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MICRO-CRACK FORMATION AND CONTROLLING OF INCONEL625 PARTS FABRICATED BY SELECTIVE LASER MELTING

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Abstract

Micro-crack is one of the most serious defects in selective laser melting (SLM), which impair the mechanical properties of the fabricated parts. In this study, Inconel625 superalloy specimens were fabricated by SLM process with progressive alternative scan strategy. The morphology of the cracks, elements distribution were detected by optical microscope (OM), scanning electron microscope (SEM) and electron back scattered diffraction (EBSD). The results showed that a large numbers of micro-cracks occurred at room temperature, with the average length of approximately 100 μm. It was found that crack formation was attribute to the local segregation of Nb and Mo element in the process of rapid solidification, resulting in the generation of low melting temperature eutectic solidification (γ+Laves). Micro-cracks grows along the interface of (γ+Laves) under the thermal stress. Base-plate preheating shows an efficient method to reduce the scales and number of cracks. The residual stress was reduced by more than 50% when preheating at 300℃.

1.Introduction

Alloy 625 is a nonmagnetic, corrosion and oxidation-resistant, nickel-based alloy. Its outstanding strength and toughness in the temperature range cryogenic to 1093℃ are derived primarily from the solid solution effects of the refractory metals, columbium and molybdenum, in a nickel-chromium matrix [1, 2]. The alloy has excellent fatigue strength and stress-corrosion cracking resistance to chloride ions. Some typical applications for alloy 625 have included heat shields, furnace hardware, gas turbine engine ducting, combustion liners and spray bars, chemical plant hardware, and special seawater applications[3, 4]. Selective Laser Melting (SLM) is an additive manufacturing technique that can print metal parts in 3D. A laser is used to melt metallic powder in specific places[5]. The laser will heat particles in specified places on a bed of metallic powder until completely melted. The CAD 3D file dictates where melting will occur. Then, the machine will successively add another bed of powder above the melted layer, until the object is completely finished[6]. It shows unique advantages in material saving, process control and parts performance. At present, extensive work has been conducted on producing nickel based superalloy parts. Yadroitsev studied the influence of different scanning interval and scanning strategies on Inconel625 powders fabricated porous components. It was identified that the tensile strength of Inconel625 is much higher than Forgings Standard Level[7, 8]. However, the elongation is much lower compare to the traditional process because of the formation of micro-cracks inside the parts. No further discussions about the phonemes were made in these papers. Micro-cracks were also found in another nickel-based alloy Waspaloy[9], by adjusting the processing parameters, the cracks were greatly suppressed, but not completely eliminated. According to the papers published by Wang, hot isostatic pressing (HIP) could effectively eliminate the micro-cracks, pores and other defects which the greatly improved properties[10]. Although the post treatment HIP was effective, the cost was too high even for additive manufacturing process. Researchers M.Shiomi and K.Osakada from Japan proved that the residual stress could be reduced by preheating the powder when used SLM to build SCM440 steel alloy. When the preheating temperature reached to 250℃, the residual stress could be reduced by almost half, effectively inhibit the generation of cracks[11]. Numbers of Nb, Mo and other strengthening elements are added to the nickel-based superalloy which contribute to a great tendency to form micro-cracks. Meanwhile, the distribution characters, formation mechanism, morphology of the cracks were not deeply analysis in the current literature. No mature theories and methods were come up to explain and control the formation of micro-cracks for nickel-based superalloy.

In this study, SLM process was used to fabricate Inconel 625 superalloy parts to explore the characters of micro-cracks and try to apply preheating to eliminate the cracks. The morphology of the cracks, elements distribution were detected by optical microscope (OM), scanning electron microscope (SEM) and electron back scattered diffraction (EBSD). A theory was come up to explain the formation of micro-cracks inside the parts and the residual stress under different preheat temperatures were explored to prove our theory and inhibition the cracks.

