Nanoscopic Strength Analysis of Work-Hardened L-Grade Austenitic Stainless Steel, 316(NG)*

Nobuo Nagashima¹ and Saburo Matsuoka²

¹National Institute for Materials Science (NIMS), Tukuba 305-0047

²Graduate School of Engineering, Kyushu University, Fukuoka 819-0395

Stress corrosion cracking (SCC) occurs in shrouds and piping of L-grade austenitic stainless steels at nuclear power plants. A work-hardened layer, where the transgranular SCC initiates, is considered to be one of the probable cause for this occurrence. In order to clarify the microstructural characteristics of work-hardened layer at the surface of shrouds or piping, the strength analysis of L-grade austenitic stainless steel, 316(NG), rolled at the reduction in area, RA , of 10, 20, 30, 40 and 50% at room temperature were conducted on a nanoscopic scale, using an ultra-microhardness tester, TEM and SEM. TEM and SEM observation showed that the microstructural parameters are the dislocation cell size, d_{cel} , coarse slip spacing, l_{csl} , and austenitic grain size, d_g . Referring $10d_{cel}$ and $10l_{csl}$, Vickers hardness, Hv , corresponding to macro strength was expressed as $Hv = Hv_{bas}^* + Hv_{sol}^* + Hv_{dis}^* + Hv_{cel}^* + Hv_{csl}^*$. Hv_{bas}^* ($= 100$) is the base hardness, Hv_{sol}^* is the solid solution strengthening hardness, Hv_{dis}^* is the dislocation strengthening hardness in the dislocation cell, and Hv_{cel}^* and Hv_{csl}^* are the fine grain strengthening hardness due to the dislocation cell and coarse slip. Hv_{sol}^* was about 50, independently of RA . Hv_{dis}^* was zero at $RA < 30\%$ and increased at $RA > 30\%$. Hv_{cel}^* and Hv_{csl}^* increased with increasing in RA and were kept constant at about 50 and 120 at $RA = 20$ and 30% , respectively. It was suggested from these results that all dislocations introduced by rolling might be dissipated for the creation of dislocation cells and coarse slips at $RA < 30\%$ and that the microstructure contributing to the fine grain strengthening due to the dislocation cell and coarse slip might be accomplished at $RA = 30\%$. The dislocation strengthening in the dislocation cell might begin to operate at $RA > 30\%$. [doi:10.2320/matertrans.47.2326]

(Received May 25, 2006; Accepted July 31, 2006; Published September 15, 2006)

Keywords: BWR(Boiling Water Reactor), SCC(Stress Corrosion Cracking), L-grade austenitic stainless steel, 316(NG), work hardening, nanoindentation, microstructural parameters

1. Introduction

Since the 1970's, there have been many cracking incidences in boiling water reactors (BWR) pressure boundary components, such as primary loop re-circulation (PLR) pipes made with stainless steels. The cracks grew along heat-affected zones, and their fractographic features were characterized by intergranular stress corrosion cracking (IGSCC). The cracking was caused by typical weld sensitization in heat affected zones due to Cr depletion along grain boundaries.¹⁾ L-grade austenitic stainless steel was developed to control the formation of Cr carbides.¹⁾ However, recently, SCC has been observed in L-grade austenitic stainless steel in core shrouds and PLR pipes in Japanese BWR plants.²⁾ An investigation of samples taken from the nuclear power plants showed that the cracking originated in the surface unit of the material, and that transgranular stress corrosion cracking (TGSCC) had occurred. TGSCC occurred in a surface heavily cold worked by machining and in an area highly strained by weld deformation. This SCC mechanism has not been fully explained.

The authors have developed ultra micro-hardness tester on the basis of an atomic force microscope (AFM).³⁾ We have also developed an empirical equation for converting an indentation force–penetration depth (F – h) curve to the Vickers hardness, Hv^* , by using a single metal crystal as a reference sample.^{4,5)} This makes it possible to obtain hardness profiles in regions ranging in size from the nano scale to the macro scale by correlating the hardness obtained by using the tester and the empirical equation with the macroscopic

hardness obtained by Vickers hardness testing. We have also made it possible to separate the various strengthening factors associated with solution, dislocation, particle dispersion, and grain boundaries (fine grain) by determining structural parameters from AFM structural observations and by correlating a hardness profile with these structural parameters. We named this set of processes “nano–meso–macro strength analysis”⁶⁾ and applied the technique to the analysis of the strength mechanism of steels, such as 1- μ m ultra-fine-grain ferrite steel,⁵⁾ Low carbon ferrite–bainite steel,⁷⁾ ferrite–pearlitic steel,⁸⁾ and tempered martensitic steel.⁹⁾

