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Effects of Niobium Additions on the Structure, Depth, and Austenite Grain Size of the Case of Carburized 0.07% C Steels

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Carbon (0.07%) steel samples containing about 0.04% Nb singly and in combination with nitrogen were carburized in a natural Titas gas atmosphere at a temperature of 1223 K (950 °C) and a pressure of about 0.10 MPa for 1/2 to 4 h, followed by slow cooling in the furnace. Their microstructures were studied by optical microscopy. The austenite grain size of the case and the case depths were determined on baseline samples of low-carbon steels and also on niobium and (Nb + N) microalloyed steel samples.

It was found that, when compared to the baseline steel, niobium alone or in combination with nitrogen decreased the thickness of cementite network near the surface of the carburized case of the steels. However, niobium in combination with nitrogen was more effective than niobium in reducing the thickness of cementite network. Niobium with or without nitrogen inhibited the formation of Widmanstätten cementite plates at grain boundaries and within the grains near the surface in the hypereutectoid zone of the case.

It was also revealed that, when compared to the baseline steel, niobium decreased the case depth of the carburized steels, but that niobium with nitrogen is more effective than niobium alone in reducing the case depth. Niobium as niobium carbide (NbC) and niobium in the presence of nitrogen as niobium carbonitride, [Nb(C,N)] particles refined the austenite grain size of the carburized case, but Nb(C,N) was more effective than NbC in inhibiting austenite grain growth.

Keywords
austenite grain size, carbon steel, carburizing, case hardening

1. Introduction

HEAVY-DUTY applications such as drive shafts, transmission gears, and bus and truck gears, demand an increased hardness and wear-resistant surface with a suitable tough core. Moreover, advanced aircraft and helicopter applications require high-duty transmission gear materials. These gear materials must be able to retain their hardness and strength at elevated temperatures up to about 588 K (315 °C). On the other hand, improved fuel efficiency for some high-consumption vehicles may be achieved by reducing the weight of the vehicle components. For this, the growing trend is to couple the engine with smaller transmissions. Consequently, higher stresses are imposed on the transmissions. Such parts made of plain carbon steel do not retain their strength and hardness at elevated temperature. Therefore, parts used for heavy-duty and high-temperature applications usually are made of low-carbon low-alloy steels. To achieve the required properties, the parts are then carburized, followed by hardening.

The carburizing behavior of plain carbon steels is well established. A number of studies (Ref 1-7) have already been made on the carburizing behavior of low-alloy steels containing nickel, chromium, molybdenum, vanadium, and so forth. Very little is known about the carburizing behavior of niobium microalloyed steel. The present work has been undertaken to study the effects of the addition of about 0.04% Nb separately and in combination with about 0.02% N on the structure, depth, and austenite grain size of the case of carburized low-carbon steels.

2. Experimental Procedure

2.1 *Materials, Specimen Preparation, and Carburization*

Three different steels with the compositions given in Table 1 were used in this work. Of the three, steel 1 is the base steel with which the carburizing behavior of the other two steels containing niobium alone or in combination with nitrogen was compared. Locally available natural Titas gas, a common carburizing medium (Ref 8) with an input composition of 97.2% methane, 1.8% ethane, 0.3% propane, 0.2% butane and higher hydrocarbons, and 0.3% nitrogen (Ref 9), was used for carburization. From the stock materials, specimens about 10 by 10 by 8 mm in size were prepared and carburized in a sealed chamber of a gas-fired gas-carburizing furnace with an automatic control system. The details of the furnace, control system, and carburizing procedure are available elsewhere (Ref 10).

To study the effect of gas flow rate on case depth, the carburization was carried out at three different flow rates—0.17, 0.28, and 0.34 m^3/h (6, 10, and 12 ft^3/h) at a constant temperature of 1223 K (950 °C) with approximately 0.10 MPa (15 psia) gas pressure for 1 h. For each case, a constant flow rate was maintained using a flowmeter.

