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REVIEW ARTICLE

TRIBOLOGICAL BEHAVIOR OF ACTIVE SCREEN PLASMA-NITRIDED OFFSHORE MATERIALS

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ABSTRACT

This review summarizes the results obtained from plasma nitriding as a potential solution to enhance the wear resistance of offshore materials without compromising their corrosion performance. The application of the active screen technique is one possible route for this purpose. To achieve a better comprehension of the topic, three recent research studies developed under the scenario of the Program of Excellence in Surface Engineering with emphasis on Plasma-Assisted Treatments were organized in the following order: i) comparing processes using a relatively low-cost substrate; ii) comparing the performance of two nitrided duplex stainless steels; and iii) analyzing the effect of the counterbody on the wear of a nickel superalloy. Although some limitations of the active screen process can be identified, the performance of layers deposited using this technique reveals a very promising application in reducing material costs. Nitriding intermediate-cost offshore materials can make them equivalent in performance to the more expensive ones.

KEYWORDS: Plasma nitriding, Corrosion-resistant alloys, Tribology.

COMPORTAMENTO TRIBOLÓGICO DE MATERIAIS OFFSHORE NITRETADOS EM GAIOLA CATÓDICA

RESUMO

Esta revisão resume os resultados obtidos da nitretação a plasma como uma solução potencial para aumentar a resistência ao desgaste de materiais offshore, sem perda em seu desempenho à corrosão. A aplicação da técnica de tela ativa é uma possível rota para esse propósito. Para alcançar uma melhor compreensão do tópico, três estudos de pesquisa recentes, desenvolvidos sob o cenário do Programa de Excelência em Engenharia de Superfícies com ênfase em tratamentos assistidos por plasma, foram organizados na seguinte ordem: i) comparação de processos usando um substrato de custo relativamente baixo; ii) comparação do desempenho de dois aços inoxidáveis duplex nitretados; e iii) análise do efeito do contracorpo no desgaste de uma superliga de níquel. Embora algumas limitações do processo de tela ativa possam ser identificadas, o desempenho de camadas depositadas usando essa técnica revela uma aplicação muito promissora na redução de custos de materiais. A nitretação de materiais *offshore* de custo intermediário pode torná-los equivalentes em desempenho aos de custo mais elevado.

PALAVRAS-CHAVE: Nitretação por plasma, Ligas resistentes à corrosão, Tribologia.

INTRODUCTION

The tribological performance of offshore components is far from being the crucial issue for selecting a material. The critical damage failures are governed by corrosion, and most probably, among them, the hydrogen-induced stress cracking can be considered the most dangerous.[1] It has been demonstrated that the corrosion depends

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Note: This paper represents most of the invited talk given at NANOSMAT24 in Barcelona, Spain.

on the H_2S partial pressure, in mixed environments (sweet/sour).[2] These failure modes define the guidelines for selecting corrosion-resistant alloys (CRAs).[3] However, in many components, friction and wear control are operational functions of a specific machine element, such as valves.[4]

The International Organization for Standardization (ISO) 15156-3[5] restricts strength and hardness in specific alloy systems. For example, nickel alloy Unified Numbering System (UNS) 625 has a maximum allowed hardness of 35 on the Rockwell C hardness scale (HRC), which can be achieved through proper heat treatment. This value also meets the structural purposes, but is likely insufficient for wear and friction control, meaning it may be an inadequate value under severe sliding conditions.[6]

When diverse failure possibilities exist within the same component, surface engineering emerges as a potential solution. Tom Bell has a quote that summarizes the spirit of complex problems involved herein[7,8]: "Surface engineering is the application of traditional or innovative technology to modify the properties of components and materials, creating a new composite material that combines the desirable characteristics of the surface and the base material in a single piece."

This review aims to provide a comprehensive document that presents the possibilities for applying plasma nitriding as a surface treatment to offshore materials, ensuring the critical requirement against H_2S attack and chloride corrosion, and enhancing the tribological performance of components in severe friction and wear conditions.

The manuscript was organized to respond to three questions:

- What is the effect of different techniques of plasma nitriding on the surface properties and performances?
- What is the effect of different substrates on the surface properties and performances?
- What is the effect of different bodies on the tribological performances?

