Effect of aging treatment on corrosion resistance of Inconel 718 fabricated by selective laser melting

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Abstract. The microstructure and corrosion resistance of Inconel 718 alloy made by selective laser melting (SLM) by aging heat treatment (AT) were studied. After the solution treatment at 1050°C, the aging treatment temperature ranges from 600°C to 800°C. Scanning electron microscopy showed that γ′ was precipitated out of the γ austenite matrix, with σ decrease in delta phase. The morphology, distribution and formation mechanism of precipitates in these microstructures were analyzed and discussed. Open circuit voltage curve (OCPT), alternating current impedance spectrum (IMP) and TAFEL curve (TAFEL) were tested by electrochemical workstation. Electrochemical corrosion results and corrosion resistance of samples after four different heat treatments were analyzed, and the best aging heat treatment mode and multistage aging treatment were discussed.

1. Introduction

SLM manufacturing technology can make up for the defects of traditional manufacturing process in high-tech fields such as aerospace, which are difficult to produce or have substandard performance, and has gradually become one of the mainstream development directions of additive manufacturing[1]. High quality metal powder materials has been avtechnical problem that selective laser melting technology development needs to overcome, and nickel based high temperature alloy has the high temperature strength, oxidation resistance, corrosion resistance and other high temperature under the condition of the use of the advantages. Inconel 718 nickel-based alloy since its launch has experienced more than 50 years of continuous improvement, already mature application in the aerospace, petrochemical and other fields [2], and is good raw materials of selective laser melting.

Although SLM has many advantages, the additive manufacturing technology is in the development stage and the technology is not mature. There are still many defects in the parts produced by SLM, such as incomplete powder melting, poor metallurgical bonding and hole formation [3]. The heat treatment process of Inconel 718 alloy need high requirements, and its microstructure is greatly affected by heat treatment. Different heat treatments will cause huge differences in morphology and quantity of strengthened phase and precipitated phase [4], resulting in differences in mechanical properties. The
immature selective laser melting manufacturing technology may aggravate these defects. The solution treatment + aging treatment is the most common heat treatment method for strengthening Inconel 718 alloy. Among them, the solution treatment is the heat treatment of Inconel 718 at 900℃~2000℃ for 1h~2h. During the experiment, the time and temperature can be adjusted and matched. Aging treatment is to conduct heat treatment for 4h~8h at 600℃~800℃. Similarly, three data curves are selected for comparison, and the experimental results are tested by an electrochemical workstation. The parts are easy to be oxidized and corroded under high temperature and high strength working conditions, so it is necessary to study the corrosion resistance of Inconel 718.

The research on Inconel 718 alloy started late in China [5]. There are few studies on the corrosion properties of Inconel 718 alloy materials manufactured by additive at home and abroad. Therefore, this paper chooses to study the influence of aging treatment on the corrosion resistance of Inconel 718 alloy prepared by selective laser melting, compares the heat treatment results, and explores the corrosion resistance rules of Inconel 718 alloy.

2. Experimental procedures

2.1. Material
The wire cutting machine was used to divide Inconel 718 alloy (ingredients as shown in table 1) into four rectangular blocks of 10 mm× 45 mm× 30 mm, and then the long strip material was cut into four pentahedral blocks with side length of 50 mm, 50 mm, 70 mm and height of 30 mm.

| Table 1. Three Scheme comparing. |
|------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| Fe | Cr | Nb | Mo | Ti | Al | C | B | Ni |
| 18.50 | 18.00 | 5.30 | 3.00 | 0.95 | 0.50 | 0.003 | 0.003 | Balance |

2.2. Heat Treatment
The box type resistance furnace of gwl-1400℃ manufactured by guoju experimental electric furnace factory in luoyang was used for heat treatment. The following four groups of samples are heat-treated with 300℃/h of heating rate according to table 2 respectively.

| Table 2. Heat treatment |
|-------------|
| Number |
| A | 1050℃×1h, AC |
| B | 1050℃×1h+620℃×8h, AC |
| C | 1050℃×1h+620℃×8h+720℃×8h, AC |
| D | 1050℃×1h+820℃×8h, AC |

2.3. Characterization Analysis
The microstructure of metallographic surface was observed by optical electron microscope. Scanning electron microscope (SEM) was used to analyze the original and aging microstructure of the alloy.

2.4. Electrochemical testing
Firstly, NaCl powder and water were used to prepare 3.5wt% sodium chloride solution as electrolyte, and electromagnetic mixer was used to mix it evenly. Then platinum electrode, mercury electrode and sample electrode were placed on the same plane and inserted into the foam plate to put the electrolyte. Finally, Shanghai chenhua CHI660E electrochemical workstation was used for testing. During the test, the initial potential OCPT of four samples obtained from 300s was measured after the point position was stabilized for a while, and the initial potential median value was set to obtain IMP and TAFEL curves.
3. Results and Discussion
Electron microscope images of the microstructure and delta phase morphology of Inconel718 alloy after solution heat treatment and aging heat treatment at three different temperatures are shown in Fig.1 (a), (b), (c) and (d).

