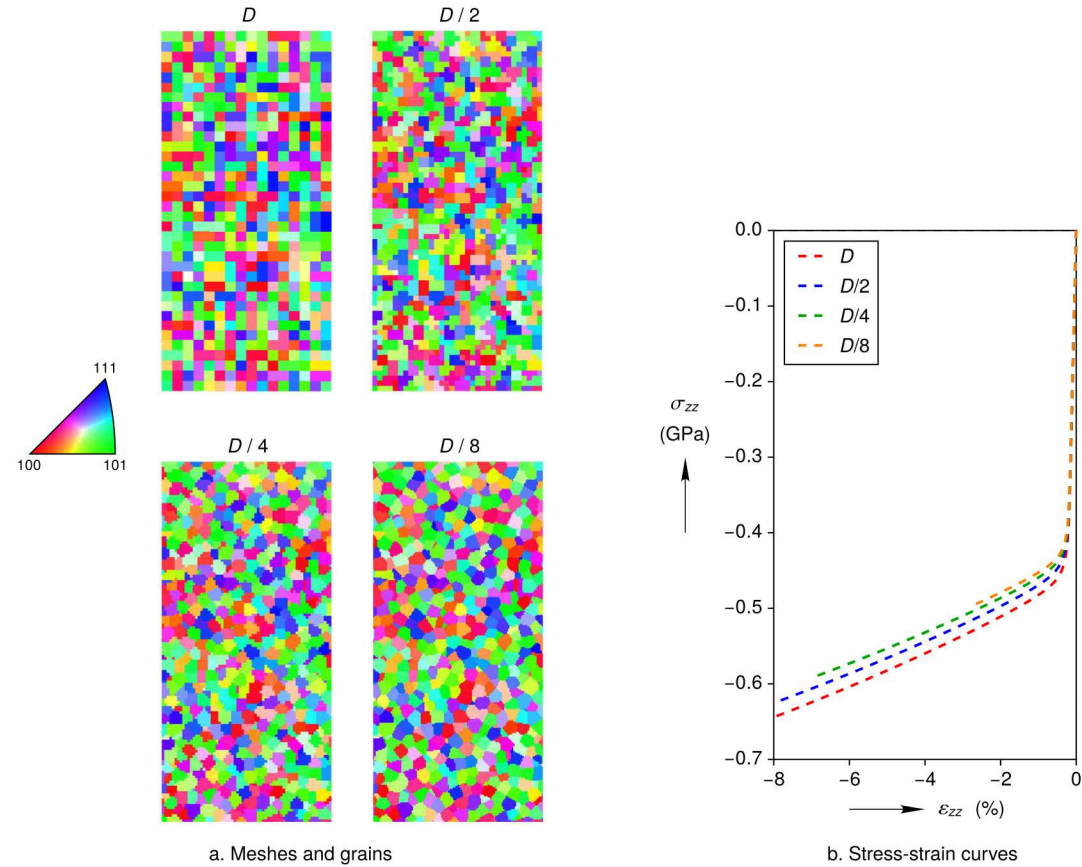


Figure 105. Mesh convergence study. Grains have inverse pole figure coloring with respect to the Z direction.

Given that mesh convergence was deemed impossible, it was decided to use $H = \frac{D}{2}$ for all further simulations as a compromise between speed and accuracy. Future improvements to Sculpt may enable meshes that conform to the grain boundaries, which should significantly increase the rate of convergence. In addition, Sandia is investing in a new crystal plasticity model that solves the constitutive differential equations more robustly, which should reduce overall simulation time.

11.1.4. Crystal Plasticity Model Calibration

Ideally, one should calibrate all crystal plasticity model parameters against single crystal experiments. Here, we were only able to use the elastic constants from single crystal experiments.

The elastic constants C_{11} , C_{12} , and C_{44} in Table 11 were measured on 304L stainless steel in [148]. Single crystals of additively manufactured 304L stainless steel were not available to measure properties such as yield strength. Even if it were possible to make a single additively manufactured crystal of 304L stainless steel, it is highly unlikely to have the same critical resolved shear stress as a polycrystalline sample. Instead, model parameters were tuned such that simulations of a polycrystalline sample in tension roughly matched an experimentally measured stress-strain curve.

Table 11: Crystal Plasticity Model Parameters

Parameter	Value
C_{11}	204.6 GPa
C_{12}	137.7 GPa
C_{44}	126.2 GPa
b	0.013
$\dot{\gamma}_0$	0.01 1/s
$\dot{\gamma}_s$	0.001 1/s
ω	0
g_0	167.37 MPa
g_{s0}	370.0 MPa
G_0	338.0 MPa

The tension test was performed by LANL on specimen A22, whose tensile axis was aligned with the X -axis of a 3.8 kW X-bar. The dog bone specimen had a diameter $2C = 3$ mm and length $L = 30$ mm (see Figure 106a). The displacement measured by the actuator was δ . An extensometer of gage length $L_e = 12.5$ mm measured the axial displacement δ_e . The experiments involved intermittently stretching the dog bone, holding $\frac{\delta}{L}$ fixed during neutron diffraction measurements, and resuming stretching. The strain rate was $\frac{\dot{\delta}}{L} = 1.77 \times 10^{-4} \text{ s}^{-1}$ until $\frac{\delta_e}{L_e} = 8.15 \%$, but it was increased to $\frac{\dot{\delta}}{L} = 3.3 \times 10^{-4} \text{ s}^{-1}$ for the remainder of the experiment. Similar to Section 11.1.3, the

axial log strain was calculated as $\varepsilon_{zz} = \ln\left(\frac{\delta_e}{L_e} + 1\right)$, and the approximate axial Cauchy stress was calculated as $\sigma_{zz} = \frac{P}{A}\left(\frac{\delta_e}{L_e} + 1\right)$.

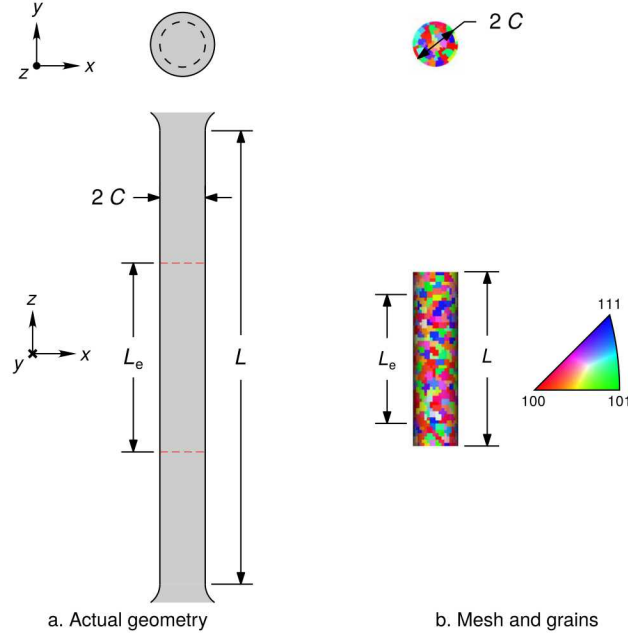


Figure 106. Specimen A22. Grains have inverse pole figure coloring with respect to the X direction.

Simulations of A22 matched many of the experiment inputs, but not all. The simulated geometry maintained the same diameter, but the extensometer gage length was reduced to $L_e = \frac{6}{\cos(\frac{\pi}{4})} = 8.485$ mm and the gage length was reduced to $L = L_e + 2C$ (see Figure 106b) for computational efficiency. The simulated extensometer gage length was chosen so that the volume of material inside the gage length was equal to the volume of material interrogated by a 6.0 mm wide neutron beam at an angle of $\frac{\pi}{4}$ to the tensile axis. Dirichlet boundary conditions were used in the z -direction to cause a displacement δ across the gage length L . Contraction in the x and y -directions, however, was left unconstrained at the boundaries.

The strain rate sensitivity parameter and hardening parameters were selected by manual tuning the simulated stress-strain curve to match the experiment. Automated optimization schemes were

considered, but the simulations ran too slowly to permit the hundreds of runs typically needed in such schemes. The strain rate sensitivity parameter b was calibrated using the stress relaxations during neutron diffraction of specimen A22. (See Section 11.2.1 for the comparison between the simulated and measured stress relaxation.) Other rate parameters $\dot{\gamma}_0$, $\dot{\gamma}_s$, and ω were simply assigned the values in Table 11. The hardness parameters g_0 , g_{s0} , and G_0 were fit by running a simulation at $\frac{\dot{\delta}}{L} = 1.77 \times 10^{-4} \text{ s}^{-1}$ up to $\frac{\delta_e}{L_e} = 8.15 \%$, and at $\frac{\dot{\delta}}{L} = 3.3 \times 10^{-4} \text{ s}^{-1}$ for the remainder. Pauses for neutron diffraction were skipped for computational efficiency. The chosen parameters are listed in Table 11.

Despite the expense of running these simulations, the resulting fit of the stress-strain curve is fairly good (see Figure 107). If one ignores the stress relaxations during neutron diffraction, the simulated curve is only little bit higher than the measured. Once the mesh convergence issues discussed in Section 11.1.3 have been resolved, it may be worth optimizing the fit further.

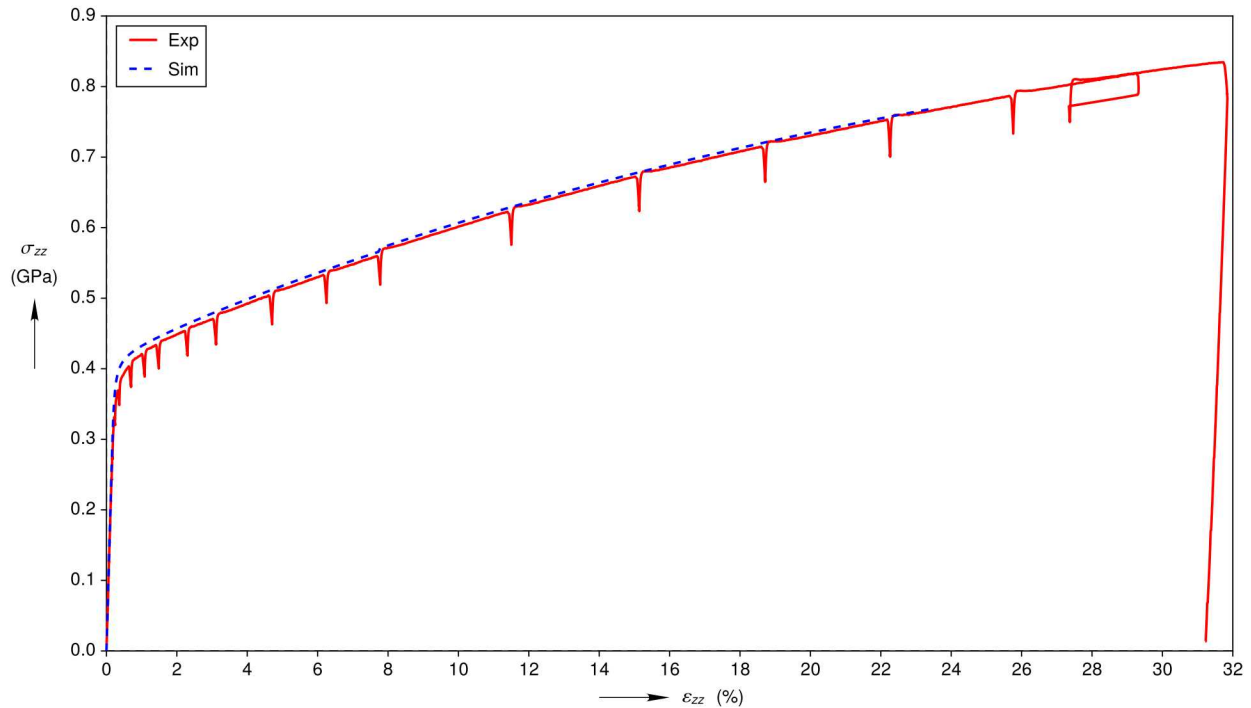


Figure 107. Simulated and measured stress-strain curves compared for specimen A22. The simulation did not include the pauses in loading that led to the stress relaxations in the experimental data.

11.1.5. Macroscopic J_2 Flow Model Calibration

The macroscopic J_2 flow model utilized was the Dynamic Strain Aging (DSA) model.[144] Initially, the viscoplastic BCJ_MEM model [146] was used. BCJ_MEM ran well when it was configured to be rate independent, but simulations of the tube tension-torsion experiments failed to converge when rate dependence was added to BCJ_MEM. The Sierra/Solid Mechanics manual warns of potential issues with rate dependence and implicit quasi-static solvers. Fortunately, tension-torsion simulations with the DSA model parameterized to be identical to BCJ_MEM ran without any issues. Presumably, the DSA model's more robust integration algorithms solved the issue.

The DSA model includes many features such as recrystallization, temperature dependence, the conversion of plastic work into heat, and damage. Here, only the isotropic elastic constants, isotropic hardening, and strain rate dependence are calibrated. At a constant strain rate, constant temperature, no kinematic hardening, no stage IV hardening, no static recovery, the flow stress is

$$\sigma_f = \left\{ \sigma_Y + \frac{H}{R_d} [1 - \exp(-R_d \varepsilon^p)] \right\} \left\{ 1 + \sinh^{-1} \left[\left(\frac{\dot{\varepsilon}^p}{f} \right)^{1/n} \right] \right\} \quad (11-12)$$

where σ_Y is the initial yield constant, H is the linear hardening modulus, R_d is the dynamic recovery constant, ε^p is the equivalent plastic strain, f is the plastic strain rate normalization parameter, and n is the plastic strain rate exponent parameter. In (11-12), the rate independent behavior is governed by the terms in the left-most curly brackets, and the rate dependent behavior is governed by the terms in the right-most curly brackets.

As discussed in Chapter 7, the properties of our additively manufactured 304L stainless steel vary considerably from specimen to specimen. Rather than struggle with the variability, the DSA model was calibrated to match crystal plasticity simulations of tension tests at three different strain rates. The RPI model parameters listed in Table 11 were used to run the simulations in Section 11.1.4 at

$$\frac{\dot{\delta}}{L} = 1.0 \times 10^{-5} \text{ s}^{-1}, 3.3 \times 10^{-4} \text{ s}^{-1}, \text{ and } 1.0 \times 10^{-3} \text{ s}^{-1}.$$

First, the elastic properties were calibrated. Young's modulus was measured in the crystal plasticity simulations as $E = 175$ GPa. Poisson's ratio in each direction could have also been extracted from the crystal plasticity simulations, but the value $\nu = 0.29$ from [148] was utilized instead. This is not expected to have a significant impact, but simulations would have to be corrected and rerun to be certain.

Second, the rate dependence and hardening were calibrated. The yield strength was measured in the crystal plasticity stress-strain curves using a 0.33 % offset. The equivalent plastic strain rate was measured by subtracting off the elastic strain from the simulated stress-strain curve to obtain ε^p and then calculating its rate at $\varepsilon_{zz} = 1$ %. The three yield strengths σ_y are plotted against the three ε^p in Figure 108a. The DSA model parameters σ_Y , f , and n were found by plugging the three pairs of $(\varepsilon^p, \sigma_y)$ values into (11-12 with $\varepsilon^p = 0$ to create a system of three equations and three unknowns. The resulting parameter values are listed in Table 12, and the J_2 flow DSA model fit is plotted in Figure 108a. Finally, the hardening parameters H and R_d were manually tuned to the crystal plasticity simulated stress-strain curve at $\frac{\dot{\delta}}{L} = 3.3 \times 10^{-4} \text{ s}^{-1}$.

Table 12: DSA Model Parameters

Parameter	Value
E	175 GPa
ν	0.29
σ_Y	214 MPa
f	1.227×10^{-5}
n	29.84
H	1235 MPa
R_d	4.8

To confirm the J_2 flow DSA model calibration, three single element simulations were run at the same strain rates as the three crystal plasticity simulations. As shown in Figure 108b, the fit is nearly perfect for the range of strains simulated. The J_2 flow model results only deviate from the crystal plasticity results near initial yield, where the J_2 flow model predicts a sharp elastic-plastic transition. The crystal plasticity simulations, on the other hand, predict a rounded elastic-plastic transition because yielding progresses from favorably aligned crystals to unfavorably aligned crystals as deformation is applied.

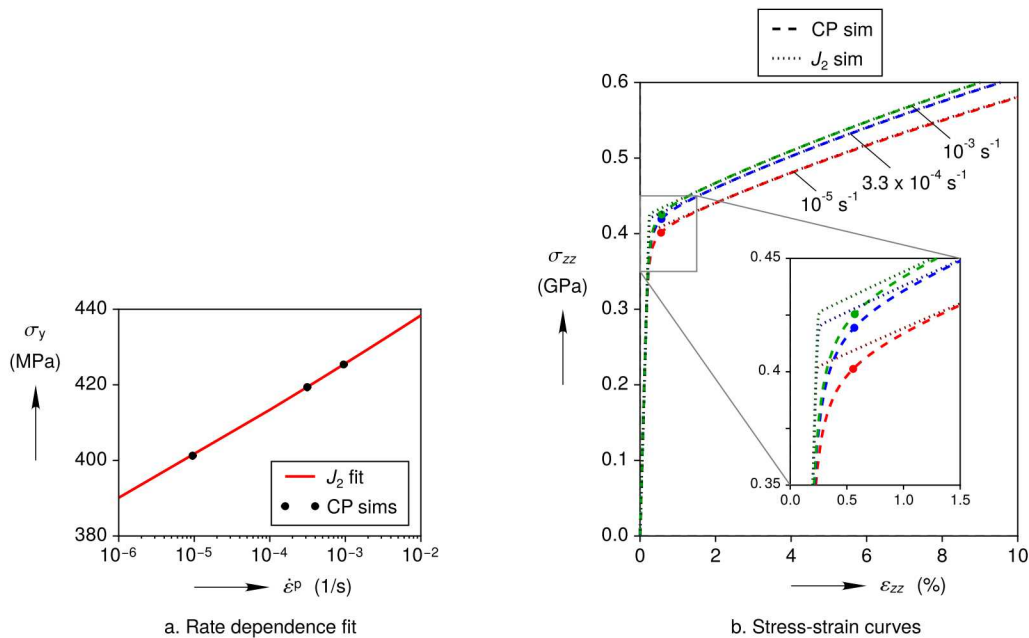


Figure 108. Macroscopic J_2 flow model calibration

11.2. Model Predictions

11.2.1. Lattice Strains

As discussed in Section 7.4, SMARTS measured the crystal lattice spacing while specimens were mechanically deformed. From those spacings, one can calculate the elastic strains suffered by the lattice, without the plastic strains. These lattice strains can be used to validate crystal plasticity simulations, so the tension test on specimen A22 was simulated.

