

# Study the Fatigue-Wear Behavior of a Plasma Electrolytic Nitrocarburized (PEN/C) 316L Stainless Steel

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In the present study, a suitable machine was developed in the laboratory to investigate the fatigue-wear behavior of the untreated 316L austenitic stainless steel and samples treated by plasma electrolytic nitrocarburizing process under different combinations of cyclic loading and contact pressure. The fracture cycles as a function of bending stress were recorded while a constant contact pressure was applied simultaneously. As a result, the PEN/C treated specimens exhibited a higher resistance (about 40% for 15.6 N contact load and about 60% for 25 N contact load) under the application of simultaneous cyclic stress and contact pressure. Also it was shown that under a range of combined fatigue and wear stresses, the specimens exhibit a better life than the conditions of performing wear or fatigue tests separately and this effect was much more observable for PEN/C-treated samples. The gravimetric weight loss values in the fatigue-wear test were also measured at intervals 5000 to 300,000 cycles (with the contact stress = 6.25 MPa and the bending stress = 87 MPa). The results showed a better wear resistance for the treated surface at the first stage of the process.

**Keywords** 316L stainless steel, contact pressure, cyclic stress, fatigue-wear, PEN/C treatment

## 1. Introduction

The problems of friction and fatigue-wear are very important in many industrial applications. Complex fatigue-wear damages emerge in systems which operate in the conditions of the contact interaction between elements (sliding, rolling, slippage, etc.) and simultaneously transmit cyclic workload. The shear stresses that initiate failure under these conditions reach a maximum value at some distance below the surface and develop cracks at subsurface microstructural discontinuities such as oxide inclusion particles (Ref 1). In order to improve the tribological performance of structural materials, such as stainless steels, a number of surface treatments such as nitriding by ion implantation, pulsed plasma nitriding, and plasma immersion ion implantation have been developed (Ref 2).

It has been pointed out (Ref 3, 4) that the improvement in fatigue properties induced by a hard PVD coating can be related to the higher mechanical strength of the coating with respect to the substrate and to the presence of residual compressive stresses within the coatings and the excellent adhesion of the films to the substrate. All these factors, especially the presence of compressive residual stresses in the coating, contribute to arrest the propagation on the cracks within the coating and

delay their transference to the substrate. Tokaji et al. (Ref 5) studied the effect of a modified gas-carburizing technique on the fatigue behavior of a 316 stainless steel, both in air and 3%NaCl aqueous solution. The fatigue strength in air of the carburized material was improved considerably compared with the untreated material and the carburizing process increased the fatigue strength by 30%. In the corrosive environment the fatigue strength of the untreated material decreased significantly, whereas no reduction of fatigue strength was observed for the carburized steel.

Modification of fatigue behavior of austenitic stainless steel also has been studied by other surface engineering techniques such as shot peening (Ref 6), laser (Ref 7), dynamic ion mixing (Ref 8), and coating (Ref 9).

In addition to those techniques, a modified plasma electrolytic technique has been developed (Ref 10) in which the coating exhibits excellent adhesion with the substrate and the deposition rate is significantly higher than that of the conventional electrolytic processes. It offers similar if not better surface morphology at a significantly lower cost as compared to the existing technologies (Ref 11).

The scope for industrial use of this technology was also quite promising due to the process flexibility, low capital cost, and environmentally friendly precursor materials utilized (Ref 12).

In the present investigation, an optimized plasma electrolyte bath (Ref 13) was used to treat the surface of 316L stainless steel fatigue specimens and its effect on the fatigue-wear properties was studied. The fatigue-wear tests were carried out with a cantilever-type rotary bending fatigue machine accompanying with different combinations of contact pressure applying wear load.

The resulting quantitative characteristics of resistance to fatigue wear damage may be useful for quality control of materials and developing materials with specified mechanical and physical behavior.

