Fatigue behaviour of duplex treated AISI 316L stainless steel

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Abstract

In this study, the effects of surface treatments on fatigue life of AISI 316L stainless steel were investigated. For this aim, the influences of the surface treatments, which are plasma nitriding, Ti-DLC thin film deposition, and duplex surface treatment (nitriding+Ti-DLC deposition) on fatigue life were determined by means of XRD, SEM, fatigue and microhardness tester. It was observed that applied surface treatments increased the fatigue strength of the 316L, in comparison with the untreated ones. Whilst the best fatigue strength was attained after duplex treatment, the fatigue life increased with increasing of the surface hardness.

Key words: duplex treatment, nitriding, CFUBMS, fatigue, 316L

1. Introduction

Plasma nitriding is a thermochemical process, which involves the diffusion of nitrogen atoms to metal based materials. Plasma nitriding treatment is one of the most effective methods to improve hardness, fatigue, wear and corrosion resistance changing properties of surface region of ferrous and non-ferrous materials used in industrial applications. The basic mechanism of plasma diffusion treatment is a reaction between species in plasma and the surface of the metal. In consequence, as a function of this interaction, the nitrided diffusion layer is formed on material surface. In addition, the plasma mass transfer has an effect on the formation behaviour of nitride layer. The mechanical, tribological and corrosion properties are related to the formed structure on the surfaces [1–6].

In the last two decades, duplex surface treatment has been used for improving fatigue, wear and corrosion properties of materials by providing better physical and chemical properties on the material surface. A duplex process is defined as a process where the surface is subsequently deposited by a thin ceramic film after a thermochemical diffusion process [7–10].

There are several studies concerned with the effect of plasma nitriding on fatigue behaviour of various steels. It has been observed that fatigue strength of materials is improved with plasma nitriding. In these studies, it has been shown that the fatigue limit of material depends on the case depth and surface hardness [11, 12]. Another study demonstrated that the fatigue life of machine parts might be increased if they are nitrided after a certain working period [13]. When the plasma nitriding treatment was used in combination with PVD hard coatings, in another words with duplex treatment, a considerable improvement of properties as fatigue and wear resistance of tool steel components has been obtained [14, 15].

Whilst there are a lot of studies about the nitriding of austenitic stainless steel, a little has been published on fatigue behaviour of films deposited on these alloys. Puchi-Carrera et al. [16] observed that fatigue properties of TiN deposited on 316L substrates increased due to compressive residual stresses in the coating and good adhesion of coating by cathodic arc deposition. Berrios et al. [17] determined that the fatigue fracture of the 316L substrate-coating composite is dominated with the fracture of TiN_x film since fatigue cracks were observed to form first within it and subsequently to propagate towards the substrate. Menthe et al. [18] studied the effect of plasma nitriding on mechanical and tribological properties of AISI 316L stainless steel by means of pulsed DC plasma nitriding. They observed that the wear rate after plasma nitriding is reduced compared to the untreated material. Moreover, it was shown that the fatigue strength in the high cycle fatigue at lower repeated loads also increased owing to plasma nitriding. Another study

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demonstrated that both fatigue and fretting fatigue limits are improved after plasma nitriding treatment [19].

In this study, AISI 316L stainless steel was nitrided, Ti-DLC deposited and duplex treated (Ti-DLC deposited after plasma nitriding treatment). After applied surface treatments, the rotating bending test machine, microhardness tester, XRD and SEM were used to investigate the changing of fatigue and structural properties of AISI 316L stainless steel.

2. Experimental details

AISI 316L stainless steel, whose chemical composition is given in Table 1, was used in the experiments. For microhardness measurements, the specimens were cut from cylindrical bars with diameter of 18 mm and thickness of 9 mm. The samples were grinded by 220–1200 mesh emery papers, and then were polished with alumina powder with 1 μm grain size.

The equipment used in the experiments for plasma nitriding was developed in our laboratory [20]. For the plasma nitriding process, after cleaning with alcohol using ultrasonic bath, the specimens were placed into the plasma nitriding chamber and the chamber was evacuated to 2.5 Pa. Then, the plasma nitriding was performed in an 80%H_2+20%N_2 gas mixture, at 500 ℃ temperature for 4 h process time.