In this study, we performed a nano–meso–macro strength analysis on cold-rolled L-grade austenitic stainless steel SUS316 (NG) as part of an investigation of the causes of TGSCC in the work-hardened layer of shrouds and PLR pipes. We also compared the results of our analysis with those of a previous investigation on the occurrence of SCC in cold-rolled austenitic stainless steel.

2. Experimental Method

2.1 Material information

The test material used was L-grade austenitic stainless steel SUS316 (NG), whose chemical composition is shown in Table 1. Nitrogen (0.109 mass%) is added to the material to compensate for the reduction in strength caused by the L-grade content. The steel was cold rolled at room temperature

Table 1 Chemical composition of SUS316(NG) steel (mass%).

C	Si	Mn	P	S	Ni	Cr	Mo	N
0.007	0.28	1.49	0.033	0.005	12.23	16.58	2.07	0.109

*This Paper was Originally Published in Japanese in J. Japan Inst. Metals
 69 (2005) 472–480.

Fig. 1 Nominal stress-strain curves of rolled SUS316(NG) steels.¹⁰⁾

Table 2 Tensile properties of rolled SUS316(NG) steels.¹⁰⁾

Rolling at ~25°C		$\sigma_{0.2}$	σ_B	δ_U	δ_T	ϕ	HV
Reduction area, RA (%)	True Strain	(MPa)	(MPa)	(%)	(%)	(%)	
0	0	285	589	55.7	78.2	83.7	144
10	0.104	508	659	37.9	57.6	82.1	226
20	0.223	651	751	19.9	44.1	82.3	278
30	0.358	773	858	2.2	29.9	80.8	314
40	0.513	846	968	1.6	22.5	78.6	341
50	0.693	907	1041	1.6	17.4	76.4	358

to give a reduction in area ($RA = (A_0 - A)/A_0$) of 10, 20, 30, 40, or 50%; each cold-rolled sheet had thickness of 10 mm and a width of 40 mm. Figure 1 shows the stress-strain curve obtained for each rolled steel.¹⁰⁾ Table 2 shows the mechanical properties, 0.2% yield strength ($\sigma_{0.2}$), tensile strength (σ_B), uniform elongation (δ_U), total elongation (δ_T), and reduction in area (ϕ). The table also shows the Vickers hardness (HV) measured with an applied indentation force of 196.1 N. The Vickers hardness of the base metal was 144, whereas that of steel with a reduction in area of 50% reached 356.

2.2 Structure observation method

The structure of cold-rolled specimens was observed by field emission scanning electron microscopy (FE-SEM) at an acceleration voltage of 20 kV. In addition, a field emission transmission electron microscopy (FE-TEM) observation was carried out at an acceleration voltage of 300 kV to investigate the details of the slip bands and dislocation cells induced by cold rolling. The FE-SEM observation was carried out on a cross section (Face C) of the specimen perpendicular to the rolling direction.

Samples for SEM were first ground using emery papers of various grinds from 230 to 600 grit and then etched in a solution of 20 vol% of HNO_3 , 60 vol% HCl and 20 vol% of H_2O at 293 K. Samples for TEM obtained from thin slices with 0.3 mm thickness were firstly mechanically thinned to 30 μm and then electrolytically polished in a solution containing 5 vol% perchloric acid and 95 vol% acetic acid,

using a twin jet apparatus at a potential of 20 V and a temperature of 298 K.

