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Effects of Nitriding and Thermal Processing on Wear and Corrosion Resistance of Vanadis 8 Steel

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Abstract: Vanadis 8 steel is a tool steel manufactured by powder metallurgical processing. Its main alloy elements are V, Cr and Mo. By implementing an experimental design with five factors—all of them are related to the thermal processing of this steel and with ionic nitriding—the effects of said factors on adhesive wear resistance and corrosion resistance were studied. For this purpose, Pin-on-Disc wear tests and lineal polarization resistance tests were carried out using an aqueous solution with 3.5% NaCl by weight. The main aim was to increase this steel use in more aggressive environmental conditions, such as in coastal environments. By means of XRD, the percentage of retained austenite was determined, and by SEM-EDX, the microstructure was revealed. The conclusion is that adhesive wear resistance is improved if thermal processing parameters are at such levels that increase austenite destabilization and reduce retained austenite content. This means to destabilize austenite at 1180 °C during 1 h, with oil quenching, tempering at 520 °C during 2 h and ionic nitriding at 520 °C during 2 h. Corrosion resistance is highly improved with ionic nitriding. At the same time, to compensate for the negative effect on corrosion resistance of a high density of primary and secondary carbides, it is essential to carry out the ionic nitriding treatment. The harmful effect of electrochemical microcells that appear in the carbide/matrix interface is compensated by the passivating effect generated by the nitrided surface.

Keywords: tool steel; secondary carbides; wear resistance; corrosion resistance; ionic nitriding



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1. Introduction

Cold work tool steels are used in the manufacture of tools used to form materials. These steels require a specific set of properties, such as high hardness and wear resistance, so their carbon contents are high. The presence of carbide-forming elements such as Cr, Mo, W and V improves hardenability and wear resistance. Vanadis 8 steel is a powder metallurgical tool steel sold by the Uddeholm firm (Hagfors, Sweden), with a high content of vanadium (8%), chrome (4.8%) and molybdenum (3.6%) [1–4]. This steel is widely used in the manufacture of dies, punches and tools because of its high hardness and excellent wear resistance. These properties turn it into the best choice for being used when adhesive wear is the main problem during long service. The main carbide present in the annealed condition is of the MC type, mainly related to V. The VC has a hardness of 2800 HV. This carbide is highly efficient for reducing grain growth during austenization treatment before cooling [5]. As a result of austenite destabilization, over 1000 °C, secondary carbides with a high level of Cr may precipitate, mainly of the M_7C_3 type. Austenite destabilization increases M_s -martensitic transformation start temperature-, reducing the retained austenite percentage [6].

Due to the high level of C content, as well as of alloy elements (Mo, Cr and V), this steel has a really low M_s temperature. Thus, it may have a high level of retained austenite after quenching [7–9]. Austenite existence is not desirable as it is soft and metastable at low temperatures. Therefore, it is subject to break down into martensite (tool weakening) [5,10].

One of the possible ways to reduce retained austenite is to carry out tempering at high temperatures, and thus, a second destabilization of austenite will take place, and it will be transformed into martensite while cooling [11]. Transformation of retained austenite begins with tempering at 470 °C [12]. During tempering over 500 °C, a secondary hardening may take place due to precipitation of carbides of the M_7C_3 [12,13] type. Nevertheless, tempering at high temperatures is not advisable for some types of steel due to the risk of high loss of hardness [10].

Corrosion resistance is not considered an important property to take into account when processing tool steels. The reason could be that most of the applications of these steels are developed in environments that are not highly aggressive. However, there are applications related to abrasive wear where it would be very interesting to increase the corrosion resistance of these steels. Some of these implementations could be mineral processing, ceramic powder compression, ore grinding, etc. [14,15]. It would also be important to highlight the possible use of these steels in coastal environments. Generally, it is considered that steels with more than 12% Cr content offer suitable corrosion resistance. Vanadis 8 steel has less than 5%, so it may be sensitive to corrosion, especially in coastal environments [16]. Thus, having a suitable combination of high wear resistance and high corrosion resistance would enable an increase in the range of implementation of these types of steel.

In the field of corrosion resistance, it has been experimentally demonstrated that austenite has a more noble behavior than ferrite. On the other hand, tempering treatment decreases corrosion resistance if compared with quenched structure [14]. This could be related to the dissolved carbon content in the martensite and the formation of galvanic cells between the precipitated carbides and the adjacent ferrite. Low-temperature tempering improves the creation of carbides of the cementitious type (M_3C). These carbides do not include Cr atoms. Thus, Cr proportion in solid solution in martensite should not be affected. Nevertheless, with higher tempering temperatures (above 500 °C), the re-dissolution of these cementitious carbides takes place due to the decomposition of these carbide atoms (Fe and C). Additionally, diffusion of Cr atoms, dissolved in a solid substitution solution, takes place, and then, the carbide precipitation of M_7C_3 with nano-metric size and greater dispersion [8]. This precipitation causes the ferrite structural hardening. If tempering takes a long time, coalescence of these carbides may take place, and it will negatively affect the ferrite corrosion resistance as they may have cathodic behavior with the adjacent ferrite [14,17].

In order to improve the adhesive wear resistance of this type of steel, it may be subjected to nitriding treatment. This treatment causes surface hardening due to the sub-nitride presence in the tempered martensite matrix. These sub-nitrides foster wear resistance increase. On the other hand, the nitrided area has no interface adhesion problems, and it has no delamination risk as it happens in the case of the layers deposited by PVD/CVD [18]. In the first nitriding stage, nano-precipitates of the CrN type appear that are coherent with the matrix. In the second stage, these precipitates will be thickened, losing their coherence with the matrix [19]. Nanometric precipitates of VN also appear, which are coherent with the matrix. These precipitates have much slower thickening than the CrN [20,21]. It has been verified in other martensitic steels that nitriding could improve corrosion resistance in chloride acid solutions due to noble behavior, compared with the substrate of nitrides, ϵ -Fe₂₋₃ (CN) and γ' -Fe₄N, present in the white layer [22,23]. At the same time, in solutions of NaCl at 3.5% in weight, though with excess regarding N content, it may reduce corrosion resistance [16,18,24–29]. In general, it seems that the influence of ϵ -carbo-nitrides against corrosion and γ' -nitrides against wear is more significant [30].