![Electron microscope images of the microstructure and delta phase morphology](image)

**Figure 1.** Metallographic microscope ×50 times (100μm)

After three heat treatment processes, the grain size of Inconel718 alloy is not significantly different from that of the solid solution treatment. It can be found from the observation of metallography that the only solid solution treatment Inconel718 alloy is mainly composed of γ-phase matrix and a small amount of carbides, while the size of other phases is too small to be observed [6]. After solid solution and aging treatment, many spherical γ′phase, disc γ″phase, rod σ phase and a large amount of carbides were separated from the dendritic γ matrix, and the strengthening γ′ phase and γ″ phase were the main factors to improve the hardness of the materials. γ″phase appeared in large quantities after heat treatment, and γ″ was the main strengthening phase of the materials after heat treatment, which was a kind of metastable phase, appearing as a face-centered cubic phase and dispersing out in the shape of a ball, which played a part in strengthening the alloy[7]. γ″ phase is metastable phase, during long-term use, γ″ phase tends to be σ phase.

### Table 3. Dynamic potential scanning parameters

| Electrochemical parameter | Ecorr (V) | Icorr (μA·cm⁻²) | Rp (KΩ·cm⁻²) |
|---------------------------|-----------|-----------------|--------------|
| A                         | -0.542    | 2.996           | 180.908      |
| B                         | -0.310    | 0.387           | 1400.517     |
| C                         | -0.310    | 0.3156          | 1717.364     |
| D                         | -0.160    | 0.17248         | 3142.393     |
The curve was fitted by Tafel epitaxial method, and the polarization parameters were obtained. $I_{corr}$ was the self-corrosion current density of the alloy, and $E_{corr}$ was the self-corrosion potential of the alloy. The corrosion rate was the thickness of the alloy corrosion every year, and the corrosion rate was proportional to the self-corrosion current density. From the perspective of corrosion dynamics, the higher the density of self-corrosion current, the faster the corrosion rate and the worse the corrosion resistance of the alloy. Alloy treated at 820 $^\circ$C had the smallest $I_{corr}$, followed by dual aging treatment at 720 $^\circ$C, and only solid solution treatment had the largest $I_{corr}$. The self-corrosion current density of alloys of different states varies from large to small: solid solution > 620$^\circ$C aging > 720$^\circ$C dual aging > 820$^\circ$C aging.

The main factors affecting the corrosion performance of materials include chemical composition and organizational structure. The corrosion resistance of Inconel 718 alloy fabricated by SLM may be affected by the porosity defects in SLM parent material samples [8]. The strengthening methods of nickel-based superalloy mainly include solution strengthening, precipitation strengthening and grain boundary strengthening. The main strengthening phase in Inconel 718 alloy is $\gamma$ phase (Ni3Nb), and the stability of $\gamma$ phase directly affects the properties of the alloy. $\gamma$ phase $\rightarrow$ $\gamma'$ phase $\rightarrow$ $\gamma''$ phase $\rightarrow$ $\sigma$ phase transition occurs during solid solution treatment. $\sigma$ phase precipitation of Inconel 718 alloy after solid solution treatment reduced the concentration of Nb element in $\gamma$ phase which could be used to precipitate Inconel 718 alloy during aging, and the $\gamma$ in grain decreased correspondingly[9], and the volume fraction of alloy strengthened phase decreased. Characteristic curves such as self - corrosion potential - corrosion time, ac impedance spectrum and tafell polarization curve are usually used to characterize the corrosion resistance of materials. The self-corrosion potential represents the corrosion tendency of the material in the corrosion solution, and the more positive the self-corrosion potential is, the weaker the corrosion tendency is[10]. When the electrode system is a sine wave voltage (current) communication signal disturbance, it produces a corresponding response signal current (voltage), and these signals can be obtained by the impedance of the electrode or admittance. The impedance spectrum generated by the sinusoidal signal of a series of frequencies is called electrochemical impedance spectrum. The greater the arc radius of the electrochemical impedance spectrum, the stronger the corrosion resistance of the material. The tuffell curve is a section of a polarization curve in which the electrode potential is changed in order to achieve a certain current, TAFEL is obtained by logarithm of current density and overpotential plot. The self-corrosion current density represents the corrosion resistance of the sample in the corrosion solution. The lower the self-corrosion current density is, the stronger the corrosion resistance of the material will be [11].

4. Conclusion
The conclusions are as follows:

(1)After 720$^\circ$C dual aging and 820$^\circ$C high-temperature aging heat treatment, $\sigma$ phase will be precipitated in the grain boundary in the form of short bars or grains, and 820$^\circ$C high-temperature aging treatment is conducive to the transformation of the metastable $\gamma''$ phase to stable $\sigma$ phase, and more $\sigma$ phase will be precipitated in the grain and grain boundary than 720$^\circ$C dual aging treatment.

(2)Since the corrosion current density changes from large to small, it is solid solution > 620$^\circ$C aging >720$^\circ$C dual aging >820$^\circ$C aging and the order of corrosion resistance tendency is 820$^\circ$C aging >720$^\circ$C dual aging >620$^\circ$C aging > solid solution. Therefore, 820$^\circ$C high-temperature aging alloy has the best corrosion resistance, while only the alloy treated with solid solution has the worst corrosion resistance.

(3)The high-temperature aging heat treatment temperature of 820$^\circ$C is higher than the dual aging temperature of 720$^\circ$C, which increases the number and size of $\sigma$ phase. The corrosion resistance of samples treated with solid solution + aging is better than that treated with solid solution only. The fractional aging has little impact on the corrosion resistance of the alloy, and the corrosion resistance of samples treated with high aging is better.
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