METALS ADDITIVE MANUFACTURING DEVELOPMENT AT HARVEST TECHNOLOGIES

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Abstract

Harvest Technologies received an EOS M280 in April of 2013 for the production of metal parts through additive manufacturing (AM). Inconel 718 was chosen as a starting material due to its high-end applications in the oil and aerospace industries. Two major areas are of high priority in understanding the machine: (1) mechanical property characterization and (2) geometrical production capability through building prototype models. The following is a working document of Harvest’ progression in developing knowledge in the field of metals AM.

1. M280 Parameter Characterization

1.1. Baseline Build

A key step in starting any new process is to create a baseline; this involves defining and documenting any input parameters and constants, running them through the process, and defining and documenting desired output variables. After documenting inputs and outputs from a baseline process, results from further testing can be compared back to the baseline in order to observe how changes in input affect output. The baseline is also a valuable troubleshooting tool; if the user suspects errors due to uncontrolled process variables, the baseline process can be run in order to verify or negate these suspicions. For these reasons, a baseline build was created and run immediately after undergoing M280 operation training.

The following considerations were made in creating a baseline build for the M280:

- Should be run with virgin powder in order that powder degradation does not affect the ability to run a baseline build.
- Should be able to run quickly in the machine. Thus, small bars built in the xy-plane are used.
- Should be amenable to obtaining output results quickly in-house. Thus, small-diameter bars are used such that they can be pulled to fracture using an in-house 10 kN capacity load cell. Furthermore, the bars are pulled at a relatively fast crosshead speed during tensile testing.
- Comparison with external properties is not of primary concern in the current stage. The primary goal of the current stage is to understand how changes in input parameters in the M280 affect output parameters.
-Although external comparison is not of utmost concern in the current stage, ultimate tensile strength (UTS) is the primary output variable tracked due to its ability to be compared with external results. Yield strength and tensile modulus can be used for internal comparison, but not for external comparison due to the relatively fast crosshead pulling speed. In addition, the lack of a small extensometer prevents accurate tracking of strain.

-Samples for microstructural evaluation should be included.

With the aforementioned considerations in mind, the following paragraphs and figures describe the baseline build geometry, input process parameters, post-processing methods, and output measurement processes.

As previously mentioned, small tensile bars were chosen for the baseline build. A reduced section length of .5” and an as-built diameter of .106” were used. Assuming a UTS of 203 ksi for Inconel 718 after Hot Isostatic Pressing (HIP) and heat treating (HT), this diameter would result in a tensile load cell requirement of 8 kN, providing a 1.25 safety factor for the load cell.

The arrangement of bars within the build chamber can be seen in Figure 1, below. As shown in Figure 1, sixteen tensile bars are oriented on the xy-plane. The long axes of eight bars are aligned parallel to the x-axis and the long axes of the remaining eight are parallel to the y-axis. Twelve rectangular samples are also spaced throughout the build. These samples are meant for microstructural examination along the xy, xz, and yz-planes.

![Figure 1. Baseline build arrangement](image)

The tensile bars are built with solid support structures on the grip sections and block supports in the middle sections, as shown in Figure 2. The solid supports prevent the bars from warping during the build process, and the block supports provide a base on which to build the reduced
sections. In order to facilitate removal of the block supports, the supports are offset ~.006” from
the downward-facing surfaces of the bars.

![Figure 2. CAD image of tensile bar set up in Magics](image)

The block supports can be easily removed using pliers, but leave residue on the reduced
section of the tensile bars. In order to obtain a smooth surface on the reduced sections, the bars
are turned on a lathe and lightly sanded, as shown in Figure 3. Figure 4 shows the
post-processing progression of the bars.

![Figure 3. Residual support removal in reduced section of tensile bar](image)
The default “EOS In718 Performance” job is used to control exposure parameters, such as laser power and scan speed. The parameters in this job were developed by EOS and are hidden from the user.

