Effect of Stacking Fault Precipitation on Hot Deformation of Austenitic Stainless Steel*

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Synopsis

The hot deformation characteristics of Nb-bearing austenitic stainless steel have been studied by means of tensile test and transmission electron microscopy in reflection to the stacking fault precipitation of NbC. Tensile tests were carried out at temperature ranging from 823 to 1 173 K at an initial strain rate of $2.1 \times 10^{-4} \text{ s}^{-1}$.

Although the yield strength of the solution-treated specimen was quite low, remarkable work hardening occurred at temperature ranging from 1 023 to 1 123 K. In this temperature region, a deep ductility trough accompanied by intergranular ductile fracture was also observed. This can be explained in terms of the dynamic precipitation of NbC within the grains as well as on the grain boundaries.

In the aged specimen, on the other hand, the yield strength was much higher than that of solution treated, but the work hardening was less significant. Such a high yield strength is probably due to the refinement of the average effective grain size, i.e., an austenite grain being separated into several regions by the hard platelets of stacking faults with fine NbC particles. The ductility trough in this case was rather shallow because of the reduction of dynamic NbC precipitation.

At temperatures above 1 123 K, the ductility of the solution-treated specimen increased with increasing deformation temperature, but that of the aged specimen was largely reduced. This ductility loss could arise from local dynamic recrystallization in the vicinity of grain boundaries.

I. Introduction

Hot ductility of the solution treated Nb-bearing austenitic stainless steels is markedly reduced by the slow strain rate deformation at temperatures around 1 000 K. This is explained in terms of the precipitation of coarse M_{23}C_6 and NbC particles on the grain boundaries and fine NbC particles within the grains with the formation of Precipitation Free Zone's (PFZ's) along the boundaries. In this case, the mobile dislocations induced mainly at the grain boundaries during hot deformation will be trapped by dynamically precipitated NbC particles within the grains and the mobility of dislocations will be significantly reduced. Thus the fresh dislocations must nucleate at the boundaries in order to continue further deformation. This process will result in the strain concentration within the soft PFZ's along the boundaries and microvoids initiating by the decohesion of the precipitate/austenite interfaces at the boundaries will coalesce, resulting in intergranular ductile fracture.^{1,2)}

The static precipitation of fine NbC particles also occurs during aging at 973 K with the simultaneous formation of extrinsic stacking faults. This phenomenon has been well known as "stacking fault precipitation", and the formation mechanism and its effects

on the mechanical properties have been the subjects of numerous studies.⁴⁻¹¹⁾ In the hot deformation of the specimen aged in such a condition, however, the dynamic precipitation of NbC particles will be largely suppressed and the deformation characteristics will markedly differ from those of solution treated materials.

In the present study, therefore the effects of stacking fault precipitation of NbC on the hot deformation properties of austenitic stainless steel have been examined in comparison with those of dynamic precipitation.

II. Experimental Procedures

The chemical composition of the steel used is given in Table 1. The steel was melted in a 20 kg vacuum induction furnace. The ingot was forged and rolled into the bars of 20 mm diameter. They were solution treated at 1 523 K for 30 min and quenched into water. Some of the solution treated bars were aged at 973 K for 100 h. From both the solution treated and the aged bars, tensile specimens were machined in the direction parallel to the rolling direction. The gauge length, the diameter and the fillet radius of the tensile specimens were 40, 6 and 5 mm, respectively. Hot tensile tests were carried out by using Instron-type tensile machine in a flowing argon atmosphere. The specimens were heated at a rate of $100 \text{ K} \cdot \text{min}^{-1}$ to the temperatures ranging from 823 to 1 173 K in the infrared imaging furnace attached to the tensile machine. After keeping for 10 min, they were isothermally pulled at a constant crosshead speed (the initial strain rate was $2.1 \times 10^{-4} \text{ s}^{-1}$) until the final fracture occurred.

The fractured specimens were examined by means of transmission electron microscopy (TEM) and scanning electron microscopy (SEM). Thin foils were prepared by a conventional twin jet electropolishing method with using the disk machined from the fracture specimens near the fracture surface, and were observed in a Hitachi H-700H microscope operating at 200 kV.