After the predetermined time, the firing was stopped and the specimens allowed to cool in the chamber to room temperature. The gas flow through the carburizing chamber was maintained during the cooling period until the temperature in the chamber reached about 773 K (500 °C). This was done as a measure of

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precaution to prevent any possible oxidation due to infiltration of air. Following the same procedure, another batch of steel samples was carburized at the identical conditions with a gas flow rate of 0.34 m^3/h (12 ft^3/h) for five different time periods: 1/2, 1, 2, 3, and 4 h.

2.2 *Optical Microscopy and Measurement of Case Depth*

The carburized and furnace-cooled specimens were cut into two pieces at right angles to reveal the section orthogonal to the treated surface. One piece of each specimen was then mounted, polished by standard techniques, and etched in 2% nital. The microstructures of these specimens were examined by optical microscope.

Case depth is usually specified as the depth below the surface of a carburized part at which a defined value of some property occurs. The depth of carbon penetration from the surface toward the interior containing carbon up to 0.4 wt% of the carburized specimens was considered to be an effective case depth (Ref 11). The effective case depth was measured linearly from the microstructures of these specimens by using a micrometer eyepiece fitted to an optical microscope (Shimadzu Model No. 3261, Shimadzu Scientific Instruments, Inc., Columbia, MD).

2.3 *Determination of Austenite Grain Size of the Carburized Case*

The three important zones of the carburized case are the hypereutectoid zone, the eutectoid zone, and the proeutectoid zone. It is very difficult to reveal the grain boundaries of eutectoid steels. The grain size of the case thus was obtained by taking the average grain size of the hypereutectoid and proeutectoid zones. For austenite grain size measurement, the mean linear intercept method (Ref 12) was employed. A total of about 300 to 400 intercepts were counted for each specimen.

3. Experimental Results

3.1 *Optical Microscopy*

The structures in the case and core of specimens of steels 1 to 3 carburized for different time periods and cooled slowly in the furnace were studied by optical microscope. The optical micrographs of specimens carburized for 1 and 4 h are presented in Fig. 1 and 2, respectively.

In these carburized specimens, three different zones were distinguished:

- *Hypereutectoid zone,* consisting of pearlite and cementite network
- *Eutectoid zone,* consisting of only lamellar pearlite
- *Hypoeutectoid zone,* consisting of ferrite and pearlite

The cementite networks of steel 1 were found to be thicker than those of steels 2 and 3. Steel 2 showed thinner cementite networks than steel 1 but slightly thicker ones than steel 3 for all the carburizing periods. In all three steels, a gradual thickening tendency of the cementite networks with increasing carburizing time was observed. In each case, the depth of hypereutectoid zone in steel 3 was found to be lower than those of steels 1 and 2.

Steel 1 carburized for 4 h showed some Widmanstätten cementite plates nucleated at the grain boundary and within the grain (Fig. 2a) in the hypereutectoid zone near the surface. No Widmanstätten cementite plates were found in the structures of steels 2 and 3 for any carburizing period.

3.2 *Case Depth*

The variation of case depth with gas flow rate for steels 1 to 3 carburized for 1 h and cooled slowly in the furnace is shown in Table 2. It is clear that the effective case depth for all the steels increases with increasing gas flow rate.

The effective case depth of steels 1 to 3 carburized for 1/2, 1, 2, 3, and 4 h at a gas flow rate of 0.34 m^3/h (12 ft^3/h) and cooled slowly in the furnace are shown in Table 3 and plotted in Fig. 3 as a function of carburizing time. It is also evident that under identical carburizing conditions, steel 1 produced higher case depth than steels 2 and 3, and that steel 2 produced somewhat higher case depth than steel 3.

3.3 *Prior Austenite Grain Size*

Prior austenite grain size is important in that it controls the structures of both the case and the core of a carburized steel. Therefore, the grain size in the case of carburized and slowly cooled specimens of steels 1 to 3 was measured for different carburizing times. The data thus obtained are listed in Table 4 and plotted in Fig. 4 as a function of carburizing time.