An MTS Insight 10 tensile testing machine is used to pull the tensile bars to fracture. A relatively fast crosshead speed of .2”/min is used in order to facilitate quick feedback prior to running further builds. An extensometer is not used to measure strain, but rather the crosshead position is used to estimate strain. The lack of an extensometer is expected to cause significant variation in elastic modulus, elongation at break, and yield strength measurements. However, the lack of an extensometer does not affect measurement of ultimate tensile strength, which is a main reason that UTS is the primary focus of the current study.

Two baseline builds were run prior to running any other builds. UTS was the most consistent property among the 32 bars, with a 2% standard deviation. Elastic modulus was the least consistent property measured, with a 17% standard deviation among the 32 bars. Variation in pre-tensile test preload is likely to be a primary cause of this deviation, because the grips must settle into the specimens before crosshead position can accurately track strain. Using an extensometer, this “settling in” would not be an issue, as the extensometer would measure the strain in the reduced section directly. Properties from the two baseline builds are shown in Figure 5. The average UTS across the two builds was 159 ksi, which closely corresponds with published values.
Figure 5. Mechanical properties from two baseline builds

1.2. General Microstructural Evaluation

The following images are optical micrographs of samples from a build using EOS-provided laser start parameters for Inconel 718 in as-built condition. The samples were polished and electrolytically etched with a solution of 70% phosphoric acid and 30% distilled water. Figures 6 and 7 show micrographs of the samples in the xy-plane. The cross-hatched scan lines can be clearly seen in Figure 6. Small cracks appear to be present in the sample as can be seen in Figure 7, which could be a result of residual stresses in the material. Figures 8 and 9 show micrographs of the samples in the xz-plane, and Figures 10 and 11 show micrographs of the samples in the yz-plane. Cross-sections of the melt pools can be seen in these vertical planes. Further microstructural examination will be highly important for studying the effects of HT and HIP.
Figure 6. Etched surface along the xy-plane at 100X

Figure 7. Etched surface along the xy-plane at 500X
Figure 8. Etched surface along the xz-plane at 100X

Figure 9. Etched surface along the xz-plane at 500X
Figure 10. Etched surface along the yz-plane at 100X

Figure 11. Etched surface along the yz-plane at 500X
1.3. Parameter Cause and Effect Study

The parameter cause and effect study aims to display how altering input parameters affects output variables. For this study, laser power, scan speed, and scan spacing were chosen as the primary input parameters to vary. These three parameters are direct contributors to the amount of energy transferred into the metal by the scanning laser, as shown in Equation 1.1 below. Layer thickness similarly affects the energy density imparted by the laser to the powder bed; however, the ability to choose layer thickness is not currently available in the M280 software. Energy density can be described as follows:

\[ E_\rho = \frac{nP}{t_s \sigma}, \quad (1.1) \]

where \( E_\rho \) represents energy density, \( P \) is laser power, \( n \) is the number of times the laser scans each cross-section, \( t \) represents layer thickness, \( s \) represents scanning speed, and \( \sigma \) is the offset between adjacent scans, or scan spacing.

A series of builds were run in which the laser power, scan speed, and scan spacing were increased or decreased by 10% with respect to start values provided by EOS. Only one parameter was varied per build. The start values provided by EOS are listed in Table 1, and the parameters used in the various characterization builds can be seen in Table 2.

**Table 1. Default EOS start parameters**

<table>
<thead>
<tr>
<th>Power (W)</th>
<th>Scan Speed (mm/s)</th>
<th>Scan Spacing (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>285</td>
<td>960</td>
<td>0.11</td>
</tr>
</tbody>
</table>