2. Experimental

Inconel 625 powder (H.C. Starck GmbH, Germany) produced by gas atomized process with the average particle size of 34.63μm was used. The distribution of the powder is tested by Particle Size Analysis (MALVERN，Master Mini). Figure 1(a) illustrates the SEM morphology of the powder. The morphology of the powder is approximately spherical with smooth surface. The powder is composed of 53.5 Ni, 21.5 Cr, 0.96 Fe, 3.71 Nb, 8.8 Mo, 0.47 Mn, 0.41 Si in wt%. The powder was heated at the temperature of 50℃for 5 hours prior to the SLM process. This procedure was to eliminate the water vapor inside the powder and get a good flow ability.

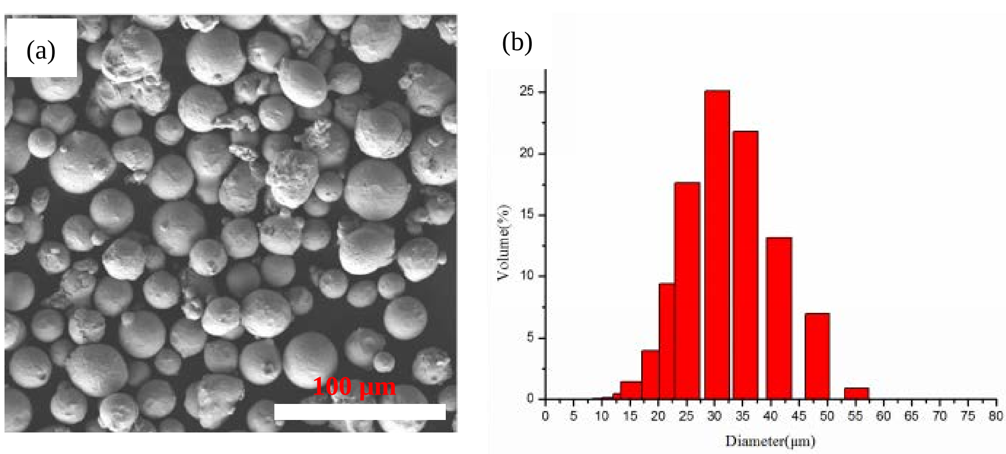


Fig.1. (a) SEM image shows the characteristic morphology of Inconel625 powder. (b) the distribution of the particle size.

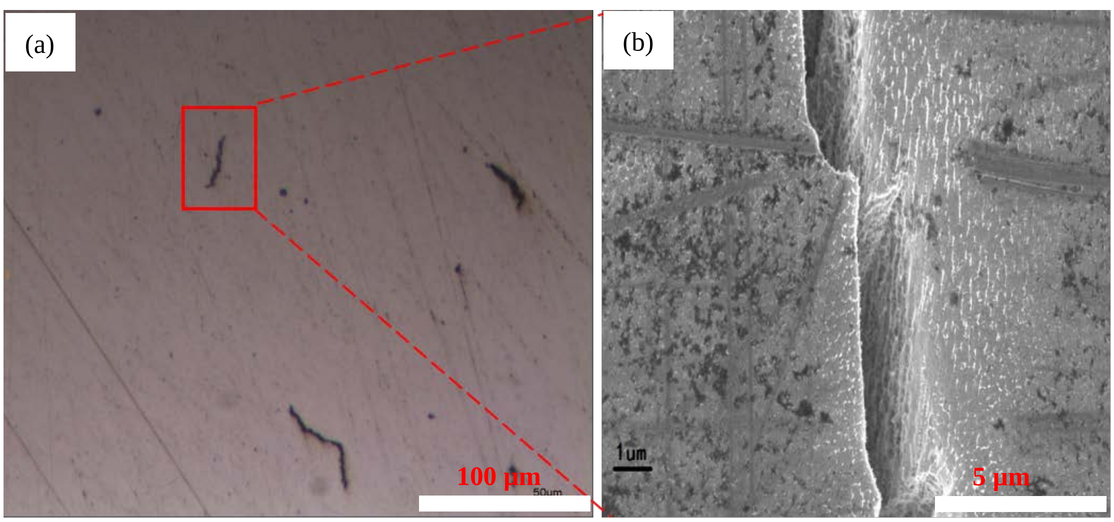
The specimen were fabricated on the HRPM-II machine developed by Huazhong University of Science and Technology (Rapid Manufacturing Center). A 200W fiber laser was equip on The machine of which the laser spot is 50-80μm in diameter and scanning speed ranges from 200 to 1000mm/s. Firstly the build chamber is vacuumed followed by filling in a high-purity argon gas to form an oxygen-free atmosphere. The optimized processing parameters obtained by our group were adopted for manufacturing of almost fully dense Inconel 625 parts, which are the laser power of 150W, the scan speed of 400mm/s, the hatch space of 0.07mm, and the layer thickness of 0.02mm. The base-plate was made of 316L stainless steel. The base-plates were preheat to 25℃(room temperature), 150℃ and 300℃,respectively, pier to the building process.

