Effect of cooling methods on mechanical and corrosion properties of Inconel 625 during solution treatment

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Abstract. Inconel 625 alloy is a nickel-based deformation superalloy which is widely used in aviation, ocean engineering, chemical engineering and other important fields. In recent years, with the continuous expansion in application field of Inconel 625 alloy, the requirements of mechanical properties, corrosion resistance and other comprehensive properties of the alloy are constantly improved. In this paper, hardness tester, tensile machine, electrochemical workstation, optical microscope, scanning electron microscope and other equipment are used to study Inconel 625 alloy, and the microstructure evolution law and mechanical properties and corrosion properties of the alloy under different heat treatment conditions are studied. The experimental results show that the as-cast Inconel 625 alloy is mainly composed of austenite which is displayed high hardness and poor corrosion resistance. Heat treatment is a common way for tuning the comprehensive properties of Inconel 625 alloy. After 1090°C heat treatment, different cooling method were used to tune the corrosion resistance of Inconel 625 alloy. The cooling rate has a great influence on the microstructure and properties of the alloy. After water cooling, the precipitation of Nb carbide at the grain boundary is retained, which improves the corrosion performance of the alloy. After air cooling or furnace cooling, chromium carbide precipitates in the alloy again, which leads to the decrease of corrosion properties of the alloy. The special treatment can effectively improve the corrosion resistance of the alloy, and improve the plasticity of the material without reducing the strength. This paper provides an effective basis for optimizing the heat treatment process of Inconel 625 alloy in production to improve the comprehensive properties of the material.

1. Introduction

Inconel 625 is a solution enhanced nickel-based heat-resisting alloy with extensive oxidation and corrosion resistance properties, which has been widely used in gas turbine and jet engines with service temperatures up to 923 K[¹]. The strength of 625 alloy is due to the solution strengthening effect of trace elements such as Mo and Ni contained in Ni-Cr alloy[²-⁴]. The Cr and Mo elements in the alloy make it resistant to chloride pitting, and the high Ni content enhances the resistance of the alloy to chloride stress corrosion cracking (SCC), so the alloy has excellent corrosion resistance[⁵,⁶]. In recent years, as a bellows material, it is mainly used in important pipelines with high temperature and highly corrosive media[⁷,⁸].
The properties of superalloys are mainly determined by their chemical composition and microstructure. When the composition of the alloy is constant, the factors that affect the microstructure of the alloy include melting process, casting process, hot working method and heat treatment process\cite{9}. Heat treatment process is the most sensitive to the evolution law of alloy microstructure. The variation of heat treatment process can change the grain size and grain boundary state of alloy through precipitation or dissolution of hardening phase\cite{10,11}. Wang Z J\cite{12} conducted solution treatment on 690 nickel-based alloy and found that after solution treatment at 1050°C−1150°C, the microstructure of all samples was fully recrystallization equiaxed grain, during solution treatment, the grain size increased uniformly from 12 to 29 μm. The effect of NiNb precipitation on the corrosion behavior of Inconel625 alloy in oxidizing environment during aging was studied by Tawancyl H M\cite{13}, results that after aging at 650°C, The corrosion rate of the alloy increases continuously, at 760−870°C, the corrosion rate tends to decrease, the reason is that at 760−870°C, the flake NiNb changes into lumpy and the content gradually increases. Liu D X\cite{14} studied the effects of the heating process on the microstructure and properties of inconel 625 alloy, the results showed that the properties of Inconel 625 alloy could be improved through the heating process concomitant with a reduced corrosion rate. It was incredibly significant to control the microstructure during various steps of solution treatments, in order to obtain a combination of favourable mechanical properties and corrosion resistance.

In this paper, Inconel 625 alloy was treated by 5 different solution treatment, the evolution of microstructure and the improvement of properties during the heat process were analyzed emphatically. This work aims to demonstrate the influence of the solution heating process on microstructure and properties of Inconel 625 alloy, which is conducive to the actual production.

