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Effect of Gas Nitriding Parameters on Microstructure and Mechanical Properties of DIN 1.2367 Hot Work Tool Steel

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Abstract – This study investigated the hardness and wear on DIN 1.2367 hot work tool steel after applying gas nitriding processes under different conditions. The impact of the gas nitriding nitrogen potential and retention time was investigated in the group of specimens. The nitriding time and potential nitrogen values in the group had changed, and the diffusion depth and hardness distribution were found to be close. In addition, wear mechanisms were investigated. As the nitriding time and nitrogen potential increase, the wear becomes more brittle. As a result, it was observed that the wear tracks were wider.

Keywords - Gas Nitriding, Friction Coefficient, DIN 1.2367 Tool Steel.

I. INTRODUCTION

Tool steels are high alloy steels used to shape other materials. Although tool steels have different rates of carbon content, they contain alloying elements such as Cr, Mo, W, Ti, V, Ni, and Co at a higher rate than other steels depending on their intended use. With applied hardening and tempering heat treatments, these steels exhibit high strength, toughness, and wear resistance. Because of these properties, tool steels are widely preferred in the manufacturing of hot and cold forming die matrices and punches, injection molds, and sheet metal cutting/punching molds. Applications in which they are used include contact conditions where various wear mechanisms occur, often due to different tribological parameters. Therefore, tool steels must have high hardness, wear resistance, and superior mechanical properties. Steels for hot work applications also need to be resistant to softening and exhibit hardness at high temperatures [1].

DIN 1.2367 (X38CrMoV5-3) is a commonly used chromium-molybdenum tool steel. The higher molybdenum content of DIN 1.2367 provides better hot hardness and temper resistance than similar tool steels such as AISI H11 and H13. DIN 1.2367 steel has gained importance in various applications such as gears, cutting knives, mandrel punches, and tool dies because of its hardness and toughness at high temperatures and good resistance to wear and thermal fatigue. The mechanical, friction, and wear properties of DIN 1.2367 tool steel have been extensively investigated in terms of the tempering process and the associated microstructure [2].

Surface engineering is a common industrial practice used to extend the life of metals and improve material performance. Nitriding is a thermochemical surface treatment that contributes significantly to fatigue strength, tribological properties, and corrosion resistance [3]. The process involves the diffusion of nitrogen species from different media types leading to the formation of nitrogen-rich phases in the near-surface region [4]. The composition and thickness of the nitrided layers are highly dependent on operating parameters such as processing temperature and time, atmospheric conditions, and substrate composition. Several studies have indicated that fatigue performance [5], tribological properties [6], and corrosion resistance [7] are closely related to these parameters.

Gas nitriding is one of the most common surface modification treatments used in the industry. It is a thermochemical process performed on steel components to enhance their surface properties, particularly hardness, wear resistance, and fatigue strength. During gas nitriding, the steel parts are placed in a furnace or a sealed chamber where they are exposed to a nitrogen-rich atmosphere at elevated temperatures typically between 500°C and 600°C. The nitrogen gas dissociates at the steel surface, and the released nitrogen atoms diffuse into the steel lattice, forming a nitride layer. The nitride layer consists mainly of iron nitrides. This layer is responsible for the improved surface hardness and wear resistance of the treated steel. The thickness and properties of the nitride layer can be controlled by adjusting process parameters such as temperature, time, and the composition of the nitrogen-rich atmosphere [8].

In this study, DIN 1.2367 hot work tool steel was applied to surface modification by gas nitriding. The effects of nitriding time and nitriding potential, which are gas nitriding parameters, on the hardness and wear resistance of steel were analysed.

II. MATERIAL AND METHODS

The material used in this research was commercially available DIN 1.2367 (X38CrMoV5-3) hot work tool steel with the chemical composition (% by weight) as specified in Table 1. Typical applications of DIN 1.2367 include small and middle-sized die-casting dies for light alloys (aluminum, zamak, brass, and magnesium), dies and molds for thermosets, and thermoplastic injection molding dies. Further applications of DIN 1.2367 hot work tool steel include light alloy extrusion tooling (e.g., liners, mandrels, pressure pads, extrusion stems, and dies), forging and hot stamping of light and heavy metals, closed dies, punches, stamps, jaws, and hot rolling rolls, as well as hot cutting applications (e.g., circular, straight, and angular cutting blades) [9].

