Petroleum Res. J. Vol. 21 (2008/2009); P. 15–24, 10 Figs.

The Enhancement of Wear Resistance of AISI-316 Austenitic Stainless Steel by Gas Nitriding: Response Surface Analysis and Optimization

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Abstract: Austenitic stainless steels are widely used in industrial applications, mainly due to their good corrosion resistance. However, its low hardness and poor wear performance impose strong limitations in many cases where tribological properties are of prior significance. Nitriding processes has gain acceptance as a technology for surface modification that seems to overcome these problems. In this regard, significant improvements on wear characteristics of stainless steels by gas nitriding have been reported by several workers.. However, previous research attempts on studying the effects of the gas nitriding process parameters on the material performance were made with the old classical method of changing one parameter at a time. This approach is time consuming and did not investigate the interaction effects between the various nitriding parameters. Furthermore, optimization particularly multi-variable type was very difficult to perform. These shortcomings are tackled in the present work by using the Response Surface Methodology (RSM) in the design of experiments (DOE) statistical method. Therefore, this work investigates the wear performance of AISI 316 stainless steel in a designed statistical approach. An austenitic stainless steel type AISI 316 was subjected to gas nitriding processes throughout temperature range of 400 – 600 C^o, nitriding time of 10- 50 hrs and flow rate of ammonia (NH_3) of 100 - 600 liter/hr. Experimental design is first created using composite design method in the response surface methodology. Then gas nitriding and wear tests were performed accordingly. Wear tests were made with an Amsler-Disc- Machine A135 under rolling and sliding conditions. The nitrided layers were investigated using optical microscopy (OM) technique. The phases built-up at the nitrided surfaces were investigated using X-ray diffraction (XRD) method. The microhardness of the nitrided layers was also measured and incorporated in the analysis. Results showed that maximum wear resistance improvement was 93% as compared with the un-nitrided specimen, which is achieved at 528 ^{o}C for 33 hrs using 260 liter/hr ammonia flow rate. It is also concluded that the RSM is a powerful tool to perform gas nitriding analysis and optimization.

Key words: Ammonia gas nitriding, AISI 316, wear tests. XRD methods, DOE, Response Surface Methodology.

INTRODUCTION

Austenitic stainless steels are well known by their corrosion resistance and, as consequence, are extensively used in chemical, nuclear and food industries. However, they have principal drawbacks, which are low hardness, low wear resistance, and high friction coefficient[1-3]. All these can be summarized as poor tribological characteristics. In the past, different methods have been tried to modify the surface and overcome the weakness mentioned, without affecting the corrosion resistance of the stainless steel. Among those methods, gaseous, salt bath, plasma, and ion beam nitriding methods are dominant. This has been demonstrated by the recent development of various low temperature surface modifications processes (including plasma, ion beam, gaseous and salt bath nitriding methods) and the increasing numbers of academic publications, reflecting rapidly expanding markets in the food, chemical, nuclear and medical sectors. Nitrided parts are being used in various applications such as in the food-processing, biomedical and automotive industries (see Figure 1). In food industry, nitrided stainless steel type 302 piston in a liquid-ammonia pump (in Coca Cola factory) lasted for more than five years when replaced with a piston made of an un-nitrided 300 series alloy that lasted approximately six months. Nitrided stainless steel type 410 cutting

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blade showed less wear after completion of five million cuts when used as an alternative of un-nitrided blade, which experienced a normal life of only one million cuts. Also, in another application, nitrided type 321, for a motor shaft used in the aeration of orange juice, exhibits very good life that prolonged nine times increase compared to un-nitrided part[3]. In biomedical sector, the austenitic microstructure of austenitic stainless steel is very important due its superior corrosion resistance and nonmagnetic property. This made it compatible for use in magnetic resonance imaging (*MRI*) systems used as diagnostic tools in the medical field[3]. Furthermore, nitriding can be beneficial in other categories as well, such as automotive parts (engine, transmission, chassis, and accessory components), cold-forming tools, and hot-forming tools. Some special applications include screws and cylinders for plastic extrusion, components for rotary internal combustion engines, and synchronizer components for transmissions are ion nitrided to meet close dimensional tolerances, reduction gears for marine steam turbines, deep-drawing punches and hot-forging dies[1-6].

The research and development of low temperature surface modification of austenitic stainless steels was focused on combining the improvements in wear, and fatigue performance parameters[1-2]. Therefore, this work is part of a comprehensive research program on gas nitriding of austenitic stainless steel, which aims to determine the multi-response (compromise) optimum settings for all performance parameters (Multi-response optimization)[2].