2. Experiment

2.1. Experimental material and technology
The test material used in this study was Inconel 625 cold-rolled plate with a thickness of 2.0 mm. The composition is shown in Table 1. Five different heat treatment processes were used in the experiment as follows: (1) heat preservation for 12min at 1090°C, furnace cooling; (2) heat preservation for 12min at 1090°C, water quenched; (3) heat preservation for 12min at 1090°C, air cooling; (4) heat preservation for 12min at 1090°C in vacuum, air cooling; (5) Special process. For simplicity, the above processes were simplified as furnace, water, air, vacuum, special, respectively.

Table 1 Composition analysis of experimental steels (mass fraction, %)

| element | Cr   | Mo   | Nb   | Fe   | C    | Al   | Ti   | Mn   | S    | P    | Ni   |
|---------|------|------|------|------|------|------|------|------|------|------|------|
| content | 20.8 | 8.65 | 3.78 | 0.3  | 0.07 | 0.23 | 0.26 | 0.18 | 0.002| 0.0015| Bal. |

2.2. Mechanics Performance Testing
Tensile tests were carried out with an Instron 5569 testing machine at room temperature at a tensile loading rate of 0.5 mm/min on specimens with a gauge width of 12 mm and gauge length of 100 mm according to the ASTM-E8 M standard. Microhardness test sample size was 20×10×5 mm. Microhardness tests were performed on the polished surface with a load of 200 g for 20 s using a LGTHVS-3A Vickers microhardness tester.

2.3. Corrosion Testing
The immersion tests refer to the national standard GB10124-88. The weight losses of the alloy samples after different heat treatments were evaluated by immersion testing in a 3.5 wt% NaCl solution at room temperature. Samples were cleaned at 96 h, 120 h and 160 h to remove the surface residues. The specimens were weighed by a FA2204B analytical balance with an accuracy of 1×10⁻⁴ g. Samples with dimensions of 10×10×5 mm and an effective working area of 100 mm² were used for the electrochemical tests. The electrochemical experiments were conducted in a PARSTAT 2273 workstation using a conventional three-electrode electrochemical system. A saturated calomel
electrode (SCE) and graphite electrode were used as the reference electrode and auxiliary electrode, respectively, and a 3.5 wt% NaCl solution was used as the electrolyte solution. The potentiodynamic polarization curves were measured at 20 mV/min. Electrochemical impedance spectroscopy (EIS) measurements were performed by applying a sinusoidal potential perturbation of 10 mV. The impedance spectra were measured with a frequency sweep from $10^2$ to $10^2$ Hz in a logarithmic increment. All electrochemical experiments were performed after stabilization of the free corrosion potential at room temperature.

2.4. Microstructure observation and analysis
Scanning electron microscopy (SEM: TESCAN VEGA III) was adopted to observe the corrosion morphology of the alloys after immersion test. The polished samples were etched with aqua regia solution at 50°C for 5~15 s, and the microstructure were observed by Olympus optical microscope.

3. Results

3.1. Effect of heat treatment on hardness of alloy
Table 2 shows the value of hardness variations of the alloy after different solution treatment. The hardness of the alloy after antivacuum condition is lower than that of the original alloy. With the increase of cooling rate (furnace<air<water), the hardness of the alloy increases gradually. Under the condition of vacuum heat treatment, the maximum hardness of the alloy is obtained, reaching 253HV. Compared with the original alloy, the microhardness of the sample after vacuum heat treatment is increased by 2.5%. After special process, the hardness of the alloy is close to the original.

| Process       | Original | Furnace | Water | Air   | Vacuum | Special |
|---------------|----------|---------|-------|-------|--------|---------|
| Hardness/Hv   | 247.67   | 218.8   | 223.55| 219.01| 253.96 | 247.58  |

3.2. Effect of heat treatment on tensile properties of alloy
Figure 1 presents the mechanical properties of the alloy under different solution treatment processes. It can be seen that the tensile strength and yield strength of the alloy after vacuum treatment is the maximum, reaching 863 MPa and 452 MPa respectively, and the elongation reaching 33%. In the antivacuum process, the tensile strength and yield strength of the alloy decrease gradually as the cooling rate slows down. Especially, the yield strength of the furnace alloy decreases by about 10.4% compared with that under vacuum condition, but the elongation increases to 35.5%. After special process, the tensile strength, yield strength and elongation of the alloy reach 832 MPa, 430 MPa and 37.5%, respectively. In general, the vacuum treatment can improve the strength of the material, and the special process can also improve the strength and toughness of the material.