Table 1. Nominal composition of the DIN 1.2367 hot work steel used in this study

C (%)	Si (%)	Mn (%)	P (%)	S (%)	Cr (%)	Mo (%)	V (%)
0.393	0.455	0.380	<0.03	<0.02	4.81	3.2	0.501

Austenitizing temperatures were chosen according to the steel manufacturer's recommendations and the stability of the carbides. The recommended austenitizing temperature for DIN 1.2367 hot work tool steel is 1030 °C. After the final preheating of the test specimens to 850 °C in a horizontal vacuum furnace, they were heated to a final austenitization temperature of 1030 °C at a heating rate of 12 °C/min. They were and held at this temperature for 2 h because the specimens had a wall thickness of 30 mm. Quenching was then performed at a rate of 3 °C/s using nitrogen gas at 45 bar pressure. Finally, tempering was applied at 520 °C for 2 h. As a result of these heat treatments, the hardness of the

DIN 1.2367 hot work tool steel was 50 HRC [10].

Two different nitriding processes were applied to the DIN 1.2367 hot work tool steel. Conditions of nitration are shown in Table 2. The nitriding temperature for both specimens was determined as 500 °C. The nitrogen potential working parameter of the first specimen was determined as Kn = 3 $(atm)^{-1/2}$, and the nitriding time was 20 h, whereas the nitrogen potential of the second specimen was determined as Kn = 6 $(atm)^{-1/2}$, and the nitriding time was 12 h (Table 2). The oven was first preheated and equilibrated to a preset temperature (450 °C), which took approximately 3 h and 10 min. The air in the retort was replaced with a mixture of ammonia and nitrogen gas. The control system calculated the filling time (1 h and 40 min) based on the furnace volume and the incoming gas flow. At each process step, temperature and gas flow was controlled to reach the set point Kn values dependent on ammonia.

Table 2. Gas nitriding test parameters

Test Specimens	Temperature	Time	Kn
GN1	500 °C	20 h	Kn = 3
GN2	500 °C	12 h	Kn = 6

For microstructure studies and hardness distribution, the starting material and processed and nitrided specimens were sanded with 180, 320, 400, 600, and 1000 grit sandpaper in a polishing device. After this process, they were polished with a broadcloth using 6-µm and 1-µm diamond pastes.

Microstructure studies were carried out under a Nikon LV 150 light microscope. The Metkon MH-3 Vickers hardness-testing machine with a load range of 0.01-1 kg was used for hardness measurements, under a 50-g load and test time of 10 s. Hardness measurements were made on the specimens at 50, 100, 150, 200, 250, and 300 µm. Five measurements were made for each hardness value, and their arithmetic average was taken. In this study, the nitriding depth according to the DIN 50 190/3 standard was accepted as the depth at which the hardness value of 50 HV above the core hardness was reached [11].

The wear tests were carried out in a dry friction environment without removing the wear product residues. Friction speeds were chosen as 12.4 mm/s and 5 mm/s. The load was selected as 15 N and the data acquisition time as 4 s. The trace diameter was determined as 18 mm. Before and after each wear test, the specimens were weighed on a precision scale, and the mass loss during the test was recorded. The wearing distance was 45 m [12].

III. RESULTS AND DISCUSSION

A. Microstructure and hardness

Figure 1 shows the microstructure of the DIN 1.2367 tool steel as-received (AR) and after heat treatment (HT). As seen in Figure 1a, AR consists of ferrite and pearlite phases. In Figure 1b, small precipitate particles are observed in the tempered steel with a martensite phase in the internal structure. Despite the improvement of the DIN 1.2367 steel, the martensite phase in the internal structure was related to the short curing time.