The aim of this work is to place the main thermal processing parameters at levels that allow the use of these steels in aggressive environmental conditions, such as coastal environments. This would allow tool and die manufacturers to define the most appropriate heat treatment to improve the in-service behavior of these steels under these conditions. For this purpose, the experimental methodology followed was the experiment design with five factors and eight experiments [31–33]. The factors analyzed include the effect of austenite

destabilization, the effect of the cooling medium, the effect of tempering conditions and the effect of ionic nitriding.

2. Materials and Methods

Table 1 shows the chemical composition of Vanadis 8.

Table 1. Chemical composition (wt.%).

C	Si	Mn	Cr	Mo	V
2.3	0.4	0.4	4.8	3.6	8

By applying a design of experiments, the aim is to deliberately modify certain working conditions in order to produce changes in some of the properties to be studied. In this case, the properties to be studied are adhesive wear resistance and corrosion resistance in coastal environments. To this end, the working conditions that were modified are shown in Table 2. In turn, this table indicates between which levels these working conditions were modified. These levels are identified as -1 and 1 [33]. The analysis of the effects that these modifications have on wear resistance and corrosion resistance allows us to place the working parameters at optimum levels. In other words, based on the analysis of these effects, it will be possible to define the most appropriate heat treatment to improve the behavior of these steels under conditions that require high wear resistance and high corrosion resistance in coastal environments.

Table 2. Factors and levels analyzed.

Code	Factors	Level -1	Level $+1$
A	Austenization temperature	1050 °C	1180 °C
B	Dwell time at austenization temperature	0.5 h	1 h
C	Quench cooling medium	air	oil
D	Tempering time (520 °C)	1 h	2 h
E	Nitriding	no	yes

In this case, a design of experiments with 5 factors was planned. In order to analyze the effects of all factors, including all interactions between factors, it would be necessary to use a factorial design with 32 experiments (2^5 experiments). The number of experiments can be reduced by assuming a loss of information, which, in industrial practice, is often irrelevant. Such experimental designs are called fractional designs. In our case, the number of experiments has been reduced to 8 (2^{5-2} experiments). Table 3 shows the matrix with these 8 experiments. The column “effect confusion pattern” shows which effects derived from interactions between two factors are confounded with the effects derived from single-factor variation. Columns D and E were generated from columns A, B and C as follows: $D = AB$ and $E = AC$ [31,32].

Table 3. Experiment matrix.

Experiment	A	B	C	D	E	Effect Confusion Pattern
1	-1	-1	-1	1	1	A + BD + CE B + AD C + AE D + AB E + AC BC + DE BE + CD
2	1	-1	-1	-1	-1	
3	-1	1	-1	-1	1	
4	1	1	-1	1	-1	
5	-1	-1	1	1	-1	
6	1	-1	1	-1	1	
7	-1	1	1	-1	-1	
8	1	1	1	1	1	

The effect of a factor on one of the properties studied, for example, wear resistance, is obtained from the variation of that property as a consequence of the variation of that factor between its levels -1 and 1 . The effect determined in this way is called the main effect. There are also effects that result from the interactions between factors. Interactions between two factors are defined as the variation between the average effect of a factor with the other factor at its level -1 and the average effect of that same factor with the other factor at level $+1$. Similarly, interactions among more than two factors will also be defined [31]. In industry, it is common that the influence of the main effects is greater than the influence of the effects derived from two-factor interactions (2nd order interactions), and these effects are, in turn, much greater than the influence of three-factor interactions and, thus, successively. The splitting of a design of experiments assumes that some of the second- and higher-order interactions are confounded in the effect of the main factors. These confounding variables are reflected in the column “effect confusion pattern” shown in Table 3. Only 2nd order interactions that are confounded with the main effects are reflected in this table. It does not include 3rd order interactions or higher.

A specific effect is defined as significant if it is “quite unusual” so as to take place by chance. Pareto charts allow the effects analyzed to be ordered according to their importance [32].

Values obtained from each experiment are subject to random variation. This variation will follow an ordinary law, where the standard deviation shows the experimental error. Effects are linear combinations of these values; therefore, they follow an ordinary law. If all the effects were non-significant, they would follow an ordinary law of zero average, so they would appear aligned on a chart made on a normal probability plot. Nevertheless, significant effects will follow an ordinary distribution with an average different from zero. Thus, they will not be aligned with the non-significant ones. This procedure allows us to discover the factors with significant effect on the property being studied. Both the statistical study and the determination of factors with significant effects were carried out using the software Statgraphics Centurion XVI (The Plains, VA, EUA) [34–36].

The material microstructure was analyzed using the reflection optical microscope Nikon Epiphot 200 (Nikon, Tokyo, Japan) and the scanning electron microscope Jeol JSM-5600 (Jeol, Nieuw-Vennep, The Netherlands), equipped with the characteristic dispersive X-ray micro-analysis system (EDX).

The “Pin-on-Disc” wear tests were carried out according to the ASTM G99 [37] standard using the Micro-Test MT/30/SCM/T tribometer (MicroTest, Madrid, Spain). The test speed was 0.27 m/s , and the force used was 30 N . As “Pin”, a WC ball of 5 mm diameter and $1600\text{--}1800\text{ HV}$ hardness was used. The total distance covered was 5 km .

One of the factors used was the ionic nitriding of the material. Table 4 shows the main parameters of the process with which said nitriding was carried out.

Table 4. Nitriding parameters.

Gas mixture	$70\%\text{N}_2 + 30\%\text{H}_2$
Gas flux (cm^3/min)	500
Temperature ($^\circ\text{C}$)	520
Pressure (Pa)	400
Time (min)	120
Output voltage (V)	500

By implementing the X-ray diffraction technique, the percentage of retained austenite was determined, following the recommendations mentioned in the ASTM E975-03 [38] standard, “Standard Practice for X-ray Determination of Retained Austenite in Steel with Near Random Crystallographic Orientation, ASTM International, 2008”. Measuring was carried out using the Stresstech Xstress 3000 G3R diffractometer (Stresstech Inc., Pittsburgh, PA, USA). Knowing the carbide percentage previously (VC), the measuring was based on estimating the area under the diffraction peaks of the ferrite and austenite stages. This area

is proportional to each stage's content. Detection of diffraction peak was carried out on the 45° position of the rotation angle, ψ , and 20 different positions of the Φ angle were measured and located, with the piece on a flat position between angles -45° and $+45^\circ$. Measures were taken on four crystallographic faces. Firstly, the two peaks corresponding to the austenite located on angles 130° and 80° , respectively, were measured, and then, the detectors were changed to the angles corresponding to the ferrite, 156.4° and 106.1° . Measures were repeated on the same 20 positions between -45° and $+45^\circ$ and always on the position of 45° of the rotation angle ψ .