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## 2. Experimental Procedure

### 2.1 The Plasma Electrolytic Nitrocarburizing (PEN/C) Process

The technique of PEN/C is traditionally based on the diffusional saturation of a metal surface by ionized species of typically nitrogen and/or carbon during high-voltage electrolysis of suitable organic electrolytes (Ref 14).

In the present investigation an optimized electrolyte bath based on urea containing  $\text{NH}_4\text{Cl}$ ,  $\text{Na}_2\text{CO}_3$ , and other additives, mainly to control the bath electrical conductivity (Ref 13), was used to treat the surface of 316L stainless steel fatigue specimens. Table 1 lists the chemical composition of the steel substrate.

### 2.2 The Fatigue-Wear Testing Machine

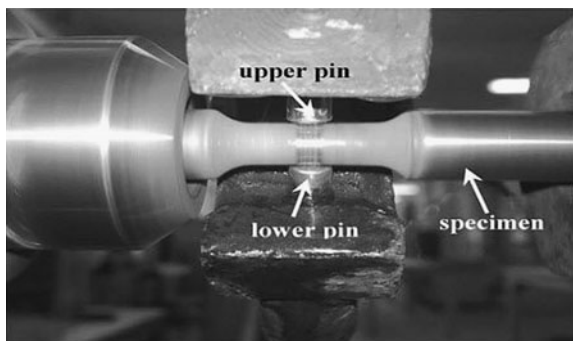
A suitable machine for fatigue-wear tests was developed in the laboratory to investigate the fatigue-wear behavior of untreated 316L austenitic stainless steel and the PEN/C-treated samples (Fig. 1). One end of a cylindrical specimen was fixed in a spindle and was allowed to rotate at the angular speed  $\omega_1$ . A vertical bending load  $Q$  (downwards) was imposed to its other end. A cylindrical pin acting as the non-rotating counterspecimen was pressed against the working zone ( $d_{\text{ave}} = 5 \text{ mm}$ ) by a contact load  $F_N$ . As shown in Fig. 1, the normal load  $F_N$  was applied by the aid of a lever arm. Thus using the lever rule, the required value of  $F_N$  can be adjusted by putting necessary weights on the corresponding load pan.

Continuous droplets of a Ringer's solution (acting as a corrosive medium and also as a lubricant) were applied on the surface of the specimen during the tests to prevent the temperature rise of the contact region. In the proposed model for fatigue-wear experimentations, the scheme of tests was configured according to Fig. 2. Concerning this scheme, it was possible to perform fatigue-wear tests and vary the values of  $F_N$  and  $Q$ .

During the tests the contact load  $F_N$  was static, i.e., constant in time. In these tests, the dynamic conditions of interactions

**Table 1** The chemical composition (wt.%) of 316L stainless steel

C	Mn	Cr	Mo	Ni	S	Si	P	Fe
0.03	2	16.75	2.69	10.27	0.04	0.63	0.45	Balance



**Fig. 1** (a) The fatigue-wear machine developed, (b) schematic configuration of the fatigue wear testing machine

between the specimen and counterspecimen can be characterized by two parameters: values of the cyclic stress as a function of the bending load  $Q$  and the mean pressure as a function of the contact load  $F_N$ . In this machine, the specimen was rotated by a DC motor with a frequency of 50 Hz. The slider stands statically in position, which simulates the sliding friction.

## 3. Specimen Preparation

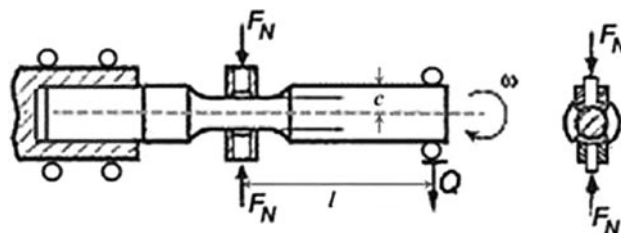
The following pieces were prepared for the tests:

- Standard (ASTM E466) cylindrical fatigue specimens from the untreated 316L stainless steel. A number of these samples were PEN/C-treated (Ref 13) for fatigue-wear tests (Fig. 3).
- Cylindrical pins with a 5 mm diameter from SAE 52100 steel as counterspecimen. Table 2 lists the chemical composition of 52100 steel.

