18th IFHTSE CONGRESS
International Federation for Heat Treatment and Surface Engineering
IFHTSE
ANAIS
PROCEEDINGS
ISSN 1516-392X

INFLUENCE OF SURFACE HARDENING DEPTH ON THE CAVITATION EROSION RESISTANCE OF A LOW ALLOY STEEL[1]

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Abstract

In this paper, the influence of surface hardening depth promoted by plasma nitriding and Cr-Al-N coating deposition on the cavitation erosion resistance of a low alloy steel was investigated. 2 and 4 hours plasma nitrided samples were produced and coated with 1 and 2 μm Cr-Al-N coatings deposited by PAPVD. The characterization was carried out by X-ray diffraction (θ-2θ and glancing angle configurations), scanning electron microscopy, Rockwell C adhesion test and 3D profilometry. Knoop microhardness tests were also performed. Cavitation erosion tests were carried out according to ASTM G32-03 Standard. The cavitation erosion rate and incubation period were determined. Coating deposition had a major influence on the incubation period, in which a higher coating thickness resulted in a longer time. Plasma nitriding treatment was more effective on reducing the average erosion rate in the accelerated period. The plasma nitriding treatment and Cr-Al-N coating in conjunction led to a decrease in both incubation period and erosion rate. The hardened systems presented mass loss up to 11 times lower than the non hardened steel for the same time. It was concluded that as ticker is the coating and as deeper is the nitrided layer better is the cavitation erosion resistance.

Key-words: Cavitation erosion; Plasma nitriding; PAPVD coating.

INFLUÊNCIA DA PROFUNDIDADE DE ENDURECIMENTO SUPERFICIAL NA RESISTÊNCIA A EROSÃO CAVITACIONAL DE UM AÇO BAIXA LIGA

Resumo

Neste trabalho, a influência da profundidade de endurecimento superficial devido à nitretação a plasma e à deposição de filme Cr-Al-N na resistência à cavitação de um aço baixa liga foi investigada. Foram produzidas amostras nitretadas a plasma por 2 e 4 horas e recobertas por PAPVD com filme de Cr-Al-N de 1 e 2 μm. A caracterização foi realizada por DRX, MEV, teste de adesão Rockwell C, perfilometria 3D e microdureza Knoop. Os testes de erosão cavitacional foram realizados de acordo com a norma ASTM G32-03. A taxa de erosão cavitacional e o tempo de incubação foram determinados. A deposição do filme teve maior influência no tempo de incubação, sendo que uma maior espessura resultou em um menor tempo. A nitretação a plasma foi mais efetiva na redução da taxa de erosão no período acelerado. A aplicação conjunta de nitretação a plasma e recobrimento Cr-Al-N levou a redução do tempo de incubação e da taxa de erosão. Os sistemas endurecidos apresentaram perda de massa até 11 vezes menor que o aço não tratado para o mesmo tempo. Concluiu-se que quanto mais espesso o recobrimento e profunda a camada nitretada melhor a resistência a erosão cavitacional.

Palavras-chave: Erosão cavitacional; Nitretação a plasma; Recobrimento Cr-Al-N.

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1 *Technical contribution to the 18th IFHTSE Congress – International Federation for Heat Treatment and Surface Engineering, 2010 July 26-30th, Rio de Janeiro, RJ, Brazil.*
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1 INTRODUCTION

Bubbles or cavities may nucleate and grow within a liquid body when the local pressure at a certain point in the liquid is reduced below its vapour pressure. This phenomenon is called cavitation. Materials can be damaged when the bubbles collapse on the surface, due to a localized increase in pressure, producing shock waves, high velocity jets of liquid and localised increase in temperature. Depending on the nature of the material, plastic deformation and mass loss can be observed. The removal of material caused by cavitation is called cavitation erosion. Cavitation erosion can be observed in a large number of hydraulic components, such as, valves, propellers, pumps, turbine blades, agitators and pipelines.[1-4]