To carry out data adjustment, an evaluation of the diffraction peaks of the austenite stage was carried out using a parabolic function to reduce surrounding noise. The Gauss function was used to adjust the relevant diffraction peaks. For the ferrite stage, diffraction peaks were adjusted by means of the PearsonVII function. A linear function for reducing noise was used [39,40]. On the other hand, it is important to mention that in the nitrided samples, the content of austenite inside the test tube was determined in order to avoid the nitrided layer thickness.

Corrosion resistance was analyzed based on a NaCl solution at 3.5% in weight. The methodology implemented was that of linear polarization resistance, according to the ASTM G59 [41] standard. Electrochemical measures were carried out by using a PalmSens4[®] potentiostat/galvanostat analyzer controlled by the PSTrace 5.7 software for the acquisition and analysis of electrochemical data. Electrochemical experiments were carried out in an ordinary electrochemical cell with three electrodes. The reference and auxiliary electrodes (counter-electrode) were the Ag(s)/AgCl(s) (3 M KCl) and a platinum thread of 150 mm, respectively. The samples corresponding to each experiment were used as working electrodes. The sample corrosion behavior was measured based on the potentiodynamic polarization technique. Corrosion tests were carried out at room temperature (about 25°C) with a potential range from -30 to $+30$ mV around the OCP with a scanning speed of 1 mV s^{-1} . Before the potentiodynamic test, the open circuit potential (OCP) was registered during 5400 s, up to the moment the interface stability was reached.

3. Results

Figure 1 shows the Vanadis 8 steel microstructure after thermal treatments. In this figure, all micrographs were taken in the cross-sectional area of the sample. These are representative samples of the microstructure found in the material after heat treatments. Figure 1a shows a high-density level of carbides in a ferritic matrix (tempered martensite). Figure 1b allows a clearer differentiation between these carbides and the matrix constituent. This figure shows the microstructure corresponding to experiment 7. Figure 1c shows the nitrided layer thickness. In all cases, such thickness was about 260 microns. Figure 1d,e show the nitrided layer microstructure of experiments 8 and 1. Both micrographs were taken at a distance of 30 microns from the outer edge of said nitrided layer. In these micrographs, points where the semiquantitative microanalysis was carried out by typical X-ray dispersion (EDX) are shown. Table 5 shows the results. Spectrum 3 shows the analysis obtained from the framed area in Figure 1b. Carbides of dark grey color (spectra 1, 8 and 11) seem to have some relation with mixed carbides related to vanadium and carbides of the MC type [8,42,43]. On the other hand, those having a lighter color may be identified as mixed carbides of the M_7C_3 type, related to Cr and Fe [8,43]. Some carbides, with dark color (spectrum 5), will correspond to the carbides of the M_2C type related to the Mo [8]. The matrix constituent has a content of Cr of about 5% (spectra 3, 9 and 12). The percentage of dissolved N in the matrix is around what is expected for this type of steel. Once the martensite is saturated with nitrogen, the carbides contribute to the absorption of nitrogen. It should be noted that in the nitrided samples, a significant presence of N in the MC-type carbides has not been verified. Nevertheless, it appears that N is disseminated in the M_7C_3 carbides. After nitriding, these carbides were converted into carbonitrides $M_7(C,N)_3$ [19]. Subsequently, nitrides of the Fe_4N , Fe_3N and CrN types begin to form [19]. These phases have a nanometric size, coherent or semi-coherent with the matrix and are not observable by SEM.