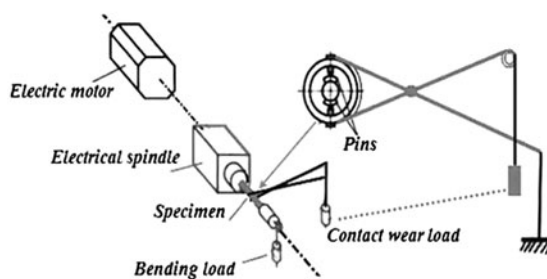
The fatigue-wear tests were performed using different combinations of contact loads (responsible for wear) and fatigue bending stresses.

## 4. Results and Discussion

Fatigue-wear tests were performed on AISI 316L stainless steel and PEN/C-treated specimens, for experimental assessment of combined effects of the processes of friction and mechanical fatigue on these samples. A representative scanning electron micrograph of surface morphology and the cross section microstructure of the PEN/C-treated AISI 316L austenitic stainless steel is reported in Fig. 4 (Ref 13). High



**Fig. 2** The scheme of fatigue-wear laboratory tests



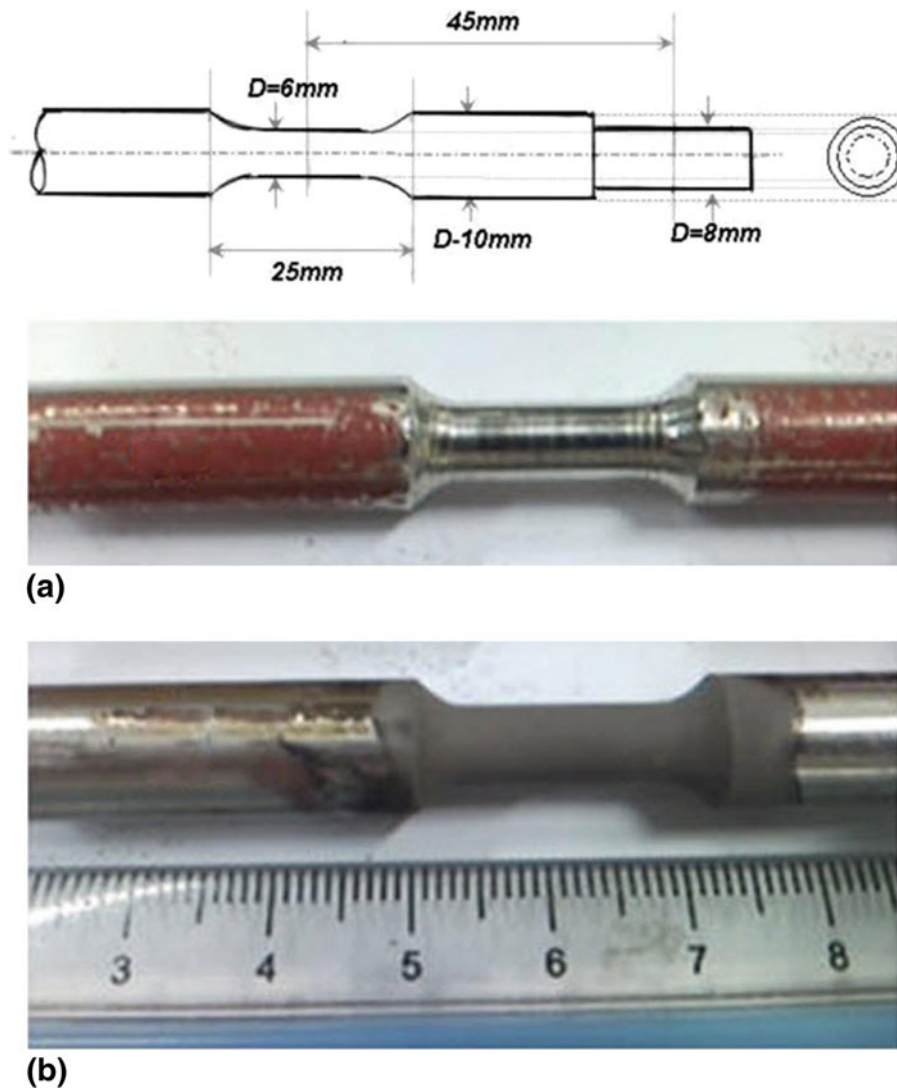


Fig. 3 The standard fatigue specimens (a) untreated sample and (b) PEN/C-treated sample

Table 2 Chemical composition (wt.%) of steel pins

C	Mn	P	S	Si	Cr	Fe
1.1	0.35	0.02	0.02	0.3	1.54	Balance

temperatures in plasma bubbles lead to localized melting of the surface layer of the specimen. After collapse of the plasma bubble, this surface is quenched by the surrounding electrolyte, leading to a unique surface microstructure (Ref 10). The following tests were carried out on the untreated and treated samples according to the scheme configured in Fig. 2.

#### 4.1 Constant Normal Load ( $F_N$ ) and Varying Vertical Bending Load ( $Q$ )

Figure 5 shows the relation between the cyclic fatigue stress and the number of cycles up to fracture. A contact load ( $F_N = 15.6$  N and 25 N) was statically applied at the middle of the cylindrical specimen during the tests to induce the wear friction process. The data in Fig. 5 indicate that the fatigue-wear

resistance of the treated samples is higher than that of the substrate and the average difference is about 40 and 65% as the test was carried out under contact loads of 15.6 and 25 N, respectively. This increase in fatigue strength could be the result of the production of a load supporting diffusion sublayer on the surface due to the PEN/C process (Ref 14) which leads to a very high surface hardness in the case depth (Ref 13, 14). This can suppress slip deformation and therefore delays the crack nucleation. Compressive residual stresses may also be induced by the PEN/C treatment in the diffusion layer and increase the surface hardness and strength. Tokaji et al. (Ref 5) measured compressive residual stresses of about 1500 MPa on a low temperature carburized 316 stainless steel.

