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# MODIFYING THE FRICTIONAL WEAR RESISTANCE OF AISI 316L AUSTENITIC STEEL

#### Keywords

AISI 316L austenitic stainless steel, glow discharge nitriding, boron nitride, pulse laser deposition, hybrid processes.

#### Abstract

The rapid technical development enhances the demands on constructional materials in terms of their resistance to frictional wear, resistance to corrosion and erosion, high hardness, etc. These demands can be satisfied by, e.g. applying various surface engineering techniques that permit to modify the microstructure, the phase and chemical composition of the surface layers of the treated parts. A prospective line of the development of surface engineering is the production of composite layers by combining various surface engineering methods. This paper presents the results of examinations of the phase composition and frictional wear resistance of the layers produced by hybrid processes, i.e. such that combine glow discharge assisted nitriding performed at 450°C and 550°C with a pulsed laser deposition of boron nitride coatings (PLD method). It has been shown that the boron nitride coatings formed on nitride 316L steel increase its frictional wear resistance.

### Introduction

Because of its high corrosion resistance, austenitic steel of the 316 grade is widely used in the food processing industry, chemical industry and medicine.

The primary drawback of this steel is its low hardness and poor frictional wear resistance. In order to improve this resistance, the steel is subjected to various surface treatments, such as glow discharge assisted nitriding. There are, however, certain temperature limitations in the use of this technique, since nitriding conducted at a temperature above 460°C results in the Fe<sub>4</sub>N and CrN nitrides being formed in the near-surface zone of the nitrided layer, which reduces the corrosion resistance of the steel [1–3]. A prospective development line that would permit obviating this drawback seems to be the fabrication of composite layers by using the so-called multiplex (hybrid) methods, which combine the glow discharge nitriding processes with the PVD, IBAD or PLD techniques. Such multiplex methods permit the microstructure, chemical composition and phase composition of the layers being produced to be fully controlled. They also enable the control of the thickness of the layers, the residual stress state prevailing in them and the topography of their surface [4].

This paper presents the results of examinations of the composite layers built of a nitrided layer, produced under glow discharge conditions, and an outer zone composed of boron nitrides. The boron nitride coatings (cubic c-BN and wurtzite w-BN) are characterised by both high corrosion resistance and high frictional wear resistance [5]. Since the boron nitride coatings produced by PLD have an adhesive character, it is necessary to introduce buffer layers, such as glow discharge nitrided layers, whose surface topography is shaped by cathode sputtering [6].

### 1. Experimental procedure

Samples made of 316L steel (0.019%C, 0.327%Si, 1.377%Mn, 17.206%Cr, 10.602%Ni, 2.052%Mo, 0.004%Ti, 0.0431%N, 0.313%Cu, 0.085%Co, 0.026P, 0.0264%S, balance Fe) were subjected to glow discharge assisted nitriding at temperatures of 450°C and 550°C. After the glow discharge nitriding at 550°C, the nitrided layers were subjected to cathode sputtering with the aim of increasing the development of the outer surface of the nitride zone. When the glow discharge nitriding was carried out at 450°C, the cathode sputtering was applied while the samples were heated to the process temperature. The next stage included the formation of boron nitride coatings using the PLD method [7, 8]. The phase composition was examined with a Brucker D8 Discover X-ray diffractometer using CuK<sub>a</sub> radiation, and a FTIR Spectrum GX Fourier infrared spectrometer (Perkin Elmer). The surface topography was analysed in an atomic force microscope (AFM) and a Form Talysurf scanning profilometer (Taylor Hobson). The frictional wear resistance was determined using the T-05 method ("block-on-ring") according to the G77-93 ASTM standard (test ring of AISI 52100 steel having Rockwell hardness of 60 HRC, outer diameter is about 34.99 mm; test block width is 6.35 mm; lubricant oil Lux 10; rotation speed is

316 rpm; wear track radius is 17.5 mm), under a unit load of 400 MPa. After examinations of the frictional wear resistance, the layer surfaces were observed in a Hitachi S-3500N scanning electron microscope. The chemical composition was determined on the cross-sections of the samples and within the abrasion area using the EDS method.