It is evident that the prior austenite grain size of steels 1 to 3 increases with increasing carburizing time. It is also clear that plain carbon steel 1 produced the coarsest grain size, whereas steel 3 produced the finest grain size of all the steels and steel 2 produced a coarser grain size than steel 3. Figure 4 shows that the prior austenite grain sizes of steels 2 and 3 are very close to each other for lower carburizing periods. However, the difference between them increases with the increasing carburizing time. Figure 2 shows the grain size of the case of steels 1 to 3 carburized for 4 h and cooled slowly in the furnace.

Table 1 Compositions of steels

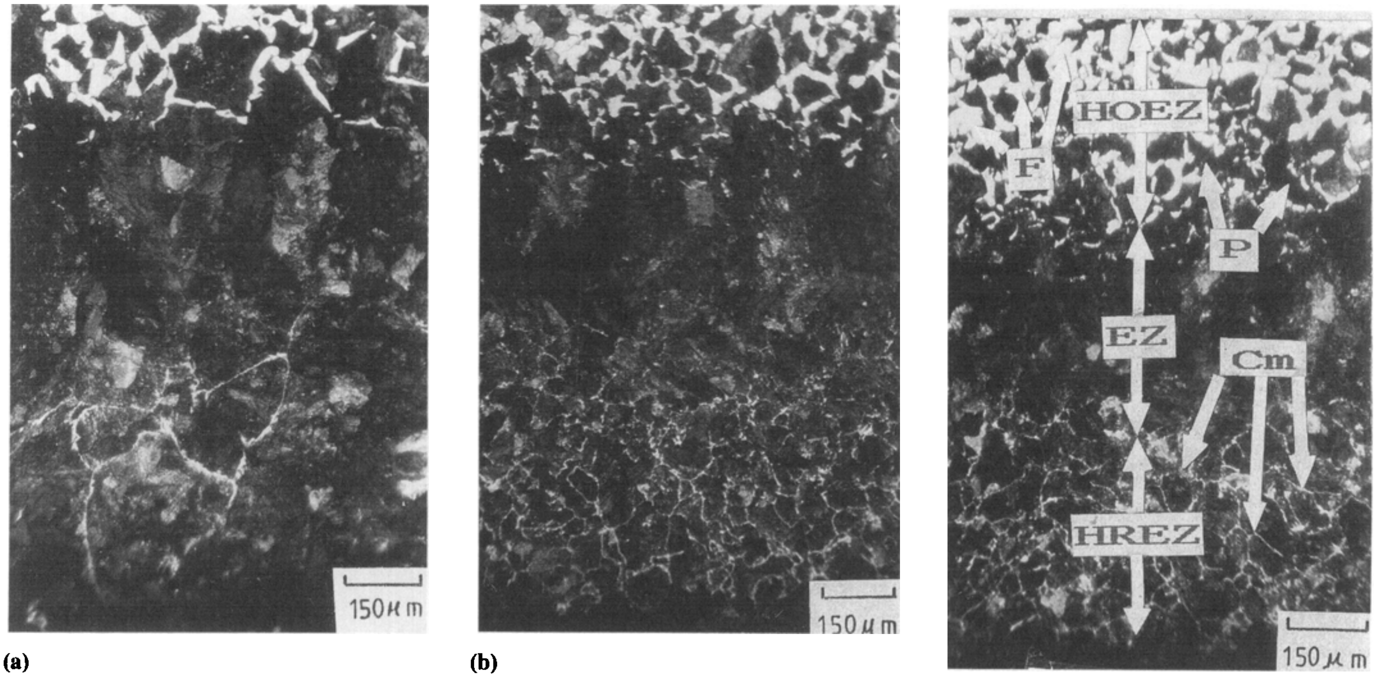
Steel No.	Composition, wt %						
	C	Si	Mn	Nb	N	S	P
1	0.07	0.22	0.55	...	0.002	0.012	0.010
2	0.07	0.23	0.58	0.04	0.001	0.015	0.016
3	0.07	0.24	0.58	0.04	0.020	0.011	0.012

