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Enhanced Wear Resistance of Mould Steel D2 via Gas Nitriding+Nitrocarburizing and Carbonitriding Processes

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Abstract

Mold steels have short service life because of being subjected to continuous impact loadings. So several industrial surface treatments are used to improve hardness and wear resistance, such as gas nitriding/nitrocarburizing/carbonitriding. This study carried gas nitriding and nitrocarburizing (GN+NC) and carbonitriding (CN) processes to D2 mold steel in industrial conditions. The results were investigated by comparing the hardness and wear properties. Nitrided and untreated samples were subjected to microstructural investigation, hardness measurements, and dry sliding wear tests. As a result of the experimental studies, it was found that different nitriding processes significantly enhanced the wear resistance of the samples. GN+NC treated D2 steel as 730 HV achieved the highest surface hardness. GN+NC treated samples had the highest wear resistance and the lowest friction coefficient.

Keywords: Wear, Gas Nitriding, Nitrocarburizing, Carbonitriding, Mould Steel D2.

1. Introduction

AISI D2 tool steel is widely used for punches and dies, forming dies, ceramic dies, cutting blades and cutters, stamping tools, etc., due to its good wear resistance and high hardenability [1-3]. The service life of the tool and die steels can be improved by various surface treatments [4-6]. The nitriding process is the most widely used technique as a surface modification to improve the lifetime of tool and die steels [3]. The researchers utilize several nitriding methods, such as gas nitriding, nitrocarburizing, carbonitriding, etc., are being used by the researchers to enhance the surface properties of AISI D2 steel [7-11].

Gas nitriding, one of the low-temperature surface hardening processes, is based on forming a hard layer on the surface by sending nitrogen atoms to the steel part surface as intermediate atoms. Ammonia (NH_3) is generally used as a nitrogen supply medium. In the nitriding process, the atomic nitrogen on the steel surface diffuses into the interior and reacts to form very fine nitride precipitates, usually 5-15 μm in size. Wear and corrosion resistance and fatigue strength are improved [12-14]. The carbonitriding process is a modified form of the carburizing process. In addition to cementation, ammonia is added to the furnace atmosphere, thus adding nitrogen and carbon to the steel surface. The difference between the process and carburizing is that it is done at lower temperatures and shorter processing times, and as a result, a thinner hardness layer is formed. The carbonitriding process is generally applied when the hardness depth is desired of 0.05-0.5 mm, and steels that are not suitable for cementation are selected. After the carburizing process, the steel is tempered to reduce the stresses in the material, and the desired final hardness value is obtained [15-18]. Nitrocarburizing is a thermochemical surface hardening process that improves engineering materials' wear, corrosion, and fatigue resistance. With the nitrocarburizing process, the simultaneous diffusion of nitrogen and carbon elements is ensured so that

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the carbon atom fills the atomic spaces and nitrogen is deposited on the surface. Thus, it forms a 10-20 μ m thick white compound layer on the material surface. This layer, which contains carbides and nitrides, provides improvements in the mechanical properties of the materials. A diffusion zone is also seen below the white layer [19-21].

In recent years, the gradual application of gas nitriding and nitrocarburization (GN+NC) has often been applied to save time and labor in industries such as the automotive, tool, and manufacturing industries, by minimizing dimensional tolerances and thus eliminating the need for post-grinding. This process, GN+NC, offers a wide range of surface hardness depending on the steel type. GN+NC provides superior improvements in wear and corrosion resistance and fatigue strength that cannot be achieved by carburizing or carbonitriding.

In this study, two different nitriding processes were applied for AISI D2 steel, and the wear performances of the samples were compared: the first process is the gradual application of gas nitriding and nitrocarburizing (GN+NC) process using ammonia and carbon dioxide gases in a vacuum nitriding furnace. The second process is applying carbonitriding (CN) process in atmosphere-controlled furnaces using endo gas and natural gas as carbon sources and ammonia as nitrogen sources.

2. Experimental Procedure

For this study, a commercially available annealed AISI D2 steel bar was selected as a base material, which had a tested hardness of 179 HV in its core region. The chemical composition of AISI D2 steel is given in Table 1.