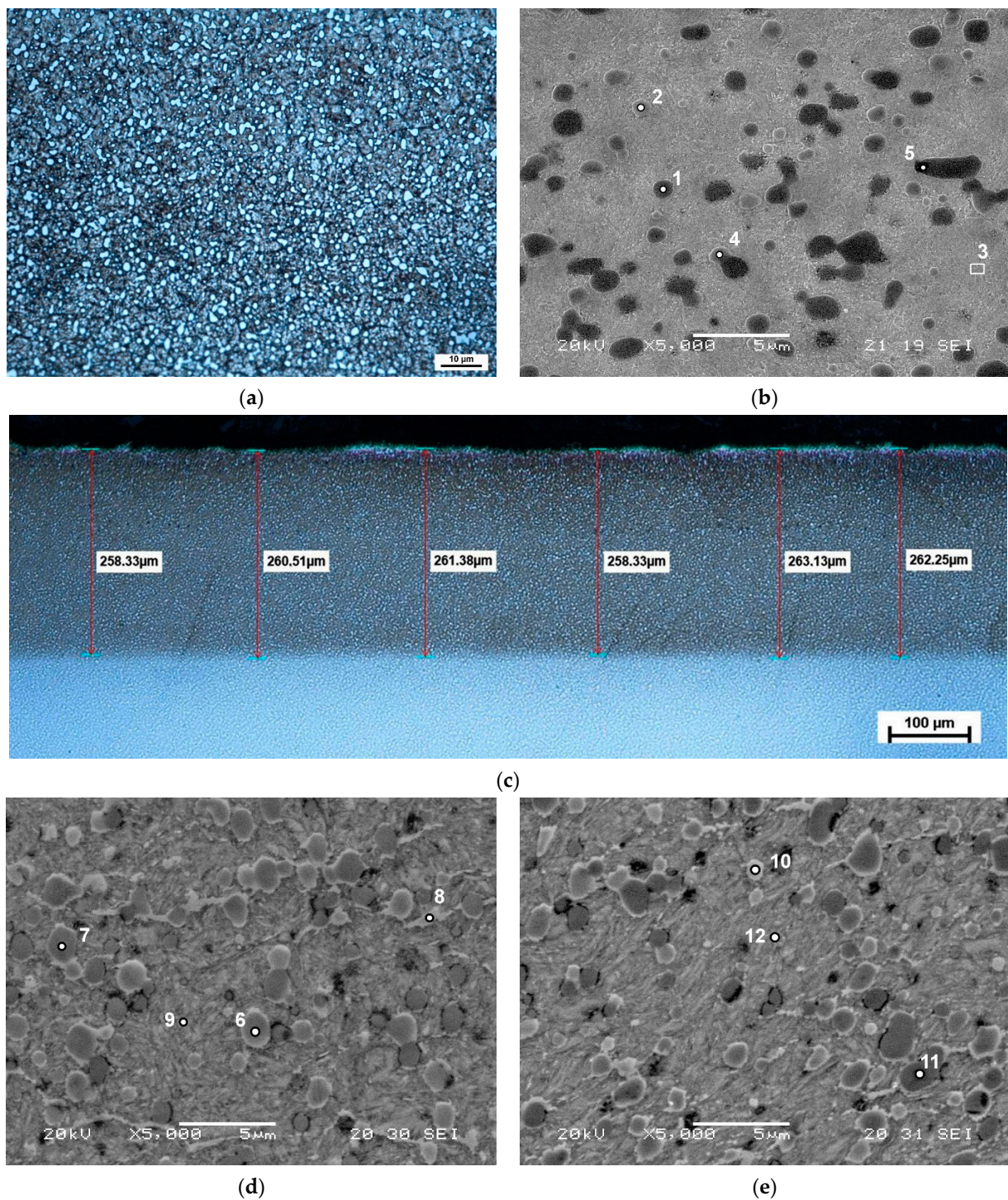


Figure 1. Microstructure of Vanadis 8 steel after thermal treatments. (a) Experiment 4 (non-nitrided sample); (b) experiment 7 (non-nitrided sample); (c) thickness of nitrided layer in experiment 6; (d) nitrided layer in experiment 8; (e) nitrided layer in experiment 1.

It should be noted that M_7C_3 carbides start to dissolve in austenite at temperatures in the vicinity of 1100 °C. However, MC carbides are “lazier” and dissolve to a lesser extent. The dissolution of Cr and part of the V allows the hardness to be increased after oil quenching. Wear resistance could be improved by favoring conditions for the precipitation of secondary chromium-rich carbides, mainly of the M_7C_3 type [43]. Prolonged destabilization could be favorable for this purpose. The destabilization of the austenite would increase the M_s temperature, reducing the percentage of retained austenite.

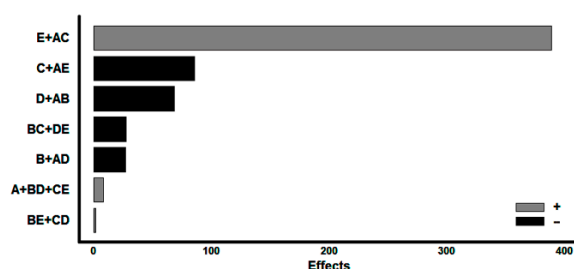
Table 5. Semi-quantitative analysis of carbides mentioned in Figure 1. Semi-quantitative micro-analysis by EDX (atomic %).

Spectrum	%C	%N	%V	%Cr	%Fe	%Mo	%Mn	Most Probable Mixed Carbide
1	Rem.	-	16.5	1.7	9.9	2.2	-	MC
2	Rem.	-	3.0	1.9	31.1	1.1	-	M ₇ C ₃
3	-	-	2.0	4.9	91.5	1.1	0.5	Matrix
4	Rem.	-	9.2	2.0	23.3	1.9	-	M ₇ C ₃
5	Rem.	-	9.6	2.1	15.8	16.5	-	M ₂ C
6	Rem.	10.7	1.6	2.2	32.2	0.6	-	M ₇ C ₃
7	Rem.	6.3	0.8	2.3	34.6	0.4	-	M ₇ C ₃
8	Rem.	-	13.6	2.5	17.8	2.3	-	MC
9	-	6.1	3.8	5.6	82.4	1.5	0.6	Matrix
10	Rem.	5.3	6.7	3.3	41	1.4	-	M ₇ C ₃
11	Rem.	-	25.8	2.9	5.1	3.8	-	MC
12	-	7.1	5.0	5.3	80.6	1.5	0.5	Matrix

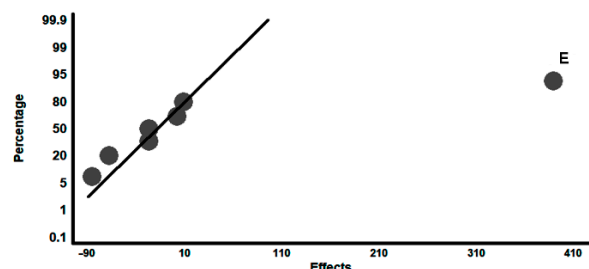
Table 6 shows the hardness results obtained from the non-nitrided samples (Vickers hardness, with a force of 300 N) and the nitrided samples (Knoop hardness, with a force of 0.5 N). Hardness corresponding to nitrided samples was obtained as the hardness average value from the first 100 microns of said layer, leaving a distance of 15 microns between the traces. This means that this hardness corresponds with the average of six hardness Knoop traces from the most superficial area of the nitrided layer up to a depth of 100 microns. The high error margin shown in the nitrided test tubes, with a 95% trust level, is due to the gradual reduction of hardness when going deeper into said nitrided layer. Figure 2 shows the graphic representation of effects, both by means of a Pareto chart, Figure 2a, as well as on a normal probability plot, Figure 2b. In the latter, the significant effect of nitriding is shown (E factor) in such a way that hardness is considerably increased when the material is subject to nitriding treatment.

Table 6. Hardness and effects. HV-300 N = Vickers hardness with a force of 300 N (non-nitrided samples); HK-0.5 N = Knoop hardness, with a force of 0.5 N, as average value in the first 100 microns of nitrided layer; CL-95% = Confidence Level at 95%.