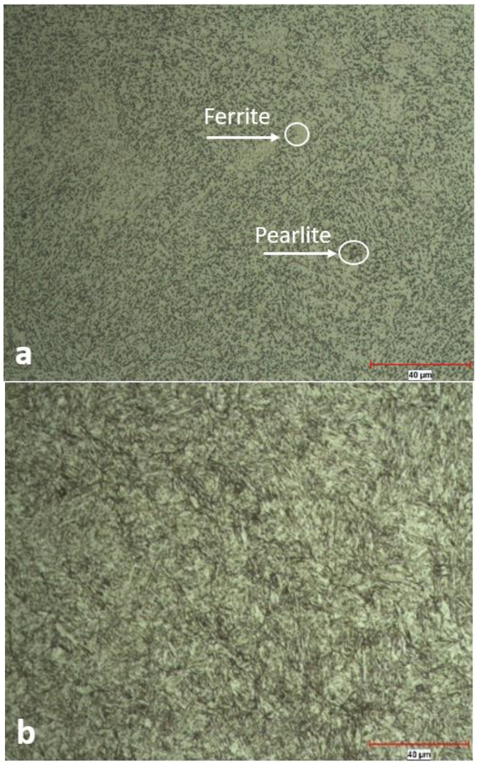

Fig. 1 Microstructure of DIN 1.2367 tool steel under the optical microscope: a) AR, b) HT

The microstructure images in Figures 2-3 show two different regions inwards from the surface. These regions consist of the diffusion layer and the matrix region. The matrix region has a martensitic structure. When the matrix region is carefully examined, precipitate particles are observed in the internal structure. The thickness of the diffusion layer of the specimen subjected to Kn = 3 and GN treatment for 20 h. at 500 °C was 200 µm (Figure 2a). The thickness of the layer was 200.7 µm at Kn = 6 and GN treatment for 12 h. (Figure 3a). For a sample with a Kn value of 12 for 20 h at 500 °C, the thickness of the diffusion layer was measured as 200 µm. This indicates that nitrogen diffusion into the steel surface results in the formation of a nitride layer extending from the surface to a depth of 200 µm. In addition, for a different treatment condition with a Kn value of 6 and a treatment time of 12 h, the thickness of the diffusion layer was measured as 200.7 µm. This resulted in a higher Kn value and a shorter treatment time, although there was a marginal difference, but still a similar diffusion layer thickness. It is understood from here that by increasing the Kn value from 3 to 6, we can achieve the same performance in a shorter time (by reducing it from 20 hours to 12 hours). The white layer thickness in both samples showed a very similar structure (Figure 2b-3b). This is in line with the study of Mridha and Khan (2008) [13].

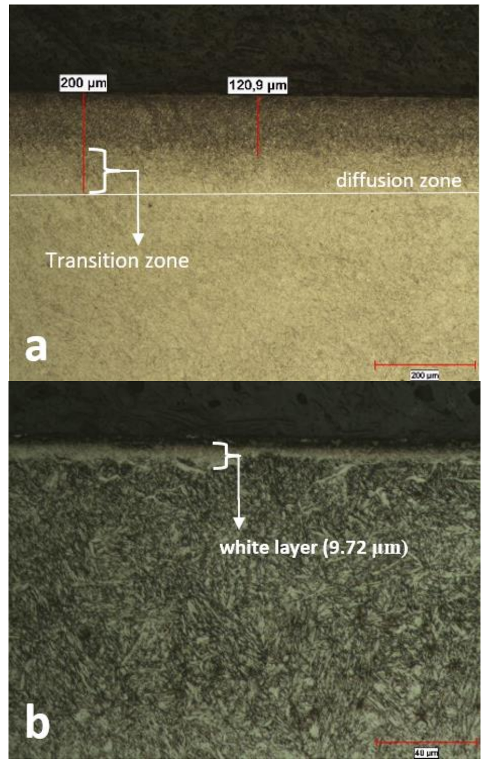

Fig. 2 Optical microscope images of the gas-nitrided specimen GN1 at 500 ºC for 12 h (Kn:4)