In order to design the experimental program of the present study, a review of the previous work is essential. Even though this research work is focused on the conventional gas nitriding, other process that would produce similar effects is included in this review. This will assist in the nitriding process conditions estimation and will assist in process understanding. In this regard, Ozbaysal[7] studied the structure and properties of ion-nitrided layers on AISI 321 austenitic stainless steel, AISI 410 martensitic stainless steel, and AISI 430 ferritic stainless steel, under varying process conditions. Microhardness-depth correlations, optical microscopy and transmission electron microscopy were used in the analysis. The process variables studied include time (2 - 10 hrs) and temperature (400 - 600 ^{o}C). He found that the highest case depth values and hardness levels were observed in martensitic stainless steels, the lowest case depths were observed in austenitic stainless steel. All three steels showed increasing case depths and decreasing surface hardness with increasing ion-nitriding temperatures and times. Nitriding depth was found to be parabolic with ion nitriding time in all three steels at all ion-nitriding temperatures investigated. Electron microscopy showed that almost no structural difference arises in the core of ferritic and austenitic stainless steels whereas recrystallization of the martensitic structure was observed in the core of martensitic steel following ion nitriding. Kuawahara[8] made comparative study between plasma nitriding and ammonia gas nitriding of austenitic stainless steel type

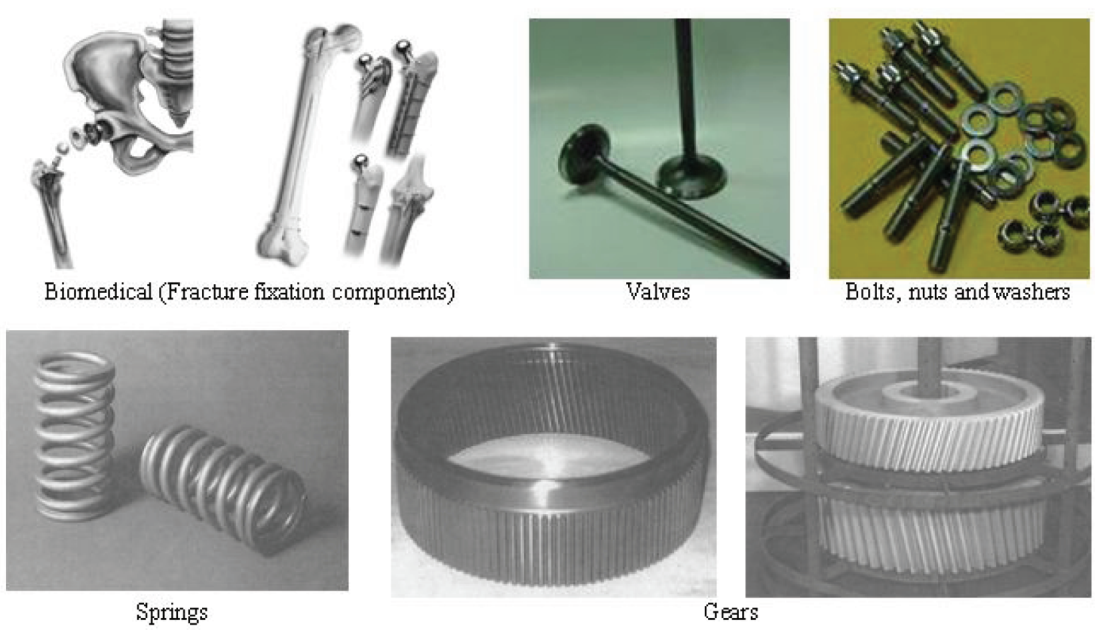


Fig. 1. Nitrided stainless steels parts.

Fe-18Cr-9Ni. Both plasma and gas nitriding were compared with respect to the growth rate of nitrided layer. He concluded that the growth rate of nitrided layer in the plasma nitriding of stainless steel type Fe-18Cr-9Ni is 1 to 2.5 times that in case of gas nitriding. However, his comparison was based on layer growth rate, which cannot be considered as strong indicator for the tribological characteristics of the nitrided stainless steel.

Wei[9] presented the results of a comparative study of beam ion implantation (BII), plasma ion nitriding (PII) and gas nitriding of AISI 304 stainless steel. He concluded that, under controlled conditions (the same treatment time of 30 to 60 min and the same treatment temperature 400°C), the microstructure produced by all techniques are similar. However, the concentrations and nitrogen detectable depths of the nitrogen-enriched layer are significantly different, depending on the process. Both BII and PII produce a thick nitrogen-enriched layer (with higher concentration) compared with gas nitriding. Again, the effect of the nitrogen-enriched layer thickness on the tribological characteristics of the nitrided stainless steel was not investigated. Finding a compromise between increased hardness, increased wear resistance and retained corrosion resistance using nitrogen plasma immersion ion implantation (PIII) on austenitic stainless steel AISI 316 at a treatment temperature of 380°C was investigated[10]. The depth and the distribution of the implanted nitrogen and the phase composition were analyzed with glow discharge optical spectroscopy (GDOS) and X-ray diffraction (XRD). Compared to untreated material, he found that the hardness can be increased up to factor of four and the wear behaviour was improved by 1-2 orders of magnitude. Only a very small fraction of chromium nitride, which is not detrimental to the corrosion resistance, was observed with XRD.