Cavitation erosion damage can be reduced by making good mechanical design and material selection. Usually, materials with high work hardening rate, high hardness (with good elastic properties) and small number of defects are expected to have good cavitation erosion resistance.[1-3] Surface engineering techniques, such as, plasma nitriding and physical vapour deposition (PVD) of coatings, can be applied to produce surfaces more resistant to cavitation erosion. Plasma nitriding is known to be effective to improve wear, corrosion and fatigue resistance of metals. Huang et al.[1] studied the cavitation behaviour of ion-nitrided carbon steel in fresh water. The authors observed that plasma nitriding reduced the steel cavitation rate. It was attributed to the high hardness of the nitrided layer and the solid solution strengthening of the diffusion layer.

Münsterer and Kohlhof[5] investigated the cavitation protection promoted by titanium-based PVD coatings deposited on ball-bearing steel substrate. The authors observed that surface topography such as, roughness and defects in film structure, influenced the cavitation behaviour and that cavitation erosion behaviour can be correlated with physical properties of the coatings such as, stoichiometry, hardness, stress and structure. Han et al.[6] studied the cavitation erosion behaviour of Cr-N and Cr coated AISI 4140 steel in fresh water. They found that the cavitation erosion resistance was improved by the coating deposition, the mass loss of the coated steels were about one third of the uncoated one after 60 minutes. However, a smooth topography and a good adhesion were necessary for the coatings to exhibit good resistance.

Krella and Czyżniewski[3] investigated the cavitation erosion resistance of Cr-N coating deposited on austenitic stainless steel. They observed the incubation period of Cr-N coating was 50% longer compared with the uncoated steel and the erosion rate of coated steel was higher than the uncoated steel. The authors have attributed the higher mass loss to the combination of cavitation erosion and solid particles erosion caused by hard particles torn off during cavitation. The longer incubation period of Cr-N coated steel was associated to the high coating hardness and good adhesion. Krella and Czyżniewski[7] investigated the influence of stainless steels substrate hardness on the cavitation erosion of TiN coating. The authors observed that coating deposition increased the incubation period. The highest prolongation occurred for the coating deposited on substrate with higher hardness, which also had the better adhesion. It was noted that with increase of the substrate hardness the adhesion increases as well. However, the authors have not found a straight correlation between substrate hardness or/and adhesion and the incubation period. According to Krella and Czyżniewski[7] the mechanism of cavitation resistance of coated materials is more complicated due to the mismatch of mechanical properties and the coating adhesion to the substrate.

Godoy et al.[8] evaluated the improvement in the cavitation erosion resistance promoted by PAPVD coating and plasma nitriding on AISI 1045 steel. The authors observed that plasma nitriding treatment, whether or not combined with PAPVD coating, increased the incubation period and decreased the cavitation erosion rates. Although plasma nitriding and PVD coatings have been applied to improve cavitation erosion resistance of metals, the cavitation erosion behaviour of Cr-Al-N coatings and of plasma nitrided layers with different depths, applied on low alloy steel alone or in conjunction, were not investigated yet. In this work, the influence of surface hardening depth, promoted by plasma nitriding and Cr-Al-N coating deposition, on the cavitation erosion resistance of a low alloy steel were investigated.

2 MATERIAL AND METHODS

The substrate was made of ground and polished AISI 4140 steel (nominal composition (wt.%): 0.38 – 0.43% C; 0.8 – 1.1% Cr, 0.75 - 1% Mn; 0.15 – 0.25% Mo, 0.03% max P; 0.040% max S; 0.15 – 0.3% Si). The plasma nitriding treatment was implemented at 500°C for 2 and 4 hours using a triode glow discharge having 60% Ar + 40% N_2 gas composition, -250 V bias voltage and 4 x 10^{-3} mbar pressure. The nominal 1 µm and 2 µm Cr-Al-N coatings were deposited by electron beam plasma-assisted physical vapour deposition (EBPAPVD), at 405°C for 100 minutes. Before the coating deposition, the substrate was sputtering cleaned with argon for 5 minutes and a Cr layer was deposited for 5 minutes. Combining plasma nitriding and coating deposition, eight systems were produced with different hardening depths. The systems produced can be assembled in 4 groups: AISI 4140 steel substrate (N0F0, reference system), plasma nitrided systems (N2F0, N4F0), coated systems (N0F1, N0F2) and duplex systems (N2F1, N2F2, N4F1, N4F2), summarized in Table 1.