The Ti-DLC film was carried out using closed field unbalanced magnetron sputtering (CFUMBS) system produced by Teer Coatings Ltd. The schematic illustration of CFUBMS system is given in Fig. 1. Before DLC deposition, Ti interlayer was deposited at 6 A current for 250 V bias and for 0.4 Pa pressure for 3 min to improve adhesion between the film and the substrate. The thickness of Ti interlayer is about 100–150 nm. Then, Ti-DLC film was deposited on plasma nitrided specimens for duplex treatment.

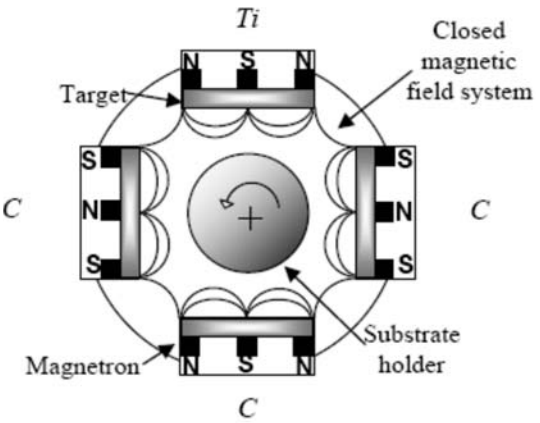

Fig. 1. CFUBMS system.

After the process, the surface hardness and the thickness of the modified layer were measured by using a Buehler Omnimet MHT1600-4980T instrument at a loading time of 15 s and loads of 10, 50 and 100 gf. Rigaku X-Ray diffractometer operated at 30 kV and 30 mA with CuK_α radiation was used for XRD analysis. The morphology of the modified layer was examined using a scanning electron microscope (SEM) Jeol 6400.

Fatigue strength was determined using a rotating bending fatigue machine. The geometry of specimen is given in Fig. 2. Rotating bending fatigue tests were performed at 5000 rev./min in laboratory air atmosphere (≈ 20 ℃, with a relative humidity of about 50 %) and carried out until the complete failure of

Table 1. Chemical composition of AISI 316L stainless steel (wt.%)

C	Si	Cr	Mn	Mo	P	S	Ni	V
0–0.08	0–0.75	16–18	0–2	2–3	0–0.045	0–0.03	10–14	0–0.06

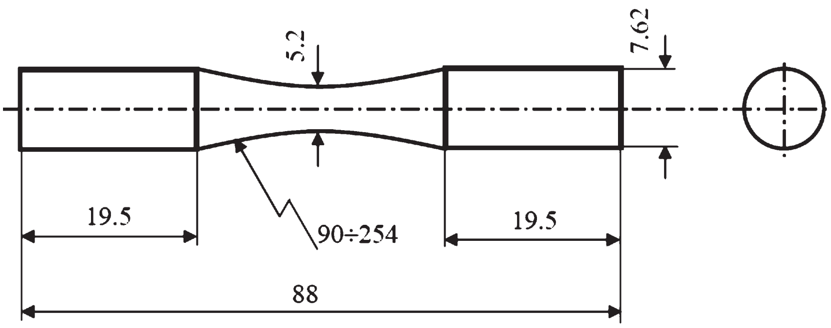

Fig. 2. Rotating bending fatigue test specimens, dimensions are in millimeters.