All issues belong to the scenario of the Program of Excellence in Surface Engineering with emphasis on Plasma-Assisted Treatments,9 a network established among four universities – Universidade Federal do Paraná, Universidade Tecnológica Federal do Paraná, Universidade Estadual de Ponta Grossa, and Pontifícia Universidade Católica do Paraná – to shed light on some industrial problems and amplify the employment of plasma nitriding.

ACTIVE SCREEN PLASMA NITRIDING

The active screen technique for nitriding (ASPN) appeared as an alternative to the conventional direct current (DC) plasma system (glow discharge). Essentially, a metal screen assumes the role of the cathode, while the workpieces float together on a working table. In both techniques, the furnace wall is the anode. Li[10] summarized the potential of ASPN in overcoming characteristic issues of the DC system: parts to be treated must be fully electrically conductive; potential of surface damage caused by arcing; non-uniform nitriding due to edge effect; non-uniform nitriding due to temperature variation; potential for overheating owing to the hollow cathode effect; and difficulties in batch treatment of a large mumber of small components. In the same overview, three factors were considered in ASPN: i) active screen material; ii) distance between screen and component; and iii) bias. Throughout this review, some strategies to increase the layer thickness will be described based on these factors.

A deep investigation into the mechanisms for nitriding using ASPN was conducted by Corujeira Gallo and Dong.[11] They studied the hardening of different regions below the screen and verified a crucial effect depending on each case. Therefore, the transferability of material from the mesh onto the treated specimens plays a significant role in hardening the nitrided surface. In this way, using a series of arrangements that included gas pressure (at a low level) and bias (at a low level), they achieved a layer thickness equivalent to that of the DC process, but with better surface quality.[12]

Apart from the processing, the key role lies in the microstructure obtained along with the nitrided layer. Most of the CRA materials indicated in the previous section are mainly composed of austenite (gamma phase). With the introduction of nitrogen from the surface, the reticulation of austenite expands, creating a new phase known as the S-phase, which is the expanded austenite.[13] It appears as a bright white layer over the austenitic substrate, indicating its good corrosion resistance. The process technique has an intimate relation to producing layers of S-phase. Dong reported that "DCPN (DC plasma nitriding) may tend to produce a non-uniform S-phase case because of the non-uniform electric discharge phenomenon in DC plasma." To overcome this issue, pulsed DC or radio frequency (RF) plasma can be applied; however, ASPN can produce a more homogeneous expanded austenite surface layer by eliminating or reducing edge effects.

To represent a significant micrograph illustrating the S-phase, one from the kinetic study performed by Lima et al.[14] appears to be representative. However, it was obtained using the DC plasma technique (Fig. 1). Besides the top layer, it is worth noting the remarkable effect of etching in revealing the austenite and ferrite constituents of super duplex stainless steel.

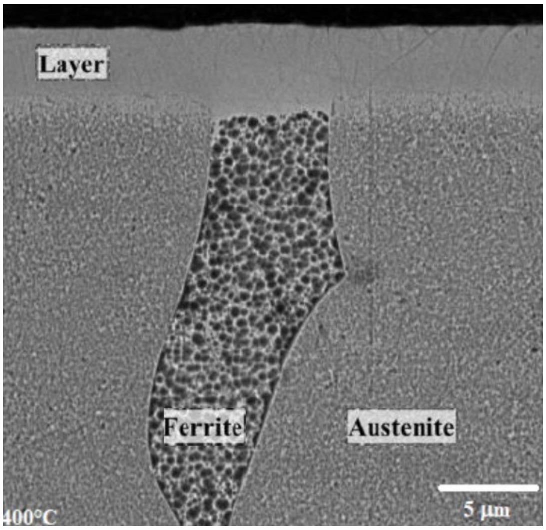

Figure 1: Cross-sectional view of low-temperature nitrided super duplex stainless steel, revealing the top layer of S-phase and the corresponding constituents of austenite and ferrite. Treatments were carried out for 4 h at 400 °C.