4. Discussion

4.1 *Metallography*

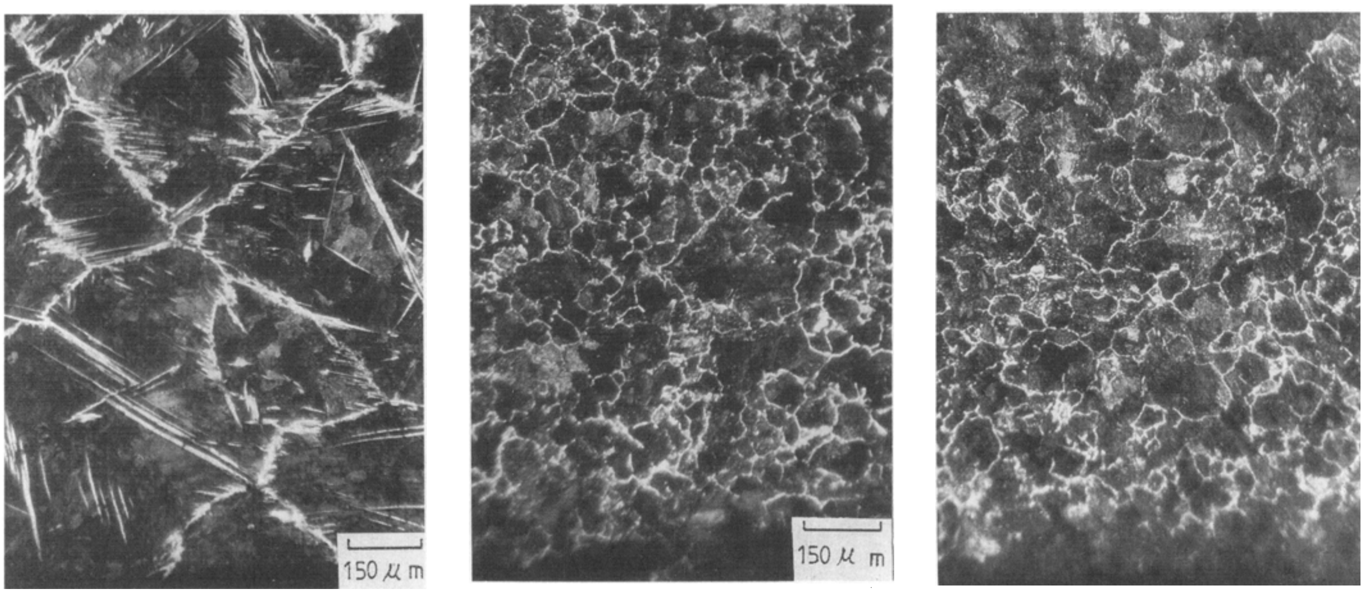
The cementite networks of steel 1 were found to be thicker than those of steels 2 and 3, and steel 2 produced thicker cementite networks than steel 3 (Fig. 1 and 2). This phenomenon indicates that the carbon concentration in the case of steel 2 is lower than that of steel 1 but higher than that of steel 3.

For all three steels, a gradual thickening tendency of the cementite networks with carburizing time was observed. This is because, at a certain temperature, the austenite can absorb a certain amount of carbon, making an interstitial-type solid solution. Any carbon in excess of the fixed amount is expelled as



(a) (b) (c)

Fig. 1 Optical micrographs showing microstructures and depth of case of steels 1 to 3 carburized at 1223 K (950 °C) for 1 h and cooled slowly in the furnace. F, ferrite; P, pearlite; Cm, cementite; EZ, eutectoid zone; HOEZ, hypoeutectoid zone; HREZ, hypereutectoid zone. (a) steel 1. (b) steel 2. (c) steel 3.



(a) (b) (c)

Fig. 2 Optical micrographs showing microstructures and prior austenite grain size in the case of steels 1 to 3 carburized at 1223 K (950 °C) for 4 h and cooled slowly in the furnace. (a) steel 1. (b) steel 2. (c) steel 3.