Table 1. Systems description

Plasma nitriding	*Cr-Al-N Coating*		
	Uncoated (F0)	1 µm (F1)	2 µm (F2)
Non nitrided (N0)	N0F0	N0F1	N0F2
2 h (N2)	N2F0	N2F1	N2F2
4 h (N4)	N4F0	N4F1	N4F2

The crystalline structure of the systems was analysed by X-ray diffraction (XRD) using Cu-Kα radiation (λ = 0.154056 nm) in θ-2θ and glancing angle (GAXRD) configurations. The GAXRD was used to assess the top layer crystalline structure, i.e. the Cr-Al-N coating and the plasma nitrided layer phases, without the influence of substrate. It was used a 2.5° angle for GAXRD. The surface morphology was investigated by scanning electron microscopy (SEM). The coating thickness was assessed by a ball crater test (Calotest). Coating adhesion was evaluated using the Rockwell C Adhesion Test; it was performed in a hardness tester with a 1471 N (150 kgf) load. The indentation marks were observed by SEM. The crack patterns were compared with the patterns described by Heinke et al.[9]

Godoy et al.[10] questioned which hardness should be measured when one wish to associate the property with the cavitation erosion performance. The authors concluded that macro and microhardness were more suitable to evaluate materials subjected to cavitation erosion. Considering that microhardness measurements were used in this study. The hardness profiles were assessed by Knoop hardness measurements on the systems surface, with applied load varying from 0.49 to 10 N. The plasma nitrided layer depth was determined using Knoop hardness measurements (0.49 N) on the cross-section of the N2F0 and N4F0 systems. The Cr-Al-N coating hardness was assessed by instrumented hardness test, using a Berkovich indenter and 50 mN load.

3D topographic surface measurements were conducted on a Hommelwerke T4000 stylus-based profilometer, using a TKU 300 pick-up (stylus tip radius: 5 μm, cone angle: 90°). The sampling area was 8 x 8 mm. The sampling interval and scanning speed were established at 160 μm and 0.50 mm/s respectively. A Gaussian filter with a cut-off length of 0.8 mm was used in all measurements. Amplitude parameters obtained from 3D roughness profile were used to describe the surface texture, before cavitation erosion tests. The following amplitude parameters were used: *Sa* (average roughness, i.e., arithmetic mean deviation of the surface from the mean plane), *Sq* (root-mean-square deviation of the surface), *Sv* (maximum valley depth), *Sp* (maximum peak height) and *St* (maximum amplitude, equal to *Sv* + *Sp*).[11,12]

The vibratory cavitation erosion resistance was evaluated according to the ASTM G32-03 Standard.[13] During the test, the sample was attached to a sonotrode, immersed in distilled water and subjected to a vibratory frequency of 20 kHz and amplitude of 45 μm. The test temperature was set at 25 ± 2°C and controlled by a thermostat. The sample weight was periodically assessed to measure the mass loss. The test duration was 1200 minutes, except for N0F0 and N0F1 systems (600 minutes). Cumulative mass loss plots were obtained as a function of cavitation erosion time.

3 RESULTS AND DISCUSSION

The α-Fe was the only phase related to AISI 4140 steel substrate identified in all systems. For the plasma nitrided substrates (N2 and N4 systems), the α-Fe lattice parameter was slightly bigger (0.2892 nm) than that calculated to N0F0 system (0.2872 nm), due to the presence of nitrogen atoms in interstitial solid solution into the body centred cubic (*bcc*) lattice. The plasma nitrided substrates also presented phases related to the formation of iron nitrides: hexagonal compact (*hcp*) ε-$Fe_{2-3}N$ and face centred cubic (*fcc*) γ'-Fe_4N. The Cr-Al-N coatings (F1 and F2) presented the *fcc*-CrAlN (prototype B1-NaCl) phase.

