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**VERIFICATION BUILD DESIGN AND DEVELOPMENT FOR ADDITIVELY
MANUFACTURED 4340 STEEL**

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Picatinny Arsenal, New Jersey

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<p>Designing an appropriate verification build layout is critical in substantiating process parameters developed for a particular material in a particular additive manufacturing machine. Two different verification builds were executed with 4340 steel process parameters developed by the U.S. Army Combat Capabilities Development Command (DEVCOM) Armaments Center (AC), Picatinny Arsenal, NJ, for laser powder bed fusion (L-PBF) on an EOS M290. The two builds had different processing conditions, with the second build improving on the first. Both verification builds included XY and Z oriented tensile coupons in five locations (four corners and the center of the build plate). The specimens were then machined, heat treated, and characterized. The Z tensile properties were equivalent to the XY orientation tensile specimens in both verification builds. All but one specimen from the first verification build and all the specimens in the second verification build met 4340 wrought minimum requirements. The same processing parameters and metal powder verified in the second build were utilized to manufacture parts, but the parts failed to build to completion. The verification build, which had met the minimum mechanical requirements, failed to capture all the effects of having tightly packed parts on the build plate and resulted in build failures. The parts were eventually built by reducing the number of parts on the build plate, which improved the gas flow. As parts become larger and closer together, the nuances of gas flow in L-PBF of 4340 will need to be better understood and incorporated into process design that accurately predicts material quality.</p>									
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INTRODUCTION

Laser powder bed fusion (L-PBF) is a metals additive manufacturing (AM) process that uses a laser to selectively fuse together layers of metal powder. The parts built in this manner, layer by layer, can be produced fully dense to net shape and include highly complex features that would be difficult or impossible to make using traditional subtractive manufacturing techniques. The AM process complements other manufacturing processes by allowing rapid tooling and prototyping (ref. 1).

4340 steel is a high-strength, low-alloy steel used in many U.S. Army structural components. However, because 4340 is not a qualified material offered by AM machine manufacturers, processing parameters needed to be developed in-house for each L-PBF-AM machine. As such, parameters including laser power, gas flow, hatch distance, and laser scan speed need to be optimized. This has been done in previous work by the U.S. Army Combat Capabilities Development Command (DEVCOM) Armaments Center (AC), Picatinny Arsenal, NJ (refs. 1 and 2), for the EOS M270 and must now be completed for the EOS M290. A range of process parameters were initially down selected based on an analysis of density and tensile properties. Verification builds were then manufactured to establish statistical confidence and evaluate the limitations of the L-PBF process parameters selected.

Process verification is an essential component for ensuring quality and consistency in the transition from prototypes to part production. For L-PBF, process verification can be a complicated task due to the wide range of variables involved, including metal powder properties, machine parameters, machine preparation, and part orientation. Challenges of process verification and part building will be discussed in this report.

BACKGROUND

Process parameters for L-PBF of 4340 for the EOS M290 were developed by first building metallurgical cubes of 4340 by varying process parameters. These parameters were hatch distance, laser scan speed, laser power, and build platform temperature resulting in 64 different iterations. The density of these cubes, as a function of the parameters, were then statistically analyzed to establish a process window of acceptable parameters, which would produce specimens with a density exceeding 99% according to Archimedes and image analysis with optical microscopy. Next, tensile testing was performed on samples built with the narrowed range of optimized parameters, and the final parameters were down selected based on the mechanical property results. Verification builds were then built with the optimized process parameters to evaluate the repeatability and reliability of the process.

EXPERIMENTAL

The build layout for the verification builds is depicted in figure 1. The layout included a cluster of tensile specimens and metallurgical cubes built at five locations (center and four corners). Each cluster included three horizontal (X-Y) and three Z-oriented tensile specimens. Feedstock powder was also characterized. Characterization techniques for the powder included particle size, morphology, oxygen content, and internal particle porosity.

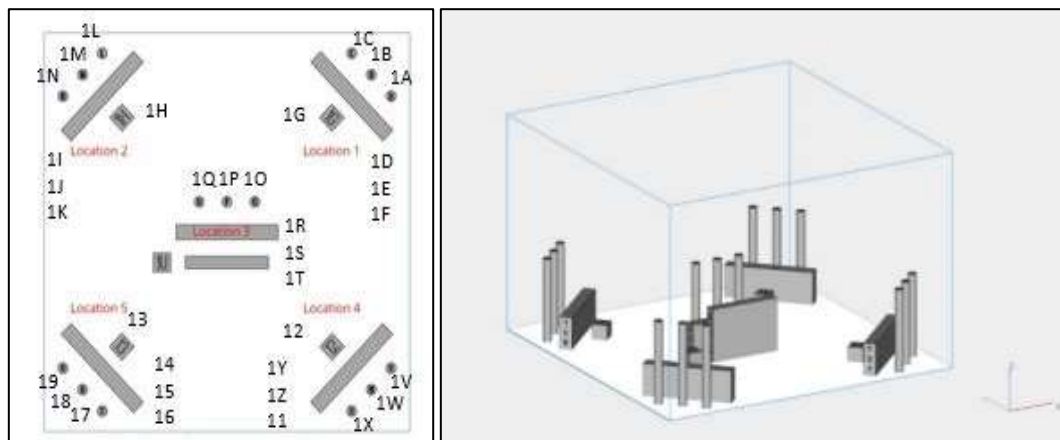


Figure 1
Schematic of top view (left) and angled view (right) of the build

The post-build heat treatment was as follows: The parts were stress relieved at 593 °C (1,099 °F) for 1 hr. Next, they were normalized at 900 °C (1652 °F) for 1 hr, furnace cooled to 650 °C (1,202 °F), and then air cooled to ambient. Finally, the specimens were austenitized at 816 °C (1,501 °F), quenched in oil, and double tempered at 190 °C (374 °F) for 2 hr each in accordance with AMS 2759 (ref. 3). The nominal hardness after heat treat was 50 HRC.

