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Article

Nitride layers formed on austenitic stainless steel: Scratch and VDI appraisal

Capas de nitruros formadas sobre un acero inoxidable austenítico: Evaluación por rasgado y VDI

García-Chávez, Jorge Alan[a], Melo-Máximo, Lizbeth[b], Solis-Romero, José[c] and Vega-Morón, Roberto Carlos[d]*

[a] Tecnológico Nacional de México / IT de Tlalnepantla • IDOUJ-9610-2025 • 0009-0006-3236-5049 • 1328579
[b] Tecnológico Nacional de México / IT de Tlalnepantla • AAF-1812-2021 • 0000-0002-7081-0661 • 299373
[c] Tecnológico Nacional de México / IT de Tlalnepantla • AGJ-8856-2022 • 0000-0002-2483-083X • 0921
[d] Tecnológico Nacional de México / IT de Tlalnepantla • AAG-1615-2020 • 0000-0003-4772-7904 • 513822

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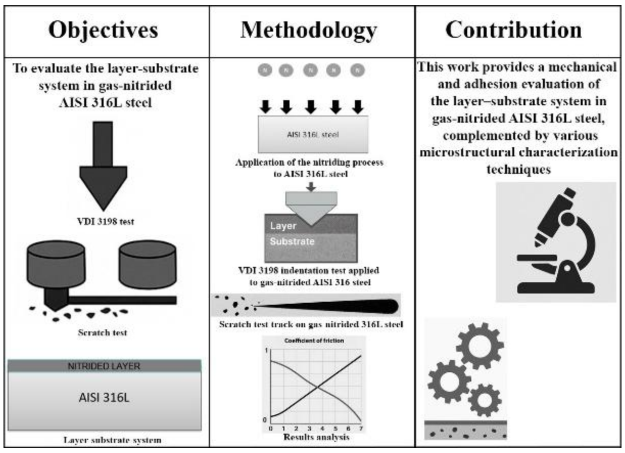
* ✉ [roberto.vm@tlalnepantla.tecnm.mx]

Abstract

This study appraised the adhesion of the layers developed on AISI 316L austenitic stainless subjected to gas nitriding at 560 °C during 24 and 72 h of exposure time. The resulting nitrided layers formed at the surface of the AISI 316L steel presented a thickness of 60.5 µm for 24 h, and 79.5 µm for 72 h. Furthermore, by applying the VDI 3198 indentation tests along with scratch testing operating in progressive load mode, the nitride layers integrity was examined. Whether 24 or 72 hours of exposure time, the layers exhibited an adhesion classified as acceptable, according to the chart disclosed in the standard, presenting radial cracks without significant spallations in both layers. Finally, only cohesive damage was produced by scratch testing, the corresponding critical loads in 24 and 72 h were determined, associated with tensile cracks, lateral spallation and angular cracks.



To evaluate the layer-substrate system in gas-nitrided AISI 316L steel

Application of the nitriding process to AISI 316L steel

VDI 3198 indentation test applied to gas-nitrided AISI 316 steel

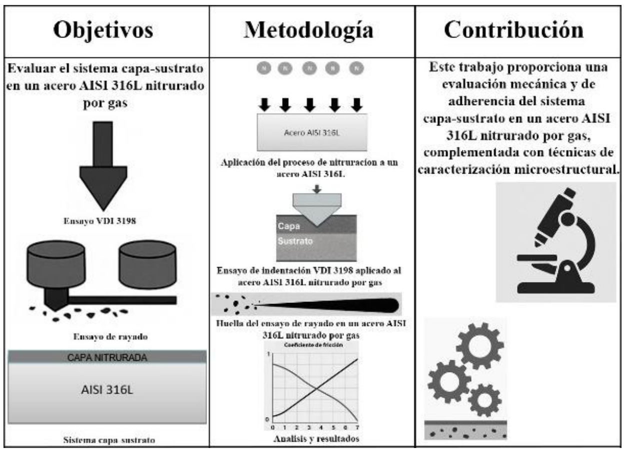
Scratch test track on gas nitrided 316L steel

