Austenitic Grain Growth behavior Immediately after Dynamic Recrystallization in HSLA Steels and Austenitic Stainless Steel

Noriki FUJITA,1) Ryoji SAHARA,2) Takayuki NARUSHIMA3) and Chiaki OUCHI(3)

1) Formerly Graduate Student, Tohoku University. Now at JFE Steel Corp., Fukuyama 721-8510 Japan.
2) Institute for Materials Research, Tohoku University, Sendai 980-8577 Japan.
3) Department of Material Processing, Graduate School of Engineering, Tohoku University, Sendai 980-8579 Japan.

(Received on March 24, 2008; accepted on July 17, 2008)

Austenitic grain growth behavior after dynamic recrystallization in HSLA steels and the austenitic stainless steel of Type 316L was investigated focusing on grain growth during a very short holding time period immediately after hot deformation. Hot compressed specimens were isothermally held at temperatures of 1 373 K, 1 423 K and 1 473 K for various time periods from 0.1 s to 1.8 ks. The grain size in all steels was coarsened by 1.5 to 2.5 times in a short holding time period of 20 s. Addition of 0.078%Zr or 0.018%Ti in HSLA steel could not prevent this rapid grain growth, which retarded grain growth in a longer holding time period over 60 s. The grain growth exponent of the n value was evaluated using a formulated grain growth kinetic equation, and two n values largely differing in the fine and coarse grain size regions were obtained in a respective temperature and alloy. The n value in the latter region ranged from 2.3 to 3.2, being almost consistent with values of various materials reported in the past studies. The very high n value over 7.7 was obtained in the fine grain size region for dynamic recrystallization in all steels, where n values for static recrystallization was relatively lower than those for dynamic recrystallization. The similar result of n values was obtained from grain growth data after dynamic recrystallization in pure nickel. The cause for very high n value and the difference of n values obtained from grain growth data after dynamic and static recrystallizations were discussed based on features of the microstructure and the grain boundary evolved by dynamic or static recrystallization and annealing.

KEY WORDS: dynamic recrystallization; austenitic grain growth; grain growth rate; HSLA steel; austenitic stainless steel; grain growth exponent.

1. Introduction

Thermo-mechanical processing in HSLA steels aims to refine the transformed microstructure through refinement of the austenitic (γ) microstructure, which consists of refinement of statically recrystallized grains during hot rolling in the high temperature region of austenite and evolution of the non-recrystallized γ microstructure by rolling at the temperature below the static recrystallization temperature of austenite in the steel. Addition of micro alloying such as Nb or Ti in HSLA steels elevates this recrystallization temperature of austenite, enhancing evolution of heavily deformed γ microstructure. That is, strict and ingenious control of static recrystallization of austenite is practiced in thermo-mechanical processing in HSLA steels.1–3) On the other hand, there has been very few practical utilization of dynamic recrystallization for the microstructural control of steels, although numerous basic studies of this behavior had been conducted.4–7) Occurrence of dynamic recrystallization in hot deformation primarily depends on the temperature, strain rate and strain, and the onset strain of dynamic recrystallization tends to reduce with the increase of the temperature and the decrease of strain rate.5) The deformation conditions in hot working processes such as hot rolling or hot forging are high strain rate and a small amount of strain in a respective deformation, indicating difficulty of occurrence of dynamic recrystallization in conventional hot working processes except hot extrusion process.

The advantageous feature of dynamic recrystallization is that the dynamically recrystallized grain size does not depend on the initial grain size, and it is simply controlled by Zener–Hollomon parameter, Z value which is determined by the deformation temperature and strain rate beside the activation energy of hot deformation.9) Currently, the control of the as cast microstructure evolved by various continuous casting processes including a thin slab caster or strip casing has become important in steel industry.5,10) The authors paid attention to this point, and studied refinement of the very coarse γ microstructure evolved in a strand casting slab of HSLA steels by use of dynamic recrystallization.11) In the experimental study, the center part of the longitudinal direction of the tensile specimen was partially melted with levitation and was deformed by a given amount of tensile strain in the austenite after solidification. This experiment was conducted under supposition to install such a rolling mill into a strand casting machine as to enable hot rolling practice with a light rolling reduction immediately after complete solidification. Strain rate corresponding to rolling speed matching with extracting speed of a slab in the strand casting process is an order of around 1 × 10^{-2} to 1 × 10^{-3} s^{-1}. The combination of this range of strain rate and the deformation temperature above 1 373 K caused dynamic
recrystallization, and very coarse γ microstructure with the grain size of mm order evolved after solidification was refined down to around 100 μm by straining in such a small strain as 0.2 to 0.3.

