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Surface coatings for 3-piece freight bogie
centre bearings

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Chapter 4: Literature review: Plasma nitriding

Plasma nitriding is a case or surface hardening method that provides improved wear- and fatigue-resistant surfaces by using glow discharge technology to introduce elemental nitrogen to the surface of a metal part for subsequent diffusion into the material [54, 55, 56]. In plasma nitriding, low pressure nitrogen and hydrogen gas is ionised by applying a high negative voltage to the component to be treated [57]. The positive nitrogen ions in the plasma are attracted to the component and upon striking the component their kinetic energy is converted to heat. This raises the temperature of the component to the necessary diffusion temperature. The positive nitrogen ions colliding with the component surface act to sputter off surface contaminants and oxides that can then be removed by the pumping system. The nitrogen ions also sputter iron atoms from the component surface. The iron atoms combine with nitrogen ions to form iron nitrides. The iron nitrides then deposit onto the component surface. This layer is known as the white layer or compound layer and can consist of either γ' (Fe_4N) or ϵ (Fe_{2-3}N) iron nitrides or both. The nitrogen in the steel then creates a diffusion gradient. The diffusing nitrogen can combine with other alloying elements to form alloy nitrides or can occupy interstitial sites within the iron matrix.

The case hardening of plasma nitrided steels is due to the formation of a surface compound layer of γ' - Fe_4N and/or ϵ - Fe_{2-3}N , and below the compound layer, in the remainder of the case known as the diffusion zone, precipitation strengthening of the steel due to the formation of iron and alloy element nitrides and interstitial (nitrogen) solid solution strengthening [58].

4.1 Glow discharge process

The glow discharge reaction is defined as one taking place in an ionized vapour composed of an equal number of positive ions and electrons. The ionized gas results from an electromagnetic field, which can be formed entirely from an electric source, or a combination of electric and magnetic sources. The electric field can be of constant frequency (DC) or an alternating one even in the radio frequency range [59]. In this plasma nitriding experimental work the electric field used to generate the glow discharge was constant frequency (DC), and the primary gas is nitrogen. Figure 40 illustrates the DC glow discharge [60].

In the case of plasma nitriding the workpiece is the cathode, and the vacuum chamber wall, being grounded, is the anode. Figure 40 (a) and (b) show schematically the primary regions and voltage characteristics, respectively, of the glow discharge. The voltage drop from the cathode dark space region to the cathode glow region provides the accelerating force driving the nitrogen ions into the workpiece (Figure 40 (b)) [60].

Within a short distance of the workpiece, the positively charged nitrogen ion then acquires an electron from the cathode (workpiece) and thus emits a photon. This photon emission during the return of nitrogen ions to their atomic state results in the visible glow discharge that is characteristic of plasma techniques. Upon collision of the nitrogen atoms with the workpiece, the kinetic energy of the atoms is converted into heat, which can be used to heat the workpiece to the nitriding temperature [56]. There are several reactions taking place simultaneously in the plasma, at the component surface and within the substrate [61]. These will be discussed further in section 4.4.4.

