The effect of grain growth on 21-4N heat resistant steel was studied by static grain growth test. The experimental results show that the temperature inhibits carbide grain growth is between 1 000 - 1 120 °C. When heat preservation time is over 40 min, the driving force of grain boundary is balanced with binding force of carbide nail, grain size will not grow up. There is no limit of grain size due to no pinning effect of carbides when the temperature is above 1 180 °C. Based on the theory of grain boundary migration, the grain growth model of 21-4N heat-resistant steel was established, and the relationship between the average grain size and the time of heat preservation at different temperatures was predicted.

**Key words:** alloy steel 21-4N; grain growth model; microstructure; carbide pinning; grain boundary migration

**INTRODUCTION**

21-4N has high carbon, nitrogen and manganese content, high deformation resistance, high strength, low plasticity and brittleness at room temperature. The effect of processing hardening is very obvious. In the range of thermal deformation temperature, cracks are easy to appear in the production process, the processing difficulty is large, the product yield is not high, and the quality is unstable [1]. Carbides have an important influence on the creep property, notch sensitivity and superplastic behavior of the material [2 - 3]. It can pinning the grain boundary, hinders the migration of grain boundary and inhibits the grain growth. Hot processing is an important method for forming 21-4N parts. During the process of hot processing, the microstructure evolution of the material will be grain growth, dynamic recrystallization, static recrystallization, carbide dissolution and so on [4]. Reasonable control of hot processing and subsequent heat treatment process parameters is the key to obtain uniform and grain parts. 21-4N alloy should often be used in engine valve, and its heat conductivity is low, which makes the heating and holding time in hot processing and high temperature after forming a long time. Therefore, it is very important to study the behavior of 21-4N grain growth, to optimize the process parameters and to obtain the parts with excellent performance.

At present, much research has been done on grain growth behavior [5 - 7]. These studies provide a good idea for developing grain growth behavior of 21-4N heat-resistant steel.

In this paper, the grain growth of 21-4N was studied by static grain growth tests under different holding temperature and holding time. The 21-4N grain growth model was established based on the grain boundary migration theory, and the average grain size under different insulation conditions under test statistics was compared and verified.

**EXPERIMENTAL PROGRAM**

The static grain growth experiment of 21-4N heat-resistant steel is to study the grain growth rule under different holding temperature and different holding time. The samples were incubated at different temperatures for different periods of time. Then the microstructure of the samples was observed, and the grain size was counted. Finally, a grain growth model was established. The specific experimental program is as follows:

1. **Holding temperature:** 1 000 °C, 1 060 °C, 1 120 °C, 1 180 °C;
2. **Holding time:** 0 min, 10 min, 20 min, 30 min, 40 min.

The size of the sample is a small cylinder of Φ 8×15 mm, and the static growth experiment is performed after the water sample is quenched to retain its high-temperature structure according to the above experimental scheme. The cooled sample was cut along the axial centerline, ground and polished, and then etched with picric acid, hydrochloric acid and alcohol solution for 10 - 15 seconds. Finally, the microstructure was observed in an optical microscope, and the average grain size was calculated using 4 points and 4 fields of view.
ANALYSIS OF GRAIN STATIC GROWTH LAW

The austenite grain grows mainly through the grain boundary migration, and its driving force comes from the interfacial energy of the austenite grain boundary. However, when there are dispersed fine precipitate particles in the steel, the coarsening tendency will be due to the existence of the resistance is weakened or eliminated, so the precipitated phase can pin the prior austenite grain boundary during heating, effectively preventing grain growth. The analysis of the evolution of austenite grain size at different temperatures has a good guiding role in the development of a reasonable process route. Figure 1 is the metallographic structure of the sample after incubation at 1 000 °C for 10 min - 40 min. Compared with the original grain size, the grain size of 21-4N at 1 000 °C for 10 min (Figure 1 (a)) and 40 min (Figure 1 (b)) increased by 9.89 μm and 11.56 μm, respectively. Growing up is not obvious.

Figure 1 The microstructure of 21-4N at different holding time at 1 000 °C

Figure 2 shows the metallographic structure of the sample after incubation at 1 120 °C for 10 min – 40 min. Compared with the original grain size, the grain size of 21-4N at 1 000 °C for 10 min (Figure 2 (a)) and 20 min (Figure 2 (b)) increased by 12.18 μm and 13.46 μm, respectively. Big is not obvious. When the holding time exceeds 40 min, the grain size is maintained at 14.73 μm to the left, and the grains no longer grow. This shows that the precipitation of M23C6 particles at 1 120 °C cannot be completely dissolved, the pinning effect inhibits grain growth, after 40 min of incubation, the grain boundary pinning force and the driving force of grain boundary migration balance, so that the grain is not grow up again.