The dynamically recrystallized microstructure evolved during straining appears to become unstable immediately after termination of hot deformation or under the unloaded condition, which may cause more rapid grain growth compared with that of statically recrystallized microstructure. Therefore, the aim of the present study is to investigate austenitic grain growth after dynamic recrystallization using four kinds of HSLA steels and Type 316L austenitic stainless steel. Particular attention focuses on detail investigations of grain growth taken place during a very short holding time period of less than 60 s after termination of hot deformation. An investigation of a variation of γ microstructure taken place in such a short holding time period was the very important in the hot working processes, but very few experimental study of this subject was conducted in the past. It has been well confirmed that both of the austenite of HSLA steels and Type 316L yielded dynamic recrystallization. The reason for the use of the latter steel in addition of HSLA steels is that it enables to investigate a variation of not only γ grain size and grain boundary morphology, but also the microstructure in the γ matrix and the substructure evolved during holding a short time period, which are unable to investigate in the former steel. HSLA steels include a 980 MPa grade steel and micro alloyed steels containing 0.078% Zr or 0.018% Ti in 0.14%C–1.45%Mn, (notation of % indicates mass percent hereafter). Hot-compressed specimens were isothermal held for various heating time periods from 0.1 s to 1.8 ks in the temperature range from 1 373 K to 1 473 K, and a variation of γ grain size with the holding time period was investigated. Grain growth after static recrystallization which was yielded by hot compression at high strain rate was also investigated using a couple of steels. The microstructural variation during such a short holding time period after dynamic recrystallization in Type 316L austenitic stainless steel was examined by observations using both the optical and transmission electron microscopes (TEM). Grain growth exponent values of n in the grain growth kinetic equation deduced by Burke12) or Burke and Turnbull13) were obtained based on the differential equation of the grain growth kinetic equation and grain growth data in all alloys, and n values obtained from the range of a short holding time period or the fine grain size region in all alloys were much larger than values reported in various metals and alloys with a single phase structure, and thus, grain growth after dynamic recrystallization of pure nickel was investigated covering the further extended range of the holding time period up to 21.6 ks. The peculiar feature of grain growth behavior immediately after termination of dynamic or static recrystallization is discussed based on all of these results, and industrial implication obtained from the present study is also referred.

2. Experimental Procedures

Chemical compositions of HSLA steels used in this study are listed in Table 1. These HSLA alloys are denoted by alloy notations shown in the left-side column of this table in the following. The 0.09C–2.26Ni steel with a grade of 980 MPa was prepared using the strand-cast slab manufactured in one of Japanese steel companies, which was hot rolled to the thickness of 12 mm by a laboratory rolling mill. The C–Mn steel, and C–Mn–0.078Zr and C–Mn–0.018Ti steels were melted by a 50 kg induction heating furnace, and these ingots were hot rolled to the thickness of 12 mm. For enabling to reveal clearly prior γ grain boundaries in quenched specimens, carbon and phosphorous contents in these steels were increased compared with those of commercial HSLA steels. The former is for the increase of hardenability and the latter is for etching enhancement of prior austenitic grain boundaries in the specimens subjected to heat treatment for temper embrittlement. Cylindrical shape specimens with the diameter of 8 mm and the height of 12 mm for hot compression test were machined from these steel plates.

Hot compression tests and the subsequent isothermal heating experiments were performed using the hot working simulator with the induction heating system under vacuum (THERMEC MASTER-Z, Fuji Electronic Industry Co.19). Hot compression specimens were heated at the temperature of 1 523 K for 180 s, and then were cooled to temperatures of 1 373 K, 1 423 K and 1 473 K. After holding for 120 s at these temperatures, specimens were hot-compressed to the true strain of 0.69 at a respective temperature, and strain rates were $1 \times 10^{-3}$ s$^{-1}$ in 0.09C–2.26Ni steel and $2 \times 10^{-3}$ s$^{-1}$ in other three HSLA steels. This hot deformation condition yielded dynamic recrystallization in all HSLA steels, which was confirmed by both the true stress–true strain (S–S) curve behavior and observation of the microstructure in specimens quenched by He gas immediately after hot deformation.20,21) For investigation of grain growth after dynamic recrystallization, hot-deformed specimens after termination of hot deformation were continuously held for various time periods at the same temperature with the hot deformation temperature, followed by He gas quenching. Holding time periods at a respective temperature were varied in the range from 0.1 s to 1.8 ks, and in particular grain growth occurring during a very short holding time period of less than 20 s was investigated in detail. After holding for various time periods at a respective temperature, specimens were quenched by He-gas. Austenitic grain growth behavior after static recrystallization was also investigated using 0.09C–2.26Ni steel. Hot compression was conducted at strain rate of $1 \times 10^{-3}$ s$^{-1}$ and the strain of 0.69, and other thermo-mechanical cycles were the same with the case of hot deformation at low strain rate including post-hot deformation holding time periods. Hot deformation at this strain rate yielded the continuous work hardening type of S-S curve, and the as-quenched microstructure revealed equiaxis and smooth prior γ grain boundaries. Hot deformed