Table 1. Properties of Used Fibers in Application

Specimen	C	Si	Mn	P	S	Cr	Mo	V	Fe
AISI D2	1,53	0,25	0,34	0,021	<0,0003	11,11	0,73	0,72	Bal.

This base material was then machined to 10 mm diameter dimensions to produce wear samples. Nitriding processes were applied in the Döksan Heat Treatment Inc. plant. A combination of gas nitriding and nitrocarburizing (GN+NC) process was conducted using the parameters shown in Fig. 1. All nitrided specimens subsequently cooled to room temperature in the furnace.

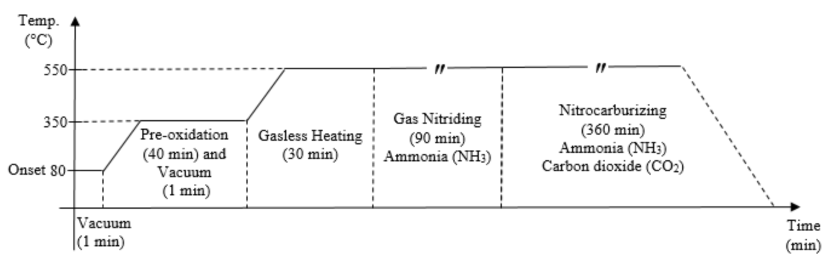

Figure 1. Gas nitriding and nitrocarburizing (GN+NC) process cycle

The parameters of the carbonitriding (CN) process applied for comparison with GN+NC samples are given in Fig. 2.

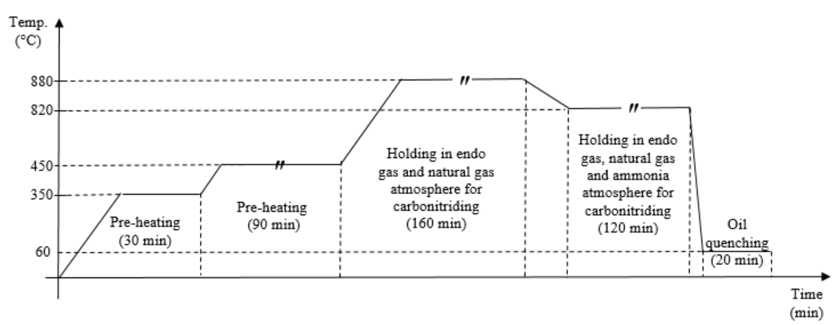

Figure 2. Carbonitriding (CN) process cycle

Wear tests and optical micrograph analyses were carried out in Pamukkale University Technology Faculty laboratories, and hardness measurements in Döksan Heat Treatment R&D Center laboratories. The hardness of the samples was measured by the Vickers method following ASTM E384 [22] standard. Hardness measurement was performed on an Emcotest - Durascan 70 G5 Vickers hardness tester with a 49.03 N (5000 gf) load and an indentation time of 15 s. The hardness values were calculated by taking the average of the measurements taken from three different regions.

The wear test of the AISI D2 steel sample prepared in Ø10 x 20 mm dimensions was performed on the pin-disc wear device given in Fig. 3. Wear test specimen dimensions are given in Fig. 4. The wear test was carried out at room temperature without using any lubricant.

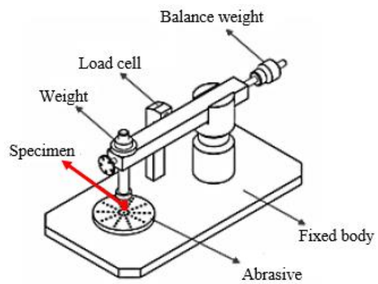

Figure 3. Schematic representation of wear test device [22, 23]

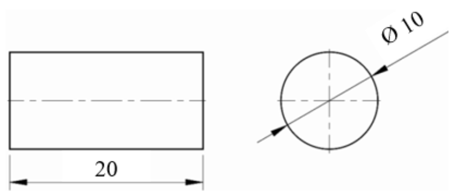

Figure 4. Wear test specimen dimensions [22]

In the abrasive wear test, 5, 10, and 15 N loads were applied to all group samples, and the sliding speed was determined as 3 m/s and the test time as 30, 60, and 90 s. The weight losses of the samples were measured on an electronic balance with an accuracy of ± 0.001 g. In addition, friction forces were measured with the load cell sensor mounted on the device to determine the friction coefficient. 240 grit sandpaper was used as abrasive.

