Effects of Molybdenum Additions on the Structure, Depth, and Austenite Grain Size of the Case of Carburized 0.13% Carbon Steels

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(Submitted 28 May 2002)

Carbon (0.13%) steel samples containing about 0.48% molybdenum (Mo) singly and in combination with nickel (Ni) were carburized in a natural Titas gas atmosphere at a temperature of 1223 K (950 °C) and at a pressure of about 0.10 MPa (15 psia) for time periods ranging from 1-4 h followed by slow cooling in the furnace. Their microstructure was studied by optical microscopy. The austenite grain size of the case and the case depths were determined. It was found that Mo and Ni alone and in combination decrease the thickness of the cementite network near the surface of the carburized case of the steels. However, Ni is found to be more effective than Mo in decreasing the thickness of cementite network. Both Mo and Ni enhance the formation of Widmanstätten cementite plates at the grain boundary and within the grains near the surface of the carburized steels. However, Ni alone is more effective than Mo in the formation of Widmanstätten cementite plates. In the presence of Ni, Mo is much more effective in the formation of Widmanstätten cementite plates than Mo in absence of Ni. It was also revealed that both Mo and Ni increased the case depth. Ni is more effective than Mo in increasing the case depth. The combined effect of Mo and Ni is much greater than that of either Mo or Ni alone in increasing case depth. Mo as Mo carbide (Mo₂C) particles refined the austenite grain size of the carburized case. Ni in solution was not found to have any effect in restricting grain growth of austenite, but the presence of Ni enhances the austenite grain size refining effect of Mo in the carburized case.

Keywords	carburization, har	dness depth,	hypoeutectoid steel,
	molybdenum, nic	kel	

1. Introduction

Machine parts used for heavy-duty applications such as drive gear, drive shafts, cam shafts, bus and truck gears, crankshafts, axles, etc., require increased hardness and wear resistance in the case and better toughness in the core than those of common parts made of low carbon (C) steel in the carburized and hardened conditions. Moreover, advanced aircraft and helicopters require high-duty transmission gear material, which must be able to retain their hardness and strength at elevated temperature up to approximately 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. Those parts made of plain C steel do not retain their strength and hardness at elevated temperatures. Therefore, the parts used for heavy-duty and high temperature applications are usually made of low-C low-alloy steels. To achieve the required properties, the parts are carburized, followed by hardening.

The carburizing behavior of plain C steel is well established. Some studies^[1-11] have already been conducted on the carburizing behavior of low-alloy steels containing nickel (Ni), chromium, molybdenum (Mo), vanadium, and niobium, but detailed information is not available about carburizing behavior of low C steels containing Mo. The effect of Ni on the carburizing behavior is also not clear. This work, therefore, was undertaken to study the effects of the addition of about 0.48% Mo and about 1.26% Ni, separately and in combination, on the structure, case depth, and austenite grain size of the case of carburized low C steels.

2. Experimental Procedure

2.1 Materials, Specimen Preparation, and Carburization

Four different steels were used in this work. The composition of the steels is given in Table 1. The steels were made in an air induction furnace. All the melts were teemed at about 1600 °C and produced sound ingots. All the ingots were rolled down to a size of about 16 mm in diameter and were used as the stock materials for carburizing. Steel 1 is the base steel with which the carburizing behavior of the other three steels (Steels 2-4) containing Mo and Ni alone, and also in combination, was compared. Locally available natural Titas gas, a successful carburizing medium^[12] with an input composition of 97.2% methane, 1.8% ethane, 0.3% propane, 0.2% butane and higher hydrocarbon, and 0.3% nitrogen,^[13] was used for carburization. From the stock materials, specimens with dimensions of about 10 × 10 × 8 mm were prepared and carburized in a sealed chamber of a gas-fired gas-carburizing furnace with an automatic control system at a constant temperature of 1223 K (950

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Table 1Composition of Steels