Upon completion of heat treatment, tensile specimens were prepared according to the ASTM¹ E8 standard with a 0.16-in. reduced section. Horizontal specimens were cut with centers at 0.235, 0.575, and 0.915 in. from the surface of the build plate. The tests were performed at room temperature on an MTS test frame with V-serrated wedges in hydraulic grips and a 50,000-lb load cell. The mechanical properties were compared to wrought 4340 heat treated to 50 HRC to determine whether the process parameters were acceptable.

RESULTS

Two verification builds were manufactured. The first verification build used metal powder with a high-phosphorus content—greater than 0.010 wt%—in a nitrogen gas environment. In addition, an original equipment manufacturer (OEM) standard gas flow nozzle was used. It was later learned that this nozzle type is not recommended for steels built using a 40- μ m layer thickness. Adjustments were made between the first verification build and the second verification build, which included using powder with a lower phosphorus content; the OEM recommended a grid flow nozzle and argon gas to lower the oxygen content. The details of these verification builds will be described in the following sections.

Verification Build 1

Powder

The powder from lot A used to manufacture the first verification build had low inter-particle porosity (fig. 2) and predominately spherical morphology. However, the phosphorus content was greater than 0.010 wt% as given in the AMS 6414 (ref. 4) standard, which can lead to embrittlement and a lower resistance to cracking (ref. 4) (table 1). An improper chemistry

¹ American Society for Testing and Materials.

specification given to the supplier allowed for phosphorus content less than 0.035 wt%. Unfortunately, phosphorus content was permitted to exceed the threshold for embrittlement, although the specification was satisfied. Previous experiments also revealed that higher build platform temperatures (160 to 175 °C / 320 to 347 °F) were necessary to eliminate widespread cracking. The powder surrounding the part in the build platform turned brownish after building in a nitrogen environment at 160 °C (320 °F), which corresponded to an increase in oxygen content (fig. 3 and table 2). After sieving, the oxygen content in the used powder was higher than virgin powder. The powder particle size did not change significantly in the build platform and after sieving.

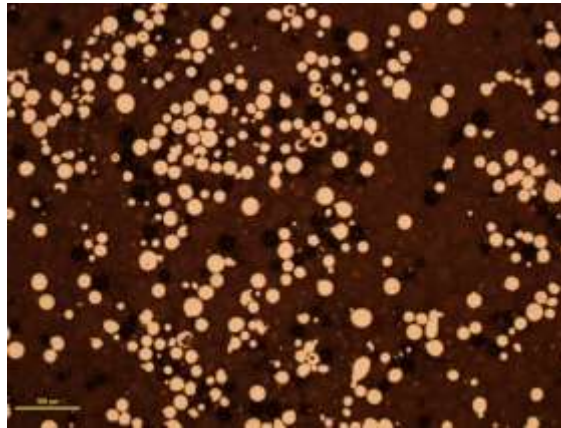
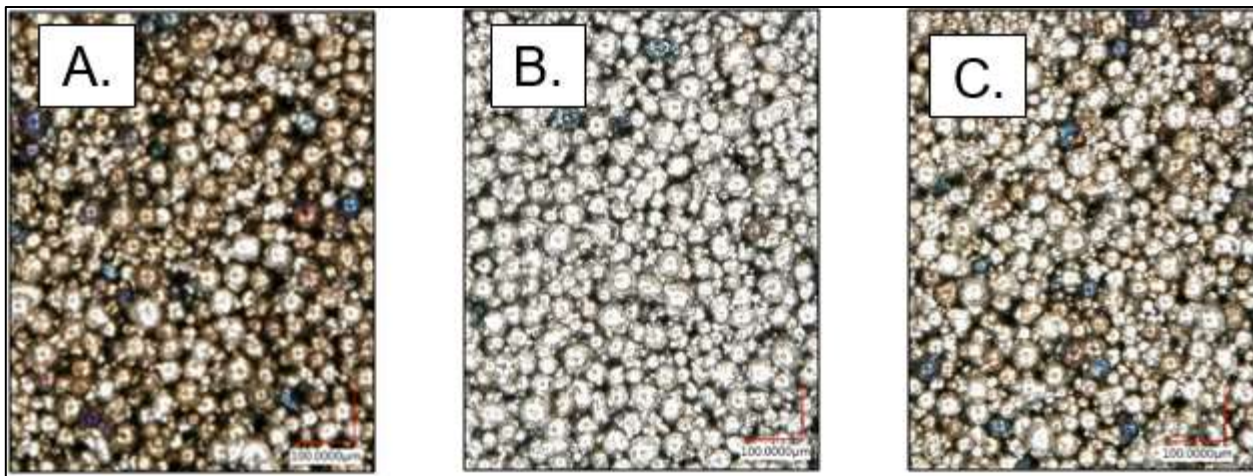


Figure 2
Optical micrograph of the cross section of lot A

Table 1
4340 powder chemistry of lot A

Element	AMS 6414	Lot A cert
C	0.38-0.43	0.41
Ni	1.65 -2.00	1.93
Cr	0.7- 0.9	0.82
Mn	0.6 -0.8	0.65
Mo	0.2-0.3	0.43
Si	0.15- 0.35	0.21
P	0.01 max	0.017
S	0.01 max	0.009

Note: Phosphorous content marked in red.



