Gradient microstructure and microhardness in a nitrided 18CrNiMo7-6 gear steel

R Yang[1, 2], G L Wu[1], X Zhang[3], W T Fu[4] and X Huang[3]

[1] College of Materials Science and Engineering, Chongqing University, Chongqing 400044, China

[2] State Key Laboratory of Mechanical Transmission, Chongqing University, Chongqing 400044, China

[3] Section for Materials Science and Advanced Characterization, Department of Wind Energy, Technical University of Denmark, Risø Campus, DK-4000 Roskilde, Denmark

[4] State Key Laboratory of Metastable Materials Science and Technology, College of Materials Science and Engineering, Yanshan University, Qinhuangdao 066004, China

Email: ranyang@dtu.dk

Abstract. A commercial gear steel (18CrNiMo7-6) containing a tempered martensite structure was nitrided using a pressurized gas nitriding process under a pressure of 5 atm at 530 ℃ for 5 hours. The mechanical properties and microstructure of the nitrided sample were characterized by Vickers hardness measurements, X-ray diffraction, and backscatter electron imaging in a scanning electron microscope. A micro-hardness gradient was identified over a distance of 500 µm with hardness values of 900 HV at the top surface and 300 HV in the core. This micro-hardness gradient corresponds to a gradient in the microstructure that changes from a nitride compound layer at the top surface (~ 20 µm thick) to a diffusion zone with a decreasing nitrogen concentration and precipitate density with distance from the surface, finally reaching the core matrix layer with a recovered martensite structure.

1. Introduction

Gear steels are required to provide an optimum combination of mechanical properties and the lowest possible cost. Some of the desired mechanical properties for gears are high wear resistance, fatigue strength, and shear strength. These properties demand a hard case on the gear surface and a tough core. A gradient in hardness decreasing from the surface to the center layer is a good option. There are mainly two types of processing to produce such hardness gradients: surface mechanical treatment, such as surface mechanical attrition treatment (SMAT) [1], surface mechanical rolling treatment (SMRT) [2], and shot peening [3], and surface thermo-chemical treatments [4,5], such as carburizing and nitriding.

Because of their low cost, easy operation and large achievable case-hardening depth, surface thermo-chemical treatments are commonly used for high quality gears to form a hard case on the gear surface. Nitriding is often used for components particularly susceptible to dimensional distortion [6,7]. Gaseous nitriding, as compared to plasma and salt-bath process variants, has the eminent advantage of low cost and

allows precise control of the microstructure via the nitriding potential [8,9]. Through the use of a nitriding treatment, different layers can be produced from the surface to interior, namely a compound layer at the surface and a diffusion zone beneath, as schematically shown in figure 1. The compound layer can be beneficial for the resistance against wear and corrosion. The diffusion zone brings about a strong increase in fatigue resistance and wear resistance [10].

In this study, a nitriding treatment is applied to a typical gear steel with an aim to investigate the microstructure and hardness gradient in the nitrided sample. To accelerate the nitriding kinetics, a newly developed nitriding process, pressurized gas nitriding (PGN) [5], is used.
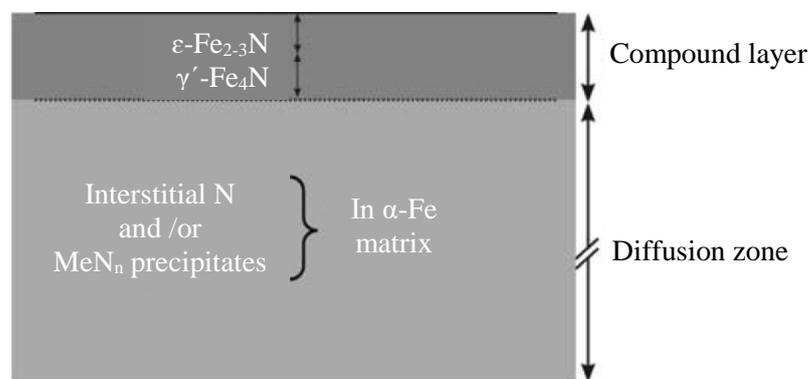

Figure 1. Schematic illustration of the surface region of a nitrided steel.

2. Experimental

A typical gear steel, 18CrNiMo7-6, was used in this investigation. Its chemical composition is listed in table 1. The starting microstructure for nitriding was tempered martensite, which was obtained by the following heat treatment: (i) solid solution treatment at 825 $^\circ$C for 1 h followed by oil cooling; (ii) tempering at 180 $^\circ$C for 2 h and then air cooling. The PGN treatment was carried out at 530 $^\circ$C for 5 hours under flowing NH_3 and H_2 with a nitriding potential of r_N=0.26 atm$^{-1/2}$ under a gas pressure of 5 atm. The dimensions of the sample for nitriding were 190 mm long, 13 mm wide and 1 mm thick.