Steel No.	Composition, wt.%						
	С	Si	Mn	Мо	Ni	S	Р
1	0.13	0.18	1.20			0.021	0.028
2	0.13	0.17	1.15	0.48		0.013	0.026
3	0.14	0.17	1.31	0.49	1.26	0.023	0.025
4	0.14	0.16	1.40		1.26	0.027	0.038

°C) with approximately 0.10 MPa (15 psia) gas pressure and a gas flow rate of 0.34 m³/h (12 ft³/h) for four different time periods: 1, 2, 3, and 4 h. After the predetermined time, the firing was stopped, and the specimens were 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 came down to about 400 °C. This was done as a measure of precaution to prevent any possible oxidation due to infiltration of air.

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 microstructure of these specimens was examined by optical microscopy. The case depth is usually specified as the depth of C penetration below the surface of a carburized part at which a definite value of some property occurs. The depth of C penetration from surface toward the interior containing C up to 0.4 wt.% of the carburized specimens was considered an effective case depth.^[14] The effective case depth was measured linearly from the microstructures of these specimens by using a micrometer eyepiece fitted to an optical microscope.

2.3 Determination of the 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 the eutectoid steels. The grain size of the case was thus obtained by taking the average grain size of hypereutectoid and proeutectoid zones. The mean linear intercept method was used for austenite grain size measurement. A total of about 400-500 intercepts were counted for each specimen.

3. Experimental Results

3.1 Optical Microscopy

The structures in the case and core of specimens of Steels 1-4 carburized for different time periods and cooled slowly in the furnace were studied by optical microscopy. Three different zones were distinguished in the microstructure of the carburized layer: (1) hypereutectoid zone, consisting of pearlite and cementite, forming a network along the former austenite grains, (2) eutectoid zone, consisting of only lamellar pearlite, and (3) hypoeutectoid zone, consisting of pearlite and ferrite. Typical optical micrographs of specimens of Steels 1-4 carburized for 3 h and cooled slowly in the furnace are presented in Fig. 1.

The cementite networks of Steel 1 were found to be the thickest, and that of Steel 4, the thinnest of all the steels. Steel 2 showed cementite networks thinner than those of Steel 1 but thicker than those of Steel 3. In all four steels, a gradual thickening tendency of the cementite networks with increasing time was observed.

In all four steels, Widmanstätten cementite plates were found to be nucleated at the grain boundary and within the grain in the hypereutectoid zone near the surface. Steel 3 showed the highest number, and Steel 1 showed the lowest number of Widmanstätten cementite plates of all the steels. Steel 4 showed a higher number of Widmanstätten cementite plates than Steel 2 did. The lowest number of Widmanstätten cementite plates was found in Steel 1. The amount of the Widmanstätten cementite plates was found to increase with an increase in carburizing period.

Heterogeneity in the grain structure was observed in the hypereutectoid zone of the case of Steel 1 carburized for time periods ranging from 1-4 h. In Steels 2 and 3, regular pearlite was not found. A ferrite-pearlite structure was observed in the core of all the carburized steels (Fig. 1).

3.2 Case Depth

The effective case depth of the carburized and furnace cooled Steels 1-4 under the identical conditions are shown in Table 2 and plotted in Fig. 2 as a function of carburizing time.

The graphical representation of the effective case depth values against the carburizing time for all the steels indicates that at the specified carburizing temperature, the growth of the carburized layer is parabolic with carburizing time. Table 2 and Fig. 2 show that Steel 2 with Mo produced higher case depth than plain C (base) Steel 1 and lower case depth than Steel 4 with Ni, while Steel 3, with both Mo and Ni, produced the highest case depth of all the steels.

3.3 Prior Austenite Grain Size

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

Table 3 and Fig. 3 show that the grain size of all the steels increases with increasing carburizing time. It is also clear that plain C Steel 1 produced the coarsest grain size, whereas Steel 3 produced the finest grain size of all the steels, and Steel 2 produced slightly coarser grains than Steel 3 did. Steel 4 produced grain size similar to that of plain C steel. Typical optical micrographs illustrating the grain size of the case of Steels 1-4 carburized for 3 h and cooled slowly in the furnace are shown in Fig. 4.