(a) Virgin lot A 4340 (b) Used lot A 4340 from build platform after build (c) Lot A 4340 powder after sieving

Figure 3
Optical images of lot A 4340 (scale bar 100 µm)

Table 2
Oxygen (O₂) content and laser diffraction of lot A powder

	Virgin powder	Collected from build platform verification Build B	Sieved powder C
d₁₀	25.30 µm	22.73 µm	24.89 µm
d₅₀	39.09 µm	35.47 µm	39.53 µm
d₉₀	59.45 µm	55.51 µm	61.42 µm
Mean	27.96 µm	25.49 µm	28.76 µm
Standard Deviation	10.77 µm	9.61 µm	10.97 µm
O₂	0.064 wt %	0.113 wt %	0.087 wt %

Mechanical Properties of Verification Build 1

Verification build 1 was built with the parameters shown in table 3. The specimens were then heat treated to 50 Rockwell C and machined as described previously in the experimental section. The average mechanical properties met the minimum wrought requirements (tables 4 and 5). However, there was one specimen that failed ductility requirements in the X-Y orientation. One major contributing factor to poor mechanical performance was defects on the fracture surface. The defects on the fracture surface had parallel lines on them (fig. 4). Afterwards, it was discovered that the standard gas flow nozzle was not recommend by the machine’s original equipment manufacturer (OEM) for their qualified steel powder built at 40-µm layer thickness. The standard gas flow nozzle likely led to inadequate gas flow to remove spatter from the build platform. (Spatter is molten

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material ejected from the melt pool.) The spatter can then fall back on the part and create an inconsistent melt pool which results in defects (ref. 5).

Table 3
Process parameters for verification build 1

Laser power	300 Watts
Laser scan speed	800 mm/sec
Hatch distance	0.11 mm
Layer thickness	0.04 mm
Stripe width	4 mm
Stripe overlap	0.08 mm
Laser beam diameter	0.08 mm
Build platform temperature	160 °C
Recoating speed	80 mm/sec
Gas pressure	0.75 mbar
Inert gas used	Nitrogen
Gas nozzle	Standard (EOS)
Hatch rotation	67 deg

Table 4
Z orientation tensile properties verification build 1

Z	Modulus (Mpsi)	Yield strength (ksi)	Ultimate tensile strength (ksi)	Elongation (%)	Strain at break
Average	27.67	226.66	286.37	8.03	9.30
Standard deviation	2.08	1.95	1.59	1.14	0.94
Relative standard deviation (%)	7.51	0.86	0.55	14.23	10.13
Wrought	30 nominal	205 minimum	235 minimum	6 minimum	6 minimum

Table 5
XY orientation tensile properties verification build 1

XY	Modulus (Mpsi)	Yield strength (ksi)	Ultimate tensile strength (ksi)	Elongation (%)	Strain at break
Average	28.41	224.90	287.33	7.43	8.76
Standard deviation	1.18	2.87	1.61	1.37	1.11
Relative standard deviation (%)	4.17	1.28	0.56	18.47	12.62
Wrought	30 nominal	205 minimum	235 minimum	6 minimum	6 minimum
Poor specimen (location 2)	28.96	221.89	286.73	3.5	5.7