Bell[5] overviewed the development of low temperature thermo-chemical surface modification processes. He reported that the surface hardness and wear resistance of the austenitic stainless steels could be much improved without lowering its corrosion resistance by low temperature nitriding. Menthe[11] investigated the influence of the plasma nitriding process on the mechanical properties of austenitic stainless steel. He conducted plasma nitriding process on AISI 304 steel in a temperature range of 375-475°C using pulsed-DC plasma with different N_2-H_2 gas mixtures and treatment times. He found that the wear resistance significantly increased even at higher loads compared to the untreated material and in the same time, the microhardness of nitrided layers had increased by factor of five as compared untreated material. Menthe‘s work was focused on the wear and microhardness characteristics and does not mention any thing about corrosion resistance issues. However, the main point which can be obtained from his work, is that wear resistance can be improved by plasma nitriding at low temperature (as low as 375°C). Baranowska[12] studied the morphology of the nitrided layers produced on austenitic steel and their mechanical and tribological behaviour. Using MFM, TEM and XRD techniques, he found that the coatings produced have complex multilayer structures. The specific wear rate was measured using a pin-on-disk test method consisting of an alumina sphere (99.6% Al_2O_3, hardness 1800HV, roughness 0.025-ìm Ra) of diameter 6mm±0.25-ìm sliding against the flat nitrided austenitic steel samples. He found that the low temperature gas nitriding combined with appropriate pre-treatment makes it possible to obtain surface layers with very complex morphology similar to those produced by plasma treatments. Furthermore, he found that by changing the conditions of the gas nitriding parameters, layers with various phase compositions could be obtained. Finally, he concluded that the mechanical properties of the layers are mainly related to nitrogen content and for all kinds of obtained phase compositions the mechanical properties were at least the same as for typical gas-nitrided layers obtained at higher temperature and containing chromium and iron nitrides. Therefore, Baranowska‘s work confirms the low temperature nitriding benefits and that gas nitriding can produce layers with comparable properties with other more advanced techniques. Ionara[13] presented results of wear tests performed on AISI 316L stainless steel samples nitrided with DC-pulsed plasma equipment for 20 hrs at 400°C in plasma formed with 25% N_2+75% H_2. The wear tests were made with an Amsler-Disc-Machine A135 under rolling and sliding conditions. The surface and subsurface were studied by optical microscope (OM) and microhardness. Wear debris generated under dry rolling-sliding condition were observed by scanning electron microscope (SEM). The results showed that plasma nitriding significantly improves the wear resistance of the surface of the austenitic stainless steel. However, Ionara work could be more beneficial if parametric studies and process optimization were performed.

Bielawski[14] conducted nitriding process on chromium steel at a temperature range of 400 – 500 °C in ammonia gas atmosphere. The microstructure of the resulted layers was investigated using scanning electron microscopy (SEM) and light microscopy analysis (LMA) techniques. Its phase build-up was checked by XRD methods, and the thickness and microhardness of the layers were measured. It was found, that it is possible to obtain layers with good mechanical properties (microhardness) and good corrosion resistance (microstructure). Moreover, it was possible, by gas nitriding treatment, to obtain uniform layers at low temperature processing. He found that by nitriding at temperature below 500°C, the obtained layers remained white after etching, which suggested their good corrosion resistance (as they resisted the acidic effect of the etching agent). All the obtained layers showed very good mechanical properties (high hardness) corresponding to a very high nitrogen content in the layers.

In summary, the previous work on gas nitriding process of stainless steel showed that the wear properties of the austenitic stainless steels could be significantly improved after nitriding depending upon the treatment conditions. In addition, there is a common conclusion that low-temperature nitriding is preferable to high-temperature nitriding from corrosion resistance point of view. However, there is no an overall agreement on the effect of other process conditions such as ammonia flow rate or time of nitriding. Furthermore, previous research work was performed using the classical methods of changing one factor at a time while holding the other factors. This methodology requires a lot of specimens and extensive experimental work, which is both costly and time consuming. In addition, the classical method is not capable of investigating the interaction effects between the factors and cannot be used to perform experiment optimization. All these drawbacks are tackled by using the response surface methodology (RSM) in the design of experiment statistical methods. Therefore, in this work an austenitic stainless steel material type AISI 316 is subjected to a conventional gas nitriding process throughout a temperature range of 400–600 °C, a nitriding time of 10-50 hrs and ammonia (NH_3) flow rate of 100-600 liter/hr; and the wear behaviour of the nitrided specimens were then examined. The ranges of processing parameters (Temperature, time, and flow rate) were estimated based on the previous literature review and few preliminary trials. Conventional gas nitriding is adopted in this research work, because it is cheap and does not require very highly skilled technicians. In addition, it can be used for mass production of industrial and biomedical parts with all sizes and shapes.