The samples were then subject to mechanical polishing, using a grit size of 1 μm. Subsequently, the polished samples were etched for 30s in a mixture comprising solution with 10ml HNO_3, 10ml HCL and 15ml CH_3COOH to vision the microstructure. The distribution characteristics of the elements were analyzed by the Energy Dispersive Spectrometer (EDS). The grain orientation, grain size and morphology of the micro-cracks were determined using the EBSD system mounted on the Scanning Electron Microscopy machine (JEOL 7600F). For this purpose mechanically pre-polished samples were electro polished for 20 s under 20 V in a 5% perchloric acid solution. The data was analyzed using HKL Channel 5. The residual stress was measured under different forming conditions (within 48 h) using X-ray stress analyzer (X-350A, Esther).

3.Results and discussions.

The morphology characters of the micro-cracks under OM can be seen in Figure 2a. It is found that the length of cracks is about 100 microns with width not exceeding 5 microns which belonged to the category of micro-cracks[12]. Such cracks have little effect to the tensile mechanical properties in room temperature, However, it shows big influence to the creep strength, fatigue resistance, as it often become sources of defects, resulting in a serious decline in performance for SLM fabricated parts[2].

Figure 2b demonstrated the characters of micro-cracks under high magnification. It can be seen that a large number of long white substances precipitate at the boarder crack. The section of crack is smooth, without obvious torn tissue. This is caused by the separation of liquid film under high temperatures which often be found for thermal cracking[13]. EDS was employed to analysis the white strip on both sides of the crack, the results were present in Figure 2 (c). At point 2, the gray area is substantially free of Nb, however, at Point 1 (white boarder) the contents of Nb, Mo elements is much higher than Point 2. According to the EDS results, it can be inferred that the white ares are a kind of Laves phase (Ni, Fe, Cr)$_2$(Nb, Mo)[12]. This phase is not stable, usually appeares under rapid solidification conditions. Although Inconel 625 is a solid solution strengthening superalloy. Precipitated does not play a key role on the mechanical properties[14]. Many studies indicated that Inconel 625 also exhibit certain properties of precipitation. The second phase precipitation strengthens the elastic modulus, ultimate strength and decreases the plasticity of the alloy.



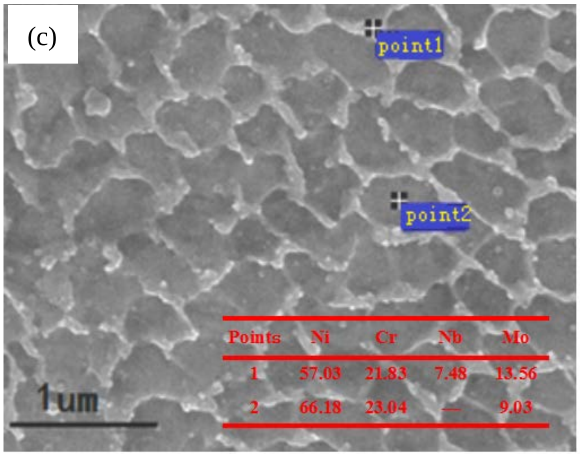


Fig.2. (a) Micro-cracks of Inconel 625 under OM image (b) morphology characterizes of micro-cracks under SEM. (c) The EDX of the white strip on both side of the cracks, the main elements of point 1 and point 2 are show in the table.

During the rapid solidification process of SLM, the path from melting pool to the solid can be described as follows[15]:

$$L \rightarrow L+\gamma \rightarrow L+\gamma+NbC+Laves \rightarrow \gamma+NbC+Laves$$

Which L means liquid.

