Microstructure and mechanical properties of Hastelloy-X produced by selective laser melting

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Abstract. The microstructure including defects and mechanical properties of as-deposited and heat treated (HT) Hastelloy-X manufactured by selective laser melting (SLM) technology were studied, in order to apply this technique into the manufacturing of gas turbine components. The processing parameters, including scan speed, laser power, scan spacing, and linear energy density were optimized to obtain less porosity. Heat treatment at different temperatures and times was executed to modify the microstructure and reduce the residual stress. When the linear laser energy density increased (>0.12 J/mm), the porosity decreased to ~0.5 %. By decreasing scan spacing to 0.06 mm, the porosity was further decreased to 0.2 %. With increased HT temperature, the microstructure changed from columnar grains through partial recrystallized grains to fully recrystallized coarsened equiaxed grains. Hardness decreased with increasing HT temperature and time in the HT conditions mainly due to grain coarsening and carbide reduction.

1. Introduction

Hastelloy-X alloy, widely used in the aero engines, e.g. combustion components[1], is a Ni-Cr-Fe-Mo solid solution strengthened superalloy, with good oxidation, corrosion resistance and high-temperature strength. It has good forming characteristics and can be forged and cold worked due to its good ductility. However, it takes a long time to make complex parts and it is hard to control the performance using casting or forging for Hastelloy-X alloy.

Among many new manufacturing methods, selective laser melting (SLM) is a rapid additive manufacturing technology which fuses fine metal powder particles into solid parts by high energy laser layer by layer from CAD data. Complex metal parts with nearly full density can be created by this technology in a short time [2-4]. SLM shows great advantage in material saving, process control and part performance compared with casing and forging[5]. Considering the merits of SLM, selective laser melted (SLM) Hastelloy-X could be used to produce the combustor and turbine of an aircraft or gas turbine engine with more complex structure, fewer parts, and less manufacturing time.

Although extensive work has been done on selective laser melted (SLM) nickel-based superalloy in terms of microstructures and mechanical behaviour [6, 7], very few papers have investigated the SLM Hastelloy X metal. Pore was one of the defects, found ubiquitously in the SLM materials and degrading the mechanical properties of the final product [8-10]. Different mechanisms were proposed
to explain the formation of pores in SLM, e.g. due to gas entrapment [11] and melt pool instability and splashing [12]. It was reported that porosity can be controlled by optimizing some parameters, such as laser energy density, but the influence of various parameters on the porosity was not investigated systematically on Hastelloy X [13-15].

In addition, heat treatment was normally carried out to reduce the residual stress induced during SLM with very high cooling rates in the heat affected zone and in the molten pool. The researchers still use the same heat treatment scheme as the forged materials. However, the SLM materials have a very different microstructure with forged material. The microstructure of SLM Hastelloy X comprises of columnar grains with fine parallel dendrites across several adjacent layers [16]. Therefore, it is necessary to investigate heat treatment schemes for SLM Hastelloy materials in order to improve its mechanical properties.

In this work, the porosities of as-SLM Hastelloy X with a series of parameters, (scan speed, laser power, scan spacing and linear energy density) were investigated. Also, the heat treatment scheme including a variety of temperatures and times was carried out to explore the microstructures in HT conditions. Finally, hardness of HT Hastelloy X materials were measured and analyzed based on the microstructure results.

2. Experiment

The Hastelloy-X powder used in the present study was gas atomized and supplied by Guangdong Institute of Materials and Processing with wt.% composition of 0.05~0.15C, 20.5~23.0Cr, 0.5~2.5Co, 0.2~1.0W, 8.0~10.0Mo, ≤0.5Al, ≤0.15Ti, 17~20Fe, ≤0.01B, ≤1.0 Mn, ≤0.025P, ≤0.015S, ≤0.5Cu, bal. Ni. The particle size of the powder is from 0 to 62 μm. An EOS M280 (200W) machine with PSW version 3.7 software has been used to carry out this study, using helium to prevent from oxidation.

The experimental parameters (800~1800 mm/s for scanning speed, 135~195 W for laser power and 60~120 μm for scan space) have been applied systematically in this study. Based on the reference parameters (scan speed=1400 mm/s, laser power=195 W, and scan spacing=0.09 mm), two of three parameters stay the same, and the third one changes, e.g. keeping scan speed=1400 mm/s and laser power=195 W, and then changing scan spacing from 60 to 120 μm. A linear laser energy density $\eta$ (J/mm) was used to represent the energy input per unit length, calculated by dividing the power (P) in Watts, by the scan speed v in mm/s, shown as follows:

$$\eta = \frac{P}{v}.$$

A layer of 20 μm thickness of Hastelloy-X powder was spread on the building platform and a meander laser scan strategy (raster with 67° rotation for each layer) was applied. Building plate was made of mild steel plates with dimensions of 250×250×(25~35) mm provided by TSC Laser Technology Development (Beijing) Co. Ltd. Samples of as-deposit Hastelloy-X with dimension of 5×5×8 mm were fabricated for investigation of microstructure and porosity. Cylindrical samples of 14 mm in diameter and 12 mm in height produced at the reference parameters were cut from the building plate and sliced into 6 pieces along the lengthwise direction for heat treated investigation at different temperatures and times, shown in Table 1, followed by air cooling.