The simulations were the same as those discussed in Section 11.1.4, except for two important differences. First, the pauses during neutron diffraction were included in the simulation. To do so, the experimentally measured engineering strain from the mechanical extensometer was simply multiplied by the simulation gage length L and used as a time varying displacement boundary condition. Second, simulations using the selective deviatoric element slowed to a crawl at the second pause ($\varepsilon_{zz} = 0.03\%$), for unknown reasons. After several attempts to troubleshoot the problem, simulations continued to run for days with virtually no progress. The mean quadrature element in Sierra/Solid Mechanics did not exhibit the problem, so it was utilized instead.

Figure 109 compares the simulated and experimentally measured macroscopic stress and strain. The engineering strain measured by the extensometer δ_e/L_e and the engineering stress P/A histories are shown in Figure 109a, while Figure 109b depicts the Cauchy stress versus log strain curves. The simulated and measured engineering strain histories are close, but not quite identical, probably because the top and bottom of the simulated geometry are slightly too close to the extensometer gage length. A more significant difference between the engineering stress histories is seen in the upper half of Figure 109a. Once yielding begins, the simulation overshoots the measured stress, and the deviation grows with increasing strain. Contrast Figure 109b with Figure 107, where the simulation results and measurements agree quite well. Clearly, the mean quadrature element, which is less accurate than the selective deviatoric element, calculates higher stresses for the same strain. Regardless, the difference is still reasonably small, and does not preclude comparing the lattice strains.

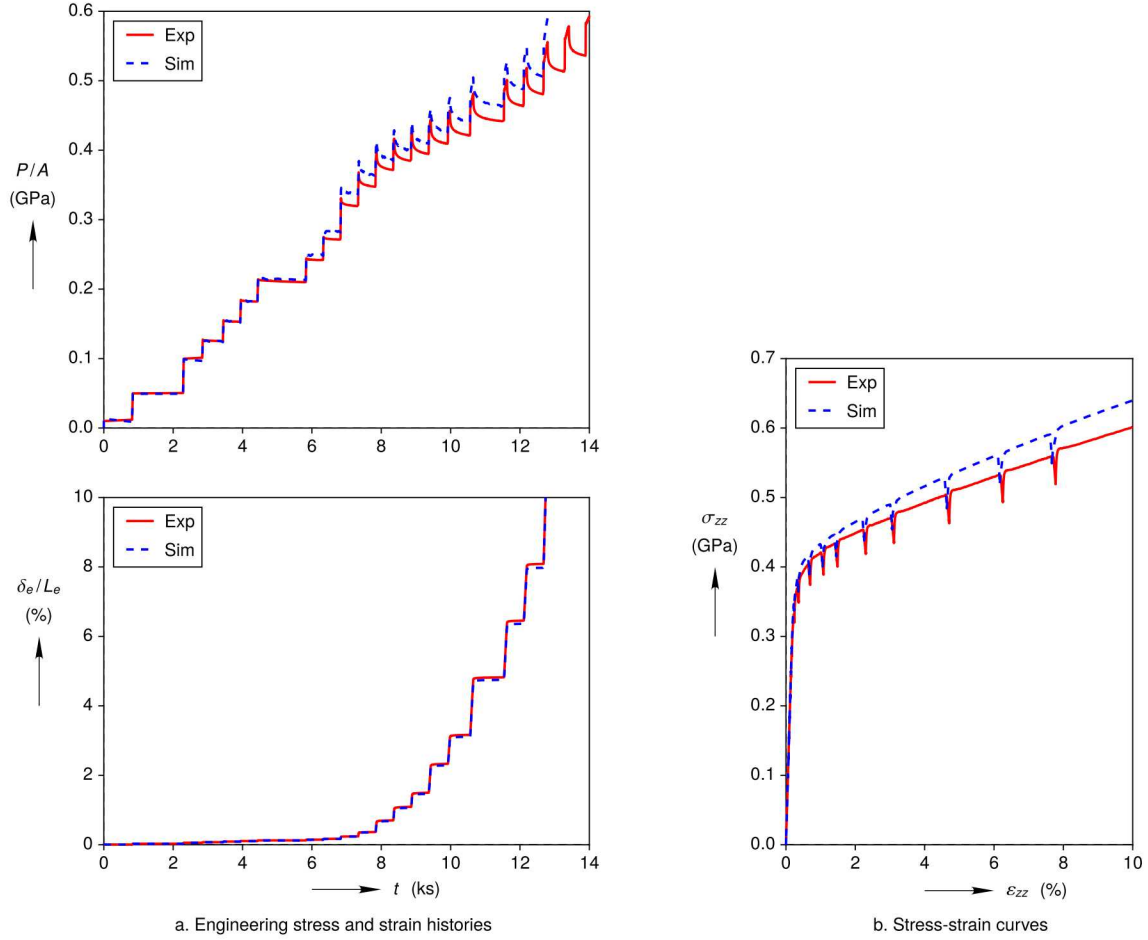


Figure 109. Simulated and measured macroscopic stress and strain compared for specimen A22. (Simulation utilized the mean quadrature element.)

The lattice strains were calculated from the simulation results in the following manner. The experiments only measured the strain perpendicular to five specific crystallographic planes whose normal was within $\pm 6.5^\circ$ of the tensile axis, so the analysis here must do the same. As mentioned in Section 11.1.1, the lattice is undisturbed by the plastic deformation gradient $\mathbf{F}^e$, so a unit vector $\mathbf{n}_0^{hkl}$ normal to a crystallographic plane (hkl) in the reference configuration coincides with the unit vector $\mathbf{n}^{hkl}$ to the same crystallographic plane in the intermediate configuration. Plane normals, however, are deformed by the elastic deformation gradient $\mathbf{F}^e$. The lattice plane normal in the current configuration was calculated as

$$\hat{\mathbf{n}}_c^{hkl} = \mathbf{F}^{e-T} \cdot \mathbf{n}_0^{hkl}, \quad (11-13)$$

and then converted into a unit vector

$$\mathbf{n}_c^{hkl} = \hat{\mathbf{n}}_c^{hkl} / \|\hat{\mathbf{n}}_c^{hkl}\|_2, \quad (11-14)$$

where $\|\cdot\|_2$ denotes the L2 norm of its argument. Note that (11-13 and (11-14 together constitute of statement of Nanson's relation. To determine whether the current lattice plane normal was within the $\pm 6.5^\circ$ tolerance, the angle between $\mathbf{n}_c^{hkl}$ and the unit vector aligned with the tensile axis $\mathbf{e}_z$ was found

$$\beta = \cos^{-1}(\mathbf{n}_c^{hkl} \cdot \mathbf{e}_z). \quad \text{Equation 15}$$

After isolating the sufficiently aligned grains, the volume averaged lattice strain was computed as

$$\varepsilon^{hkl} = \frac{1}{V} \int_{\Omega} \mathbf{n}_c^{hkl} \cdot \mathbf{E}^e \cdot \mathbf{n}_c^{hkl} dV, \quad \text{Equation 16}$$

where Ω is the domain within the extensometer and V is its volume. Recall that the extensometer gage length L_e was specifically chosen so that V would be equal to the volume of material interrogated by the neutron diffraction beam.

The computed lattice strains agree reasonably well with the measured lattice strains. are proportional to stresses, so the plot in

Figure 110a resembles a stress-strain curve, where the deviation from linearity indicates the onset of yielding. Note, however, the lattice strain magnitude on a plane should not be interpreted as the magnitude of the stress, because lattice strains are *tensorially* proportional to stresses. The lattice strains on all planes are over predicted by varying amounts in

Figure 110a. With a finer mesh, the lattice strains (and stress in Figure 109) would be reduced, but maybe not enough to completely eliminate the differences. Regardless, it is encouraging that the crystal plasticity simulation captures the grouping of the curves.

Figure 110b is another representation of the data where the elastic anisotropy prior to the start of yielding is more apparent. The $\{311\}$ and $\{200\}$ planes are elastically more compliant than the $\{111\}$, $\{220\}$, and $\{331\}$ planes. At around 310 MPa, the $\{111\}$, $\{220\}$, and $\{331\}$ planes yield first, as evidenced by their reduced rate of elastic strain accumulation. The $\{311\}$ planes and the $\{200\}$ planes accrue more elastic strain while the other planes yield, and then the $\{311\}$ and $\{200\}$ planes yield at roughly 380 MPa. The simulation captures the elastic anisotropy, yield, and post-yield fairly well.

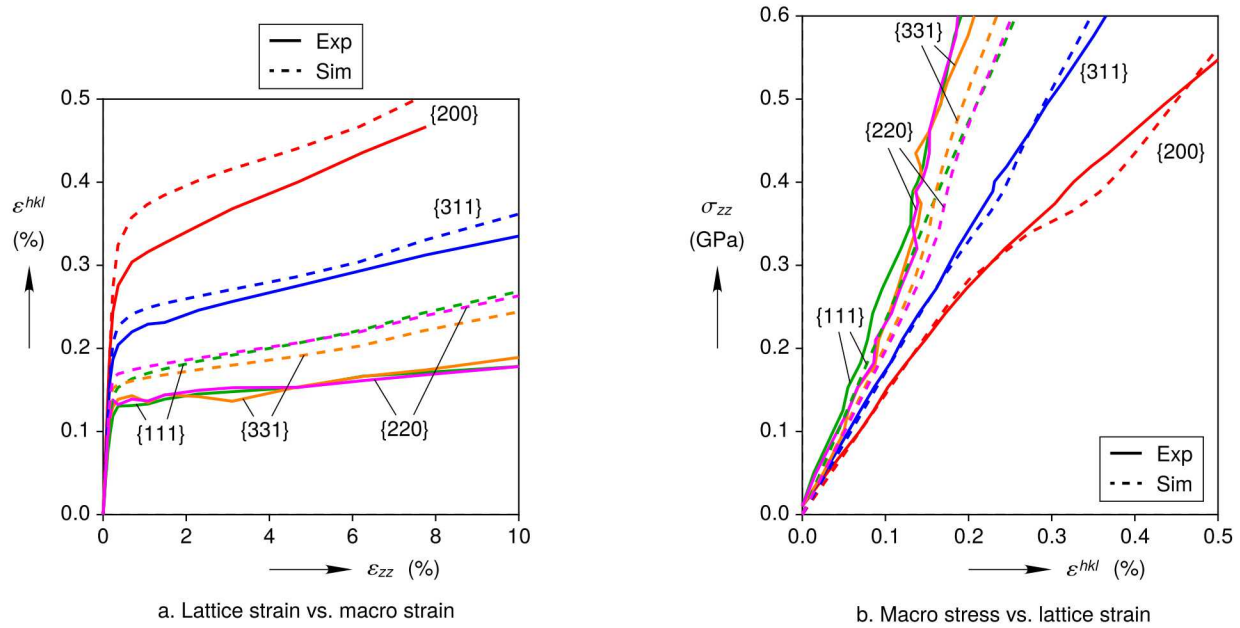


Figure 110. Simulated and measured lattice strains compared for specimen A22. (Simulation utilized the mean quadrature element.)

11.2.2. Anisotropy

The pole figures in Section 5.1.4 revealed a significant degree of texture, which means the mechanical behavior is likely to exhibit anisotropy. Attempts were made to predict the anisotropy using a cube shaped representative volume element (RVE) with the experimentally measured texture. However, the periodic boundary conditions in Sierra/Solid Mechanics did not perform as expected, so a different approach was taken. Experimental measurements of the anisotropy have been collected, so the experiments were simulated. Seven compression specimens, each with a different orientation, were cut from a 3.8 kW X-bar to characterize the behavior in seven different directions. They have been tested, but the experimental data is still being post-processed. At this juncture, only the simulation predictions will be shown.

The orientations of the seven cuboid compression specimens are displayed in Figure 111. These cuboid specimens are the same as those described in Section 11.1.3. As previously noted, two coordinate systems are defined. Lower case (x, y, z) coordinates denote directions with respect to the specimen, while upper case (X, Y, Z) coordinates continue to denote directions with respect to the additive manufacturing build process. The z -axis is always aligned with the $L = 14$ mm

dimension (the compression axis), while the x-axis is always aligned with the $T = 6.3$ mm dimension. The names for the cuboids refer to the orientation of the z-axis within the (X, Y, Z) coordinate system. For example, the X-cuboid's z-axis is aligned with the X -axis, and the ZX-cuboid's z-axis is 45° between the X and Z axes. Although the z-axis orientations in Figure 111 match those of the actual experimental specimens, the x-axis orientation may be off by 90° . Thus, Figure 111 should only be used to interpret the simulations presented here.

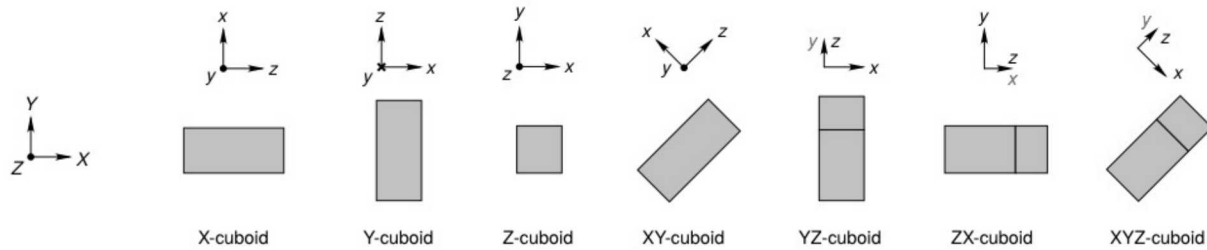


Figure 111. Cuboid compression specimen orientations

The simulations were conducted in the same manner as Section 11.1.3. The virtual extensometer gage length was $L_e = 7.0$ mm, and the specimen was compressed at an engineering strain rate of $\frac{\dot{\delta}}{L} = -1 \times 10^{-3} \text{ s}^{-1}$. Sliding was not allowed between the specimen and the platens.

As expected, the simulations predict a fair degree of anisotropy. The stress-strain curves are shown in Figure 112a, where the differences in flow stress are apparent. At $\epsilon_{zz} = -8\%$, the spread in flow stress is about 8 % of the average flow stress. The elastic modulus of each specimen was measured by fitting a line through the origin down to $\epsilon_{zz} = -0.3\%$, resulting in the measurements in Figure 112b. These moduli were then used to measure the initial yield stress using a $\epsilon_{zz} = -0.2\%$ offset, resulting in the measurements in Figure 112c. Although the moduli appear to be more sensitive to the texture than the yield stress, the two correlate with one another. For example, the X and YZ cuboids both have the lowest modulus and lowest yield strength, while the XY cuboid has the highest modulus and highest yield strength.

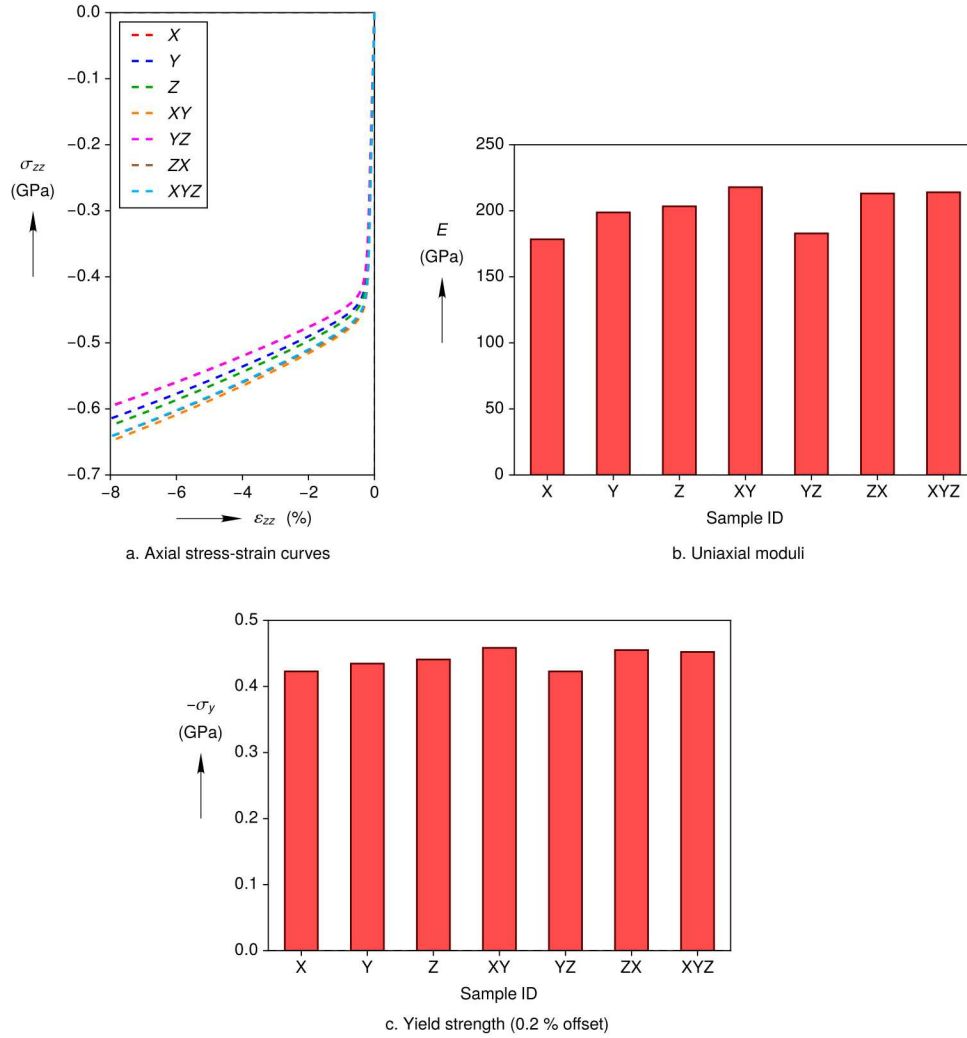


Figure 112. Predictions of anisotropy in the elastic stiffness and yield strength of cuboid compression specimens

The lateral strains (see Figure 113) show more sensitivity to texture than the yield strength or the uniaxial elastic moduli. In contrast to the axial strain ϵ_{zz} , which was measured by tracking the displacement of an extensometer on the surface of the specimen, the lateral strains $\bar{\epsilon}_{xx}$ and $\bar{\epsilon}_{yy}$ were measured by taking a volume average of the logarithmic strain at the Gauss quadrature points. When the volume averaged axial strain $\bar{\epsilon}_{zz}$ was compared against ϵ_{zz} , they differed by about 7 % at $\epsilon_{zz} = 8$ % strain. Therefore, these two measurement methods are not consistent and further work needs to be done to reconcile them. For the time being, however, the curves in Figure 113 can still be analyzed cautiously. An isotropic material laterally expands the same percentage in both directions when it is compressed, so it would have a slope of one in Figure 113. The anisotropic cuboid slopes, however, vary from 0.43 to 1.88. The lateral strains also have a

connection to concepts from macroscopic plasticity. If one calculates the lateral plastic strains ε_{xx}^p and ε_{yy}^p , the slope $r = \frac{d\varepsilon_{xx}^p}{d\varepsilon_{yy}^p}$ is the Lankford ratio. The yield stresses Figure 112c locate a point on the yield surface, while the Lankford ratios are related to the yield surface normal at that point.