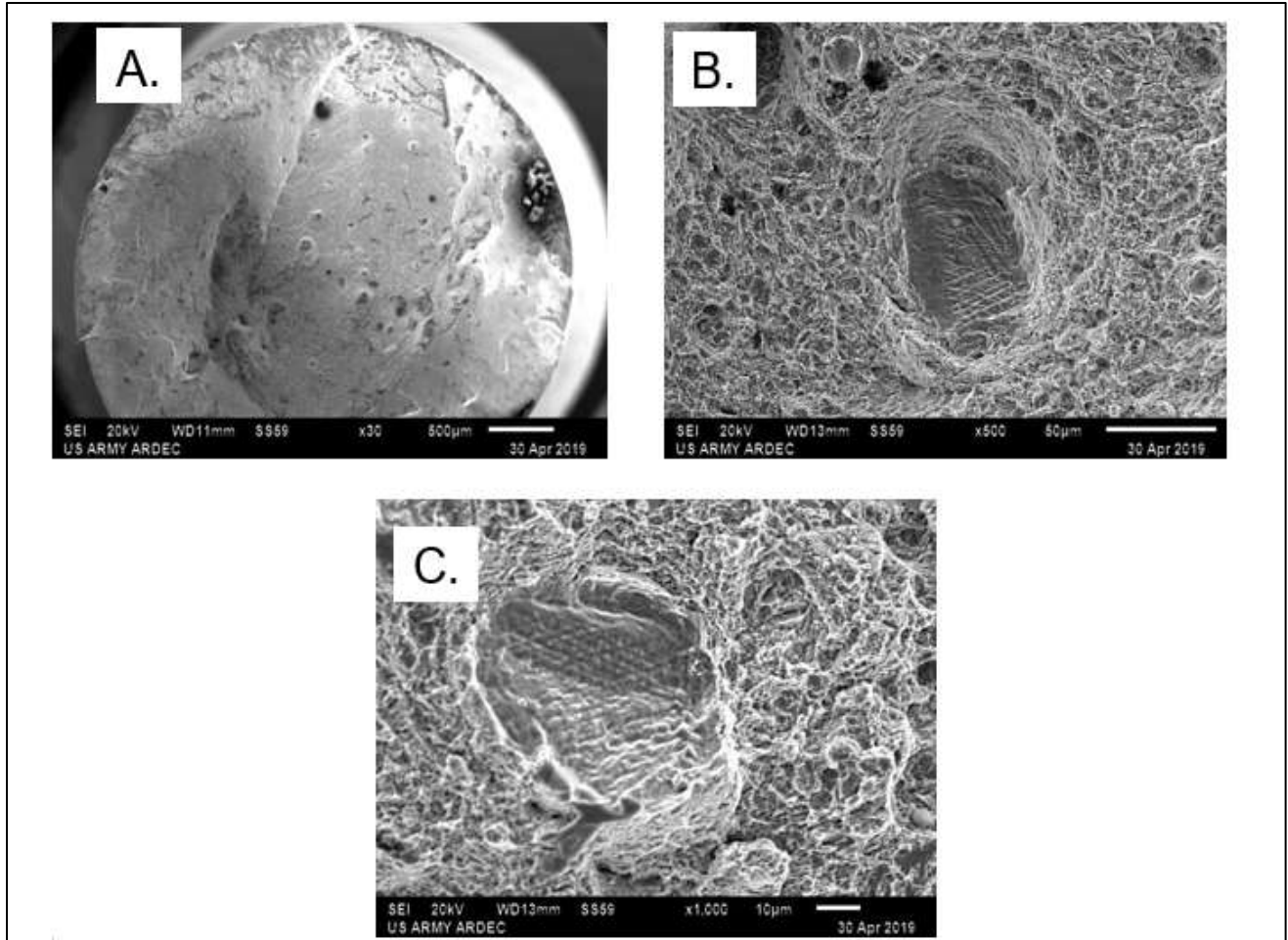


Figure 4

(A) Low magnification SEM micrograph of the fracture surface of worst performing sample, (B) 500x mag SEM micrograph of the fracture surface, (C) and 1,000x magnification SEM micrograph of the fracture surface

There were several modifications from build 1 that were implemented for verification build 2. First, AMS 6414 was specified in the acquisition of 4340 powder, which specifies low content (<0.01 wt%) for tramp elements including sulfur and phosphorus to prevent embrittlement. Next, argon gas was used to make the build chamber inert instead of nitrogen to lower build chamber oxygen content. Finally, the OEM recommended gas flow grid nozzle was used instead of the standard nozzle to improve gas flow and thereby spatter removal from the build chamber.

Verification Build 2

Powder

Powder lot B was employed for verification build 2. The powder certification complied with AMS 6414 requirements (table 6). In addition, the powder of lot B had low-internal porosity with spherical morphology (figs. 5 and 6). The processing of parts in an argon environment at 160 °C (320 °F) did not change the color of the powder surface significantly except for the first several millimeters on the powder bed. The oxygen content and powder size were also similar between the powder from the build platform, the virgin powder, and the sieved powder (table 7). It is likely that

argon reduces the acquisition of oxygen by the powder at an elevated build platform temperature (160 °C / 320 °F).

Table 6
4340 lot B chemistry

Element	AMS 6414	Powder certification
C	0.38-0.43	0.39
Ni	1.65-2.00	1.83
Cr	0.7-0.9	0.81
Mn	0.6-0.8	0.69
Mo	0.2-0.3	0.28
Si	0.15-0.35	0.21
P	0.01 maximum	0.008
S	0.01 maximum	<0.01

Note: The phosphorus content in lot B is below the 0.01 wt% maximum, as marked in red.