This work provides a mechanical and adhesion evaluation of the layer–substrate system in gas-nitrided AISI 316L steel, complemented by various microstructural characterization techniques

Nitriding, scratch, adhesion

Resumen

Este estudio evaluó la adhesión de las capas desarrolladas en acero inoxidable austenítico AISI 316L sometido a nitruración gaseosa a 560 °C durante 24 y 72 h. Las capas formadas en la superficie del acero AISI 316L presentaron espesores de 60.5 µm para 24 h y 79.5 µm para 72 h. Además, mediante la aplicación de ensayos de indentación según la norma VDI 3198, junto con ensayos de rasgado en modo de carga progresiva, se examinó la integridad de las capas nitruradas. Tanto para 24 y 72 horas de exposición, las capas mostraron una adhesión clasificada como aceptable, presentando grietas radiales sin desprendimientos significativos en ambos casos. Finalmente, en los ensayos de rasgado únicamente se generaron fallas cohesivas y se determinaron las cargas críticas correspondientes para 24 y 72 h, asociadas a grietas de tensión, desprendimiento lateral y grietas angulares.



Evaluar el sistema capa-sustrato en un acero AISI 316L nitrurado por gas

Aplicación del proceso de nitruracion a un acero AISI 316L

Ensayo de indentación VDI 3198 aplicado al acero AISI 316L nitrurado por gas

Huella del ensayo de rayado en un acero AISI 316L nitrurado por gas

Este trabajo proporciona una evaluación mecánica y de adherencia del sistema capa-sustrato en un acero AISI 316L nitrurado por gas, complementada con técnicas de caracterización microestructural.

Nitrurado, rasgado, adhesión

Area: Promotion of frontier research and basic science in all fields of knowledge

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Introduction

AISI 316L steel belongs to the family of austenitic steels and is used in a wide variety of industries, such as aerospace, automotive, marine, and medical. Among its remarkable properties are its high corrosion resistance and biocompatibility with the human body, which make it a valuable material for engineering applications. However, despite these attractive properties, it presents an important limitation for long-term applications, since it possesses low mechanical and tribological properties (D'Andrea, 2023; Rodríguez-Castro et al., 2016)

There are different ways to address this problem through surface modifications; one of them is thermochemical treatments, which allow improvement of the tribological properties of steels (Mertgenç, 2025). One of these thermochemical treatments is boriding, carried out at high temperatures that enable the diffusion of boron atoms into the base material. When applied to steels, a layer of iron borides is produced with a hardness that can exceed 20 GPa, resulting in improved wear resistance (Garcia-Bustos et al., 2013). Another thermochemical treatment is nitriding, in which nitrogen atoms diffuse into the substrate. This can be performed through different methods, one of the most common being gas nitriding. This process is well known for surface hardening and can be applied to austenitic steels to increase surface hardness and improve wear resistance. Among its advantages are its low cost and the ability to be applied to parts of various sizes on a large scale (Shuaeib & Benyounis, 2016)

On the other hand, when a material is coated or subjected to a thermochemical treatment, it is essential that there is good adhesion between the coating or layer and the substrate, since this will determine whether the process is functional or not. Adhesion in a layer or coating is a key factor in ensuring coating quality; there are several methods to evaluate adhesion in engineering coatings, and the scratch test is one of the most common and effective methods, as it allows a semi-quantitative evaluation (Mittal, 2001). This method consists of producing controlled damage on the coating surface using a diamond counterpart, generally a Rockwell C indenter (with a tip radius of 200 µm), which travels across the surface applying a constant or progressive load.

In addition, another way to determine adhesion in a material is through a quick and reliable test called the Rockwell C indentation test, which allows a qualitative evaluation of the coating. This test is described in the VDI 3198 standard; it is a destructive test of coating quality, consisting of indenting the surface of the material with a conical diamond tip, causing plastic deformation of the material and fracture of the layer. The volume of the failure zone allows the adhesion of the layer to be evaluated through optical microscopy inspection, although scanning electron microscopy can also be used (Vidakis et al., 2003).

