INVESTIGATION ON MICROSTRUCTURE AND MECHANICAL BEHAVIOUR OF ONE WROUGHT NICKEL BASED SUPERALLOY OBTAINED BY SELECTIVE LASER MELTING PROCESS

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ABSTRACT

Selective laser melting (SLM) technology has been known as a promising Additive Manufacture (AM) technology for components production and prototype design, especially for the hot gas path (HGP) parts with complex geometry feature that serve in aero-engine or land-based gas turbine. But the metallurgical defects caused by complicated solidification process during SLM effect the alloy’s microstructure stability severely, prone to be crack initials and lead to mechanical properties decrease eventually. How to control or eliminate those defects is a critical issue for the actual application of SLMed component.

In this paper, nickel based superalloy HX was selected for SLM process, the related microstructure and mechanical behaviour were analysed on as SLMed and HIPed samples. It is found that pores and un-melting powder are two main kinds of defects in this SLMed alloy, which decrease SLMed HX alloy’s mechanical properties. And then the effects of powder size, energy input, and post-treatments on the microstructure and mechanical property of SLMed samples were investigated. Result shows that higher relative density (which means fewer defects) will be obtained by using smaller size powder and appropriate line energy density $\rho (\rho=P/v, \quad "P" \quad \text{is} \quad \text{laser power input and} \quad \text{"v"} \quad \text{is} \quad \text{laser scanning speed})$. In addition, hot isostatic pressing (HIP) was also proved that can further eliminate metallurgical defects to improve SLMed alloy’s relative density and enhance ductility. Tensile test results shows that HIPed samples have much better room temperature elongation than SLMed ones, which even run up to the level of forged alloy.

INTRODUCTION

The performance upgrade of land-based Gas Turbine is heavily influenced by the material and manufacturing capabilities. Meanwhile, as a free-form process, Additive Manufacturing has been proven to be a prospective technology that can significantly increase design freedom level and reduce lead time & cost for components with complex geometries and small-lot production, such as HGP parts (normally made of nickel or cobalt-based superalloy) used in land-based Gas Turbine.

Selective laser melting (SLM) is one of the typical AM technology where metal powder is melted by a laser source layer by layer, forming a solid, dense component. Compared to other metal-based AM process (such as Laser Metal Deposition) SLM offers higher dimension accuracy and lower surface roughness, which is more suitable for HGP parts fabricating and expected to be widely used in fields of rapid prototyping, manufacturing, repairing or preparing spare parts. However, due to the complicated melting and solidification process, metallurgical defects (pores, inclusions, micro crack) formed easily and residual stress exists in SLMed materials, which lead to mechanical property degrade. How to control or eliminate inner metallurgical defects in SLMed superalloy is a critical problem to be solved for the actual application of SLMed HGP parts.
HX alloy is a common used nickel-based superalloy for Gas Turbine combustor and other HGP parts. As mentioned above, SLMed HX alloy might be widely used in HGP components’ fabricating in the future. In order to find the proper solutions to control the defects and improve properties of SLMed HX alloy, the process parameter, microstructure and related mechanical behaviours had been investigated by researchers. It is found that SLMed HX has better tensile strength property than forged alloy under proper printing process, but with worse tensile ductility (Wang, 2011), and the material’s property will be further improved with energy density optimization (Wang, 2016, Tomus, 2013). Post-treatments’ effect on SLMed HX was also investigated and results showed that precipitates formed during SLM process will be homogenized after hot isostatic pressing (HIP), but the relationship between precipitates and mechanical property did not be implied (Hou, 2016). Post-treatments can also remove the dendrites boundaries and molten pool boundaries, the anisotropy of SLMed HX alloy disappeared accordingly and ductility upgraded (Tomus, 2016). On the other hand, by reduction the content of Mn and Si in HX alloy, the crack initial tendency will also decreased (Harrison, 2015). However, the formation mechanism of other defects and their influence to SLM material’s mechanical property are still needed to be further researched.

For the current study, SLM process parameters (Laser power and scanning speed), powder size and HIP’ effect on SLMed HX alloy’s microstructure are comprehensively investigated and tensile property were compared among SLMed HX alloy, forged HX alloy and post-treated ones, the results will be presented in detail in this paper.

**METHODOLOGY**

Two kinds of pre-alloyed HX spherical powder (Table 1) were adopted for fabricated SLMed samples. Figure 1 shows the particle morphology, and the respective chemical composition of powder A and B can be seen in Table 2. The powder was dried before used for SLM process with temperature 85±5°C and hold for 3h.