As shown in Fig. 5 the ultimate stress during fatigue-wear tests is higher than the ultimate stress during common fatigue tests ( $F_N = 0$ ) within a relatively broad range of variations of fatigue bending stresses and this is much more observable for the treated steels than for untreated steels. The fatigue limit of the specimens during common fatigue tests is shown by the horizontal dashed line.

When the field of contact pressure superimpose on the bending stress field, a large part of the applied energy is going



to dissipate in the surface layer of the material and cause the localization of the wear process and cracking in the surface layer. Due to the high hardness and unique surface morphology of the PEN/C-treated surface, the deformation energy preferably tends to flatten the spheroids, fill the craters, and shattering the grain fragments into finer particles than to allow the crack penetrates into the depth of the specimen. This may therefore cause the delay or even the prevention (depending on the conditions of loading) of the formation of large pits which are the main sources of stress concentration. Under pure wear condition when there was no contribution of fatigue stress, the crack propagation accelerated even at lower contact pressures and sudden fracture occurred.

It has been proved (Ref 15) that friction and wear are the phenomena capable of mechano-physico-chemical interactions. It is the result of these interactions that complex fatigue-wear damage of the material occurs.

#### 4.2 Constant Bending Stress ( $\sigma$ ) and Varying Normal Contact Load ( $F_N$ )

The fatigue-wear tests were carried out on treated surfaces with constant cyclic stresses of 500 and 180 MPa. To obtain each point of the corresponding  $F-N_f$  curve, the test was repeated under different values of contact wear loads applied at the middle of the specimen.