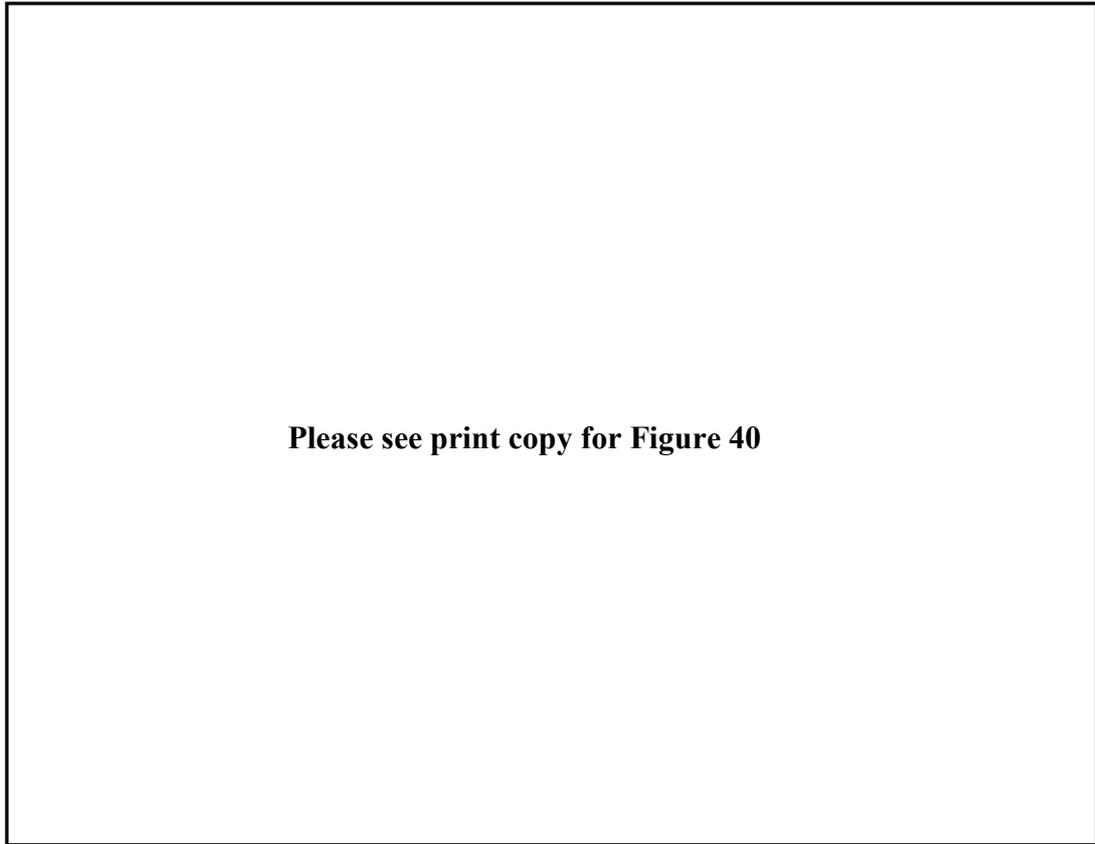


Figure 40 Schematic illustration of the (a) primary regions, (b) voltage characteristics, and (c) net space charges for a DC glow discharge. [60]

4.2 AISI 4016 molybdenum steel substrate

A typical chemical composition of the AISI 4016 molybdenum steel substrate is shown in Table 8.

Table 8 Chemical composition (in wt%) of AISI 4016 molybdenum steel.

C	Mn	Si	P	S	Cr	Ni	Mo	B	Fe
0.16	1.10	0.20	0.010	0.003	-	-	0.20	0.0010	Balance

4.3 Fe-N binary phase diagram

Given the low carbon concentration in the AISI 4016 molybdenum steel it is acceptable to use the Fe-N binary phase diagram as a guide in determining the phases present in the plasma nitrided steel rather than the Fe-C-N ternary equilibrium phase diagram. The Fe-N binary equilibrium phase diagram is presented in Figure 41 [62]. The eutectoid temperature for the Fe-N binary alloy is 592 °C. According to the Wever classification of Fe-X alloys, nitrogen, like carbon, is a type A2 austenite stabilizer [63]. Significant nitrogen diffusion into the steel matrix can occur at temperatures above 450 °C [59]. The maximum plasma nitriding temperature is maintained around 10-15 °C below the Fe-N binary eutectoid temperature of 592 °C to prevent distortion that would occur quenching from the austenite phase field. In this research, the plasma nitriding temperatures used were 450, 500, 550 and 580 °C.

4.4 Plasma nitriding process description

The main parts of a plasma nitriding system, as shown in Figure 42, are: gas supply, vacuum chamber with viewing port, rotary vacuum pump, DC power supply, vacuum and temperature gauges, and a process control unit. The workpiece to be plasma nitrided is ultrasonically cleaned in degreaser, followed by distilled water, then acetone. The subsequent plasma nitriding process can be divided into five steps: vacuum chamber evacuation, sputter cleaning, workpiece heating, nitriding reactions, and workpiece cooling. These steps will be described based upon the University of Wollongong plasma nitriding equipment used in the experimental procedure.