Source: Adapted from Lima et al.[14]

Plasma nitriding as an effective barrier against hydrogen embrittlement

There is a significant amount of evidence that the nitriding treatment is an effective barrier against hydrogen embrittlement. As a first result, Michler[15] applied pulsed plasma nitriding on austenitic stainless steel. The process produced a 3-layered structure, with the γ/γC (original austenite/austenite expanded by carbon) layer serving as the intermediate layer between a γ_N (austenite expanded by nitrogen or S-phase) top layer and a diffusion layer. Surprisingly, since this is not a predicted microconstituent, the intermediate layer exhibited ductile behavior under severe plastic deformation. Following a tensile test in gaseous hydrogen, no cracks were observed in the γ/γ_C-layer regions.

A different approach was noted in the works of Asgari et al.[16,17] These researchers developed an *in-situ* hydrogen charging instrument that permeates the hydrogen during nanoindentation cycles. For austenitic stainless steel[16] or duplex stainless steel,[17] the nitrided layer can soften in the presence of hydrogen. An interesting result is that the removal of hydrogen reversed the softening effect, turning nitriding into a promising way to control the embrittlement of stainless steels.

More recently, we can observe the application of hydrogen permeation in various plasma nitriding techniques. Kurelo et al.[18] used plasma immersion ion implantation (PIII) nitriding, while Braceras and Bautista[19] used ASPN technology. Concerning the PIII nitriding, the processing temperature had a significant effect on hydrogen embrittlement. In the PIII 300 °C case, the hydrogen attack was less severe than in the untreated sample. On the other hand, on the PIII 400 °C surface, after hydrogenation, cracking, delamination, and detachment of the modified layer were observed. Braceras and Bautista[19] also observed a temperature effect on the hydrogen permeation of a nitrided ferritic steel, but this effect was strongly dependent on the sample thickness, even though a significant permeation reduction factor of 210 times was achieved using ASPN.

All described studies presented promising possibilities to block the hydrogen damage on offshore components using plasma nitriding, but the resistance of a nitrided layer against sulfide stress cracking was confirmed by Coseglio et al.[20] These researchers modified the surface of 17-4 PH stainless steel (H1150D condition) by processing at 420 °C (low-temperature plasma nitriding) or 500 °C (high-temperature plasma nitriding [HTPN]) for 10 h using the active screen technique. No sulfide stress cracks were observed on the specimens following exposure (720 h) to synthetic produced water saturated with 3.5 kPa partial pressure of H_2S (CO_2 as balance gas) and pH 4.5 after HTPN processing (Fig. 2).[21] Two factors could explain the success of plasma nitriding: i) the barrier effect of the plasma nitride layer – previously observed in different hydrogen-charging systems (as described herein and even in other studies compiled in Coseglio[22]); and ii) the compressive residual stress induced after the treatment.

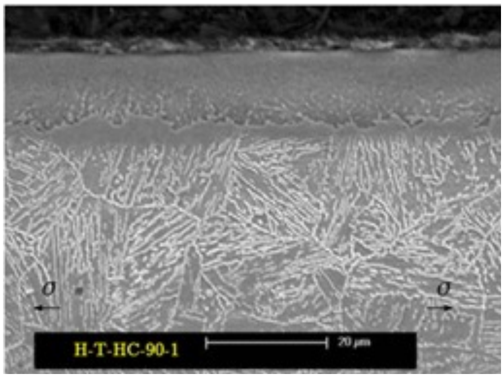

Figure 2: Cross-sectional view of active screen plasma nitrided PH 17-4 stainless steel loaded to 90% of yield stress after SSC tensile in pH 4.5. Absolutely no cracks are noted.

Source: Adapted from Coseglio.[21]

Nitriding of offshore materials

An experimental study that allows for building a summary of the CRAs subject to thermo-chemical treatment (nitrocarburizing) was performed by Singh and Marya.[23] They applied a salt-bath nitrocarburizing treatment, using 580 °C for 120 minutes, to five stainless steels (13Cr, S13Cr, 17-4PH, 304, and 316 grades) and four nickel-based alloys (925, 935, 718, and 625Plus grades). Fig. 3 shows the variation in the compound layer thickness as a function of pitting resistance equivalence number (PREN).