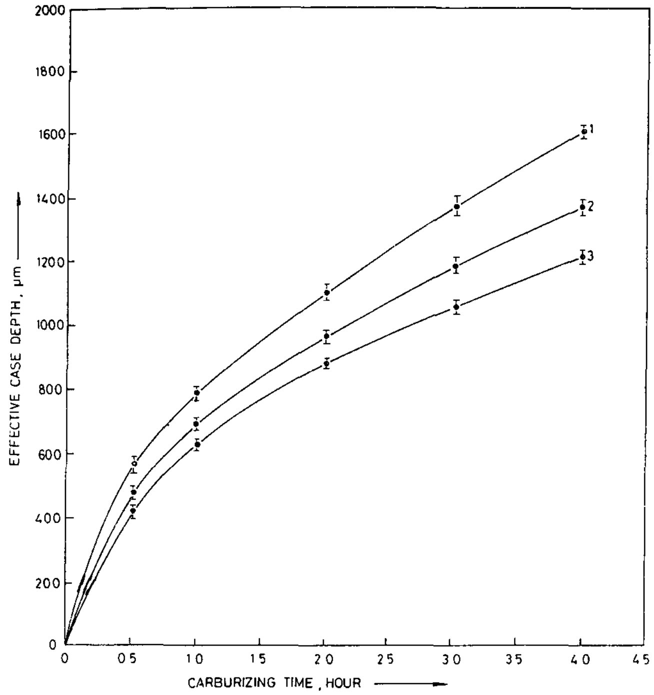


Fig. 3 Variation of effective case depth with carburizing time of steels 1 to 3. Error bars are incorporated.

Table 2 Effective case depth of steels 1 to 3 carburized for 1 h at different gas flow rates
Temperature, 950 °C; pressure, 0.10 MPa

Steel No.	Case depth, μm 0.17 m^3/h	 0.28 m^3/h	 0.34 m^3/h
1	510	700	790
2	430	600	690
3	400	560	610

cementite precipitates at the austenite grain boundaries. As the carburizing time increases, more and more carbon is expelled as cementite precipitates, making the cementite network thicker.

Some Widmanstätten cementite plates nucleated at the grain boundary and within the grains near the surface of steel 1 carburized for 4 h and cooled slowly in the furnace. This nucleation was due to the supersaturation of austenite with respect to carbon. Because Widmanstätten cementite plates were present only in steel 1, it is clear that under identical conditions, the carbon-absorbing capacity of plain carbon steel 1 is much higher than that of microalloyed steel containing niobium or niobium in combination with nitrogen. This higher carbon-absorbing capacity of steel 1 makes the austenite supersaturated more readily than in steels 2 and 3. After carburizing and furnace cooling, the austenite transforms to pearlite while the Widmanstätten cementite plates remain unchanged. The absence of Widmanstätten cementite plates in the carburized and furnace-cooled steels 2 and 3 indicates that niobium with or without nitrogen decreases carbon activity in austenite, thus causing it to be less saturated with carbon.