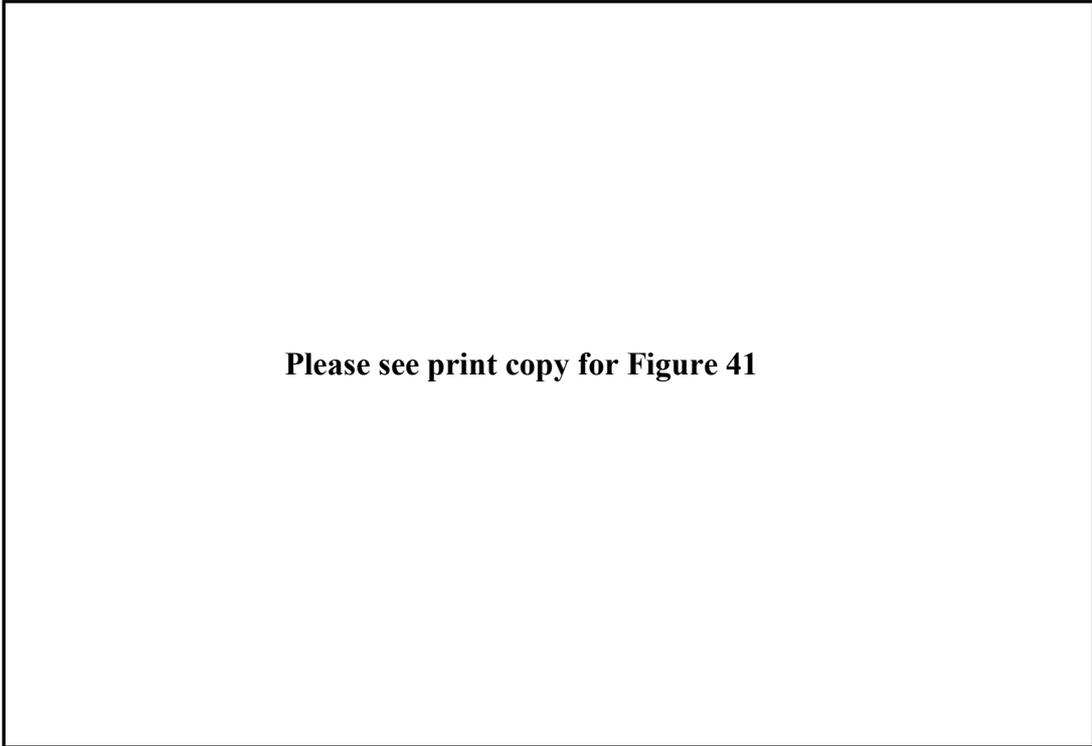


Figure 41 Fe-N binary phase diagram [62].

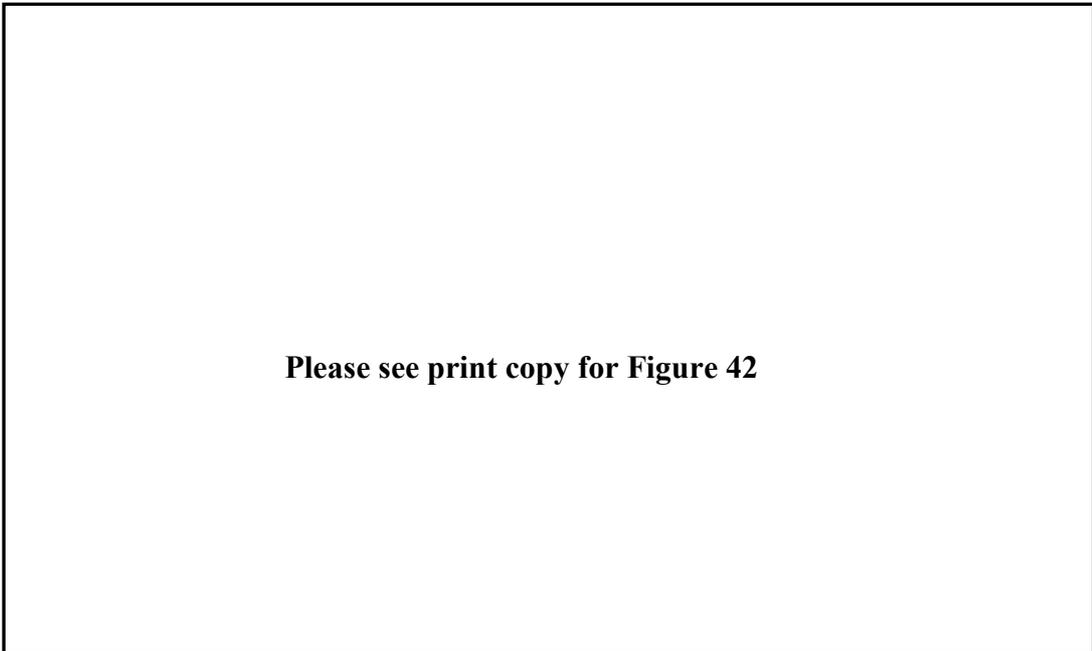


Figure 42 Typical plasma nitriding system. [56]

4.4.1 Vacuum chamber evacuation

This is performed using a rotary pump so that the pressure is reduced to a level of 0.2 torr (mm of mercury). This is necessary to remove most of the air and contaminants from the vacuum chamber.