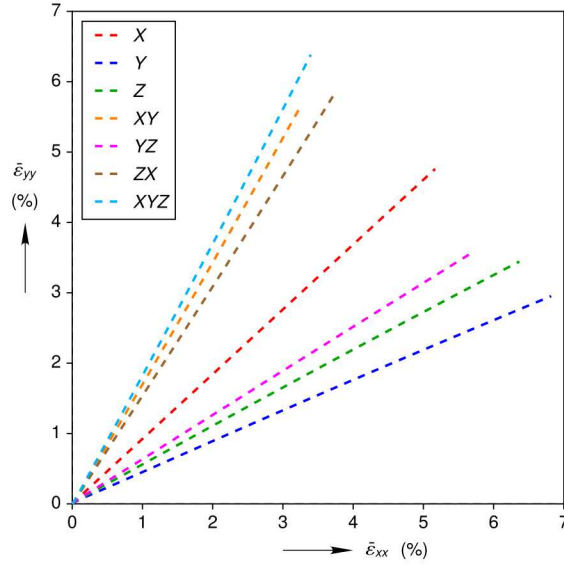


Figure 113. Predictions of anisotropy in the lateral strains of cuboid compression specimens

11.2.3. Axial-Torsion and Torsion-Axial Tests

The crystal plasticity finite element method (CPFEM) is attractive because it has a stronger physical underpinning than classical macroscopic plasticity models. One can predict the anisotropy of a material based on a single tension test and a texture measurement. On the other hand, CPFEM still employs many assumptions so it may or may not be more accurate than an empirically calibrated macroscopic model. CPFEM also requires orders of magnitude more computational resources, making it relegated to mostly research applications. In order to assess whether the extra computational cost produces greater accuracy, the RPI crystal plasticity model and the DSA J_2 flow model were both used to simulate two non-proportional experiments on tubes.

The tube geometry is shown in Figure 114a, along with a cylindrical coordinate system. The length of the tube (the z -axis) was aligned with the X -axis of a 3.8 kW X-bar before it was cut out using electrical discharge machining. The wall thickness in the thin section was $B = 0.508$ mm. The

length of the smooth section was $L_s = 10.16$ mm. Other tube features are referenced off the thickness B . An axial actuator stretched the tube, and a rotary actuator twisted the tube. The axial displacement δ and angle of twist ϕ were measured at the actuators where they were used for control purposes. While actuating, the axial displacement rate was $\dot{\delta} = 0.33$ mm/ks, and the twist rate was $\dot{\phi} = 0.94^\circ/\text{ks}$.

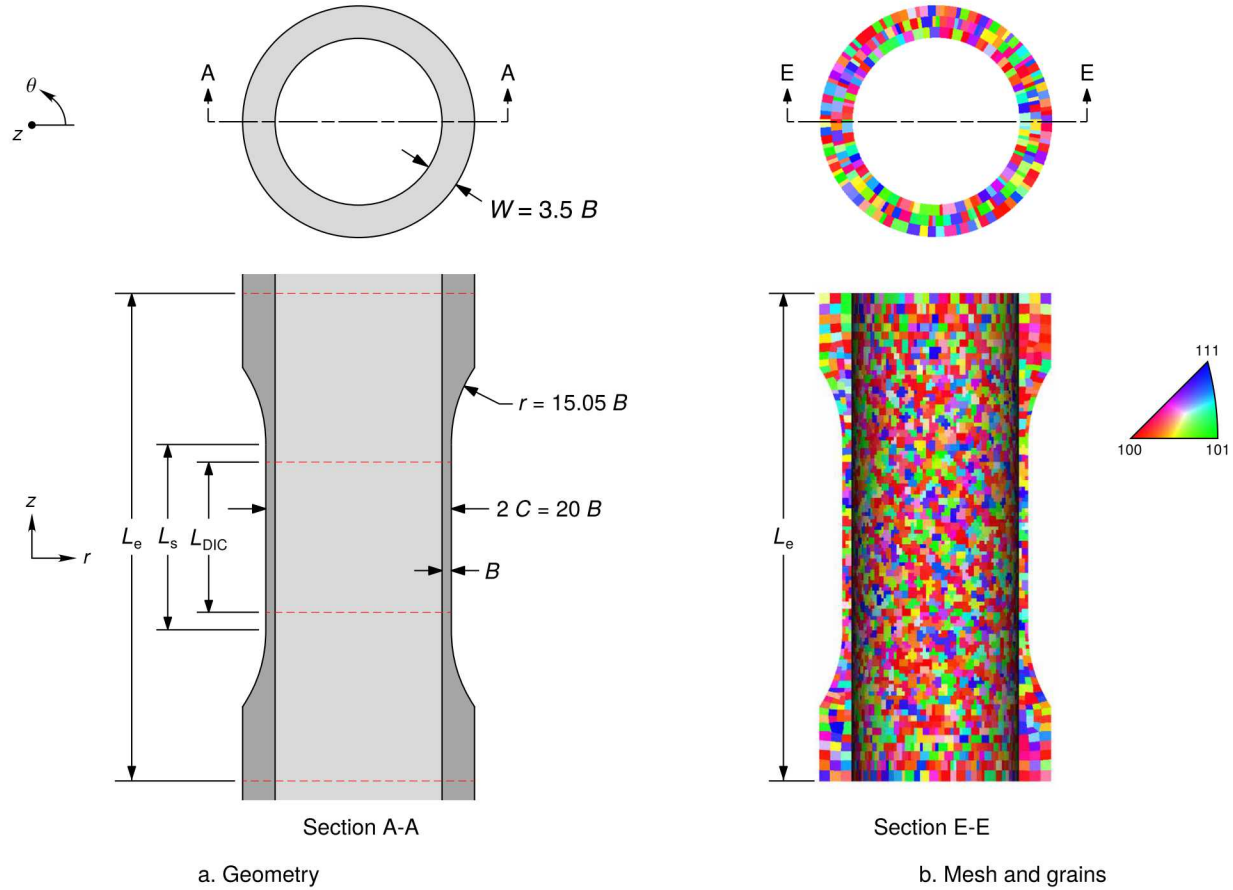


Figure 114. Tube specimen. Grains have inverse pole figure coloring with respect to the X direction.

The deformation of the tube's outer surface was monitored using stereo digital image correlation (DIC) [12]. A DIC "virtual" extensometer measured the axial displacement δ_e and the transverse displacement δ_x between two points separated by L_e . The angle of twist between the two points was calculated as $\phi_e = \sin(\delta_x/\eta)$, where $\eta = C - B + W$ is the tube radius in the thick wall region. DIC was also used to measure the deformation on the outer surface of the thin wall section. The DIC software returned the Biot strain, which was averaged over a gage length L_{DIC} ,

to obtain the axial strain ε_{zz}^B and the shear strain $\varepsilon_{\theta z}^B$. The lengths L_e and L_{DIC} varied, so they are listed for the Axial-Torsion experiment and the Torsion-Axial experiment in Table 13.

Table 13: Tube Gage Lengths

Experiment ID	L_e	L_{DIC}
Axial-Torsion	26.73 mm	8.23 mm
Torsion-Axial	26.39 mm	7.15 mm

The finite element mesh used in the crystal plasticity simulations is shown in Figure 114b. Rather than simulate the entire specimen length between the grips, the mesh includes only the length L_e . With the top and bottom of the length L_e exposed, the experimentally measured values of δ_e and ϕ_e were applied as boundary conditions. Contractions in the r and θ -directions were left unconstrained at the boundaries. Due to the thin wall along L_s , only one or two grains, and three elements, existed through the wall thickness.

The same mesh was used in the J_2 flow model simulations, except the top half was discarded by taking advantage of the symmetry plane halfway along L_e . Although a mesh convergence study was not performed for the J_2 flow model simulations, Reedlunn and Lu [151] simulated a similar tube geometry under tension-torsion using a different J_2 flow model. Appendix B in [151] shows that a single Q1P0 element (similar to the selective deviatoric element) through the thickness was sufficient for the moderate deformations applied here.

Sierra/Solid Mechanics can output the Biot strain, but only in a Cartesian coordinate system and at the Gauss quadrature points. In order to compare directly to the experiments, the Biot strain field was extrapolated to the nodes via a L2 projection built into Sierra/Solid Mechanics. Then, user-defined functions were used to transform the components into a cylindrical coordinate system. Finally, the Biot strain fields were averaged over outer surface of the tube, along the length L_{DIC} . This proved to be worthwhile because engineering strain measures differ significantly from the Biot strain under the axial-torsion deformations applied here.

The programmed actuator paths and the measured virtual extensometer paths did not quite agree in the experiments. In the Axial-Torsion experiment (see Figure 115a), the specimen was stretched and then twisted. The virtual extensometer registered 20 % less axial displacement than the axial actuator and then followed a non-linear path during twisting. In the Torsion-Axial experiment (see Figure 115b), the specimen was twisted and then stretched. The ϕ_e measurement almost matched the ϕ measurement, but the δ_e measurement differed from the δ measurement by 31 % by the end of the experiment. These differences are likely due to slipping at the grips and compliance in the load stack (especially in the axial direction), which are persistent issues in any experiment of this nature. By utilizing the δ_e and ϕ_e histories as boundary conditions in the simulations, the discrepancy between the actuators and the virtual extensometers should not affect the comparison between the experiments and the simulations.

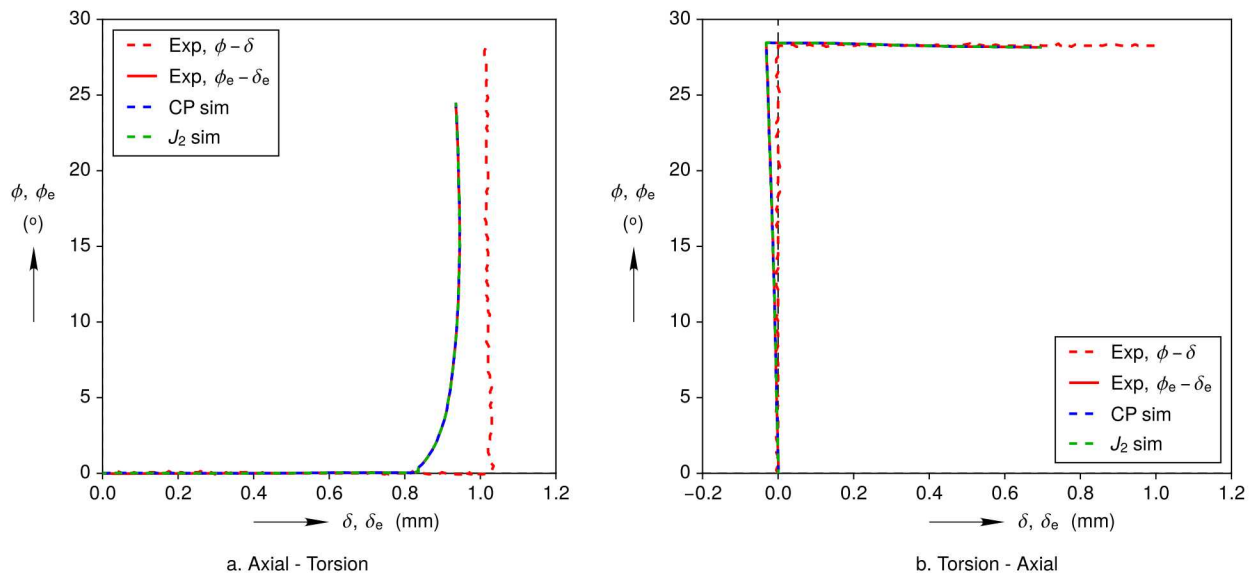


Figure 115. Actuator and extensometer displacement paths

Figure 116 and

Figure 117 show the comparison for the Axial-Torsion and Torsion-Axial experiments, respectively. The axial force P is normalized by the cross sectional area A of the thin wall portion of the tube to become an average engineering axial stress P/A . The torque M is normalized by C and the polar moment of inertia of the thin wall portion of the tube J to become an average engineering shear stress MC/J .

Note that the stresses and strains are not uniform along the length L_s in either the experiments or the simulations, due to the thick wall shoulders neighboring the thin wall portion. For example, an axial displacement δ_e causes a tendency for the thin wall portion to laterally contract in the hoop direction. The thick wall sections of the tube, however, restrain this lateral contraction and cause a hoop stress to develop, especially near the fillets.

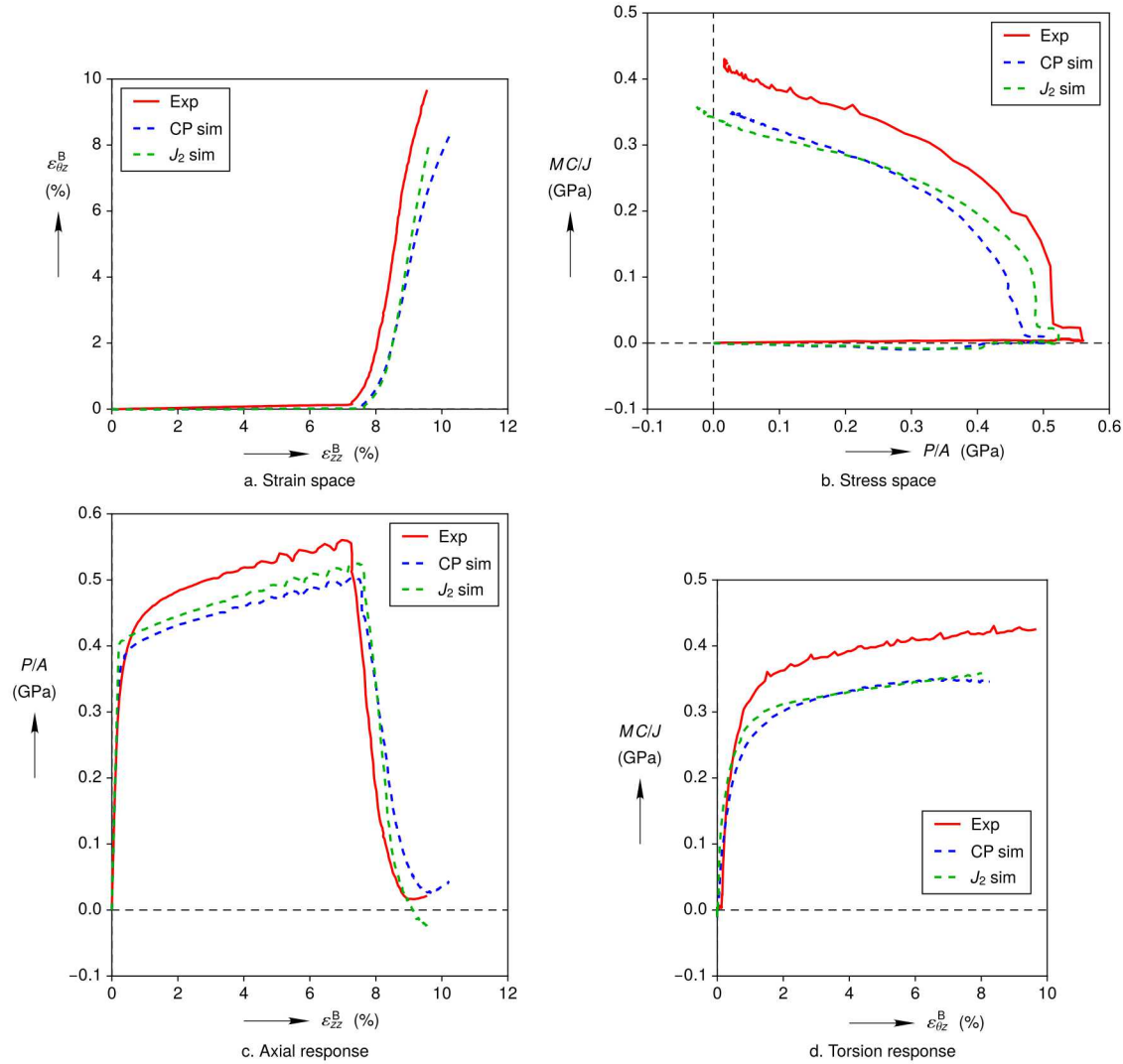


Figure 116. Axial-Torsion results

Both models under-predict the onset of yielding during the Axial-Torsion experiment (see Figure 116c). This may be because the hoop stresses previously alluded to cause the material to hit a different point on the yield surface. If so, one would expect the crystal plasticity model to perform better since it supposedly captures the yield surface shape more accurately, but the J_2 flow model

actually performs slightly better here. The discrepancy is more likely due to specimen-to-specimen variation. Both models were calibrated against a tension test on a specimen extracted from a different 3.8 kW X-bar.

Upon twisting, the axial stress drops in the Axial-Torsion experiment and the shear stress increases rapidly. Both models under-predict the torque, but that is to be expected given that they under-predicted the axial load. In stress space (Figure 116b), the stresses curve around counter clockwise. The J_2 flow model seems to predict the initial shape of the counter clockwise sweep slightly better than the crystal plasticity model. Near the end of the stress path, however, the crystal plasticity model predicts the stress path slope better.

