

Development of Anti-coarsening Extra-fine Steel for Carburizing

Manabu KUBOTA*¹Tatsuro OCHI*¹

Abstract

There are large needs for omitting intermediate heat treatments in manufacturing process of carburizing units and increasing the carburizing treatment temperature. However, since grain coarsening which often occurs in these processes gives negative impacts on property of units, it is necessary to develop techniques to prevent the grain coarsening. Therefore, we have developed a new technique effective in preventing the grain coarsening by fining precipitate size and increasing its volume. The developed steel shows good property in preventing the grain coarsening in various manufacturing processes of units. In particular, the developed steel proved to prevent the grain coarsening at 1,323K high temperature carburizing.

1. Introduction

Carburizing is widely applied to the components of an automobile drive system such as gears, shafts and bearings to enhance their fatigue strength and wear resistance. One of the important issues related to this kind of car components is the simplification of their manufacturing processes for purposes such as cost reduction and environmental protection.

It is widely known that when a component formed by cold working is carburized, some austenite grains are likely to grow excessively during the carburizing. "Coarse grains" are also formed. The coarsening of grains has various harmful effects: the fatigue and impact properties of the product components are deteriorated and increase of heat treatment distortion make dimensional accuracy worse. For this reason, it has been a normal practice to apply normalizing or annealing to cold-worked components to remove the effects of cold working prior to carburizing.

Another issue is that if the carburizing temperature can be made higher, processing time is shortened and as a result, productivity and energy saving are enhanced. Raising the carburizing temperature is effective especially in the case of the components for which a large carburized case depth is required. For instance, when a carburized case depth of 1 mm is required, it takes about 12 h for carbon to diffuse at 1,203K, which is the carburizing temperature in usual prac-

tice. In contrast, at 1,323K, it takes about 3 h, only one quarter thereof, to obtain the same carbon concentration distribution. Therefore productivity is improved significantly. However, coarse grains form more easily as the carburizing temperature becomes higher, and for this reason, after the carburizing at a high temperature, it has been necessary to refine a coarsened microstructure by cooling once to a temperature lower than the transformation temperature and then reheating to a lower austenitizing temperature than carburizing temperature.

This paper reports Nippon Steel's research and development activities regarding the developed anti-coarsening steel for carburizing applications, with which coarse grains are prevented and thus, the intermediate heat treatment before carburizing and the reheating after carburizing can be eliminated.

2. Prevention of Grain Coarsening

It is widely known that when a polycrystalline structure contains second phase particles such as fine precipitates dispersed therein, grain boundaries are pinned and the growth of matrix grains is inhibited; Zener's analysis is known as one of the typical theories of steady state grain growth¹⁾. In the same way, many researchers have pointed out the relationship between the abnormal grain growth and the pinning effect of secondary phase particles. Among them, Gladman²⁾

*¹ Muroan R&D Lab.

obtained the following equation as the critical condition of abnormal grain growth:

$$r_{crit} = \frac{6R_0 f}{\pi} \left(\frac{3}{2} - \frac{2}{Z} \right)^{-1} \quad (1)$$

where r_{crit} is the critical radius of precipitate grains, R_0 the crystal grain radius of the matrix, f the volume fraction of the precipitates, and Z the ratio of radii of growing and matrix grains at an initial stage (the radius of growing crystal grains (R) / the radius of matrix crystal grains (R_0)).

Because abnormal grain growth occurs when the radius r of the fine precipitates that pin the crystal grain boundaries becomes larger than r_{crit} in equation (1) for some reason, grains coarsen more easily as r_{crit} becomes smaller. In other words, the grain coarsening is likely to occur when:

- (1) the matrix crystal grains that form as a result of the $\alpha \rightarrow \gamma$ transformation during the heating for carburizing are fine (R_0 is small),
- (2) the degree of mixed grain size, or the ratio of radii of growing and matrix grains, of γ grains is large (Z is large), or
- (3) the number of the pinning particles is small (either f is small or r is large).