According to the above, there are some studies in the literature on adhesion and stainless steels subjected to thermochemical treatments. For example, Rodríguez et al., (2016) studied the borided layer of an AISI 304 steel using Rockwell C indentation and scratch tests. The authors concluded that despite having critical failures in the FeB phase, the borided layer reduces scratch resistance. They also highlighted that the exposure time during the boriding process is an important factor, since at 6 hours better results were obtained than at 2 hours.

On the other hand, Manfrinato et al., (2022) performed scratch tests on nitrided and nitrocarburized AISI 321 steel, both treatments carried out for 6 hours at temperatures of 400 and 500 °C. The results showed that at 500 °C a higher residual depth and a more irregular coefficient of friction (COF) were obtained, while the best results were obtained in the nitrocarburized sample at 400 °C, which exhibited better integrity after the test.

Likewise, regarding AISI 316L steel, Yildiz & Alsaran, (2010) performed multipass scratch tests on AISI 316L steel and Ti6Al4V alloy to study the coefficient of friction and compare the results between both samples after being subjected to plasma nitriding. The temperatures applied were 400 and 450 °C for the steel and 700 and 750 °C for the Ti6Al4V alloy. According to the results, the nitrided layer of AISI 316L steel proved to be unstable due to the significant presence of CrN and Fe_4N, causing fractures in the contact area. In contrast, the Ti6Al4V alloy showed greater stability in the sample treated at 750 °C, achieving a lower and more stable coefficient of friction.

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Therefore, although the literature reports work related to surface modifications through thermochemical treatments applied to austenitic stainless steels, there is limited information regarding the qualitative and semi-quantitative adhesion of AISI 316L steel subjected to gas nitriding treatment. Hence, the objective of this work is the evaluation of adhesion, through scratch and VDI 3198 tests, of the layer obtained by gas nitriding under two treatment conditions.

Methodology

For this study, the specimens were prepared from an AISI 316L stainless steel bar with a diameter of 25.4 mm, which was sectioned into 7-mm-thick disks. The samples were progressively ground to achieve a mirror-like surface finish, suitable for the subsequent thermochemical nitriding process. Prior to nitriding, the steel surfaces were subjected to a chemical pickling treatment.

After, the gas nitriding process was performed at a temperature of 560 °C and two exposure times: 24 h, corresponding to a single cycle (named 1C hereinafter), and 72 h, corresponding to three cycles (identified 3C hereinafter).

Next, a conventional metallographic process of the nitrided AISI 316L samples was conducted. The samples were cross-sectioned, mounted and ground sequentially with emery papers, from 120 to 1000 grit. The mirror finish was achieved by polishing with 0.25 µm diamond paste. The chemical etching was performed with Marble's reagent (HCl, $CuSO_4$, H_2O). The nitrided layer formed on each specimen was observed through optical microscopy, and its thickness was measured using ImagePro Plus software. Next, X-ray diffraction tests (XRD) were conducted (D8 Advance, Bruker), using Cu-Kα ($\lambda = 0.154$ nm) radiation, and a 2θ from 30 to 80°.

Later, the first adhesion assessment method, VDI indentation test, was conducted using a DISITEC DIS-DUR hardness tester, applying a load of 150 kgf with a Rockwell C diamond indenter, oriented perpendicular to the nitrided surfaces. The indentation imprints were examined by scanning electron microscopy and compared with the chart disclosed in the VDI 3198 procedure.

Additionally, the cross-sectional residual depth profile of each indentation was acquired by optical profilometry (GT-K Contour 3D, Bruker). Finally, progressive-load scratch tests (Revetest Xpress+, CSM Instruments) were conducted to investigate the practical adhesion resistance of the nitride layers. The tests employed an initial load of 5 N and a final load of 150 N, with a scratch length of 7 mm at a speed of 1.21 $mm \cdot min^{-1}$, using a Rockwell C diamond indenter with a 200 µm tip radius. Three repetitions were performed for each sample. The resulting scratch tracks were analyzed using optical microscopy, scanning electron microscopy (SEM), and optical profilometry.