**Table 1 Particle size of the powder adopted for SLM**

<table>
<thead>
<tr>
<th>Powder</th>
<th>Particle size range (μm)</th>
<th>D10 (μm)</th>
<th>D50 (μm)</th>
<th>D90 (μm)</th>
<th>Tapping density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>15-53</td>
<td>17.9</td>
<td>36.7</td>
<td>64.4</td>
<td>5.09</td>
</tr>
<tr>
<td>B</td>
<td>10-45</td>
<td>18.0</td>
<td>30.2</td>
<td>51</td>
<td>5.27</td>
</tr>
</tbody>
</table>

*Particle size was measured by Malvern Mastersizer 3000 and tapping density was measured by HY-100D

**Table 2 Chemical composition in wt% of Powder A and B**

<table>
<thead>
<tr>
<th>Element</th>
<th>Cr</th>
<th>Fe</th>
<th>Mo</th>
<th>Co</th>
<th>Mn</th>
<th>Si</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>21.5</td>
<td>18.8</td>
<td>9.0</td>
<td>1.16</td>
<td>0.87</td>
<td>0.83</td>
<td>0.82</td>
</tr>
<tr>
<td>B</td>
<td>21.1</td>
<td>19.4</td>
<td>9.0</td>
<td>1.28</td>
<td>0.96</td>
<td>0.86</td>
<td>0.62</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Element</th>
<th>O</th>
<th>N</th>
<th>C</th>
<th>P</th>
<th>S</th>
<th>Ni</th>
</tr>
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<tbody>
<tr>
<td>A</td>
<td>0.08</td>
<td>0.08</td>
<td>0.06</td>
<td>0.007</td>
<td>0.003</td>
<td>Bal</td>
</tr>
<tr>
<td>B</td>
<td>0.16</td>
<td>0.08</td>
<td>0.03</td>
<td>0.009</td>
<td>0.004</td>
<td>Bal</td>
</tr>
</tbody>
</table>

**Figure 1 Particle morphology of the pre-alloyed atomized powder (a: Powder A; b: Powder B)**

In this study, cylindrical and rectangular samples were fabricated by 3D system ProX200, a commercial SLM equipment with maximum printing size 140mm×140mm×20mm. By adjusting the Laser power P and scanning speed v, the line energy density will maintain in the range of 90~120J/m. And all the samples will be produced above a stainless steel sub plate which need to be polished before printing. Samples were cut off from the sub plate after SLM process by wire-cutting, some were further machined for tensile tests. For some tensile test samples, hot isostatic pressing was performed by using TP530203S HIP furnace, with the parameters 1175°C/150MPa/3h.

Optical microscope (LEICA-DM-6000M) and scanning electrical microscope (TESCAN VEGA3) were adopted for SLMed samples’ microstructure analysis. The etchant used was 30 ml hydrochloric acid, 20 ml acetic acid and 20 ml of nitric acid. For each sample, the relative density is based on the average value of three image’s statistical results.

Tensile tests were carried out using tensile equipment DNS100, the value of Rm, Rp and elongation were obtained based on standard GB/T 228.1.

**RESULTS AND DISCUSSIONS**

**Microstructure**

Optical micrographs of SLMed and HIPed samples are shown in Fig. 2. Molten pool boundaries (MPB) can be observed in SLMed samples with typical arch-shape curves for vertical directions in Fig. 2(a). Columnar grains with several dendrites of uniform crystallographic orientation across multiple MPBs also can be observed in some local amplification areas. MPBs are formed during solidification process because of the segregation of the different positions in the molten pool during different cooling conditions. In the process of laser additive manufacture, the solidification environment of the molten pool is approximately directionally solidified, and the columnar grains grew along the favourable orientation. Because the cooling rate of SLM can reach 105/s or even higher, the solidification structure is fine dendrite. The disappearance of MPBs and dendrites morphology with the change of grain morphology from columnar to equiaxed grains can be observed in HIPed samples (Fig. 2(b)). In the process of HIP treatment, MPBs and metastable dendrites were transformed to the stable microstructure with lower free energy at high temperature and pressure. Meanwhile, the
columnar grains were changed to the equiaxed grains due to recrystallization.

Fig. 2 Microstructure of HX alloy: (a) SLMed; (b) HIPed

In addition to MPBs, epitaxial growth, columnar grains with dendrites, two kinds of defects such as pores and un-molten powders, can be found in SLMed samples. Fig. 3(a) shows the pores with black dots in the optical micrographs. The black dots can be seen at MPBs or in molten pools, which implies the pores distributed randomly. Fig. 3(b) shows un-molten powders with sub-spherical morphology and well-developed secondary dendrites, which are different from matrix microstructure.

Fig. 3 Defects of SLMed HX alloy: (a) pores; (b) Un-molten powder;

Whether pores or un-molten powders will influence HX alloy’s properties severely. The pores formed during solidification due to the high solidification rate in SLM process which limit the gas get out from the molten. While un-molten powders will appear in some regions where energy input is not enough for melting during SLM. According to these defects formation mechanism, relevant research on controlling defects will be one key step of the application of SLMed HX alloy. As inner metallurgical defects, pores and un-molten powder decrease SLMed alloy’s relative density, and prone to be crack initials, reduce alloy’s ductility eventually.

Defects Control

Energy input

In this paper, laser power (P) and scan speed (V) were the only two experimental variables, while other variables such laser beam diameter, layer thickness and incident angle were fixed at constant values. Through the model of line energy density (Sun, 2005) \( \rho = P/V \), laser power and scan speed are combined into one variable, which represents energy input in the experiments.