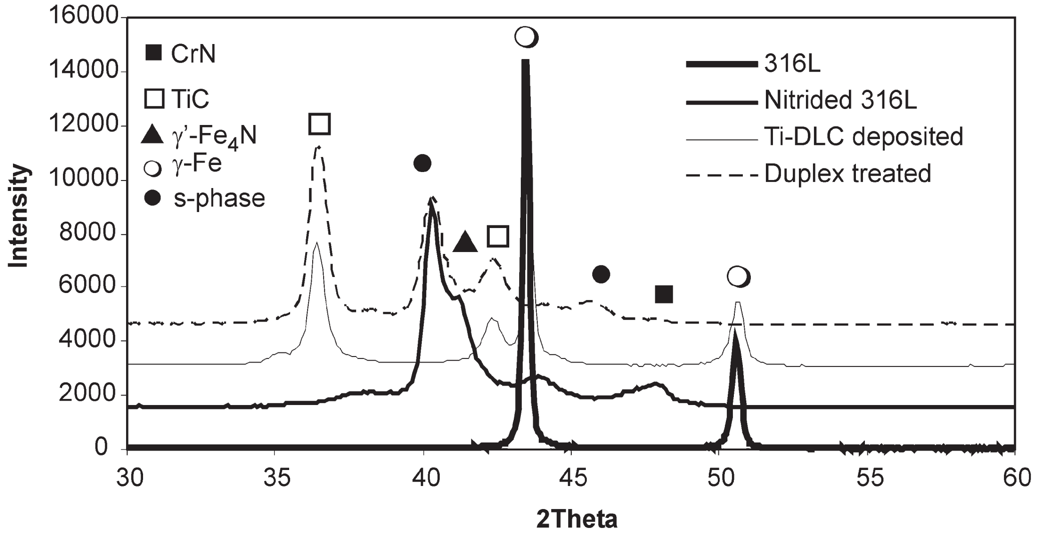

Fig. 3. XRD results of untreated, plasma nitrided, Ti-DLC coated and duplex treated 316L.

Table 2. Microhardness values of untreated and different surface treated 316L

Surface treatment	Microhardness, $HK_{0.01}$		
	10 gf	50 gf	100 gf
Untreated	270–300	270–300	270–300
Ti-DLC deposited	1050–1100	800–900	500–600
Nitrided	1400–1450	1300–1380	1050–1150
Duplex surface treated	2850–2900	2350–2500	1800–2000

specimens. The fatigue machine stopped automatically as soon as the specimen failure occurred. The machine is provided with a digital counter that shows the number of load cycles endured by the test specimen. Twenty-six specimens were used to determine the S-N curve for plasma nitrided and untreated, Ti-DLC deposited and duplex treated specimens; i.e. 15 specimens for the fatigue life (three specimens at each of five levels of stress amplitude), and 11 specimens for the fatigue limit region. The staircase method was employed to determine fatigue limit. In each test, the number of cycles to fatigue failure was noted on semi-log (S, Log$N$) graphs. The fracture surfaces of the specimens were examined by SEM.

3. Results and discussion

3.1. XRD analysis

Figure 3 shows XRD patterns of plasma nitrided, Ti-DLC deposited, duplex treated (Ti-DLC deposited after plasma nitriding) and untreated 316L stainless steel. The phases formed on the surface of AISI 316L after plasma nitriding are s-phase (expanded austenite), CrN and Fe_4N nitrides. Whilst the phase with the highest intensity is detected as TiC, s-phase formed with nitriding and γ-Fe diffracted from substrate were also observed in the duplex treated samples. In the Ti-DLC deposited samples, the main phase is γ-Fe diffracted from substrate.

3.2. Microhardness

The microhardness values of plasma nitrided, Ti-DLC deposited and duplex treated 316L stainless steel are given in Table 2. While the microhardness of untreated 316L was determined between 270–300 $HK_{0.01}$, the microhardness of surface increased 3–10 times as dependent of process type after the surface treatments. The surface hardness of the plasma nitrided samples is higher than Ti-DLC deposited ones because of the high hardness of $CrN+Fe_4N$ dual phase structure. The highest microhardness results were measured after duplex treatment. The hardness of duplex surface treated samples was between 2850–2900 $HK_{0.01}$, and the hardness of Ti-DLC deposited samples was between 1050–1100 $HK_{0.01}$. The surface hardness values measured with different loads (10, 50 and 100 gf) show that the modified layer, which is formed during the plasma nitriding, supports the film and then the load bearing capacity increases. With