The wear test diagrams were characterized using the following equations [23, 24].

The friction coefficient is $\mu = \frac{F}{P}$ (1)

where F is the friction force measured by the load cell and P is the normal load applied to the samples.

Worn surfaces and nitrided layers were examined by optical micrograph analyses on a Nikon Eclipse LV150NL microscope.

3. Results and Discussion

Fig. 5 shows the hardness test results of AISI D2 steel with different surface treatments. As shown in Fig. 5, an impressive increase in the hardness of the samples was obtained due to the GN+NC process. It is known that an oxide layer can be produced on the top of the nitrocarburized case during GN+NC treatment, which other nitriding methods cannot regularly produce. It should be emphasized that temperature and time control the nitrogen concentration and diffusion rate on the surface layer, which determines the phase composition and results in excellent hardness of nitrocarburized specimens. These parameters determine nitrogen concentration, the most critical factor in hardness [25]. However, the high Cr content in AISI D2 steel favors the formation of dominant chromium carbide structures that increase hardness [26]. GN+NC treated AISI D2 specimens have a thicker compound layer than the CN process. In GN+NC processed specimens, carbon diffuses more than nitrogen and penetrates more profound than the surface. Studies suggest that nitrogen pushes carbon deeper into the bulk [27]. However, nitrogen inhibits carbon diffusion in the CN process, so a shallower case is usually seen on the surface than is typical for carburized parts. As a result, in the GN+NC process, nitrogen is the predominant element in the carbonitride layer. At the same time, carbon predominates in the formation of the martensitic layer in the CN process, so the GN+NC process represents higher total layer thickness and higher surface hardness than the CN process.

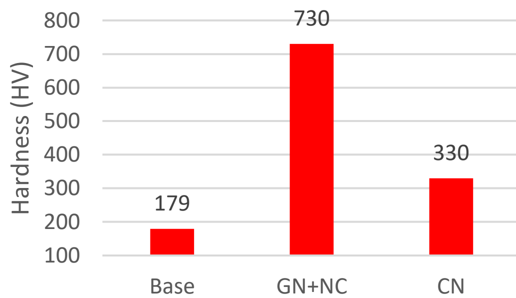

Figure 5. Hardness values of AISI D2 steel with different surface treatment

The wear tests are compatible with the hardness obtained thanks to the applied surface treatments. Fig. 6 shows the weight loss results as a function of sliding distance and applied loads in wear tests of AISI D2 specimens with different surface treatments. A significant reduction in wear volume is observed in GN+NC treated samples. The weight loss decreased sharply in the GN+NC treated samples due to the increased sliding distance. However, in general, the wear behavior of both GN+NC and CN treated specimens was significantly improved relative to the base metal. This result is attributed to the hard compound layer obtained on the surface in the GN+NC process and the formation of secondary phases such as carbonitride and intermetallic compounds in the CN process. (Fig. 7). These layers and compounds are considered to delay surface degradation and prevent carbides in the core region from being exposed to the wear contact surface [28]. The experimental results show that the weight loss of samples was highly dependent on the surface treatment, applied load, and sliding distance.