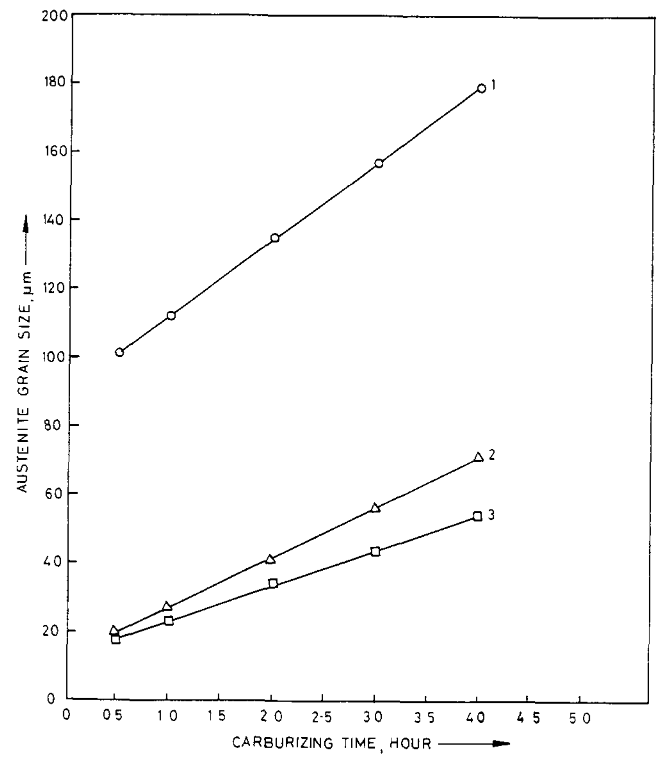


Fig. 4 Variation of prior austenite grain size with carburizing time of steels 1 to 3

4.2 *Case Depth*

From Table 2 it is clear that with increasing gas flow rate, the effective case depth increased for all the steels. With the increase in gas flow rate, the reservoir of available carburizing gas inside the carburizing chamber was increased. This increase in concentration increased the carburizing rate.

It is clear that the microalloyed steels 2 and 3 produced smaller case depths than the plain carbon steel 1 (Table 3 and Fig. 3). This indicates that the microalloying element niobium with or without nitrogen reduces the case depth. The highest case depth in steel 1 is due to the much higher concentration of carbon at its surface, which is clearly indicated by the presence of a thicker cementite network and Widmanstätten cementite plates in its carburized case.

The formation of vanadium carbide reduces the solubility of carbon in austenite (Ref 13) and hence decreases the carbon concentration of the surface of the steel specimen (Ref 5). The much lower case depth in steel 2 with niobium carbide (NbC) than the base steel 1 indicates that the formation of NbC also reduces the solubility of carbon in austenite.

Steel 3 is basically steel 2 with nitrogen added to it. This steel produced lower case depth than steel 2. The lower case depth of steel 3 is also due to the lower concentration of carbon at its surface, which is indicated by the presence of a thinner cementite network in the case of this steel. The lower concentration of carbon and the consequent lower case depth in steel 3 are clearly due to the presence of nitrogen, which is thought to decrease the solubility of carbon in austenite. The presence of nitrogen as niobium carbonitride [Nb(C,N)] is also thought to decrease the diffusion coefficient by increasing the activation

Table 3 Effective case depth of steels 1 to 3 for different carburizing periods with 95% confidence limit

Steel No.	Case depth, μm 1/2 h	1 h	2 h	3 h	4 h
1	550 ± 15	790 ± 14	1100 ± 15	1380 ± 18	1610 ± 14
2	450 ± 13	690 ± 13	950 ± 14	1180 ± 15	1370 ± 16
3	415 ± 13	610 ± 13	880 ± 12	1060 ± 16	1210 ± 16

energy; consequently, a case depth lower than that of steel 2 is obtained in steel 3.

4.3 *Prior Austenite Grain Size*

Steel 1 is a plain carbon steel having no second-phase particles to inhibit austenite grain growth, and thus the austenite grain size of this steel increases rapidly with increasing time at the carburizing temperature (Fig. 4). Microalloyed steel 2 contains niobium, which combines with carbon to form NbC, and steel 3 contains niobium in combination with nitrogen, which forms Nb(C,N). These second-phase particles pin the prior austenite grain boundaries and inhibit grain growth, causing the steels to retain a fine grain size, which is found to increase slightly during carburizing with successively longer holding times.

It is also evident from the experimental results that microalloyed steel 3 produced slightly finer austenite grains than steel 2. It has been reported that the temperatures for the precipitates in steels 2 and 3 to dissolve completely are 1408 and 1435 K (1135 and 1162 °C, respectively) (Ref 14). Therefore, it is expected that grain-growth restriction in steel 3 will persist to a higher temperature than in steel 2. However, at the lower temperature of 1223 K (950 °C), where the precipitates in these two steels were still effective as grain-growth inhibitors, steel 3 produces a finer grain size than steel 2, suggesting that Nb(C,N) is more effective than NbC in controlling the grain growth of austenite. The prior austenite grain sizes of steels 1 to 3 at 1223 K (950 °C) prior to any treatment are reported to be 46, 15, and 9 μm, respectively (Ref 14). This confirms that Nb(C,N) particles are more effective than NbC particles in restricting the grain growth of austenite. Rios and Honeycomb (Ref 15) also observed the grain-refining effect of niobium. Bepari and Whiteman (Ref 16) reported that the undissolved particles of Nb(C,N) in steel 3 are finer than the undissolved particles of NbC in steel 2. The greater effect of these Nb(C,N) particles compared to NbC particles in restricting grain growth is clearly due to their finer size.

