Effect of Thermal Cycling on Microstructures and Mechanical Properties of Lath and Lenticular Martensites in Fe–Ni Alloys

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(Received on July 28, 2000; accepted in final form on January 17, 2001)

Effects of thermal cycling on the microstructures and mechanical properties of Fe–15%Ni and Fe–31%Ni alloys having lath and lenticular martensite, respectively, have been studied. The average width of laths in lath martensite of Fe–15%Ni alloy decreased with the increase in number of thermal cycles, and the width of internal twins in lenticular martensite of Fe–31%Ni alloy also decreased with thermal cycling. The hardness of martensite increased up to 3 thermal cycles in Fe–31%Ni alloy, over which it remained constant. On the other hand, the hardness of martensite in Fe–15%Ni alloy increased up to 1 cycle, and remained nearly constant on further cycles. The tensile strength of martensite in Fe–31%Ni alloy increased up to 3 cycles, showing a similar tendency of the hardness variation. However, the tensile strength of martensite in Fe–15%Ni alloy decreased with increasing the number of thermal cycles in spite of the increase in hardness with thermal cycling. The reason is that no strain hardening occurred in the thermal-cycled specimens owing to the brittle fracture due to the segregation of sulfur at prior austenite grain boundaries.

KEY WORDS: Fe–Ni alloy; repetition of transformation; thermal cycling; sulfur segregation; mechanical properties; martensite.

1. Introduction

The effect of thermal cycling (\(\alpha' \leftrightarrow \gamma'\)) on the mechanical properties of the reverted austenite has been studied by a number of workers in Fe–Ni–C alloys with \(M_s\) temperatures around \(-30^\circ C\), and it was reported that the strength (hardness) of the reverted austenite increased with increasing thermal cycle. On the other hand, some workers have investigated the mechanical properties of martensite after thermal cycling, using the Fe–Ni–C alloys with plate martensitic structures, and have reported that the strength of the martensite also increased with the increase in thermal cycle. Few studies, however have compared the mechanical properties of lath martensite (not reversed austenite) and those of plate martensite after thermal cycling.

In the present paper, we prepared an Fe–15%Ni alloy with lath martensitic structure and an Fe–31%Ni alloy with plate martensitic structure, and examined the microstructures and mechanical properties of those martensites after thermal cycling. The obtained results were discussed in relation to the different martensitic structures.

2. Experimental Procedures

Fe–15%Ni and Fe–31%Ni alloys (in weight) were prepared by high frequency induction melting in a vacuum atmosphere. The ingots of 5 kg were homogenized at \(1100^\circ C\) for 24 hr, and then hot-rolled to sheets with 3 mm in thickness. From the sheets, various specimens for microstructural observation, X-ray diffractometry (XRD), and tensile test were prepared by rolling and machining. Martensitic transformation temperatures (\(M_s, A_s, A_f\)) were determined using a differential scanning calorimeter (DSC). The chemical compositions and transformation temperatures of the experimental alloys are listed in Table 1. All specimens were solution-treated at \(1100^\circ C\) for 1 hr, quenched into water at room temperature, and subzero-treated at liquid nitrogen (\(-196^\circ C\)). Thermal cycling (\(\alpha' \leftrightarrow \gamma'\)) was conducted by dipping the martensitic specimen into a salt bath held at 670 or 430°C just above \(A_s\) temperature and then quenching into water followed by subzero cooling at liquid nitrogen. The duration time at each temperature was 5 min. A sample for optical micrography was electro-polished in a solution of 10%HClO\(_4\)/90%CH\(_3\)COOH and etched with a 3% nital solution. Foils for transmission electron microscopy (TEM) were jet-polished in a 20%HClO\(_4\)/80%CH\(_3\)COOH solution and observed with a JEOL JEM-200CX operating at 200 kV. The volume fraction of constituent phases such as \(\gamma\) austenite (fcc) and \(\alpha'\) martensite (bcc) was measured by a X-ray diffraction (XRD) method using the integrated in-

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**Table 1.** Chemical composition (wt%) and martensitic transformation temperatures (°C) of experimental alloys.