The GAXRD patterns for plasma nitrided, coated and duplex systems were assessed using an incident angle of 2.5°. The maximum x-ray penetration depth was estimated in 2 μm. The plasma nitrided systems presented a very similar patterns, indicating that at both systems have the same crystalline phases, identified as ε-$Fe_{2-3}N$ and γ'-Fe_4N. For the coated systems, the N0F1 system presented the *fcc*-CrAlN and a peak that was related to α-Cr phase. The presence of Cr can be explained by the deposition of a Cr layer between the steel substrate and the coatings and its detection was due the thinner thickness of F1 film. The N0F2 system presented only the peaks related to the *fcc*-CrAlN phase. For the duplex systems, the ε-$Fe_{2-3}N$ and *fcc*-CrAlN phases were identified, suggesting that the ε-$Fe_{2-3}N$ is the more external phase of the nitrided layer.

The cross-section microstructures for all systems are shown in Fig. 1. For the plasma nitrided systems, it was observed the formation of a compound layer, the compact layer on the top of the surface, and the presence of needles of nitrides growing towards the sample centre. Both the compound layer thickness and the needles size increased with the increasing of plasma nitriding treatment duration. Below the compound layer, there is a diffusion layer, where nitrogen atoms are in solid solution. The nitrided layer depth for N2 and N4 systems were estimated in 90 µm and 126 µm, respectively. The criteria used to determine the nitrided layer depth was a hardness value 10% higher than the AISI 4140 steel hardness. The nitrided layer depth increased with the increase of treatment time in a non-linear basis. The N0F0, N0F1 and N0F2 systems presented similar steel microstructure, suggesting that the PAPVD process has not modified the AISI 4140 steel substrate. The F2 coating was apparently twice as thick as F1 coating in the photomicrography. The coating thickness measured using ball crater tests were 0.85 µm and 1.70 µm for F1 and F2 films respectively. For the duplex systems, it was observed a compound layer and needles of nitrides, which were also observed in the plasma nitrided steel, and the Cr-Al-N coatings on the top of the systems.