2.3 Hardness measurement method

In hardness measurement, an ultra micro-hardness tester (indentation force: 0.1 to 5 mN), a micro-hardness tester (1 to 2000 mN), and a Vickers hardness tester (0.196 to 490 N) were used to investigate the dependence of the hardness on the indent size over the nano-, meso-, and the macro-sized regions, respectively. The hardness values obtained by using the three hardness testers are called to as the nano hardness, the meso hardness, and the macro hardness, respectively. Hardness measurements were made on Face C, as in the structural observation. The samples, cut to a thickness of 2 mm, were buffed and then electrolytically polished in a solution of 8 vol% perchloric acid, 10 vol% 2-butoxyethanol, 70 vol% ethanol, and 12 vol% distilled water, at a potential of 40 V and a temperature of 273 K. Electropolishing of the surfaces was intended to remove work-hardened layers formed during the processes from cold rolling through buffing. The ultra micro-hardness test was carried out by using the AFM ultra micro-hardness tester with special levers developed by the authors.⁴⁾ Two types of special levers were used that had triangular pyramidal diamond indenters with apical angles of 60 and 115°, respectively, in the lever center. The lever with the triangular pyramidal indenter with an apical angle of 60° is referred to as lever No. 1 and the one with an apical angle of 115° as lever No. 2. The micro-hardness test was carried out by using a Shimadzu DUH201 instrument with a triangular pyramidal indenter with an apical angle of 115°. An Akashi micro Vickers hardness tester was used for the Vickers hardness test. The diamond indenter was of a quadrangular pyramidal shape with a face angle of 136°.

According to the definition of the Vickers hardness, the hardness calculated by dividing the indentation force by the surface area of the indent. In the ultra micro-hardness test, this hardness calculated shows a size effect in which the hardness increases with decreasing indentation depth.¹¹⁾ It is suggested that this size effect occurs because the radius of the indenter tip has an effect that appears as a result of the shallow indentation depth.¹¹⁾ To eliminate this effect, we have proposed the following empirical equation to convert indentation force-depth (F - h) curves (see Fig. 7) obtained in ultra micro-hardness tests to the Vickers hardness Hv^* , using several electrolytically polished single metal crystals as reference specimens.^{4,5)}

$$Hv^* = \left\{ \frac{F}{p(h+q)^2} \right\}^n \quad (1)$$

where p , q , and n are constants and need to be specified for each indenter. This approach can be applied to F - h curves (see Fig. 8) obtained in a micro-hardness test.^{8,9)} Table 3 shows p , q , and n values for each of the pyramidal indenters.

3. Experimental Results

3.1 Structural observation

Figure 2 shows an SEM image of the microstructure of the SUS316 (NG) base metal (*i.e.*, a sample with a reduction in

Table 3 Constants p , q and n in an experimental equation, $Hv = \{F/[p(h+q)^2]\}^n$

Hardness tester	Indenter	p	q	n
Nano-micro	60° indenter, No. 1 lever	9.011×10^{-5}	66.6	1.262
	115° indenter, No. 2 lever	6.886×10^{-4}	49.51	1.151
Micro	115° indenter	5.530×10^{-7}	255.2	1.175

Fig. 2 SEM image of microstructure in parent SUS316(NG) steel.

Fig. 3 SEM images of microstructure in 30% rolled SUS316(NG) steel. (a) Low magnification (b) High magnification.

Fig. 4 TEM images of 30% rolled SUS316(NG) steel. (a) Low magnification (b) Medium magnification (c) High magnification.

area of 0%). The SEM image shows that the austenitic grain size is about 150 μm.

Figures 3 and 4 show SEM and TEM images of the microstructure of a sample with a reduction in area of 30%, it's a typical example for cold-rolled SUS316 (NG) steel. Figure 5 shows the slip deformation model¹²⁾ observed on the

Fig. 5 Slip deformation model.¹²⁾

surface of aluminum, which has the same face-centered cubic structure as SUS316 (NG) steel.

Figure 5 shows that large slip lines with a height difference of 200 nm are formed at a strain ϵ of less than 10%. As the strain increases, the spacing between the large slip lines decreases and saturates at a spacing of 2 μm at a strain of 10%. As the strain increases beyond 10%, 20 nm-spaced fine slip lines are formed in the root of the 2 μm -spaced slip lines. As shown by the SEM image in Fig. 3 and the TEM image in Fig. 4(a), 2 μm -spaced large slip lines are also formed in the SUS316 (NG) specimen with a reduction in area of 30% (a true strain of 35.8%). In addition, the TEM image in Fig. 4(b) shows that 20 nm-spaced fine slip lines are formed in the root of the 2 μm -spaced slip lines.