From the above, it is understood as follows: the reason why coarse grains form in cold-worked steel is that γ grains forming as a result of the $\alpha \rightarrow \gamma$ transformation during the heating for carburizing are unavoidably made fine (R_0 becomes small) by cold working, thus, r_{crit} becomes smaller and as a result, the radius r of the precipitates becomes larger than r_{crit} ; and the reason why coarse grains form during carburizing at a high temperature is that either the precipitate radius r becomes larger than r_{crit} during heating to a high temperature owing to the Ostwald ripening³⁾ or a part of the precipitates solves in the matrix and as a result, f becomes smaller and as a consequence, r_{crit} becomes smaller than the radius r of the precipitates.

Therefore, what is essential for preventing the coarsening of crystal grains is to control so that the radius r of the precipitates does not exceed r_{crit} by dispersing fine precipitates (small r) in the matrix in a great amount (large f) during carburizing. This is accomplished by controlling the chemical composition, the production conditions of steel and production conditions of parts. Fig. 1 shows the concept for dispersing a great amount of precipitates in fine particles, which are:

- (1) to completely solutionize once during the reheating for bar rolling the coarse precipitates that have inevitably precipitated in a bloom,
- (2) to raise the reheating temperature of bar rolling so that as much

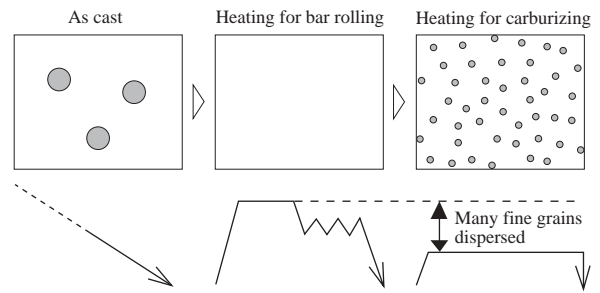


Fig. 1 Concept of dispersion of many fine precipitates

precipitates as possible are solutionized, and for this end, limit the addition amounts of precipitate forming elements to respective maximum soluble amounts at the reheating temperature, in accordance with the solubility of each of them, and

- (3) then, to make the precipitates precipitate in fine particles during the annealing or normalizing at the production of the parts or the heating for carburizing.

AlN, Nb(CN), Ti(CN), V(CN) and the like are given as the compounds that form the fine precipitates that can be used for pinning matrix grain boundaries; it is desirable to choose the one that is capable of completely solutionizing at the time of the reheating for hot rolling and precipitating in a large amount at the heating for carburizing. Since AlN is the most popular and best suits this requirement among the above compounds, the authors studied its use as the main pinning particles. In view of a possibility that AlN alone cannot provide a sufficient amount of pinning particles, use of Nb(CN) in addition to AlN was also studied.

3. Test Methods

Specimens for examining the formation behavior of coarse grains were prepared on the basis of JIS SCr 420, changing the addition amounts of Al, Nb and N. Their chemical compositions are shown in Table 1. In specimens Nos. 1 to 6, the addition amounts of Al and N were changed for the purpose of examining the effect of the AlN amount, setting the ratio of their addition amounts at the stoichiometric composition (Al/N = 1.94). Specimens Nos. 7 to 10 were prepared based on specimen No. 5 changing the addition amount of Nb, and specimen No. 11 based on specimen No. 10 decreasing the addition amount of Al.