4. Discussion

4.1 Metallography

Optical microscopy revealed thinner cementite networks surrounding pearlite near the surface in the hypereutectoid zone of Steels 2-4 than in Steel 1. Steel 3 produced thinner cementite



Fig. 1 Optical micrographs showing microstructure and depth of case of Steels 1-4 carburized at 1223 K (950 °C) for 3 h and cooled slowly in the furnace: (a) Steel 1, (b) Steel 2, (c) Steel 3, (d) Steel 4

networks than Steel 2 did. For all four 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 C, making an interstitial-type solid solution. Any C in excess of the fixed amount is expelled as cementite precipitates at the austenite grain boundaries. As the carburizing time increases, more and more C is expelled as cementite precipitates, making the cementite network thicker. The presence of Widmanstätten cementite plates nucleated at the grain boundary and within the grains in the hypereutectoid zone near the surface of all the steels is due to the supersaturation of austenite with respect to C. Steel 1 showed smaller numbers of Widmanstätten cementite plates than the other three steels did, indicating clearly that the supersaturation of austenite with respect to C in plain C Steel 1 is less than that in other steels.

Among Steels 2-4, Steel 4 produced a higher number of

Widmanstätten cementite plates than Steel 2 and a lower number of Widmanstätten cementite plates than Steel 3. This indicates that the supersaturation of austenite with respect to C in Steel 4 is higher than that in Steel 2 and lower than that in Steel 3.

Thus, it is clear that both Mo and Ni enhance the formation of Widmanstätten cementite plates both at the grain boundary and within the grains. However, Ni is more effective than Mo in the formation of Widmanstätten cementite plates. The combined effect of Mo and Ni in the formation of Widmanstätten cementite plates is greater than that of either Mo or Ni alone.

4.2 Case Depth

The effective case depth of Steels 1-4 is plotted in Fig. 2 as a function of carburizing time and presented in Table 2. Steel 1 is the base steel to which the carburizing behavior of the other steels (Steels 2-4) with Mo and Ni alone and in combination are compared. Table 2 and Fig. 2 show that Steels 2-4 produced higher case depth than the plain C Steel 1. Among Steels 2-4, Steel 4 with Ni produced higher case depth than Steel 2 with Mo did. This indicates clearly that both Mo and Ni alone increase the thickness of the carburized layer, and Ni is more

Table 2Effective Case Depth of Steels 1-4 for DifferentCarburizing Periods

Steel No.	Case Depth, µm			
	1 h	2 h	3 h	4 h
1	620	930	1175	1300
2	710	1055	1320	1460
3	940	1300	1580	1720
4	810	1170	1460	1580



Fig. 2 Variation of effective case depth with carburizing time of Steels 1-4

effective than Mo in increasing the case depth. The lowest case depth in Steel 1 is due to the much lower concentration of C at its surface, which is clearly indicated by the presence of the smallest number of Widmanstätten cementite plates in its carburized case.

Ni increases the diffusion coefficient, D_{γ} , of C in austenite by reducing the activation energy of diffusion.^[15] The case depth of Steel 4 with Ni higher than that of the plain C Steel 1 is thus obviously due to the higher diffusion coefficient.

Steel 2 showed higher number of Widmanstätten cementite plates than Steel 1 and lower number of Widmanstätten cementite plates than Steel 4. This indicates that the C concentration at the surface of Steel 2 is greater than that of Steel 1 and lower than that of Steel 4. The higher case depth in Steel 2 than that of Steel 1 and lower case depth than that of Steel 4 is thus clearly due to the above variation of C concentration.

The case depth in Steel 2 higher than that of Steel 1 and lower than that of Steel 4 indicates that Mo increases the diffusion coefficient, D_{γ} , of C in austenite by reducing the activation energy of diffusion, but not as high as that caused by Ni.