The TEM images in Figs. 4(b) and 4(c) show the formation of dislocation cells (subgrains). Fig. 6 shows the relationship between the dislocation cell size and the reduction in area. In each rolled specimen, five dislocation cells were randomly selected, and their average size was used as the dislocation cell size. Figure 6 also shows the tensile-test results for low-alloy steel obtained by Bassim *et al.*¹³⁾ They cut a fractured tensile-test specimen at several positions in the reduction area, and performed TEM observations of the cross-section of each sample cut. They then calculated the reduction in area by using the diameter of each sample and the initial diameter of the test specimen. This showed that the dislocation cell size decreases with increasing reduction in area and becomes constant at a spacing of nearly 0.4 μm for a reduction in area of more than 20%. In comparison with this result, the dislocation cell size in the cold-rolled SUS316 (NG) steel is

Fig. 6 Relationships between dislocation cell size and reduction in area for rolled SUS316(NG) steels.

as small as 0.15 to 0.25 μm , and the reduction in area giving a constant dislocation cell size is estimated to be 10 to 20%.

As mentioned in the introduction, structural parameters play an important role in the nano-meso-macro strength analysis.⁵⁻⁹⁾ On the basis of the above results, the structural parameters of cold-rolled SUS316 (NG) steel are an austenitic grain size of 150 μm , slip spacings of 2 μm and 20 nm, and dislocation cell sizes of 0.15 to 0.25 μm .

3.2 Hardness measurement

Figure 7 shows typical F-h curves obtained using the AFM ultra micro-hardness tester. In order to show the results clearly, F-h curves for the base metal and for specimens with reductions in area of 10, 30, and 50% are shown in Fig. 7(a), and those for the base metal and specimens with reductions in area of 20 and 40% are shown in Fig. 7(b). In the F-h curves, if the indentation force is the same, the material is harder for shallower indentation depths. Therefore, the results of hardness measurement in the nano region in Fig. 7 show that specimens with reductions in area of 10, 20, or 30% are as hard as the base metal and those with reductions in area of 40 or 50% are harder than the base metal. As shown in Table 2, the Vickers hardness increases with increasing reduction in area. These results mean that the dependence of the hardness on the reduction in area differs between the nano and the macro regions.

Figure 8 shows typical F-h curves obtained by using the micro-hardness tester. A comparison between Fig. 7 and Fig. 8 shows that the maximum indentation force is 500 times larger in the micro-hardness test than in the ultra micro-hardness test. In the meso-hardness obtained micro-hardness tester, the indentation depth decreases and the hardness increases in proportion to the reduction in area. This tendency agrees with that of the macro-hardness obtained with the micro-Vickers hardness tester (Table 2).

Figure 9 shows the indent size dependency of hardness over the nano-meso-macro regions. Fig. 9(a) shows the results for the base metal and specimens with reductions in area of 10, 30, and 50%, and Fig. 9(b) shows those for the base metal and specimens with reduction in area of 20 and 40%. The indent size on the horizontal axis is expressed as

Fig. 7 Force–penetration depth curves obtained by ultra-microhardness tester for parent and rolled SUS316(NG) steels. (a) Reduction in area of 0, 10, 30 and 50% (b) Reduction in area of 0, 20 and 40%.

Fig. 8 Force–penetration depth curves obtained by microhardness tester for parent and rolled SUS316(NG) steels. (a) Reduction in area of 0, 10, 30 and 50% (b) Reduction in area of 0, 20 and 40%.

the square root of the projected area of the indent in order to show test results for indenters of different shapes in a unified manner. The nano-hardness obtained in the ultra microhardness test, the meso-hardness obtained in the microhardness test, and the macro-hardness obtained in the Vickers hardness test are shown for indent sizes smaller than $2\mu\text{m}$, $2\text{--}20\mu\text{m}$, and larger than $20\mu\text{m}$, respectively. The nano-hardness and the meso-hardness were converted to the Vickers hardness by applying eq. (1) to the F – h curves shown in Figs. 7 and 8. The structural parameters in Fig. 9 are the dislocation cell size $d_{cel} = 0.15$ to $0.25\mu\text{m}$, $10d_{cel}$, which is 10 times d_{cel} , and $10l_{csl}$, which is 10 times the $2\text{-}\mu\text{m}$ large slip spacing l_{csl} . d_{cel} is averaged at $0.2\mu\text{m}$. $10d_{cel}$ or $10l_{csl}$ shows the position where fine grain strengthening by dislocation cells or slip spacing saturates and the hardness becomes constant. This will be explained in detail in the Discussion section below.