A similar abnormal behavior was found for the sample tested under the cyclic stress of 180 MPa. The corresponding curve in Fig. 6 indicates the positive contribution of the wear load with cyclic fatigue stress to cause fracture, during a relatively broad range of applied contact pressure. In this case, the characteristics of friction and wear have been changed under the effect of the fatigue processes. In other words, the fatigue limit of the specimen can either grow or fall or remain unchanged in response to the conditions of fatigue-wear tests and origin of contacting materials (Ref 15).

The samples tested under cyclic stress of 500 MPa did not fail at the working cross section but fracture occurred at the end of the specimen. In fact, due to the higher cyclic bending stress, crack initiation, and propagation accelerated at the surface of the specimen in this region, leading to the failure of the working sample at the ending cross section. At the same time, in the early stage of wear, the compressive stress produced by contact pressure on the working surface might have prevented crack initiation. The fatigue curve in this condition behaved like common fatigue curves. The SEM micrographs of the fractured cross sections for a pure mechanical fatigue tested specimen

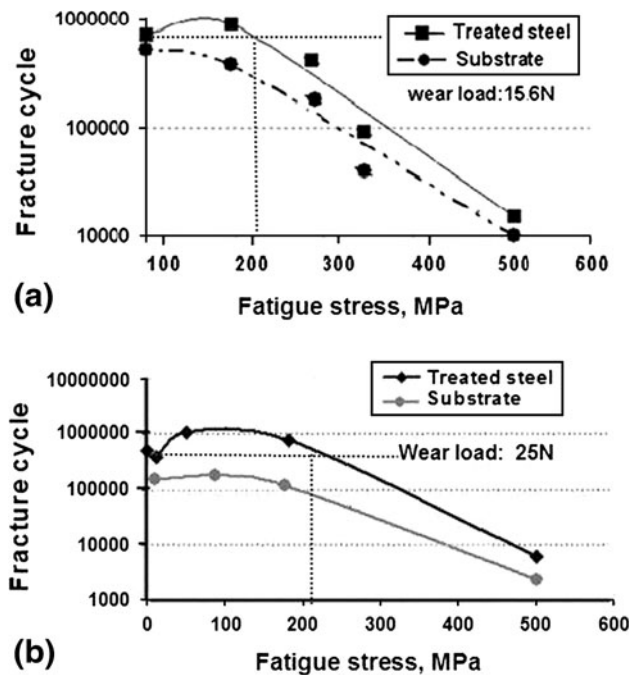


Fig. 5 The results of fatigue-wear tests for the substrate and treated steel against AISI 52100 steel pins: (a)  $F_N = 15.6$  N and (b)  $F_N = 25$  N

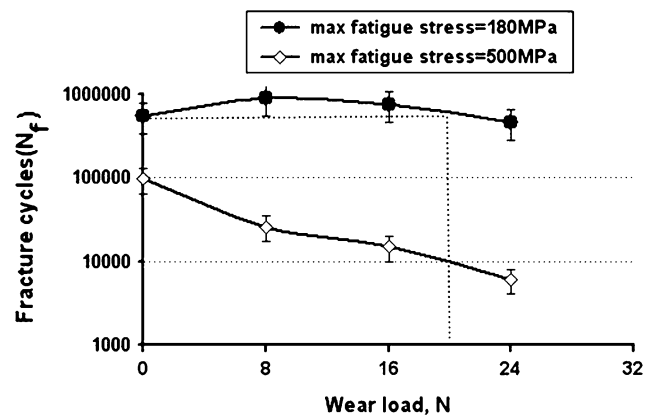


Fig. 6 The results of fatigue-wear tests on the PEN/C-treated samples (constant bending fatigue stress,  $\sigma_{max}$ )