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cu</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Zr</th>
<th>Ti</th>
<th>sol Al</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.09C–2.26Ni</td>
<td>0.088</td>
<td>0.075</td>
<td>1.14</td>
<td>0.005</td>
<td>0.001</td>
<td>0.17</td>
<td>2.26</td>
<td>0.55</td>
<td>0.54</td>
<td>0.045</td>
<td>0.072</td>
<td>0.0028</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C–Mn</td>
<td>0.151</td>
<td>0.25</td>
<td>1.48</td>
<td>0.016</td>
<td>0.004</td>
<td>0.20</td>
<td>0.09</td>
<td>0.20</td>
<td>0.09</td>
<td>0.20</td>
<td>0.32</td>
<td>0.0030</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C–Mn–0.078Zr</td>
<td>0.134</td>
<td>0.24</td>
<td>1.41</td>
<td>0.016</td>
<td>0.004</td>
<td>0.18</td>
<td>0.078</td>
<td>0.03</td>
<td>0.0033</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C–Mn–0.018Ti</td>
<td>0.138</td>
<td>0.24</td>
<td>1.41</td>
<td>0.023</td>
<td>0.001</td>
<td>0.20</td>
<td>0.018</td>
<td>0.026</td>
<td>0.0032</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Grain growth behavior after dynamic and static recrystallizations in Type 316L austenitic stainless steel was investigated using hot compression specimens with the same shape with that of HSLA steels, which were machined from commercial Type 316L steel bar with Fe-13.64%Cr-9.40%Ni-1.74%Mo. Hot compression tests at the temperature of 1273 K were performed at both strain rates of $2 \times 10^{-3} \text{s}^{-1}$ and $1 \times 10^{-1} \text{s}^{-1}$, which yielded dynamic and static recrystallizations, respectively. Hot deformed specimens used for grain growth study were held at this temperature for the same time periods as the case of HSLA steels. The effect of the temperature on grain growth after dynamic recrystallization of this steel was also investigated under the same hot deformation and post-deformation isothermal holding conditions with those in HSLA steels.

Hot compressed specimens of HSLA steels were subjected to heat treatment for temper embrittlement at the temperature of 723 K for 86.4 ks, which revealed clearly the prior γ grain boundaries in the martensitic or lower bainitic microstructure. All specimens were halved by cutting parallel to the loading direction, and the microstructure in the center part of the specimen was observed by an optical microscope. The prior γ grain boundaries in HSLA steels were revealed by etching in an aqueous solution of picric acid, and the etching solution used for Type 316 steel was 10% hydrochloric acid with a small amount of ferric chloride. Grain size of γ was measured by an intercept method and the average γ grain size was obtained by measuring the number of the γ grain over 40. TEM observations were conducted using a Hitachi H-800 electron microscope operated at 200 kV. The thin foil was prepared using twin-jet electron polishing equipment, and electric polishing was performed using a solution of perchloric acid and methanol with a composition of 1:10 in volume at the temperature of 253 K.

Precipitates formed in C–Mn–0.078Zr and –0.018Ti steels were analyzed by EPMA (EPMA-8705, Shimadzu Co.).

3. Results

3.1. Grain Growth Behavior after Dynamic Recrystallization in HSLA Steels

Figures 1(a) and 1(b) show variations of the γ grain size with a holding time period after dynamic and static recrystallizations in 0.09C–2.26Ni steel. Strain rates in hot compression tests in the former and the latter were $1 \times 10^{-3} \text{s}^{-1}$ and $1 \times 10^{-1} \text{s}^{-1}$, respectively. Variations of the γ grain size occurring during a very short holding time period of less than 20 s are shown in graphs inserted into the top side in each figure. The dynamically recrystallized grain size obtained at three deformation temperatures ranges from 70 to 110 μm, and it is coarsened by almost twice after the holding time period of 20 s. A variation of the γ grain size with a holding time period shown by a nominal scale exhibits three stages: a very rapid grain growth in a very short time period, then a sluggish grain growth in an intermediate time period and again a relatively high grain growth rate in the further extended time period. The γ grain size obtained by holding for the time period of 1.8 ks ranges from 430 to 630 μm at three temperatures. On the other hand, the statically recrystallized grain size obtained at three deformation temperatures ranges from 45 to 60 μm, being much smaller than the dynamically recrystallized grain size. The grain growth rate in a short holding time period of less than 20 s is higher than that for dynamic recrystallization, and the grain size is coarsened by around 2.5 to 3 times during this time period. The holding time period exhibiting the second stage of grain growth is relatively short, and that of the third stage is more extended compared with those after dynamically recrystallization. The γ grain size obtained after holding for 1.8 ks ranges from 400 to 550 μm at three temperatures.

Results of grain growth behavior after dynamic recrystallization in C–Mn, C–Mn–0.078Zr and C–Mn–0.018Ti steels are shown in Figs. 2, 3 and 4, respectively. Strain rate in hot compression tests for these three steels was higher by twice than that in 0.09C–2.26Ni steel. Figure 2 shows the result of C–Mn steel. The dynamically recrystallized grain size obtained at three deformation temperatures ranges from 50 to 80 μm, and it is coarsened by around 1.5 times after a holding time period of 20 s at all temperatures. Growth behavior during a holding time period up to 1.8 ks in this steel is very similar to that in 0.09C–2.26Ni steel, but the grain size obtained after holding for 1.8 ks is almost half of those in 0.09C–2.28Ni steel at all temperatures. On the other hand, results of grain growth behavior in two micro-alloyed HSLA steels shown in Figs. 3 and 4 are...
markedly different from those of 0.09C–2.26Ni and C–Mn steels. Both of C–Mn–0.078%Zr and C–Mn–0.018%Ti steels exhibit much retarded grain growth except a very short holding time period immediately after dynamic recrystallization in all temperatures. The dynamically recrystallized grain size obtained by deformation at three temperatures ranges from around 35 to 70 μm, which coarsens to around 60 to 95 μm after a holding time period of 20 s. Grain growth progresses very slowly during a further extension of the holding time period up to 1.8 ks, and the grain size obtained after holding for 1.8 ks at three temperatures ranges from 115 to 130 μm. Grain growth behavior in C–Mn–0.018Ti steel shown in Fig. 4 is very similar to that of C–Mn–0.078Zr steel. The dynamically recrystallized grain size is around 30 to 40 μm at three deformation temperature. The grain size is largely coarsened in a very short holding time period less than 20 s, and the grain size after holding for 20 s at three temperatures ranges from around 50 to 80 μm. But grain growth during a further extension of the holding time period up to 1.8 ks is markedly retarded, and the grain size obtained after holding for 1.8 ks at three temperatures ranges from 90 to 110 μm. Consequently, it is found that the increment in the grain size occurring during the holding time period from 20 s to 1.8 ks at a respective temperature is very small in this steel, while the grain growth rate in the holding time period of less than 20 s is very large.