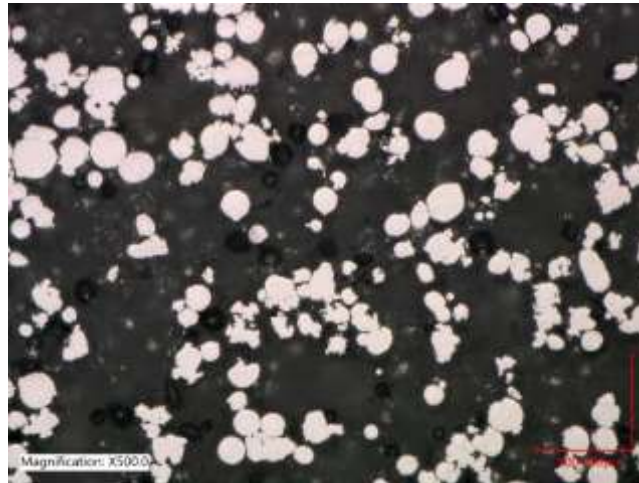
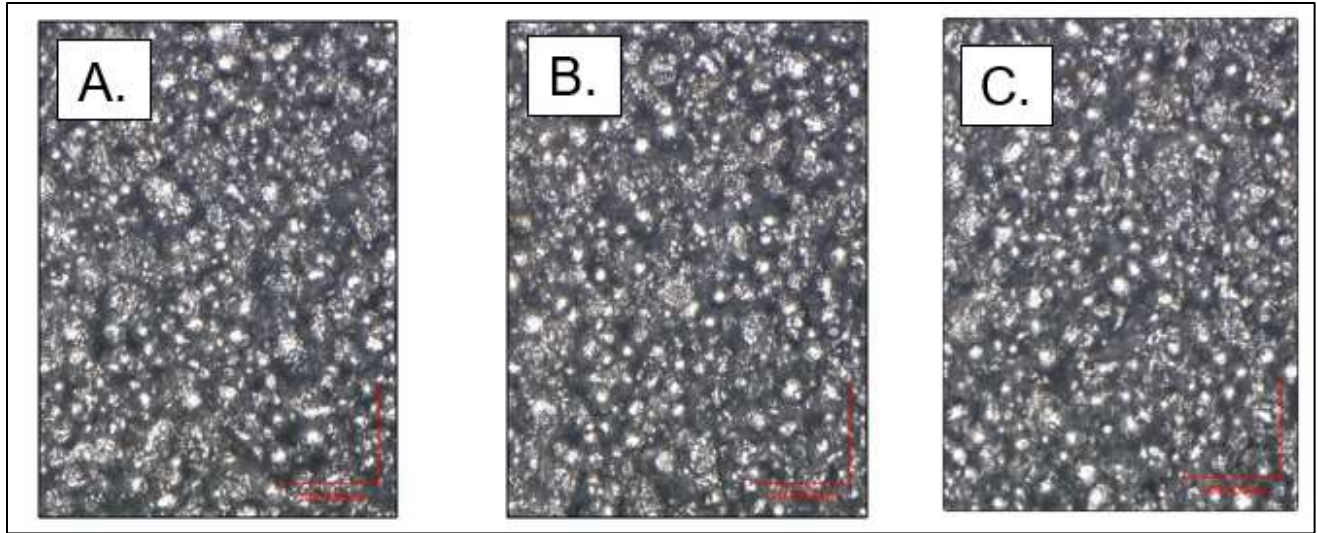


Figure 5
Optical micrograph (scale bar 100 μm) of the cross section of 4340 powder lot B



(a) Virgin lot B 4340 (b) Lot B 4340 from build platform after build (c) Lot B 4340 from the dispenser after sieving

Figure 6
Optical images (scale bar 100 μm) of lot B 4340 powder

Table 7
Oxygen (O₂) content and laser diffraction of AMS 6414 compliant power

	New lot 17099 A	Collected from build platform Build B	Sieved powder C
d₁₀	26.18 μm	24.81 μm	25.13 μm
d₅₀	38.14 μm	35.74 μm	36.96 μm
d₉₀	56.90 μm	53.15 μm	55.27 μm
Mean	26.63 μm	28.2 μm	28.76 μm
Standard deviation	9.82 μm	9.00 μm	9.48 μm

Mechanical Properties

Process parameters for verification build 2 are included in table 8. Verification build 2 mechanical properties show significant improvement in mechanical properties in comparison to build 1. All tensile specimens exceeded minimum wrought requirements. Furthermore, the ductility was approximately 4% greater and more consistent in build 2 versus build 1 (tables 9 through 11), as marked in red in the tables. The mechanical properties are isotropic as well after heat treatment. The mechanical properties were considered acceptable to build 4340 components in subsequent builds.

Table 8
Process parameters for the verification build 2

Laser power	300 Watts
Laser scan speed	800 mm/sec
Hatch distance	0.11 mm
Layer thickness	0.04 mm
Stripe width	4 mm
Stripe overlap	0.08 mm
Laser beam diameter	0.08 mm
Build platform temperature	160 °C
Recoating speed	80 mm/sec
Gas pressure	0.75 mbar
Inert gas used	Argon
Gas nozzle	Grid
Hatch rotation	47 deg

Table 9
Z orientation tensile properties low phosphorous 4340, Argon, grid nozzle

Z	Modulus (Mpsi)	Yield strength (ksi)	Ultimate tensile strength (ksi)	Elongation (%)	Strain at break
Average	28.05	219.51	278.44	12.67	12.20
Standard deviation	1.39	1.58	1.45	0.65	0.42
Relative standard deviation (%)	4.96	0.72	0.52	5.13	3.44
Wrought	30 nominal	205 minimum	235 minimum	6 minimum	6 minimum

Table 10
XY orientation tensile properties low phosphorus 4340, Ar, grid nozzle

XY	Modulus (Mpsi)	Yield strength (ksi)	Ultimate tensile strength (ksi)	Elongation (%)	Strain at break
Average	27.99	219.05	280.85	11.81	11.83
Standard deviation	1.80	1.84	1.48	1.12	0.88
Relative standard deviation (%)	6.43	0.84	0.53	9.48	7.44
Wrought	30 nominal	205 minimum	235 minimum	6 minimum	6 minimum

Table 11
Ductility comparison of verification build 1 and build 2

Build-orientation	Elongation (%)	Relative standard deviation elongation (%)	Strain at break	Relative standard deviation elongation (%)
Build 1 Z	8.03	14.23	9.30	10.13
Build 2 Z	12.67	5.13	12.20	3.44
Build 1 XY	7.43	18.47	8.76	12.62
Build 2 XY	11.81	9.48	11.83	7.44

Component Build

After the successful second verification build, a subsequent job loaded with parts was built with the same processing conditions and powder lot. However, the job failed because the recoater blade jammed on spatter, which landed on the parts in the front of the build platform (fig. 7); it can land back on the part to create disturbances in the melt pool. The presence of large spatter indicates that the gas flow was not adequate to remove it from the build (fig. 8). There were also regions of poor gas flow (dark), which had a higher concentration of melt pool defects in contrast to lighter regions (figs. 7 and 9).