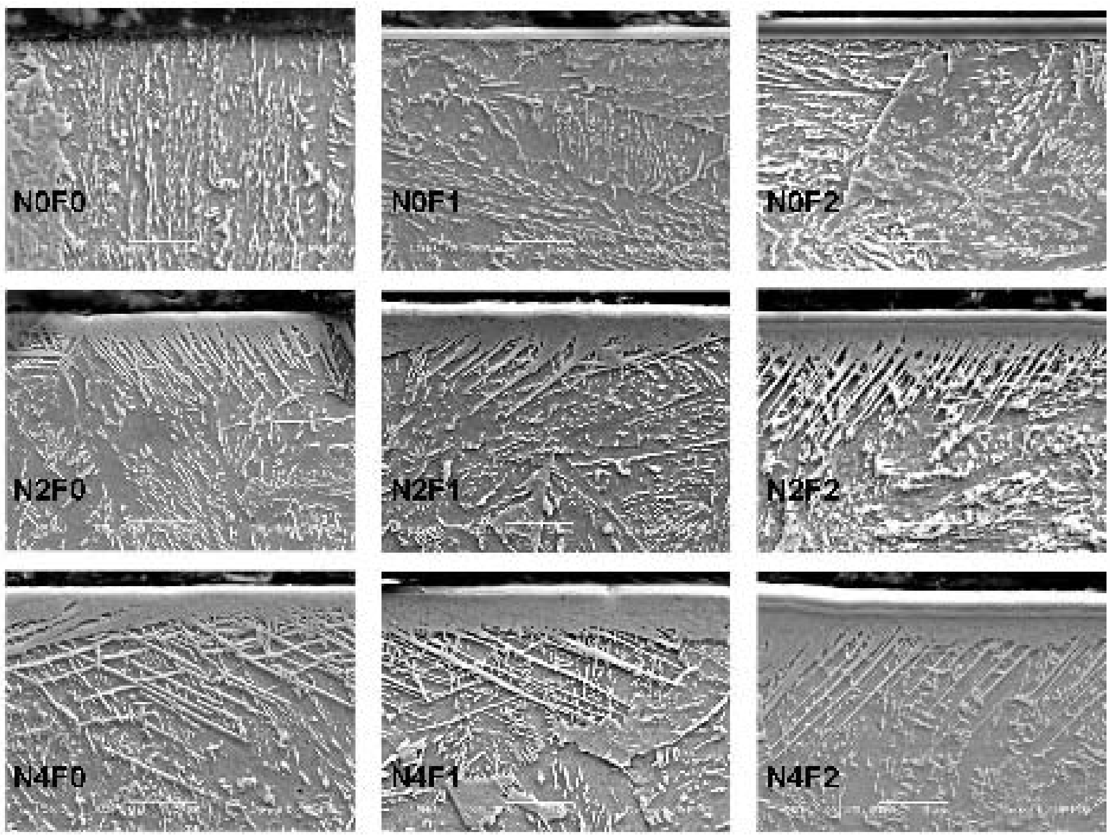
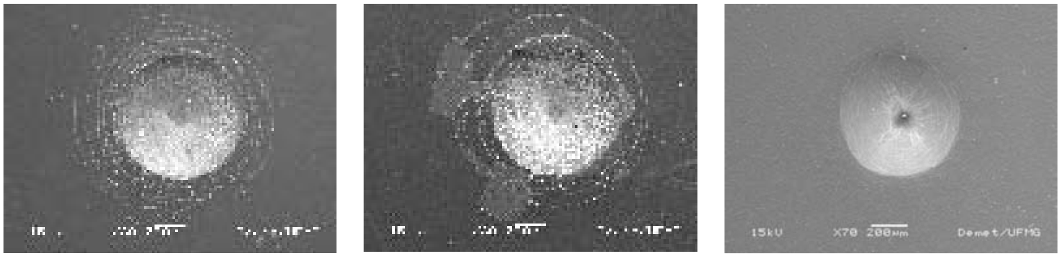


Figure 1. Cross-section microstructures for all systems.

The Rockwell C indentations marks on the surface of Cr-Al-N coated and duplex systems were analysed using SEM (Fig. 2). The N0F1 system presented several circular cracks and small regions with coating delamination, indicating good adhesion (Fig. 2a). The N0F2 system presented circular cracks and large regions with delamination, indicating poor adhesion (Fig. 2b). EDS analysis carried out in the delaminated region detected a large amount of Cr, suggesting that the adhesive failure occurred at the interface between Cr-Al-N coating and the Cr interlayer. All the duplex systems presented very few radial cracks, indicating a very good adhesion (Fig. 2c).

ISSN 1516-392X



(a) (b) (c)

Figure 2. Rockwell C adhesion test indentation marks of the (a) N0F1 showing circular cracks, (b) N0F2 showing circular cracks and delamination (bright grey areas) and (c) duplex systems showing radial cracks.

The Knoop hardness (KH) profiles measured at the systems surface, with applied load varying from 0.49 to 9.8 N, are shown in Fig. 3. The given results are based on an average of 5 measurements. The KH profiles can be divided in two groups: the first one presenting lower hardness values, comprising the systems with non plasma nitrided substrate (N0 systems); and the second one with higher hardness, being the plasma nitrided systems (N2 and N4 systems). For the N0F0, N0F1 and N0F2 systems, it was observed that the high Cr-Al-N coating hardness (~30 GPa) and the coating thickness have influenced the superficial hardness for applied loads lower than 1.9 N. For higher loads, the N0 systems hardness values were very similar to the AISI 4140 steel hardness (382 KH). For the N2F0 and N4F0 systems, it was observed that the increase in the plasma nitriding treatment time led to an increase in the superficial hardness for all applied loads, which can be attributed to the thicker compound layer and the deeper diffusion zone of the N4F0 system. The duplex systems presented the highest KH values for all loads. For loads up to 1.9 N, as thicker was the coating as higher was the KH measured. For higher loads, the N2 and N4 systems hardness presented close values, suggesting a major contribution of the nitrided layer hardness. The results indicated the surface hardness incorporate contributions from both coating and substrate hardness, showing that substrate/coating systems surface hardness depends on the individual properties of each component like hardness and thickness as well as the load applied.