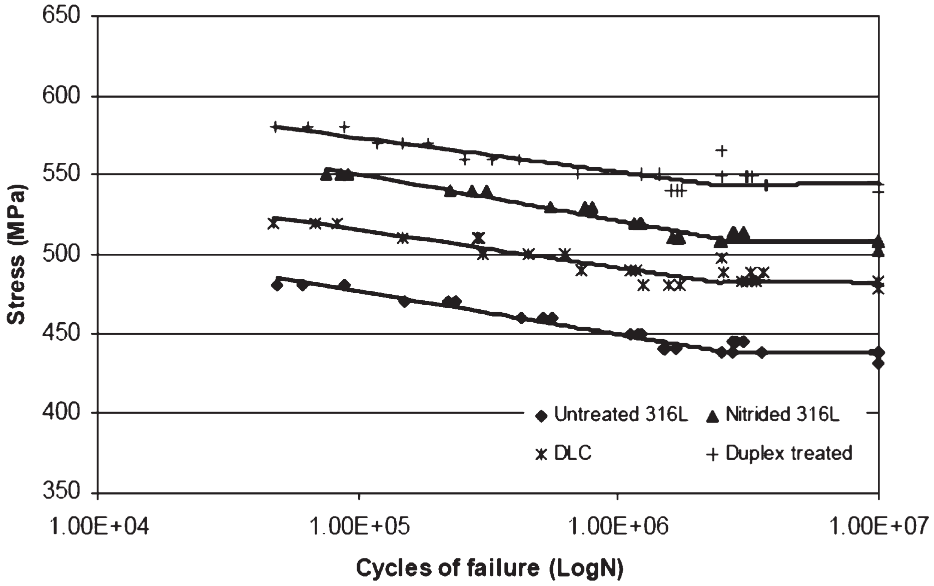

Fig. 4. S-N curves of untreated, plasma nitrided, Ti-DLC coated and duplex treated 316L.

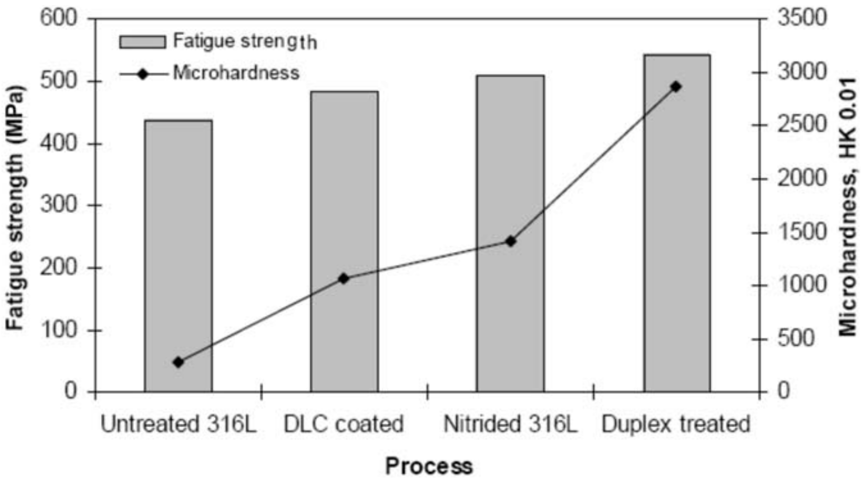

Fig. 5. The relationship between fatigue strength and surface hardness as depending on applied surface treatment.

increasing applied load, the effect of substrate on microhardness values increases.

3.3. Fatigue behaviour

The S-N curves obtained from the rotating bending fatigue tests are illustrated in Fig. 4. The fatigue strength completely depends on the applying surface treatments. While the fatigue limit of untreated samples was 438 MPa, this value increased to 483 MPa with Ti-DLC film deposition, 508 MPa plasma nitriding and 544 MPa duplex surface treatments.