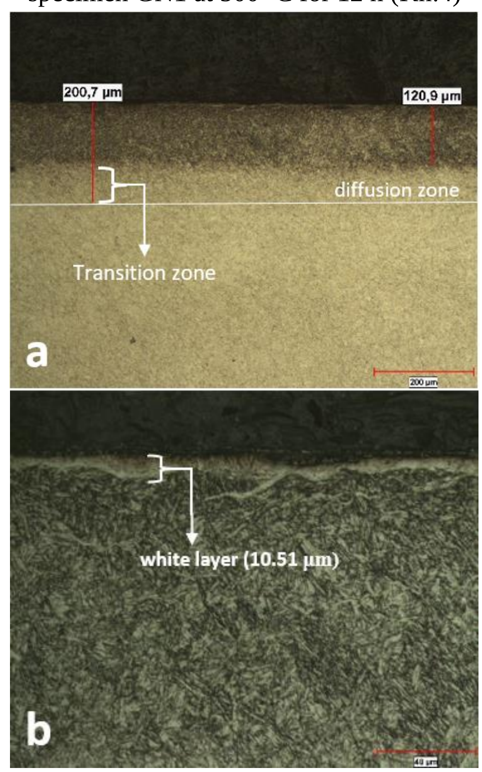

Fig. 3 Optical microscope images of the gas-nitrided specimen GN2 at 500 ºC for 12 h (Kn:18)

B. Hardness measurements

The average hardness of the starting material was measured as 196.28 HV and that of the tempered steel as 514.46 HV. Figure 7 shows that the surface hardness of the materials decreased as the nitriding temperature increased. The surface hardness value of GN1 was 1070.2 HV and that of GN2 was 1188.6. According to these results, the surface hardness increased in parallel with the nitriding potential. Mridha also reported similar results [13]. The high hardness of this resulting nitrided layer was attributed to the precipitation of the excellent dispersion of CrN and to the maximum surface hardness that increased with the increase in the chromium content, i.e., to the increase in the density of the CrN precipitation at the surface [14].

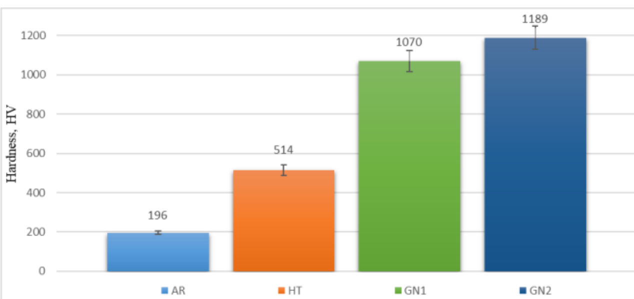

Fig. 4 Hardness results of the test specimens

When the hardness distributions of GN1 and GN2 samples are examined; hardness distributions of the numen were almost the same. As can be seen here, the same hardness value was obtained by reducing the nitriding time from 20 hours to 12 hours and increasing the Kn value from 3 to 6. In this way, the same performance can be achieved in a shorter time.

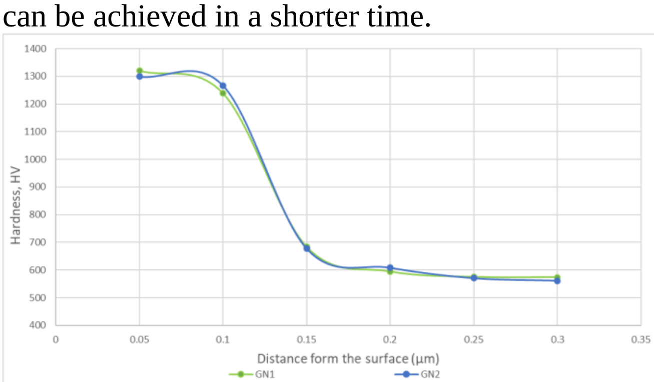

Fig. 5 Hardness distribution of plasma- and gas-nitrided DIN 1.2367 steel specimens