4.4.2 Sputter cleaning

Nitrogen and hydrogen ions from the plasma or glow discharge are used to sputter clean the workpiece. The required gas pressure is 0.3 torr and the applied DC potential is 650 volts. A mixture of nitrogen and hydrogen gas is admitted continuously into the vacuum chamber. The rotary pump maintains the gas pressure at 0.3 torr. A negative DC potential of 300 volts is applied to the workpiece to form the plasma consisting of nitrogen and hydrogen gas and ions and electrons. The applied voltage is linearly increased to 650 volts over a period of 5 minutes. The voltage is held at this value for 10 minutes during which time accelerating nitrogen and hydrogen ions sputter any oxides or contaminants from the workpiece surface that are then removed by the rotary pump. At the same time the ion bombardment acts to heat the workpiece. By the end of the 10 minute sputtering step the temperature of the workpiece is around 75-100 °C.

In some other plasma nitriding systems argon ions are used to sputter clean the workpiece surface in addition to nitrogen and hydrogen ions. Argon ions being heavier than the nitrogen and hydrogen ions are more effective at sputtering [64].

4.4.3 Workpiece heating

Once the sputter cleaning step is complete, the voltage is decreased to 500 volts to stabilize the plasma. To heat the workpiece of dimensions 35 x 35 x 4.8 mm³ to the nitriding temperature of 500 °C, the gas pressure, voltage and subsequently the current

(proportional to the nitrogen ion flux) are increased gradually from 0.3 to 2.5 torr, 500 to 540 volts, and 0.5 to 1.8 amps, respectively over a period of 2 hours. Significant nitrogen diffusion into the steel matrix can occur at temperatures above 450 °C [59].

Resistance heaters or cathode shields are also used in some other plasma nitriding systems to bring the workpiece up to temperature before the glow discharge or plasma is formed [56]. In this case there is no nitrogen diffusion during workpiece heating.

4.4.4 Nitriding reactions

The molybdenum alloy steel plasma nitrided in this research can be classed as a low alloy steel. Sun and Bell have developed a numerical model of plasma nitriding of low alloy steels by considering the kinetics and thermodynamics of the reactions: nitrogen mass transfer from the plasma to the workpiece surface, diffusion of nitrogen in the matrix, chemical reactions on the surface, and chemical reactions in the diffusion zone [61].

There are many reactions that occur within the plasma including ionization, neutralization, collision, activation, charge transfer, excitation, decomposition and recombination of various species to generate a variety of nitrogen carrying ions, atoms or molecules such as N^+ , N_2^+ , NH_j^+ , N, N_2 , and NH_j . [65]. Some of these reactions are shown schematically in Figure 43. There are several forward reactions which increase the nitrogen concentration at the plasma-solid interface, and there are backward reactions including sputtering and denitriding that remove nitrogen from this surface [61]. Plasma nitriding experiments on En40B steel have shown that the forward reactions proceed much faster than the backward reactions to the extent that the contribution by the latter to the overall nitrogen mass transfer can be negligible at least

at the early stage of nitriding [61].

Due to the nitrogen mass transfer from the plasma to the workpiece surface, a nitrogen concentration gradient is built up which is the driving force for the diffusion of nitrogen in α -Fe or tempered martensite into the substrate. The nitriding case depth increases with processing time, and generally temperature however this is not true for all steels. There is no technical limit to the depth of case that is obtainable with plasma nitriding. [58]. In practice case depths of around 100-500 μm are used.

Figure 44 shows the relationship between total transferred nitrogen and plasma nitriding time for En40B steel. The relationship from the start is linear, however then becomes parabolic. The transition between the two corresponds to the initiation of γ' -Fe₄N phase on the surface [61]. During the linear period, the surface nitrogen concentration gradually increases with time up to the solubility limit, accompanied with the initiation of iron nitride on the surface. A new compound layer / diffusion zone boundary is generated. The time taken to reach this point is referred to as the incubation time.

Figure 45 schematically illustrates the development of the compound layer [61]. Based on the Fe-N binary phase diagram, the concentration of nitrogen in the γ' -Fe₄N compound layer is approximately 5.8 wt%, whilst the maximum concentration in the α -Fe is approximately 0.10 wt% [62]. The compound layer is being sputtered away at the same time that it is growing into the substrate. Typically the γ' -Fe₄N compound layer is less than 10 μm thick [61].