Table 2. Parameters used in characterization builds

<table>
<thead>
<tr>
<th>Build ID</th>
<th>Power (W)</th>
<th>Scan Speed (mm/s)</th>
<th>Scan Spacing (mm)</th>
<th>Powder Quality</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>EOS IN 718</td>
<td>EOS IN 718</td>
<td>EOS IN 718</td>
<td>Virgin</td>
</tr>
<tr>
<td>2</td>
<td>EOS IN 718</td>
<td>EOS IN 718</td>
<td>EOS IN 718</td>
<td>Virgin</td>
</tr>
<tr>
<td>3</td>
<td>EOS IN 718</td>
<td>EOS IN 718</td>
<td>EOS IN 718</td>
<td>Used/sifted</td>
</tr>
<tr>
<td>4</td>
<td>EOS IN 718</td>
<td>EOS IN 718</td>
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<td>Used/sifted</td>
</tr>
<tr>
<td>5</td>
<td>256.5</td>
<td>960</td>
<td>0.11</td>
<td>Used/sifted</td>
</tr>
<tr>
<td>6</td>
<td>313.5</td>
<td>960</td>
<td>0.11</td>
<td>Used/sifted</td>
</tr>
<tr>
<td>7</td>
<td>285</td>
<td>864</td>
<td>0.11</td>
<td>Used/sifted</td>
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<tr>
<td>8</td>
<td>285</td>
<td>1056</td>
<td>0.11</td>
<td>Used/sifted</td>
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<td>10</td>
<td>285</td>
<td>960</td>
<td>0.099</td>
<td>Used/sifted</td>
</tr>
<tr>
<td>13</td>
<td>285</td>
<td>960</td>
<td>0.11</td>
<td>Used/sifted</td>
</tr>
<tr>
<td>14</td>
<td>EOS IN 718</td>
<td>EOS IN 718</td>
<td>EOS IN 718</td>
<td>Used/sifted</td>
</tr>
</tbody>
</table>

Builds 1 and 2 listed in Table 2 are the baseline builds previously discussed, whose data can be seen in Figure 5. Builds 3 and 4 repeat the two baseline builds from a parameter standpoint, but were run with used powder instead of virgin to study whether powder quality had significantly changed over the course of the first two builds. Builds 5 through 8, 10, and 11, and 13 are used for parameter cause and effect studies. Build 14 is a repeat of builds 3 and 4, to study whether powder quality had significantly changed over the course of the study. Based on the data displayed in Figure 12 and Figure 13, neither the used powder nor 10% parameter changes appear to significantly affect xy-plane tensile properties. Figure 12 displays the average UTS per build. Figure 13 displays the average UTS per build normalized by a value of 154.2 ksi, which is the value published by Morris Technologies. Input parameters are also displayed in Figure 13, normalized by the EOS provided default starting values listed in Table 1.
Additive manufacturing processes typically produce anisotropic materials, with less desirable mechanical properties along the z-axis (where the z-axis is parallel to the build direction). Thus, z-axis properties are often used to verify build quality. In order to obtain an initial estimate of
how close the as-built z-properties are to expected values, a build with vertically oriented tensile bars was run. The resulting properties can be seen in Figure 14, where the average z-axis UTS of 146 ksi is roughly 10% lower than the average xy-plane UTS.

![UTS vs. Orientation](image)

**Figure 14.** Average xy-plane vs. z-plane UTS

A parameter characterization study was done on the vertical bars as well. Seven sets of eight vertically oriented bars were built in which laser power, scan speed, and scan spacing were increased or decreased from nominal as listed in Table 3.

**Table 3.** Exposure parameter variations for vertically oriented bars

<table>
<thead>
<tr>
<th>Set</th>
<th>Power (W)</th>
<th>Scan Speed (mm/s)</th>
<th>Scan Spacing (mm)</th>
<th>Powder Quality</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>213.75</td>
<td>960</td>
<td>0.11</td>
<td>Used/sifted</td>
</tr>
<tr>
<td>2</td>
<td>356.25</td>
<td>960</td>
<td>0.11</td>
<td>Used/sifted</td>
</tr>
<tr>
<td>3</td>
<td>285</td>
<td>720</td>
<td>0.11</td>
<td>Used/sifted</td>
</tr>
<tr>
<td>4</td>
<td>285</td>
<td>1200</td>
<td>0.11</td>
<td>Used/sifted</td>
</tr>
<tr>
<td>5</td>
<td>285</td>
<td>960</td>
<td>0.088</td>
<td>Used/sifted</td>
</tr>
<tr>
<td>6</td>
<td>285</td>
<td>960</td>
<td>0.132</td>
<td>Used/sifted</td>
</tr>
<tr>
<td>7</td>
<td>71.25</td>
<td>960</td>
<td>0.11</td>
<td>Used/sifted</td>
</tr>
</tbody>
</table>

Average UTS for the various sets of vertically oriented bars can be seen in Figure 15. Although the typical variation of parameters is roughly ±25% of nominal, UTS is not largely affected by the parameter changes. Set 7, however, in which laser power is decreased by 75%, yields a significant decrease in UTS. Thus, a plateau appears to exist for UTS such that if energy
density is above a critical value, UTS remains relatively constant. This phenomenon can be seen in Figure 16, where UTS is plotted vs. energy density for the eight cases shown in Figure 15. Additional data must be gathered to validate this behavior.