Results

The nitrided layers formed on the surface of AISI 316L steel subjected to the gas nitriding treatment are showed in Figure 1. The thicknesses were obtained through optical microscope, obtaining 60.5 µm for the process of to 24 h (1C), and 79.5 µm in the 72 h (3C) treatment. After chemical etching, the layer presented a dark tonality, which revealed the precipitation of the CrN phase.

Box 1

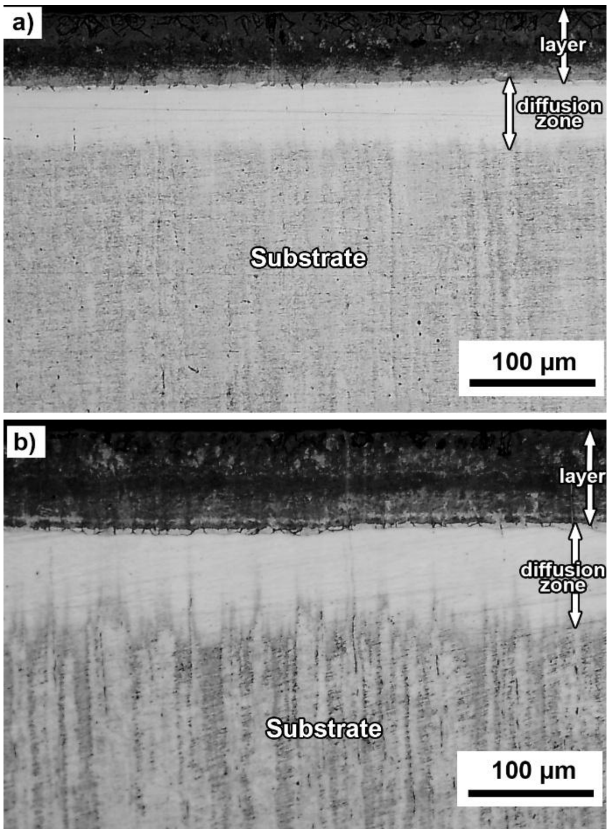

Figure 1

Cross-section micrographs of the AISI 316L subjected to nitriding: a) 24 h (1C); b) 72 h (3C).

The results allow to establish that nitriding is a diffusion-controlled process; since longer exposure times resulted in higher thicknesses. However, it has been reported that when the CrN phase precipitates, other properties, such as corrosion resistance, might be compromised.

In Figure 2 is presented the XRD spectra obtained for 1C and 3C layers. It can be seen that iron nitrides Fe_3N and Fe_4N were identified, along with some oxides associated to the process (Fe_2O_3 and Cr_2O_3), but more noticeable, the precipitation of CrN phase occurred, as suggested by the dark tonality seen in the metallographic process. Although this phase presents a high hardness, it also can compromise the corrosion properties of the steel, since the available Cr used to produce and regenerate the passivation layer, is used to form this compound (Larisch et al., 1999).