Experiment	Type of Hardness	Hardness	CL-95%	Effects
1	HK-0.5 N	1375.8	±108.7	1178.28
2	HV-300 N	1065.9	±4.1	8.4
3	HK-0.5 N	1446.4	±58.5	−27.1
4	HV-300 N	996.1	±3.9	−85.5
5	HV-300 N	931.4	±5.3	−68.4
6	HK-0.5 N	1394.6	±102.3	388.5
7	HV-300 N	943.0	±4.3	−27.4
8	HK-0.5 N	1273.4	±92.2	1.8



(a)



(b)

Figure 2. Graphic representation of effects on hardness: (a) representation on Pareto chart; (b) representation on normal probability plot.

Table 7 shows the results of retained austenite. In those experiments with nitrided samples (experiments 1, 3, 6 and 8), austenite was determined outside the nitrided layer at a depth of 1 mm below the inner limit of said layer. Figure 3 shows the graphic representation of effects by the Pareto chart and the normal probability plot. In the latter, those factors with significant effects are underlined.

Table 7. Content of retained austenite; CL-95% = Confidence Level at 95%.

Experiment	wt. (%)	CL-95%	Effects	
1	10.6	± 1.6	7.6	average
2	12.3	± 2.2	−0.15	A + BD + CE
3	7.5	± 1.8	−2.15	B + AD
4	8.5	± 2.0	−4.25	C + AE
5	7.0	± 2.0	0.1	D + AB
6	4.8	± 1.1	−1.5	E + AC
7	5.6	± 1.3	1.3	BC + DE
8	4.5	± 1.1	0.45	BE + CD

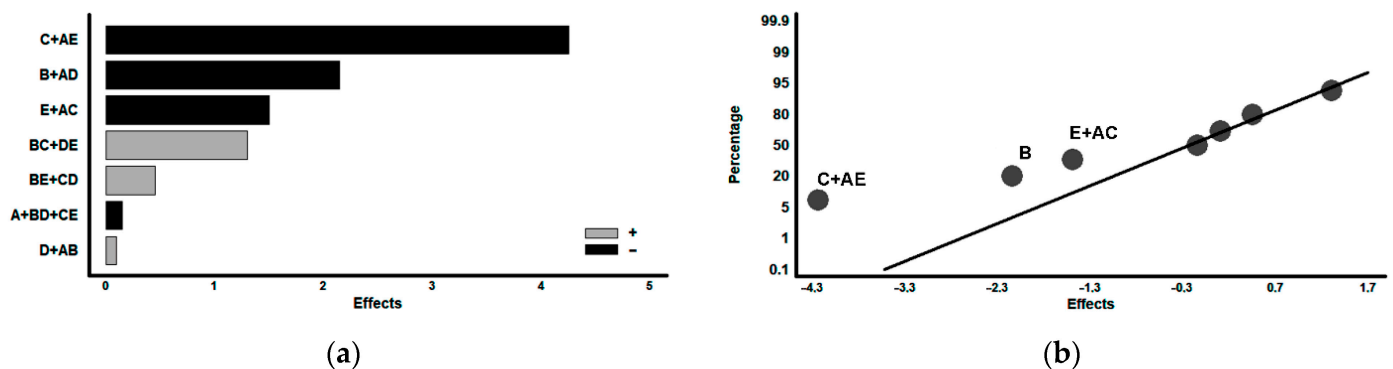


Figure 3. Graphical representation of the effects on retained austenite: (a) representation on Pareto chart; (b) representation on normal probability plot.

The main factors with significant effects are C (cooling medium), B (dwell time at austenitizing temperature) and E (nitriding). Thus, if said factors are at their −1 level, the retained austenite content will increase. Therefore, retained austenite will be considerably increased if these factors are at their respective −1 levels (time at the austenite destabilization temperature of 30 min, air cooling and with no nitriding).

In this way, the conclusion is that with 1 h of destabilization, oil cooling and nitriding treatment, the amount of retained austenite is reduced. Nitriding treatment is equivalent to a second austenite destabilization, improving its cooling transformation. The effect of the E factor (nitriding) hides the effect of the AC second-order interaction. Considering that the C factor (cooling medium) is significant, it is advisable to analyze this AC interaction. Similarly, the C factor effect (cooling medium) hides the effect of the AE second-order interaction. As the E factor (nitriding) is significant, it is also advisable to analyze this interaction. Table 8 shows a detailed analysis of both interactions. It is evident that when the A factor is at its +1 level (austenite destabilization at 1180 °C), together with the C and E factors at their +1 levels, there is a reduction in the retained austenite. Therefore, it may be concluded that there is an “additional” reduction of austenite if austenite destabilization takes place at 1180 °C, together with oil cooling and nitriding treatment.

Table 8. Analysis of the effect of AC and AE interactions on austenite percentage.

A(↓) × C(→)	−1	+1	A(↓) × E(→)	−1	+1
−1	9.1	6.3	−1	6.3	9.1
+1	10.4	4.7	+1	10.4	4.7

Table 9 shows the weight loss results obtained with the Pin-on-Disc test (adhesive wear). Figure 4 shows the graphic representation of effects by means of a Pareto chart and normal probability plot. In the latter, those factors with significant effect are pointed out. The “importance” of the effects of main factors B (austenite destabilization period), C (cooling medium) and A (destabilization temperature) is underlined. In such a way that, if said factors are at their -1 levels, a greater weight loss takes place due to the wear suffered during the Pin-on-Disc test. Therefore, if these same factors are at their $+1$ levels (destabilization at $1180\text{ }^{\circ}\text{C}$ during 1 h and oil cooling), an increase in the wear resistance takes place. These three factors, at said levels, also benefit the reduction in retained austenite.

Table 9. Adhesive wear resistance. Weight loss in mg.