![Normalized Z-axis UTS and Input Parameters](image)

**Figure 15.** Average UTS of vertically oriented bars with varying input parameters

![Z-axis UTS vs. Energy Density](image)

**Figure 16.** Average UTS vs. Energy Density for vertically oriented bars
1.5. Larger bars for ASTM testing

While the baseline build is useful for in-house parameter affect studies, the smaller bars present more room for error during post-processing and testing. Furthermore, the fast crosshead speed used in the baseline build does not conform to the speeds specified in ASTM E8. Thus, larger bars of .25” diameter were also built in the xy-plane and tested for comparison. These bars were pulled at a .015 in/in/min strain rate until reaching a stress of 130 ksi, at which the speed was increased to .25 in/in/min. In the next phase of M280 characterization, these tensile bars and pulling parameters will be used to record and report tensile data. The average UTS for the larger bars is shown in comparison to the smaller bars in Figure 17.

![UTS (ksi)](chart)

**Figure 17. Average UTS of .25” diameter bars vs. .106” diameter bars**

Average UTS, elongation at break, yield strength, and elastic modulus values for the .25” diameter bars are listed in Table 4. Measurement of yield strength and elastic modulus are challenging due to the existence of a pre-yield “shoulder” at which the slope changes from one linear segment to another. In calculating modulus and yield strength, the segment of data prior to the shoulder was avoided by using a 1% strain offset rather than the standard .2% offset, and elastic modulus was calculated between 1% and 9% strain. It is suspected that the first shoulder occurs due to stress relaxation within the as-built tensile specimens, but it could also be due to the lack of an extensometer. An extensometer will be used in further studies to verify or negate this speculation. The pre-yield shoulder can be seen in the tensile curve shown in Figure 18.

**Table 4. Average mechanical properties for .25” diameter specimen**

<table>
<thead>
<tr>
<th>UTS (ksi)</th>
<th>Elongation at Break (%)</th>
<th>Yield Stress (1% offset - ksi)</th>
<th>Elastic modulus (1% to 9% - ksi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>150.1</td>
<td>33.9</td>
<td>112.0</td>
<td>1301.7</td>
</tr>
</tbody>
</table>
Initial steps of characterizing an EOS M280 using Inconel 718 are documented in this report. A baseline build was created and run in order to provide a point of reference for further parameter characterization. The average UTS across two baseline builds was 159 ksi, which closely corresponds with published data. Optical micrographs were produced for a baseline build as well, revealing fine columnar structures produced by high cooling rates. Small cracks appear to be present in the higher magnification images which could be a result of residual stresses in the material.

The variation of parameters in a cause and effect study generally appeared to have little influence on the UTS of the tensile bars. Laser power, scan speed, and scan spacing were varied independently, typically by ±10% of the default values for bars built in the xy-plane and by ±20-25% for bars built along the z-axis. A significant drop in UTS did occur, however, when laser power in bars built along the z-axis was reduced by 75%. Based on these results, it appears that when energy density is above a critical value, UTS is largely unaffected by varying input parameters. This behavior should be validated by further study. The average UTS of tensile bars
built parallel to the z-axis is generally 10% lower than that of bars built in the xy-plane. This anisotropy is typical of additively manufactured parts.

In further study, an extensometer will be used to record the strain dependent tensile properties of the material. Impact, hardness, and fatigue will be studied as well. Stress relief, heat treating and hot isostatic pressing will be used to improve mechanical properties, and the resulting microstructures will be compared with those in as-built specimens.