Electron Microscopic Study of Structural Changes in 18-8 Austenite Stainless Steel after Solution and Precipitation Treatment

By Toshimi Yamane* and Jitsuhiko Ueda*

In order to investigate lattice defects of 18-8 stainless steel after solution, precipitation and resolution treatments, thin films prepared from bulk specimens after the heat treatments were examined by means of transmission electron microscopy. In addition, measurements of the internal friction, specific gravity and electrical resistivity as well as Huey corrosion tests were conducted. The experimental results obtained are as follows:

(1) The 18-8 austenite stainless steel has the strain amplitude dependent internal friction after the solution or precipitation treatment.

(2) The precipitation of carbides (after annealing at 600°C~800°C) may give rise to a great decrease in corrosion resistance and a decreases in specific gravity. The strain amplitude independent internal friction decreases with rise in the annealing temperature up to 400°C after the solution treatment, while the precipitation of carbide does not affect the internal friction.

(3) A large number of twins are observed in a specimen solution-treated, but after annealing at 700°C (precipitation treatment) and at 930°C (resolution treatment), a few twins and dislocations are observed.

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I. Introduction

Although 18 Cr-8 Ni austenite stainless steel excells in corrosion resistivity when subjected to the solution treatment, the carbide precipitated during aging at temperatures between 600°C and 800°C results in a remarkable decrease of the corrosion resistivity(1). A number of research have dealt with the precipitation 18-8 stainless steel in the light of the composition (2), crystal structure(3) and form of the precipitate, and of the variation in the concentration of the carbon and chromium atoms in the matrix due to the precipitation(4). However, little has been known on the matrix and carbide observed directly by transmission electron microscopy.

It is generally known that the carbide precipitation of 18-8 stainless steel does not influence the mechanical properties such as tensile strength, hardness and impact value, but causes marked changes in the Huey corrosion resistivity and structure. In this regard, the authors deduced that the carbide precipitation would induce, not only variations in the concentration of chromium and carbon atoms around the precipitates but also the nucleation of vacancies and dislocations or the local expansion and contraction. Ainslie et al. reported the diffusion of sulfur in iron(5), which was accordant in principle to the authors' point of view in regard to the diffusion of vacancies. In this paper, the structural changes around the precipitates of 18-8 stainless steel were examined by means of transmission electron microscopy.

II. Specimens and Experimental Procedure

Specimens used in this experiment were all prepared from a hot rolled 18-8 stainless steel (AISI-304) 1 mm in thickness. The chemical composition of the specimens is shown in Table 1. The specimens for electron microscopic observation were electrochemically thinned in 6:4 solution of concentrated phosphoric acid and sulfuric acid at 9V and 1.5 A/cm² according to Bollmann’s method(6). Throughout the microscopic observations, a Hitachi H.U.-10 type transmission electron microscope was used at 75 KV. The electrical resistivity of specimens 1.5 mm × 30 mm × 80 mm was measured at a room temperature of 18°C after specified heat treatments. The internal friction was determined in the following way(7). A transversal resonance vibration was given to a specimen 1 mm × 10 mm × 200 mm and then the electricity in the driving coil was switched off. During the course of free damped vibration, measurement was made on the time t₀ within which the amplitude reached 1/n of the initial value. The internal friction Q⁻¹ can be obtained from the following equation:

\[ Q^{-1} = \ln \frac{n}{\pi} f_0 t_0, \]

where \( f_0 \) is a frequency of the resonance vibration. The measurements were all carried out at a room temperature of 20°C.

III. Experimental Results and Discussion

Table 1 Chemical composition of specimens, in wt %.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Ni</th>
<th>Cr</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.061</td>
<td>0.62</td>
<td>0.87</td>
<td>0.041</td>
<td>0.011</td>
<td>8.93</td>
<td>18.93</td>
</tr>
</tbody>
</table>


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1. Changes in some properties on annealing after solution treatment

Internal friction of a specimen quenched from 1100°C in water at 20°C was measured at 20°C and then the specimen was annealed for 2 hr at 200°C for the subsequent measurement. In this way, the measurements of internal friction was carried out repeatedly on the same specimen with increasing annealing temperatures. The strain amplitude dependence of the internal friction thus measured is shown in Fig. 1. The strain amplitude dependence of internal friction exists irrespective of the solution treatment (water quenched from 1100°C), precipitation treatment (annealing at 600°C and 700°C) and resolution treatment (annealing at 1000°C). Variations with carbide precipitation owing to annealing at 600°C~800°C are observed in the specific gravity and Huey corrosion resistivity. The decrease in specific gravity at about 700°C may be caused by the volume expansion which is due to the carbide precipitation; it is likely that the precipitation of chromium and iron atoms produces vacancies and hence the specific gravity decreases with increase in the volume of carbide precipitation. The increase in volume loss by corrosion at around 700°C may result from the formation of the local cell which is due to contact of the matrix with carbide as already stated(1). The electrical resistivity, on the other hand, falls after annealing at about 700°C.