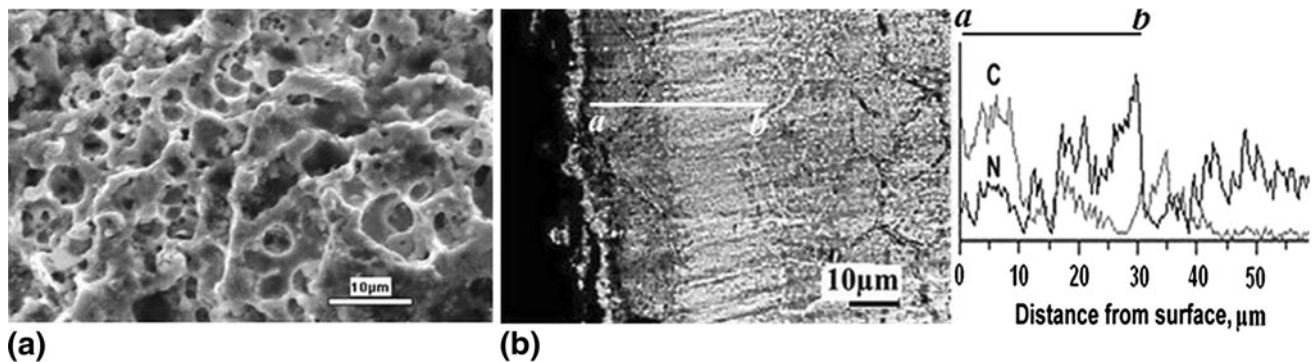
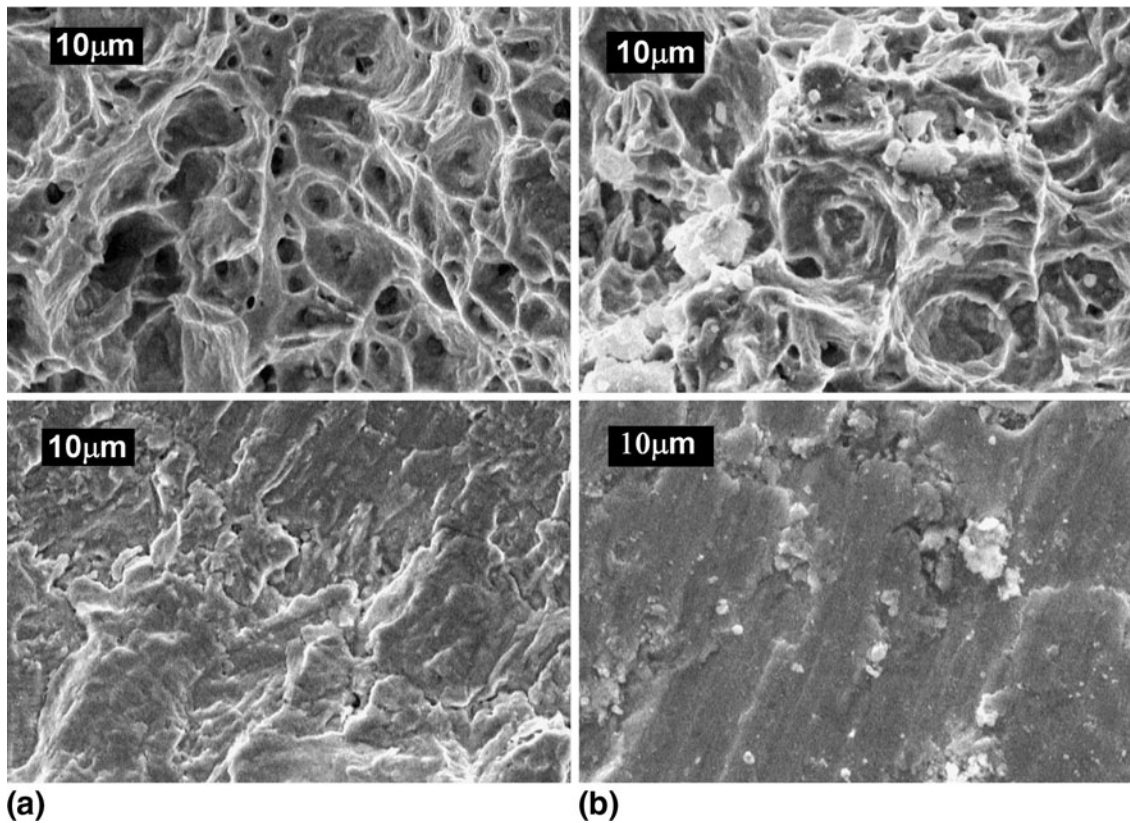