Table 1. Chemical compisition of the material used. (wt%)

C	Si	Mn	P	S	Ni	Cr	Nb
0.048	0.50	1.35	0.004	0.005	11.03	18.59	0.46

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III. Experimental Results

1. Tensile Properties

Figure 1 illustrates stress-strain curves in the solution treated and the aged specimens. Although the solution treated specimens exhibit rather low yield stress, significantly large work hardening occurs during the following deformation in the temperature range from 823 to 1 023 K. Such a high work hardening rate decreases with raising deformation temperature above 1 073 K and no work hardening is observed at 1 173 K. The yield strengths of the aged specimens are higher than those of the solution treated, but the work hardening rates of the formers are much smaller than those of the latters. It should be noted that the serrations of the stress-strain curves can clearly be observed in the case of deformation temperatures between 923 K and 823 K in both cases.

Variations of 0.2 % proof stress (YS), the ultimate tensile stress (UTS) and the reduction of area (RA) with deformation temperature are illustrated in Fig. 2. The YS's of the aged specimens are much higher than those of the solution treated but the difference between them decreases with raising the deformation temperature.

Although the RA's of both the specimens decrease with raising the deformation temperature up to 1 000 K, the decreasing rate for the aged specimens is much smaller than that for the solution treated. The RA of the as-solution-treated specimens increases gradually with deformation temperature above 1 000 K while that of the aged shows the maximum value at 1 073 K and then decreases with the further increase of deformation temperature.

Figure 3 shows variations of work hardening rate at 4 % strain, $(\partial\sigma/\partial\varepsilon)_{\varepsilon=0.04}$, with deformation temperature. The work hardening rate of the solution treated specimen is much larger than that of the

aged in all the temperature range examined, and the rates for both specimens decrease with increasing deformation temperature above 900 K.

2. Fractography

Photograph 1 shows the effect of deformation temperature on the fracture mode of the solution treated specimen. Large dimples arising from transgranular ductile fracture can be seen in the specimen deformed at 873 K, while the fraction of intergranular fracture increases with raising deformation temperature (Photo. 1(b)). The detailed examination of the intergranular facets reveals that they can be classified into two types: the intergranular facets with small dimples at 1 073 K and those with wavy wrinkles at 1 173 K as in Photos. 1(c) and (d), respectively. Such wavy patterns on the intergranular facets tend to increase with increasing deformation temperature.

Photograph 2 shows the effect of deformation

Fig. 1. Stress-strain curves.

Open: Solution treated, Solid: Aged

Fig. 2. Variation of tensile properties with deformation temperature.

Fig. 3. Variation of work hardening rate with deformation temperature.

temperature on the fracture mode of the aged specimens. The specimens deformed at temperature below 873 K show the transgranular mode of fracture similar to that of the solution treated (Photo. 2(a)), and the fraction of intergranular fracture increases with raising deformation temperature. Although

some transgranular ductile facets are observed in the specimen deformed at 1 073 K (Photo. 2(b)), fracture mode in the specimen deformed at temperatures above 1 123 K is mostly intergranular as in Photo. 2(c), and wavy wrinkles can be observed on the intergranular facets (Photo. 2(d)).

3. Metallography

The grain size of the solution treated specimen was rather coarse and the mean linear intercept of the boundaries was $83\ \mu\text{m}$. Photograph 3 shows the microstructures of the aged specimen. Rather coarse M_{23}C_6 particles are formed discretely on the random grain boundaries as in Photos. 3(a) and (b). These M_{23}C_6 particles exhibit the cube-cube crystallographic orientation relationship with one of the γ grains, and grow into the other grain without any relationship as can be seen in the dark field image (Photo. 3(b)). While particles formed on the incoherent twin boundaries are much finer in size as in Photo. 3(c) and the precipitates cannot be observed on the coherent twin boundaries. It is interesting to note that a number of stacking faults with NbC precipitation forms closely to the coherent twin boundaries as Photo. 3(d).

Fine NbC particles also precipitate on the dislocations introduced by the growth of M_{23}C_6 particles as indicated by arrows in Photo. 3(b). Although NbC particles can be observed on neither the random boundary nor the incoherent twin boundaries, they frequently form on the coherent twin boundaries.

Photograph 4 illustrates an example of close examination of the stacking faults with NbC precipitates. Four different variants of stacking faults can clearly be recognized. From the dark field illumination with using a $\{111\}_\gamma$ and a $\{111\}_{\text{NbC}}$ reflections, these stacking faults were confirmed to be parallel to the $\{111\}$ planes (Photo. 4(b)). Each austenite grain is divided into much smaller regions by these stacking faults. High density of fine NbC particles

on the stacking faults can be seen. It should be noted that the diffraction contrast from some parts of the stacking faults disappears and only the weak thickness fringes due to the NbC particles on the

(a) Bright field image (b) Dark field image
Photo. 4. TEM micrographs showing the stacking fault precipitation in the aged specimen.