Since the formation behavior of coarse grains and the distribution of the precipitates were expected to differ significantly at each of production stages depending on the solution/precipitation of the

Table 1 Chemical compositions of steels

No.	C	Si	Mn	P	S	Cr	Sol.Al	Nb	N	Remarks
1	0.21	0.26	0.81	0.015	0.014	1.05	0.014	Tr.	0.0056	AlN type
2	0.20	0.26	0.80	0.014	0.015	1.05	0.019	Tr.	0.0112	
3	0.19	0.26	0.81	0.014	0.015	1.05	0.024	Tr.	0.0132	
4	0.20	0.26	0.81	0.014	0.016	1.05	0.028	Tr.	0.0137	
5	0.21	0.26	0.82	0.016	0.013	1.05	0.031	Tr.	0.0152	
6	0.20	0.26	0.82	0.015	0.012	1.05	0.048	Tr.	0.0239	
7	0.18	0.26	0.79	0.016	0.014	1.03	0.028	0.005	0.0136	Nb-addition type
8	0.20	0.26	0.80	0.016	0.014	1.04	0.027	0.010	0.0144	
9	0.21	0.25	0.82	0.015	0.013	1.04	0.029	0.020	0.0160	
10	0.20	0.26	0.82	0.015	0.013	1.05	0.029	0.041	0.0155	
11	0.21	0.25	0.80	0.014	0.014	1.03	0.011	0.041	0.0112	

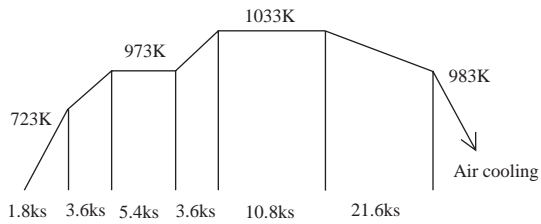


Fig. 2 Heat pattern of SA

precipitates and the influences of the matrix microstructure⁴⁻⁷⁾, the production history of each specimen was controlled to simulate the actual conditions of commercial production to the best possible extent.

The steels were melted in a laboratory vacuum melting furnace, cast into ingots 150 kg in weight, hot forged into 50 mm × 50 mm square bars, then heated to 1,573K for homogenization, cooled in air, and machined into rolling materials 44 mm × 44 mm in section. The rolling materials were subjected to a 2-pass hot rolling using a laboratory rolling mill to simulate the bar rolling or hot forging of commercial products. The two passes of the hot rolling were conducted, after reheating to 1,498K for 1.2 ks, at 1,373 and 1,233K under the same reduction ratio of 25% using plane rolls, and the steels were cooled in air thereafter. The average cooling rate from 1,073 to 773K was 0.46K/s. Some of the rolled steel sheets were subjected to a spheroid annealing (SA) under the condition shown in Fig. 2 or a normalizing treatment, wherein they were heated at 1,193K for 3.6 ks and then cooled in air.

Then, cylindrical test pieces 16 mm in diameter and 24 mm in length were cut out from the rolled steel sheets. The test pieces of specimens for cold working applications underwent upsetting cold working under an upsetting ratio of 50% and a strain velocity of 1.0.

For simulating a real carburizing treatment, the austenite structure after the simulative carburizing was frozen in the form of pre-austenite grain boundaries in a martensitic structure by heating and holding the cylindrical test pieces at 1,223 to 1,323K for 18 ks and water cooling immediately thereafter. For some test pieces, additional tests were carried out at higher heating temperatures.

The pre-austenite of the test pieces that underwent the simulative carburizing treatment was observed in a section in the rolling direction and its grain size number was determined according to JIS. A grain size corresponding to a JIS grain size number of No. 5 or lower was defined as being coarse, and a test piece was judged as having coarse grains if such a coarse grain size number was obtained in any one field of observation on the entire section. The observation began with the test piece that was heated to the lowest temperature at the simulative carburizing in the ascending order of the heating temperature, and the heating temperature of the test piece in which coarse grains were detected for the first time was defined as a coarse grain forming temperature. The coarse grain forming temperature was determined through observations of two test pieces for each of the different chemical compositions and production conditions.

4. Test Results and Discussions

4.1 Solution-precipitation behaviors of AlN and Nb(CN)

It is essential for controlling the distribution of fine precipitates to understand their solution-precipitation behaviors. The behaviors of AlN and Nb(CN) were studied for this end.

