Developing Processing Parameters for Nickel-base Superalloys for the Electron Beam Melting Additive Manufacturing Process

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Abstract

A methodology was established to assist in the development of process parameters for nickel-base superalloys in the electron beam melting (EBM) process. The methodology was demonstrated on the Ni-base superalloy Alloy 718. The methodology includes characterization of the feedstock material, material parameter development, and build sample analysis. All steps were completed in order to understand the material's behavior in the EBM system and to produce defect free parts. Extensive research has been done on Alloy 718 has been characterized and used in an Arcam A2X system. Build themes have been developed to produce components with mechanical properties meeting the AMS 5662 and ASTM F3055-14a testing requirements.

Introduction

Over the last few years, the manufacture of material and components in a layer-by-layer fashion termed additive manufacturing (AM) has seen tremendous growth in applications of the technology and for advanced structural materials. Metal AM processes typically use a laser or an electron beam as a heat source to melt metal powder. Laser based techniques are generally performed near room temperature and experience rapid solidification as a result. Much work has been done on laser based Alloy 718 material production and characterization, using both Powder Bed Fusion and Direct Energy Deposition.

Alloy 718 was chosen as a candidate alloy due to the high industrial demand. It is a solution annealed and precipitation hardened material designed for high temperature applications. Alloy 718 is hardened by the precipitation of secondary phases (e.g. gamma prime and gamma double-prime) into the metal matrix. This alloy is the workhorse of nickel super-alloys and it is widely used for gas turbine components, jet engines, pump bodies and parts, rocket motors, thrust reversers, nuclear fuel element spacers, hot extrusion tooling and other applications requiring oxidation and corrosion resistance. The advantages of Alloy 718 are mechanical and chemical stability at elevated temperatures.

The present study concerns the development of a methodical approach for developing EBM processing parameters for high temperature Ni-base superalloys. Utilizing the approach presented here, material themes for processing Alloy 718 have been developed and demonstrated to produce material with desirable tensile properties when post-processed.

Experimental

Feedstock Material

The feedstock material utilized in this study was plasma wire Alloy 718 powder with a size distribution of 40-120 μm (-100+325 mesh) and nominal chemical composition as given in Table I. The powder was manufactured by Advanced Powders and Coatings (AP&C) of Quebec, Canada. In general, the powder is highly spherical as shown in Figure 1 and contains small amounts of entrapped gas as a result of the manufacturing process. Plasma atomized powder was selected for the EBM technology for its spherical morphology, fully dense particles, packing and flowability characteristics.

Table I: Nominal chemical compositions of the Alloy 718 powder used in this work given as weight percent.

Cr	Fe	Nb	Mo	Ti	Cu	Al	C	Ni
18.5	18.5	5	3	1	0.15	0.5	0.05	Bal

Figure 1: Cross section of AP&C Alloy 718 plasma wire atomized feedstock material used in this study.

In selecting the AP&C powder, several characteristics of the powder were determined to confirm the suitability of the Alloy 718 powder for use in the EBM process. Detailed in Figure 2 are the steps followed for evaluating the powder. To determine the powder's susceptibility to explosion, a minimum ignition energy test was performed following standard ASTM E2019

Figure 2: Process flow diagram for analyzing suitability of candidate feedstock powder for the EBM process.

Figure 3: Process flow diagram indicating the order of process parameter development used in developing material themes for Alloy 718 for use in the EBM process.

[1]. The test measured the ease of ignition of a dust cloud by electrical and electrostatic discharge. Electrodes were connected to a circuit that produced a spark with a known energy as a cloud of powder blown past the spark. In the case of the Alloy 718 powder used in this study, ignition did not occur.

Directly impacting the ability to process the powder is its flowability during the raking stage and within the gravity fed powder hoppers. The minimum requirement for powder flowability is 25 s/50 gm. Following ASTM B213, a Hall flow meter was used to test flowability [2]. The powder must also have high apparent density (apparent density must be >50% of the density of solid material) to be able to melt the powder and obtain fully dense parts. Further, the powder particle size distribution should be between 45-105 μm with a normal distribution curve. Lastly, the powder was mounted and polished to check for internal porosity. Ideally the powder should have low amounts of internal porosity due to entrapped gases not being able to escape while solidifying.