| Table 1. Investigated heat treatment temperature T (°C) and time (h) |
|---|---|---|---|---|---|
| T | 1077 °C | 1127 °C | 1177 °C | 1227 °C |
| t | 1 h | 1 h | 2 h | 1 h |
| | | | | 3 h |

as-deposit and heat-treated samples were sliced perpendicular to z axis (building direction) and parallel to z axis (building direction) to obtain the horizontal and vertical sections. The fresh sections were then mounted, ground, polished to 1 μm water soluble diamond polishing paste finish and etched (3 ml HNO3, 5 ml H2SO4, 90 ml HCl) for optical and SEM examination. The microstructure of as-deposited and heat-treated samples was investigated by optical microscopy (MZ4000) and SEM (JSM-
The porosity volume fractions in vertical and horizontal sections were examined on polished surfaces of microstructure samples. 5 images (10.9 mm² in total) from the middle of polished surfaces were taken for each sample by optical microscopy (MZ 4000). The porosity volume fractions, and pore size were measured on each image using Image J software. Microhardness was carried out on the polished surface by a HV-1000A machine with a load of 0.5 kg. 5 dents were made for each sample.

3. Results and Discussion

3.1. Microstructure of as-deposited samples

Microstructure of as-deposited samples in vertical sections and horizontal sections is shown in Figure 1. Molten pool boundaries can be seen with typical arch-shape curves in vertical sections and lines with different angels in horizontal sections. The microstructures of as-deposited samples are columnar grains with finer primary dendrites (less than 1 μm diameter) along the laser beam direction (z axis) through several adjacent layers, indicated by the black rectangle in Figure 1 (a). The horizontal sample, Figure 1 (d), shows the cross section of the primary dendrites.

3.2. Porosity

The pores in the horizontal section of samples at low laser power 135 W is shown in Figure 2. The samples at high scan speed e.g. 1800 mm/s and scan spacing e.g. 0.12 mm show similar pore distribution and morphology. There are two types of pore morphologies: one type is irregular-shaped with sharp angles and large size regarded as bonding defect with adjacent layers and tracks; another type is small spherical gas pores (generally <20 μm in diameter), shown in Figure 2. Irregular-shaped pores with sharp angles and large size were formed at the melt pool boundaries due to decreased energy input, incomplete re-melting and lack of fusion [17]. This irregular-shape bonding defect disappeared, and small spherical pores dominated with increasing energy input (increasing linear energy density) and decreasing scan spacing at which less powders were melted between adjacent laser scan tracks. The small spherical pores were dispersed over the whole investigated area. Accordingly, in Figure 3, the porosity (volume fraction) decreases with increasing linear energy density (increasing scan speed, decreasing laser power), and decreasing scan spacing. Figure 3 c shows that when the linear energy density η is above 0.12 J/mm, the porosity reaches to the lowest value~ 0.5% and could not be improved. Keeping η constant and further decreasing the scan spacing reduce the porosity to lower value of 0.2%.
Figure 2. Optical images of sample using laser power 135 W. Dark areas are pores.

Figure 3. The porosity (volume fraction) as a function of scan speed (a), laser power (b), laser linear energy density (combining the data in (a) and (b)) (c), and scan spacing (d).

The reason of the formation of large irregular bonding defects could be that when the linear energy density is low, the dynamic viscosity $\mu$ of the molten metal increased [18]. The higher dynamic viscosity $\mu$ impedes the liquid from spreading out smoothly, which leads to bonding defects with adjacent layers and tracks, especially irregular-shaped pores, in Figure 2. Also, the balling may tend to occur when the width of the liquid melt pool is small in order to reduce the surface energy. The shape of the melting pool is mainly dependent on the linear energy input (laser power and scan speed), and increases in dimensions with higher linear energy input [19].

The main reason for the increased porosity, according to previous studies, is the melt pool instability and splashing [17]. The low line energy density (high scan speed and low laser power) can lead to the irregular laser scanned tracks and unstable melt flow [12]. Therefore, increasing line energy density could decrease the porosity by stabilizing the melt pool flow.

The influence of the scan spacing on the melt behavior could be explained by the amount of powder material melted by the laser beam which affect melt pool stability [12]. With smaller scan spacing, less powder materials will be melted by the laser beam with less powder surface area, which decreases the evaporation and Marangoni force, responsible for the melt splashing [12]. At the same time, with less powder particles and less gas in between them, the gas expansion would be less obvious, which further stabilizes the melt flow [17]. The reference parameter with porosity of ~0.5% was adopted for the heat treatment investigation in next section.
3.3. Microstructure of heat treated SLM Hastelloy X