Experiment	mg	Effects	
1	19.3	8.37	average
2	11.7	−4.8	A + BD + CE
3	8.1	−7.9	B + AD
4	6.3	−5.9	C + AE
5	14	3.7	D + AB
6	4.4	−0.1	E + AC
7	1.8	0.3	BC + DE
8	1.4	0.8	BE + CD

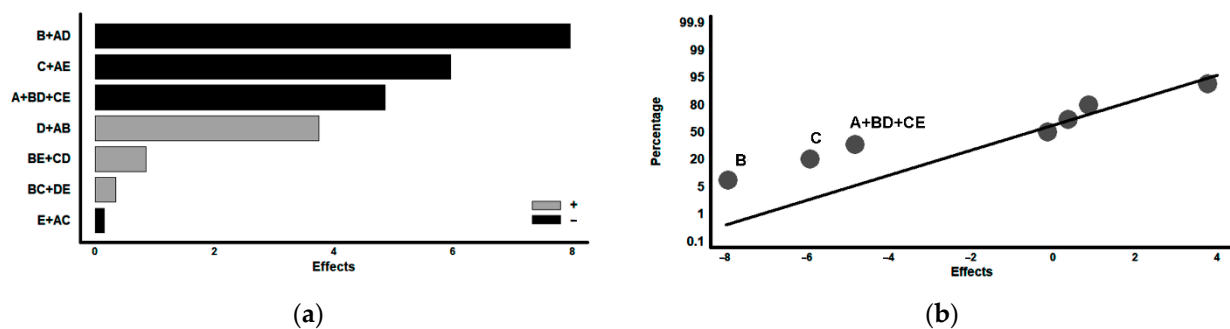


Figure 4. Graphic representation of the effects on the adhesive wear resistance: (a) representation on the Pareto chart; (b) representation on the normal probability plot. In the latter, factors with significant effects are underlined.

Therefore, it may implicitly be suspected that there is a direct relation between adhesive wear resistance and the amount of austenite present. It is important to take into account that the effect of the main factor A hides the effects of the BD and CE interactions.

Considering that the main factors B and C have a significant effect, and both factors are present in said interactions, it is advisable to analyze these interactions separately. Table 10 shows said analysis. The result is that, apart from destabilizing at $1180\text{ }^{\circ}\text{C}$, the significant effect of the B and C factors is “reinforced” if the tempering period is of about 2 h and if nitriding is carried out. It should be taken into consideration that, in this type of steel, tempering has a very significant influence on the amount of austenite retained. During tempering, secondary hardening can occur due to the precipitation of carbides of the M_7C_3 and MC types [43]. A remarkable example of this analysis could be the case of experiment 5, which shows a high weight loss (14 mg) despite being oil quenched ($C = +1$). This sample was austenitised at $1050\text{ }^{\circ}\text{C}$ ($A = -1$) for 0.5 h ($B = -1$). Such low destabilization times (0.5 h) favor an increase in retained austenite. In addition, the CE interaction has a significant effect, so that if $C = +1$ and $E = -1$ (without nitriding), there is a significant increase in wear. It should be noted that the nitriding treatment is equivalent to a second destabilization of the austenite. Table 11 shows a summary of the results obtained. Therefore, the conclusion is that those conditions that benefit austenite destabilization

(austenization at 1180 °C during 1 h and tempering during 2 h), together with oil cooling and further nitriding, provide more wear resistance to the material. The formation of CrN is associated with nitriding temperatures above 500 °C. This nitride promotes an additional increase in hardness and wear resistance [44–46]. One of the factors analyzed in our work was nitriding at 520 °C for 2 h, so the presence of this type of nitride in the diffusion layer cannot be ruled out [19,44,45]. It should be noted that the increase in wear resistance reflected in the results due to the effect of ion nitriding is comparable to that of other vanadium powder metallurgy steels and is superior to the results obtained with other hard PVD coatings [47] despite having a lower hardness than these [48]. Regarding the effect of diamond-like carbon coating (5.94 µm thickness) on the wear resistance at dry sliding conditions, it was found that the DLC coating may result in a significant reduction, reaching 70% in weight loss in a high carbon low-alloy steel [49]. However, in the present study, wear resistance was improved after combining thermal processing with ionic nitriding. The average thickness of the nitrided layer was 260 µm, positively impacting wear and also in corrosion properties. In our case, the improvement percentage of weight loss was higher than 90% in series austenitized at 1180 °C for 1 h, quenched in oil, tempered at 520 °C for 2 h and, subsequently, nitrided at 520 °C for 2 h. This suggests that in comparison to DLC coatings, the ionic nitriding process can also be used to improve wear resistance.

Table 10. Analysis of the effects of BD + CE interactions on the adhesive wear resistance.

B(↓) × D(→)	−1	+1	C(↓) × E(→)	−1	+1
−1	15.5	16.65	−1	9	13.7
+1	4.95	1.6	+1	7.9	2.9

Table 11. Factors with significant effects on adhesive wear resistance and levels for which wear is reduced.

Factors	Interactions
A = +1; destabilization at 1180 °C B = +1; destabilization period of 1 h C = +1; oil cooling	BD = +1; destabilization period of 1 h and 2 h for tempering CE = +1; oil cooling and nitriding

Table 12 shows the results obtained from the polarization resistance test. Figure 5 shows the polarization curves, and Figure 6 shows the graphic representation of effects by using the Pareto chart and by means of the normal probability plot. In the latter, factors with significant effects are underlined. Figure 6b shows the clearly significant effect of nitriding on potential corrosion in such a way that nitriding increases said potential. Figure 6d shows the same significant effects of nitriding on polarization resistance (factor E) in such a way that this thermochemical treatment significantly increases said resistance. Factor D also shows the same effect on polarization resistance (tempering time) in such a way that if the said period is increased up to 2 h, an increase in polarization resistance takes place. In several scientific articles, it was published that carbide existence has a harmful effect on corrosion resistance as micro-electrochemical cells establish in the carbide/matrix interfaces [14,50,51]. Nevertheless, limits between the thin tempering carbides and the matrix tend to be attacked with less strength by corrosion, thus helping to reduce the corrosion rate if they are present in sufficiently high amounts [14]. Additionally, the significant effect of some of the BE and CD second-order interactions may also be observed. Table 13 shows the independent analysis of both interactions. It may be observed that it seems that BE interaction is the one that shows a significant effect in such a way that if austenite destabilization is carried out during 1 h, it is advisable for the material to be treated with nitriding to increase polarization resistance. Figure 6f shows the significant effects of nitriding over corrosion intensity in such a way that the latter considerably decreases when said thermochemical treatment is carried out. At the same time, it is also observed that any of the second-order interactions, BE or CD, may have a significant

effect on said corrosion intensity. Table 14 shows the analysis of the effects separately. Results show that, again, the BE interaction is the one with said significant effects. Thus, if destabilization treatment is carried out during 1 h ($B = +1$) and nitriding treatment is not carried out ($E = -1$), a significant increase in corrosion intensity takes place. It seems to be confirmed that if the destabilization period is 1 h, it is essential to carry out nitriding treatment in order to avoid the increase in corrosion kinetics.