Grain growth behavior after dynamic recrystallization at the temperature of 1 373 K is compared among C–Mn, C–Mn–0.078Zr and C–Mn–0.018Ti steels in Fig. 5. It is found that all steels yields very rapid grain growth in a short holding time period of less than 20 s and that the subsequent grain growth behavior during the holding time period up to 1.8 ks is largely different between C–Mn steel and micro-alloyed steels. The continuous grain growth observed in C–Mn steel is prevented by addition of micro-alloying elements. Ti-bearing steel always yields the smallest grain size among three steels in the whole range of the holding time period, and the grain size obtained by holding for 1.8 ks in this steel is around one third of that of C–Mn steel.

3.2. Grain Growth Behavior after Dynamic and Static Recrystallizations of Type 316L Steel

Figure 6 shows grain size variations with the holding time period after dynamic and static recrystallizations in Type 316L steel. The hot compression test and an isothermal holding after deformation were conducted at 1 273 K. The statically recrystallized grain size is around 5 μm, which is much finer than dynamically recrystallized grain.
size obtained in this steel. The grain growth rate of the statistically recrystallized grain in a short holding time period of less than 20 s is much higher than that of dynamically recrystallized grain. The grain size obtained by holding for 20 s becomes larger by around 6 times and 1.5 times compared with the initial grain size for static and dynamic recrystallizations, respectively. These results of Type 316L steel as well as HSLA steels appears to indicate that the grain growth rate in a short holding time period of less than 20 s becomes higher with a reduction of the initial grain size in spite of the dynamically or statically recrystallized microstructure. Grain growth behavior during a longer holding time period up to 1.8 ks after dynamic recrystallization is very similar to that of 0.09C–2.26Ni steel, although the grain size of Type 316L steel is much finer than that of 0.09C–2.26Ni steel in the whole range of the holding time period. On the other hand, grain growth during the holding time period from 60 s to 1.8 ks after static recrystallization is rather sluggish, and rapid grain growth in the third stage is not seen. However, the cause for this difference between grain growth behavior after static and dynamic recrystallizations was not made clear. The effect of the temperature on grain growth after dynamic recrystallization in this steel is shown in Fig. 7. Although the dynamically recrystallized grain size becomes larger with an elevation of the deformation temperature, subsequent grain growth behavior is very similar at all temperatures. The grain growth rate in the holding time period of less than 20 s tends to become higher with a reduction of the initial grain size. The grain size obtained by holding for 1.8 ks at a respective temperature is much smaller than that of C–Mn steel and almost the same with that of C–Mn–0.078Zr steel.

Figure 8 shows a variation of the optical microstructure during a very short holding time period at 1 373 K after dynamic recrystallization in Type 316L steel. As seen in Fig. 8(a), the dynamically recrystallized microstructure shows an irregular shape of grain boundary morphology and no twin formation inside grains.51 Figures 8(b) and 8(c) show the microstructures for holding time periods of 0.1 s and 1.0 s, respectively. Marked grain coarsening evidently takes place during such a short holding time period, which accompanies with the smoother shape of grain boundary morphology and formation of annealing twins inside grains. As shown in Fig. 6, the grain size in the microstructure evolved after holding for 1.0 s becomes larger by around 1.6 times compared with that the initial grain size, and annealing twins tend to be formed inside grains with the larger grain size. On the other hand, the microstructural variation during a short holding time period after static recrystallization in this steel showed much marked grain coarsening with maintaining smooth grain boundary morphology, and annealing twins were also formed inside grains with the increase of the grain size.

A variation of TEM micrographs during a short holding time period at 1 373 K after dynamic recrystallization in Type 316L steel is shown in Fig. 9. The dynamically recrystallized microstructure shown in Fig. 9(a) shows relatively high dislocation density nearby grain boundaries, and a dislocation network and a number of isolated dislocations are formed inside grains.5,12 Figures 9(b)–9(d) show TEM micrographs after holding for 0.1 s, 0.5 s and 1.0 s, respectively. An extension of the holding time in such a short holding time period continuously causes smoothing of the
3.3. Evaluation of the Grain Growth Exponent $n$ in Grain Growth Kinetic Equation

Grain growth behavior after dynamic recrystallization is analyzed based on theory of the grain growth kinetic equation by Burke et al.\textsuperscript{13,14} which was deduced under an assumption that the driving pressure on the boundary was arisen from the curvature of the boundary. This is expressed by the following equation.