For Inconel 625 alloy, when the powder is melted, Nb, Mo elements has a strong tendency to segregation in front of the crystallization[15]. Nb and Mo atoms continuously move by diffusion from the coagulation region to liquid region. In a micro area, the composition of melted Inconel 625 has a great distinction with the original one which accelerated the process of solidifying path. It is easy to form the Laves phase and NbC phase inside the parts. Due to short cooling time, the carbide NbC has limited time to grow. On the other hand, the superalloy has a very low content of C element. According to the solidifiction path, less NbC also contribute to the formation of Laves. For SLM process, the solidfication path also can be short to

$$L \rightarrow \gamma+Laves+NbC\ (Trace)$$

Studies have shown that, the reaction of eutectic (γ+Laves) occurs at 1157℃[16]. Such a low melting point eutectic provides internal condition for the formation of microcracks. Meanwhile Laves phase is a kind of brittle hcp metal compound which greatly reduces the elongation of the alloy at room temperature. It can hardly bear any deformation. When the content of Laves phase exceeds 2% -3%, it will greatly weaken the mechanical properties. Weak performance area will be formed around the Lave phase. Researchers Mercelis and Kruth pointed that the temperature gradient mechanism (TGM) leads to residual stress attributed to the layer by layer process[17]. Temperature gradient leads to the generation of residual stress, which is the external reason for the cracks. Due to the residual stress caused by the rapid solidification, it is likely to form stress concentration around the strip eutectic (γ+Laves) at high temperature. After the formation of cracks, residual thermal stress has been released, the crack stop the expansion. Meanwhile, the alloy is cooling rapidly, the rapid rise in the ultimate tensile strength, also prevents further extension of the crack. Therefore, the overall scale of the crack is very small, less than 100 um.

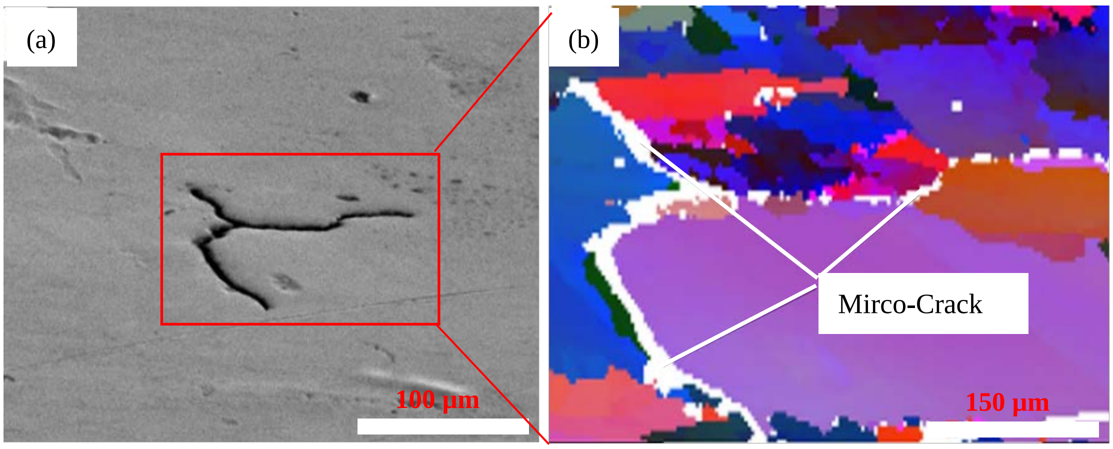


Fig.3. (a) The crack morphology of the SLM formed Inconel625 superalloy. (b) Inverse pole figure (IPF) colored map of the grain around the crack.

The micro-crack was also detected by EBSD which was shown in Figure 3. The white striped zone was the micro-crack. In the inverse pole figure , different color represents different grain[5]. From the image, it is clear to see the cracks started from the grain boundary and extend along it. Because the grain boundary is the severe area of the segregation, a large amount of Nb, Mo and other elements enriched at the grain boundaries. The tendency to form low temperature eutectic is largest contribution to the formation of cracks.