The relative density of SLMed samples fabricated by Powder A (15-53μm) powder is shown in Fig. 4. Through Image-J software, the relative density is obtained by the binarization processing and differential statistics of the optical micrographs of polished SLMed samples (pores and un-molten powders are black, while matrix are bright). In a word, the relative density of SLMed samples increase along with the increase of line energy density (Fig. 4). The relative density of SLMed samples with a few dark dots is 95.2% when \( \rho \) value increase to 90 J/m, while the relative density reach to 99.2% when \( \rho \) value run up to 118 J/m. When \( \rho \) is higher than 118 J/m, the relative density of SLMed samples appear to be unchanged, which implies the defects were well controlled.

Fig. 4 Relative density of SLMed samples fabricated by Powder A (15-53μm)

Powder size

Powder size is one of the most important character of metal powder. In this paper, two kinds of powders, Powder A (10-45μm size) and Powder B (15-53μm size) were selected for comparative experiments, the distribution curves of powder size are shown in Fig. 5. There are mainly two differences between these powders. Firstly, the D50 of 10-45 size powder is 30.2 μm, while the D50 of 15-53 size powder is 36.7 μm. Secondly, the particle size distributions of these powders were the range of 18μm-51μm and 18μm-64μm, respectively. Obviously, the 10-45 size powder is more fine and concentrated.

The relative density trend diagram of SLMed samples fabricated by 15-53 and 10-45 size powders are shown in Fig. 6. When the value of \( \rho \) is 90 J/m, the relative density of SLMed samples fabricated by 10-45 size powders reach to 97.5% which is 3% higher than that of 15-53 size powders. When the relative density of SLMed samples fabricated by 10-45 size powders reach to 99%, the \( \rho \) is up to 95 J/m, which is 25 J/m
lower than that of 15-53 size powders. Although the relative densities of two powders increase, the difference between them is smaller with the increase of ρ value. The relative density of 10-45 size powder is higher than that of 15-53 size powder at same ρ value between 90 to 125 J/m.

Fig. 5 Distribution curves of powder size

Fig. 6 Comparison of relative density

Because of the compaction by roller during paving process, tap density is used for the quantitative analysis of monolayer powders. In Table 2, the tap density of 10-45 size powder is 5.27 g/cm³, which is higher than that of 15-53 size powder. Because of the relative density of SLMed samples fabricated by 10-45 size powders, it is assumed that the relative density of SLMed samples is related to the tap density of powders. In addition, the size range of 15-53 size powder is larger than that of 10-45 size powder, for which the possibility of defects is greater when used the former powders with same energy input since the melting time and melting degree of powders with different particle sizes are different.

Hot isostatic pressing

Fig. 7 Tensile properties of HX alloy samples at room temperature

Fracture surfaces of HX alloy after room temperature tensile are shown in fig. 8. Compared with HIPed samples in fig. 8(b), the fracture surface of SLMed samples is flat in fig. 8(a), which indicates that the former absorbed more damage energy before fracture. The fracture surface of SLMed samples has less dimples with shallower morphology (fig. 8(c)), while the fracture surface of HIPed samples has more dimples with deeper morphology, implying that the latter has a greater plastic deformation before fracture. The yield strength, ultimate stress and elongation of HIPed samples were 572 MPa, 636 MPa and 46%, respectively. As a conclusion, the tensile properties of HIP samples are better.

The yield strength and tensile strength of SLMed samples are higher because of the metastable structure like MPBs and dendrites. In the process of deformation, those substructure can effectively impede dislocation movement and increase the difficulty of materials deformation. However the local stress concentration at those substructure will lead to crack initiation and low elongation because of the higher dislocation density. The elongation of HIPed samples is higher because the MPBs, dendrite boundaries and even some pores are eliminated and the local stress concentration is effectively alleviated. In addition, the equiaxed grain can better release the coordination deformation between grains.

Fig. 8 Fracture surfaces of HX alloy after room temperature tensile: (a), (c) SLMed; (b), (d) HIPed
CONCLUSIONS

Result shows that molten pool boundaries, epitaxial growth, columnar grains with dendrites are typical microstructure for SLMed HX alloys, while pores and un-molten powders are two common kinds of defects due to the high solidification rate and local insufficient energy input during SLM process. By increasing line energy density and select more fine and concentrated powder can significantly control or eliminate those two kinds of defect: For SLMed samples fabricated by 10-53μm size powder, the relative density reach to 99.2% when line energy density is up to 118 J/m. And the relative density could reach to 99% when line energy density is only 95 J/m by changing the powders from 15-53 to 10-45 size. In addition, the ductility of SLMed HX alloy can be enhanced by HIP effectively, test results show that elongation of HIPed samples at room temperature almost twice higher than SLMed ones, even reach to the level of the forged alloy standard.

NOMENCLATURE

AM       Additive Manufacturing
SLM       Selective Laser Melting
HIP       Hot Isostatic Pressing
P          Laser power in SLM process (W)
v          Laser scanning speed in SLM process(m/s)
ρ          Laser line energy density (J/m)
R_p0.2     Yield Stress in MPa
R_m        Ultimate tensile stress in MPa
A          Elongation (%)

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