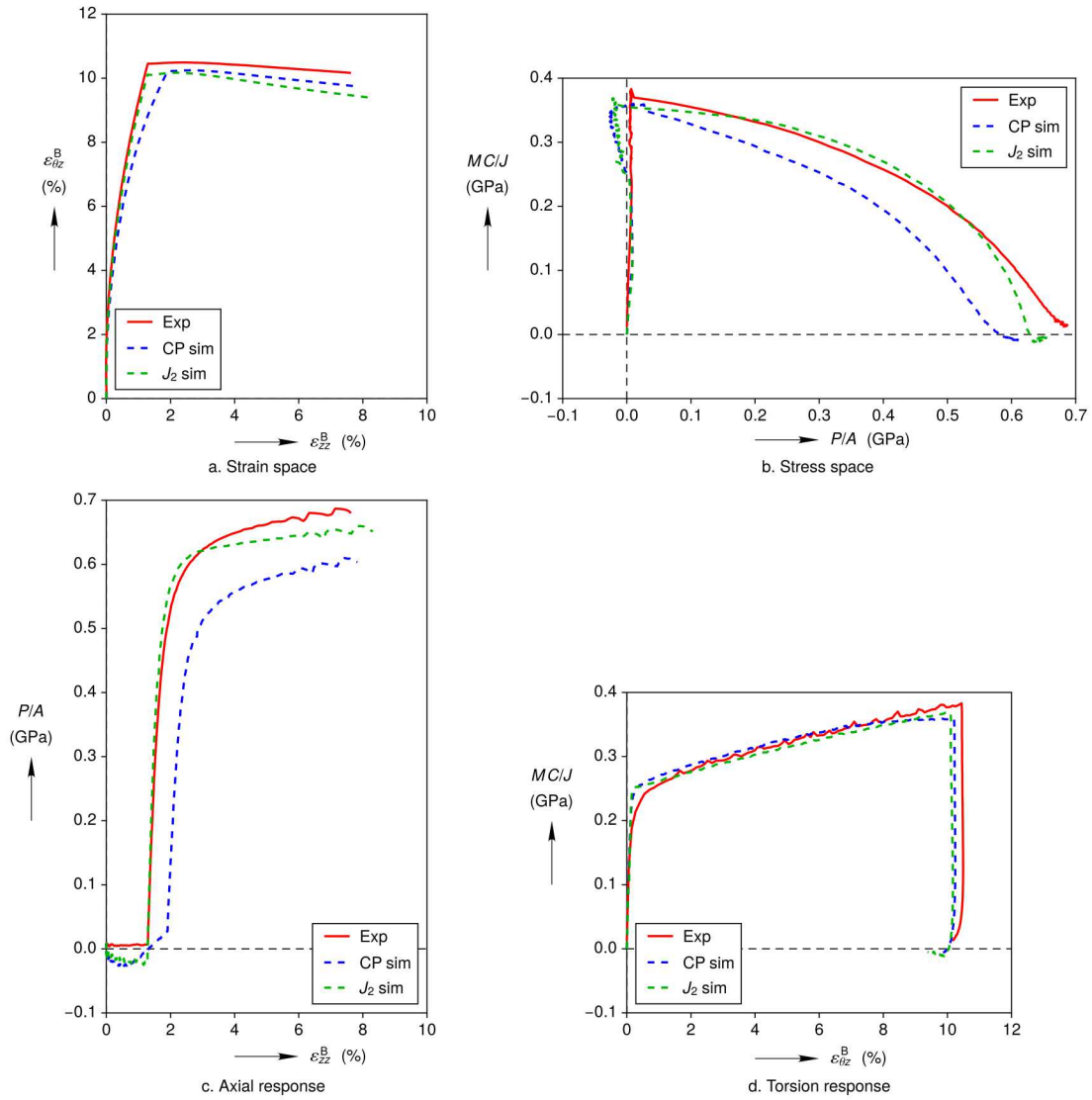


Figure 117. Torsion-Axial results

In the Torsion-Axial experiment, both models start by predicting the torsion response in Figure 117d. Once axial loading begins, the stress swings clockwise in stress space (Figure 117b). Similar to the Axial-Torsion experiment, the J_2 flow model predicts the initial shape of the stress path better than the crystal plasticity model. Yet, near the end of the stress path, the crystal plasticity model predicts the stress path slope better.

Before closing this section, it should be noted that the tube exhibited small torsional both crystal plasticity simulations. The shear stress has a local maximum at $\varepsilon_{\theta z}^B \approx 6.2\%$ 116d, and at $\varepsilon_{\theta z}^B \approx 9.7\%$ in

Figure 117d. Both of these local maxima correspond to helical lobes forming in the thin wall portion of the tube, similar to what occurs when one twists a thin aluminum can. The J_2 flow model did not predict such buckling either in the scoping simulations before the experiments, or in the J_2 flow model simulations presented here. Therefore, the buckling was likely brought on by the anisotropy in the crystal plasticity model.

11.2.4. Conclusions

In Section 11.2.1, the crystal plasticity modeling approach was partially validated by comparing against lattice strains measured by neutron diffraction. The simulations captured the relative magnitudes of the lattice strains, engendering confidence in the model formulation. Further work to achieve mesh convergence may improve the match between the simulations and measurements.

Section 11.2.2 demonstrated that the model predicts a significant degree of anisotropy. Simulated compression tests along seven different directions resulted in a 20 % variation in uniaxial elastic modulus, an 8 % variation in yield strength, and a 144 % variation in lateral strain slopes.

Section 11.2.3 compared crystal plasticity simulations and J_2 flow model simulations against non-proportional axial-torsion experiments on tubes. On the whole, it was difficult to say which model more accurately captured the experiments. The J_2 flow model performed better at the onset of non-proportional loading, while the crystal plasticity model performed better towards the end.

Perhaps the crystal plasticity model would out-perform the J_2 flow model if larger deformations were applied.

12. CONCLUSIONS

This LDRD investigated the mechanical response of additively manufactured stainless steel 304L produced by several high power, laser-based DED processes. Experiments covered a broad range of loading conditions covering 10 decades of strain rate. Direct comparisons with wrought stainless steel 304L demonstrate a greater strength and reduced ductility in the additively manufactured material. Increased strength is found in AM stainless steel for all strain rates utilized for testing, in compression and tension.

A detailed investigation of microstructure, phase, and composition revealed differences between AM and wrought that may contribute to the differing mechanical behaviors. Compared with the baseline wrought material, dense AM stainless steel 304L has a more complex grain structure with substantial sub-structure, a fine dispersion of ferrite, increased dislocation density, and oxide dispersions. Several potential strengthening mechanisms have emerged from characterization experiments. *In-situ* neutron diffraction studies conducted during quasi-static loading show strong evidence for dislocation density being responsible for the increased strength of AM material. The AM stainless steel has a larger dislocation density prior to mechanical testing. Furthermore, the flow strength of both forms has been correlated with dislocation density through a square root dependence (akin to a Taylor-like relationship) thereby demonstrating the likely role of defect structure on yield stress. In addition to defect structure, solid solution strengthening may play a role. Additively manufactured stainless steel had nearly double the concentration of nitrogen than that of wrought as determined by Leco. Based on published literature, a measured concentration of 0.089 wt.% N (versus 0.04 wt.% in wrought) is considered to be significant in that there are potential ramifications in the form of solute drag and its effects on response. The elevated nitrogen concentration is likely developed as a result of using nitrogen – atomized powder for AM processing.

Crystal plasticity simulations were also used to study how the AM material's exotic microstructure affects its mechanical behavior. Potts model simulations with a moving heat source produced grain morphologies that agreed well with experimental observations, but

attempts to convert the grain structures into finite element meshes were unsuccessful. Instead, crystal plasticity simulations used equiaxed Voronoi grains imprinted with the experimentally measured crystallographic texture. This texture caused the crystal plasticity model to predict a significant degree of elastic and plastic anisotropy that could affect the performance of additively manufactured components. The modeling approach was validated in two ways. First, simulations of a tensile test correctly predicted the relative magnitudes of lattice strains before, during, and after yield, as compared with *in-situ* neutron diffraction measurements. Second, axial-torsion experiments on the AM material were simulated using the crystal plasticity model and a macroscopic plasticity model. The macroscopic model matched the experiments better early in the deformation, while the crystal plasticity model appeared to perform better towards the end of the deformation.

Experiments completed in this LDRD have, for the first time, explored the high strain response of an additively manufactured material. Hopkinson tests at $\dot{\epsilon} = 500$ and 2500 s^{-1} also show a greater strength for the AM stainless steel 304L. Although, the differences compared with wrought are reduced with increasing strain rate. Gas gun impact tests, including reverse ballistic, forward ballistic and spall tests, further demonstrated a larger strength in the AM material. The Hugoniot Elastic Limit (HEL) of AM SS 304L exceeds that of wrought material. Forward ballistic testing demonstrates spall strengths of AM material (3.27 – 3.91 GPa) that exceed that of the wrought material (2.63 – 2.88 GPa). The Hugoniot equation-of-state for AM samples matches archived data for this metal alloy. The dynamic mechanical response of AM stainless steel 304L varied considerably depending on sample orientation.

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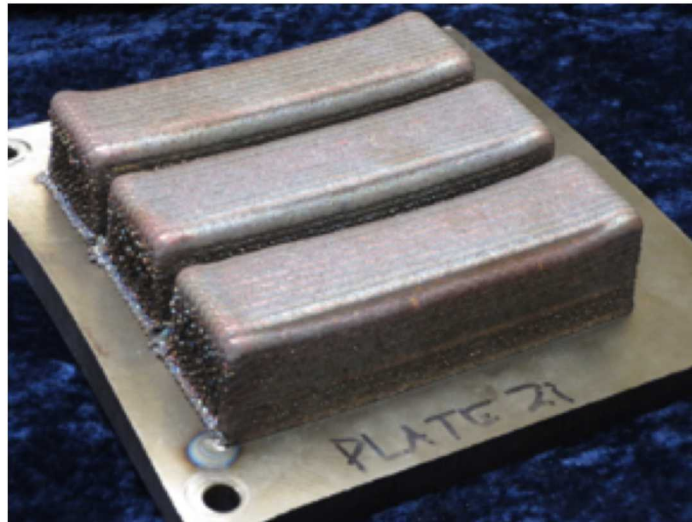
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14. APPENDICES

14.1. Photographs of various rectangular bars fabricated for study.

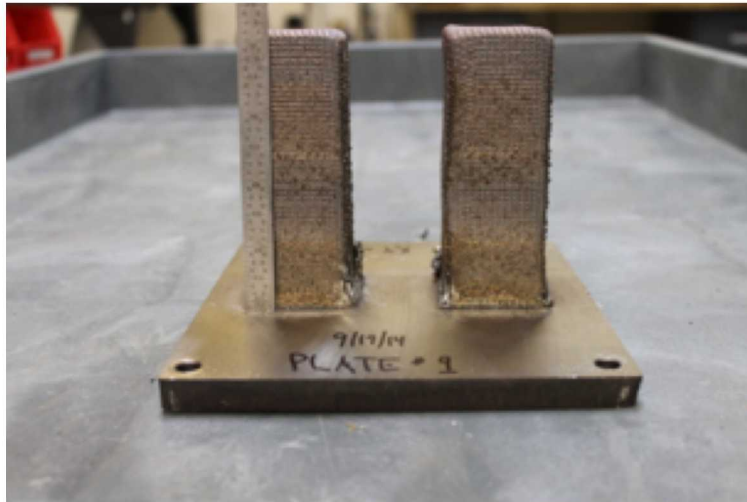
The bars shown below were machined to make compression and/or tension test samples for quasi-static and/or Hopkinson tests. For reference the base plates are 15.25 x 15.25 x 1.0 cm.



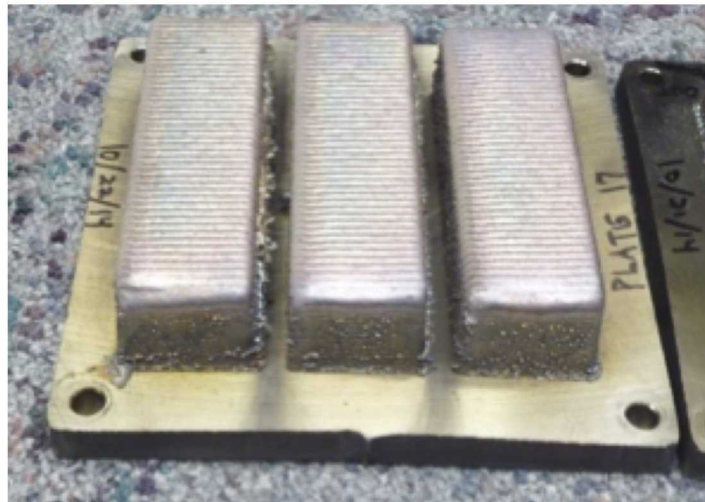
Photograph showing three X bars that are approx. 11 cm long made using a 2.0 kW cross hatched process.



Photograph showing three X bars made using a 3.8 kW parallel line process.



Photograph showing two Z bars made using a 2.0 kW cross hatched process.



Photograph showing three Y bars made using a 2.0 kW cross hatched process.

14.2. Photograph of square bar fabricated for study.

The bars shown below were machined to make circular test samples for gas gun impact testing.



Plate 19. For reference the base plates are 15.25 x 15.25 x 1.0 cm.

14.3. Summary of Builds by Penn State University

Plate ID	Bar types	Nominal Laser Power (kW)	Travel speed (ipm)	Powder flow rate (g/min)	Spot Size (mm)	Thickness/ Layer (inches)
1	X, Z	3.8	25	23	~4	0.050
2	X, X	3.8	25	23	~4	0.050
5	X	3.8	25	23	~4	0.050
6	X, X	3.8	25	23	~4	0.050
7	X, X	3.8	25	23	~4	0.050
8	X, X	3.8	25	23	~4	0.050
9	Z, Z	2	20	18	~4	0.035
10	Z, Z	2	20	18	~4	0.035
11	Z, Z	2	20	18	~4	0.035
12	Z, Z	2	20	18	~4	0.035
14	Z, Z	2	20	18	~4	0.035
15	Z, Z	2	20	18	~4	0.035
16	Y, Y, Y*	2	20	18	~4	0.035
17	Y, Y, Y*	2	20	18	~4	0.035
18	Y, Y, Y*	2	20	18	~4	0.035
19	2.5"	3.8	25	23	~4	0.050
20	2.5"	3.8	25	23	~4	0.050
21	X, X, X	2	20	18	~4	0.035

* The Y bar nomenclature used here is equivalent to X when using a 2.0 kW process

14.4. Results from ultrasound tests of AM SS 304L fabricated with $P_{avg} = 3.8$ kW.

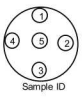
The following two pages include results from sound testing of AM material. Results include determined longitudinal velocity, shear velocity, Young's modulus, shear and bulk modulus.

Sample ID#	Density (kg/m ³)	2x Thickness (mm)	Position	Longitudinal ToF (μsec)	Longitudinal Velocity (mm/μsec)	Shear ToF (μsec)	Shear Velocity (mm/μsec)	Poisson Ratio (ν)	Young's Modulus (GPa)	Shear Modulus (GPa)	Bulk Modulus (GPa)
L1, Shear Parallel to ID Stamp 	7833.30	6.241	1	1.0440	5.9776	2.3350	2.6726	0.375	153.879	55.953	205.292
			2	1.0320	6.0471	2.3410	2.6658	0.379	153.572	55.667	212.221
			3	1.0285	6.0677	2.1800	2.8627	0.357	174.198	64.193	202.806
			4	1.0465	5.9633	2.3415	2.6652	0.375	153.039	55.643	204.370
			5	1.0400	6.0006	2.3475	2.6584	0.378	152.558	55.359	208.241
			Average	1.0382	6.0112	2.3090	2.7049	0.373	157.449	57.363	206.586
L2, Shear Parallel to ID Stamp	7828.10	6.268	1	1.0340	6.0623	1.8630	3.3647	0.277	226.414	88.623	169.529
			2	1.0250	6.1155	1.8655	3.3602	0.284	226.933	88.385	174.920
			3	1.0285	6.0947	1.8500	3.3883	0.276	229.414	89.872	170.948
			4	1.0310	6.0799	1.8645	3.3620	0.280	226.470	88.480	171.396
			5	1.0270	6.1036	1.8705	3.3512	0.284	225.801	87.913	174.410
			Average	1.0291	6.0912	1.8627	3.3653	0.280	227.006	88.655	172.241
L3, Shear Parallel to ID Stamp	7822.00	6.188	1	1.0040	6.1633	1.8955	3.2646	0.305	217.579	83.363	185.983
			2	1.0045	6.1603	1.8795	3.2924	0.300	220.461	84.788	183.787
			3	1.0065	6.1480	1.9055	3.2474	0.307	215.549	82.490	185.672
			4	1.0115	6.1176	1.8965	3.2629	0.301	216.718	83.275	181.710
			5	1.0095	6.1298	1.9035	3.2509	0.304	215.642	82.663	183.686
			Average	1.0072	6.1438	1.8961	3.2636	0.303	217.190	83.316	184.168
Z1, Disk 7, Shear Parallel to scan line	7824.20	3.056	1	0.5070	6.0276	0.9490	3.2202	0.300	211.001	81.136	176.088
			2	0.5055	6.0455	0.9590	3.1867	0.308	207.789	79.453	180.022
			3	0.5065	6.0336	0.9550	3.2000	0.304	209.002	80.120	178.005
			4	0.5065	6.0336	0.9530	3.2067	0.303	209.696	80.456	177.556
			5	0.5070	6.0276	0.9490	3.2202	0.300	211.001	81.136	176.088
			Average	0.5065	6.0336	0.9530	3.2068	0.303	209.698	80.460	177.552
Z2, Disk 8, Shear Parallel to scan line	7816.30	3.063	1	0.5035	6.0838	1.0105	3.0314	0.335	191.755	71.826	193.536
			2	0.5055	6.0597	1.0360	2.9568	0.344	183.648	68.333	195.907
			3	0.5050	6.0657	1.0210	3.0002	0.338	188.281	70.356	193.779
			4	0.5060	6.0538	1.0115	3.0284	0.333	191.125	71.684	190.873
			5	0.5035	6.0838	1.0180	3.0090	0.338	189.394	70.771	194.941
			Average	0.5047	6.0694	1.0194	3.0051	0.338	188.841	70.594	193.807
Z3, Disk 9, Shear Parallel to scan line	7843.40	3.051	1	0.4980	6.1261	0.9755	3.1274	0.324	203.103	76.714	192.070
			2	0.4995	6.1077	0.9995	3.0523	0.334	194.898	73.075	195.158
			3	0.4995	6.1077	0.9635	3.1664	0.316	207.009	78.637	187.741
			4	0.4985	6.1200	0.9720	3.1387	0.322	204.227	77.268	190.742
			5	0.4985	6.1200	0.9955	3.0646	0.333	196.336	73.663	195.549
			Average	0.4988	6.1163	0.9812	3.1099	0.326	201.115	75.871	192.252
Z1, Disk 4, Shear Parallel to scan line	7842.80	4.117	1	0.7020	5.8652	1.3290	3.0981	0.307	196.702	75.278	169.430
			2	0.7015	5.8694	1.3285	3.0993	0.307	196.877	75.335	169.739
			3	0.7015	5.8694	1.3200	3.1192	0.303	198.890	76.308	168.442
			4	0.7010	5.8736	1.3250	3.1075	0.306	197.762	75.733	169.594
			5	0.7015	5.8694	1.3230	3.1122	0.304	198.177	75.962	168.902
			Average	0.7015	5.8694	1.3251	3.1055	0.3055	197.4897	75.6401	169.2037
Z2, Disk 5, Shear Parallel to scan line	7850.40	4.066	1	0.6945	5.8540	1.3240	3.0707	0.310	193.970	74.023	170.330
			2	0.6925	5.8709	1.3280	3.0614	0.313	193.253	73.577	172.480
			3	0.6935	5.8624	1.3250	3.0684	0.311	193.846	73.911	171.256
			4	0.6935	5.8624	1.3380	3.0386	0.316	190.821	72.482	173.161
			5	0.6940	5.8582	1.3355	3.0443	0.315	191.346	72.753	172.411
			Average	0.6936	5.8616	1.3301	3.0567	0.313	192.647	73.349	171.928
Z3, Disk 6, Shear Parallel to scan line	7833.20	4.074	1	0.6945	5.8667	1.3165	3.0949	0.307	196.154	75.028	169.564
			2	0.6920	5.8879	1.3250	3.0750	0.312	194.426	74.069	172.795
			3	0.6940	5.8709	1.3270	3.0704	0.312	193.735	73.846	171.529
			4	0.6925	5.8836	1.3280	3.0681	0.313	193.665	73.734	172.848
			5	0.6950	5.8624	1.3245	3.0762	0.310	194.210	74.125	170.381
			Average	0.6936	5.8743	1.3242	3.0769	0.311	194.438	74.160	171.423