Electron Beam Melting

Electron beam melting (EBM) is an AM technology that selectively consolidates metal powders such as titanium and cobalt alloys to fabricate 3D structures. Compared to conventional manufacturing processes, the EBM process is capable of fabricating low-volume, high-value articles at reduced lead times. The technology was invented in 1993 in

Sweden at the University of Technology in Gothenburg. Arcam was founded in 1997 and sold its first commercial system in 2002. The company currently has over 200 systems installed around the world [3,4].

All samples were built using an Arcam A2X EBM machine. An interior schematic of the EBM system is shown in Figure 3. The EBM system consists of an electron beam gun, vacuum chamber ($\approx 0.05^{-4}$ Pa), build tank, and powder distribution mechanisms (powder hoppers and rake). Within the electron beam gun, a tungsten filament is heated to emit electrons accelerated at high voltage (60 kV), resulting in an electron beam (carrying high kinetic energy) that is focused by electromagnetic lenses. The system is capable of achieving scan speeds up to 8000 m/s, electron beams positioning accuracy of ± 25 μm and layer thicknesses ranging from 50-200 μm [3]. Additionally, due to the high power of the electron gun, metals with melting temperatures up to 3,400 $^{\circ}\text{C}$ can be melted [4]. The build volume of the A2X is 200x200x380 mm.

Figure 4: Interior schematic of the Arcam EBM vacuum system.

Process Parameter Development

Comprising the build sequence in the EBM process, are three main steps: 1) Initial plate preheating 2) Layer pre-heating 3) Layer melting. Identified in Figure 4 are the steps taken to develop the processing themes for Alloy 718. The first item identified was the maximum current that can be used with the powder before smoking occurs. Smoke takes place when the charge distribution density exceeds the critical limit of the powder. As a result, an electrical discharge occurs since the powder particles repel each other, causing a powder explosion inside the chamber. To conduct the smoke test, the powder was scanned with the electron beam under vacuum at room temperature. The smoke test consisted of using various machine parameter settings of line order, line offset, beam speed, and focus offset to determine which settings were within the “smoke-free” parameter window.

Next, a sintering test was conducted to identify the temperature required to pre-heat the build platform and sinter the Alloy 718 powder underneath the build plate for a period of 20 minutes. In the case of Alloy 718, a temperature of 1000

°C was determined to be ideal due to the ability to suppress precipitation of large needle like δ that has the ability to span many grains. Once the correct start temperature and start plate materials were identified, the first process build was prepared. The first build must be a simple geometry, like blocks with no negative surfaces or supports. There are three main build themes for every build: the pre-heat theme, the melt theme, and the support theme. The advantage of the Arcam system is that theme parameters can be modified while the system is building. For every build step there is a theme that provides the parameters to the machine. The initial themes were modified to reflect the findings in the smoke test, so that they were within the parameter window, to eliminate smoke during the build. Next, the preheat theme was modified by taking the average current obtained in the sintering test and using it as the beam current.

Powder sintering was controlled by the pre-heating theme parameters: min/max current, focus offset, and number of pre-heating repetitions. The pre-heating current controls the amount of energy that is delivered during one cycle run of the heating process. The pre-heat focus offset controls the electron beam diameter during pre-heating. The focus offset parameter provides the area where energy is being distributed and can be set to deliver maximum energy without hard-sintering the material. The number of repetitions conveys how many times the build is pre-heated and is used to raise and stabilize the bed temperature. These parameters control the temperature and stabilize the powder before the melting process begins.

Figure 5: Cube layout used for optimizing the focus offset of the electron beam. Each block was processed with a different focus offset with circled block 5 yielding the best top surface finish.

