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The Effect of Aging Heat Treatment on the Microstructure and Mechanical Properties of 10Cr20Ni25Mo1.5NbN Austenitic Steel

Abstract: The effect of aging heat treatment on the microstructure and mechanical properties of 10Cr20Ni25Mo1.5 NbN austenitic steel was investigated in this article. The microstructure was characterized by scanning electron microscopy, energy dispersive spectrometry and transmission electron microscopy. Results show that the microstructure of 10Cr20Ni25Mo1.5NbN austenitic is composed of austenite. This steel was strengthened by precipitates of secondary phases that were mainly M23C6 carbides and NbCrN nitrides. As aging treatment time increased, the tensile strength first rose (0–3,000 h) and then fell (3,000–5,000 h) due to the decrease of high density of dislocations. The impact absorbed energy decreased sharply, causing the sulfides to precipitate at the grain boundary. Therefore, the content of sulfur should be strictly controlled in the steelmaking process.

Keywords: 10Cr20Ni25Mo1.5NbN austenitic, aging treatment, microstructure, mechanical property

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Introduction

Developing the ultra-supercritical technology is of great interest to improve the boiler efficiency and reduce the pollutant emission [1–3]. High-temperature materials are one of important factors for improving the steam parameter. In the twenty-first century, many countries are devoting to developing the newly austenitic heat-resistant steel for ultra-supercritical coal-fired (USC) power plants [4, 5]. 10Cr20Ni25Mo1.5NbN austenitic steel is a new 20Cr–25Ni-type austenitic heat-resistant steel, which was developed on the basis of the conventional steel by strict controlling of constituents and impurities [6]. High creep strength of 10Cr20Ni25Mo1.5NbN austenitic steel was obtained by precipitation strengthening of (Nb, Ti) carbonitrides and grain boundary (GB) strengthening of element boron [7–10]. Fireside and steam-side corrosion resistance of 10Cr20Ni25Mo1.5NbN austenitic steel was enhanced due to high Cr and Ni contents [10–13]. In addition, 10Cr20Ni25Mo1.5NbN austenitic steel shows an equivalent welding property with the 18–8-type heat-resistant steel [14]. However, little literature is available on the effect of aging treatment on the performance of 10Cr20Ni25Mo1.5NbN steel.

The purpose of this paper is to obtain the relationship between the microstructure and mechanical properties of 10Cr20Ni25Mo1.5NbN austenitic steel after aging heat treatment.

Materials and methods

10Cr20Ni25Mo1.5NbN austenitic steel plate was smelted with a size of 500 × 300 × 10 mm, and its compositions in accordance with ASME CC 2581 standard are shown in Table 1. The heat treatment of 10Cr20Ni25Mo1.5NbN steel is described below: Solution treatment at 1,250℃ followed by water cooling.

In this article, designed aging temperature was 650℃ because 10Cr20Ni25Mo1.5NbN austenitic steel was applied for the USC power plant with a steam temperature of 600℃ or above. The periods of aging treatment were 1,000, 3,000 and 5,000 h. After aging treatment, the tensile and impact specimens (5 mm × 10 mm × 55 mm–2 mm V type) were
Table 1 The compositions of experimental 10Cr20Ni25Mo1.5NbN austenitic steel in mass %

<table>
<thead>
<tr>
<th>Element</th>
<th>10Cr20Ni25Mo1.5NbN</th>
<th>ASMECC 2581</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.027</td>
<td>&lt; 0.10</td>
</tr>
<tr>
<td>Si</td>
<td>0.404</td>
<td>&lt; 1.00</td>
</tr>
<tr>
<td>Mn</td>
<td>0.98</td>
<td>&lt; 1.50</td>
</tr>
<tr>
<td>P</td>
<td>0.007</td>
<td>&lt; 0.030</td>
</tr>
<tr>
<td>S</td>
<td>0.006</td>
<td>&lt; 0.030</td>
</tr>
<tr>
<td>Cr</td>
<td>23.01</td>
<td>19.5–23.0</td>
</tr>
<tr>
<td>Mo</td>
<td>1.486</td>
<td>1.00–2.00</td>
</tr>
<tr>
<td>Ni</td>
<td>25.5</td>
<td>23–26</td>
</tr>
<tr>
<td>Ti</td>
<td>0.006</td>
<td>&lt; 0.20</td>
</tr>
<tr>
<td>N</td>
<td>0.133</td>
<td>0.10–0.25</td>
</tr>
<tr>
<td>B</td>
<td>0.0058</td>
<td>0.002–0.010</td>
</tr>
<tr>
<td>Nb</td>
<td>0.289</td>
<td>0.10–0.40</td>
</tr>
</tbody>
</table>