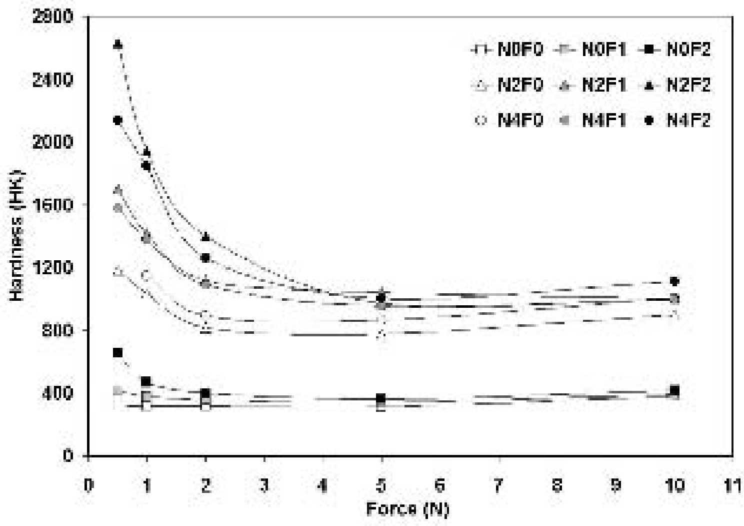
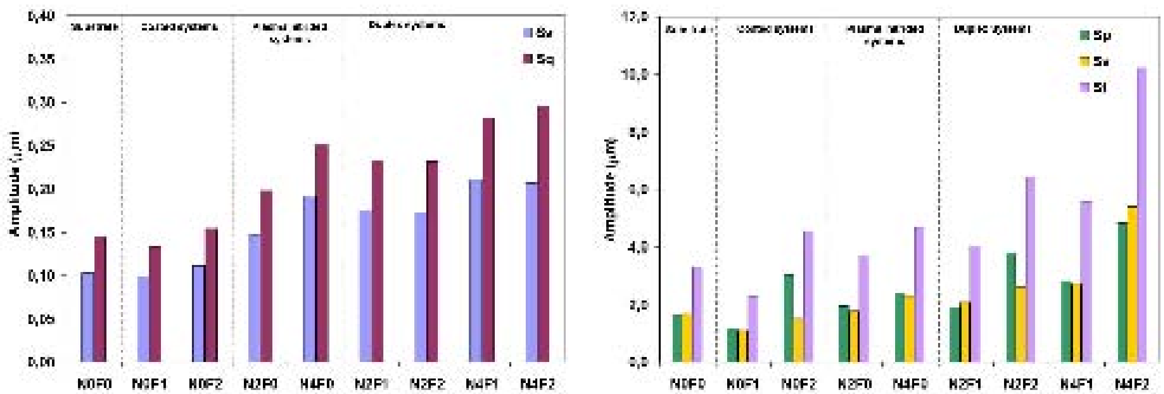


Figure 3. Surface Knoop hardness profile for all systems.