Please see print copy for Figure 43

Figure 43 Glow-discharge ion-nitriding mechanisms [56].

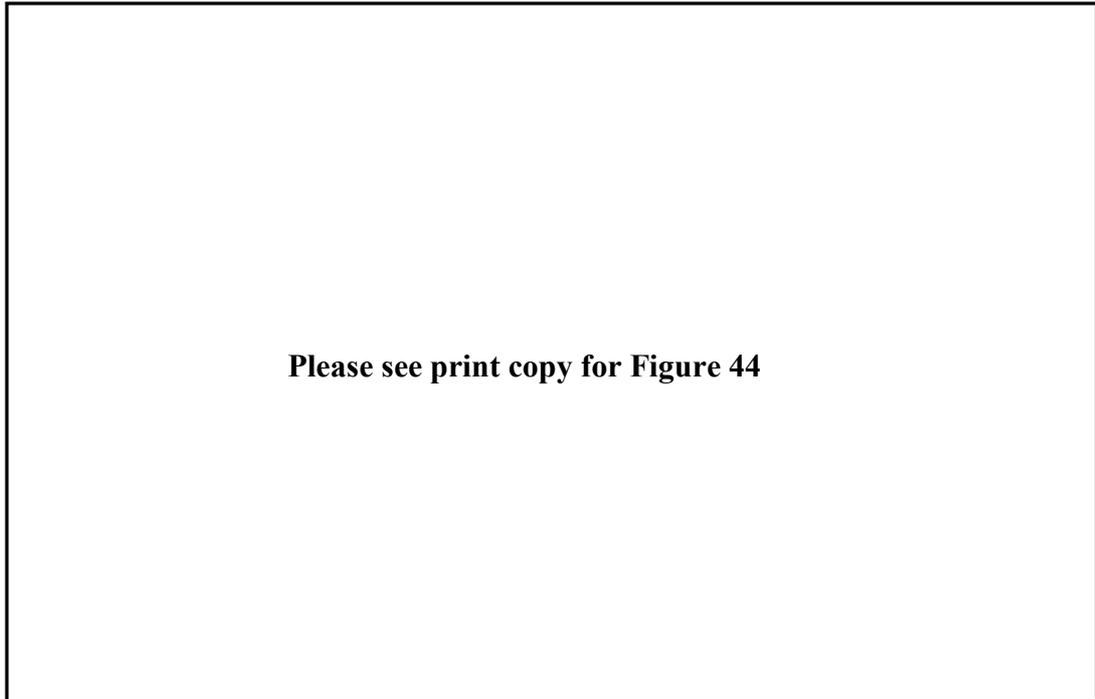


Figure 44 Total transferred nitrogen vs. nitriding time for En40B steel.