$$D^n - D_0^n = Kt$$

(1)

\text{where $D_0$ and $D$ are the grain size at $t=0$ and the grain size obtained by holding for the time period of $t$, respectively, and $K$ and $n$ are constant values. The grain growth rate $dD/dt$ in the grain size of $D$ is obtained by differentiating Eq. (1) by $t$, which is shown by the following equation.}

$$\ln(dD/dt) = \ln(K/n) - (n - 1) \ln D$$

(2)

This equation indicates that the grain growth rate decreases at the constant rate with the increase of the grain size and that this constant value of $n$ can be obtained from the slope of the straight line in log–log plots of the grain growth rate versus the grain size. Grain growth data after both dynamic and static recrystallizations at a respective temperature in all alloys were formulated by the equation of $D = kr^n$, and values of $k$ and $m$ in HSLA and Type 316L steels were obtained by data fitting. These values are summarized in Table 2. Grain growth data of 0.09C–2.26Ni and C–Mn steels were approximated by two equations according to the range of the holding time period of below or above 0.5 ks, and those of micro-alloyed HSLA steels and Type 316L steel were formulated by one equation in the whole range of the holding time period.

The relationship between $dD/dt$ and the time period of $t$ can be obtained by differentiating the formulated equation with $t$. This enables to calculate the value of $ddt/D$ in $t$, and the grain size in $t$ can be obtained from grain growth data. Logarithmic plots of the calculated grain growth rate $(dD/dt)$ versus the grain size obtained by holding for various time periods after dynamic recrystallization in HSLA steels are shown from Figs. 10(a) to 10(d). The grain growth rate markedly and linearly decreases with the increase of the grain size in all HSLA steels in the fine grain size region, followed by the knick point of the straight line in a particular grain size at a respective temperature in all HSLA steels. In the coarse grain size region, the slope of the straight line becomes much smaller, and it tends to converge to the constant value at all temperatures in any HSLA alloy. Data in the coarse grain size region in all steels were relatively few, and thus an average $n$ value was evaluated using all data obtained at three temperatures in a steel. Values of $lnD$ in the knick point range from 4.1 to 4.8 (60 to 121 $\mu$m in $D$ value) in C–Mn and two micro alloyed steels, while its values of 0.09C–2.26Ni steel are 5.3 to 5.7 (200 to 299 $\mu$m in $D$ value). The grain growth exponent $n$ can be obtained from the slope of the straight line in these figures, which is noted together with the temperature inside a respective figure. Values of $n$ in the fine grain size region in C–Mn and two micro alloyed steels range from 11.0 to 22.0, and those of 0.09C–2.26Ni steel are 7.7 to 8.5.

Figure 11 shows logarithmic plots of the calculated grain growth rate versus the grain size obtained by holding for various time periods after dynamic recrystallization in Type 316L steel. All data points obtained at three temperatures ranging from 1373 to 1473 K are approximated by two straight lines with a largely different slope similarly to results of HSLA steels. That is, very high $n$ value ranging from 14.8 to 17.2 in the fine grain size region and $n$ value of 3.5 in the coarse grain size region are obtained.

Figures 12(a) and 12(b) show logarithmic plots of the

---

**Table 2.** Values of $k$ and $m$ in the formulated equation of $D = kr^n$ in HSLA steels, Type 316L steel and pure nickel.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Recrystallization</th>
<th>Range of holding time period</th>
<th>Temperature</th>
<th>1123 K</th>
<th>1273 K</th>
<th>1373 K</th>
<th>1423 K</th>
<th>1473 K</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>k</td>
<td>m</td>
<td>k</td>
<td>m</td>
<td>k</td>
<td>m</td>
<td>k</td>
</tr>
<tr>
<td>0.09C–2.26Ni</td>
<td>dynamic</td>
<td>0.1–0.5 ks</td>
<td>0.5–1.8 ks</td>
<td>73.4</td>
<td>0.231</td>
<td>93.7</td>
<td>0.210</td>
<td>105.9</td>
</tr>
<tr>
<td></td>
<td>static</td>
<td>0.1–0.5 ks</td>
<td>0.5–1.8 ks</td>
<td>93.9</td>
<td>0.194</td>
<td>146.6</td>
<td>0.160</td>
<td>27.5</td>
</tr>
<tr>
<td>C–Mn</td>
<td>dynamic</td>
<td>0.1–0.5 ks</td>
<td>0.5–1.8 ks</td>
<td>76.0</td>
<td>0.085</td>
<td>96.8</td>
<td>0.057</td>
<td>111.8</td>
</tr>
<tr>
<td></td>
<td>static</td>
<td>0.1–0.5 ks</td>
<td>0.5–1.8 ks</td>
<td>92.1</td>
<td>0.0336</td>
<td>23.4</td>
<td>0.303</td>
<td>65.8</td>
</tr>
<tr>
<td>C–Mn-0.078%Cr</td>
<td>dynamic</td>
<td>0.1–1.8 ks</td>
<td>18.6</td>
<td>65.6</td>
<td>0.069</td>
<td>73.6</td>
<td>0.040</td>
<td>87.6</td>
</tr>
<tr>
<td>C–Mn-0.018%Ti</td>
<td>dynamic</td>
<td>0.1–1.8 ks</td>
<td>18.6</td>
<td>65.6</td>
<td>0.069</td>
<td>73.6</td>
<td>0.040</td>
<td>87.6</td>
</tr>
<tr>
<td>Type 316L</td>
<td>dynamic</td>
<td>0.1–1.8 ks</td>
<td>0.112</td>
<td>61.5</td>
<td>17.1</td>
<td>0.267</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pure nickel</td>
<td>dynamic</td>
<td>0.1–1.2 ks</td>
<td>1.2–21.6</td>
<td>61.5</td>
<td>17.1</td>
<td>0.267</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
calculated grain growth rate versus the grain size obtained by holding for various time periods after static recrystallization in Type 316L and 0.09C–2.26Ni steels, respectively. Values of \( k \) and \( m \) in formulated equations of grain growth kinetics used for calculation of the grain growth rate are listed in Table 2. Figure 12(a) shows both results after static and dynamic recrystallizations at the temperature of 1273 K. All data for static recrystallization are approximated by a single straight line, yielding the \( n \) value of 4.1. The grain size obtained by holding for the longest time period was relatively small for static recrystallization in this steel. Therefore, it may be needed to conduct the grain growth experiment after static recrystallization under a further extended time period, then becoming possible to make clear existence of another straight line with a lower \( n \) value in the coarse grain size region. On the other hand, for grain growth after dynamic recrystallization, data are approximated by two straight lines, of which \( n \) values are 9.5 and 2.5 in the fine and coarse grain size regions, respectively. The value of \( \ln D \) at the knick point is around 4.2. Data of 0.09C–2.26Ni steel are approximated by two straight lines similarly to the case of grain growth after dynamic recrystallization, and \( n \) values in the fine grain size region are relatively lower than those after dynamic recrystallization shown in Fig. 11. Consequently, for grain growth after static recrystallization, both steels showed the lower \( n \) value in the fine grain size region compared with the \( n \) value for grain growth after dynamic recrystallization.