4.1.1 Solution behavior during heating

The solution behaviors of AlN and Nb(CN) during heating were

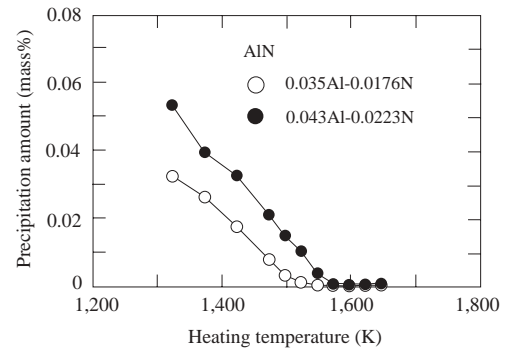


Fig. 3 Solution behavior of precipitates in AlN type steels

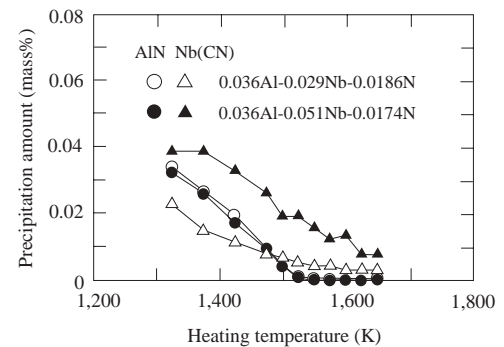


Fig. 4 Solution behavior of precipitates in Nb-addition type steels

examined by holding test pieces of the AlN and Nb-addition type specimens at different temperatures for 1.2 ks, cooling water immediately thereafter and measuring their precipitation amounts. Figs. 3 and 4 show the results. The complete solution temperature of the precipitates was higher as the addition amounts of Al, Nb and N increased. The solution temperature sometimes exceeded 1,500K when the addition amounts were large; with such a chemical composition it was found difficult to have the precipitates solve during the reheating for rolling. The solution behavior as described above can be predicted more or less accurately based on the solubility of the precipitates when the steel chemical composition is comparatively simple, but when elements such as Al, Nb, N and C are added in combination, the behavior of the precipitates becomes complicated and in such a case, use of a calculation software such as ThermoCalc is effective.

4.1.2 Precipitation behavior during cooling

The precipitation behaviors of AlN and Nb(CN) during cooling were investigated using a thermomechanical simulator, Thermecmaster-Z (Fuji Electronic Industrial Co., Ltd.). The Nb-addition type specimens were heated to 1,523K to have AlN and Nb(CN) solve once and upset at 1,273K at a reduction ratio of 50% to simulate rolling or hot forging, and then cooled at different cooling rates to obtain a CCT diagram. Samples for chemical analysis were cut out from the test pieces after cooling and the precipitation amounts of AlN and Nb(CN) were measured. Their precipitation rates at the different cooling rates were calculated relative to their respective maximum deposition amounts, which were measured separately using samples that were heated to 1,123K for 7.2 ks and then water cooled.

It is understood based on Fig. 5 and Table 2 that the AlN that is

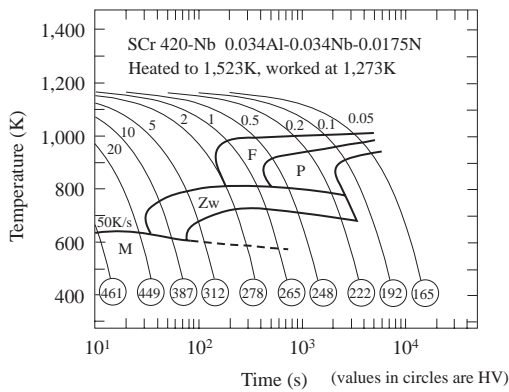


Fig. 5 Transformation behavior of SCR 420-Nb steel

Table 2 Precipitation rates of AlN and Nb(CN) during cooling

	Cooling rate(K/s)									
	50	20	10	5	2	1	0.5	0.2	0.1	0.05
AlN	2	1	2	1	1	2	2	4	4	7
Nb(CN)	24	29	32	32	32	41	44	50	68	82

