熱軋與回火對 13% Cr 不銹鋼機械性質的影響
Effect of Hot Rolling and Tempering on the Mechanical Properties of 13% Cr Stainless Steel

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Abstract

The effect of prior hot rolling and tempering on the mechanical properties of 13% Cr MSS was studied as a function of tempering temperature and grain size. The results indicate that a secondary strengthening phenomenon in stress and hardness occurs at tempering temperature of 500 °C. The grain size decreases with increasing in the rolling reduction. Fine grain leads to an increase in the density of grain boundary and the homogeneous dispersion of carbide particles, that provides a dominant action against the crack propagation and increasing the mechanical properties.

Keywords: Hot rolling; Mechanical property; Stainless steel; Grain size

摘 要

本文探討 13% Cr 麻田散鐵不銹鋼經熱軋與 200~600 °C 回火處理後的機械性質，分別討論晶粒尺寸與回火溫度的影響。結果顯示，回火於 500 °C 時有明顯的二次強化現象，使本實驗材料具有較高之硬度和強度與相對較低之延性和韌性；回火前的熱軋有明顯細晶效果，晶粒尺寸隨熱軋量增加而減小，細晶伴隨的高密度晶界與均勻析出的碳化物可有效阻礙裂縫連結進而提升其機械性質。

關鍵字: 熱軋，不銹鋼，回火，二次硬化，晶粒尺寸

1. Introduction

Martensitic stainless steels (MSS) are extensively used as end fitting and valve
body in the chemical and power industries and as compressor blades in modern aircraft engines [1-3]. Moreover, they are generally heat treated to provide moderate corrosion resistance and a good combination of mechanical properties. This treatment often involves an austenitizing, followed by quenching, and then tempering processes. The austenitizing leads to carbide coarsening, which make difficult chemical composition homogenization in the austenite. Therefore, the original microstructure can transform to austenite single phase or austenite plus ferrite duplex system. Finally, these phases may lead to duplex microstructures formation of martensite and ferrite. Tempering of the as-quenched martensitic steel can bring about secondary hardening by the precipitation of alloy carbide in the material [4], which can moderately improve the mechanical properties. Another important factor correlated to phase transformation is hot forming process. During hot working, various microstructural changes such as dynamic recovery and recrystallization may occur and alter the final microstructure and mechanical properties [5]. However, the growth of grain during austenitizing can affect adversely the mechanical properties. It is common knowledge that the grain of matrix can be refined by mechanical deformation such as hot rolling. Hence, an improvement of mechanical properties can be obtained reasonably.

13% Cr MSS is one of these MSS with about 11-13% Cr, 0.1% C and a small amount of austenite-stabilizing elements such as manganese and nickel. According to the Schaeffler diagram [6] as show in Fig.1, the microstructure of 13% Cr MSS mainly consists of the mixture of martensite and ferrite. Due to the different property of these two phases, the mechanical properties of 13% Cr 403 MSS are affected not only by the volume fraction of phases but also through the morphology and the property difference between phases. In general, that cannot be determined on the basis of simple law of mixture. So their mechanical properties as function of tempering effect and grain size are not fairly well understand.

There have been several researchers to study the behavior of steels under hot working conditions [7-12]. In these works, the metal flow behavior during deformation was mainly taken into account. Tempering behavior of 13% Cr MSS has been investigated in terms of carbide precipitation and second hardening effect [13,14]. To the best of our knowledge only a few reports have yet been made to characterize the effect of hot rolling and tempering on the mechanical properties of stainless steels [15,16]. In this study, the 13% Cr MSS was processed with hot rolling and then
tempering, in which mechanical properties as a function of tempering temperature and grain size will be discussed.

2. Experimental Procedures

2.1 Material and specimen preparation

In order to get the final form of 14 mm thick after rolling, slabs with three thickness (16.5, 20 and 28mm), 200 mm long and 150 mm wide were prepared for rolling to give 15,30 and 50% reduction in the thickness, respectively. These slabs were first homogenized to eliminate the segregation by holding at 1000°C for 24h, and then air cooling down to room temperature. After hot rolling and tempering, at least 2 mm thick on each side was machined out to remove the decarburization layer. Subsize tensile test specimen with 6.25 mm wide and 5 mm thick according to ASTM E-8 standard [17] and type A Charpy impact test specimen according to ASTM E-23 standard [18] were cut parallel to the previous rolling direction. Metallography and hardness specimens with dimension 20 × 20 × 10mm were cut from the middle of the slabs

2.2 Hot rolling

The slabs were given a single pass rolling in a 700 tons Nippon rolling mill with rolls of radius 360 mm. The rolling speed was set at 40 rpm. Throughout the processing, the temperature of the slabs was measured using K-type thermocouple, which was inserted to the center of the slabs. The slabs were preheated to 1010°C for 2h, and then removed from the furnace and rolled at entry temperature of 900°C. After rolling the slabs were quenched in water. To compare the reduction effect, after preheating, one set slabs were quenched directly without rolling, which is designated as the 0% reduction in this study.