MATERIALS AND METHODS

The material used for this investigation was austenitic stainless steel type AISI 316 with a chemical composition shown in Table 1.

The as received material microstructure was fully austenitic and its hardness was 200 HV. The as received material was then stress relieved for 3 hrs at 1100 °C in nitrogen atmosphere then oil quenched to avoid oxidation. The wear test specimens were fabricated in accordance with Amsler-Disc-Machine A135 specimens' requirements. Specimens were prepared with the shape of discs, 38mm diameter and 10mm thickness machined from annealed stainless steel bar. All specimens were subjected to a pickling pre-treatment using a hot hydrochloric acid (70 °C and 50%) for oxide film breaking as required by nitriding process. Specimen shape and dimensions are shown schematically in Figure 2 and the physical specimens after nitriding in Figure 3.

Table 1. Chemical composition of AISI 316 austenitic stainless steel used in the experiments.

Alloying element	C	Mn	P	S	Si	Cr	Ni	Mo
Wt.%	0.08	1.53	0.023	0.024	0.69	16.4	10.4	2.00

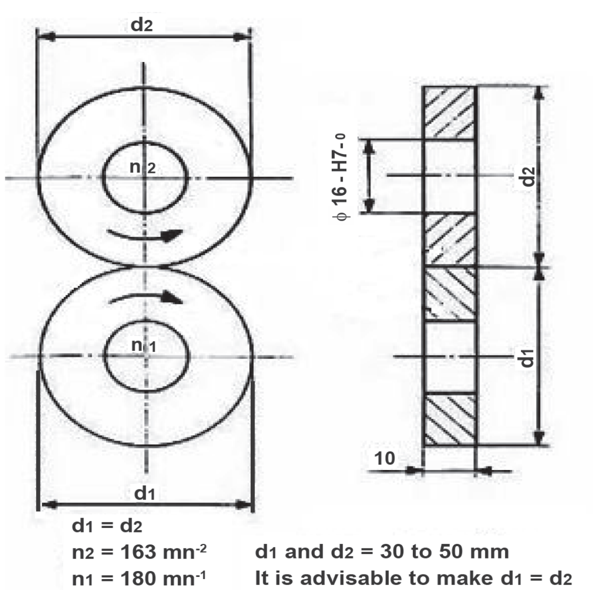


Fig. 2. Rolling and sliding wear test specimen's shape and dimensions.

The response surface methodology (RSM) was then employed to determine the required points of experiments (design points) within the previously mentioned ranges of nitriding temperature, nitriding time and ammonia flow rate. The resulted matrix is shown in Table 2. Anhydrous ammonia gas was used to accomplish the gas nitriding processes. The nitriding processes were conducted using a SIB Furnace (pit furnace type 752) shown in Figure 4.

Table 2. RSM matrix of gas nitriding process with results of wear resistance and microhardness. experimental program.

Exp. No	Time (hrs)	Temperature (^oC)	Flow rate (litre /hr)	Wear resistance (relative weight loss) %	Microhardness (HV $_{200\ gr.}$)
1	30	500	350	7	1298.0
2	18	441	201	49	339.9
3	10	500	350	47	458.4
4	42	441	499	52	633.3
5	18	559	201	22	1080.0
6	30	500	100	9	973.0
7	42	559	499	18	1010.0
8	50	500	350	25	1089.0
9	30	500	600	26	915.5
10	42	559	201	12	1061.0
11	30	600	350	23	1081.4
12	30	400	350	71	312.9
13	42	441	201	37	647.0
14	18	559	499	29	1001.0
15	18	441	499	62	388.1
16	30	500	350	9	1118.7
17	30	500	350	8	1166.8

Fig. 3. Wear test specimen after nitriding.

The wear tests were performed on an Amsler Disc-Machine model A 135 shown in Figure 5a. The arrangements of samples are as shown in Figure 5b, where the top sample rotated at 200 rpm and. The bottom sample rotated at 220 rpm to obtain 10% sliding. The wear tests were performed under 500 N load and the wear tests duration was fixed to 2 hours for all specimens. After wear test, the specimens were cleaned with alcohol and acetone using an ultrasonic bath; the specimens were weighted with an ADAM type AAA 260 LE balance (0.0001g sensitivity). After wear test completion, the preparation of specimens for metallography was made by cutting them parallel to the rotation plane (longitudinal) and in a transverse direction. The specimens were then polished up to grade 0.3ì and etched (at room temperature) in an aqueous solution of $FeCl_3$ and NITAL 2% solution (2 ml nitric acid + 98 ml ethanol) to reveal the thickness of nitride layer and the microstructure.