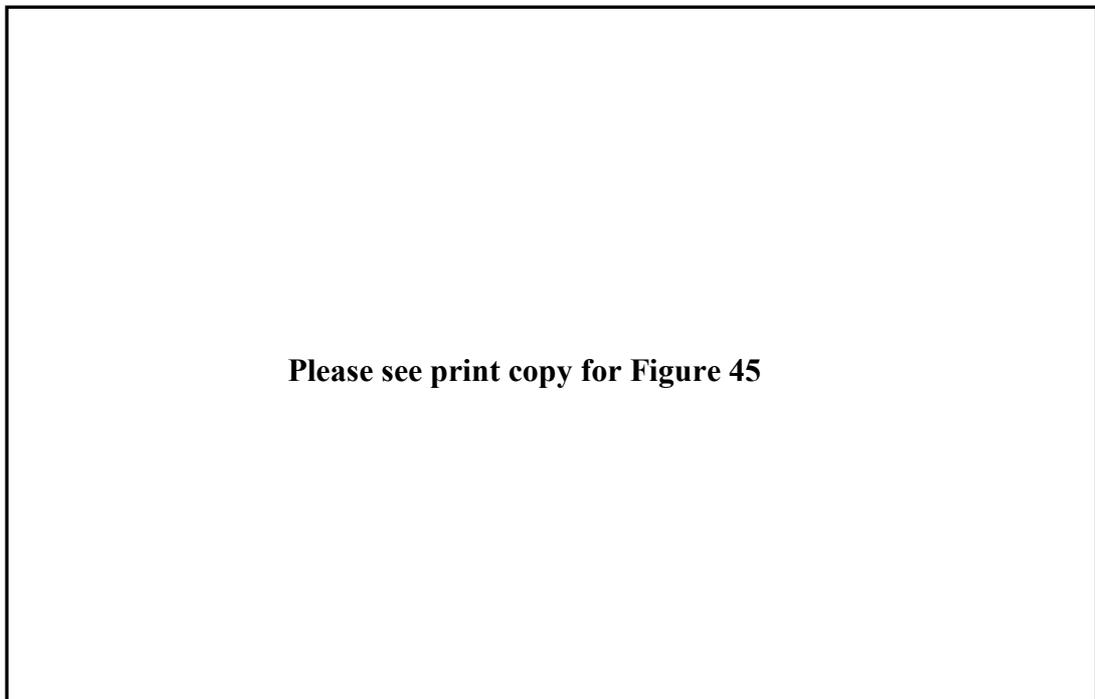


Figure 45 Schematic illustration of γ' -layer growth, taking into account cathode sputtering [61].

During diffusion of nitrogen into the α -Fe, chemical reactions also occur in the diffusion zone. Fine scale alloy nitride particles like TiN, VN, and CrN precipitate. Due to thermodynamic constraints, the stronger nitride forming elements will combine with nitrogen first [61]. The main nitride forming elements are: aluminium, chromium, molybdenum, vanadium, tungsten and titanium. [56]. The amount of these strong nitride forming alloying elements present dictates the depth of the nitrided case for a given plasma nitriding temperature and time. The higher the concentration of the strong nitride forming elements, the shallower the plasma nitrided case.

4.4.5 Workpiece cooling

The applied DC potential is switched off extinguishing the plasma. High purity nitrogen gas is admitted continuously to the vacuum chamber, whilst the flow of the mixture of nitrogen and hydrogen gas is stopped. The rotary pump maintains the nitrogen gas pressure at 1.5 torr. When the sample has cooled below 100 °C the vacuum chamber can be vented to air. Initially the cooling rate is around 12 °C/min, nearer to 100 °C it is around 0.5 °C/min. For a plasma nitriding temperature of 450 and 580 °C it takes around 3 and 4 hours, respectively, to cool the workpiece to 100 °C.

It has been reported that for another plasma nitriding system cooling also occurs by the circulation of nitrogen gas, however at a pressure of 715 torr and for a period of 2 hours or so [64].

4.5 Hardness profile in low alloy steels

AISI 4016 molybdenum steel has a low concentration of the strong nitride forming alloying elements such as aluminium, chromium, molybdenum, vanadium, tungsten and titanium. Berg et. al. reported that plasma nitriding of AISI 1020 steel for 15 hours with

80% N₂: 20% H₂ atmosphere at 500 V and 525 °C provided a surface hardness of around 500 HV, and case depth of around 650 μm [50]. By comparison, under the same processing conditions, plasma nitriding of a nitriding steel (0.33% C, 1.65% Cr, 1.0% Ni, 1.0% Al, 1.0% Mo), provided a much greater surface hardness of around 1150 HV, and a shallower case depth of around 260 μm [50].

Plasma nitriding of En19 steel (0.40% C, 0.87% Mn, 0.95% Cr, 0.20% Mo) for 5 hours with 75% N₂: 20% H₂ atmosphere at 550 °C achieved a surface microhardness of around 750 HV_{0.5} compared to original microhardness of 375 HV_{0.5}, and a case depth of around 550 μm [54].

4.6 Wear resistance of plasma nitrided steels

In adhesive wear, the wear resistance of the compound layer is very high compared with non-nitrided parts. This is because the inclination to microwelding decreases due to the change in the electron configuration on the surface [55]. A compound layer consisting of ε-Fe₂₋₃N is best for wear and fatigue applications that are relatively free from impact loading and high localized stresses. The γ'-Fe₄N compound layer is softer and less wear resistant, but its better toughness provides better service in severe loading conditions [56]. With regards to abrasive wear, the increased hardness of the compound layer is largely responsible for the improved wear resistance compared to non-nitrided parts [66].

In the diffusion layer, the steel is strengthened by precipitation hardening and interstitial solid solution strengthening. This increased strength improves the adhesive, abrasive and subsurface fatigue wear resistance compared to non-nitrided parts [55]. The formation of nitride precipitates in the diffusion zone causes a volume expansion of the affected steel. This results in compressive stresses in the case that can improve surface

fatigue resistance. Surface fatigue cracks start when a surface is subject to tensile stresses. If a surface contains residual compressive stresses, a fatigue crack will not start until the applied stress overcomes the negative (compressive) stress in the surface [58]. Increasing the case depth allows the component to withstand higher surface pressures without sacrificing wear resistance [55].