Figure 7 shows the areas of experiments 7 (non-nitrided sample) and 8 (nitrided sample) after polarization tests. In Figure 7a, it may be observed that in the area of experiment 7, carbides have a thin rust layer that provides them a cathodic behavior with reference to the matrix, fostering its anodic attack. It is informed that carbide/matrix limits are preferably attacked by corrosion [14]. In the matrix, some whitish “needles” may be seen, though they were not visible before polarization. Therefore, these carbides may have been taken from the austenite destabilization [52].

Table 12. Results of the polarization resistance test.

Exp.	Ecorr		Rp		Icorr		Effects
	mV vs. OCP	Effects	$k\Omega \cdot cm^2$	Effects	$\mu A/cm^2$	Effects	
1	−276	−467.25	27.0	20.97	0.22	0.46	average
2	−687	−10.0	9.9	6.4	0.52	−0.07	A + BD + CE
3	−233	29.0	23.4	6.7	0.20	0.14	B + AD
4	−689	8.0	9.0	7.3	0.81	0.05	C + AE
5	−690	−12.5	14.7	12.8	0.50	−0.13	D + AB
6	−274	423.5	18.9	22.1	0.32	0.52	E + AC
7	−650	8.5	60	8.9	1.08	0.01	BC + DE
8	−239	10.0	58.9	11.5	0.06	−0.29	BE + CD

Table 13. Analysis of the effect of BE + CD interactions on polarization resistance.

$B(\downarrow) \times E(\rightarrow)$	−1	+1	$C(\downarrow) \times D(\rightarrow)$	−1	+1
−1	12.26	22.99	−1	16.64	18.03
+1	7.50	41.16	+1	12.46	36.78

Table 14. Analysis of the effect of BE + CD interactions on corrosion intensity.

$B(\downarrow) \times E(\rightarrow)$	−1	+1	$C(\downarrow) \times D(\rightarrow)$	−1	+1
−1	0.51	0.27	−1	0.36	0.51
+1	0.94	0.13	+1	0.70	0.28

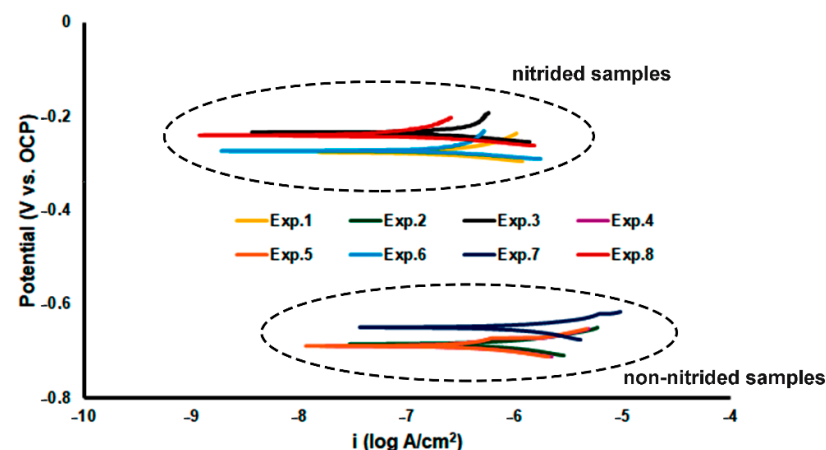


Figure 5. Polarization curves. Curves with a higher percentage of corrosion correspond to the nitrided samples.