The specimens were examined by optical microscopy (OM). In addition, X-ray diffraction analysis of nitrided surface was performed using a Philips X-ray diffractometer (type PW 1800) with a Cu-radiation. A two theta (2è) range of 2^o to 80^o was selected with step 0.02^o. The hardening effect of the nitrided layer was evaluated by surface hardness measurements using the conventional Vickers micro-hardness technique. Microhardness testing was carried out under an indentation load of 200g. The microhardness measurements were made normal to the surface and on transverse sections of the samples using an equipment Leica VMHT (type 302801) apparatus.

RESULTS AND DISCUSSION

For each wear test, two specimens of each nitriding process were used, and their corresponding weight loss relative to un-nitriding material for wear resistance is determined. Furthermore, the nitrided layers microhardness was measured. The obtained results of the wear resistance of nitrided specimens are entered into the previously created design matrix and then analyzed using the RSM**.** The wear resistance (weight loss relative to un-nitriding material, the wear %) and the microhardness ($HV_{200gr.}$) of nitrided specimens are presented in Table 2.

As shown in Table 2, generally, for all nitriding processes performed, the wear resistance significantly improved as compared with un-nitriding

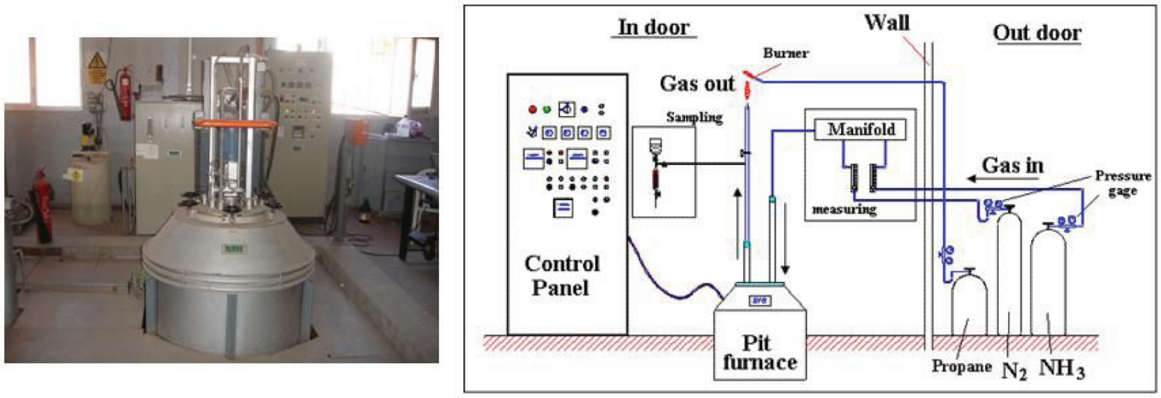
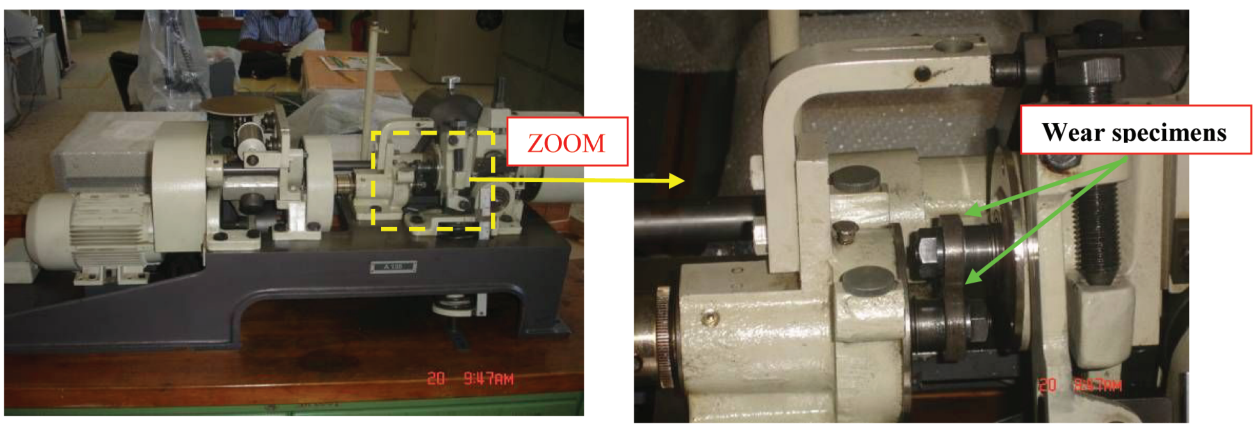


Fig. 4. The SIB Furnace (pit furnace type 752).