Figure 2 The microstructure of 21-4N at different holding time at 1 120 °C

Figure 3 shows the metallographic structure of 21-4N after incubation at 1 180 °C for 10 min, 20 min, 30 min, and 40 min. From the metallographic map, it can be seen that the average grain size of 21-4N austenite increases significantly with the increase of holding time. The average grain size grows to 67.05 μm (Figure 3 (a)) and 72.27 μm (Figure 3 (b)) and 90.027 μm (Figure 3 (c)), respectively. 92.8 μm (Figure 3 (d)). This is
because the carbides pinned around the grain boundary dissolve at a high temperature, resulting in an abnormal grain growth.

The microstructure of 21-4N after incubation at 1 000 °C, 1 060 °C, 1 120 °C and 1 180 °C for 40 minutes is shown in Figure 4. The average grain size was 11.56 μm, 12.36 μm, 14.73 μm, and 92.82 μm, respectively. The grain size below 1 120 °C hardly grows, but the grain size above 1 180 °C increases.

Statistics show that the average grain size of 21-4N at different temperatures varies with the holding time. The specific statistics are shown in Table 1. Within 1 120 °C, the grain growth is small, and the average grain size is basically the same do not grow to reach a balance. It shows that when the heating temperature is within 1 120 °C, the M23C6 phase particles are not completely dissolved, and pinning around the grain boundary inhibits the grain growth, and thus the grain size reaches a stable value. When the temperature is higher than 1 120 °C, the stability of M23C6 phase is poor under high temperature, and a large amount of dissolution occurs. The pinning effect on austenite is severely weakened, the grain growth is obvious, and even grain coarsening is induced, and coarse-grained mixed crystals appear, the result are shown in Table 1. Therefore, the rolling temperature of 21-4N should be controlled below 1 120 °C to avoid grain coarsening and mixed crystal defects. At the same time, the small crystall grain size can also improve the yield strength and fatigue strength of the steel during deformation. At the same time, the steel has high plasticity and impact toughness, and can reduce the brittle transition temperature of the steel.

<table>
<thead>
<tr>
<th>Heating Temperature/°C</th>
<th>Soaking time/min</th>
<th>0</th>
<th>10</th>
<th>20</th>
<th>30</th>
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<td>1 000</td>
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<td>9.95</td>
<td>10.69</td>
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<tr>
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<td>11.53</td>
<td>11.80</td>
<td>12.36</td>
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<tr>
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<td>12.18</td>
<td>13.46</td>
<td>14.56</td>
<td>14.73</td>
<td></td>
</tr>
<tr>
<td>1 180</td>
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<td>67.05</td>
<td>82.23</td>
<td>90.02</td>
<td>92.82</td>
<td></td>
</tr>
</tbody>
</table>

Figure 5 compares the predicted and experimental values of the 21-4N grain growth model, and the model can predict well.

Describe the microstructure evolution of metal materials, and the grain size is an important consideration.
In the thermoforming of metal materials, the growth process of grains usually includes the static growth of crystal grains and the recrystallization refinement. These two processes are crystal-clear granules play an important role in evolution. The static grain growth has been studied above. Recrystallization is the grain refinement process. The static grain growth has been researched above. Recrystallization is the grain refinement process. Considering the effect of grain static growth and fractional rate of recrystallization on grain evolution, the evolution rate of grain size is expressed as follows:

$$d = G_1 (d_1 / d)^{\psi_1} - G_2 (d / d_0)^{\psi_2}$$  \hspace{1cm} (5)

In the equation:
- $G_1$, $G_2$, $d_1$, $d_0$, $\psi_1$, $\psi_2$ – Material constant.

CONCLUSIONS

Through the experimental study, the following conclusions are obtained:

1. At 1 000 ~ 1 120 °C, the grain growth is not obvious. As a result of pinning, the grain growth is inhibited. When the holding time exceeds 40 min, the grains will not grow up to a limit.
2. The grain growth rate decreases with the prolongation of the heat preservation time when the carbides are kept above 1 180 °C. There is no limit value for the grain size due to the absence of the pinning effect of carbides.
3. The grain growth model of 21-4N stainless steel is established, and the model has a good prediction effect.

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REFERENCES


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