Fig. 4 The surface morphology (a) and the cross section microstructure (b) of the PEN/C-treated AISI 316L austenitic stainless steel



**Fig. 7** SEM micrographs of the fractured cross sections for (a) a pure fatigue tested sample,  $\sigma_{\max} = 180$  MPa and (b) a fatigue-wear tested sample,  $\sigma_{\max} = 180$  MPa and  $F_N = 5$  N

and a fatigue-wear tested one are shown in Fig. 7. The deformed shapes of the dimples and lines of crack propagation in Fig. 7(b) could indicate the effect of the applied wear load.

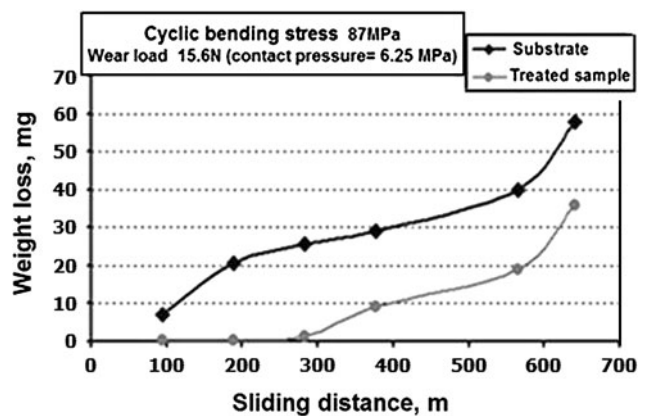
## 5. Wear Rate Measurement

In the present investigation, the weight loss in the fatigue wear test was measured gravimetrically at intervals of 5000 cycles, up to 300,000 cycles while the normal wear load ( $F_N = 15.6$  N or  $P = 6.25$  MPa) and fatigue bending stress ( $\sigma = 87$  MPa) were applied on the system at the same time. The experimental results are depicted in Fig. 8.

For the treated surface, the wear rate at the first period was very low. The weight loss in this region (up to 16,000 cycles) was significantly lower than that of the substrate. This could be due to the high wear resistance of surface layers produced by PEN/C treatment. Although the whole wear rate diagram for the treated sample was seen to be below that of the substrate, the wear rate for both treated and untreated samples was approximately the same, after passing through the treated depth.

## 6. Conclusions

In the present study, fatigue-wear tests were performed using 316L stainless steel and plasma electrolytic nitrocarburized samples and the effects of PEN/C treatment on fatigue-wear strength were discussed. The main conclusions can be made as follows:



**Fig. 8** The weight loss versus sliding distance for untreated and PEN/C-treated samples

The PEN/C treatment enhances the fatigue strength of the 316L steel with respect to the untreated steels due to the special surface morphology and the high surface hardness in the case depth.

In these types of fatigue wear tests, the induced surface plastic deformations due to the superposition of compressive and bending stresses harden the working portion of the specimen. Also the favorable residual compressive stresses in the surface layer may postpone or even prevent the propagation of primary fatigue cracks.