| Alloy   | Ni | C  | Si | Mn | Cr | S  | P  | Fe  | Ms  | As  | Af  |
|---------|----|----|----|----|----|----|----|-----|-----|-----|-----|
| Fe-15%Ni | 15.15 | 0.003 | 0.06 | 0.01 | 0.03 | 0.007 | 0.004 | 0.004 | 584 | 588 | 640 |
| Fe-31%Ni | 31.36 | 0.004 | 0.05 | 0.01 | 0.02 | 0.006 | 0.006 | 0.006 | 360 | 400 | 580 |
Tensile tests were conducted at room temperature with ASTM-E8 type tensile specimens (thickness: 2 mm, gauge length: 25 mm) using an Instron-type machine at a strain rate of $10^{-3}$/s. Fracture surfaces of the tensile specimens were observed by scanning electron microscopy (SEM) to determine the fracture mode, and were analyzed by Auger electron spectroscopy (AES) to investigate prior austenite grain boundary segregation.

### 3. Results and Discussion

**Figure 1** shows the change in optical microstructures with number of thermal cycles in Fe–15%Ni and Fe–31%Ni alloys. The morphology of lath martensite in the Fe–15%Ni alloy is characterized by packets in austenite grains, within which numerous fine blocks are observed as shown in Fig. 1(a), whereas the lenticular martensite plates with midribs are shown in the Fe–31%Ni alloy, as shown in Fig. 1(d). It is observed in Fig. 1 that the average size of prior austenite grains decreases with increasing number of thermal cycles in the both alloys, which is listed in **Table 2**. The bright field TEM images of the lath and lenticular martensites with respect to number of thermal cycles are shown in **Fig. 2**. In the case of lath martensite, individual laths distributed in parallel with one other contain high density of dislocations (Fig. 2(a)). It is noted in Figs. 2(a), 2(b), and 2(c) that the average width of laths slightly decreases with the increase in number of thermal cycles, but no appreciable change in dislocation density is observed. The decrease in average width of laths with thermal cycling is presumably due to the refinement of prior austenite grains. For the lenticular martensite, the martensite plate consists of internal twins across midrib and high dislocation density near the martensite boundary (Fig. 2(d)). From Figs. 2(d), 2(e), and 2(f), it is obvious that after the thermal cycling, a large number of dislocations were additionally introduced into

![Fig. 1. Change in optical microstructures with number of thermal cycles in Fe–15%Ni and Fe–31%Ni alloys.](image)

(a) Fe–15%Ni alloy before thermal cycling
(b) Fe–15%Ni alloy after 1 thermal cycle
(c) Fe–15%Ni alloy after 7 thermal cycles
(d) Fe–31%Ni alloy before thermal cycling
(e) Fe–31%Ni alloy after 1 thermal cycle
(f) Fe–31%Ni alloy after 7 thermal cycles.

| Cycle No. | Fe-15%Ni | Fe-31%Ni |
|-----------|----------|----------|
|           | Grain diameter (μm) | ASTM No. | Grain diameter (μm) | ASTM No. |
| Non-cycled | 68.88    | 5        | 87.00    | 4        |
| 1 cycle    | 32.63    | 7        | 61.63    | 5        |
| 7 cycles   | 20.66    | 8        | 50.75    | 6        |

**Table 2.** Variation in prior austenite grain size with number of thermal cycles in Fe–15%Ni and Fe–31%Ni alloys.
entire region of lenticular martensite, while the width of pre-existing internal twins gradually decreased, as shown in Fig. 3.

The variation in martensite volume fraction with number of thermal cycles in the Fe–15%Ni and Fe–31%Ni alloys was measured by a XRD method, and the results are given in Table 3. The Fe–15%Ni alloy maintains 100% martensite regardless of thermal cycling, but 1% of retained austenite in the non-cycled specimen of Fe–31%Ni alloy slightly increases to 3% after 7 thermal cycles. The increase in retained austenite for the Fe–31%Ni alloy is ascribed to the austenite stabilization due to the increased dislocation density. This is well consistent with the previous report, wherein the amount of martensite produced from the reverted austenite (γ') in Fe–Ni alloy decreased with increasing number of thermal cycles.