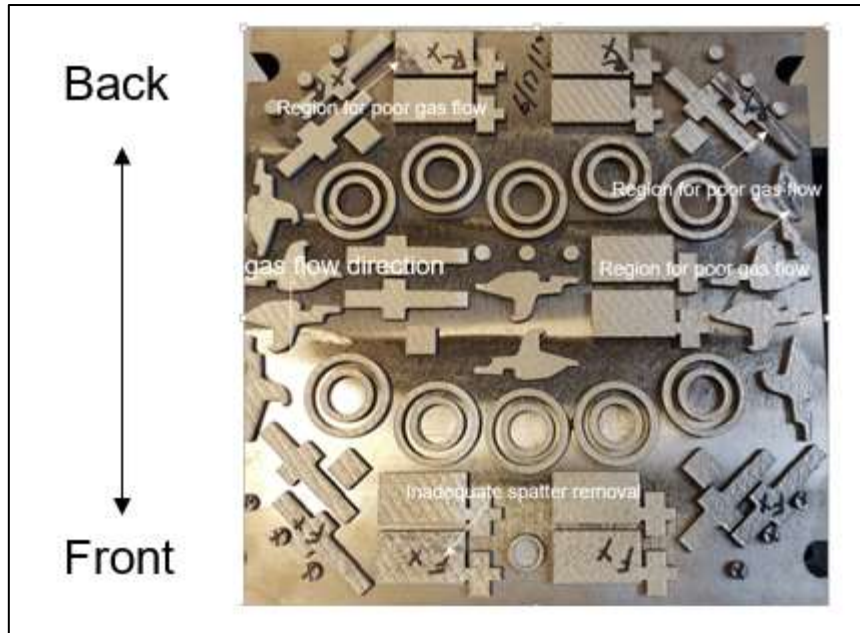
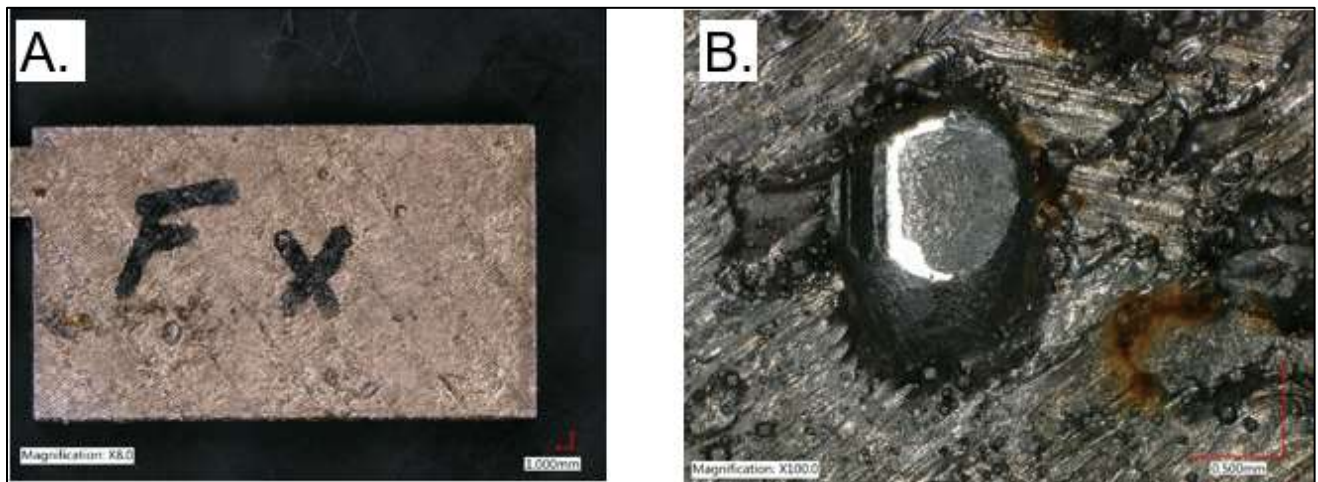


Figure 7
Overall photograph component of build 1 (gas flow = 0.75 mbar)