in a solute state in the γ phase little precipitates under any cooling rate within a practicable range. From the above, we can say that the AlN that has deposited in an as-rolled or as-forged material is that which was not solute in the matrix at the heating stage for rolling or forging. Therefore, the precipitation amount of AlN in an as-rolled or as-forged material can be used as an indicator for judging if the precipitate has been solved sufficiently or not during the heating for rolling or forging. With respect to Nb(CN), on the other hand, it is seen in the figure and table that, although the deposition amount of the Nb(CN) that is solute in the γ phase is comparatively small when the cooling rate is comparatively high, its precipitation amount increases at the $\gamma \rightarrow \alpha$ transformation.

4.1.3 Precipitation behavior during heating

The precipitation behaviors of AlN and Nb(CN) during heating were investigated using the Nb-addition type specimens. Their precipitation amounts at different temperatures were measured by heating the steels to 1,573K for 1.8 ks to have the precipitates become solute, cooling in air, heating again at a uniform heating rate and then water cooling immediately after hitting the different designated temperatures. From Fig. 6 it is understood that the precipitation amounts of both AlN and Nb(CN) increase drastically with the $\alpha \rightarrow \gamma$

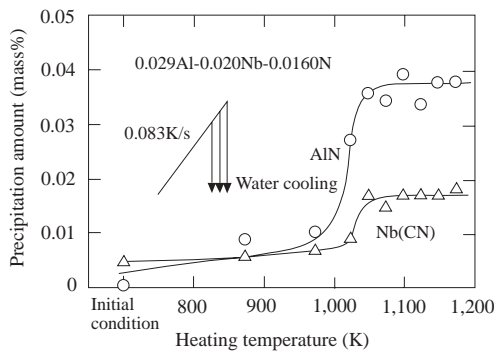


Fig. 6 Precipitation behavior of AlN and Nb(CN) during heating

transformation during heating. It follows that a deposition treatment prior to the heating for carburizing is not necessary, because even if the precipitates are in a solute state before said heating, they will precipitate quickly during the heating for carburizing.

4.2 Formation of coarse grains at production routes

The distribution of precipitates and the microstructure of the matrix are different at different production routes depending on the working and thermal history of the material. In view of this fact, the formation of coarse grains was examined at each of the production routes of typical process routes.

4.2.1 Process route comprising hot rolling, cold working and carburizing

This process route corresponds to that of the shafts that undergo drawing work and then carburizing.

Fig. 7 shows the influence of the Al addition amount over the coarse grain forming temperature of specimens Nos. 1 to 6 (AlN type). Since the addition amount of N was increased as that of Al was increased, Fig. 7 indicates that the coarse grain forming temperature becomes higher as the amount of AlN increases. This is presumably because the volume fraction of the precipitates (f) increases during carburizing. However, if Al and N are added in excess of certain amounts, the coarse grain forming temperature falls rather than rises. Fig. 8 shows the amount of non-solute AlN after rolling. When Al and N are added in great amounts as in the case of specimen No. 6, AlN cannot be solved completely at the reheating temperature for rolling of the authors' tests (1,498K). This is presumably because AlN, which would otherwise precipitate in fine

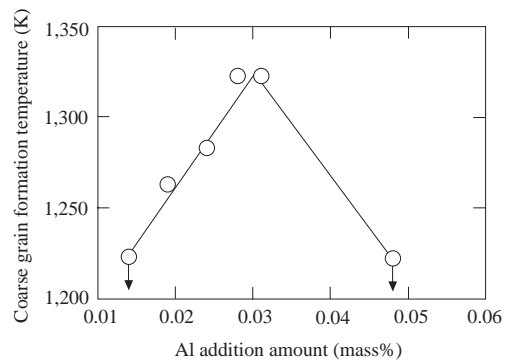


Fig. 7 Relationship between Al addition amount and coarse grain forming temperature (specimens Nos. 1 to 6)