(a), (b) Random grain boundary
(c) Twin boundary
(d) Coherent twin boundary

Photo. 3. TEM micrographs showing M_{23}C_6 and NbC precipitations at grain boundaries in the aged specimen.

previously existed stacking faults can be seen as indicated by circle in Photo. 4(a).

Photographs 5 to 7 show the microstructures of the solution treated specimen deformed at 873, 1 073 and 1 173 K, respectively. In the specimen deformed at 873 K, stacking faults form frequently with high density of dislocations, and no precipitates can be observed (Photo. 5). The deformation at 1 073 K, where deep ductility trough appears, produces very fine NbC particles precipitated dynamically on the dislocations within the grains and rather coarse particles on the grain boundaries with the precipitation free zones (PFZ's) along them as in Photos. 6(a) and (b). Photographs 6(c) and (d) show the trace of grain boundary migration. The dark field image using a reflection from the grain in the lower part in Photo. 6(c) indicates that the NbC particles within the upper grain aligned almost parallel to the boundary and were in the cube-cube orientation

relationship¹²⁾ with the lower grain (Photo. 6(d)). This suggests that the particles had formed on the grain boundary at the initial position in contact with the lower grain and then the boundary migrated with leaving the row of the precipitates.

In the specimen deformed at 1 173 K, a typical recovered structure consisting of dislocation networks can be observed as in Photo. 7.

Photograph 8 shows the microstructure of the aged specimen deformed at 873 K. In this case the dislocation density is quite high and formation of dislocation cell structures can be clearly seen. The stacking faults were observed to meander due to the deformation (the arrow in Photo. 8(a)).

Although small amounts of dynamically precipitated NbC can be seen within the grains in the specimens deformed at higher temperatures, most of the precipitates are on the stacking faults which formed prior to the deformation similarly to the case of

Photo. 5. TEM micrograph of the solution-treated specimen deformed at 873 K.

Photo. 7. Dislocation network of solution-treated specimen deformed at 1 173 K.

(a), (c) Bright field image (b), (d) Weak-beam dark field image

Photo. 6. NbC precipitation at random grain boundary (a, b) and at twin boundary (c, d).

deformation at 873 K (Photo. 9). It should be noted that the dislocation density of the specimen fractured at 1 173 K was quite low because the final fracture occurred at the early stage of straining.

(a) Bright field image (b) Dark field image

Photo. 8. Deformation structure of the aged specimen deformed at 873 K.

IV. Discussion

1. $M_{23}C_6$ and NbC Precipitation during Aging

Aging at 973 K after the solution treatment at 1 523 K yields both $M_{23}C_6$ and NbC precipitations within the grains as well as on the grain boundaries. The preferential nucleation site for $M_{23}C_6$ is of course the random grain boundaries, but the coherent twin interfaces are almost free from the precipitates as already reported by other investigations.^{13,14)} Whereas NbC particles do not precipitate on the grain boundaries except the coherent twin interfaces and majority of them is in the form of the stacking fault precipitation within the grains. These facts suggest that $M_{23}C_6$ particles can precipitate more easily at the incoherent grain boundaries than NbC particles, and that, once $M_{23}C_6$ particles nucleate, their growth will continuously consume C atoms in the vicinity of the boundaries, which may suppress precipitation of NbC. It is also interesting to note that the stacking fault precipitation of NbC particles cannot be observed in the vicinity of the random grain boundaries where the precipitation of $M_{23}C_6$ particles likely occurs prior to that of NbC (Photos. 3(a), (b)). This indicates that depletion of Cr atoms by precipitation of $M_{23}C_6$ particles increases the stacking fault energy and suppresses the stacking fault precipitation of NbC. In fact, the formation of PFZ along the boundaries supports this model. Such effects of alloying elements on the stacking fault precipitation have already been reported by Harding and Honeycombe⁵⁾ and Silcock *et al.*,¹⁵⁾ who demonstrated that the addition of U or Si accelerates the stacking fault precipitation in the austenitic stainless steels by lowering the stacking fault energy.

(a), (c) Bright field image (b), (d) Weak-beam dark field image

Photo. 9. Deformation structure of the aged specimen at 1 073 K (a, b) and 1 173 K (c, d).