In Fig. 9, the hardness of the base metal is nearly constant over the nano and macro regions, with a value of 150. The hardness of the cold-rolled specimens with reduction in area of 10–50% increases with increasing indent size. The hardness appears to change hierarchically in three steps corresponding to d_{cel} , $10d_{cel}$, and $10l_{csl}$. In the nano-hardness region, in the first step, the hardness of the specimens with reductions in area of 10–30% corresponds to that of the specimen with reduction in area of 0% (the base metal), whereas the hardness of specimens with reductions in area of 40 and 50% is greater than that of the base metal. The meso-hardness in the second step and the macro-hardness in the third step increase linearly with increasing reduction in area.

4. Discussion

The authors first applied the nano–meso–macro strength

Fig. 9 Relationships between converted hardness or Vickers Hardness and indent size for parent and rolled SUS316(NG) steels. (a) Reduction in area of 0, 10, 30 and 50%. (b) Reduction in area of 0, 20 and 40%.

Fig. 10 Relationship between converted hardness or Vickers hardness and indent size for 1.2 μm fine-grained ferritic steel.⁶⁾

analysis to 1- μm ultra fine-grain ferrite steel.⁶⁾ Figure 10 shows the indent size-dependency of the hardness of 1- μm ultra fine-grain ferrite steel and 30- μm large-grain ferrite steel. The side length l_h of a triangular indent and the vertical angle length l_d of a rectangular indent were used to represent the indent size in the ultra micro-hardness test and in the

Fig. 11 Relationships between converted hardness or Vickers hardness and indent size for parent and 50% rolled SUS316(NG) steels.

Vickers hardness test, respectively. The hardness of the large-grain ferrite steel is independent of the indent size and nearly corresponds to the base hardness of iron, which has a value of 100. The hardness of the ultra fine-grain ferrite steel begins to increase near the grain diameter d_α and saturates at $10d_\alpha$. It then reaches the macro-hardness obtained in the Vickers hardness test. Miyazaki *et al.*¹⁴⁾ investigated the effect of the ratio of the crystal grain size d to the thickness B on the tensile strength by using plate specimens of Cu, Cu-13at% Al, and Fe, and showed that the tensile strength saturates when the thickness is nearly five times the grain size, *i.e.* $B = 5d$, and coincides with the macro-characteristics. Their results, as well as the finding that a plastic zone twice the indent size is formed around the indent,¹⁵⁾ can explain the hardness saturation at $10d_\alpha$ in ultra fine-grain ferrite steel; the results also show that d_α and $10d_\alpha$ are important structural parameters. Based on these two structural parameters, the macro-hardness of ultra fine-grain ferrite steel or the Vickers hardness H_v can be split up into the base hardness H_v^{*bas} of iron, the solid solution strengthening hardness H_v^{*sol} , the dislocation strengthening hardness H_v^{*dis} , and the fine grain strengthening hardness H_v^{*fer} .

In particular, the locking parameter k in the following Hall-Petch equation was obtained from the fine grain strengthening hardness H_v^{*fer} , and the value agreed with that from the tensile test.⁶⁾

$$\sigma = \sigma_0 + \frac{k}{d_\alpha^{1/2}} \quad (2)$$

This analysis was applicable to several tempered martensitic steels with different strengths.⁹⁾

A figure similar to Fig. 10 was created by using the results for the SUS316 (NG) steel base metal and the specimen with a reduction in area of 50%. This is shown in Fig. 11. In Fig. 11, the structural parameters are the dislocation cell size d_{cel} , $10d_{cel}$, which is 10 times d_{cel} , and $10l_{csl}$, which is 10 times the large slip spacing l_{csl} .

The Vickers hardness H_v of the specimen with a reduction in area of 50% is split up into the base hardness H_v^{*bas} of iron, the solid solution strengthening hardness H_v^{*sol} , the dislocation strengthening hardness H_v^{*dis} , the fine grain strengthening hardness resulting from dislocation cells H_v^{*cel} , and the fine grain strengthening hardness resulting from the large slip line spacing H_v^{*csl} .