Box 2

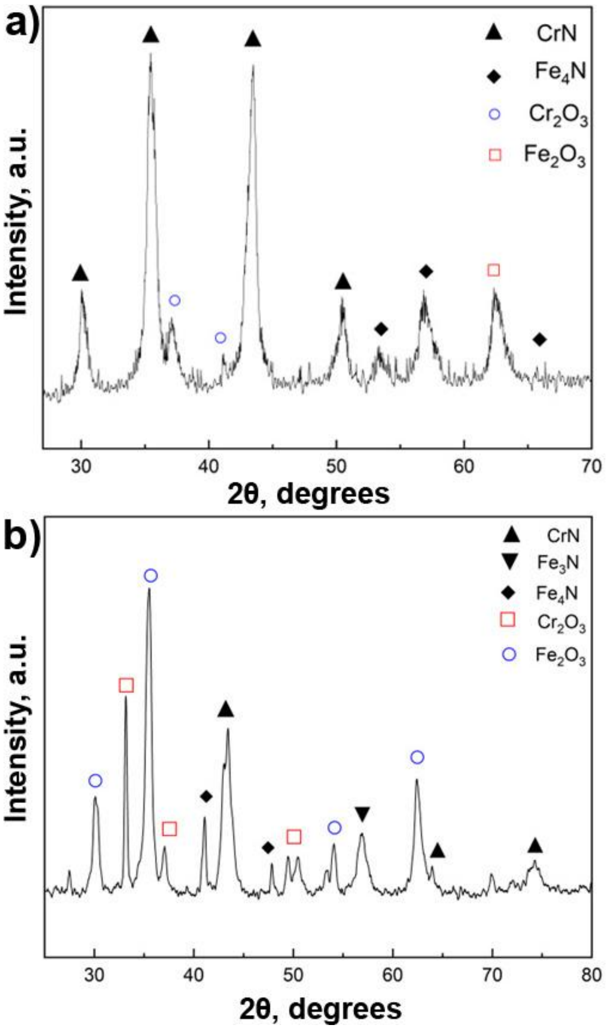

Figure 2
X-ray diffraction patterns: a) 24 h (1C); b) 72 h (3C).

In the Fig. 3 is presented the damage classification chart disclosed in the VDI 3198 adhesion test standard. The imprints obtained in the AISI 316L steel samples subjected to gas nitriding were compared with the chart, and after a visual inspection by optical microscopy, the damage was classified as acceptable or not acceptable (from HF1 to HF6), providing then a qualitative evaluation of the adhesion of the layers.

Box 3

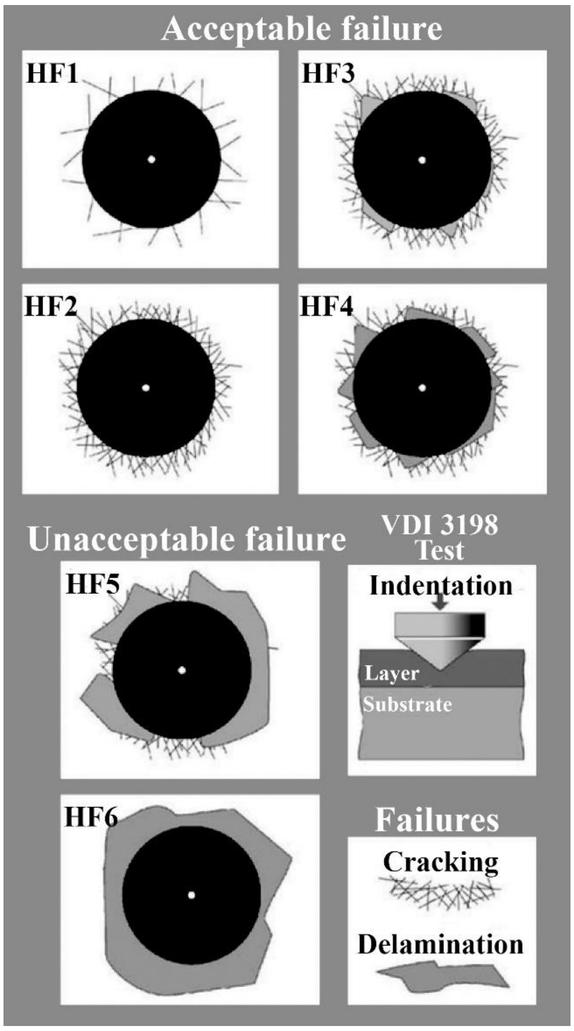

Figure 3
VDI 3198 failure chart.

A representative imprint obtained by SEM at the surface of the 1C sample is presented in Figure 4, along with details of the failures and their cross-sectional profile obtained by optical profilometry. As can be seen, only ring-shaped cracking occurred on the layer. Although a considerable number of cracks were registered, no partial or full delamination of the layer was detected.

Therefore, for this 1C layer, an acceptable adhesion was determined. From the residual depth profile, it can be seen that the deformation exceeded the layer thickness, however this indicates remarkable adhesion properties, since only cohesive failures occurred; the release of the deformation energy occurred by cracking rather than detaching the layer.