The weight loss in the first region (up to 16,000 cycles) for PEN/C-treated samples was significantly lower than that of the

substrate. This confirms the higher wear resistance of the surface layers produced by PEN/C treatment.

## References

1. ASM Handbook, *Heat Treating*, Vol 4, ASM International, 1991, p 853
2. G. Terwagne, J. Colaux, G.A. Collins, and F. Bodart, Structural and Quantitative Analysis of Nitrided Stainless Steel Coatings Deposited by dc-Magnetron Sputtering, *Thin Solid Films*, 2000, **377–378**, p 441–446
3. J.A. Berrios, D.G. Teer, and E.S. Puchi-Cabrera, Fatigue Properties of a 316L Stainless Steel Coated with Different TiN<sub>x</sub> Deposits, *Surf. Coat. Technol.*, 2001, **148**(2–3), p 179–190
4. S. Puchi-Cabrera, F. Matnez, I. Herrera, J.A. Berrios, S. Dixit, and D. Bhat, On the Fatigue Behaviour of an AISI, 316L Stainless Steel Coated with a PVD TiN Deposit, *Surf. Coat. Technol.*, 2004, **182**, p 276–286
5. K. Tokaji, K. Kohyama, and M. Akita, Fatigue Behaviour and Fracture Mechanism of a 316 Stainless Steel Hardened by Carburizing, *Int. J. Fatigue*, 2004, **26**(5), p 543–551
6. M. Akita and K. Tokaji, Effect of Carburizing on Notch Fatigue Behaviour in AISI, 316 Austenitic Stainless Steel, *Surf. Coat. Technol.*, 2006, **200**(20–21), p 6073–6078
7. K. Masaki, Y. Ochi, and A. Ishii, Fatigue Properties of Hard Shot-Peened SUS316L Behaviors of Hardness Distribution, Residual Stress Distribution and Fatigue Cracks During the Fatigue Process, *Mater. Sci. Res. Int.*, 1998, **4**(3), p 200–205
8. H. Stamm, U. Holzwarth, D.J. Boerman, F. Dos Santos Marques, A. Olchini, and R. Zausch, Effect of Laser Surface Treatment on High Cycle Fatigue of AISI, 316L Stainless Steel, *Fatigue Fract. Eng. Mater. Struct.*, 1996, **19**(8), p 985–995
9. P. Villechaise, J. Mendez, and J. Delafond, *Surface Modification Technologies*, Vol IV, The Minerals, Metals and Materials Society, Warrendale, 1991, p 335
10. A.L. Yerokhin, X. Nie, A. Leyland, A. Matthews, and S.J. Dowey, Plasma Electrolysis for Surface Engineering—Review, *Surf. Coat. Technol.*, 1999, **122**(2–3), p 73–93
11. P. Gupta, G. Tenhundfeld, E.O. Daigle, and D. Ryabkov, Electrolytic Plasma Technology: Science and Engineering—An Overview, *Surf. Coat. Technol.*, 2007, **201**(21), p 8746–8760
12. IGR Report: GR/R15696/02 Novel Plasma Electrolytic Treatment Processes to Improve the Wear and Corrosion Performance of Light-Alloys
13. F. Mahzoon, M.E. Bahrololoom, and S. Javadpour, Optimization of a Novel Bath for Plasma Electrolytic Nitrocarburising of 316L Stainless Steel and Study of Tribological Properties of the Treated Steel Surfaces, *Surf. Eng.*, 2009, **25**(8), p 628–633
14. A.L. Yerokhin, A. Leyland, C. Tsotsos, A.D. Wilson, X. Nie, and A. Matthews, Duplex Surface Treatments Combining Plasma Electrolytic Nitrocarburising and Plasma-Immersion Ion-Assisted Deposition, *Surf. Coat. Technol.*, 2001, **142–144**, p 1129–1136
15. L.A. Sosnovskiy, *Fundamentals of Tribo-Fatigue*, Springer, Berlin, 2005