(a)
Low optical micrograph

(b)
High optical micrograph

Figure 8
Low and high magnification optical micrographs of part from build 1 with poor spatter removal
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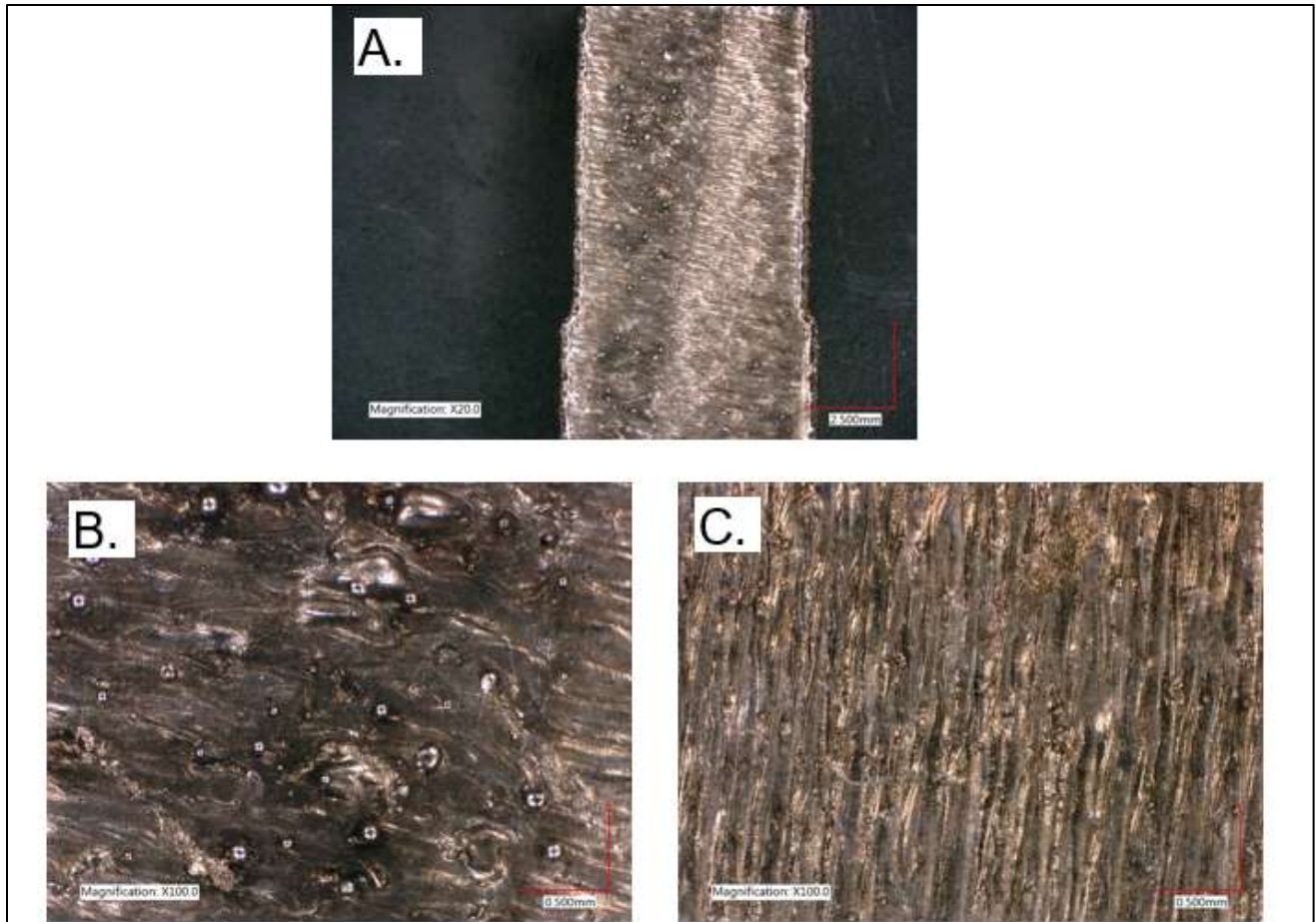


Figure 9

Low magnification (100x) optical micrograph of dark color area from component build 1 (A), detail showing optical micrograph of dark region with localized poor gas flow (B), and detail showing high magnification of region with adequate gas flow (C)

The gas flow was increased to 1.0 mbar from 0.75 mbar for component build 2 (fig. 10). All other parameters between build 1 and 2 were the same. The dark regions of poor gas flow decreased in size from build 1, but they were still present (figs. 10 and 11). There was also the presence of large protruded particles in front of the build plate (fig. 12). The build failed in the same manner as component build 1, recoater jam. It was then decided that the parts in the front needed to be removed for the next build.

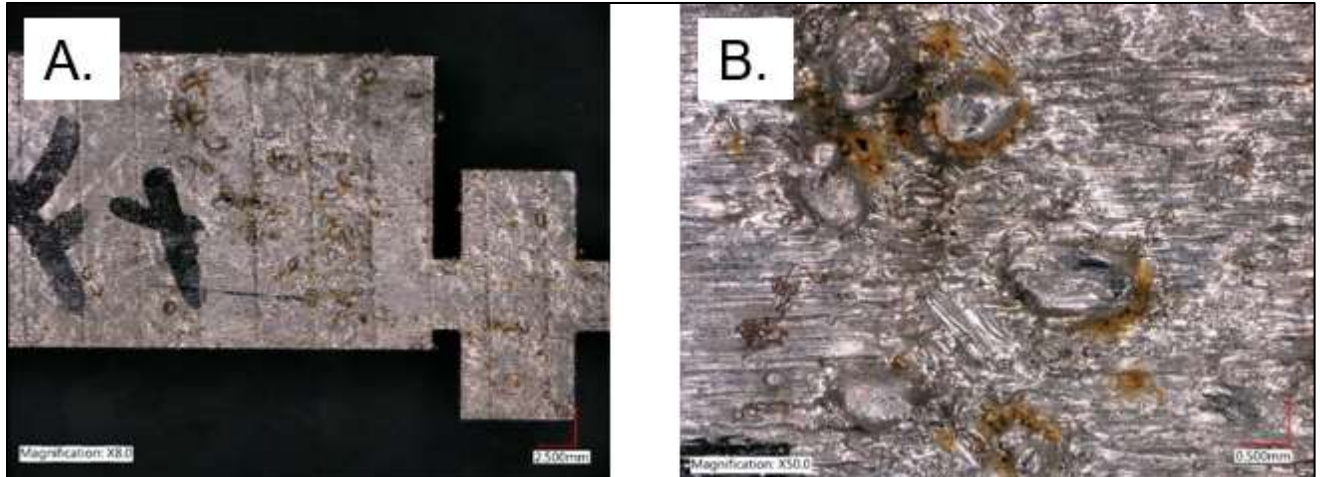


Figure 12

Low magnification optical micrograph of part with poor spatter removal build 2 (A) and detail showing higher magnification with poor spatter removal (B)

After removing the parts located at the front of the build plate, build 3 completed to full build height (fig. 13). The mechanical properties of the Z-oriented specimens satisfied wrought 4340 heat treated to 51 Rockwell C (table 12). Most of the parts were built below the height where the reduced section of the tensile specimen was built. Therefore, the Z-oriented tensile specimens' results do not demonstrate the effectiveness of the process performance.