L1, Shear 90d ID Stamp	7833.30	6.241	1	1.0440	5.9776	1.8785	3.3221	0.277	220.720	86.452	164.627
			2	1.0320	6.0471	1.9500	3.2003	0.305	209.472	80.228	179.472
			3	1.0285	6.0677	1.9615	3.1815	0.310	207.805	79.290	182.675
			4	1.0465	5.9633	1.9570	3.1889	0.300	207.067	79.656	172.353
			5	1.0400	6.0006	1.9000	3.2845	0.286	217.369	84.450	169.378
			Average	1.0382	6.0112	1.9294	3.2355	0.296	212.487	82.015	173.701
L2, Shear 90d ID Stamp	7828.10	6.268	1	1.0340	6.0623	2.0380	3.0758	0.327	196.497	74.056	188.951
			2	1.0250	6.1155	2.0435	3.0675	0.332	196.213	73.658	194.556
			3	1.0285	6.0947	1.9745	3.1747	0.314	207.311	78.896	185.583
			4	1.0310	6.0799	2.0105	3.1178	0.322	201.137	76.096	187.908
			5	1.0270	6.1036	2.0295	3.0886	0.328	198.328	74.620	192.057
			Average	1.0291	6.0912	2.0192	3.1049	0.324	199.897	75.465	189.811
L3, Shear 90d ID Stamp	7822.00	6.188	1	1.0040	6.1633	2.2530	2.7466	0.376	162.397	59.006	218.458
			2	1.0045	6.1603	2.2365	2.7668	0.374	164.508	59.880	216.998
			3	1.0065	6.1480	2.2435	2.7582	0.374	163.525	59.507	216.316
			4	1.0115	6.1176	2.2520	2.7478	0.374	162.250	59.058	213.999
			5	1.0095	6.1298	2.2480	2.7527	0.374	162.835	59.269	214.879
			Average	1.0072	6.1438	2.2466	2.7544	0.374	163.103	59.344	216.130
Z1, Disk 7, Shear 90deg to screen line	7824.20	3.056	1	0.5070	6.0276	0.9775	3.1263	0.316	201.277	76.474	182.305
			2	0.5055	6.0455	0.9875	3.0947	0.322	198.191	74.933	186.049
			3	0.5065	6.0336	0.9870	3.0963	0.321	198.212	75.009	184.820
			4	0.5065	6.0336	0.9795	3.1200	0.318	200.687	76.162	183.282
			5	0.5070	6.0276	0.9705	3.1489	0.312	203.623	77.581	180.828
			Average	0.5065	6.0336	0.9804	3.1172	0.318	200.398	76.032	183.457
Z2, Disk 8, Shear 90deg to screen line	7816.30	3.063	1	0.5035	6.0838	0.9795	3.1273	0.320	201.879	76.444	187.378
			2	0.5055	6.0597	0.9790	3.1289	0.318	201.748	76.522	184.989
			3	0.5050	6.0657	0.9720	3.1514	0.315	204.183	77.628	184.083
			4	0.5080	6.0538	0.9730	3.1482	0.315	203.689	77.469	183.160
			5	0.5035	6.0838	0.9750	3.1417	0.318	203.397	77.151	186.435
			Average	0.5047	6.0694	0.9757	3.1395	0.317	202.979	77.043	185.209
Z3, Disk 9, Shear 90deg to screen line	7843.40	3.051	1	0.4980	6.1261	0.9410	3.2421	0.305	215.255	82.443	184.432
			2	0.4995	6.1077	0.9670	3.1549	0.318	205.796	78.069	188.499
			3	0.4995	6.1077	0.9605	3.1763	0.315	208.055	79.129	187.085
			4	0.4985	6.1200	0.9560	3.1912	0.313	209.799	79.876	187.265
			5	0.4985	6.1200	0.9525	3.2029	0.311	211.039	80.464	186.481
			Average	0.4988	6.1163	0.9554	3.1935	0.313	209.989	79.996	186.752
Z1, Disk 4, Shear 90deg to screen line	7842.80	4.117	1	0.7020	5.8652	1.2765	3.2255	0.283	209.415	81.597	161.004
			2	0.7015	5.8694	1.2785	3.2205	0.285	208.989	81.342	161.729
			3	0.7015	5.8694	1.2855	3.2030	0.288	207.255	80.459	162.907
			4	0.7010	5.8736	1.2735	3.2331	0.283	210.308	81.982	161.261
			5	0.7015	5.8694	1.2755	3.2281	0.283	209.736	81.725	161.218
			Average	0.7015	5.8694	1.2779	3.2220	0.284	209.141	81.421	161.624
Z2, Disk 5, Shear 90deg to screen line	7850.40	4.066	1	0.6945	5.8540	1.2820	3.1713	0.292	204.062	78.952	163.758
			2	0.6925	5.8709	1.2770	3.1837	0.292	205.567	79.572	164.488
			3	0.6935	5.8624	1.2825	3.1701	0.293	204.072	78.891	164.616
			4	0.6935	5.8624	1.2735	3.1925	0.289	206.300	80.010	163.124
			5	0.6940	5.8582	1.2985	3.1310	0.300	200.102	76.959	166.804
			Average	0.6936	5.8616	1.2827	3.1697	0.293	204.021	78.877	164.558
Z3, Disk 6, Shear 90deg to screen line	7833.20	4.074	1	0.6945	5.8667	1.2855	3.1695	0.294	203.637	78.691	164.681
			2	0.6920	5.8879	1.2770	3.1906	0.292	206.074	79.742	165.231
			3	0.6940	5.8709	1.2665	3.2171	0.285	208.419	81.069	161.898
			4	0.6925	5.8836	1.2635	3.2247	0.285	209.390	81.455	162.555
			5	0.6950	5.8624	1.2780	3.1881	0.290	205.417	79.617	163.058
			Average	0.6936	5.8743	1.2741	3.1980	0.289	206.587	80.115	163.484

14.5. Results from ultrasound tests of wrought SS 304L used for mechanical testing.

The following page includes results from sound testing of wrought SS 304L. Results include determined longitudinal velocity, shear velocity, Young's modulus, shear and bulk modulus.

Sample ID#	Density (kg/m ³)	2x Thickness (mm)	Position	Longitudinal ToF (μsec)	Longitudinal Velocity (mm/μsec)	Shear ToF (μsec)	Shear Velocity (mm/μsec)	Poisson Ratio (ν)	Young's Modulus (GPa)	Shear Modulus (GPa)	Bulk Modulus (GPa)
W1, 24x2  Sample ID#	7882.40	3.980	1	0.6825	5.8321	1.2655	3.1453	0.295	201.958	77.981	164.132
			2	0.6820	5.8364	1.2670	3.1416	0.296	201.652	77.796	164.771
			3	0.6820	5.8364	1.2670	3.1416	0.296	201.652	77.796	164.771
			4	0.6820	5.8364	1.2675	3.1404	0.296	201.528	77.735	164.853
			5	0.6820	5.8364	1.2665	3.1428	0.296	201.776	77.858	164.689
			Average	0.6821	5.8355	1.2667	3.1423	0.296	201.713	77.833	164.643
W2, 24x2	7882.20	3.963	1	0.6800	5.8282	1.2600	3.1454	0.295	201.901	77.983	163.768
			2	0.6775	5.8497	1.2605	3.1441	0.297	202.107	77.921	165.830
			3	0.6770	5.8541	1.2605	3.1441	0.297	202.173	77.921	166.229
			4	0.6800	5.8282	1.2605	3.1441	0.295	201.777	77.921	163.851
			5	0.6795	5.8325	1.2610	3.1429	0.295	201.719	77.859	164.327
			Average	0.6788	5.8386	1.2605	3.1441	0.296	201.935	77.921	164.801
W3, 24x2	7877.90	3.980	1	0.6830	5.8278	1.2645	3.1478	0.294	202.024	78.060	163.481
			2	0.6805	5.8492	1.2645	3.1478	0.296	202.355	78.060	165.451
			3	0.6815	5.8406	1.2655	3.1453	0.296	201.975	77.936	164.825
			4	0.6810	5.8449	1.2665	3.1428	0.297	201.792	77.813	165.384
			5	0.6825	5.8321	1.2660	3.1441	0.295	201.719	77.875	164.120
			Average	0.6817	5.8389	1.2654	3.1456	0.296	201.973	77.949	164.652
W1, 24x1.5	7870.80	2.972	1	0.4995	5.9491	0.9430	3.1512	0.305	203.994	78.158	174.355
			2	0.4970	5.9791	0.9425	3.1529	0.307	204.588	78.241	177.054
			3	0.4965	5.9851	0.9435	3.1495	0.309	204.326	78.076	177.842
			4	0.5020	5.9195	0.9430	3.1512	0.302	203.567	78.158	171.587
			5	0.5010	5.9313	0.9435	3.1495	0.304	203.568	78.076	172.800
			Average	0.4992	5.9528	0.9431	3.1509	0.305	204.009	78.142	174.728
W2, 24x1.5	7888.20	2.972	1	0.5080	5.8504	0.9465	3.1400	0.298	201.853	77.774	166.292
			2	0.5085	5.8446	0.9465	3.1400	0.297	201.766	77.774	165.761
			3	0.5070	5.8619	0.9465	3.1400	0.299	202.027	77.774	167.358
			4	0.5080	5.8504	0.9470	3.1383	0.298	201.687	77.692	166.401
			5	0.5090	5.8389	0.9470	3.1383	0.297	201.513	77.692	165.341
			Average	0.5081	5.8493	0.9467	3.1393	0.298	201.769	77.741	166.231
W3, 24x1.5	7877.10	2.984	1	0.5095	5.8567	0.9495	3.1427	0.298	201.937	77.799	166.462
			2	0.5080	5.8740	0.9500	3.1411	0.300	202.030	77.717	168.169
			3	0.5095	5.8567	0.9500	3.1411	0.298	201.771	77.717	166.571
			4	0.5105	5.8452	0.9500	3.1411	0.297	201.598	77.717	165.514
			5	0.5015	5.9501	0.9495	3.1427	0.307	203.297	77.799	175.151
			Average	0.5078	5.8766	0.9498	3.1417	0.300	202.127	77.750	168.373
W1, 57x3	7879.20	6.015	1	1.0375	5.7978	1.9120	3.1460	0.291	201.408	77.984	160.875
			2	1.0365	5.8034	1.9105	3.1485	0.291	201.741	78.107	161.223
			3	1.0345	5.8146	1.9125	3.1452	0.293	201.592	77.943	162.468
			4	1.0365	5.8034	1.9105	3.1485	0.291	201.741	78.107	161.223
			5	1.0365	5.8034	1.9130	3.1444	0.292	201.334	77.903	161.495
			Average	1.0363	5.8045	1.9117	3.1466	0.2920	201.5806	78.0114	161.5217
W2, 57x3	7881.60	6.024	1	1.0350	5.8207	1.9145	3.1467	0.294	201.900	78.043	162.974
			2	1.0350	5.8207	1.9150	3.1459	0.294	201.818	78.002	163.028
			3	1.0305	5.8461	1.9155	3.1451	0.296	202.129	77.961	165.420
			4	1.0330	5.8319	1.9145	3.1467	0.295	202.075	78.043	164.009
			5	1.0345	5.8235	1.9150	3.1459	0.294	201.862	78.002	163.287
			Average	1.0336	5.8286	1.9149	3.1461	0.294	201.957	78.010	163.744
W3, 57x3	7883.00	6.034	1	1.0385	5.8107	1.9205	3.1421	0.293	201.320	77.827	162.393
			2	1.0375	5.8163	1.9210	3.1413	0.294	201.327	77.787	162.960
			3	1.0370	5.8191	1.9200	3.1429	0.294	201.533	77.868	163.109
			4	1.0390	5.8079	1.9190	3.1446	0.293	201.520	77.949	161.974
			5	1.0360	5.8247	1.9205	3.1421	0.295	201.538	77.827	163.679
			Average	1.0376	5.8157	1.9202	3.1426	0.294	201.448	77.851	162.823

14.6. Cumulative sample log for static and Kolsky bar samples

EDM BAR = ID of EDM Bar (Ref. Drawing location from Appendix 14.7)

Bar = PSU AM Deposit Block ID

“A” bars are 0.280 in. diameter blanks, “B” bars are 0.550 in. diameter blanks

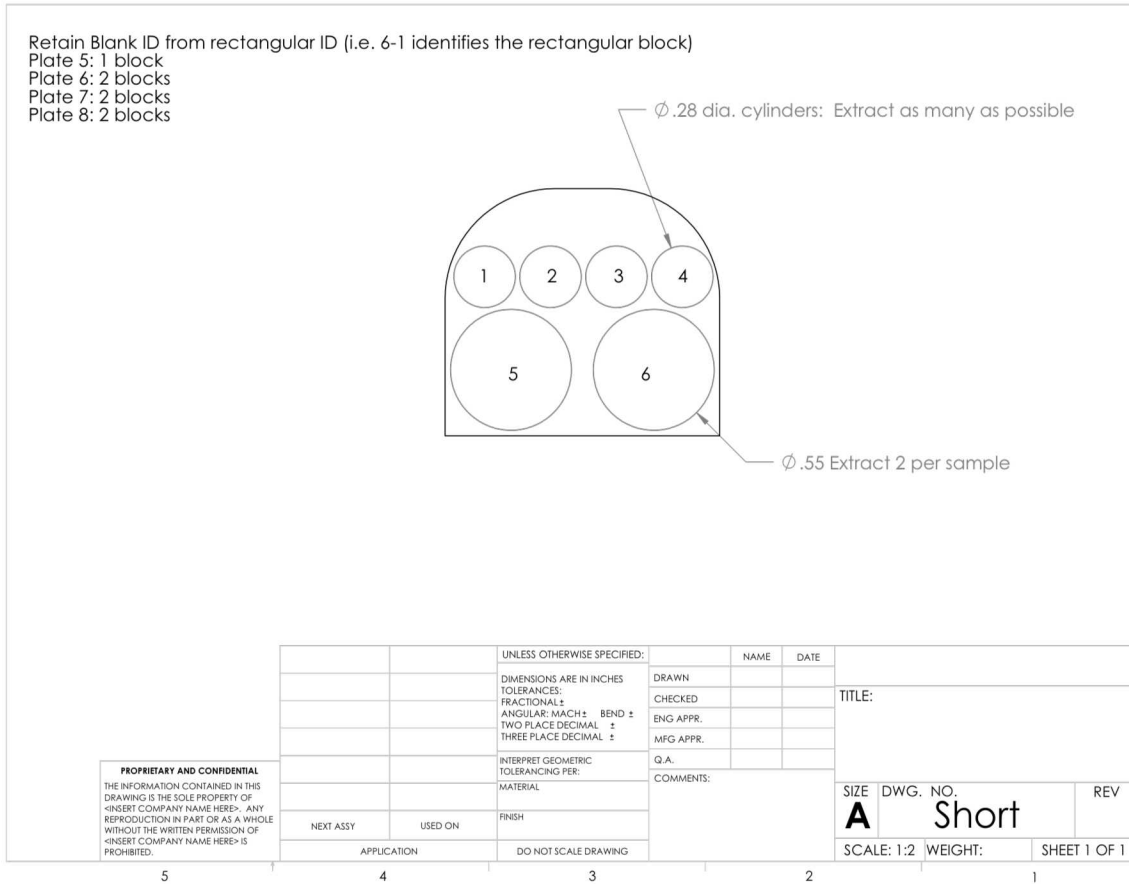
3.8 kW Parallel Hatch										
X Blocks - Scan direction along 4" dimension										
0.280 dia blanks						0.550 dia blanks				
EDM BAR	Loc.					EDM BAR	Loc.			
	Bar	Loc.			Test pcs		Bar	Loc.	Use	Test pcs
A1	5	1	Edge	RT Tensile	2	B1	5	5	N/A	Tensile 2
A3	5	2	Center	RT Tensile	2	B2	5	6	N/A	Tensile 2
A4	5	3	Center	RT Tensile	2					
A6	6-1	2	Center	RT Comp	4					
A5R	6-1	4	Edge	RT Comp	4					
A2R	8-1	4	Edge	RT Tensile	2					
A22	8-2	1	Edge	LANL	2					
Y Blocks - Scan direction perpendicular to 4" dimension										
0.280 dia blanks						0.550 dia blanks				
EDM BAR	Loc.					EDM BAR	Loc.			
	Bar	Loc.			Test pcs		Bar	Loc.	Use	Test pcs
F1	26A	3	Edge	RT Tensile	2	E1	26A	1	N/A	Tensile 2
F3	26A	4	Center	RT Tensile	2	E2	26A	2	N/A	Tensile 2
F4	26A	5	Center	RT Tensile	2	E3	26B	1	N/A	Tensile 2
F5	26B	3	Edge	RT Comp	4					
F6	26B	6	Center	RT Comp	4					
F13	26C	3	Edge	Comp	15					
F2R	28B	3	Edge	RT Tensile	2					
Z Blocks										
0.280 dia blanks						0.550 dia blanks				
EDM BAR	Loc.					EDM BAR	Loc.			
	Bar	Loc.			Test pcs		Bar	Loc.	Use	Test pcs
F7	24A	1	Edge	RT Tensile	2	E4	23	1	N/A	Tensile 2
F8	24A	2	Edge	RT Tensile	2	E5	23	2	N/A	Tensile 2
F9	24A	3	Edge	RT Tensile	2	E6	23	3	N/A	Tensile 2
F10	24A	4	Edge	RT Tensile	2					
F11	24A	6	Edge	RT Comp	4					
F12	24A	7	Edge	RT Comp	4					
F14R	28A	6	Edge	Comp	15					

2.0 kW Cross Hatch					
X Blocks					
0.280 dia blanks					
EDM BAR	Bar	Loc.			Test pcs
A23	22-3	8	Edge	LANL	2