(a) (b)

Fig. 5. Photographs showing an Amsler machine model A135 Wear Testing Machine, (a) overall shape of the wear apparatus, (b) zoomed to illustrate the specimens postions.

material. High and low wear resistances were obtained around nitriding process No.1and No.2, respectively. In addition, Table 2 shows the highest and the lowest values obtained from No. 1 and No. 2 nitriding process. Detailed analysis of these results is provided in a separate section hereafter. The point of maximum wear resistance and microhardness (point No.1) will be considered from the microstructure analysis point of view. Further layer thickness and microstructural analysis is provided in another publication. Therefore, Figure 6 shows an optical microscopy (OM) micrograph of a cross section of nitrided specimen (nitriding process No.1 of Table 2). The layer appears uniform and continuous with a total layer thickness of 72 ìm and the diffusion zone can easily be distinguished from the substrate. Figure 7 shows a typical microhardness profile of a sample cross section (nitrided sample experiment No.1 in the design matrix of Table 2). The sharp transition from the high hardness in the surface to the low hardness of the substrate can be observed which is associated with the presence of the nitrided layer.

Figure 8 shows X-ray diffraction patterns for the same nitriding process No 1. The detected chromium nitride (Cr N) is the most responsible for the increased hardness and wear resistance. Ion nitride ã'-Fe_4N is shown, However, the most interesting phase appearing in the nitriding layer is the expanded austenite phase ($ã_N$), which is named after the lattice expansion of the original austenite lattice, as indicated by a shift of X-ray diffraction peaks towards smaller diffraction angles. This phase is reported to retain the corrosion resistance of the nitrided material[2]. Therefore, its presence in the nitrided layer of the maximum wear resistance specimen as depicted by the XRD analysis is a significant indication on the good efficiency of the nitriding process.

Parametric Study

The results of Table 2 were analyzed using response surface methodology (RSM), and the interactions of nitriding processing parameters (nitriding temperature, nitriding time and ammonia flow rate) were identified. The effects of nitriding

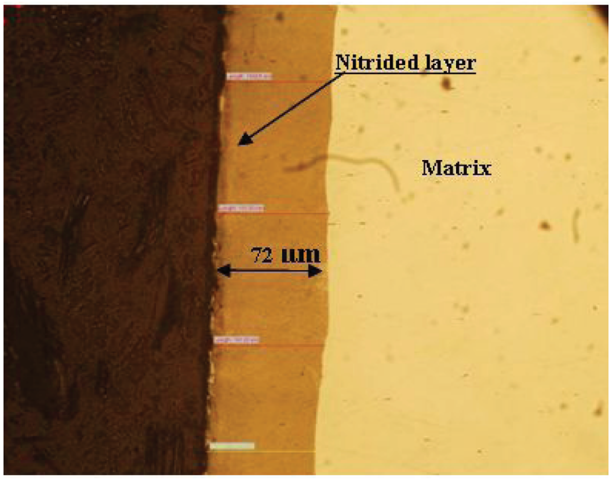


Fig. 6. Optical micrographs (OM) of a nitrided layer of AISI 316 (process No.1).

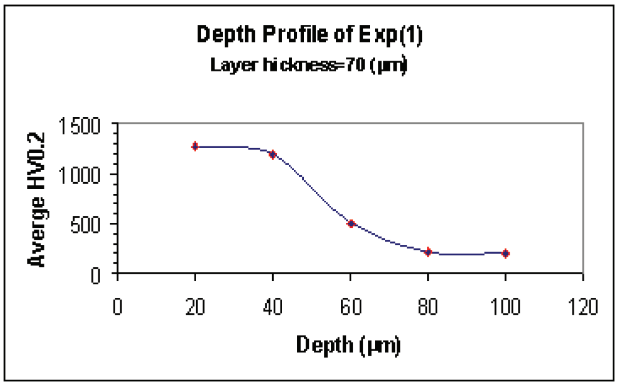


Fig. 7. Hardness profile across the nitrided layer (nitriding process No.1).

processing parameters on wear resistance are shown in three-dimensional graphs and contours in Figure 9a, b, c, d, e, and f. It is clear that an optimum region for the wear resistance do exist (Figs. 9a and b). This is evident from both the decreasing contour circles and the downward curvature of the 3D surface plot. More specifically, referring to Figure 9a, the time and temperature, which provide optimum region of wear resistance (less than 13.991%), is about 32 hrs and 535 ^{o}C respectively. This is also shown in the 3D surface plot of Figure 9b. However, these plots need to be correlated with the other graphs for conclusions. Figure 9c shows the effect of the time and the flow rate on the wear resistance. As shown the time and the flow rate, which provide optimum (minimum wear) region of wear resistance (less than 15%) is about 34 hrs and 260 l/hr respectively. This is also agrees with the 3D surface plot of Figure 9d. However, these plots need to be correlated with the other graphs for conclusion as well. Figure 9e, shows the effect of the temperature and the flow rate on the wear resistance. As shown the temperature and the flow rate, which provide optimum region of wear resistance (less than 18%) is about 530 ^{o}C and 300 l/hr respectively. This is also agrees with the 3D surface plot of Figure 9f. These plots have provided a comprehensive view on the parameters effects and an optimization plot will further confirm these findings,