4. Discussion

4.1. A Possible Cause for High Value of Grain Growth Exponent Obtained in the Fine Grain Size Region

Grain growth mechanism in metals and alloys is different according to their microstructures which are classified by having a single- or two-phase structure and containing dispersive particles. The basic study of grain growth in materials having a single phase structure without dispersive particles was mostly conducted over 30 years ago. Since then very few study of this subject was conducted, and Licke\(^1\)\(^5\) and Humphreys et al.\(^1\)\(^6\) made excellent reviews of this subject including current studies in this field. The present grain growth study using HSLA and Type 316L steels was conducted focusing on grain growth occurring during a very short holding time period immediately after dynamic recrystallization in austenite, and it was found that two differ-
ent $n$ values according to the fine and coarse grain size regions were obtained in all steels investigated in this study. The theoretical value of the grain growth exponent $n$ in Eq. (1) is 2.0 in metals and alloys with a single phase structure. Humphreys et al. summarized the experimentally measured $n$ values in various pure metals and alloys, finding that these values were mostly higher than 2, ranging from 2 to 4 with an average of 2.4±0.4. The cause for arising the difference between the theoretical $n$ values and measured these values had been discussed. Values of $n$ in the coarse grain size region ranged from 2.3 to 3.2 at all temperatures and in all alloys investigated in this study, which almost agreed with measured values of other metals and alloys. On the other hand, $n$ values obtained in the fine grain size region were much higher than reported values. However, as seen from Figs. 10 to 12, data in the coarse grain size region were not always enough to evaluate an exact $n$ value in all HSLA and Type 316L steels. To fill up for this point and also to reconfirm peculiar grain growth behavior observed in the present study, grain growth behavior after dynamic recrystallization in pure nickel was investigated covering a much extended holding time period up to 21.6 ks. The reason for use of pure nickel is that basic studies of grain growth behavior and evaluation of $n$ value including the impurity effect on grain growth had been mostly conducted using pure metals. In addition, this metal has the same FCC structure with austenite and is a typical dynamic recrystallization type of metals.

Specimens machined from a commercial pure nickel bar with purity of 99.9% were heated at 1 123 K for 0.3 ks, followed by hot compression in the strain of 0.69 and strain rate $1 \times 10^{-3}$ s$^{-1}$. Figure 13 shows a variation of the grain size with the holding time in pure nickel. The dynamically recrystallized grain size is around 45 μm, which rapidly coarsens during a very short holding time immediately after dynamic recrystallization as seen in the graph inserted in the top of this figure. The grain growth rate continuously decreases with an extension of the holding time. The grain growth kinetics was formulated by two equations according to the region of the holding time period as listed in Table 2.

The relationship between the grain growth rate and the grain size was obtained by the similar method as the case of HSLA steels, of which the result is shown in Fig. 14. All data plots are evidently approximated by two straight lines with a knick point in an ln $D$ value of 4.75 (116 μm in $D$ value). Values of $n$ in the fine and coarse grain size regions obtained from this figure are 9.0 and 2.6, respectively. Consequently, it is confirmed in this pure metal that very high $n$ value is obtained in the fine grain size region, while $n$ value in the coarse grain size region is almost the same with $n$ values in various materials reported in the past studies.