#### 2. Results and discussion

The glow discharge assisted process applied to 316 steel gave surface layers of a thickness of 14  $\mu$ m after nitriding at 450°C and 20  $\mu$ m after nitriding at 550°C. The surface hardness of the layers was 1080 HV0.05 and 1180 HV0.05, respectively. Figs. 1 and 2 show the appearance of the steel surface after the glow discharge assisted nitriding at temperatures of 450°C and 550°C, and after the formation of BN layers on it. Table 1 gives the stereometric parameters of the nitrided layers.



Fig. 1. Surface of the 316L steel samples after nitriding at 450°C: a) without and b)with a BN coating



Fig. 2. Surface of the 316L steel samples after nitriding at 550°C: a) without and b) with a BN coating

Parameter	Starting state [µm]	Nitriding at 450°C	Nitriding at 550°C
R <sub>a</sub>	0.0591	0.0937	0.333
R <sub>q</sub>	0.077	0.12	0.43
R <sub>p</sub>	0.965	0.546	3.2
R <sub>v</sub>	0.53	0.907	1.64
R <sub>t</sub>	1.5	1.45	4.84
Rz	1.12	1.28	4.58

Table 1. Stereometric parameters characterising the surface topography of 316L steel in the starting state and after glow discharge assisted nitriding carried out at temperatures of 450°C and 550°C

 $R_a$  – arithmetic mean deviation of the roughness profile,  $R_q^-$  square mean deviation of the roughness profile,  $R_p^-$  maximum height of the roughness profile,  $R_v$  – maximum depth of the roughness profile,  $R_t$  – maximum distance between the lowest and the highest points of the roughness profile ( $R_t = R_p + R_v$ ),  $R_z$  – mean difference between the five highest points and 5 deepest points of the roughness profile (in accord with ISO 4287/11984).

An analysis of the phase composition of the nitrided layers produced at a temperature of 450°C (Fig. 3) identified so-called nitrogen austenite, known as the phase S, with a thin surface layer composed of chromium nitride (CrN) and iron nitride (Fe4N) (about 0.05  $\mu$ m). In the layers produced at a temperature of 550°C (Fig. 4), the outer zone was also composed of nitrides but its thickness was about 10  $\mu$ m.

Figure 5 shows two-dimensional (2D) and three-dimensional (3D) topographies (in two magnifications) of the surfaces of the boron nitride layers about 200 nm thick, and the relevant phase maps. The topographies were imaged using the contact-less measuring mode ("tapping mode") which permits a phase



Fig. 3. Distribution of iron, chromium and nitrogen in a nitrided layer produced at a temperature of 450°C (a) and a diffraction image of this layer (b)



Fig. 4. Distribution of iron, chromium and nitrogen in a nitrided layer produced at a temperature of 550°C (a), and a diffraction image of this layer (b)



Fig. 5. Topography of the surface of a BN layer produced on the surface of the 316L steel earlier subjected to glow discharge assisted nitriding at 550°C: a) topography 2D, b) topography 3D, c) phase map

analysis to be done at the same time ("phase imaging"). The phase map is prepared based on the differences in the interactions of the measuring edge of the microscope with the layer surface examined, recorded during each measurement. Then the measured values of the interaction forces are displayed as different shades of grey colour (or 'pseudo-colours') in the form of a twodimensional phase map. This map of the regions differing in shades of their grey colour (Fig. 5c) gives information about the varied physical and chemical structure of the layer, and thus, in our case, about its phase structure.