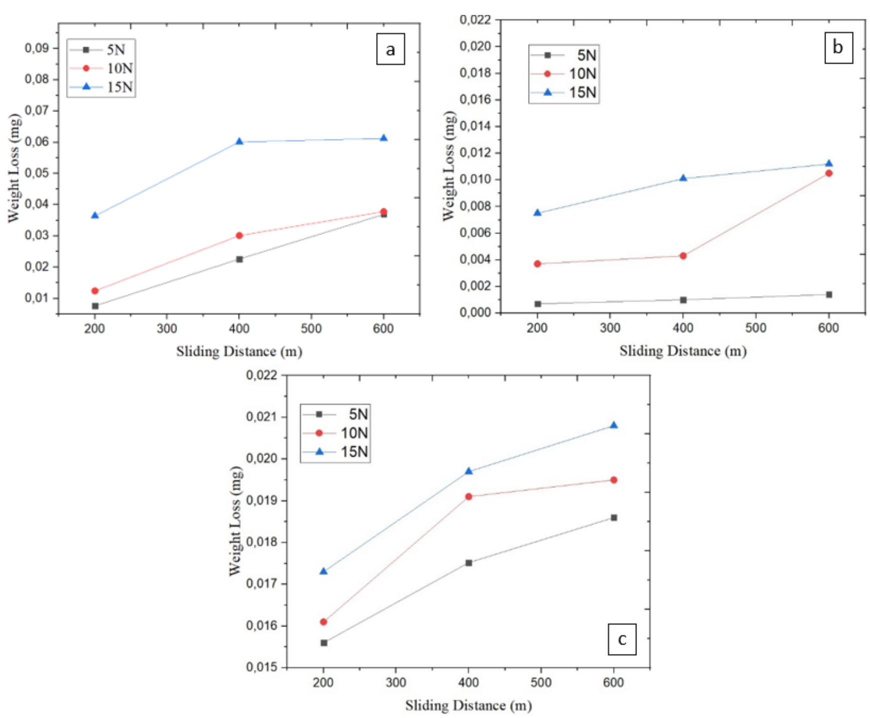

Figure 6. The effect of surface treatments on the weight loss as a function of sliding distance and load:
(a) Base; (b) GN+NC; (c) CN

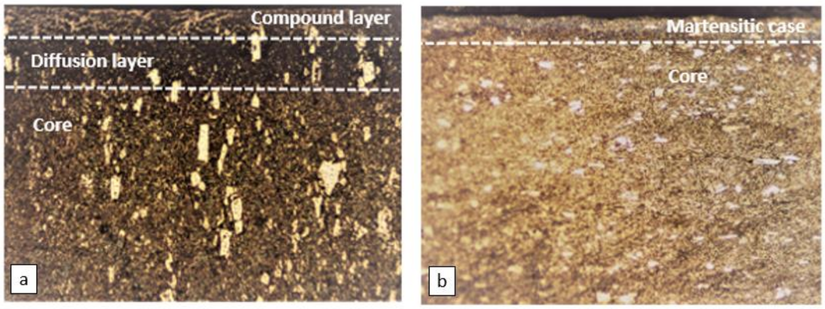

Figure 7. Cross-sectional optical micrographs of (a) GN+NC (b) CN (200x)

The friction coefficient is a widely used evaluation parameter for the wear characterization of steel. Fig. 8 shows the variation in the friction coefficient to the sliding distance under a constant load for i) base, ii) GN+NC, and iii) CN treated AISI D2 steel. The samples selected for friction coefficient evaluation were for 300 m sliding distance, and at 15 N. There is a relationship between the coefficient of friction, wear loads, and sliding distances. The friction coefficient decreases as the wear time and the associated sliding distance increase with applied loads. These parameters should be well determined in selecting materials exposed to wear and in design applications. The lowest friction coefficient was obtained in GN+NC samples. The results can be attributed to the higher hardness of GN+NC samples. It is defined that the friction coefficient shows fluctuations for the same trend in all samples. In surface-treated samples, wear coefficient values are lower than the base metal, regardless of their volumetric hardness. However, it should be noted that, due to the high chromium content in AISI D2 steel, the wear coefficient and other wear mechanisms are negatively affected by the presence of coarse chromium carbides in the structure [28]. Thus, as the sliding distance and contact load increase, these randomly distributed chromium carbides may be released locally, and the mechanical properties may deteriorate.

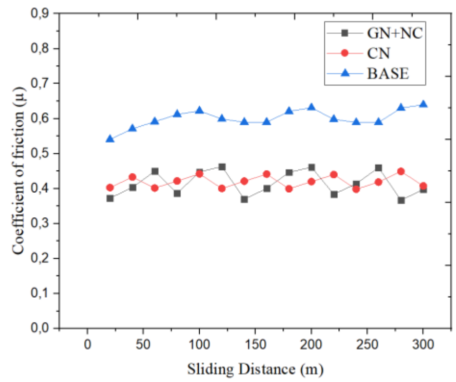

Figure 8. Variation of friction coefficient as a function of sliding distance and material's treatment.