C. Wear resistance

The friction coefficient variation graphs from the wear tests are given in Figures 5 - 8 for a 15-N load. The friction coefficient increased rapidly from the start of the test in both the starting material and nitrided specimens, and after a short time, switched to steady-state conditions (Figures 9 - 18). During friction, hard, abrasive particles are released, disrupting the contact conditions. Thus, the coefficient of friction changes and the abrasive wear character emerges (Figures 19 - 24). This situation was also reported in the study of Karaoğlu [15]. The results of tests performed at a 15-N load and 5-mm/s and 12.4-mm/s sliding speeds show similar characteristics. When the friction coefficients were compared, they were found to be directly related to the surface hardness. The friction coefficients of specimens GN1 and GN2 which have roughly equal hardness distribution at 0.05 µm, were very similar in these two specimens. The initial friction coefficient value of these specimens was high due to surface roughness, but was then reduced to a steady value after about 10 m of sliding distance [16]. Because of the high hardness of the sublayer, the oxide layers were protected and acted as a solid lubricant that was very effective in reducing the contact area, which decreased the friction coefficient and the wear rate of these specimens. With regard to the process parameters of the GN1 and GN2 specimens subjected to the gas nitriding process, changing the Kn ratio did not significantly affect the friction coefficient [17].

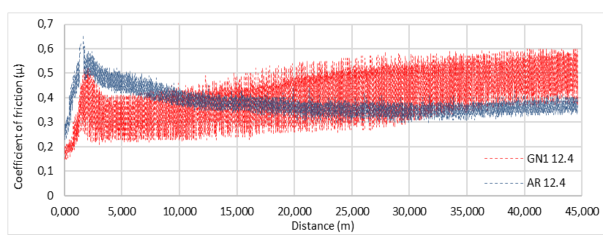

Fig. 5 Friction coefficient changes of AR and GN1 specimens at 12.4 mm/s sliding speed

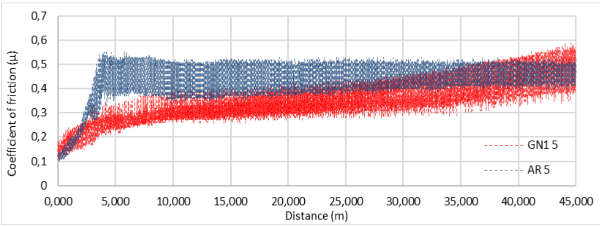

Fig. 6 Friction coefficient changes of AR and GN1 specimens at 5 mm/s sliding speed

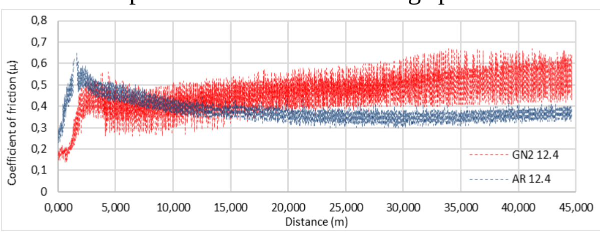

Fig. 7 Friction coefficient changes of AR and GN2 specimens at 12.4 mm/s sliding speed

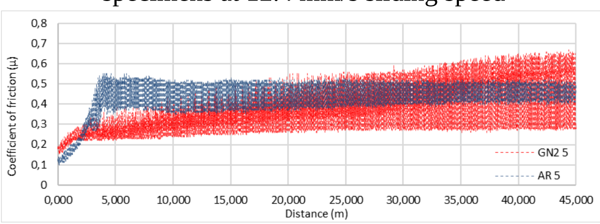

Fig. 8 Friction coefficient changes of AR and GN2 specimens at 5 mm/s sliding speed

Figures 9 -10 show that, according to the nitriding type and parameters determined in this study, the DIN 1.2367 nitrided steel exhibited much higher wear resistance than the AR. When the microscope images of the AR specimen are examined (Figure 9), abrasive wear can be seen in addition to adhesive wear. When the wear areas of the analyzed specimens are compared, the nitration process is shown to contribute to the tribological properties of the specimens. The wear trace of the GN2 specimen (Figure 10), which had the highest surface hardness, was wider than that of the other nitrided specimen. With the applied load, the hard and brittle structure formed caused fractures and abrasive fragments. Intense abrasive and adhesive wear can be seen in the microstructure images. In addition, since the wear products were not removed during the experiment, the particles in question acted to increase the wear in the test system. Figures 9 - 10 show that these particles adhered to the test specimen with abrasive effects causing the formation of cavities [18].