2. Electron microscopic observation

A specimen was cooled in water from 1100°C at which the specimen had been solution-treated. The specimen was electrochemically etched in saturated solution of oxalic acid and a carbon replica of the specimen was prepared for electron microscopic observation. The microstructures of specimens subjected to the solution, precipitation and resolution treatment are shown in Photo. 1. In the structures for solution treatment (a) and for annealing at 930°C at which the precipitates are redissolved (e), the precipitates are not observed until annealing at about 700°C ((b), (c) and (d)). Photos. 2, 3 and 4 show transmission electron micrographs of thin films prepared from a bulk specimen solution-treated. Photo. 2 shows dislocations along a grain boundary (G), and a dislocation net work in a twin (T-T) which might be produced by
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Photo. 1 Electron microscopic structures by carbon replica. (×2500)
(a) Quenched in water from 1100°C.
(b) Aged at 600°C for 30 min after the quenching.
(c) Aged at 700°C for 30 min after the quenching.
(d) Aged at 800°C for 30 min after the quenching.
(e) Aged at 930°C for 30 min after ageing at 700°C.

Rapid cooling is observed in Photo. 3. Photo. 4 shows dislocations around twin boundaries (T). A number of the twin boundaries are observed in Photos. 3 and 4 for the specimen solution-treated. It should be noted that in Photo. 4, a striped pattern shown as S is seen between two twin boundaries T and T’. This pattern S is just the same as that observed in a Cu-Al alloy by Swann(8). There are two cases for such a pattern: (1) stacking fault(9) and (2) inclined grain boundary or inclined twin boundary as shown in Fig. 3 (a). It is not clear which case is applicable to the present pattern. The formation of a number of twins in the specimen solution treated corresponds to the high value of the strain amplitude independent internal friction in the quenched state as shown in Fig. 2. The transmission electron microphotographs of specimens annealed for 30 min at 700°C after the solution treatment are shown in Photos. 5, 6 and 7. A white area on the right side (B) in Photo. 5 corresponds to the precipitation product which was removed by an electrochemical polishing. The partial absence of the thin film was confirmed by tilting the specimen and by the dark field observation. Many dislocation lines are connected with a grain boundary (B-B). It should be emphasized that there are parallel lines which consist of dislocation networks. These facts may be caused by a volume expansion which is due to the carbide precipitation at a grain boundary and by the diffusion of the carbides.

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Photos 2, 3, 4: Quenched in water from 1100°C. (×18000)

Photos 5, 6, 7: Annealed at 700°C for 30 min after quenching from 1100°C. (×18000)

Photos 8, 9, 10, 11: Annealed for 30 min at 930°C after annealing at 700°C after quenching from 1100°C. (×18000)
of chromium and iron atoms to the precipitates. Dislocation lines around a grain boundary (B-B) are also observed in Photo. 6. Photo. 7 shows the dislocations connected with carbides.

These phenomena caused by the precipitation of carbides, as mentioned above, have influence on the hydronitric acid resisting property and the decrease in specific gravity with the decreasing concentration of chromium atoms near grain boundaries (10) owing to the precipitation of chromium carbides.

A specimen which had been annealed at 700°C after the solution treatment was again annealed for 30 min at 930°C at which the carbide dissolves in matrix, and then was cooled in air. A thin foil structure of the specimen is shown in Photos. 8, 9, 10 and 11. As already mentioned, the characteristic distribution of dislocations is observed, which results from the carbide precipitation accompanied by volume expansion. It is expected that a characteristic redistribution of dislocations may occur with the redissolution of carbon atoms into the matrix. Photos. 8, 9, 10 and 11 show the dislocations around grain boundaries. The twin boundaries are shown as T in Photo. 8. The piled-up dislocations toward a grain boundary are shown as P in Photo. 11. Although a number of dislocations are observed in Photos. 9, 10 and 11, it is recognized that the distribution and form of the dislocations somewhat differ from those in a precipitated structure. This difference might be attributed to the redissolution of the carbides into the matrix and the slow cooling from a high temperature.

IV. Summary

The experimental results are summarized as follows.
(1) 304 type of 18-8 austenite stainless steel has the strain amplitude dependent internal friction for both solution treatment and precipitation treatment.
(2) In the relation between annealing temperatures after solution treatments and several properties, it was shown that the thickness decrease in Huey corrosion tests became unusually large, and the specific gravity decreased after annealing at about 700°C. The strain amplitude independent internal friction at a lower amplitude decreased with rise in annealing temperature up to 400°C and became constant after annealing at above 500°C.
(3) A large number of twins were observed in the transmission electron microstructures of the specimen solution treated and dislocations around grain boundary. In the specimen annealed at 700°C and 930°C, a few twins and many dislocations were observed, and there was a difference in the distribution and form of dislocations between solution treatment and precipitation treatment.

Acknowledgment

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