Table 1. Chemical composition of 18CrNiMo7-6 steel (wt.%).

	C	Si	Mn	S	Cr	Ni	Mo	H	Fe
18CrNiMo 7-6	0.15-0.21	0.17-0.35	0.50-0.90	≤0.015	1.50-1.80	1.40-1.70	0.25-0.35	≤2.0ppm	Balanced

Optical microscopy (OM) and backscatter electron (BSE) imaging in a scanning electron microscope (SEM) were used to observe the microstructure. Specimens for OM and SEM-BSE observations were etched by 3% nital after polishing.

The phase composition of the compound layer, as well the diffusion zone and the core were examined by X-ray diffraction (XRD) using Cu-K$_\alpha$ radiation (λ = 1.5406 Å) operated at 40 kV and 40 mA. XRD patterns were recorded with a step size of 0.009° and a step duration of 1 s over an angular range of 30°-90°.

Microhardness profiles as a function of distance from the top surface were measured on cross-sections of the sample using a Vickers microhardness tester with a load of 100 g. The case depth was determined according to standard procedures [11] by measuring the distance from the surface to the point where the hardness is 50 HV higher than the hardness of the core substrate.

3. Results

3.1. Microstructure gradient
Figure 2 shows the tempered martensitic microstructure prior to nitriding. The hardness of this tempered martensite is 442 HV.
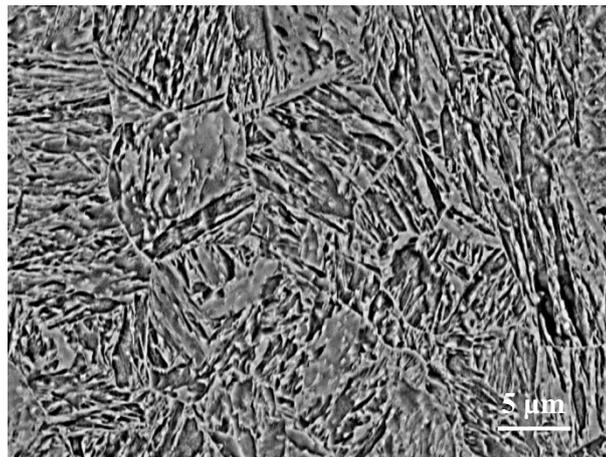

Figure 2. SEM-BSE image showing the tempered martensite prior to nitriding.
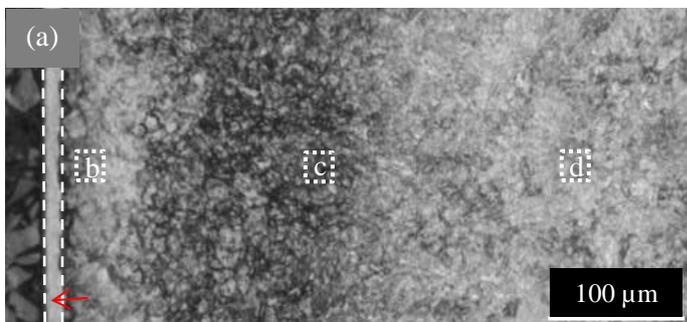