It is known that the most important factor in increase of the fatigue life is compressive residual stress produced at the surface [5]. Especially, after the nitriding treatment, fatigue crack initiation tends to shift from the specimen surface to the subsurface region [21]. It is commonly believed that the intrusions and extrusions produced by sequential slip are responsible for the formation of microcracks on the surface. However, in the nitrided specimens, the intrusions and extrusions are limited by the surrounding hard elastic material. In addition, in this region, the formation of hard precipitates can prevent dislocation movement. As a result, intrusions that normally act as stress concentrators promoting crack nucleation cannot be piled up easily, compared with the untreated homogeneous material [22]. Especially after nitriding treatment the fatigue life increases, because the crack initiation occurs from the subsurface region. The coating process causes improving fatigue life due to producing compressive residual stresses with the effect of ion bombardment [23, 24]. The relationship between the fatigue life and the surface hardness is given in Fig. 5.

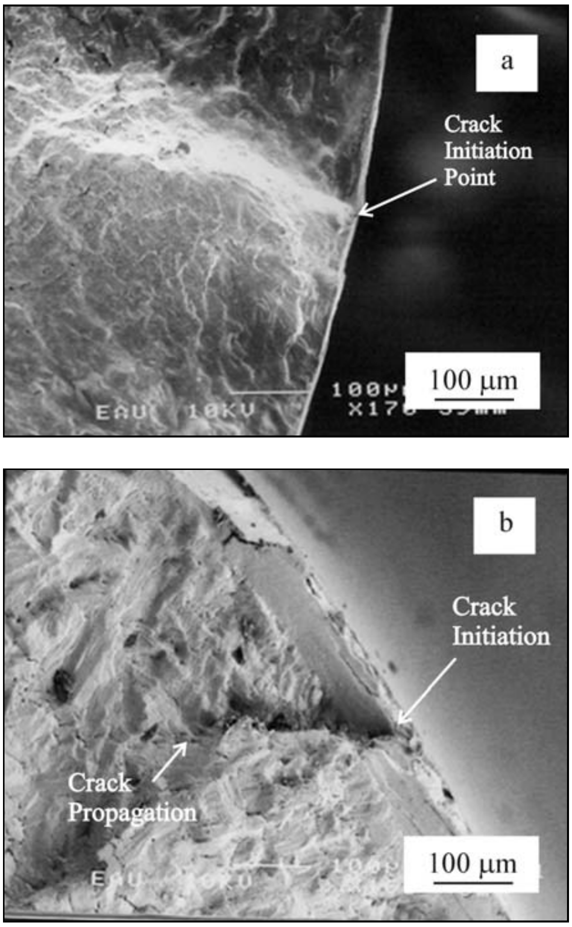

Fig. 6. SEM micrograph of fatigue fracture surface of 316L stainless steel: (a) Ti-DLC coated, (b) Duplex treated 316L.

It was seen that the fatigue strength of a specimen depends on the surface hardness. The fatigue strength of the material increases with increasing surface hardness. The hard modified layer causes improvement of the fatigue strength by preventing dislocation movement.

When the fracture surface of specimens is examined, it has been observed that fatigue crack initiates on the surface of untreated specimens and the formation of beach marks has been observed as well. The fatigue cracks initiate beneath the modified surface after plasma nitriding. Figure 6a shows the crack initiation and propagation in the Ti-DLC deposited specimens. The fracture process is dominated by a single crack which initiates in the core, under the hard deposited film. After duplex surface treatment, it has been observed that crack initiates in the film or interface between film and diffusion layer, and then propagates towards the substrate as indicated by arrows (Fig. 6b).

4. Conclusion

The changing of fatigue and structural properties of AISI 316L stainless steel with the surface treatments, which are Ti-DLC deposition, plasma nitriding and duplex treatment, have been investigated. The following conclusions can be derived from the experimental results:

– After the surface treatments, the microhardness of surface increased 3–10 times comparing to untreated samples and the highest result with 2900 $\text{HK}_{0.01}$ was obtained after duplex surface treatment.

– The fatigue strength of AISI 316L stainless steel was increased by 10 %, 16 % and 24 %, with Ti-DLC deposition, plasma nitriding and duplex surface treatment, respectively.

– The fatigue strength increases with increasing surface hardness.

– SEM micrographs illustrated that fatigue crack initiated beneath the case region after plasma nitriding, whilst initiated in film after Ti-DLC coating and duplex surface treatment.

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