Figure 4 shows the plot of hardness versus number of thermal cycles in the Fe–15%Ni and Fe–31%Ni alloys. In the case of lenticular martensite, the hardness steeply increased up to 3 cycles, over which it maintains a constant value. Considering that the hardness of as-quenched lenticular martensite increases with increasing reduction of thickness after rolling at room temperature in Fig. 5, the lenticular martensite is not saturated with dislocations in as-quenched state. It is, thus, evident that the increase in hard-
ness up to 3 cycles is due to introduction of additional dislocations into martensite during cyclic transformation. On the other hand, the hardness of lath martensite rapidly increases up to 1 cycle and remains nearly constant on further thermal cycles, as shown in Fig. 4. From the fact that the hardness of as-quenched lath martensite of Fe–15%Ni alloy remains constant regardless of reduction in thickness, as shown in Fig. 5, it is believed that the lath martensite is saturated with dislocations in as-quenched state, indicating that no additional dislocations are introduced during thermal cycling. Therefore, the increase in hardness after 1 cycle is attributed to refinement of prior austenite grain size in Table 2, which results in the refinement of block (and packet) size that is responsible for strengthening.10–13)

Figure 6 shows the plot of tensile strength versus number of thermal cycles in both alloys. It is observed that the change in tensile strength with number of thermal cycles in lenticular martensite is well consistent with the hardness change in Fig. 4. However, the tensile strength of lath martensite decreases with increasing the number of thermal cycles in spite of the increase in hardness with thermal cycling in Fig. 4. The reason is that no strain hardening occurs in the cycled specimens of lath martensite, as shown in Fig. 7, because of almost zero elongation as shown in Fig. 8.

Figure 9 shows scanning electron micrographs of fracture surfaces of the tensile specimens for the Fe–15%Ni and Fe–31%Ni alloys. It is observed that all the specimens of Fe–31%Ni alloy and the non-cycled specimen of Fe–
15%Ni alloy were fractured in a ductile mode by formation of dimples with various sizes. However, the cycled specimens of Fe–15%Ni alloy show intergranular fracture along prior austenite grain boundaries as shown in Fig. 9(b) which is a typical structure. This is well consistent with zero elongation of the specimens in Fig. 8.

Figure 10 shows the Auger analysis of the fracture surface of the 3-cycled specimen of Fe–15%Ni alloy. It is seen that the concentration of sulfur at the prior austenite grain boundaries is as high as eight times that of grain interior by comparing the height of sulfur peak at the grain boundary with the peak obtained from the grain interior. However, no segregation of other elements including phosphorus at the grain boundaries is detectable.

A number of studies\(^\text{14–21}\) have been performed on the tempered martensite embrittlement of low-alloy steels. There are two different results in the works: One group\(^\text{14–18}\) has suggested that the temper embrittlement is attributed to the segregation of phosphorus at prior austenite grain boundaries, while the other group\(^\text{19–21}\) insisted that the segregation of sulfur at austenite grain boundaries causes the temper embrittlement. Noticeable points in their works are that the composition of the steels used in the works are different from work to work, and that the tempering temperatures are also different in a wide range from 250°C to 700°C. This suggests that the different segregation elements may be attributed to the alloying elements contained in the steels and tempering temperatures.

In this study, it is apparent that the embrittlement of cycled specimens of the Fe–15%Ni alloy is associated with segregation of sulfur at prior austenite grain boundaries, owing to the fact that no segregation of other elements except for sulfur occurred as shown in Fig. 10.

4. Conclusions

(1) The average width of laths in lath martensite of Fe–15%Ni alloy decreased with the increase in number of thermal cycles, and the width of internal twins in lenticular martensite of Fe–31%Ni alloy also decreased with thermal cycling.

(2) The hardness of martensite increased up to 3 thermal cycles in Fe–31%Ni alloy, over which it remained constant. On the other hand, the hardness of martensite in Fe–15%Ni alloy increased up to 1 cycle, and remained nearly constant on further cycles.

(3) The tensile strength of martensite in Fe–31%Ni alloy increased up to 3 cycles, showing a similar tendency of the hardness variation, while the tensile strength of martensite in Fe–15%Ni alloy decreased with increasing the number of thermal cycles in spite of the increase in hardness with thermal cycling.

(4) The martensitic specimens of Fe–31%Ni alloy were fractured in ductile mode regardless of thermal cycles. However, the thermal-cycled martensitic specimens of Fe–15%Ni alloy were fractured in brittle mode due to the segregation of sulfur at prior austenite grain boundaries.

Acknowledgements

The helpful assistance from Mr. Jong-Bin Noh in the experiment is gratefully acknowledged. One of the authors (J. H. Jun) would like to thank Korea Science and Engineering Foundation (KOSEF) for the financial support of postdoctoral study at University of Illinois at Urbana-Champaign in 2000.

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