The amplitude parameters assessed by 3D profilometry for all systems is shown in Fig. 4. The surfaces were characterized before the cavitation erosion tests. Comparing with the reference system (N0F0), the Cr-Al-N coating deposition has not significantly modified the average roughness parameters *Sa* and *Sq* of the N0F1 and N0F2 systems (Fig. 4a). However, for the extreme roughness parameters (Fig. 4b), it was observed that the F1 film deposition flattened the profile, decreasing the N0F1 system total amplitude (*St*). On the other hand, the F2 film deposition led to an increase of the maximum peak height (*Sp*) and, consequently, an increase in the total amplitude of the N0F2 system. The plasma nitriding treatment led to a remarkable increase in the surface roughness. The *Sa* and *Sq* parameters increased approximately 40% and 85% for the 2 and 4 hours treatment time, respectively. The *Sv*, *Sp* and *St* parameters also increased with the increase of plasma nitriding time. The duplex systems presented the highest surface roughness parameters values; this was expected, once both surface engineering techniques applied promoted changes in the surface topography. In this study, the increase of plasma nitriding time and of Cr-Al-N coating thickness resulted in higher 3D amplitude roughness parameters. The cavitation erosion behaviour is highly influenced by the surface topography, because the implosion of bubbles is promoted at edges (asperities) of the surface.[5]



(a) (b)

Figure 4. Amplitude parameters (a) *Sa*, *Sq* and (b) *Sp*, *Sv* and *St* before the cavitation test extracted from the 3D roughness profile for all systems.

Typically, plots of cumulative mass loss as a function of the cavitation exposure time exhibit two stages. In the first stage, called incubation period, the erosion rate is zero or negligible when compared to the erosion rate of the second stage (accelerated period).[13] In this study, the criterion used to determine the incubation time was a mass loss equal to 0.40 mg, since the criterion described in the ASTM G32-03 standard was not suitable for the data set. The plots of cumulative mass loss versus cavitation time are shown in Fig. 5. The incubation time and erosion rates for all systems are summarized in Table 2. The AISI 4140 steel (N0F0) presented incubation period equal to 60 minutes and the highest erosion rate in the accelerated period (0.094 mg/min). For the coated systems, the incubation time increased with the increase of the coating thickness, being 120 min for N0F1 system and 210 min for N0F2 system. Otherwise, N0F1 and N0F2 systems erosion rates in the accelerated period (0.081 mg/min) were close to the one presented by N0F0 system. For the plasma nitrided systems, the incubation time did not varied significantly comparing with N0F0 system, but the erosion rate in the accelerated period were much lower, 0.013 mg/min and 0.009 mg/min for N2F0 and N4F0 systems respectively. The increase in the plasma nitriding treatment time resulted in a significant decrease in the erosion rate. For the duplex systems it was observed a slight increase in the incubation period (up to 90 min for N4F2 system), as well as a significant decrease in the erosion rate (up to 0.007 mg/min for N4F2 system).

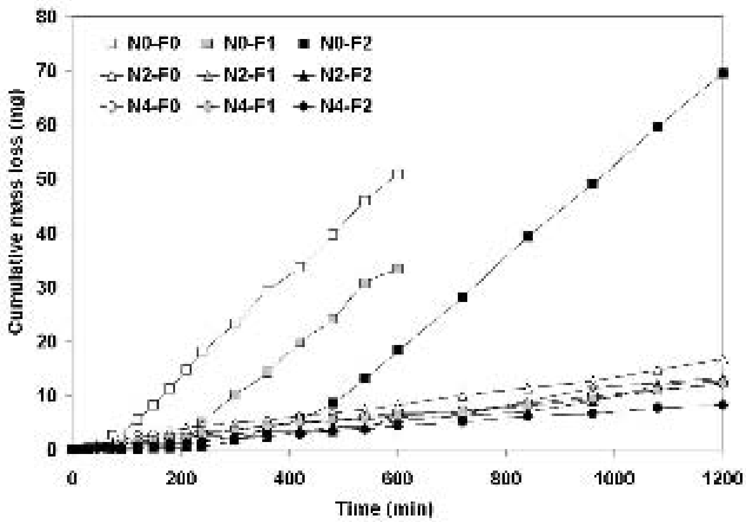


Figure 5. Cumulative mass loss during cavitation erosion test for all systems.

Table 2. Incubation period, erosion rate in the accelerated period and normalized erosion resistance for all systems

Systems	*Incubation period (min)*	*Erosion rate (mg/min)*	*Normalized erosion resistance*
N0F0	60	0.094	0.52
N0F1	120	0.081	0.61
N0F2	210	0.081	0.61
N2F0	50	0.013	3.80
N4F0	45	0.009	5.50
N2F1	80	0.013	3.80
N2F2	35	0.015	3.28
N4F1	60	0.009	5.50
N4F2	90	0.007	7.04
Stellite 6	-	-	3.80
308 SS*	-	-	1.00**

* SS: stainless steel; **Reference material.