The melt step in the EBM process commences at the conclusion of pre-heat II and generally utilizes a focused electron beam with a lower current. During this process, the powder surface must be even and smooth to obtain fully dense parts. The key process variables during the melting process are the speed function and focus offset for the electron beam. The speed function can be considered an equation that determines the electron beam current and velocity as a function of line scan length. Higher values for the speed function provide

higher speed and deliver less energy when melting. The focus offset of the electron beam is adjusted to control the melt by providing an even and smooth upper surface. Focus offset calibration was done by modifying the focus offset in each cube to obtain the best top surface finish, as seen in previously-built samples in Figure 5, where the best surface is the one circled.

Mechanical Test Sample Verification Build

Once the parameters were determined to be sufficient, a verification build was manufactured using a 150x150 mm 316 stainless steel start plate. The verification build (Figure 6) consisted of four rectangular blocks of 100x25 mm and 32 cylinders of 10 mm in diameter and 105 mm in height. Additionally, four cubes are also included for chemistry and microstructure analysis. The stainless steel plate was given an initial plate pre-heat temperature of 950 °C and a sinter temperature of ≈ 20 minutes. Overall, the build time was ≈ 30 hrs with a ≈ 6 hour period required for cool-down of the build. For the present build, a layer thickness of 75 μm was chosen as a compromise between build speed and surface quality.

Figure 6: a) CAD model of the mechanical test sample verification build. b) Alloy 718 mechanical test sample build completed in this study.

Material Post-processing

To remove potential porosity the as-built material was hot isostatically pressed (HIP) at 1200 °C for 240 minutes at a pressure of 100 MPa. Following the HIP, the material was given a solution and aging treatment. Solutioning was done at 1066 °C for a 2 hour period followed by a precipitation hardening cycle at 760 °C for 10 hrs, and 650 °C for 20 hrs.

Microstructure Characterization

Samples were sectioned using an Allied High Tech TECHCUT 5 and mounted in KonductoMet using a Buehler SimpliMet XPS1. The mounted samples were metallographically prepared using successively finer silicon carbide grinding paper and given a final polish with 1 μm diamond on an Allied High Tech MetPrep 4. Chemical etching via submersion using a mixture of HCl, acetic acid, and HNO_3 (1:1:1) was used to reveal the microstructure. Microscopy was conducted using a Leica DM4000M optical microscope and a Hitachi S4800 field emission scanning electron microscope.

Tensile Testing

Specimens for monotonic tensile testing were machined from the prismatic blocks perpendicular to the build direction. From each block, five specimen blanks were removed with a center-to-center spacing of 15 mm. The specimens were then machined into cylindrical dogbone geometry with a gauge diameter of 5.6 mm and gage length of 42 mm. Room temperature monotonic tensile experiments were conducted according to ASTM E8-13a [6]. In all cases, the tensile experiments were done in open air conditions and at a strain rate of 0.005 /min. All tensile experiments were conducted on a model 5582 Instron load frame equipped with a 100 kN load cell and an Instron 2620 extensometer.

Results and Discussion

As-built Microstructure

Shown in Figure 7 is the as-built microstructure along the build (XZ) direction. The grain structure consists of elongated grains with their [001] aligned with the build direction. Aligned parallel to the grain boundaries are strings of carbides. Additionally, characteristic of the as-built microstructure is a microstructure gradient that forms as a function of build height. The variation in microstructure results from the extended high temperature (>900 °C) exposure throughout the building process. Representative micrographs taken along the vertical height are shown in Figure 8-10. Based on the characteristics, the gradient can be classified into three regions as described by Kirka et al. [7]:

- Region 1: The last layers of the build solidify in a dendritic manner (Figure 8) with a change in the primary growth direction occurring within the last two layers. Kirka et al. [7] has reported γ'' to have precipitated within the dendritic cores, with carbides and Laves phase precipitating within the interdendritic regions (Figure 11).
- Region 2: An intermediate transition zone, which is characterized by a diffuse dendritic structure

(Figure 9) largely lacking secondary dendrite arms and networks of fine δ needles precipitating at the grain boundaries.