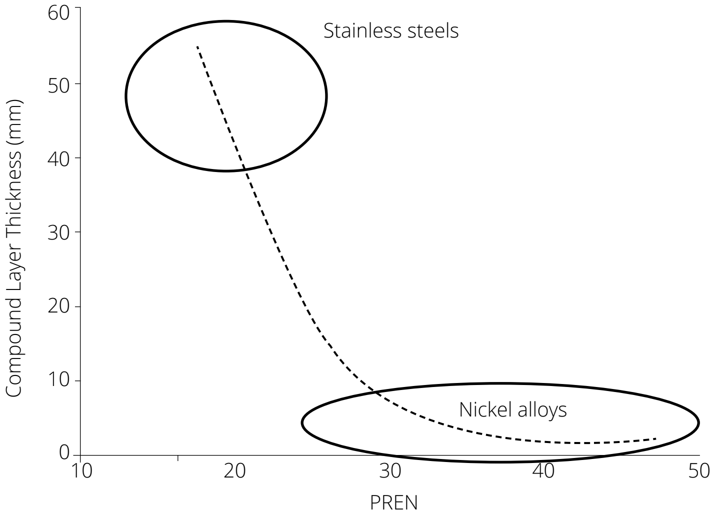

Figure 3: Variation of nitrocarburized layer thickness for offshore materials as a function of PREN.

Source: Adapted from Singh and Marya.[23]

An expected result was found by Singh and Marya[23]: the higher the PREN, the thinner the layer. For the same processing variables, diffusion becomes more complex with the addition of more alloying elements. As some nickel alloys are classified as superalloys,[24] increasing the compound layer is a significantly more challenging task. Besides, the effect of liquid nitrocarburizing on corrosion resistance was beneficial for stainless steels but detrimental to nickel alloys. It is possible that the effect of a thin layer on wear would be the same, i.e., thinner layers could be less effective against the wear process.[25]

Calabokis et al.[26] reviewed the application of nitriding to Inconel 718 alloy. Looking only at the temperature effect, they identified some investigations that report surpassing the 10 μm layer determined by Singh and Marya[23] using the nitrocarburizing process. In the case of liquid nitriding, it appears relatively easy to form thicker layers; in contrast, using plasma techniques, it is not trivial to find many investigations that achieve the same.

One notable case is that of Maniee et al.,2[7] who utilized a hot wall for nitriding. The chamber wall temperature was set to 400 °C in all studied conditions. Among all nitriding temperatures, 450 °C was considered to promote a balance between wear and corrosion resistance for a layer of 11 μm. The increase in temperature can precipitate detrimental constituents that affect corrosion performance; therefore, it is crucial to investigate how different plasma techniques can achieve a proper balance between thickness and microstructure.

It was this question that Kurelo et al.[28] focused on. These researchers compared two surface modification techniques, glow discharge plasma nitriding (GDPN) and cathodic cage plasma nitriding (CCPN, equivalent to ASPN), in terms of the mechanical and tribological behavior of layers produced on AISI 316 stainless steel. They tested at relatively low temperatures (350, 400, and 450 °C for 6 h), aiming to prevent the formation of precipitates that can affect corrosion resistance. In all cases, the thickness of modified layers was smaller using the CCPN process; depending on the temperature, GDPN promotes approximately double or even six times greater layers. These results directly affected the scratch resistance of the layers.

Investigating the same substrate as Kurelo et al.[28] – AISI 316 stainless steel – Sato et al.[29] used the active screen for 20 h at 400 °C, achieving layers with a thickness of 6 μm. Upon scratching the surface, a critical load of 6 N was determined, indicating that increasing layer thickness can enhance the abrasive wear resistance of the expanded austenite.

To surpass the low thickening, Hamashima and Nishimoto[30] proposed modifying the material of the screen. While Kurelo et al.28 manufactured the screen using AISI 1008 steel, the later researchers compared the results obtained using a nickel screen. Unfortunately, for the current purposes, Hamashima and Nishimoto30 only performed corrosion tests; they did not address any wear issues. Although it is essential to describe their findings here, primarily because they measured the hardness, they applied the technique to three stainless steels: austenitic stainless steel (SUS304), ferritic stainless steel (SUS430), and duplex stainless steel (SUS329J4L). The use of the Ni screen increased the thickness of the nitrided layer from 2.1 to 3.6 μm for SUS304 steel. For duplex steel, the change in thickness depended on the constituent. Considering the gamma phase, it was 2.5 μm when using a steel screen, and with Ni, it turned to 3.4 μm. In ferrite, the change was from 2.9 to 3.7 μm thick.