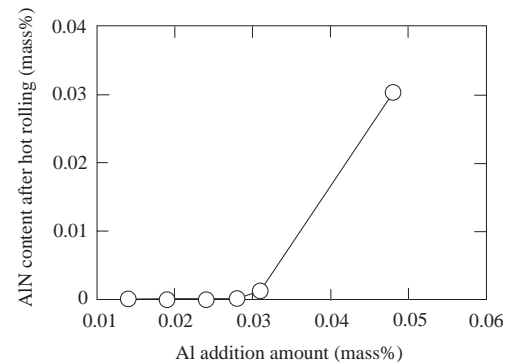


Fig. 8 Relationship between Al addition amount and non-solute AlN (specimens Nos. 1 to 6)

grains during the heating for carburizing, precipitate around nuclei of comparatively coarse non-solute AlN and as a consequence, the number of fine AlN grains that form during carburizing does not increase but decreases, lowering the coarse grain forming temperature. This indicates that excessive additions of Al and N are harmful, and there are optimum addition amounts of these elements for preventing coarse grains.

Fig. 9 shows the influence of the addition amount of Nb over the coarse grain forming temperature. The coarse grain forming temperature falls as the Nb amount increases. However, when specimen No. 9 is slow cooled (at an average cooling rate of 0.12K/s after rolling, its coarse grain forming temperature rises to the same level of specimen No. 5, to which Nb is not added.

Photos 1(a), (b) and (c) show microstructures after rolling. Whereas the microstructure of specimen No. 5 not containing Nb is a compound structure of ferrite and pearlite, that of specimen No. 9 containing Nb is a compound structure of ferrite, pearlite and bainite. Bainite is presumed to have formed as a result of an increase in hardening owing to Nb, which has solved during the heating for rolling. When the compound structure of ferrite and pearlite is formed through slow cooling, then the coarse grain forming temperature becomes higher and from this, it is understood that structural factors are involved in the fall of the coarse grain forming temperature caused by the Nb addition.

Photos 1(d), (e) and (f) show austenitic grains of materials that

were cold worked by 50% upsetting taken at the initial stage of the heating for the simulative carburizing. It is seen here that the crystal grains of the matrix of the material that contained bainite in the rolled structure have become markedly fine, and its degree of grain size mix is large. This is presumably because R_0 in equation (1) was small and Z was large and as a consequence, r_{crit} was small, resulting in the formation of coarse grains. As a conclusion, in the case of this process route, it is necessary, in addition to controlling the behavior of the precipitates, not to allow bainite to form and for this reason, due consideration must be paid in commercial production to the hardenability of the material and rolling conditions.

4.2.2 Process route comprising hot rolling, SA, cold working and carburizing

This process route corresponds to that of comparatively small gears and carburized bearings that are formed through cold working, and is characterized by including SA.

Fig. 10 shows the influence of Nb over the coarse grain forming temperature of materials that have undergone SA. The authors also confirmed that coarse grains formed at the simulative carburizing at 1,223K in all the AlN type specimens other than No. 5 (Nos. 1 to 4 and 6), which is not shown in the figure. In contrast to the process route discussed in the previous section comprising rolling, cold working and carburizing, to prevent coarse grains from forming in the process route of the present section, the use of AlN alone is insufficient and the addition of Nb is necessary. However, from the fact

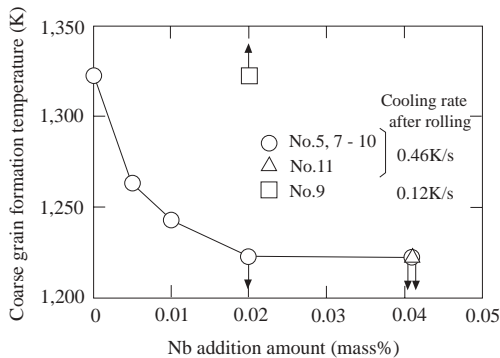


Fig. 9 Relationship between Nb addition amount and coarse grain forming temperature (specimens Nos. 5 and 7 to 11)