Box 4

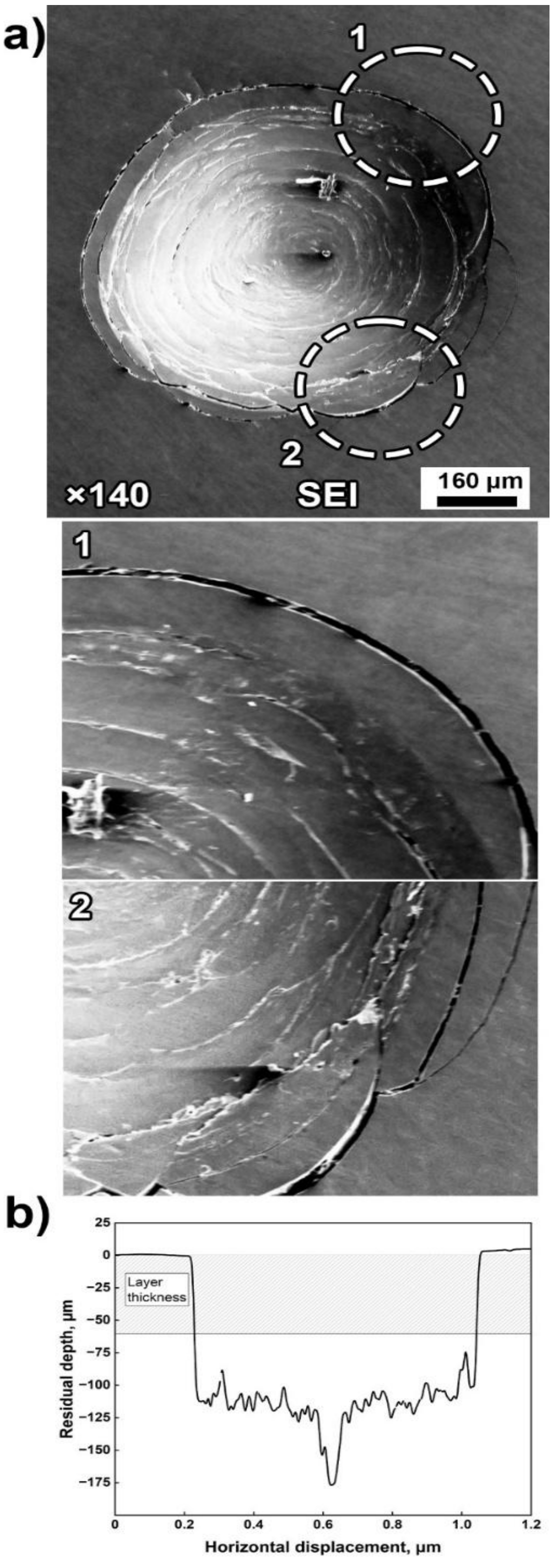

Figure 4

VDI results of 1C sample a) SEM micrographs; b) residual depth profile.

Moreover, in the Figure 5 is presented a representative indentation imprint of the 3C layer and its corresponding failures and cross-sectional profile. Ring-shaped cracking was again observed, although more severe than in 1C layer. However, these cracks did not produced delamination of the layer. Since no significant detachment was observed around the indentation edges, the adhesion was qualitatively considered as acceptable. Further, the indentation depth exceeded the layer thickness; however, severe delamination did not occur. Comparing both layers, the integrity of the 3C nitrided layer was more impaired than 1C, based on the number and size of cracks. Literature reports indicate that although the CrN phase can reach high hardness values, it is associated with more brittle behavior.