2.3 Heat treatment

All slabs after rolling were double-tempered at 200, 300, 400, 500, and 600°C, respectively. The heat treatment procedure is shown in Fig.2.
2.4 Microstructural and image analysis

Optical microscopy (OM), Scanning electron microscopy (SEM) and electron probe microanalysis (EPMA) were utilized to examine the microstructure of the specimens after polishing and etching. Image analysis was performed in the metallography specimen using an automatic image analyzer to determine grain size and volume fraction of ferrite. The grain size was measured by the method of mean grain intercept. Each datum was the average of at least twenty measured results.

2.5 Mechanical properties testing

Hardness testing of the constituents and bulk material were performed using standard Vickers (100g) and Rockwell hardness testers, respectively. Before testing, the specimens were polished and etched in the same way as for the metallographic examination. All the Rockwell hardness readings were converted to Vickers hardness numbers. At least five hardness reading were taken, and then averaged. Tensile tests were carried out using a tensile machine (MTS model 810) at a constant strain rate of $10^{-3} \text{s}^{-1}$. Impact tests were performed using a Charpy impact tester. For each condition, every tensile and impact tests were repeated at least three times and then averaged.

3. Results and Discussion

3.1 Microstructure and Chemical Composition

The chemical composition of the experimental material is listed in Table 1. The Cr- and Ni-equivalent values by using Schaeffler formula are 13.755 and 3.24, respectively [6]. A typical microstructure of the as-received experimental material is shown in Fig.3(a), which shows a banded microstructure consists of approximately the average amount of 70% martensite and 30% ferrite and the result is accordant with the Schaeffler diagram (Fig.1). Figure 3(b) and (c) shows the tempering effect on microstructure change. After tempering at 200°C, some ferrite islands are found in martensite plate, as shown in Fig. 3(b). The forming of low carbon ferrite means that the martensite had decomposed into tempered martensite and Cr-rich carbide. The result of line scan by EPMA also suggests the distribution of C and Cr both being more turbulent.
than that of Fe, providing the evidence for precipitation of carbides. These carbides were identified as Cr$_2$C$_6$ by Miao [14]. Similar phenomenon is also observed in other specimens tempered within 300~500°C, wherein the precipitation of alloyed carbide increases with increasing in the tempering temperature up to 500°C. While tempered at 600°C, the carbide decreases and the ferrite island increases (Fig. 3(c)), and this leads to the decreasing in hardness and strength. In Fig. 3(c), the packet boundaries of the martensitic structure can be seen with careful observation. However, it is difficult to examine their size accurately.

The microstructure of specimens after hot rolling is shown in Fig. 4. The ferrite forming well defined grain boundaries with the martensitic matrix. This lined up of the ferrite is agreement with the rolling direction. The resulting average grain size is 28.6, 19.4, 13.8, and 8.5µm for 0, 15, 30 and 50% reduction in the thickness, respectively. Larger deformation increases the dynamic recrystallization of austenite getting in grain refining. A good relationship between grain size and reduction is shown in Fig. 5. It is note that due to the packet boundaries of the martensitic structure are not easily to distinguish, hence only ferrite grain was measured in this measurement. As mentioned by Cardoso [16], the chemical composition and prior heating microstructure will affect the ferrite formation. In this study, these conditions are consistent. Therefore, the content of martensite and ferrite for all of four rolling reductions are change not remarkably and that is closed to the content of as-received microstructure. The effect of grain size and microstructural variations on the mechanical properties were further discussed in the following section.

3.2 Hardness behavior

Table 2 lists the partial hardness data of constituents and bulk material after rolling and tempering as well as the value estimated by the law of mixture. The estimated values are not consistent with bulk readings, it supports that the mechanical properties of experimental material cannot be determined on the basis of simple law of mixture.

Figure 6 shows the hardness versus tempering temperature. Apart the 0% reduction specimens, the hardness increases with increasing in the reduction for all tempering temperature. This is since a higher grain boundary density provides more carbide precipitation site and a shorter diffusion distance. Fine grain and homogeneous dispersion of carbide particles produce an increase of hardness. However, the hardness
of the 0% reduction specimens is higher than that of the others, i.e. even though the hardness can improve with grain refining by hot rolling but that can not excess the hardness of direct quenching specimens. It is assumed that such behavior is related to the presence of ferrite, this ferrite softens by dynamic recovery, while austenite by dynamic recrystalization. On the other hand, the quenching temperature for the 0% reduction specimens is higher than that of the other reduction samples. Larger driving force make martensite lath to refine, which leads to hardness increase. In addition, tempering effect on hardness is as following changes. The hardness of all four reductions exhibit similar tendency, those first increase with increasing in the tempering temperature until to 500°C and then decrease. A typical secondary hardening is produced by formation of alloy carbides within martensite or at martensite/ferrite interface. It is agreement with the study by Miao [14]. After 600°C tempering, the hardness descends rapidly, mainly resulting from the softening of the martensite.