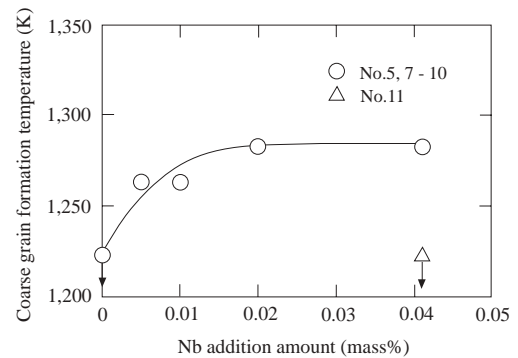


Fig. 10 Relationship between Nb addition amount and coarse grain forming temperature (specimens Nos. 5 and 7 to 11)

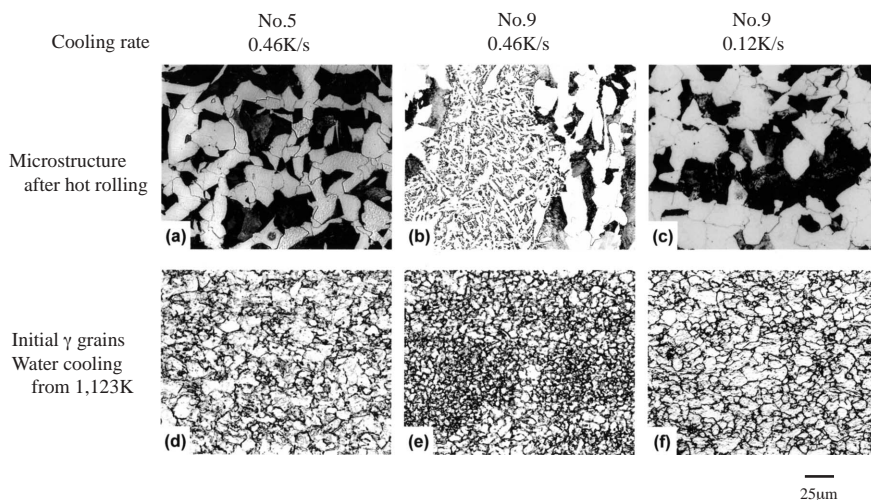


Photo 1 Effect of Nb content and cooling rate after hot rolling on microstructure and initial γ grains

that the coarse grain forming temperature of specimen No. 11, in which the addition amount of Al was decreased, is very low in spite of its high Nb content, AlN is considered to function effectively as the pinning grains also in the process route including SA.

The coarse grain forming temperature tends to increase as the addition amount of Nb increases, but the temperature stays substantially constant in the Nb content range from 0.02 to 0.04%; it is presumed from this that the coarse grain prevention effect little increases if Nb is added in excess of 0.02%. It is also noted that the coarse grain forming temperature is lower in this process route than in the process route of the previous section not including SA, and that coarse grains form more easily when SA is applied. The reasons for this are, conceivably, that the pinning particles grow coarse during the long heating time for SA, and that the distribution of the precipitates becomes biased as a result of a holding time at the α/γ two-phase temperature range. As a conclusion, in the case of the process route including SA, it is necessary to use AlN and Nb(CN) in combination for preventing coarse grains.

4.2.3 Process route comprising hot forging, normalizing and high temperature carburizing

This process route corresponds to that of large gears. Because the process route does not include cold forging, coarse grains are less likely to form. However, carburizing at high temperatures is required recently for enhancing productivity^{8,9)} and as a consequence, the prevention of coarse grains is an important issue also in this process route. The high temperature carburizing is required, in particular, for the power transmission components of a continuously variable transmission (CVT), which require a large carburized case depth¹⁰⁾.