3.2. The influence of preheating on the micro-crack

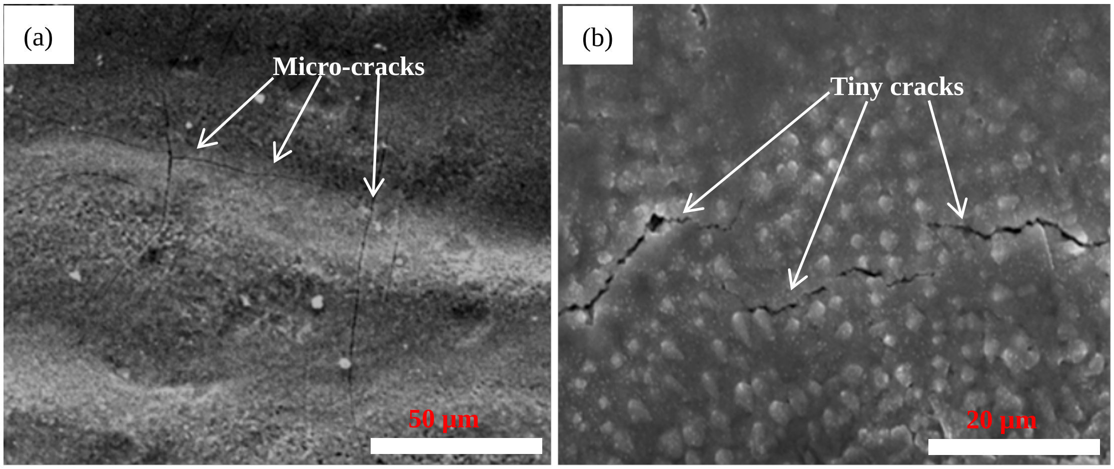


Fig.3. (a) The crack morphology of the SLM formed Inconel625 superalloy under different preheating temperature: (a) micro-cracks when preheat at 150 ℃; (b) Tiny cracks appear at 300 ℃

Figure 4 (a), (b) show the internal crack morphology of samples preheat 150℃ and 300℃. In the preheating 150℃ conditions, certain amount of cracks are still exist, although the crack length is relatively shorter than room temperature. No obvious cracks are found at when preheat at 300℃ at low magnification. When observe at high-mag, tiny cracks which is short than 10 microns can be observed. Those tiny cracks are separate and no connection between them which have much less harmful to the mechanical properties compared to no preheating condition. Vasinonta pointed that the residual stress decrease as the thermal gradient become lower[18]. The residual stress under different preheating temperatures were 396±23Mpa for room temperature, 356±4Mpa for 150℃ preheating temperature, 160±25Mpa for 300℃ preheating temperature. Every part was test three times to obtain the average value. The results show that the thermal residual stresses are tensile stress. Under room temperature conditions, the stress can reach (396±23) MPa. As the preheating temperature increases, the residual stress decreased. When the preheating temperature reached 300℃, the residual stress is reduced by more than 50% ((160 ± 25) MPa). The reducing of stress contribute to the lack of sufficient external energy to form and expand the cracks. Thus, only tiny cracks occur when preheat at 300℃. For

Inconel625 superalloy, when the preheating temperature reaches 300℃, the residual stress is greatly reduced and crack formation and expansion are suppressed.

4.Conclusion

In this study, SLM process was used to fabricate Inconel 625 superalloy parts to explore the formation of micro-cracks and applied preheating to eliminate the cracks. The main found can be summarized as follows:

(1) High density Inconel625 superalloy part can be obtain using SLM process under certain parameters, however, micro-cracks also can be found inside the part. The length is not more than 100 microns, severely impair the mechanical properties of the alloy.

(2) The internal reason for the crack is the low melting point eutectic (γ+Laves) which caused by the segregation of Nb, Mo in front of the crystallization. At the same time, stress concentrates around the brittle Laves phase, resulting in solidification cracking along the grain boundaries. The external reason is thermal stress caused by the non-uniform temperature gradient which provides the energy for the cracking.

(3) By preheating the substrate, the thermal residual stress has been reduced. When the preheating temperature reached 300℃, the residual stress is reduced by more than 50% (160 ± 25) MPa. The number and the length of crack significantly reduced, compared to the previous length. Preheating is an effective way to inhibition the micro-crack when fabricate Inconel 625superalloy.

5.References

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