![Figure 1 The effect of heat treatment on alloy tensile properties](image)
3.3. *Effect of heat treatment on corrosion of alloy*

Figure 2 reveals the results of the full-immersion tests as the weight loss of the alloys after different solution treatments as a function of the duration time in 3.5 wt% NaCl. The experimental results show that all samples have weight loss in the initial stage, and the weight loss of the original and vacuum alloy is the largest, and the weight loss of the special alloy is the least. However, the mass of alloys begins to increase (e.g., at approximately 100 h), which indicates that the corrosion products cannot be removed completely because pitting corrosion may have allowed the electrolyte to permeate into the interior of the alloys.

![Figure 2](image)

*Figure 2 Full-immersion corrosion weight loss curves for different heat-treated alloys*

Figure 3 shows the SEM corrosion morphology of the corroded surface of different solution treated alloys soaked in 3.5wt % NaCl solution for 160 h. A large number of corrosion cracks can be observed on the surface of the original alloy, and the surface layer is relatively loose. As shown in figure 3(b), the surface state of furnace alloy is similar to the original state. As shown in figure 3(d), large corrosion products appear on the surface of the air alloy. There are fine corrosion products on the surface of water alloy. After vacuum treatment, The surface appears partial peeling phenomenon. The surface of special process sample is smooth and no obvious corrosion was observed.

![Figure 3](image)

*Figure 3 The corroded surface morphologies of alloys that were soaked in 3.5 wt % NaCl for 160h (a) Original state, (b) Furnace cooling, (c) Water quenched, (d) Air cooling, (e) Vacuum heat treatment, (f) Special process*
The potentiodynamic polarization curves of Inconel 625 alloy after different heat treatments in 3.5wt% NaCl solution are shown in figure 4. In order to study the corrosion behavior of alloys subjected to different solution treatments, polarization curves were fitted, and the results were compiled and are shown in Table 3. From the perspective of thermodynamics, the lower the corrosion potential \(E_{\text{corr}}\), the greater the corrosion tendency of the material. According to the table 3, after antivacuum solution treatment, the corrosion potential of the alloy is higher than that of the original alloy, and the corrosion current density \(I_{\text{corr}}\) is an order of magnitude lower than it, indicating that the antivacuum solution treatment is beneficial to improve the corrosion resistance. With the decrease of cooling rate, the corrosion potential of the alloy moves to the positive direction, and the corrosion current density increases. The \(I_{\text{corr}}\) of the alloy after water cooling is \(3.8\times10^{-7}\) A/cm\(^2\), and the \(E_{\text{corr}}\) is 0.17 V. The corrosion resistance of the alloy decreases after vacuum solution treatment. After special treatment, the corrosion current density of the alloy is \(4.2\times10^{-7}\) A/cm\(^2\), and the \(E_{\text{corr}}\) reaches 0.31 V. The results show that with the increase of cooling rate, the corrosion potential can be increased, the corrosion current density can be decreased, and the corrosion resistance of the alloy improved effectively.

![Figure 4 Polarization curves of alloys after heat treatment processes](image)

**Table 3 Electrochemical parameters of alloys with different treatment**

| Process          | \(I_{\text{corr}}/\text{A/cm}^2\) | \(E_{\text{corr}}/\text{V}_\text{SCE}\) |
|------------------|-----------------------------------|-----------------------------------------|
| Original         | \(1.2\times10^{-6}\)             | -0.18                                   |
| Furnace          | \(5.1\times10^{-7}\)             | -0.08                                   |
| Water            | \(3.8\times10^{-7}\)             | 0.17                                    |
| Air              | \(6.3\times10^{-7}\)             | -0.01                                   |
| Vacuum           | \(6.7\times10^{-6}\)             | -0.48                                   |
| Special          | \(4.2\times10^{-7}\)             | 0.31                                    |