The microstructure of samples after heat treatment at varied temperatures and times were investigated. Samples heat treated at 1177 °C for longer time, e.g. 2 and 3 hours shows similar microstructure with the one at heat treatment condition of 1177°C/1h. The same holds true for the microstructure of samples at HT condition of 1077 °C/1h and 1127 °C/1h. Therefore, three HT conditions, 1127 °C/1h, 1177 °C/1h, 1227 °C/1h show the representative horizontal and vertical microstructures in Figure 4. After HT, the molten pool boundaries disappeared, and primary dendrites dissolved. The heat treatment may remove the interdendritic segregation present in the as-deposited condition and thus lead to the primary dendrite dissolution. At the lower HT temperature of 1127°C, the grains at vertical section are parallel to the building direction, Figure 4 (b). Grain morphologies in horizontal sections, Figure 4 (a), seem to have a bimodal distribution, ribbon-like shapes in the molten pools with a length of about 100 μm, and equiaxed grains situated at the pool boundaries. This is due to impurities mainly concentrated in the boundary between molten pools during the solidification process providing heterogeneous cores for the grains, and then small grains grow up along the boundary.

![Figure 4. Optical images of the microstructure of samples after heat treatment at 1127 °C/1 h (a) and (b), 1177 °C/1 h (c) and (d), and 1227 °C/1 h (e) and (f). Horizontal sections are shown in (a), (c) and (e), whereas vertical sections are shown in (b), (d) and (f).](image)

When increasing heat treatment temperature to 1177 °C and 1227 °C, the microstructure are mainly composed of large equiaxed grains, shown in Figure 4 (c)-(f), which indicate the occurrence of recrystallization. The thermal residual stress is considered to be the driving force of recrystallization for the SLM-fabricated iron part during the annealing process.

At 1177°C, significant grain coarsening and more equiaxed grain shapes are observed and the alignment of the grains in building direction is still present, but evidently reduced. Also, the grains have a feature of zigzag grain boundary which may be caused by pinning of carbides at grain boundaries, shown in Figure 5. The carbides are assumed as M6C according to the Time-temperature-precipitation diagram of Hastelloy-X[1, 20].
At 1227 °C, equiaxed recrystallized grains in both horizontal and vertical sections with more uniform grain size were observed. The grain boundaries are straighter than those at 1177 °C, which could be explained by less carbides at 1227 °C. Thermo-Calc software using database TTNI8 was applied to predict the phase amount versus the temperature at equilibrium condition, Figure 6. It confirms that M6C comes out from 700 to 1400 °C and as temperature increase from ~800 °C, the amount of M6C decreases.

Figure 5. The carbides in the sample after heat treatment at 1177 °C/2h

Figure 6. The phase amount (wt. %) as a function of temperature (°C) in Hastelloy-X predicted by Thermo-Calc.

3.4. Microhardness

Vickers hardness were measured for as-deposited and HT samples in the vertical and horizontal sections. Compared with as-SLM sample, the hardness after heat treatment stays similar ~250 HV at 1027 °C and decreases to ~170 HV at 1227 °C, shown in Figure 7. The hardness is slightly lower with longer heat treatment of 2 and 3 hours at 1177 °C.

After heat treatment, the high level of single dislocations in as-SLM materials could be strongly reduced and thus the residual stress decreased[16]. The residual stress reduction decrease the hardness [21]. Meanwhile the carbides M23C6 and M6C precipitate during heat treatment according to the predicted phase diagram and time-temperature-precipitation diagram. Therefore, residual stress reduction, dendrite and molten pool boundaries dissolution and precipitation strengthening all contribute to the hardness and counteract each other’s effect at 1027 °C, which accounts for no hardness difference between as-SLM material and HT one at 1027 °C.

With increasing HT temperature, recrystallization with grain coarsening occurs, Figure 4. After heat treatment, homogenization has taken place (the primary dendrites disappeared). Also, at higher temperature, amount of M6C decrease as predicted and observed with straighter grain boundaries. Both grain coarsening and precipitate reduction could lead to the decrease of hardness. Therefore, the
sample after heat treatment at 1177°C for 1~3 hour could obtain both good equiaxed grains and suitable hardness.

Figure 7. Vicker hardness of horizontal (H) and vertical (V) samples with a 0.5kg load (HV0.5) in as-deposited condition and in HT conditions at various heat treatment temperatures and times.

4. Conclusions

1) By controlling the processing parameters, the porosity of SLM Hastelloy-X alloy could be optimized. The porosity decreased with increasing linear energy density (increasing scan speed and decreasing laser power) and decreasing scan spacing. When the linear energy density was higher than 0.12 J/mm and the scan distance decreased to 60 μm, the porosity could reach the lowest value of 0.2 %.

2) Heat treatment could be used to tailor the microstructure of SLM Hastelloy-X material. By increasing HT temperature, the microstructure was modified from columnar grains inherited from as-deposited microstructure, through partly recrystallized grains, to fully recrystallized microstructure with coarsened equiaxed grains.

3) The hardness of the HT samples decreased from ~250 HV0.5 (same with hardness of as-deposited sample) to ~170 HV0.5 with increasing HT temperature (1027~1227°C) or time (1~3 h). The optimized heat treatment scheme of 1177°C/1~3h was obtained to get both the equiaxed grain structure and suitable hardness.

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