The highest case depth in Steel 3 containing both Mo and Ni is due to much higher concentration of C at its surface, which is clearly indicated by the presence of higher number of Widmanstätten cementite plates at the grain boundaries and within the grains in its carburized case.

Table 3Prior Austenite Grain Size of the CarburizedCase of Steels 1-4 for Different Carburizing Periods

Steel No.	Prior Austenite Grain Size, µm				
	1 h	2 h	3 h	4 h	
1	70	84	94	105	
2	51	59	68	73	
3	46	52	54	59	
4	72	83	95	105	



Fig. 3 Variation of prior austenite grain size with carburizing time of Steels 1-4



Fig. 4 Optical micrographs showing the prior austenite grain size in the case of Steels 1-4 carburized at 1223 K (950 °C) for 3 h and cooled slowly in the furnace: (a) Steel 1, (b) Steel 2, (c) Steel 3, (d) Steel 4

The case depth in Steel 3 containing both Mo and Ni, which is higher than that of Steel 2 with Mo, clearly indicates that in the presence of Ni, Mo is more effective in increasing the case depth than Mo in absence of Ni. The presence of Ni is thought to enhance the effect of Mo in the increments of the diffusion coefficient.

It is also evident from Fig. 2 that as the time at carburizing temperature increases, the case depth of the steels increases.

This is due to the fact that as carburizing time increases, C atoms have more time to diffuse into a greater depth, and hence the case depth is higher.

4.3 Prior Austenite Grain Size

Steel 1 is the base steel with which the austenite grain sizes of the other steels (Steels 2-4) have been compared. This is a plain C steel with no addition of alloying elements, so it does not contain any second-phase particles to inhibit austenite grain growth. Thus the austenite grain size of this steel increases rapidly, as shown in Fig. 3 with the increase of time at the carburizing temperature.

It is also evident from Fig. 3 and 4 that Steel 2 produces much finer austenite grain size than Steel 1. Steel 2 contains Mo, which combines with C and forms Mo carbide, Mo₂C. These undissolved Mo carbide precipitates pin the prior austenite grain boundaries, inhibit grain growth, and keep the grain size small. Steel 4 produces a grain size similar to that of plain C Steel 1. This steel contains Ni. Ni does not combine with C to form any second-phase particles. It remains in solution in austenite; thus there is no second phase particle to inhibit austenite grain growth.

Steel 3 produces the finest grain size of all the steels and finer grain size than Steel 2 with Mo. Steel 3 contains both Mo and Ni. Ni remains in solution in austenite, and Mo combines with C forming Mo₂C particles. The presence of Ni in solution is thought to produce finer Mo₂C particles. Fine particles are more effective than coarse particles in restricting grain growth. The fact that the grain-refining effect found in Steel 3 is greater than that found in Steel 2 is due to the finer Mo₂C particles caused by the presence of Ni. It is also evident from Fig. 3 that the austenite grain size of Steels 2 and 4 increases with increasing carburizing time because the carbide particles coarsen with increasing time at the carburizing temperature.

5. Conclusions

- Mo and Ni, alone and in combination, decrease the thickness of the cementite network near the surface of the carburized case of the steels, but Ni is more effective than Mo in decreasing the thickness of the cementite network.
- Both Mo and Ni enhance the formation of Widmanstätten cementite plates at the grain boundary and within the grains near the surface of the carburized steel, but Ni alone is more effective than Mo in the formation of Widmanstätten cementite plates.
- In presence of Ni, Mo is much more effective in the formation of Widmanstätten cementite plates than Mo is in the absence of Ni.
- Both Mo and Ni increase the case depth. Ni is more effective than Mo in increasing the case depth. The com-

bined effect of Mo and Ni is much greater than that of either Mo or Ni alone in increasing case depth.

- Mo as Mo carbide (Mo₂C) particles (undissolved particles) refines the austenite grain size in the carburized case.
- Ni in solution does not have any effect on restricting grain growth of austenite.
- In the presence of Ni, Mo is more effective in retarding grain growth of austenite than Mo is in the absence of Ni.

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