Box 5

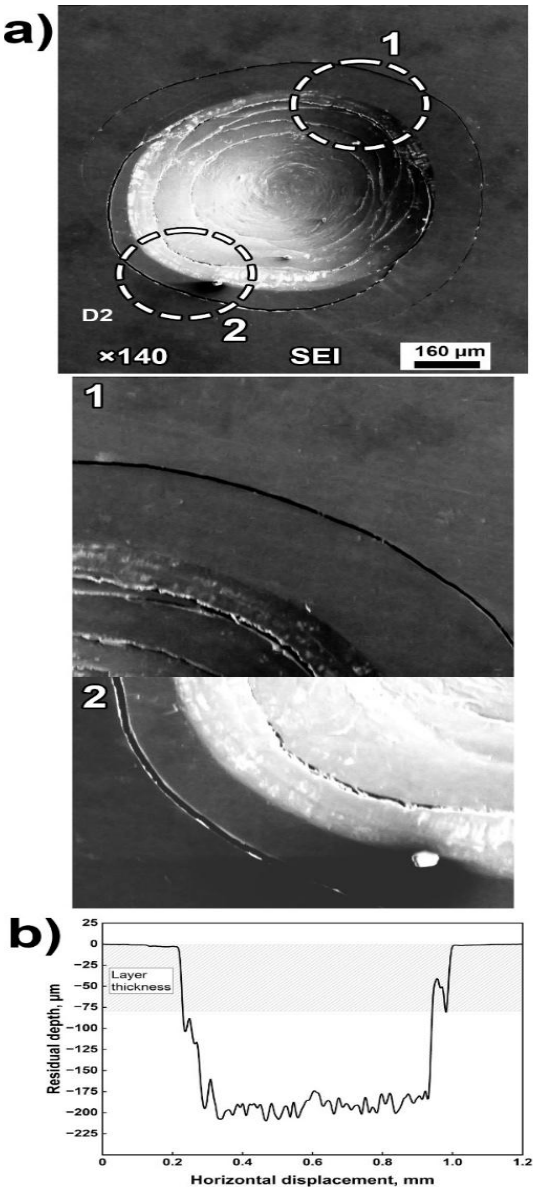

Figure 5

VDI results of 3C sample a) SEM micrographs; b) residual depth profile

Representative scratch tracks obtained from the samples are presented in Figure 6a, along with the corresponding coefficient of friction and residual depth profiles (Figure 6b). For the 1C sample, an attenuated coefficient of friction (COF) behavior was observed, with no severe fluctuations. Some small variations appeared around 3.5 mm of displacement, but its amplitude is limited.

The COF fluctuations during a scratch test are associated with failures occurring within the layer. The final coefficient of friction value was around 0.33. From the residual depth profile can be concluded that under experimental conditions employed, the indenter finished within the layer thickness. Although the scratch test did not completely exceed the layer, it still allows to evaluate the practical adhesion and the types of failure that occur under applied loading conditions. On the other hand, for the 3C layer, a final coefficient of friction of 0.35 was recorded. In this layer, the attenuated behavior can be associated with the absence of severe spallations or detachments of the layer.

Box 6

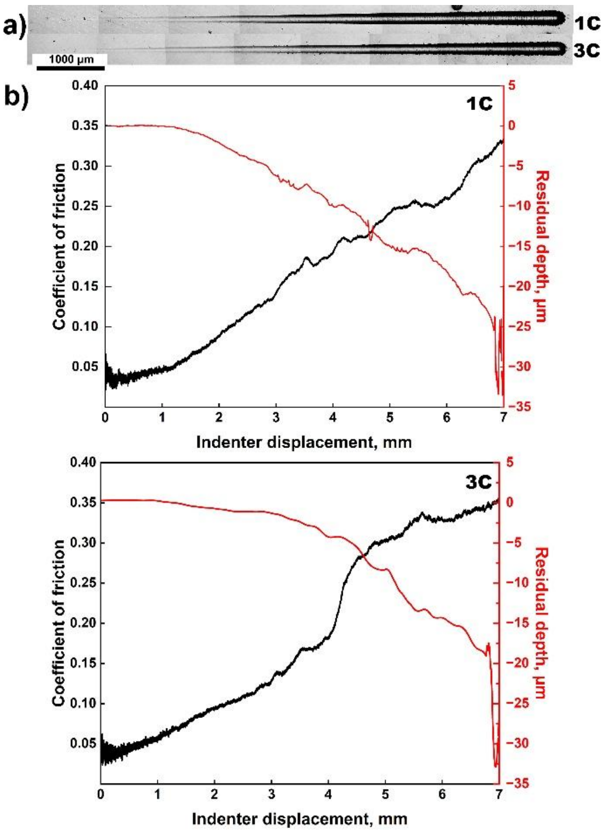

Figure 6

a) Representative scratch tracks; b) COF and residual depth behavior

Subsequently, the critical loads were calculated, according to the expression disclosed in the ASTM C1624 standard:

$$L_{c_n} = \left(\frac{l_n}{X_n} \cdot L_r\right) + L_i \quad (1)$$

where l_n is the critical distance (mm), or the distance where the particular failure appears; X_n is the linear speed (mm•min^{-1}) of the indenter; L_r is the load rate (N•min^{-1}), and L_i is the initial load (N). The Table 1 presents the critical loads for both layers.

Box 7

Table 1

Critical loads

Failure mechanism	Nitride layer	
	1C	3C
Tensile cracks	70±4 N	83.4±0.4 N
Chipping	95±5 N	66±2 N
Angular cracks	102±5 N	--------

It can be observed that although the tensile cracking increased its critical load with treatment time, the lateral spalling occurred earlier for 3C. This may be associated with the fact that 3C could better absorb deformation imposed by the indenter and also having a greater thickness. Figure 7 shows details of the scratch tracks obtained by SEM. The tensile cracks are formed behind the indenter during displacement movement, whereas the angular cracks occur due to high stress concentration behind the indenter and at the edge of the scratch track. Finally, chipping is the response of the layer–substrate system to release stored energy caused by compressive stresses ahead of the indenter (Bull, 1997).

Box 8

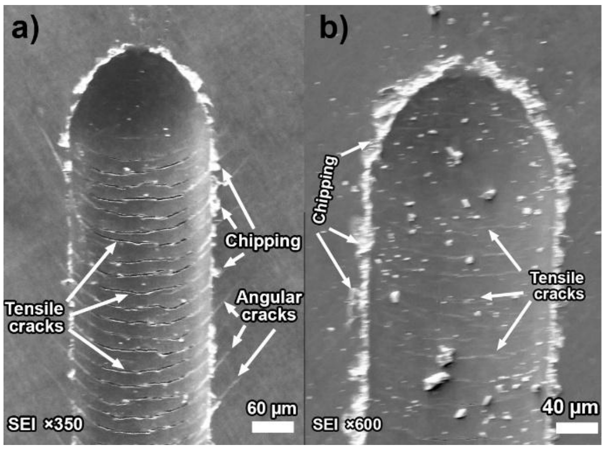

Figure 7

SEM micrograph of the scratch track a) 1C layer; b) 3C layer

Conclusions

This work presented the adhesion appraisal of the nitride layers formed on the surface of AISI 316L austenitic stainless-steel formed by gas nitriding. The treatments were performed at a temperature of 560 °C with two exposure times (24 and 72 h), obtaining thicknesses of 60.5 and 79.5 µm, respectively. Through VDI 3198 tests, a qualitative adhesion examination was conducted, obtaining failures that were classified as acceptable, since no full delamination of the layer occurred. Furthermore, the practical adhesion was studied by the scratch test, where only cohesive-type failures were observed, i.e. along the layer thickness. Although a good integrity was observed after both adhesion tests, the formation of CrN compromise other properties, therefore this treatment under the experimental setup employed should be carefully driven according to the application required.

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author Contribution

García Chávez Jorge Alan: Adhesion tests, data formatting and writing original draft

Melo Máximo Lizbeth: Microstructural characterization (SEM and XRD), formal analysis, data curation

Solis Romero José: Nitriding treatment supervision, formal analysis, methodology

Vega Morón Roberto Carlos: Project idea, resources, formal analysis, data curation and writing, review & editing of the manuscript.

Availability of data and materials

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Abbreviations

AISI	American Iron and Steel Institute
COF	Coefficient of Friction
SEM	Scanning Elctronic Microscope
VDI	Verein Deutscher Ingenieure
XRD	X-ray Diffraction

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