In a base metal with a solution-treated austenitic structure there are almost no dislocations or precipitations and therefore dislocation strengthening and precipitation strengthening can be ignored. Also, because the grain size is as large as 150 μm , grain strengthening barely operates. Therefore, the difference between the hardness of the base metal and the base hardness of iron Hv^*_{bas} is thought to be accounted for by the solid solution strengthening hardness Hv^*_{sol} . Of course, the solid solution strengthening hardness also exists in the specimen with a reduction in area of 50%. According to Fig. 2.2 in Pickering's book,¹⁶⁾ the chemical components that make significant contributions to Hv^*_{sol} are C, N, and Mo in Table 1. When cold-rolling is performed, the number of dislocations increases, and therefore the difference in hardness between the base metal and the first flat section in the specimen with a reduction in area of 50% is thought to be the result of the dislocation strengthening hardness Hv^*_{dis} . As the indent size increases, the hardness of the specimen with a reduction in area of 50% begins to increase near d_{cel} and saturates at $10d_{cel}$. This tendency agrees with that of the ultra fine-grain ferrite steel in Fig. 10, suggesting that the difference in hardness between the first and second flat sections is the fine grain strengthening hardness resulting from dislocation cells, Hv^*_{cel} . Eventually, the hardness of the specimen with a reduction in area of 50% reaches the macro-hardness at $10l_{csl}$. This suggests that the difference in hardness between the second and third flat sections is slip-related hardness. Thompson *et al.*¹⁷⁾ found that stress-strain curves for copper, aluminum, and brass can be well expressed by the following equation, which includes the contribution of slip lines in the Hall-Petch eq. (2).

$$\sigma = \sigma_0 + \left(1 - \frac{\lambda}{d}\right) \left(\frac{k_1}{\lambda}\right) + \frac{\lambda}{d} \left(\frac{k_2}{d^{1/2}}\right) \quad (3)$$

where d is the crystal grain size, and λ is the slip line length and is proportional to the slip spacing. For smaller strains, λ approaches d , and consequently eq. (3) becomes the Hall-Petch eq. (2). For larger strains, λ becomes smaller, resulting in an increased effect of the slip line on the strength. With these points in mind, the difference in hardness between the second and third flat sections was determined to be the fine grain strengthening hardness resulting from the large slip line spacing, Hv^*_{csl} .

The relationships of the dislocation strengthening hardness Hv^*_{dis} , the fine grain strengthening hardness resulting from dislocation cells Hv^*_{cel} , and fine grain strengthening hardness resulting from the large slip line spacing Hv^*_{csl} to the reduction in area were obtained by using Figs. 9 and 11 and are shown in Figs. 12–14. The average values in Fig. 9 were used to obtain these hardness values. Of these three strengthening factors, the fine grain strengthening by dislocation cells is the largest, the fine grain strengthening by large slip line spacing is the second largest, and the dislocation strengthening is the smallest. The three strengthening factors are all related to dislocation and, could all be taken as dislocation strengthening. However, as described below, the strength-production mechanism in cold-rolled SUS316 (NG) steel can be clearly explained by splitting the strengthening into the three factors and discussing them individually.

Fig. 12 Dislocation strengthening hardness, Hv^*_{dis} , against reduction in area.

Fig. 13 Fine-grain strengthening hardness due to dislocation cell, Hv^*_{cel} , against reduction in area.

Fig. 14 Fine-grain strengthening hardness due to coarse slip spacing, Hv^*_{csl} , against reduction in area.

In Fig. 12, the dislocation strengthening hardness Hv^*_{dis} is zero at a reduction in area of less than 30% and increases for larger reductions in area. Hv^*_{dis} was obtained by using an indent size smaller than the dislocation cell size, and can therefore be considered to be the hardness within dislocation

cells. It follows from this that at a reduction in area of less than 30%, dislocations induced by cold rolling do not exist within dislocation cells, and all of them are used for the formation of dislocation cell boundaries and slip bands. As the reduction in area increases beyond 30%, dislocations begin to exist within dislocation cells, and their number increases.

As shown in Fig. 13, the fine grain strengthening hardness by dislocation cells Hv^*_{cel} is somewhat low at a reduction in area of 10%, but saturates at nearly 120 at a reduction in area of more than 20%. Fig. 6 shows that the dislocation cell size is constant at a reduction in area of more than 20%. This, as well as the fact that at a reduction in area of more than 20% the dislocation density on the dislocation cell boundaries saturates and the locking parameter k in the Hall–Petch equation becomes constant, can explain the constant value of Hv^*_{cel} for a reduction in area of more than 20%. The drop in Hv^*_{cel} at a reduction in area of 10% is likely to be the result of the low dislocation density on the dislocation cell boundaries and the small locking parameter k , because the dislocation cell size at a 10% reduction in area is nearly the same as that for a reduction in area of more than 20%.