Response Optimaztion (Minmum Wear)

A third and comprehensive method of presenting the nitriding process parameters effects is by developing the optimization chart of the wear resistance with the nitriding conditions, which is shown in Figure 10. This Figure shows the optimization chart for the performed wear tests on the gas nitrided specimens. The optimization result is shown in the lift column; while the optimum setting of each parameter is shown at meddle of the top row. The behaviour curve of each factor is shown hereunder. As shown, the optimum nitriding time is 33.4 hrs, the optimum temperature setting is 528.2^{o}C and the optimum gas flow rate setting is 260 liter/hr, which resulted in minmum wear of 1.98%. This achievement means that by setting the nitriding conditions to the previous values, a material with an optimum wear resistance will be produced (1.98% wear as compared with the 100% wear of the un-nitrided samples). Furthermore, up to the authors' knowledge, the optimum 33 hrs nitriding times and the 260 l/hr ammonia gas flow rate have not been mentioned in the previous work, which is considered as a further contribution

CONCLUSIONS

From this study, we can conclude that:

- The ammonia gas nitriding, if suitably performed, can improve the wear resistance of austenitic stainless steel type AISI 316. From the results of this study, the considered nitriding process parameters were nitriding temperature, nitriding time and ammonia flow rate and the two dominate factors found to be the nitriding temperature, and the nitriding time, while the ammonia flow rate was found to be of less significance.
- The maximum wear resistance experimentally obtained was 93% (point No.1 in Table 2) as compared with the un-nitrided specimens and the RSM optimization predicts that an optimum wear 1.98% (H" 98% wear resistance) as compared with

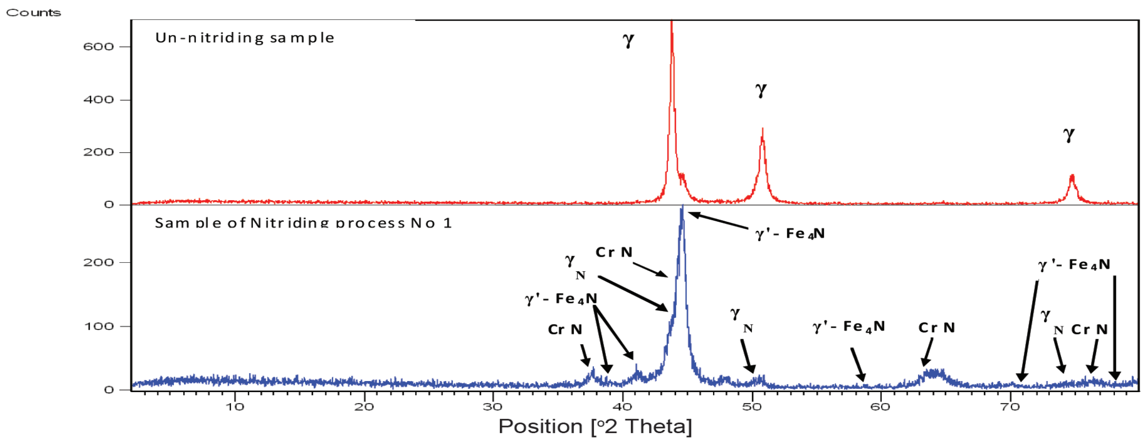


Fig. 8. X-ray diffraction patterns of a nitrided layer of nitriding process No. 1.