4. Conclusions

The friction and wear behavior of the AISI D2 steel during dry sliding subjected to two different surface treatments, one with a combination of gas nitriding and nitrocarburizing (GN+NC) and one with carbonitriding (CN), were investigated. A summary of the main findings is presented below:

- The wear resistance of AISI D2, treated with GN+NC, enhanced significantly.
- GN+NC treated AISI D2 has excellent hardness compared to the base metal and CN treated samples. The surface hardness of the AISI D2 increased from around 179 HV (Base) to more than 730 HV (GN+NC treated.).
- GN+NC process represents higher total layer thickness and higher surface hardness than the CN process (330 HV).
- GN+NC process significantly increases wear resistance compared to the CN process.
- Weight loss for all samples depended on the surface treatment, applied load, and sliding distance.

- The wear resistance of AISI D2 can be optimized with surface treatment and wear conditions.
- The highest wear resistance was obtained at GN+NC samples, while the lowest wear resistance at base samples. The results can be related to surface treatment effects on the hardness of samples.
- The lowest friction coefficient was obtained in GN+NC samples.

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References

[1] https://industeel.arcelormittal.com/wp-content/uploads/2021/02/DS-TOOL-D2.pdf

[2] J.C.Díaz-Guillén, M.Naeem, H.M.Hdz-García, J.L.Acevedo-Davila, M.R.Díaz-Guillén, M.A.Khan, Javed Iqbal, A.I.Mtz-Enriquez (2020). Duplex plasma treatment of AISI D2 tool steel by combining plasma nitriding (with and without white layer) and post-oxidation. Surf. Coat. Technol; 385: pp. 1-14.

[3] K.T. Cho, K. Song, S.H. Oh, Y.-K. Lee, W.B. Lee. (2014). Enhanced surface hardening of AISI D2 steel by atomic attrition during ion nitriding. Surf. Coat. Technol; 251 pp: 115-121.

[4] M. Stoiber, J. Wagner, C. Mitterer, K. Gammer, H. Hutter, C. Lugmair, R. Kullmer. (2003). Plasma-assisted pre-treatment for PACVD TiN coatings on tool steel. Surf. Coat. Technol; 174–175: pp. 687-693.

[5] M.H. Staia, Y. Perez-Delgado, C. Sanchez, A. Castro, E. Le Bourhis, E.S. Puchi-Cabrera. (2009). Hardness properties and high-temperature wear behavior of nitrided AISI D2 tool steel, prior and after PAPVD coating. Wear, 267: pp. 1452-1461.

[6] M. Uma Devi, T.K. Chakraborty, O.N. Mohanty. (1999). Wear behaviour of plasma nitrided tool steels. Surf. Coat. Technol; 116–119: pp. 212-221.

[7] [7] Mathew Alphonse, V.K Bupesh Raja, Manoj Gupta. 2020. Optimization of plasma nitrided, liquid nitrided & PVD TiN coated H13-D2 friction drilling tool on AZ31B magnesium alloy. Materials Today, Proceedings.

[8] M.H. Staia, Y. Pérez-Delgado, C. Sanchez, A. Castro, E. Le Bourhis, E.S. Puchi-Cabrera. (2009). Hardness properties and high-temperature wear behavior of nitrided AISI D2 tool steel, prior and after PAPVD coating. Wear, 267(9–10): pp. 1452-1461.

[9] Daniel Toboła, Bogusz Kania. (2018). Phase composition and stress state in the surface layers of burnished and gas nitrided Sverker 21 and Vanadis 6 tool steels. Surf. Coat. Technol; 353: pp. 105-115.

[10] P.A. Dearnley, A. Matthews, A. Leyland. (2015). 5 - Tribological behaviour of thermochemically surface engineered steels. Editor(s): Eric J. Mittemeijer, Marcel A.J. Somers, Thermochemical Surface Engineering of Steels, Woodhead Publishing; pp. 241-266.

[11] Daniel Toboła, Witold Brostow, Kazimierz Czechowski, Piotr Rusek. (2017). Improvement of wear resistance of some cold working tool steels. Wear, Volumes 382–383: pp. 29-39.