2. Effects of NbC Precipitation on Tensile Properties

The aged specimen exhibits much higher yield strength due to the stacking fault precipitation of NbC at temperatures below 1 050 K. Dispersion of NbC particles in this case, however, is quite inhomogeneous, and the mean interparticle spacing is quite large, $0.5 \sim 1 \mu\text{m}$, thus the strengthening cannot be explained in terms of Orowan's mechanism which strongly depends on the interparticle spacing. It should be noted that stacking faults with densely distributed NbC particles can act as hard platelets within the grains. These platelets, lying on four $\{111\}$ planes, will separate a grain into much smaller regions as has been suggested by Harding *et al.*,⁵⁾ and will act as the barriers for dislocation motion. In fact, a number of dislocations clinging to these faults can frequently be observed (Photos. 8 and 9). It has commonly been accepted that flow stress during small strain rate deformation at elevated temperatures can be determined mainly by the average size of either subgrains or recrystallized grains forming during deformation and that the initial grain size dependence of flow stress is less significant. In the case of yielding at temperatures where the recrystallization does not occur, however, the initial grain size should dominate the yield stress because the subgrains have not well developed when yielding initiates. Thus it is of interest to argue the increase of yielding stress with the grain size effect. If the areas separated by the hard platelets of stacking fault precipitation can act as small grains, the yield stress increase can reasonably be explained by Hall-Petch relationship^{16,17)} since the effective grain size, $0.91 \mu\text{m}$, is about 10^{-2} times smaller than the grain size of the solution treated specimen.

The work hardening rates of the solution treated specimen at temperature between 900 K and 1 100 K on the other hand are much larger than those of the aged specimens. This is probably due to the dynamic precipitation of NbC particles, *i.e.*, the dislocations nucleating at the grain boundaries will be subsequently pinned by the fine precipitation of NbC particles on them and further dislocation multiplication will be required to continue the deformation, resulting in a marked increase of dislocation density. In the aged specimens, on the other hand, such effect of dynamic precipitation is negligibly small, because most NbC particles have precipitated in the form of the stacking fault precipitation prior to the deformation. Therefore the dislocations emerging at the boundaries will be hardly pinned down by fine NbC particles and can move more freely. This will lead to the small work hardening.

Hot ductility is also closely related to the precipitation of NbC. In the solution treated specimens the ductility is reduced by the deformation at temperature ranging from 950 to 1 150 K. This ductility trough can be explained as follows.

Since the matrix is strengthened by the dynamic precipitation of fine NbC particles, the strain will concentrate within the softer PFZ's along the grain boundaries. Then microvoids will be formed by the

decohesion of the interfaces between the matrix and the coarse precipitates such as M_{23}C_6 and NbC on the boundaries. The coalescence of them will lead to intergranular ductile fracture in keeping with the observation in Photo. 1(c). In the aged specimens, the ductility trough is rather shallow. This can be explained mainly by the decrease of NbC dynamic precipitation, because of static stacking fault precipitation of NbC prior to the deformation.

Although the ductility of the solution treated specimens is improved by raising the deformation temperature up to 1 173 K, that of the aged specimens is markedly reduced above 1 123 K, which accompanies with the increase in fraction of the intergranular fracture along the original boundaries. The detailed mechanism for it is not clear at present, but it is probably due to the dynamic recrystallization in the vicinity of the grain boundaries. The intergranular mode of fracture with wavy wrinkles may be the results of such a microstructural change near the grain boundaries.

V. Conclusions

(1) Although the yield strength of the solution-treated specimen is quite low, remarkable work hardening occurs during the deformation at temperature ranging from 1 023 to 1 123 K. In this temperature region, deep ductility trough accompanying the intergranular ductile mode of fracture is observed. These facts can be explained in terms of the dynamic precipitation of NbC within the grains, the PFZ's formation and the grain boundary precipitation of coarse M_{23}C_6 and NbC particles.

(2) The yield strength of the aged specimen is considerably higher than that of the as-solution-treated specimen but the work hardening rate during the deformation is less significant. The increase of the yield strength by aging can be explained by the decrease in the effective grain size by the presence of the platelets comprising the stacking fault precipitation of fine NbC particles. The ductility trough in this case is rather shallow because of the decrease of the dynamic NbC precipitation.

(3) At temperatures above 1 123 K the ductility of the solution-treated specimen increases with increasing deformation temperature, but that of the aged specimen is largely reduced. This is probably due to the microstructural change in the vicinity of the grain boundaries.

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