As a result of the higher nitrogen concentration at the surface using the Ni screen, a higher hardness was determined for this technique. In addition, the researchers identified a non-homogeneous hardness distribution after the steel screen was used, which was not observed in the case of Ni application. Exemplifying the hardness increase, SUS304 after nitriding with a steel screen exhibited a hardness of ~ 750 $HV_{0.005}$, which increased to ~ 1100 $HV_{0.005}$ for the Ni case.

The diversity of commercially available duplex stainless steels raises a question about the differences in how each grade behaves during nitriding. Even with the challenge in increasing the layer thickness using the active screen technique, the results delivered by Kurelo et al.[28] showed that a balance of the required performances for offshore applications can be achieved using ASPN instead of glow techniques.

To address this issue, Pintaude et al.[31] tested two grades of duplex stainless steels (SAF 2205 and SAF 2507) using scratch tests after applying ASPN. The chosen temperature was 380 °C to avoid any precipitation during the treatment. To achieve a reasonable thickness, 10 h of processing were necessary. Homogeneous thin layers of 1.48 ± 0.14 μm for SAF 2507 and 1.62 ± 0.26 μm for SAF 2205 were created.

It is important to note that SAF 2507 steel is harder than SAF 2205; the former has a hardness of 429 $HV_{0.025}$, while the latter has a hardness of 333 $HV_{0.025}$. This difference was relevant in providing support for the scratching resistance of the formed nitrided layer. After nitriding, the layer of SAF 2507 remained harder; however, it was also important to check for a significant difference in microconstituent hardness compared to SAF 2205 steel. For this steel, the nitrided austenite was harder than the ferrite. Therefore, beyond the bearing support provided by a harder substrate in the case of SAF 2507 steel, the heterogeneous distribution of the hardness layer is also a reasonable reason for poorer performance during scratch tests, as softer regions were put in contact with the diamond tip. All factors resulted in a higher frequency of surface cracking of SAF 2205 steel substrate compared to the SAF 2507 after the scratching test, as shown in Fig. 4.

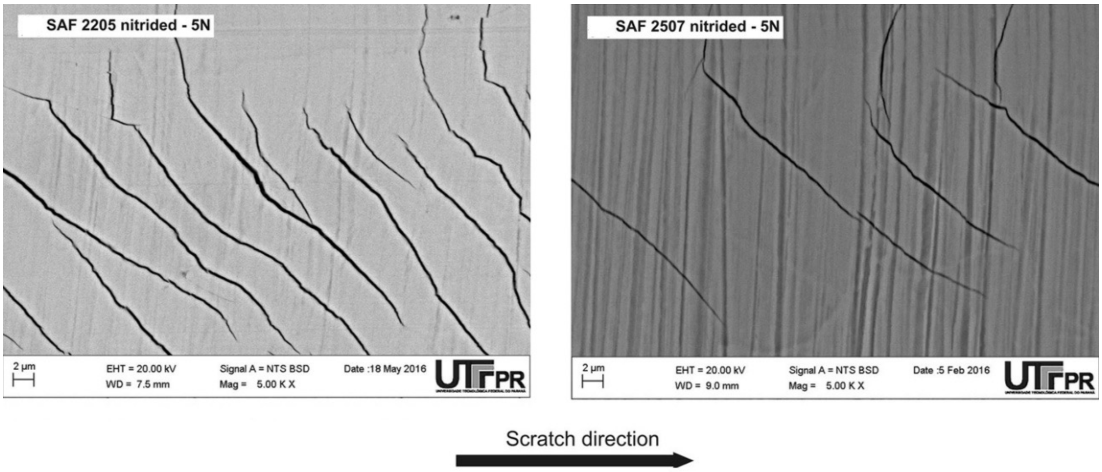

Figure 4: Crack patterns after scratching tests performed over S-phase layers on SAF 2205 and SAF 2507 steels using a 5 N applied load.

Source: Adapted from Pintaude et al.[31]

The evaluation of the critical load during the scratching test was also performed by Nuñez de la Rosa et al.[32] for nitrided Inconel 718, using the glow discharge technique. The nitriding temperature was the same as that of two other investigations[32,33] – 400 °C – allowing for valid comparisons, considering this alloy an important CRA, primarily because different techniques were used: a triode plasma system (used to deposit physical vapor deposition [PVD] films) and ASPN. The layer thickness and hardness, as well as the processing times, are presented in Table 1. Another common aspect of all investigations was the determination of corrosion rates under a saline solution.