For the lower carburizing period, the prior austenite grain size of steels 2 and 3 are very close to each other. With increasing carburizing time, the difference between the grain sizes of the two steels increases. This is thought to be due to the fact that Nb(C,N) particles coarsen less rapidly with increasing carburizing time because of their higher solution temperature compared to NbC particles.

Table 4 Prior austenite grain size of the carburized case of steels 1 to 3 for different carburizing periods

Steel No.	Austenite grain size, μm 1/2 h	1 h	2 h	3 h	4 h
1	101	112	135	158	180
2	19	27	42	56	71
3	18	24	34	43	53

5. Conclusions

- Niobium alone or in combination with nitrogen decreases the thickness of the cementite network near the surface of the carburized steels, but niobium with nitrogen is more effective than niobium in reducing the thickness of the cementite network.
- Niobium with or without nitrogen inhibits the formation of Widmanstätten cementite at the grain boundary and within the grains near the surface of the carburized steels.
- Niobium with or without nitrogen reduces the case depth of the carburized steels. Niobium with nitrogen is more effective than niobium alone in reducing the case depth.
- Undissolved particles of NbC and Nb(C,N) inhibit the grain growth of austenite in the carburized cases of low-carbon steels. However, Nb(C,N) is more effective than NbC in reducing the grain growth of austenite.
- As the carburizing time increases, the grain-restricting capacity of NbC decreases more readily than that of Nb(C,N).

References

1. S. Preston, *Mater. Sci. Technol.*, Vol 9, July 1993, p 615-619
2. V.I. Slavov, V.N. Zadorozhnaya, and A.I. Slavova, *Russ. Metall.*, Vol 3, 1992, p 83-88
3. B.M. Khusid, *Wear*, Vol 165, 1993, p 109-112
4. S. Preston, *J. Heat Treat.*, Vol 8 (No. 2), 1990, p 93-99
5. M.N. Haque, M.Sc. engineering thesis, Bangladesh University of Engineering and Technology, Dhaka, 1988
6. M.N. Haque and M.M.A. Bepari, *J. Inst. Eng., (Bangladesh)*, Vol 17 (No. 1), Jan 1989, p 37-47
7. N. Geru and T. Vasile, *Metallurgica (Bucharest)*, Vol 10, 1994, p 10-11
8. M.M.A. Bepari and M. Ibrahim, *J. Inst. Eng. (Bangladesh)*, Vol 11 (No. 2), April 1983, p 23-35
9. "Natural Resources and Industrial Prospects in Bangladesh," Seminar, Department of Chemical Engineering, Bangladesh University of Engineering and Technology, Dhaka, 1972, p B-2
10. M.A. Islam, M.Sc. engineering thesis, Bangladesh University of Engineering and Technology, Dhaka, 1994
11. C.A. Stickels, in *ASM Handbook*, Vol 4, ASM International, 1991, p 312-324
12. M.M.A. Bepari, M.Met. thesis, University of Sheffield, U.K., 1978
13. J.H. Woodhead, "Vanadium in High Strength Steel," in seminar proceedings (Chicago), 1979, p 3
14. M.Z. Haque, M.Sc. engineering thesis, Bangladesh University of Engineering and Technology, Dhaka, 1989
15. P.R. Rios and R.W.K. Honeycomb, *Mater. Sci. Technol.*, Vol 8, 1992, p 1057-1062
16. M.M.A. Bepari, and J.A. Whiteman, *J. of Mat. Proc. Tech.*, Vol. PRO 056/1-4, 1996, p 834-846