Higgins reported the temperature dependence of the $n$ value for isothermal grain growth in a variety of materials, where the inverse value of $n$ was plotted against normalizing annealing temperature $T_T/m$ where $T_m$ was the melting point. While the inverse value of $n$ decreased with a reduction of the temperature in the low temperature region, it became constant in the high temperature region, where $n$ values ranged from 2 to 4 in a variety of metals and alloys. The temperature or $T_T/m$ adopted in the investigation of austenitic grain growth in HSLA and Type 316L steels is relatively high, and so the $n$ value obtained in the coarse grain size region in the present alloys is found to be consisted with values obtained in the high temperature region in past studied materials. Therefore, it is needed to find causes for high $n$ values obtained in the fine grain size region corresponding to the very short holding time period region. First, it is important to note that the present study was conducted focusing on grain growth behavior after hot deformation using a hot working simulator, which enabled to investigate grain growth occurring during a very short holding time period immediately after termination of hot deformation. On the other hand, the most of the past grain growth studies in various metals and alloys were conducted by isothermal holding in heat treatment such as annealing using the various heating furnaces. The minimum holding time period taken in isothermal heating was mostly over 100 s in these studies, because the grain growth study including such a short holding time period as taken in the present investigation is not easy for the conventional heating method. This indicates that $n$ values reported in the past studies may be obtained by the holding time period condition corresponding to the coarse grain size region in the present results, and that evaluation of the $n$ value including grain growth during a very short holding time period is very few.

Then, it turns out to find the cause for very high $n$ value obtained in the fine grain size region. This appears to be
arisen by the difference of grain boundary contamination due to impurity or solute elements in the initial microstructures evolved by annealing and recrystallization. Aust et al. referred that a “pure grain boundary”, i.e., one which entirely free of impurities, rarely exists even in the purest metals. This appears to be true for a grain boundary in the microstructure evolved by annealing. However, it may possibly assume that the grain boundary formed immediately after recrystallization in spite of static or dynamic one is extremely clean and that this boundary has the higher mobility due to a lack of solute drag effect compared with the contaminated grain boundary. So the initial grain growth rate immediately after termination of recrystallization may be very high compared with that of the contaminated grain boundary, and then the grain growth rate may trap various solutes during its migration in grain growth, resulting in a rapid and continuous reduction of the grain growth rate. This may induce a large reduction of the grain growth rate with the grain coarsening, that is high n value. The continuous boundary contamination with an extension of the holding time period may be accelerated by grain coarsening, because the impurity or solute concentration on the grain boundary becomes higher with the increase of grain size. Grain coarsening in the fine grain size region yielding a high n value terminates when the grain size gets to the particular size which may be determined by both solute contents of impurity and alloying elements in the alloy. The subsequent grain growth in an extended holding time period turns out to be the same one in grain growth behavior observed in the annealing experiment which yields a low n value.

It was found from comparison of grain growth after dynamic and static recrystallizations that the n value of the former in the fine grain size region was higher than that of the latter. As shown in Figs. 8 and 9, the microstructure evolved by dynamic recrystallization has an irregular shape of grain boundaries and contains a relatively high dislocation density inside grains and nearby grain boundaries compared with that of static recrystallization. Both factors give rise to the higher driving force for grain boundary migration due to the higher boundary energy and enhanced diffusivity of solvent atoms, respectively. This may result in the very high initial grain growth rate immediately after dynamic recrystallization. But both enhanced factors of grain growth tend to disappear during a very short holding time period of a couple of seconds, and the microstructure becomes almost the same with that of static recrystallization. Thus, the grain growth rate reduces at very high rate with the increase of the grain size, resulting in the higher n value in grain growth after dynamic recrystallization than that obtained after static recrystallization.

4.2. The Effect of Addition of Micro-alloying Elements on Austenitic Grain Growth

It was found that very rapid austenitic grain growth took place during a very short holding time period after dynamic recrystallization in all of HSLA steels and Type 316L steel, and the grain size after holding for 20 s became larger by 1.5 to 3 times compared with the initial grain size or dynamically recrystallized grain size in a respective alloy. Addition of micro alloying elements such as Zr or Ti in HSLA steels could not prevent this rapid grain growth immediately after dynamic recrystallization, and the marked retardation effect of austenitic grain growth due to Zr or Ti addition was observed in a longer holding time period over 60 s. Figures 15(a) and 15(b) show BSE images obtained by EPMA analysis in C–Mn–0.078Zr and C–Mn–0.018Ti steels, respectively. Very strong sulfur spots with the white color are observed in C–Mn–0.078Zr steel. Thus, this precipitate appears to be ZrS, although C and N analysis was not performed. The morphology of this precipitate is the elongated rod shape as well as its fragment-like particles. TiN precipitates with a square shape are observed in C–Mn–0.018Ti steel. However, fresh grain boundaries evolved immediately after recrystallization appear not to be pinned by not only solute elements but also these precipitates, and so very rapid grain growth takes place during a short holding time period even in micro-alloyed steels. Continuously migrating grain boundaries may be pinned by precipitates during a further extended holding time period, then grain growth being retarded. The grain size in the alloy containing particles is primarily controlled by the volume fraction and the size of particles, and so the finer size and the larger number of TiN precipitates over ZrS precipitates appears to cause the more marked retardation of grain growth in C–Mn–0.018Ti steel. As seen in Fig. 5, the γ grain size after holding at 1 373 K for 1.8 ks in C–Mn–0.018Ti steel became by one third times smaller than that of C–Mn steel. Currently, Nakashima et al. reported similar marked austenitic grain growth retardation due to 2% Cu addition in low carbon steel, and they concluded that this grain growth retardation was attributed to the dragging effect of solute Cu atoms rather than the pinning effect of Cu precipitates, because of fully dissolution of Cu precipitates during austenization. However, in the present case, both precipitates of ZrS and TiN may precipitate during solidification and remain not to dissolve at the reheating temperature before hot deformation. Thus, these precipitates cause grain boundary pinning after holding for over 100 s. Consequently, if a rapid grain growth occurring immediately after dynamic or static recrystallization can be suppressed by the same method, more pronounced grain refinement is possibly achieved in all alloys, which is discussed in the following.