Results

Microstructure

Figure 1 shows the microstructure of 10Cr20Ni25Mo1.5NbN austenitic steel after aged for different times. The microstructure was austenite with twins in grains. Austenitic grains grew slightly with increasing aged time. However, the precipitation and growth of the carbide particles along the GBs were evident. The precipitates in austenitic grains also increased. In Figure 1(d), coarsening of GBs was observed, and some place is even up to 2 μm. A large number of gray precipitates appeared at the interface of white typical carbides and the matrix, leading to lesser bonding strength between grains. Growth of those precipitates was found along direction perpendicular to the GB, as shown in Figure 1(d).

Figure 2 displays the dislocation distribution of 10Cr20Ni25Mo1.5NbN austenitic steel through TEM pictures. As the aged time increased, the dislocation density increased (0–3,000 h) and then decreased greatly (3,000–5,000 h). In Figure 2(a), precipitates of 10Cr20Ni25Mo1.5NbN austenitic steel were not detectable in the grain or along the GB. However, there were many dislocations distributed in the grain. Partial dislocation coarsened. Moreover, the tiny precipitates in the GBs became bigger, which pinned the dislocation strongly.
Figure 3 shows the morphology of $M_23C_6$ carbides after different aging times. $M_23C_6$ carbides were both observed in the grain and along the GBs of all aged specimens. After aged for 1,000 h, $M_23C_6$ carbides in grains and along the GBs were mainly discrete granular and lamina, respectively. $M_23C_6$ carbides were still discrete granular after aged for 3,000 h. However, when it comes to the specimens aged for 5,000 h, $M_23C_6$ carbides in GBs joined and evolved to chains with a few micrometers in length. Moreover, the amount of $M_23C_6$ particles in grains increased distinctly whose size stays almost invariable. Some even interfaced with dislocations, as shown in Figure 3(f). With increasing aging time, $M_23C_6$ carbides in GBs coarsened from granular to chains.

We also found clavate NbCrN phases in grains and dislocations of 10Cr20Ni25Mo1.5NbN austenitic steel, which was stable with aging treatment time, as shown in Figure 4. These phases played a significant role in improving tensile strength. However, in this steel Nb(C, N) phases were not observed in the grains which could enhance the creep strength to some extent [10, 11]. We deduced that the precipitates $M_23C_6$ in grains and GBs consumed too much element C, resulting in the C shortage for forming Nb(C, N) phases.

**Mechanical properties**

Figure 5 shows the tensile results at room temperature after different aging times. As the aging time increased, the tensile strength of 10Cr20Ni25Mo1.5NbN austenitic steel first increased (0–3,000 h) and then decreased (3,000–5,000 h). Tensile ductility fell greatly (0–1,000 h) and then rose (1,000–5,000 h). Elongation and reduction of the cross sections both decreased. The change of reduction was largest from 0 to 1,000 h. Figure 6 shows the impact and B-hardness of 10Cr20Ni25Mo1.5NbN austenitic steel under aging conditions. The impact absorbed energy (IAE) decreased with increasing aging time up to 5,000 h. After 1,000 h aging treatment, the IAE decreased sharply, only 26.8% was kept. And the value of hardness was the smallest. As the aging time increased, the hardness of 10Cr20Ni25Mo1.5NbN austenitic steel displayed a similar tendency to the tensile ductility.

Figure 7 shows fractographs of impact specimens after aged for different times. The fractographs showed typical ductile fracture while the aged specimens showed brittle fractures. As aged time increased, the impact toughness decreased quickly which was in agreement with their IAEs. Many precipitates were observed in the dimples from Figure 7. EDS results display that they were composed of Fe or Mn sulfides.