Table 3 shows the JIS crystal grain size numbers of specimens Nos. 5 and 9 after simulative carburizing at high temperatures. In either of specimens Nos. 5 (AlN type) and 9 (Nb-addition type), coarse grains do not appear up to a carburizing temperature of 1,343K, but specimen No. 9 is considered better for preventing the formation of coarse grains stably because f in equation (1) is larger. Thus, it has been confirmed that with the developed steel, as a result of dispersing fine precipitates in a great amount by optimizing the chemical composition and production processes, coarse grains are prevented from forming even when carburizing at temperatures as high as 1,323 to 1,343K is applied.

Table 3 JIS grain size number after simulative carburizing at high temperatures

Steels	1,323K × 5.4ks	1,333K × 5.4ks	1,343K × 5.4ks	1,343K × 18ks
No.5	#8.4	#8.4	#8.5	#7.5 + 6.4
No.9	#8.9	#9.0	#8.6	#7.7

5. Summary

Based on the technique for dispersing fine precipitates in a great amount by optimizing the chemical composition and production processes of the steels, the authors investigated the behavior of forming of coarse grains in steels for carburizing use. The following have been made clear as a result:

- (1) Whereas the AlN that is in a solute state in the γ phase does not precipitate during cooling at any cooling rate within a practicable range, Nb(CN) precipitates partially during the $\gamma \rightarrow \alpha$ transformation. At a heating process, on the other hand, the deposition of both AlN and Nb(CN) increases rapidly at the $\alpha \rightarrow \gamma$ transformation.
- (2) Process route comprising hot rolling, cold working and carburizing: the coarse grain forming temperature becomes higher as the amount of AlN increases, but, Al and N have harmful effects when added excessively, and there are optimum addition amounts of these elements for preventing coarse grains. The coarse grain forming temperature is lowered when Nb is added. This is because bainite forms, and R_0 in equation (1) decreases and Z increases during carburizing.
- (3) Process route comprising hot rolling, SA, cold working and carburizing: the coarse grain forming temperature becomes higher as the addition amount of Nb increases, but AlN also work effectively as the pinning particles. The effect of Nb tends to be saturated when Nb is added by 0.02% or so.
- (4) Process route comprising hot forging, normalizing and high temperature carburizing: coarse grains did not form in either of specimens Nos. 5 (AlN type) and 9 (Nb-addition type) even when the temperature of the simulative carburizing was as high as 1,343K. It has been confirmed that, with the developed steel, coarse grains are prevented from forming even when high temperature carburizing at 1,323 to 1,343K is applied.

References

- 1) Nishizawa, T.: Tetsu-to-Hagané. 70(15), 1984(1984)
- 2) Gladman, T.: Proc. Roy. Soc. A. 294, 298(1966)
- 3) Gladman, T., Pickering, F. B.: JISI. 205(6), 653(1967)
- 4) Kinoshita, S., Ueda, T., Suzuki, A.: J. of Japan Institute of Metals. 36(5), 403(1972)
- 5) Kinoshita, S., Ueda, T., Suzuki, A.: Tetsu-to-Hagané. 59(8), 1125(1973)
- 6) Fujita, K., Sakari, T., Ohnishi, M., Noma, T.: Tetsu-to-Hagané. 59(8), 1131(1973)
- 7) Koyasu, Y.: Heat Treatment. 26(6), 405(1986)
- 8) Harada, N., Yamada, Y., Katoh, K.: Mitsubishi Motors Technical Review. 12, 47(2000)
- 9) Ureshino, Y., Nakano, O., Fukuda, K., Fujiwara, T., Fukuda, Y.: Toyota Technical Review. 52(1), 84(2002)
- 10) Fujiwara, K., Date, Y., Ariyasu, T.: Mitsubishi Motors Technical Review. 13, 114(2001)