Comparing all systems at 600 minutes cavitation time, the mass loss of the N0F1 and N0F2 systems were 1.5 and 2.8 times smaller than that of the reference system (N0F0). And the incubation time was 2 times longer for the N0F1 system and 3.5 times longer for N0F2 system. The poor adhesion observed for F2 coating (Fig. 2) seemed to have not influenced significantly the N0F2 performance. The plasma nitrided systems presented mass loss 6-7 times smaller and the duplex systems 8-11 times smaller than the AISI 4140 steel. The higher roughness observed for the plasma nitrided systems (Fig. 4) have contributed to the short incubation time observed, once rough surfaces present more sites to bubble collapse. Basically, the increase in incubation time can be attributed to the Cr-Al-N coating deposition and the erosion rate increase related to the surface hardening promoted by plasma nitriding.

The cavitation erosion resistance of the systems was compared with the resistance of two materials commonly used in applications that require good cavitation erosion resistance, namely, Stellite 6 alloy and 308 stainless steel (SS). The normalized erosion resistance for all systems, Stellite 6 alloy and 308 SS are shown in Table 2. The normalized cavitation erosion resistance was calculated using the method described by Mancosu[14] that takes in account the erosion rate and the material density, considering the 308 SS as reference. The N0F0, N0F1 and N0F2 systems presented normalized resistances inferior to the 308 SS one, suggesting that both the AISI 4140 steel and the Cr-Al-N coated systems would not have a good performance in the real application. Otherwise, all systems with plasma nitrided substrates (N2 and N4 systems) presented superior normalized resistances, even comparing with the Stellite 6 alloy, which is considered one of the best materials to resist cavitation erosion.

The Knoop hardness profiles presented a good correlation with the cavitation erosion behaviour of all systems. The systems with lower hardness values were the ones with bigger mass losses in the cavitation erosion tests and vice-versa. It was also observed that systems with a deeper superficial hardening, i.e., with a thicker coating and a deeper plasma nitride layer showed the best performances. In this study, the mass losses were found to be inversely proportional to the surface hardening depth. However, the role of plasma nitriding treatment was more prominent than that of the PAPVD Cr-Al-N coating deposition in the cavitation erosion resistance improvement. Apparently, the surface roughness has not significantly influenced the cavitation erosion resistance, or its effect was much less intense than the surface hardening effect, once the rougher systems (N2 and N4 systems) presented the higher cavitation erosion resistances.

4 CONCLUSIONS

The Cr-Al-N coating deposition improved the cavitation erosion resistance of the AISI 4140 low alloy steel. The coating deposition greatly influenced on the increase of incubation time and promoted a small reduction in erosion rate in the accelerated period. The increase in the coating thickness led to an increase in the incubation period. The plasma nitriding treatment also improved the cavitation erosion resistance of the AISI 4140 steel. Plasma nitriding promoted a deep surface hardening that greatly affected the erosion rate in the accelerated period. The increase in plasma nitriding treatment time and, consequently, the increase in the surface hardening depth, led to a decrease in the cavitation erosion rate. The increase in the surface roughness caused by plasma nitriding treatment has not significantly influenced the cavitation erosion resistance. The use of plasma surface engineering techniques in a low alloy steel promoted a reduction of mass loss up to 11 times for a 600 minutes cavitation erosion test. The 4 hours plasma nitrided systems (uncoated and coated) presented normalized cavitation erosion resistance superior to that of the Stellite 6 alloy.

ISSN 1516-392X

Acknowledgements

The authors are greatly indebted to CNPq - Conselho Nacional de Desenvolvimento Tecnológico, FAPEMIG - Fundação de Amparo à Pesquisa de Minas Gerais, Brazil, for the financial support.

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