Table 1: Layer thicknesses and hardness resulting from different plasma nitriding techniques applied to Inconel 718 alloy at 400 °C.

Ref.	Nitriding technique	Thickness (mm)	Hardness
32	Glow discharge (4 h)	7.17 ± 0.89	12 GPa*
33	Triode (PVD system) (4 h)	1.80 ± 0.10	No data
33	Triode (PVD system) (20 h)	4.20 ± 0.20	1,560 $HV_{0.01}$
34	Active screen (20 h)	4.43	1,200 ± 100 $HV_{0.005}$ /12.1 ± 0.5 GPa*

Source: Elaborated by the authors. *Via nanoindentation.

An interesting aspect when comparing the investigations performed under DC and ASPN systems is that the Inconel substrate was extracted from the same original bars, and they had the same initial hardness (530 HV).

From Table 1, a thicker layer was formed using the DC process compared to the others. Using a processing time five times shorter, the DC layer was about 40% thicker. However, this increase did not translate to an overall improvement in the performance of nitrided Inconel. The nanohardness was equivalent to that determined for the ASPN layer, for example. Regarding the critical load during scratch tests, Nuñez de la Rosa et al.[32] verified cracks from a 3 N load in a progressive test conducted up to 8 N. Using the same range of loads, Oikava[35] verified a crack only at a 3-mm track, which corresponds to a load of 4.48 N (Fig. 5). This critical load is equivalent to Lc1, using the proper nomenclature for scratch tests.[36] Therefore, the increase in thickness via the DC technique cannot assure a higher critical load, as expected considering substrate deformation.

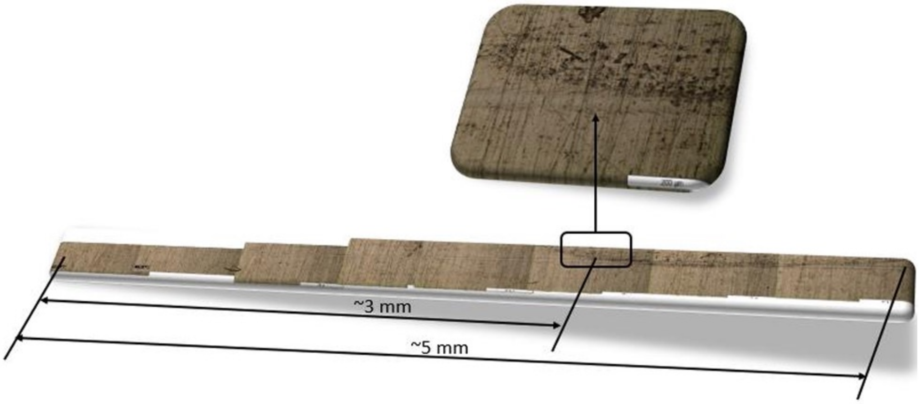

Figure 5: Evolution of scratched track of ASPN Inconel 718 using progressive load (2-8 N).

Source: Adapted from Oikava.[35]

A more recent study on the scratch resistance of nitrided Inconel 718 was conducted by Luis-Pantoja et al.,[37] using liquid nitriding. They produced a two-layer system consisting of CrN (with a thickness of 7.6 ± 0.5 µm) and an expanded austenite, called S-phase (with a thickness of 1.31 ± 0.1 µm), resulting in 18.2 ± 0.2 GPa of hardness. The critical load corresponding to Lc1 was 4.3 ± 0.3 N, close to the value reported in Oikava et al.[34] They reported much higher loads up to Lc4, but this result would not be significant considering the corrosion performances that CrN would achieve. Otherwise, liquid thermochemical processes are much more polluting than ASPN.