Figure 3. Microstructure of the nitrided specimen. (a) Optical micrograph showing the cross-sectional microstructure. (b) SEM-BSE image of the microstructure over the depth of 30-50 µm. (c) The microstructure over the depth of 180-200 µm. (d) The microstructure of the un-nitrided or lightly-nitrided core zone.
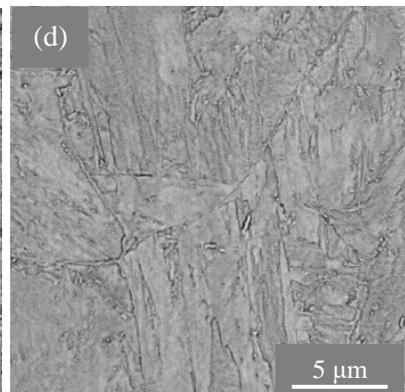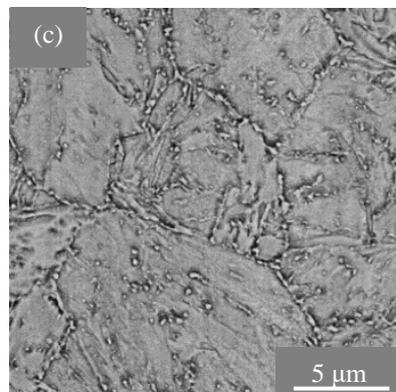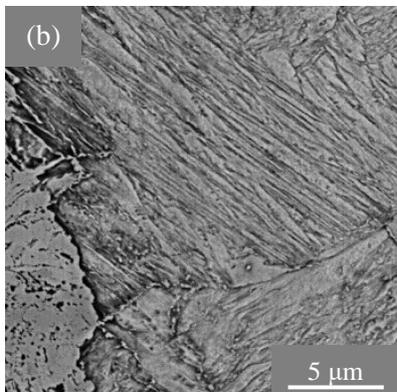
Figure 3 shows the microstructure of a nitrided sample. A thin compound layer of about 20 µm in thickness, indicated by two parallel white dashed lines in the figure, was formed on the top surface (figure 3a). The composition of this compound layer is dependent on the nitriding processing parameters such as temperature and nitriding potential. For the current nitrided sample, as indicated in figure 4, only one series of X-ray diffraction peaks are obtained from the compound layer, which corresponds to the γ'-Fe_4N iron nitride phase. Accordingly the compound layer is single phase γ'-Fe_4N.
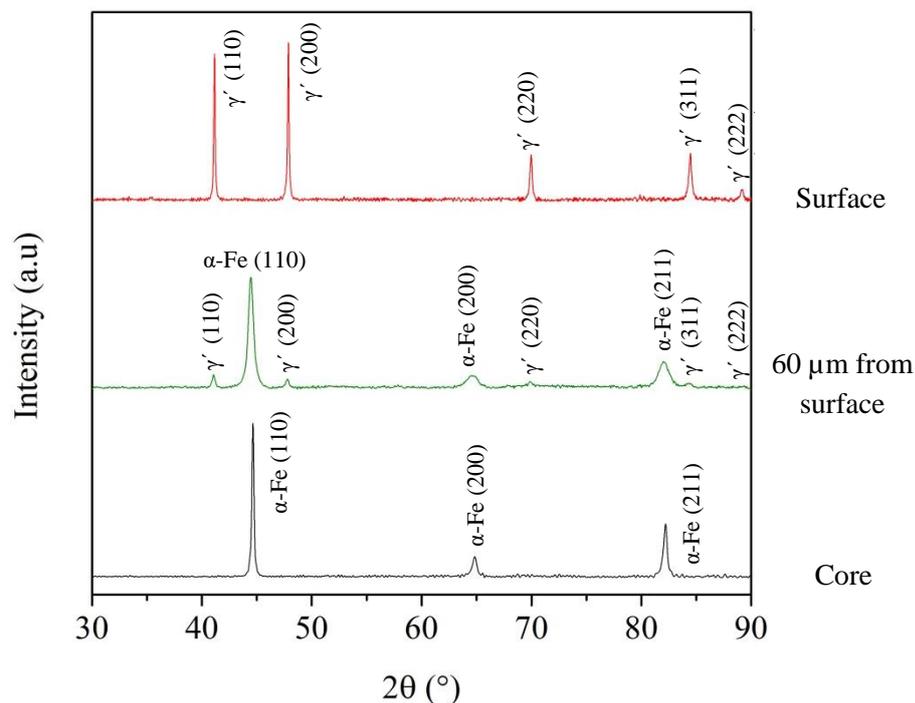

Figure 4. XRD patterns taken from the surface, 60 µm from surface and in the core of the nitrided sample. Only one series of diffraction peaks are seen from the surface data, corresponding to the γ'-Fe_4N iron nitride phase. The diffraction patterns of the core correspond to α-Fe (tempered martensite). At the depth of about 60 µm below the surface, diffraction peaks both from γ'-Fe_4N and α-Fe (tempered martensite) are identified.

Beneath the compound layer there is a diffusion zone (20-350 µm below the surface). Figure 3b gives a representative image of the microstructure over the depth of 30-150 µm below the surface, which is a recovered martensite structure containing dispersed precipitates of γ'-Fe_4N, as verified by the XRD diffraction peaks obtained at a depth of about 60 µm below the surface (see figure 4). In this diffusion zone, the diffraction peaks from the α-Fe (tempered martensite) are wider than those obtained from the core. This means a higher concentration of nitrogen in the interstitial sites of iron in the diffusion zone. At deeper locations (190-350 µm below the surface), in figure 3c, there exists a carbon-rich zone. The presence of nitrogen causes the chromium-containing carbides to dissolve allowing the chromium to react with nitrogen because of the affinity of chromium with nitrogen. The carbon is then free to diffuse to sites with lower chemical potential, for example towards to the surface and the nitriding front, leading to the decarburization of the nitrided case, and to the formation of a carbon-rich zone in the nitriding front as well as to the formation of grain boundary phases [12,13]. The precipitation particles can be seen at the boundaries in figure 3c.