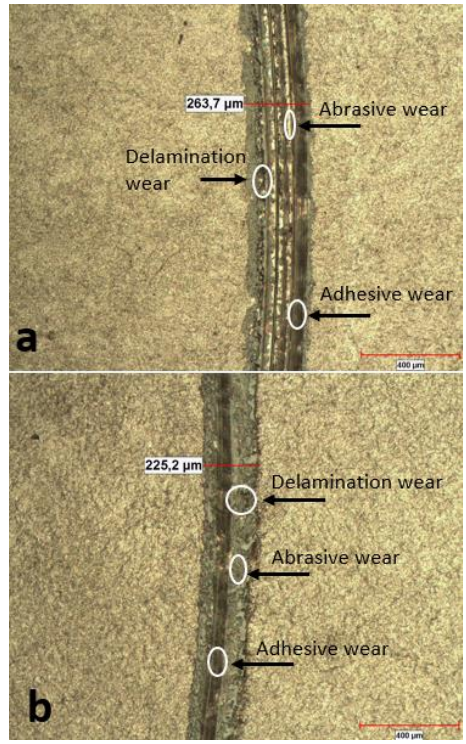

Fig. 9 Optical microscope images of the wear groove and track width for GN1 specimen: (a) 12.4 mm/s, (b) 5 mm/s

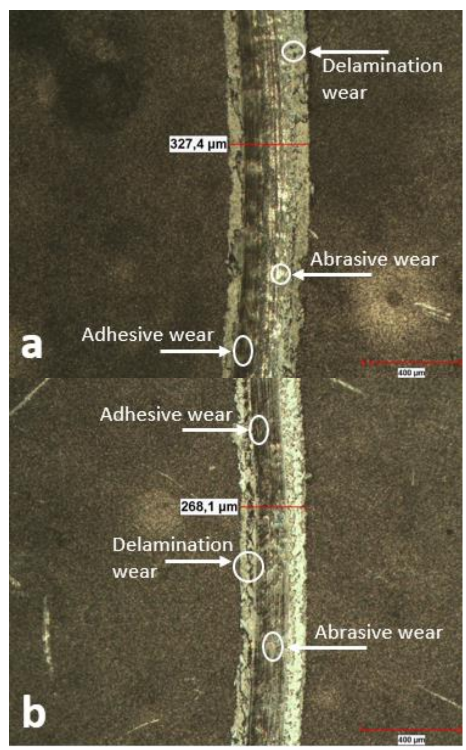

Fig. 10 Optical microscope images of the wear groove and track width for GN2 specimen: (a) 12.4 mm/s, (b) 5 mm/s

IV. CONCLUSION

This study examined the effect of the nitriding conditions on the hardness, diffusion depth, and wear properties of DIN 1.2367 hot work tool steel.

In our study, between GN1 and GN2 samples, the Kn value was increased from 3 to 6 and the nitriding time was shortened by 8 hours. As a result, the same hardness and microstructure were obtained in a shorter time.

A white layer of approximately 10 microns was formed in the GN1 and GN2 samples.

Among all the specimens, the highest surface hardness under all nitriding conditions was obtained from the GN2 specimen (gas nitriding at 500 °C, Kn 6 and 12 h).

The nitriding hardening of the DIN 1.2367 hot work tool steel significantly contributed to the wear resistance property of the material compared to the untreated steel. specimen.

In the gas nitriding process, the hardness distribution was independent of the nitriding nitrogen potential; however, the surface hardness increased with the increase of the nitriding nitrogen potential.

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