4.3. Industrial Implications obtained from the Present Results

As noted in Introduction, thermo-mechanical processing in HSLA steels primarily utilizes static recrystallization of austenite, and it is very important to prevent grain growth immediately after the static recrystallization. Rapid grain growth occurring during a very short holding time period was observed after not only dynamic recrystallization but
also static recrystallization in 0.09C–2.26%Ni steel and Type 316L steel. The grain size after holding for 20 s became larger by 2.5 to 6 times than the initial grain size obtained by static recrystallization in a respective alloy. This appears not to be prevented by addition of micro-alloying elements, although grain growth behavior after static recrystallization in micro-alloyed steels was not investigated. Senuma et al. studied the effect of the onset time of cooling after final rolling on the grain size in a plain ultralow-carbon steel, finding that a reduction of the onset time of cooling in the range of less than 1.5 s continuously decreased the grain size. The present result indicates that this ferrite grain refinement is possibly yielded by the austenite grain refinement accompanied with a reduction of the onset time of cooling. For prevention of rapid grain growth immediately after hot deformation, hot rolled steel products are needed to cool down as soon as possible after termination of hot rolling. For this purpose, an accelerated cooling facility in a plate mill or the run-out cooling facility in hot strip mill is favorable to stand in the most close position to the exist of finish-rolling mill stand, and then the time delay between termination of hot rolling and the onset of rapid cooling is minimized. The increase of the hot rolling speed or the traveling speed of rolled products is also favorable for minimizing this time delay.

The present study was conducted with a purpose to refine the very coarse γ microstructure evolved in a strand cast slab in use of dynamic recrystallization under supposition to install such a rolling mill into a strand casting machine as to enable hot rolling practice with a light rolling reduction immediately after complete solidification. It may be very useful to install cooling facility just behind rolling mill in this case, enabling to cool down the temperature of the rolled slab immediately after termination of hot rolling. The amount of the temperature reduction due to rapid cooling is not needed to be so large, because the grain growth rate markedly reduces with the decrease of the temperature. The most important factor is to minimize the time delay between the finish of hot rolling and the onset of rapid cooling.

5. Conclusions

Austenitic grain growth behavior after dynamic recrystallization in HSLA steels and Type 316L austenitic stainless steel was investigated focusing on grain growth taken place during a very short holding time period immediately after hot deformation. Grain growth behavior after static recrystallization was also investigated in a couple of steels. Main results are summarized as follows.

(1) The grain size obtained after holding for 20 s in all temperatures and alloys is coarsened by 1.5 to 2.5 times compared with the initial grain size obtained by dynamic recrystallizations. For static recrystallization, it becomes larger by 2.5 to 6 times than the initial grain size.

(2) Addition of 0.078% Zr or 0.018% Ti in HSLA steel cannot prevent this rapid grain growth occurring in a short holding time period after dynamic recrystallization, and it markedly retards grain growth taken place in the longer holding time period over 60 s. In particular, the grain size after holding for 1.8 ks in C–Mn–0.018Ti steel becomes smaller by one third compared with that of HSLA steel containing no micro alloying element.

(3) It is found by TEM observations of Type 316L steel that the microstructural features of dynamic recrystallization such as the irregular shape of grain boundaries, relatively high dislocation density or non-twining formation disappear in a very short holding time of less than 1 s, the microstructure evolved by holding for 1 s being almost the same with the microstructure obtained by annealing.

(4) The grain growth exponent of n value was evaluated using a formulated grain growth kinetic equation. Two n values which largely differ according to the fine and coarse grain size regions are obtained in a respective temperature and alloy. The n value in the coarse grain size region ranges from 2.3 to 3.2, being almost consistent with values reported in the past studies.

(5) The very high n value over 7.7 was obtained in the fine grain size region for dynamic recrystallization in all steels, and n values for static recrystallization in this region were relatively lower than those for dynamic recrystallization.

(6) The cause for very high n value in the fine grain size region appears to be brought about by very clean grain boundaries formed by recrystallization, yielding the very high grain boundary migration rate. As the holding time is extended or the grain size increases, grain boundaries may be trapped by various solutes and then, grain growth kinetics turn out to be controlled by the low n value.

Acknowledgements

The authors are grateful to Professor T. Sakai of the University of Electro-Communications for useful discussion and Dr. S. Mitao of JFE Steel for a kind supply of HSLA steels used in this study.

REFERENCES