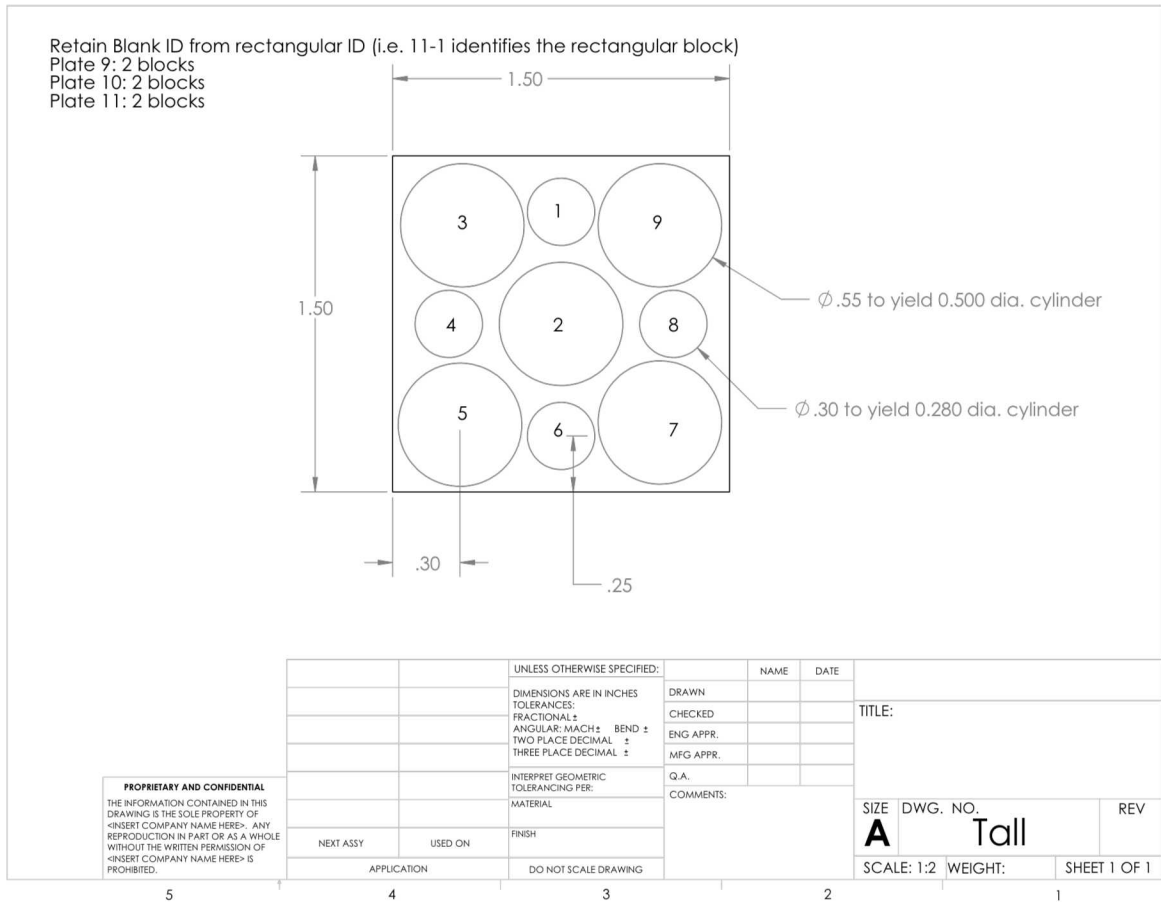
2.0 kW Cross Hatch										
Z Blocks										
0.280 dia blanks						0.550 dia blanks				
EDM BAR	Bar	Loc.			Test pcs	EDM BAR	Bar	Loc.	Use	Test pcs
A7	9-1	1	Edge	RT Tensile	2	B3	9-2	2	Center	Tensile
A8	9-1	4	Edge	RT Tensile	2	B4	10-2	2	Center	Tensile
A9	9-1	6	Edge	RT Comp	4	B5	11-1	2	Center	Comp
						B6	9-1	3	Edge	Tensile
						B7	9-1	5	Edge	Tensile
						B8	9-1	9	Edge	Comp

14.7. Drawings showing location of extracted cylinders removed from AM SS304L blocks.

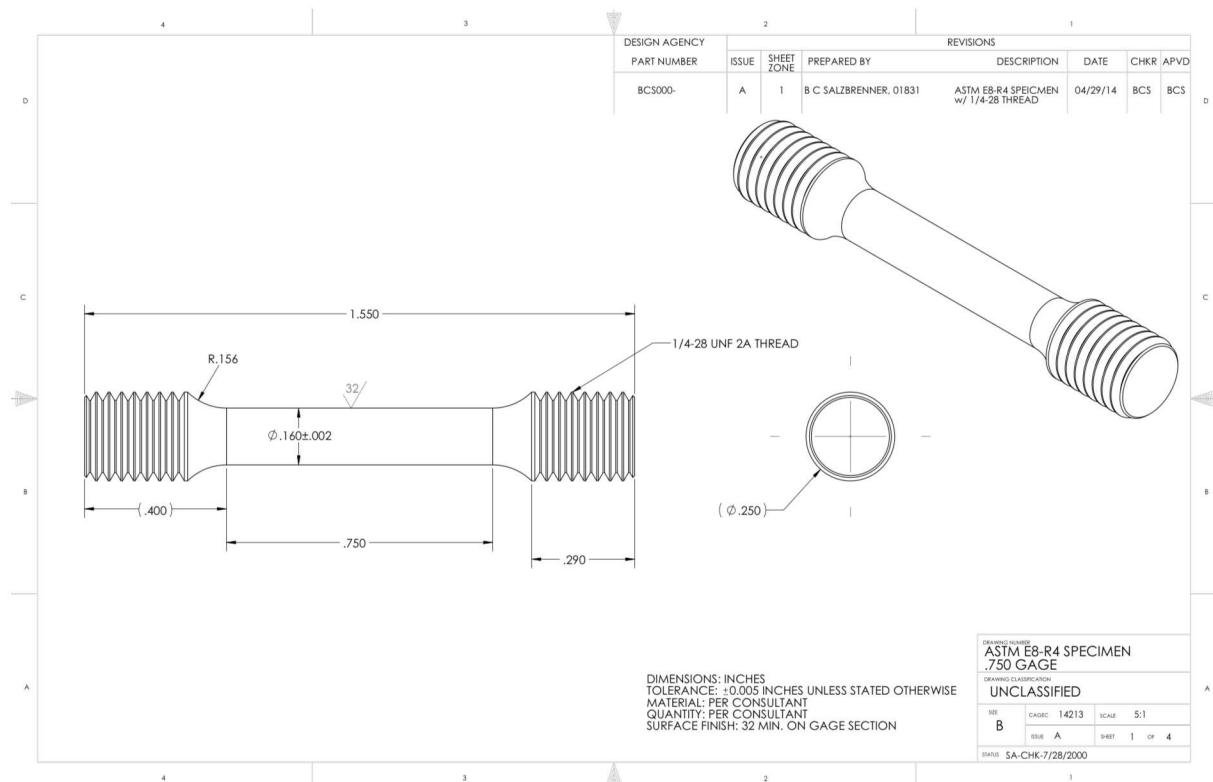
Drawing showing the location of different cylinders extracted from X and Y bars. Cylinders were used to make static and Kolsky bar test samples.

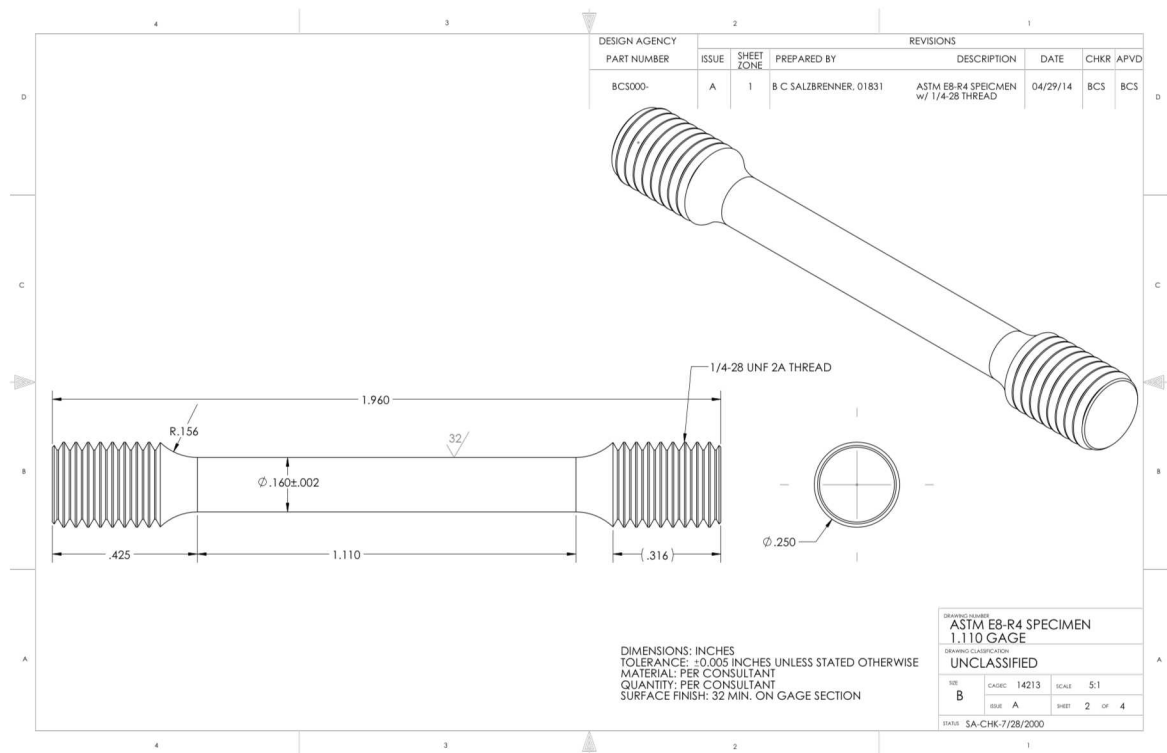


Drawing shows the location of different cylinders extracted from Z bars.

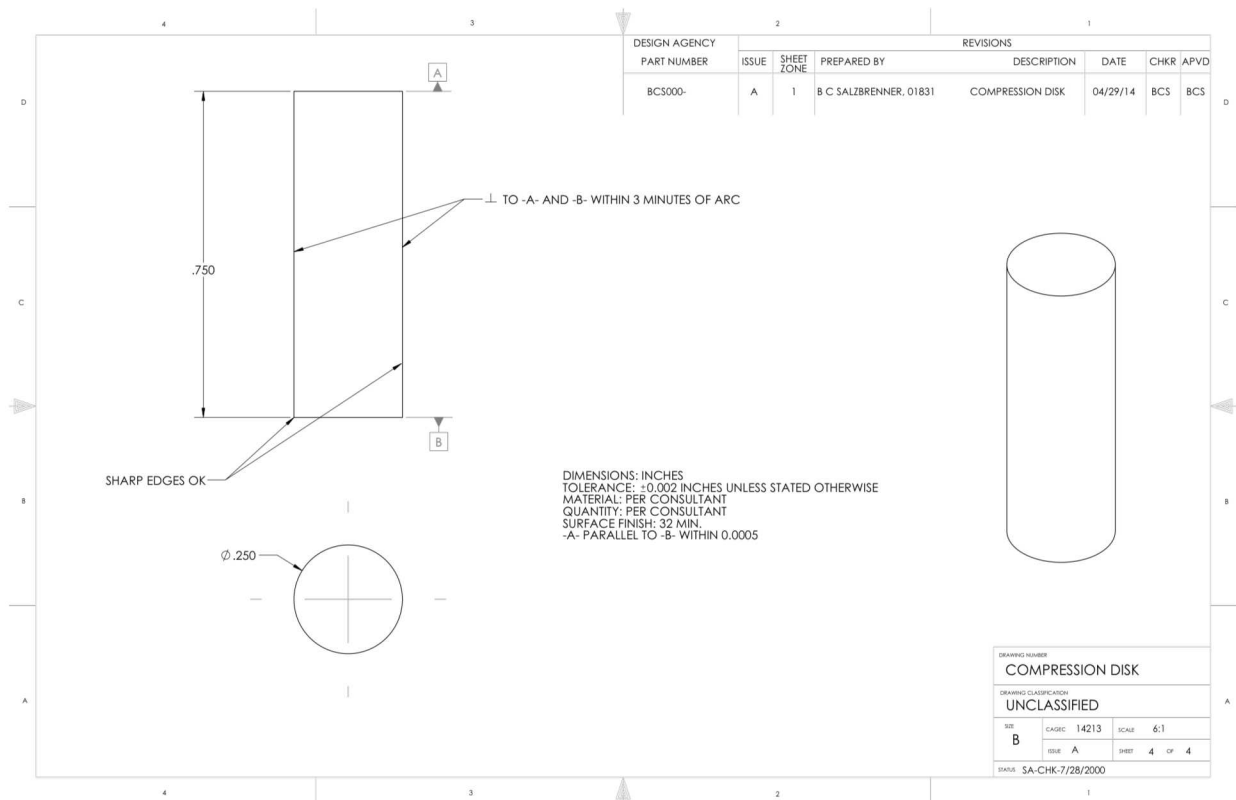


14.8. Machine drawings of tensile samples used for quasi-static testing



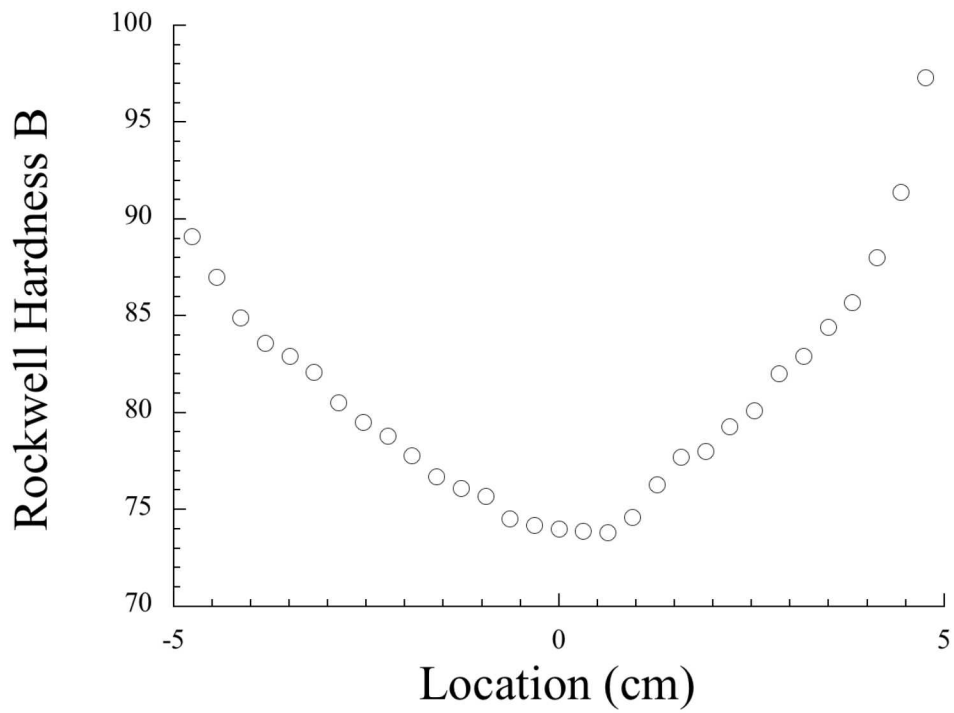


14.9. Machine drawing of compression specimen used for quasi-static testing



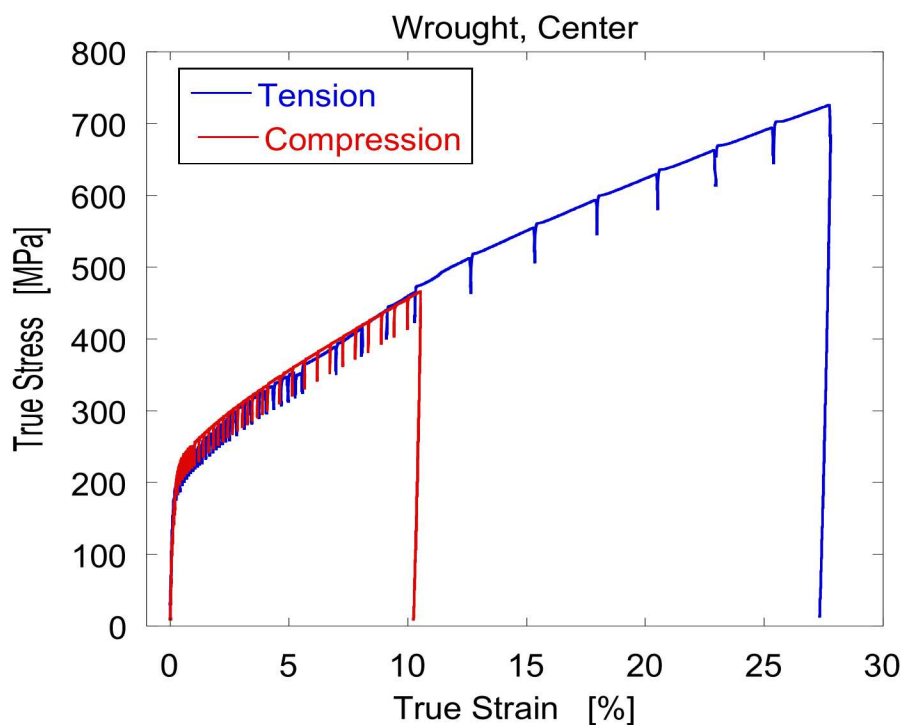
14.10. Hardness of wrought SS 304L

The following plot is a hardness trace from the wrought stainless steel billet. Hardness is plotted as a function of radial position with 0 being at center of cylinder.