- Region 3: Bulk grain structure (Figure 10) consisting of columnar grains lacking characteristics of dendritic segregation. Networks of δ consisting of both globular and small needle-like δ morphologies have precipitated throughout the interior of the grains.

Figure 7: OM micrograph of the microstructure gradient observed at the top of EBM Alloy 718 block with the three distinct regions discernible [7].

Figure 8: SEM micrograph depicting the dendritic structure in the last two layers of the EBM build [7].

Post-processed Microstructure

Shown in Figure 12 is the resultant microstructure after the HIP and heat-treatment post-processing. Retained was the columnar grain structure despite the super-solvus treatment, which has been reported to lead to significant grain growth in traditionally processed Alloy 718. However, it has also been reported that carbides can pin the grain boundaries in Alloy 718 and suppress grain growth [8]. Further, grain growth during heat-treatment of EBM Alloy 718 can be considered sensitive to the materials thermal history as Unocic et al. [9] reported significant grain growth in their study. Further, the post-process treatment successfully resulted in dissolution of

the δ networks from the interior of the grains and precipitation of the strengthening γ'' precipitates.

Figure 9: SEM micrograph of the diffuse dendritic structure characteristic of Region 2 [7].

Figure 10: SEM micrographs showing spurious δ networks spreading throughout the Alloy 718 grain structure in Region 3 [7].

Figure 11: TEM micrograph of the matrix and interdenritic regions as viewed within the plane of the build in the second to last layer [7].

Figure 12: SEM micrograph of the HIP and heat-treated EBM Alloy 718 with the columnar structure retained.

Tensile Properties

Depicted in Figure 13 are the tensile results for the as-built material along the build direction (XZ) and within the plane of the build (XY) at room temperature. However, as a function of build height, a tensile property gradient was observed within the XY-plane. Both the yield and ultimate stress exhibited a height dependence starting 30 mm below the top of the build. Additionally, the elongation at fracture exhibits the general trends of progressively declining with increasing distance from the top surface of the build. Comparatively, the yield and tensile strengths for the as-built EBM Alloy 718 are both 30 % less on average than wrought Alloy 718 in the peak-aged state [10]. The general progressive decline in the monotonic properties can be attributed to the increasing amounts of precipitated δ at the cost of the strengthening γ'' . Mechanically, the δ phases is largely considered to be detrimental in Alloy 718 due to the brittle nature of the phase [11-14].

Figure 13: Variation in the tensile properties of EBM Alloy 718 in the as-built state within the XY plane. Average XZ properties are denoted by squares with shape corresponding to property.

Shown in Figure 14 are the tensile results for the material in the HIP and heat-treated state. Compared to the as-built material, the property gradient within the XY plane has been drastically reduced as a result of the HIP and heat-treatment. Further, for material tested aligned with the build direction specifications were also meet.

Figure 14: Variation in the tensile properties of HIP and heat-treated EBM Alloy 718 within the XY plane. Average XZ properties are denoted by squares with shapes corresponding to property.

Conclusions

In this work a methodology was established and implemented for the development of material processing parameters for the Ni-base superalloy Alloy 718 in the EBM process through which defect free material was manufactured. Before material processing parameters were developed, the candidate feedstock powder required characterization such that flammability, flowability, and size distribution were within acceptable limits. The process parameter development involved determining the maximum allowable beam current at which smoking does not occur, temperature and time at which the material sufficiently sinters to retain the stainless steel start plate within the powder bed, and acceptable values for the speed function and electron beam focus offset such that defect free material is obtained.

Utilizing, the optimized build parameters a mechanical test sample build was completed. Microstructural analysis revealed that the as-built Alloy 718 material exhibited a largely columnar grain structure with the [001] aligned with the direction of the build. Further, due to the high temperature exposure during the build process, the as-built material was found to exhibit a microstructure gradient as a function of build height. Subsequent post-processing was observed to largely homogenize the microstructure. Mechanical testing of the as-built material at room temperature indicated that the as-built material exhibited a tensile property gradient within the XY-direction. Subsequent heat-treatment was found to homogenize the mechanical properties within the XY-direction.

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