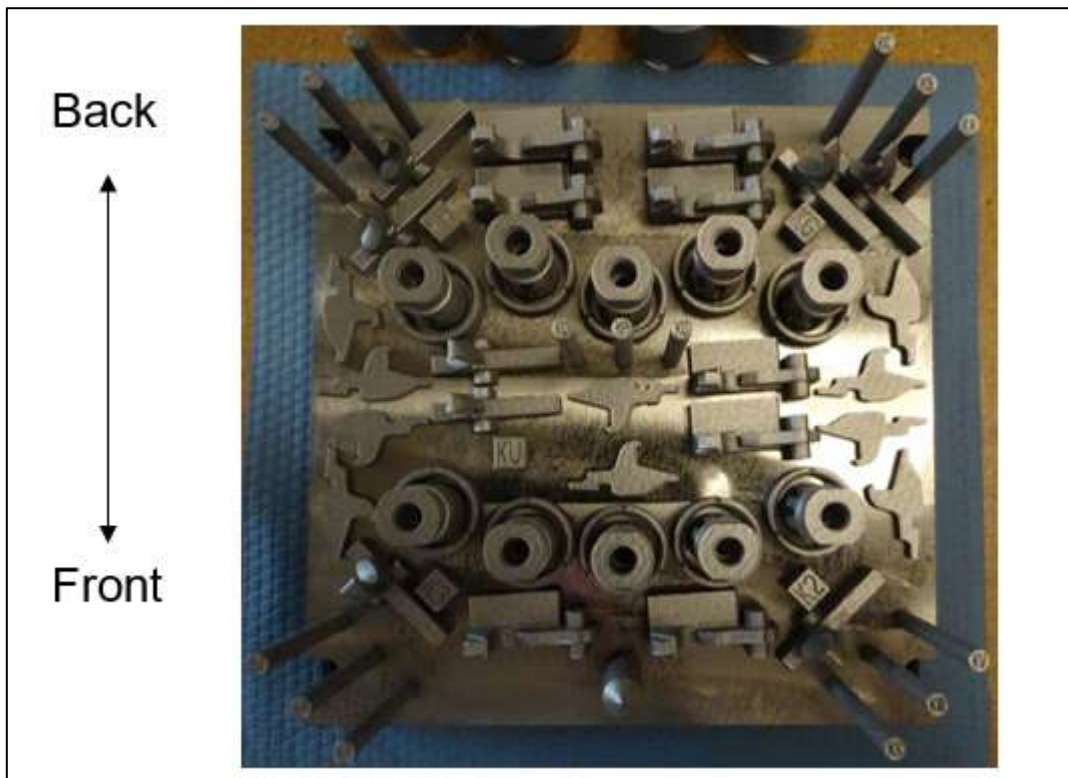


Figure 13

Overall photograph of completed component build 3 (gas flow = 1.0 mbar)

Table 12
Z orientation tensile properties of part build 3 as compared to wrought

Z	Modulus (Mpsi)	Yield strength (ksi)	Ultimate tensile strength (ksi)	Elongation (%)	Strain at break
Average	28.84	214.44	276.89	12.02	11.62
Standard deviation	0.71	2.12	1.53	0.54	0.45
Relative standard deviation (%)	2.46	0.99	0.55	4.49	3.87
Wrought	30 nominal	205 minimum	235 minimum	6 minimum	6 minimum

CONCLUSIONS

Two verification builds were manufactured to substantiate the process parameters developed in-house for laser powder bed fusion (L-PBF) of 4340 on the EOS M290 and validate the metal powder chemistry. The first verification build had one specimen fail to meet 4340 wrought minimum requirements, and improvements were incorporated for the second verification build. All the specimens of the second build met the minimum requirements. The same parameters used in the second verification build were then used to manufacture parts. Despite the success in the second verification build, the first attempt to build parts failed when the recoating blade jammed on spatter that had landed on components at the front of the build plate. The presence of spatter indicated that the gas flow was inadequate. In addition, there were dark regions of the build brought about by disturbances in the melt pool due to poor gas flow. Even though the gas flow was increased for the second build attempt, the recoater blade jammed again on spatter. For the third attempt, a work around was implemented of removing the specimens from the front of the build plate, which had the spatter that had jammed the recoater blade. This allowed for a dead zone in the build chamber and the remainder of the parts were successfully built and exhibited material properties stronger than wrought.

The need to implement a work around shows that the verification build results did not directly translate to build performance when the build is loaded with parts. A different verification build configuration (i.e., with parts that occupy a greater cross-sectional area of the build plate) may be needed to ensure that the process parameters (particularly, gas flow) are optimized. The many variables that affect adequate gas flow will need to be addressed in the objective to design a comprehensive verification build layout.

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REVIEW AND APPROVAL OF ARDEC TECHNICAL REPORTS

Verification Build Design and Development for Additively Manufactured 4340 Steel
Title

Date received by LCSD

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