Figure 5 shows the impedance diagrams of different solution treated alloys in 3.5wt%NaCl. It can be seen from Nyquist diagram that the curvature radius of the original alloy and the vacuum treated alloy is significantly smaller than that of the alloy treated in antivacuum condition, indicating that the corrosion resistance of the alloy increases and the corrosion rate decreases. The maximum impedance curvature radius of the special process alloy indicates that the special process can effectively improve the corrosion resistance of Inconel 625 alloy among the five heat treatment processes.
3.4. Effect of heat treatment on microstructure of alloy
Metallographic structure of alloy after different solution treatments are presented in Figure 6. Figure 6(a) shows that the microstructure of the original alloy is mainly composed of large irregular cellular crystals, characterized by a lot of deformation twins. Compared to original alloy, the degree of recrystallization of Inconel 625 alloy in antivacuum condition is excessive, and uniform equiax grains can be obtained, the size of the recrystallization grain is bigger, about 50 μm. After air cooling, the precipitates at grain boundaries increase. Under vacuum solution treatment, the austenite grains in the alloy are fine and evenly distributed. After special treatment, the austenite grain distribution in the alloy is fine and uniform without twinning structure, and the grain boundary precipitation is obvious.

4. Discussion
Some defects in plasticity and corrosion resistance are caused by segregation and a large number of blocky second phases in microstructure of original alloy\textsuperscript{[15]}. After the solution treatment, the alloy is recrystallized, which leads to the increase of plasticity after the solution treatment. The M\textsubscript{23}C\textsubscript{6} carbide,
generated in the heating process, was retained and distributed at the grain boundary during the process, which had an important influence on both elongation and corrosion resistance. The carbide and segregation Nb did not have enough time to diffuse when the cooling time was short, a large number of carbides at the grain boundary which can be nailed dislocation to improve the strength of the alloy. With the decrease of the cooling rate, the M23C6 and segregation Nb dissolved into the matrix to make a stronger solid solution strengthening and improving the strength. After vacuum treatment, a large number of twins appear in the alloy. Twins can hinder the subsequent dislocation glide, and stress concentration will arise at the twin boundary where dislocations are more and more heavily concentrated. Concentration of dislocation will cross the twin boundary and slip again when the stress concentration is large enough. The stress concentration at the twin boundary increases and a large concentration of dislocation can pass through the twin boundary when the crack extends to the twin boundary. There is difference in the crystal orientation on both sides of the twin boundary, which leads to crack deflection when the crack crosses the twin boundary. Therefore, vacuum alloy is both in well strength and toughness.

The relative dissolution or passivation of a surface has been linked to the total grain boundary of alloy, which is considered as a representation for the overall surface reactivity and diffusivity and plays a critical role in the corrosion and passivation processes. Depending on specific material and environment combinations, an increased grain boundary density can either increase or decrease the corrosion. The main corrosion mechanism of superalloys has been proven to be corrosion occurring in hot corrosive environment. The M23C6 carbide contains Cr element at the grain boundary and has a more important effect on the corrosion rate by forming Cr2O3 oxide film. Nb forms a protective surface film, and a decrease in the chemical dissolution rate of oxide films has been shown with the increase of the Nb content.

5. Conclusion
In this paper, the Inconel 625 alloy is taken as the research object, and the influences of 5 solution treatment methods on the microstructure and properties of the alloy are explored respectively, and the main conclusions are as follows:

(1) The microstructure of the original Inconel 625 alloy is mainly austenitic, and the hardness of the alloy is high, but the corrosion resistance is poor. Vacuum heat treatment increases the hardness and strength of the alloy, but decreases the corrosion resistance.

(2) At 1090℃, the cooling rate has a great effect on the microstructure and properties of the alloy. After water cooling, the precipitated niobium carbide is retained at the grain boundary during heat treatment, which improves the corrosion property of the alloy. After air cooling or furnace cooling, chromium carbide precipitates in the alloy again, which leads to the decrease of corrosion properties of the alloy.

(3) Special processing can improve the plasticity and corrosion resistance of the alloy without reducing the strength of the alloy.

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