In Fig. 14, as the reduction in area increases, the fine grain strengthening hardness as a result of large slip line spacing Hv^*_{csl} increases and saturates at a reduction in area of 30%. According to the slip deformation model in Fig. 5, the spacing of large slips saturates at 2 μm for a strain of 10%, or a reduction in area of nearly 10% (see Table 1), and the number of fine slip lines increases in the roots of large slip lines for larger strains. This is also observable in the TEM image of the specimen rolled to a reduction in area of 30% (a true strain of 35.8%). With the Hall–Petch equation in mind and assuming that large slip lines act as crystal grain boundaries and the locking parameter k increases as the number of fine slip lines increases in the root of large slip lines, it is understandable that Hv^*_{csl} increases even at a reduction in area of more than 10%. In the TEM observations, the number of slip lines increases even in the case of a reduction in area of more than 30%. Therefore, Hv^*_{csl} should increase, but it actually saturates at a reduction in area of 30%. However, this can be resolved by considering that fine slip lines do not contribute to increasing the locking parameter k when they are formed far away from large slip lines.

The above discussion needs refinement, yet its key points can be summarized as follows:

(1) For a reduction in area of 20%, the formation of dislocation cells ends, and fine grain strengthening by dislocation cells saturates (Fig. 13). (2) At a reduction in area of 30%, fine grain strengthening by a combination of 2 μm -spaced large slip lines and fine slip lines formed in their root saturates (Fig. 14). In other words, the structure contributing to fine grain strengthening is completed at a reduction in area of 30%. (3) At a reduction in area of more than 30%, dislocations begin to exist within dislocation cells, and dislocation strengthening occurs (Fig. 12). Therefore, a reduction in area of 30% is a key value for the strength mechanism of cold-rolled SUS316 (NG) steel. As mentioned in the introduction, this study was motivated by the fact that SCC was observed in the work-hardened layer of shrouds and

recirculation pipes. A reduction in area of 30% is also an important value for SCC.

Because SCC has been observed in the work-hardened layer of L-grade austenitic stainless steel shrouds and recirculation pipes, attention has been given to studies on the occurrence and crack growth of SCC in cold-rolled austenitic stainless steel.^{18–20} Kuniya *et al.*¹⁸ performed a crevice bent beam test on cold-rolled SUS304 steel under high-temperature and high-pressure water, simulating an actual usage environment. Their results show that the occurrence of TG SCC and the length of cracks formed at the same time increased rapidly for a reduction in area of more than 30%. That is, the SCC sensitivity of cold-rolled steel increases at a reduction in area of more than 30%. Tsubota *et al.*²⁰ obtained nearly identical results on L-grade stainless steels SUS304L and SUS316L. Therefore, a reduction in area of 30% appears to be a key point in structure formation and the occurrence of SCC in cold-rolled L-grade SUS316 steel. Although this point remains to be clarified, one of the important factors for SCC is believed to be that at a reduction in area of 30%, fine grain strengthening by dislocation cells and large slip lines saturates, and a high-strength producing structure is completed.

5. Conclusions

In this study, we performed ultra micro-hardness, micro-hardness, and Vickers hardness tests and SEM and TEM analyses on the nano–meso–macro strength of L-grade austenitic stainless steel SUS316 (NG) cold-rolled to reductions in area of 10 to 50%. The results obtained are as follows:

- (1) Structural parameters play an important role in the nano–meso–macro strength analysis. The SEM and TEM observations identified an austenitic grain size of 150 μm , slip spacings of 2 μm and 20 nm, and dislocation cell sizes of 0.15 to 0.25 μm as the structural parameters of cold-rolled SUS316 (NG) steel.
- (2) As a result of the ultra micro-hardness, micro-hardness, and Vickers hardness tests, the relationships between the hardness and indent size, *i.e.* the hardness profiles, were obtained over the nano–meso–macro regions. The hardness of the base metal was independent of the indent size and was constant at nearly 150. The hardness of specimens cold-rolled to a reduction in area of 10 to 50% increased with increasing indent size. On the basis of the structural parameters (the dislocation cell size d_{cel} , $10d_{cel}$, which is 10 times d_{cel} , and $10l_{csl}$, which is 10 times the 2- μm large slip spacing l_{csl}), the hardness profiles of cold-rolled specimens showed a three-step hierarchical structure. As a result, the Vickers hardness HV corresponding to macro strength was split up into the base hardness Hv^*_{bas} of iron, the solid solution strengthening hardness Hv^*_{sol} , the dislocation strengthening hardness Hv^*_{dis} , the fine grain strengthening hardness resulting from dislocation cells Hv^*_{cel} , and fine grain strengthening hardness resulting from the large slip spacing Hv^*_{csl} .
- (3) Hv^*_{cel} and Hv^*_{csl} saturated at reductions in area of 20 and 30%, respectively, and then became constant at

values of about 120 and 50. This suggests that the formation of dislocation cells and slip bands ends at reductions in area of 20 and 30%, respectively, and thus the cold-rolled structure contributing to fine grain strengthening is completed at a reduction in area of 30%.

- (4) Hv_{dis}^* was zero at up to a reduction in area of 30% and then increased. This suggests that dislocations induced by cold rolling are used entirely for the formation of dislocation cells and slip bands, and that dislocations begin to exist within dislocation cells at a reduction in area of more than 30%, and dislocation strengthening occurs.

REFERENCES

- 1) M. Kowaka: *Kinzoku no fusyoku sonsyou to bousyoku gijyutu*, (Agune, Tokyo, 1993) 377–404.
- 2) *Sougo shigen energy tyousakai genshiryoku anzen hanabukai. 7th gennshiryokusetubi no kenzensei hyouka ni kannsuru syouiinnkai* (2003.3.10) genshiryoku anzen hoanin.
- 3) K. Miyahara, S. Matsuoka, N. Nagashima and S. Mishima: Trans. Jpn. Soc. Mech. Engrs. A **61** (1995) 2321–2328.
- 4) K. Miyahara, N. Nagashima, S. Matsuoka and T. Ohmura: Trans. Jpn. Soc. Mech. Engrs. A **64** (1998) 2567–2573.
- 5) K. Miyahara, S. Matsuoka and T. Hayashi: Metallurgical and Mater. Trans. A **32** (2001) 761–768.
- 6) S. Matsuoka: *ISIJ 53th Shiraisi kinen kouza* (2004) 51–67.
- 7) N. Nagashima, K. Miyahara, S. Matsuoka, K. Tsuzaki, T. Hara and T. Takahashi: Tetsu to hagane **86** (2000) 815–821.
- 8) H. Hirukawa and S. Matsuoka: Trans. Jpn. Soc. Mech. Engrs. A **68** (2002) 1038–1045.
- 9) H. Hirukawa, S. Matsuoka, Y. Furuya and K. Miyahara: Trans. Jpn. Soc. Mech. Engrs. A **68** (2002) 1473–1480.
- 10) S. Matsuoka: Trans. Jpn. Soc. Mech. Engrs. A **70** (2004) 1535–1541.
- 11) W. C. Oliver and G. M. Pharr: J. Matter. Res. **7** (1992) 1564–1583.
- 12) B. Jaoul: *Kinzoku no sosei*, (Marusen Tokyo 1969) 205.
- 13) M. N. Bassim and R. J. Klassen: Mater. Sci. Eng. **81** (1986) 163–167.
- 14) K. Miyazaki, K. Shibata and H. Fujita: Acta Met. **27** (1978) 855–862.
- 15) D. Tabor: *The Hardness of Metals* (Clarendon Press, Oxford, 1951) 34–37.
- 16) F. B. Pickering: *Physical Metallurgy and the Design of Steels* (Applied Science Publishers Ltd, London, 1978) 10–12.
- 17) A. W. Thompson, M. I. Baskes and W. F. Flanagan: Acta Metall. **21** (1973) 1017–1028.
- 18) J. Kuniya, I. Masaoka and R. Sasaki: Corrosion **44** (1988) 21–28.
- 19) Y. Katayama, M. Tsubota and Y. Saito: Zairyou to kankyou (2004) 149–158.
- 20) M. Tsubota, Y. Kanazawa and H. Inoue: 7th International Symposium on Environmental Degradation of Materials in Nuclear Power System **1** (1995) 519–527.