**Discussion**

The strengthening mechanism of 10Cr20Ni25Mo1.5NbN austenitic steel was mainly dislocation and secondary
Figure 3  The $M_2C_6$ morphologies of 10Cr20Ni25Mo1.5NbN austenitic steel after different aging times: (a) 1,000 h GB, (b) 1,000 h grain, (c) 3,000 h GB, (d) 3,000 h grain, (e) 5,000 h GB and (f) 5,000 h grain

Figure 4  NbCrN-phase morphologies after aging treatment: (a) grain and (b) dislocation
Figure 5  Tensile results at room temperature after different aging times

Figure 6  The impact and B-hardness of 10Cr20Ni25Mo1.5NbN austenitic steel under aging conditions

Figure 7  Fractographs of impact specimens after aged for different times and EDS result in the dimple: (a) 0 h, (b) 1,000 h, (c) 3,000 h, (d) 5,000 h and (e) EDS analysis of particles in dimples
phase strengthening from microstructure analysis. Strengthening increment $Y_{S\text{PO}}$ caused by the secondary phase particles (SPPs) has a relationship with its volume percent $f$ and size $d$, as shown in the following:

$$Y_{S\text{PO}} \propto f^2d^{-1}\cdot \ln d$$  \hspace{1cm} (1)

Strengthening increment $Y_{S\text{D}}$ caused by dislocations correlate with density $\rho$ and coefficient $\alpha$ as shown in the following:

$$Y_{S\text{D}} = 2\alpha Gb\rho^{0.5}$$  \hspace{1cm} (2)

where $G$ is the shear modulus and $b$ is the absolute value of Burgers vector [15].

The mechanism of secondary phase strengthening was believed to be that the SPPs in the matrix hinder dislocation slip [15]. The coherent relationship between SPPs and the matrix was damaged by the big SPPs which ultimately became nondeformation particles. Dislocations only passed over these SPPs under bigger external stress.

From eq. (1), enlargement of SPPs volume fraction improves the tensile strength of 10Cr20Ni25Mo1.5NbN austenitic steel. The morphology of $M_23C_6$ precipitates evolved from particles to chains in GBs. The size of precipitates in grains including $M_23C_6$ and NbCrN phases was almost invariant with increasing aging time. The amount of $M_23C_6$ particles in grains increased distinctly. What's more, the small precipitates in dislocations pinned partial dislocations. Therefore, the tensile strength was enhanced from the perspective of SPPs.

The $Y_{S\text{D}}$ has a positive correlation with dislocation density $\rho$ and coefficient $\alpha$. In Figure 2, dislocation density increased (0–3,000 h) and decreased sharply (3,000–5,000 h). Dislocation reaction in some direction may take responsibility for the decreased dislocation density. Under high dislocation density (3,000 h), per unit length in dislocation lines of strain energy decreased because of restriction of long-distance stress field, leading to a decrease in coefficient $\alpha$ [15]. So the $Y_{S\text{D}}$ (5,000 h) was lower than $Y_{S\text{D}}$ (3,000 h). As a result, the tensile strength increased (0–3,000 h) and decreased (3,000–5,000 h) considering two above strengthening factors.

The decrease in impact toughness had a direct relationship with precipitated inclusions and SPPs during the aging period. The IAE decreased sharply (0–1,000 h) because of MS (M: Mn, Fe) precipitates in GBs, as shown in Figure 7e. Early MS precipitates weakened the bonding force between GBs or carbides/matrix, promoting the nucleation of cavities in these positions. With a long aging time, the cavities grew and evolved to microcracks. It agrees well with the fractographs of impact specimens, as shown in Figure 7(b). The decrease of IAE also correlated with $M_23C_6$ carbides which coarsened from granular to chains in GBs.

### Conclusions

1. The microstructure of 10Cr20Ni25Mo1.5NbN austenitic steel consists of austenite with some twins. The growth of austenitic and reduction of twins occurred during the aging treatment.

2. This steel was strengthened by precipitates of secondary phase containing typical $M_23C_6$ and NbCrN phases. The sizes of SPPs in grains were invariant, whereas the $M_23C_6$ carbides evolved from particles to chains with increasing aging time. The decrease in dislocation density caused the decrease in tensile strength of 10Cr20Ni25Mo1.5NbN austenitic steel.

3. The IAE decreased with increasing aging time (0–5,000 h). The fractographs of impact specimens showed typical ductile fracture while the aged specimens showed brittle fractures. We suggest that MS (M: Mn, Fe) precipitates in GBs lead to the lower IAEs.

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### References