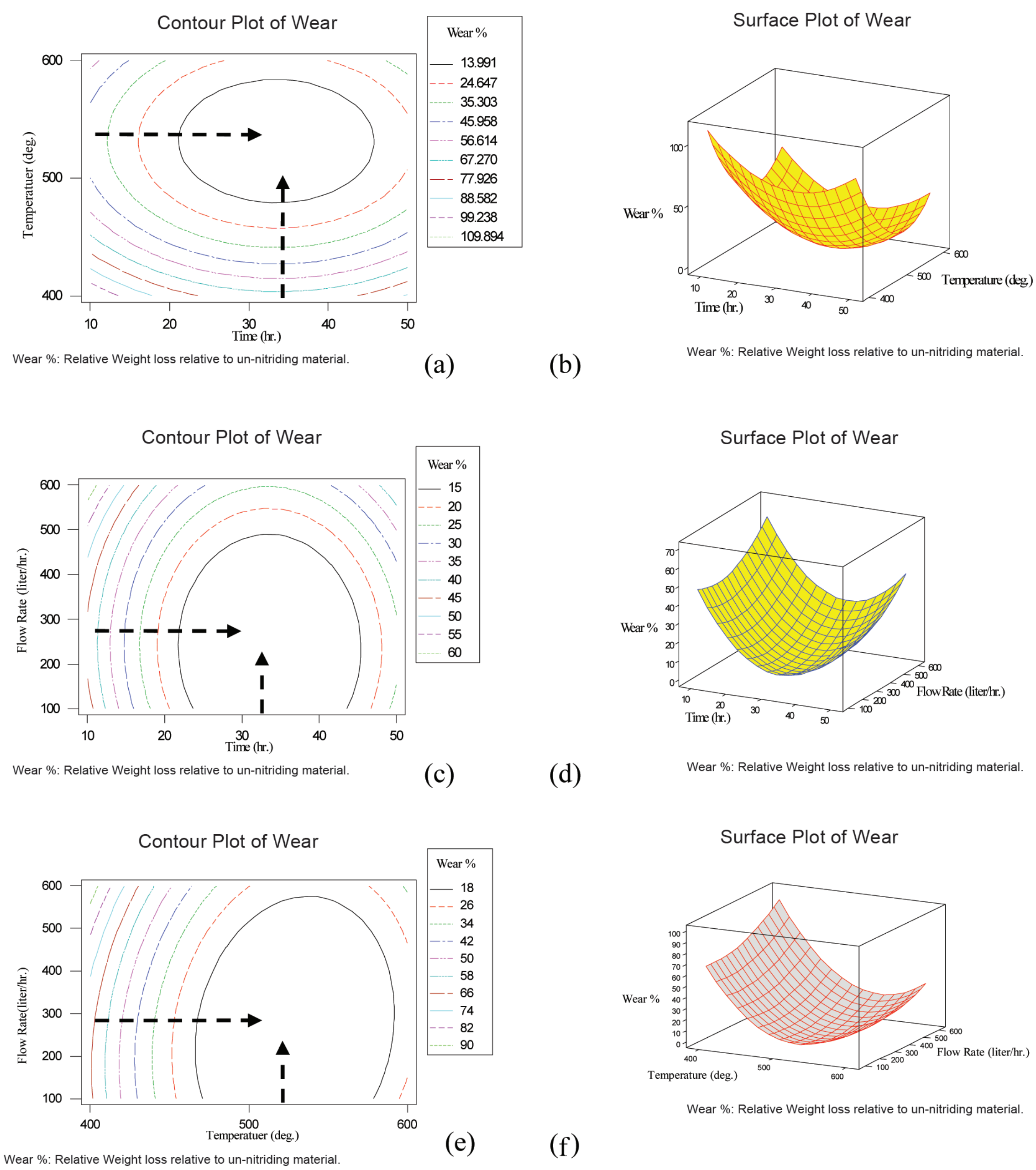


Fig. 9. Effects of nitriding processing parameters on wear resistance (MINITAB).

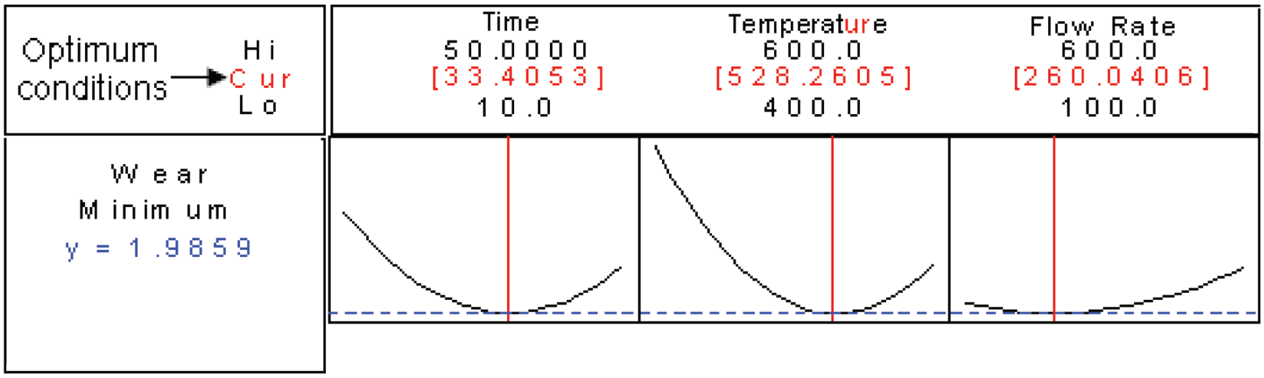


Fig. 10. Optimum nitriding conditions for minimum wear (1.9859 %).

the 100% wear of the un-nitrided specimens could be achieved at 528°C nitriding temperature for 33.4 hrs using 260 liter/hr ammonia flow rate.

- Gas nitriding of AISI 316 stainless steel has been shown to produce the ($\tilde{a}_N$) S-phase. This is reported to improve both corrosion and wear resistance of the of AISI 316 steel. However, a compromise between the wear and corrosion resistance can be achieved by controlling the nitriding process to produce a material suitable for the intended application.

ACKNOWLEDGEMENTS

The authors are thankful to all staff of the Libyan Technical Research Centre-Mechanical Research Branch, and the Libyan Petroleum Institute, Tripoli, for their help and co-operation throughout this research program.

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