[12] S. Ataş Bakdemir, D. Özkan, E. Uzun, S. Salman. (2020). Effect of the nitriding process in the wear behaviour of dın 1.2344 hot work steel. Journal of Naval Sciences and Engineering; 16(1): pp. 45-70.

[13] A.F.M. Arif, S. S. Akhtar, B. S. Yilbas. (2010). Effect of process variables on gas nitriding of H13 tool steel with controlled nitriding potential. International Journal of Surface Science and Engineering; 4: pp. 396–415.

[14] L.O. B. Rodrigo, O. P. Heloise, S. Vanessa, J.R. B. Israel, A.C.A. Silvia, S. de S. Fernando, S. Almir, A. F. Carlos, G. Cristiano, (2010). Microstructure and corrosion behaviour of pulsed plasma-nitrided AISI H13 tool steel. Corrosion Science; 52, pp. 3133–3139.

[15] B. Wang, B. Liu, J. Gu, M. A.J. Somer. (2022). Gaseous carbonitriding of high carbon chromium bearing steel: Correlation between composition, microstructure and stability. Surface and Coatings Technology; 438: 128408.

[16] H. Wan, H. Lu, Y. Ren, C. Ma, Y. Chen, Z. Xin, L. Cheng, K. He, X. Tu, Q. Han, Y. Ye, Y. Tao, X. Ren. (2022). Strengthening mechanisms and tensile properties of 20Cr2Mn2Mo processed by laser shock peening and vacuum carbonitriding. Surface and Coatings Technology; 439: 128462.

[17] B. Liu, B. Wang, J. Gu. (2019). Effect of ammonia addition on microstructure and wear performance of carbonitrided high carbon bearing steel AISI 52100. Surface and Coatings Technology; 361: pp. 112-118.

[18] K. Rajan, V. Joshi, A. Ghosh. (2013). Effect of carbonitriding on endurance life of ball bearing produced from SAE 52100 bearing steels. Journal of Surface Engineered Materials and Advanced Technology; 3: pp. 172-177.
[19] Y. Kaplan, A. Yıldırım, S. Aksöz. (2020). The effect of oxidation process after nitrocarburization on tribological properties of AISI 4140 steel. Journal of Polytechnic; 23(4): pp. 1357-1362.
[20] H. Z. Sun, J. Zheng, Y. Song, J. Chi, Y. D. Fu. (2019). Effect of the deformation on nitrocarburizing microstructure of the cold deformed Ti-6Al-4V alloy. Surface and Coatings Technology; 362: pp. 234-238.
[21] G. Chen, J. Wang, H. Fan, D. Wang, X. Li, H. Dong. (2019). Combat molten aluminum corrosion of AISI H13 steel by low-temperature liquid nitrocarburizing. Journal of Alloys and Compounds; 776: pp. 702-711.
[22] Annual book of ASTM standards. (2017). ASTM E384-99: Standard test method for microindentation hardness of materials. ASTM Int.
[23] E. Ersöz, İ. Ovalı. (2018). The effects of the industrial cryogenic process on the wear behaviours of AISI D2 cold work tool steels. Journal of Polytechnic; 21(2):411-417.
[24] Y. Kaplan. (2020). Role of Reinforcement Materials on Mechanical and Tribological Properties of PTFE Composites. Polymer Korea; 44(4): 436-444.
[25] Guang Chen, Jun Wang, Hongyuan Fan, Danqi Wang, Xiaoying Li, Hanshan Dong. (2019). Combat molten aluminum corrosion of AISI H13 steel by low-temperature liquid nitrocarburizing. Journal of Alloys and Compounds; Volume 776: pp. 702-711.
[26] P. Nurthen, O. Bergman, I. Hauer. (2008). Carbide design in wear resistant powder materials. in: Proceedings of the PM 2008 World Congress in Washington, DC, pp. 1–16.
[27] Insup Lee. (2019). Combination of plasma nitriding and nitrocarburizing treatments of AISI 630 martensitic precipitation hardening stainless steel. Surf. Coat. Technol; Volume 376: pp. 8-14.
[28] Karamboiki, C.-M. et al. (2013). Influence of microstructure on the sliding wear behavior of nitrocarburized tool steels. Wear 303: 560-568.