Regarding the corrosion performance reported by the studies comprised in Table 1, Nuñez de la Rosa et al.[32] observed traces of CrN precipitates, even using a relatively low temperature. On the other hand, there is no evidence of precipitation using ASPN.[34] This difference impacted the corrosion rates. Using the DC system, a value of 2.55 ± 0.12 (mm/year) × 10^{-4} was determined, whereas after ASPN, the corrosion rate was 1.49 ± 0.08 (mm/year) × 10^{-4}. This comparison is necessary to affirm categorically that for offshore components, any trace of precipitation is unacceptable, since the possibility of hydrogen embrittlement would be more dangerous than any damage by saline corrosion.

The completeness of the investigation conducted by Oikava et al.[34] was due to the reciprocating wear tests, conducted by varying the counterbodies. They applied, under the same testing conditions, different pairs in saline contact: i) AISI 52100 ball × Inconel 718; ii) AISI 52100 ball × nitrided Inconel 718; iii) silicon nitride × Inconel 718; and iv) silicon nitride × nitrided Inconel 718. Different contacts resulted in different wear mechanisms, leading to varying wear rates. These systems can represent real ones in terms of different levels of mechanical deformation. Apart from the improved performances obtained with the ASPN layer, its presence can also enhance tribological performance, depending on a systematic analysis of the contact. It was very clear from this investigation that a nil contribution of nitriding occurred when a ceramic was used as the counterbody. On the other hand, when a deformable body is present (quenched and tempered steel), the ASPN process makes a crucial difference in the wear rates, as it changes the wear mechanism from adhesion/oxidation to scoring (Fig. 6).

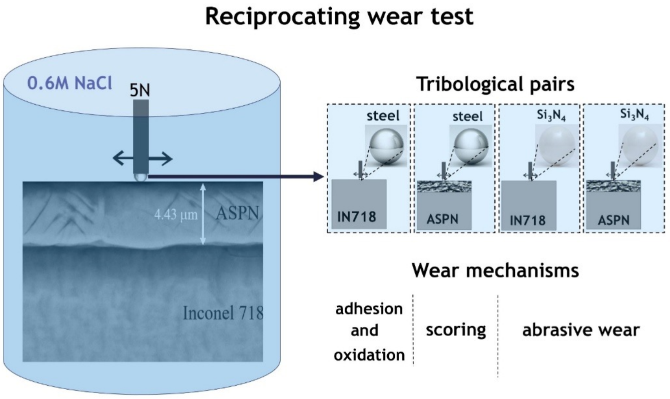

Figure 6: Summary of wear mechanisms varying the counterbodies under the reciprocating test of Inconel 718.

Source: Adapted from Oikava et al.[34]

Although it was not concerned with the ASPN technique, a tribological investigation about nitrided layers can help to complete the understanding of the findings summarized in Fig. 6. Berton et al.[38] applied solution heat treatment after plasma nitriding (SHTPN) to AISI 409 steel using the DC technique. They produced 10 different nitrided layers, including a single nitrided layer (not post-heat-treated). All conditions and the original substrate were tested in a reciprocating system against AISI 52100 steel balls under PAO8 base oil lubrication. Excluding the non-post-treated nitrided layer, the others contemplate a gradient of properties from the surface to the bulk, with a much smoother hardness profile. With this characteristic, the layers are less susceptible to the eggshell effect, avoiding cracking during mechanical contact. On the other hand, post-treatment can soften the initial nitrided layer, resulting in a probable lower resistance.

Most of the SHTPN conditions resulted in hardness closer to the bearing ball (Fig. 7), and the tribological system produced mild abrasion as the primary mechanism of wear. However, the untreated nitrided layer, the hardest tested condition, performed similarly to another that was twice as soft. The ineffectiveness in increasing the hardness of worn material repeats the same situation observed by Oikava et al.,[34] when abrasion was the dominant wear mechanism.