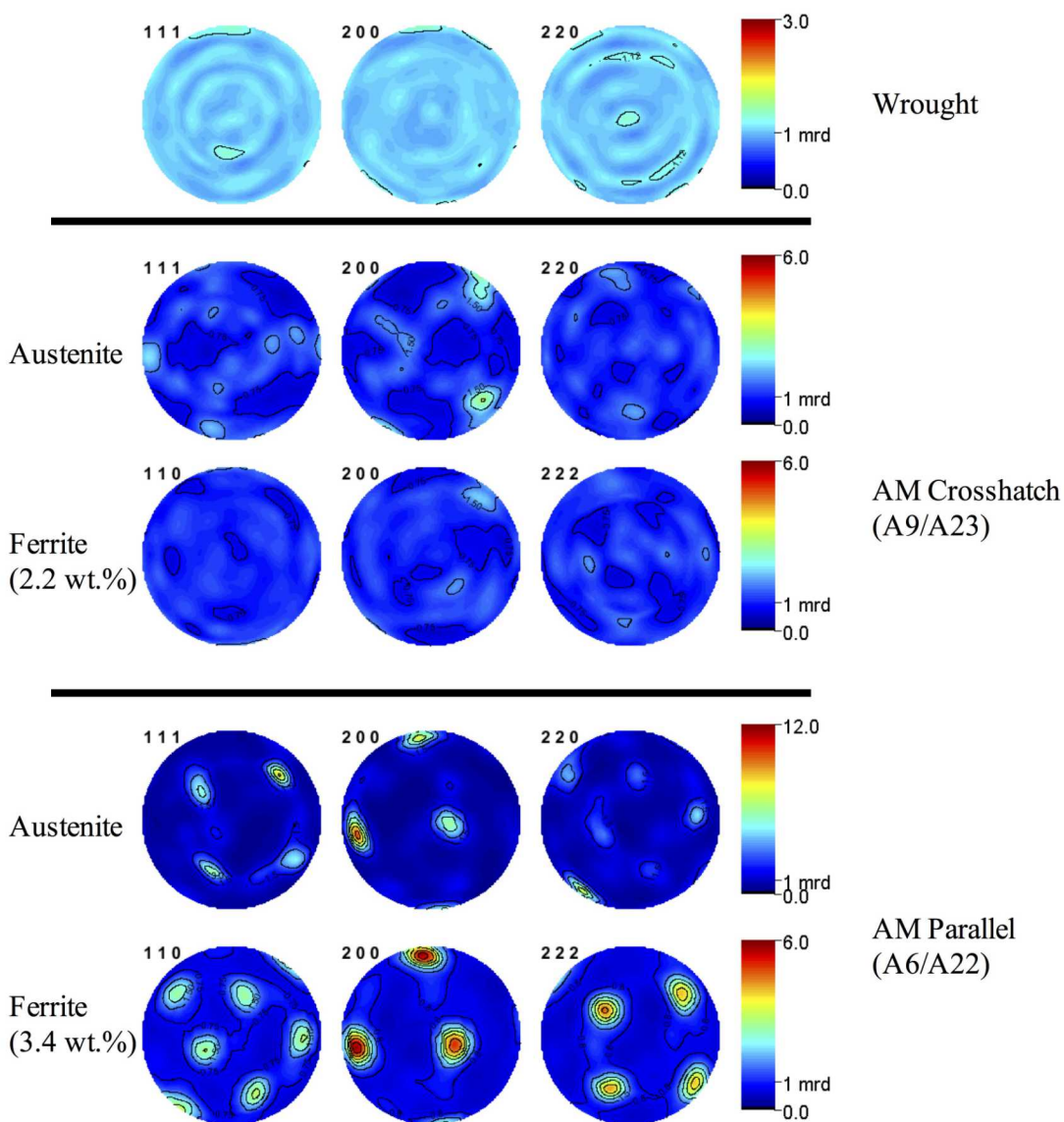
14.11. Results from quasi-static testing of wrought, center samples at LANSCE

The following true stress vs. true strain data was obtained from different wrought (center) samples. One was tested in compression and the other was in tension. Samples were probed using in-situ neutron diffraction during mechanical testing.

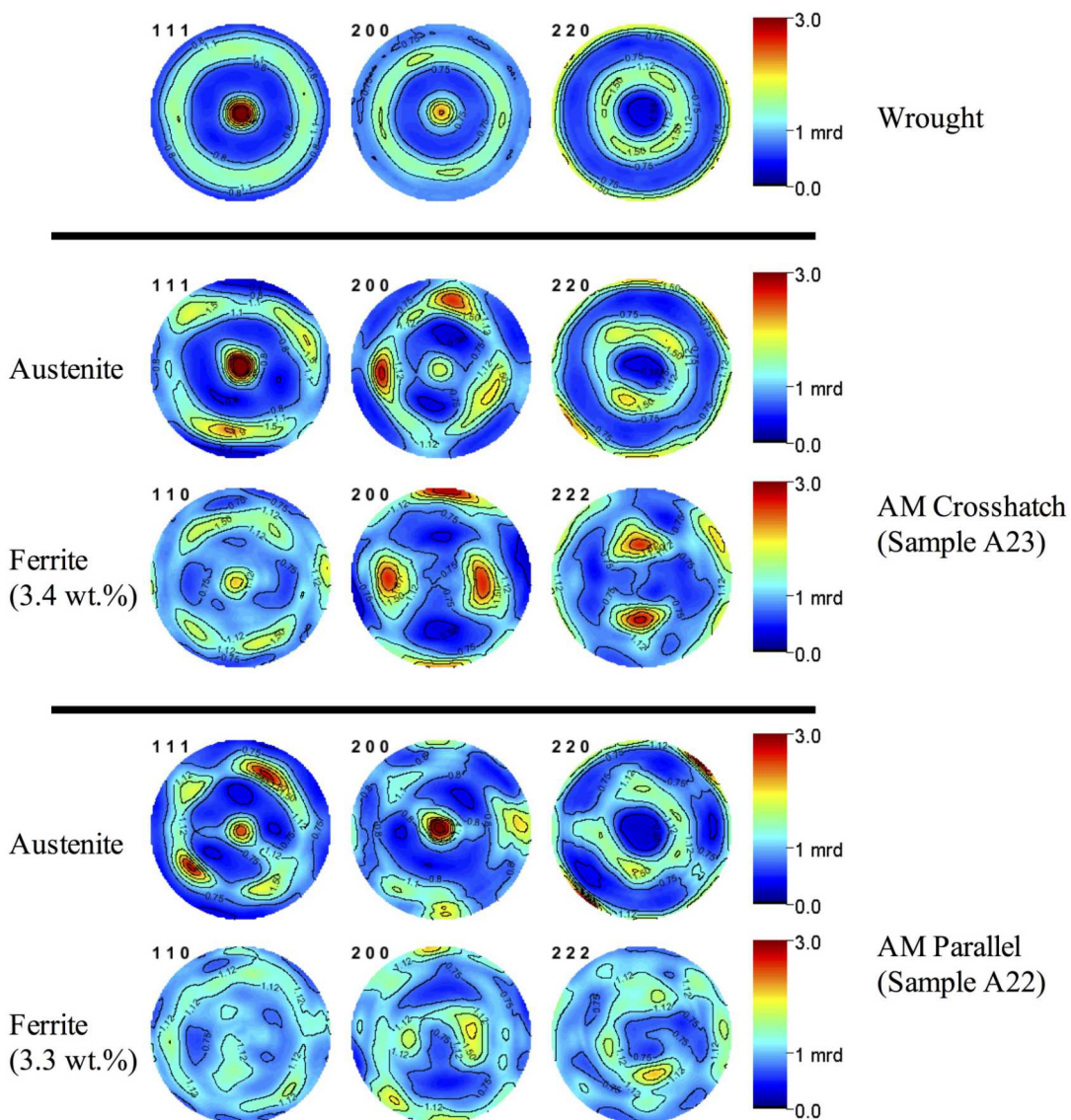


14.12. Starting and deformation textures of wrought and AM stainless

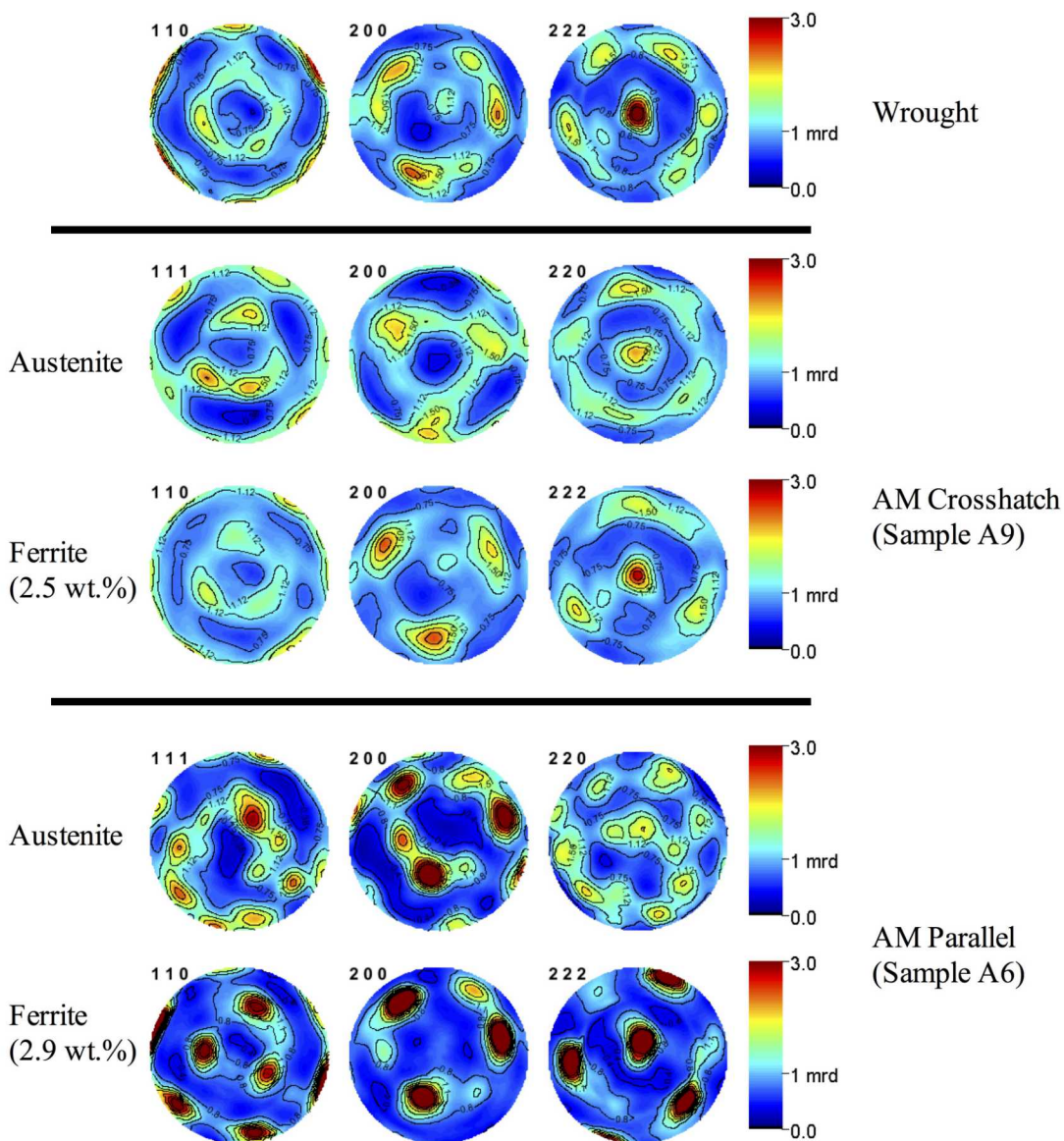
This page includes pole figures obtained prior to deformation. Samples A6 and A22 are derived from X bars made with a 3.8 kW, parallel scan process and are evaluated along the X direction. Samples A9 and A23 are derived from Z bars made with a 2.0 kW, crosshatch scan process and are evaluated along the Z direction.



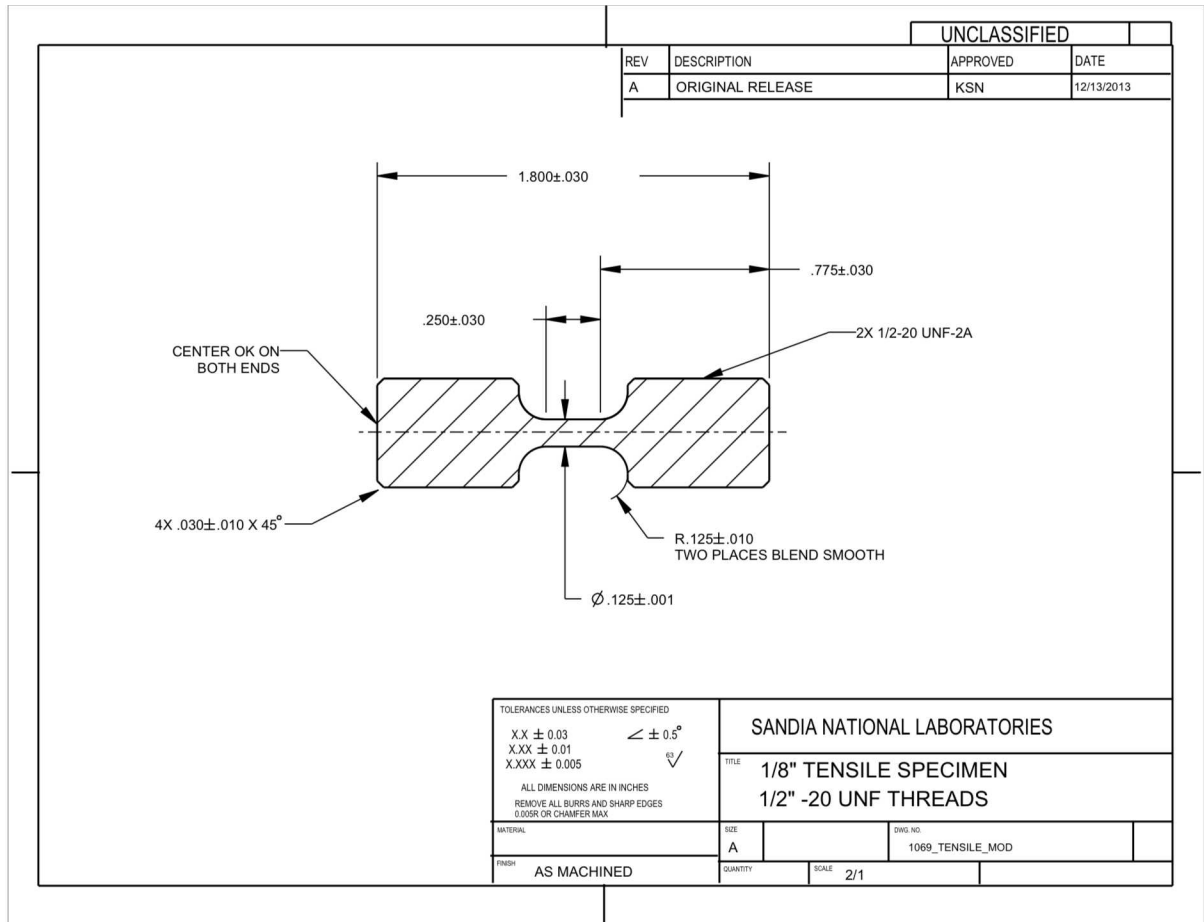
This page includes pole figures of the wrought and AM SS 304L following 0.32 tensile strain. Sample A22 is derived from an X bar made with a 3.8 kW, parallel scan process and is evaluated along the X direction. Sample A23 is derived from a Z bar made with a 2.0 kW, crosshatch scan process and is evaluated along the Z direction.



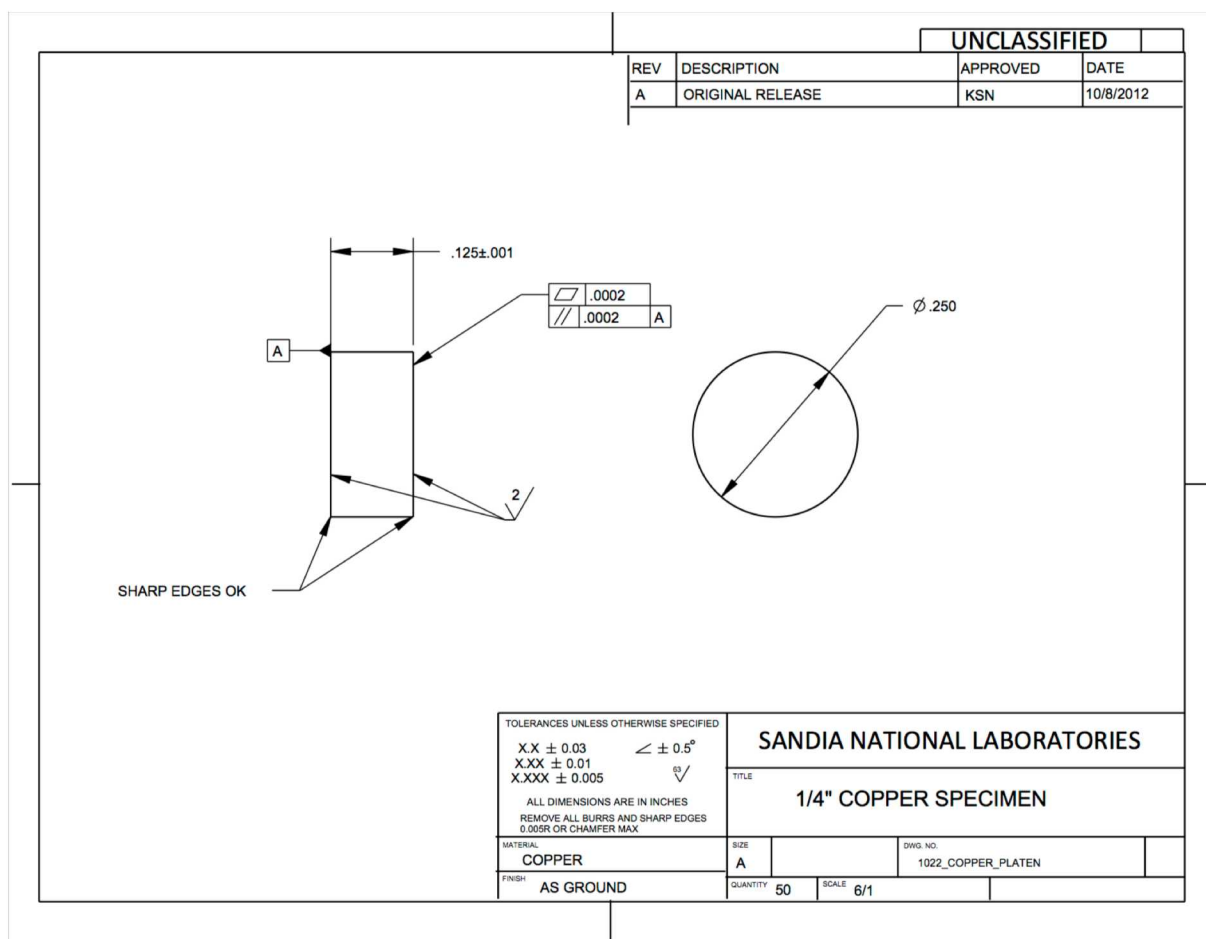
This page includes pole figures of the wrought and AM steel following 0.12 compressive strain. Sample A6 is derived from an X bar made with a 3.8 kW, parallel scan process and is evaluated along the X direction. Samples A9 is derived from a Z bar made with a 2.0 kW, crosshatch scan process and is evaluated along the Z direction.