### 3.3 Tensile behavior

The tensile strength and elongation of the specimens as a function of tempering temperature and reduction are plotted in Fig. 7. For all of four reduction specimens, a secondary strengthening phenomenon evidently occurs in the specimens tempered at 500°C (Fig. 7(a)). The reason is that the harden effect of martensite and the re-precipitated carbides retards tensile micro-cracking connections, resulting in detour of the cracking path and increasing the tensile strength. However, due to the tempered martensite embrittlement effect, the ductility of the specimens is also obviously reduced, as shown in Fig. 7(b). When reduction effect is considered, an interesting result different from Fig. 6 is found. After rolling, both strength and ductility increase to higher than those of the 0% reduction specimens. It is contributed to the higher grain boundary density and homogeneous dispersion of carbide particles, hence, the ability of retarding the crack propagation increases.

### 3.4 Toughness behavior

The impact toughness against tempering temperature is plotted in Fig. 8. The results show that the variations of toughness have a similar trend as the ductility. The brittleness phenomenon occurs at tempering temperature of 500°C, in which,
precipitation carbides cause cracking connection and tearing rapidly under impact fracturing, and the toughness is reduced evidently. Whereas, the grain boundary can retard the crack propagation and alter the cracking path, therefore, the toughness increases with increasing in the reduction.

3.5 Relationship between grain size and maximum mechanical properties

From observation the overall mechanical properties, maximum hardness and strength and maximum ductility and toughness of each reductions occur at tempering temperature of 500 and 600 °C, respectively. Figure 9 shows the relationship between the maximum mechanical properties and grain size. It can be found that the grain size has remarkable effect in the maximum mechanical property of the specimens after rolling. The values of the maximum mechanical properties increase with decreasing in the grain size and exceed those of the direct quenching specimens apart from the hardness.

4. Conclusions

The effect of prior hot rolling on the mechanical properties of 13% Cr MSS was studied as a function of tempering temperature and grain size. The following conclusions are drawn.

(1) The microstructure of the experimental material consists of martensite and ferrite and there are Cr-carbides precipitated within martensite or at martensite/ferrite interface. When the material is tempered at 500 °C, there is a secondary strengthening phenomenon in stress and hardness occurred because the martensite decomposed into tempered martensite and carbide. In the case of tempering at 600 °C, the Cr-carbides are not re-precipitated and thus both martensite and ferrite are softer, which gets the maximum values of ductility and toughness.

(2) The prior hot rolling has a remarkable grain refine effect on the experimental material. The grain size decreases with increasing in the reduction. Fine grain leads to an increase in the density of grain boundary and homogeneous dispersion of carbide particles, that provides a dominant action against the crack propagation and
increasing the mechanical properties. Except hardness, the maximum mechanical properties of each rolling reductions are better than that of the 0% reduction specimens treated with direct quenching and then tempering.

5. REFERENCES

Table 1 Chemical composition of experimental material. (wt%)

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<tr>
<th>C</th>
<th>Cr</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
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Table 2 Hardness (Hv) of 30 % rolling reduction specimens after tempering

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<tr>
<td>600</td>
<td>martensite</td>
<td>278</td>
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![Schaeffler diagram](image)

Fig. 1. The microstructure estimated of experimental material by Schaeffler diagram.
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Fig. 2. Heat treatment Procedure in this experiment.

Fig. 3. OM and SEM metallography of the experimental material (a) as-received, (b) tempered at 200 °C, and (c) tempered at 600 °C.
Fig. 4. SEM metallography of the experimental material after rolling with variant reductions
(a) 0% (70%M, 30%F), (b) 15% (73%M, 27%F), (c) 30% (67%M, 33%F), and (d) 50% (69%M, 31%F).

Fig. 5. Relationship between the ferrite grain size and the reductions.
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Fig. 6. Effect of tempering temperature and reductions on hardness of the specimens.

Fig. 7. Effect of tempering temperature and reduction on tensile properties of the specimens (a) tensile strength, and (b) elongation.
Fig. 8. Effect of tempering temperature and reductions on impact toughness of the specimens.

Fig. 9. Relationship between the maximum mechanical properties and the grain size
(a) hardness and tensile strength, and (b) ductility and toughness.
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