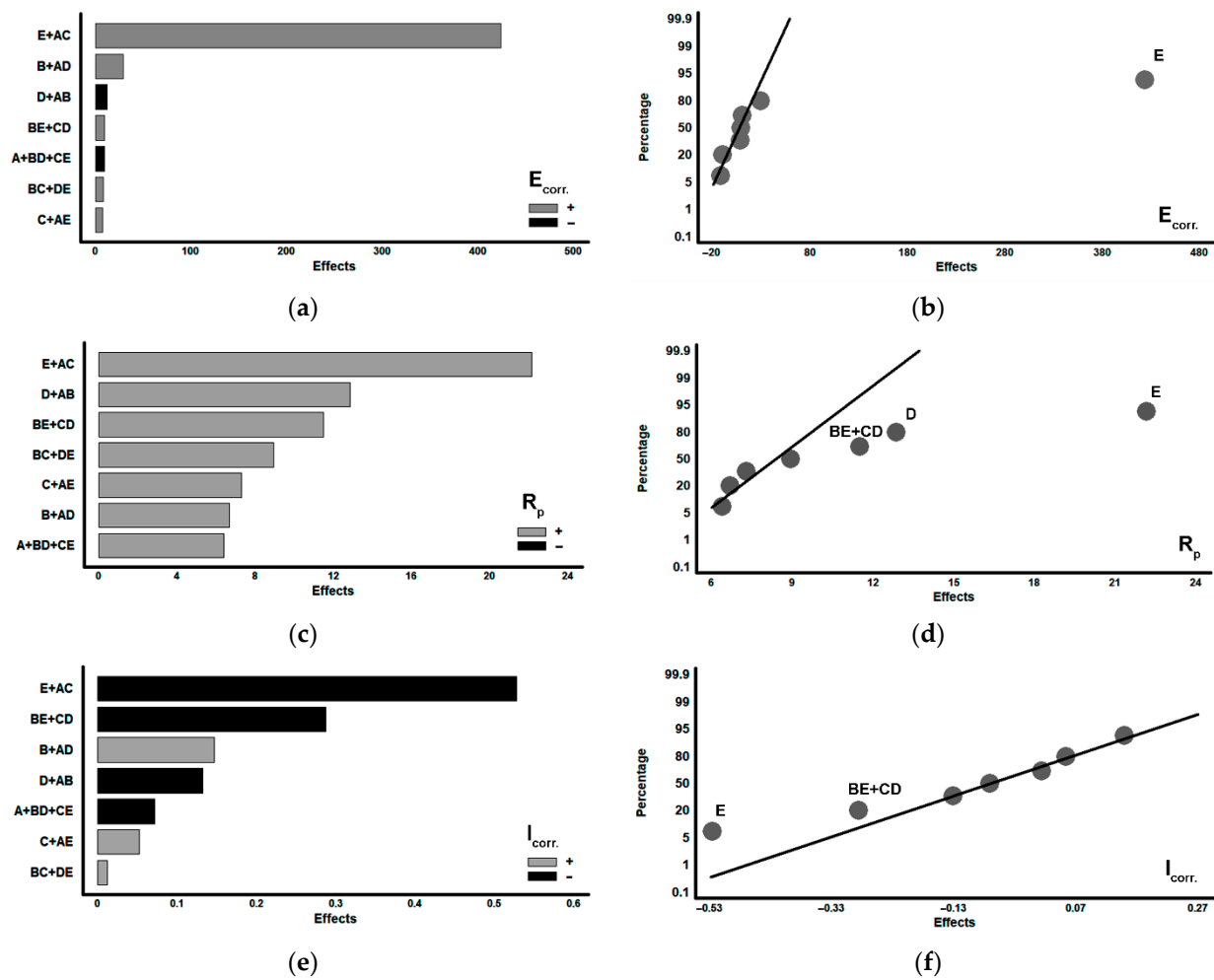
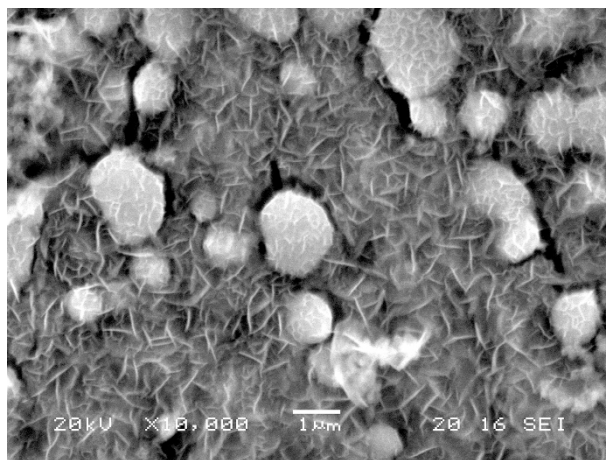
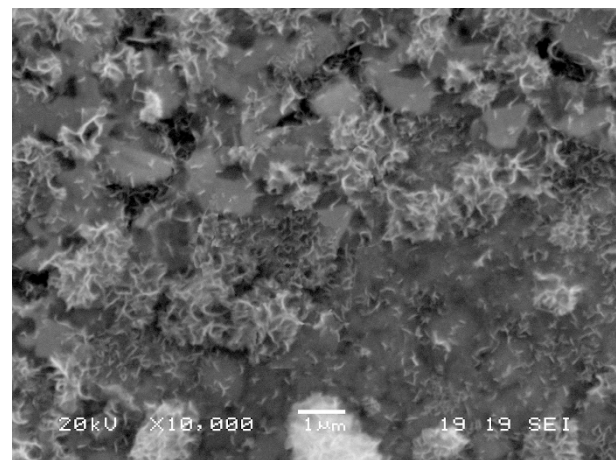


Figure 6. Graphical representation of the effects on corrosion potential (E_{corr}), polarization resistance (R_p) and corrosion intensity (I_{corr}). (a,c,e) show the representation on Pareto charts; (b,d,f) show the representation on normal probability plots. In the last diagram, factors with significant effects are underlined.



(a)



(b)

Figure 7. Surface after 4 h of being in touch with electrolyte. (a) Experiment 7 (non-nitrided), $\times 10,000$; (b) experiment 8 (nitrided), $\times 10,000$.

Figure 7b shows the area of one of the nitrided samples, in this case, of experiment 8. Its corrosion potential is considerably higher than that of sample 7 (non-nitrided sample). The nitrided surface is made up of nitrogen and iron nitrides in a solid solution, which is responsible for the improvement of steel corrosion resistance [18,53,54], giving it a passive behavior [15,29,30,55]. When the nitriding temperature exceeds 500 °C, CrN can form, which reduces the amount of Cr in solid solution, potentially negatively affecting corrosion behavior [44,45]. In this case, the temperature used in the nitriding process (520 °C) does not seem to have negatively affected corrosion resistance.

It should be noted that the non-nitrided samples exhibited an average roughness between 0.05 and 0.15 microns. However, the average roughness of the nitrided samples increased slightly to values ranging from 0.1 to 0.5 microns. Therefore, it must be emphasized that the small increase in roughness resulting from the nitriding treatment does not have a negative influence on corrosion resistance.

4. Conclusions

With the ultimate goal of increasing the use of the Vanadis 8 powder metallurgic steel under more aggressive environmental conditions, such as coastal environments, an experimental design with five factors—related to thermal processing and ionic nitriding of this steel—was carried out. The effect of said factors on adhesive wear resistance and corrosion resistance was analyzed. The main conclusions were:

1. The nitrided layer thickness has an average size of 260 microns. Mixed carbides of MC stoichiometry related to V have no significant N content. Nevertheless, mixed carbides of M_7C_3 stoichiometry have an N content similar to that of the matrix constituent.
2. Adhesive wear resistance is increased if thermal processing parameters are at such levels that foster austenite destabilization and the reduction of retained austenite content. This means carrying out austenite destabilization at 1180 °C during 1 h, with oil quenching, tempering at 520 °C during 2 h and a thermochemical treatment of ionic nitriding.
3. Ionic nitriding improves the increase in corrosion potential and polarization resistance in an aqueous solution at 3.5% NaCl.
4. Polarization resistance is increased with tempering periods of 2 h. If the density of fine-tempering carbides is high, interfaces between these carbides and the matrix are attacked with less intensity, and the corrosion rate is reduced.
5. Nevertheless, to reduce the negative effect on the corrosion rate, which implies a high density of primary and secondary carbides that are bigger than previous ones appearing during the austenite destabilization, it is essential to carry out the ionic nitriding treatment to increase polarization resistance and reduce said corrosion rate.

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