A phase analysis made earlier by HRTEM [9, 10] showed that the layers contained regions with an increased concentration of hard phases (w-BN and c-BN) embedded in a matrix composed of a mixture of the h-BN and a-BN (amorphous boron nitride) phases. The proposed technique of producing boron nitride layers yields layers with the matrix composed of h-BN reinforced with hard phases of the w-BN and c-BN types.

The absorption spectrum (Fig.6), measured with a Fourier infrared (FTIR) spectrometer, confirms that the three boron nitride phases: h-BN, w-BN and c-BN are present in the layers.

As can be seen from Fig. 7, the boron nitride layer, about 200 nm thick, formed on the surface of the nitrided layer, increases the frictional wear resistance, with the greater improvement of this resistance being observed in the 316L steel nitrided at 450°C, where the basic component of the nitrided layer is



Fig. 6. Example of the FTIR spectrum obtained for a BN coating produced on the surface of 316L steel that was pre-nitrided under glow discharge conditions at 550°C

the phase S, known as nitrogen austenite (nitrogen content of about 4.59%). Fig. 8 indicates that it is just the phase S that is responsible for the increase of the frictional wear resistance. During a test conducted under a unit load of 400 MPa, the surface nitride zone, and similarly the thin boron nitride layer undergo chipping. This is why, with the nitrided layers produced at 550°C, in which the compound zone has a thickness of 10  $\mu$ m, the thin BN coating affects insignificantly the wear resistance of the nitrided layer (Fig. 8).

In the analysis of the chemical composition of the wear traces that occur in the nitrided layers, bare and BN-coated, we can see that the basic role in the improvement of the frictional wear resistance is played by the diffusion zone of the nitrided layer, i.e. the phase S, and the thin BN layer produced by the PLD method (Figs. 7 and 8).



Fig. 7. Volumetric wear of 316L steel glow discharge nitrided at 450°C and 550°C with a BN coating and without a BN coating - T-05 method at a unit load of 400 MPa



Fig. 8. Distribution of the elements in the wear region observed in 316L steel nitrided at: a) 450°C and b) at 550°C), (both coated with a BN layer, after a wear test by the T-05 method under a unit load of 400 MPa)

# Conclusions

- Glow discharge assisted nitriding of 316L steel produces nitrided layers with a hardness between 1080 and 1180 HV0.05 and a high frictional wear resistance dependent on the phase composition of the layer.
- A thin coating composed of a mixture of boron nitrides (c-BN, w-BN and h-BN) produced on pre-nitrided 316L steel increases significantly its frictional wear resistance, provided that the principal component of the nitrided layer is the phase S, which can be achieved when the nitriding process is conducted at a low temperature (450°C).
- When dealing with precision parts made of 316L steel, the prospective method of improving their frictional wear resistance seems to be the hybrid method, which combines a low-temperature glow discharge nitriding with the PLD method, the more so since low-temperature nitriding ensures a high corrosion resistance of this steel [11].

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### Kształtowanie odporności na zużycie przez tarcie stali austenitycznej typu 316L

### Słowa kluczowe

Stal austenityczna 316L, azotowanie jarzeniowe, azotek boru, osadzanie laserem impulsowym, metody hybrydowe.

### Streszczenie

Procesy azotowania jarzeniowego stali austenitycznych pozwalają na wytworzenie powierzchniowych warstw dyfuzyjnych zwiększających twardość i odporność na zużycie przez tarcie. Prowadzenie tych obróbek w temperaturach powyżej 460°C powoduje jednak spadek odporności korozyjnej stali. W artykule przedstawiono wyniki badań składu fazowego oraz odporności na zużycie przez tarcie warstw wytworzonych w procesach hybrydowych łączących metodę azotowania jarzeniowego w temperaturach 450°C i 550°C z procesem impulsowego laserowego osadzania powłok azotku boru (metoda PLD). Wykazano, że wytworzenie powłok azotku boru na warstwach azotowanych wytworzonych na stali 316L zwiększa ich odporność na zużycie przez tarcie.