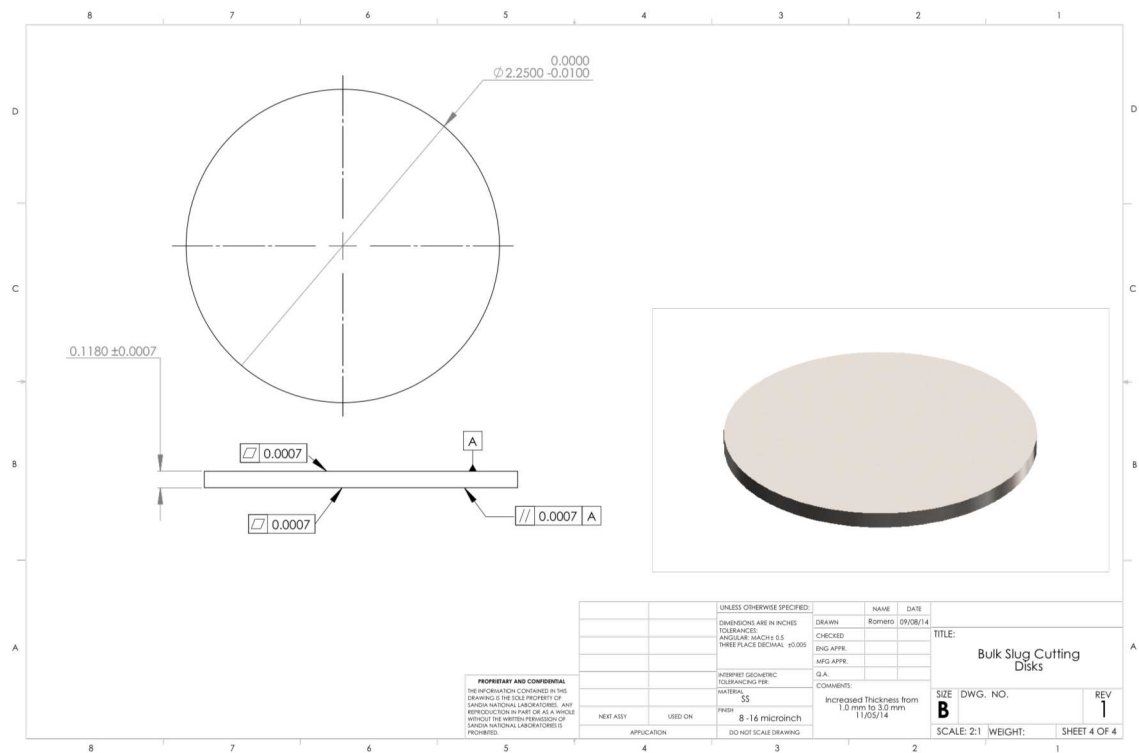
14.13 Machine drawing of tensile sample used for high strain rate testing



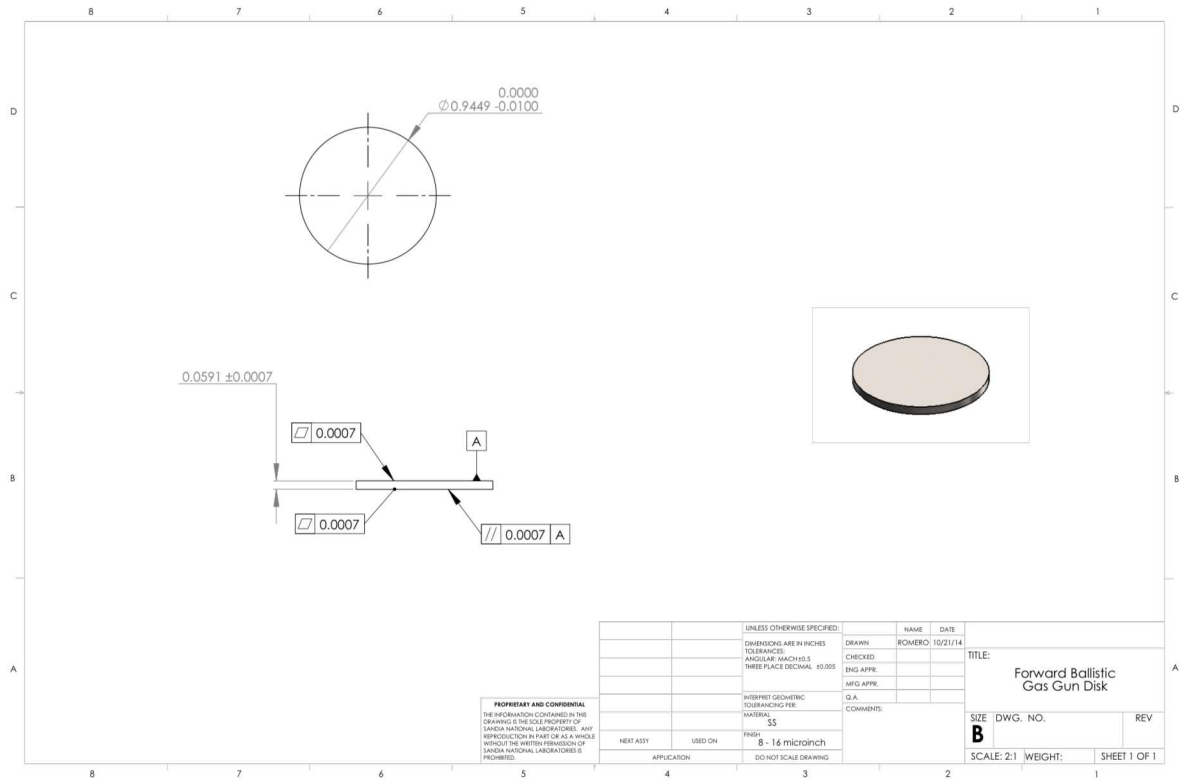
14.14. Machine drawing of compression sample used for high strain rate testing



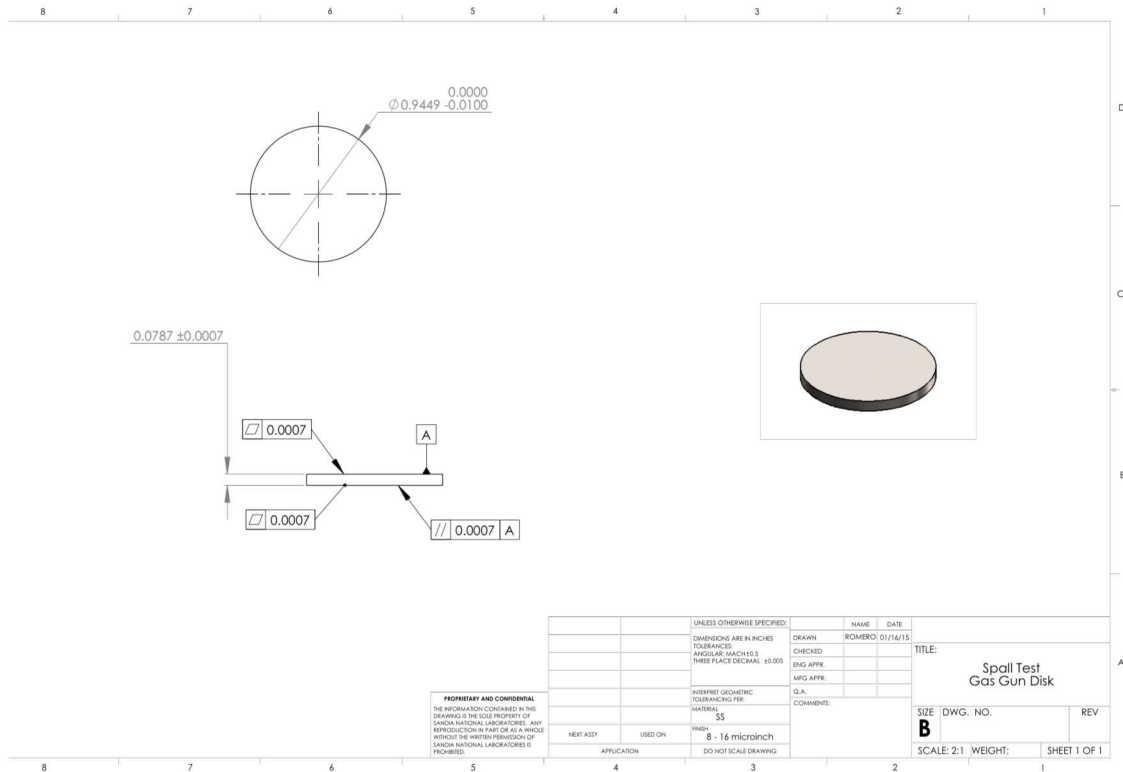
14.15. Machine drawing of discs used for reverse ballistic testing



14.16. Machine drawing of discs used for forward ballistic testing on gas gun instrument

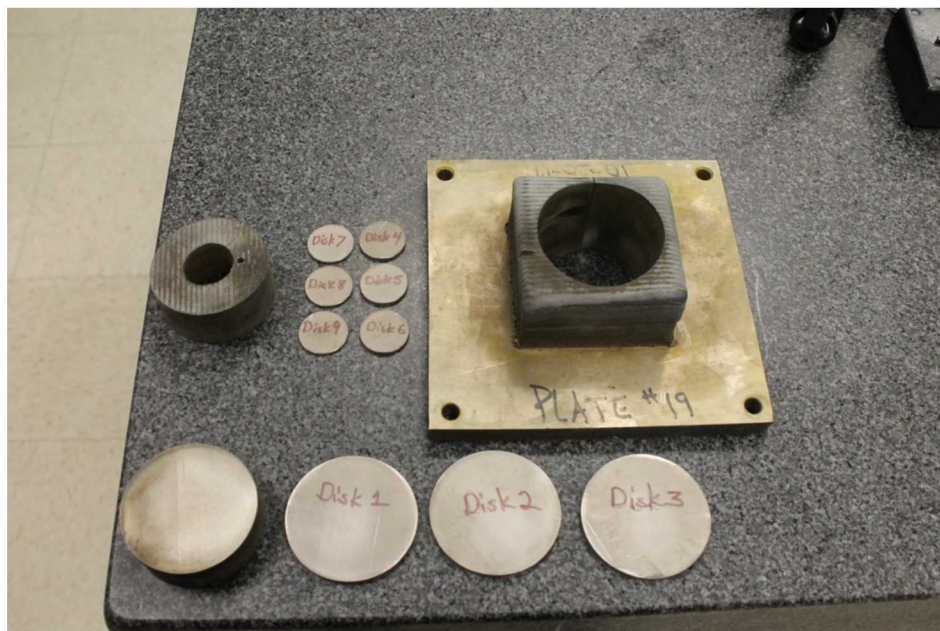
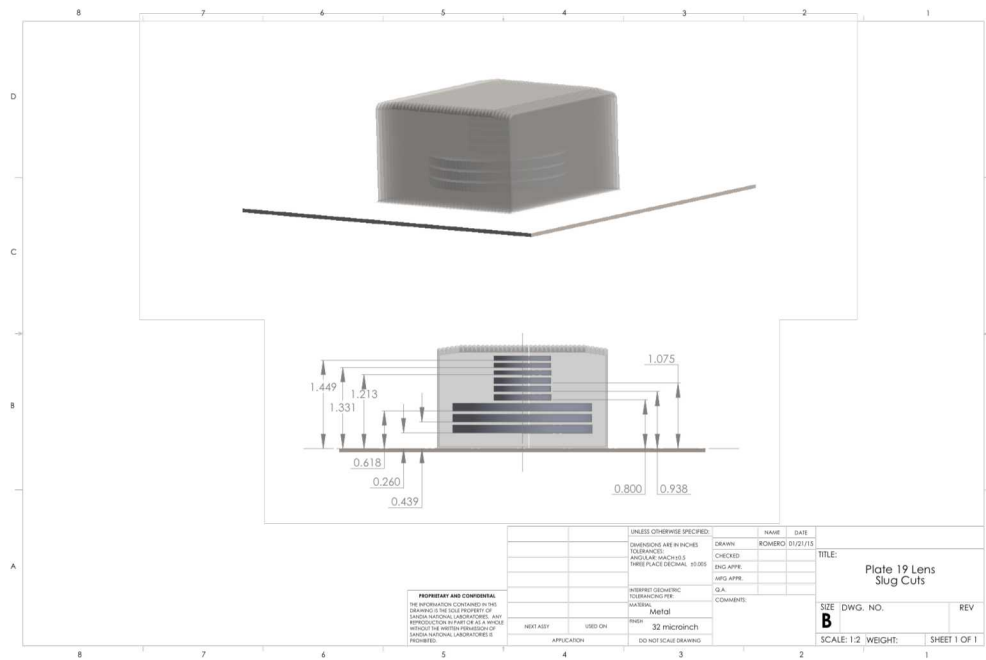


14.17. Machine drawing of discs used for spall testing on gas gun instrument

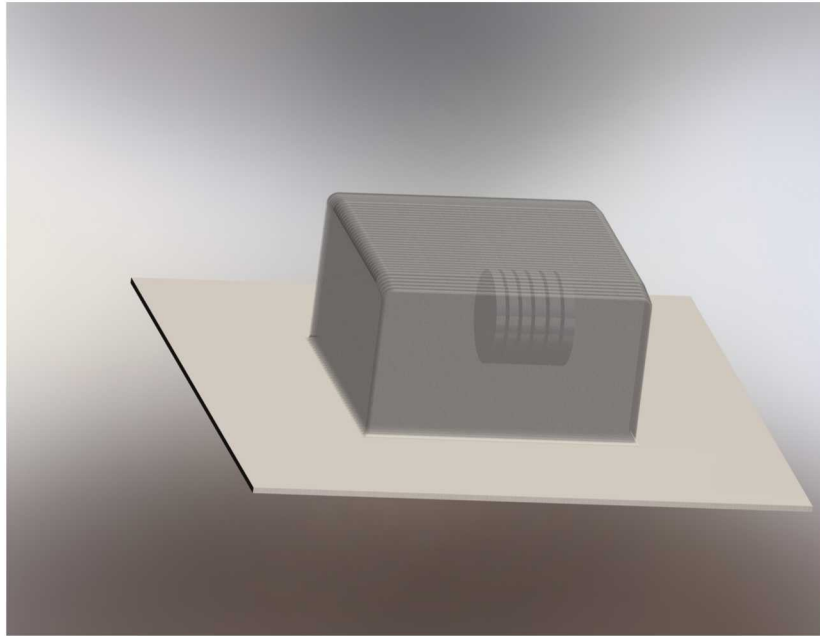


14.18. Drawings and photographs showing location of Z-cut discs (inside original AM block) used for impact tests on gas gun instrument

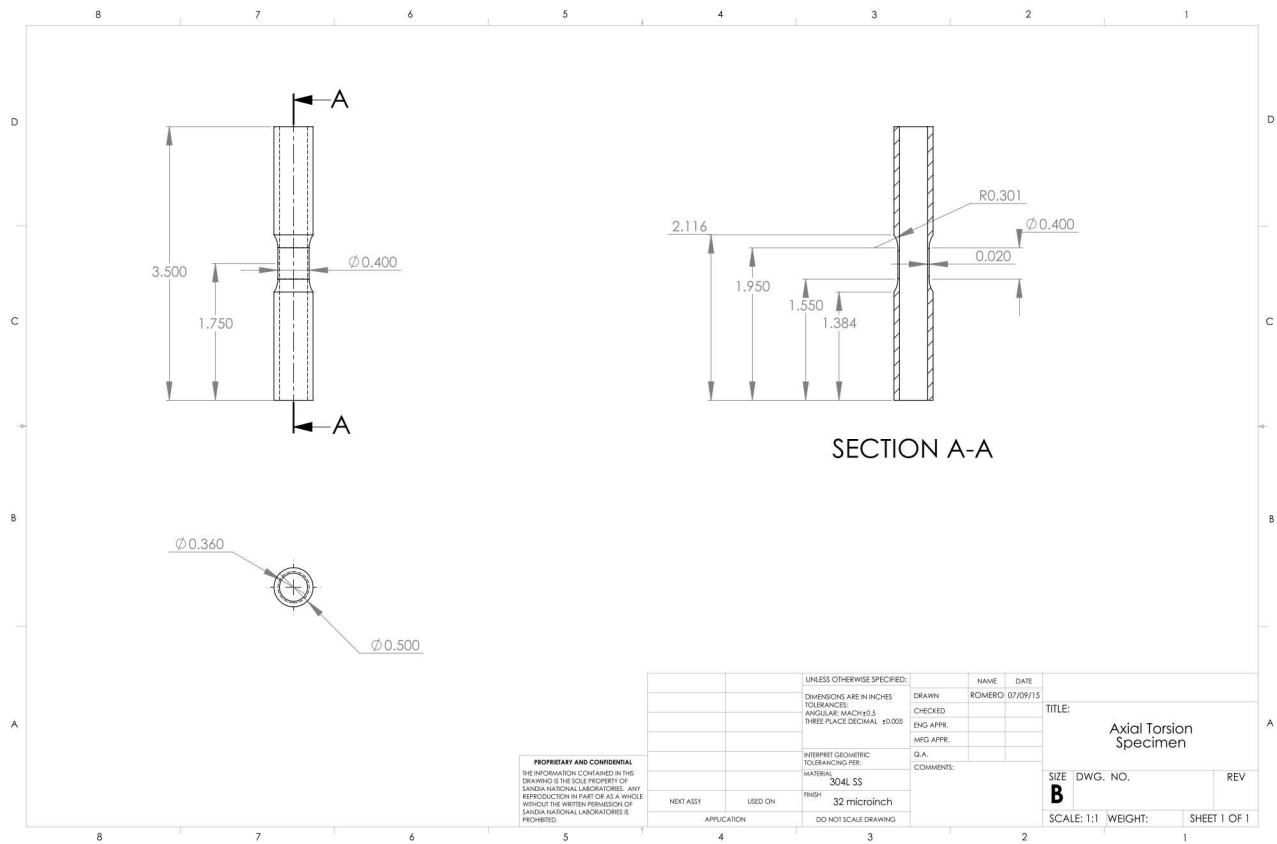
Circular samples shown in this appendix are Z-cut discs because their normal direction is aligned with the Z – direction of building (parallel with the laser vector).



14.19. Drawings showing approximate location of X-cut discs (inside original AM block) used for impact tests on gas gun instrument



14.20. Machine drawing of tension-torsion specimen used for testing



14.21. Photographs of Axial-Torsion test samples

The first two images show axial-torsion samples taken from the edge (first image) and the center (second image) regions of the wrought billet. The third image shows the two axial-torsion samples machined from an AM stainless steel 304L X-bar made using $P_{avg} = 3.8$ kW (Plate 2).





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