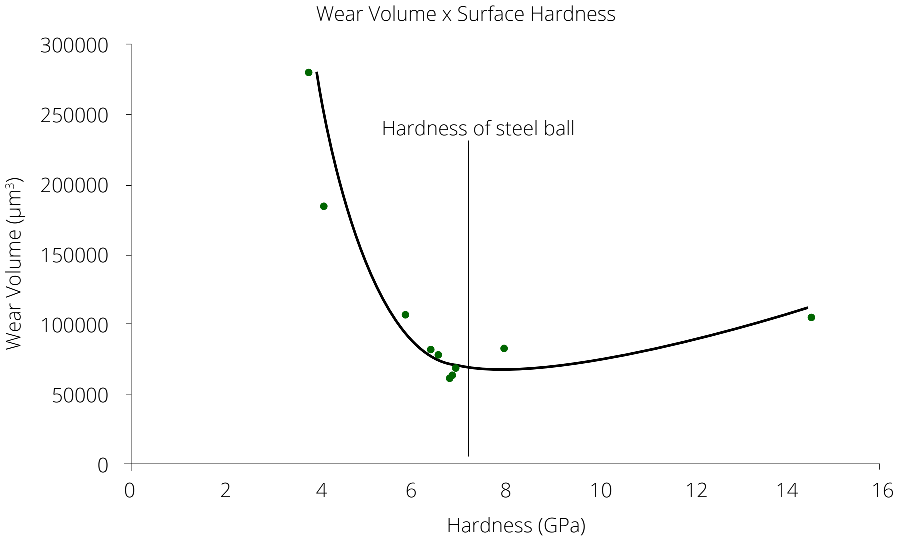

Figure 7: Wear rates as a function of the hardness of nitrided AISI 409 martensitic steel after reciprocating tests. The steel hardness of the ball counterface is indicated.

Source: Adapted from Berton et al.[38]

The remaining question in terms of microstructure/properties is: how to produce hard surfaces with good tribological performance? Logically, this answer depends on the tribological system; however, we can observe some impressive hardness values after applying some plasma nitriding techniques. An expressive result was reported by Kurelo et al.,[39] who found a value of around 15 GPa after PIII nitriding for a SAF 2507 duplex stainless steel. This substrate was identical to that used by Pintaude et al.,[31] with a hardness value of 429 HV. The limiting hardness for Inconel 718 alloy (35 HRC) is higher than SAF 2507 stainless steel (25 HRC) for sulfide stress cracking following NACE/ISO recommendations,[5] therefore, an application of the PIII technique for achieving the same level of hardness that was reported in Schibicheski Kurelo et al.[39] would create an interesting perspective for replacing some current surface engineering solutions used in the offshore industry.[40] The bearing support provided by Inconel is expected to be higher than that provided by duplex stainless steel.

Considering the active screen technology, the investigations conducted by Gao et al.[41,42] reveal that for the Ti-6Al-4V alloy, adjusting the bias voltage and the screen height makes it possible to achieve hardness of the same order of magnitude as described by Schibicheski Kurelo et al.[39] Therefore, nitrided layers can reach or surpass the hardness of hard coatings with compositions that serve only tribological purposes, without blocking hydrogen action and chloride corrosion simultaneously.[43]

CONCLUDING REMARKS AND POTENTIAL FUTURE INVESTIGATIONS

CRAs susceptible to hydrogen embrittlement in offshore applications can be enhanced with plasma nitriding when the component is subjected to wear processes. Processing temperatures that promote the precipitation of constituents, which hinder corrosion performance, are prohibitive. Then, the challenge is to achieve a sufficient thickness to prevent substrate deformation during the wear process, as higher temperatures can increase the thickness but also promote deleterious constituents.

The active screen technique has several advantages over other plasma techniques. In some cases, applying the same variables of the DC technique, for example, is not sufficient to obtain a robust layer against wear. For these cases, the literature showed some strategies, such as modifying the screen material, bias voltage, and screen height. The adjustment of processing variables enables the achievement of a hardness equivalent to that of hard coatings used solely for tribological purposes. However, nitriding can simultaneously block hydrogen action and chloride corrosion.

Future possibilities include the additive manufacturing (AM) of CRAs. Some investigations have studied the effect of plasma nitriding on materials manufactured with laser powder bed fusion (LPBF).[44,45] Compared to the traditional process, LPBF produced a harder CRA, enabling the substrate to serve as a more effective bearing support for mechanical loads under the nitrided layer, in line with the results observed by Pintaude et al.[30] However, in the case of AM technology, there is no necessity to use a more expensive material to achieve a high level of hardness.

CONFLICT OF INTEREST

Nothing to declare.

DECLARATION OF USE OF INTELLIGENCE ARTIFICIAL TOOLS

The author declares the use of Grammarly software to revise the English.

AVAILABILITY OF DATA AND MATERIALS

All dataset were generated or analyzed in the current study.

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