In the un-nitrided or lightly nitrided core zone (350 µm below the surface), the martensitic microstructure was coarsened during the nitriding process, as shown figure 3d.

3.2. Hardness gradient
The Vickers microhardness profile from surface to center direction is shown in figure 5. The hardness shows a maximum (900 HV) near the top surface and decreases gradually toward the sample core. The surface hardness corresponds to a hardness increase of about 600 HV or an enhancement of 200% compared with the core hardness value (300 HV). Note that the core hardness is lower than that prior to nitriding (442 HV). The hardness-depth profile shows a sharp transition from the compound layer to the diffusion zone. The case depth was identified to be about 370 µm as marked by the vertical dashed line in figure 5.
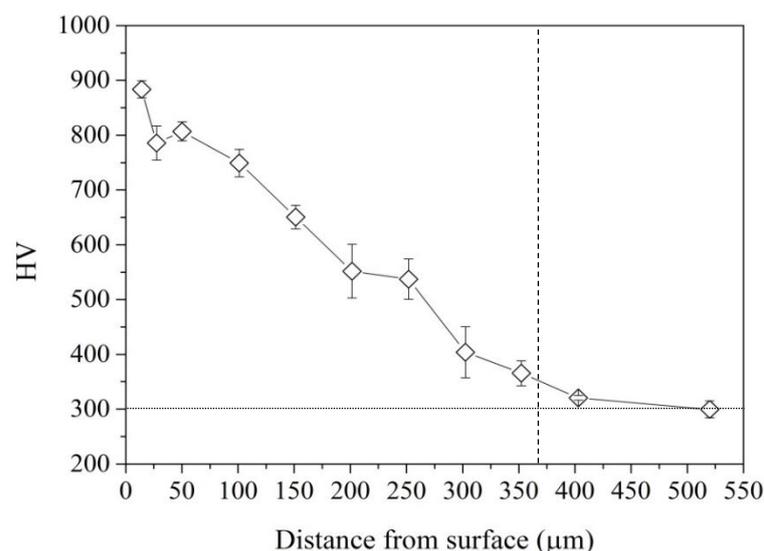

Figure 5. Variation in Vickers microhardness as a function of depth from the surface. The horizontal dotted line indicates the core hardness of 300 HV. The case depth is about 370 µm as marked by the vertical dashed line.

4. Discussion
The nitrided gear steel shows a gradient in microhardness, decreasing from the surface to the center. At the same time there is also a gradient in microstructure along the depth. The microhardness decreases corresponding to the change of microstructure.

Case-hardening of the gear steel 18CrNiMo7-6 is most commonly achieved by carburization [14,15]. In this work, nitriding by the PGN process was applied to create a hardened case. The results show that a typical nitrided layer structure is formed consisting of a compound layer adjacent the surface and diffusion zone beneath this layer.

The case depth produced in the present 18CrNiMo7-6 gear steel is similar to that obtained in a typical nitrided steel (38CrMoAlA) nitrided by PGN using the same nitriding equipment and similar nitriding conditions [5]. It can be concluded that PGN is also feasible method for the generation of case-hardened surfaces for the 18CrNiMo7-6 gear steel with a high efficiency. However for large scale gears carrying high loads, a much thicker case depth is normally desired. In the future efforts to increase the case-hardening depth for this gear steel will be made by varying the nitriding potential, the temperature and the time in the PGN process.

It should be pointed out that the hard and brittle compound layer may spall off in service and therefore is not desirable for gear applications. It is possible to perform the PGN process so that the formation of a compound layer can be eliminated [5] by controlling the nitriding potential. Furthermore, additional plastic deformation after PGN can be used to modify the microstructure of the hardened case and enhance the wear resistance [5]. An appropriate degree of additional plastic deformation is also expected to work harden the core, restoring some of the strength lost as a result of the nitriding process, and as such this possibility also requires further investigation.

5. Conclusions

A gear steel (18CrNiMo7-6) with tempered martensite structure was nitrided using a pressurized gaseous nitriding process. After nitriding, three different layers were observed as expected. The top surface layer with a hardness of about 900 HV is a compound layer consisting of the γ'-Fe$_4$N phase. Below the compound layer, there is a diffusion zone where the nitrogen concentration and precipitate density decrease with increasing depth, resulting in a gradual decrease of hardness. The core part of the nitrided sample is non-nitrided or lightly nitride, but tempered at the nitriding temperature, causing a decrease in hardness compared with the tempered martensite prior to nitriding. A case depth of about 370 µm was obtained for the conditions used in this study.

Acknowledgments

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