



Mechanical and tribological properties of duplex treated TiN, nc-TiN/a-SiN_x and nc-TiCN/a-SiCN coatings deposited on 410 low alloy stainless steel

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ABSTRACT

The use of hard and superhard nanocomposite (nc) coatings with tailored functional properties is limited when applied to low alloy steel substrates due to their low load carrying capacity. Specifically in this work, in order to enhance the performance of martensitic SS410 substrates, we applied a duplex process which consisted of surface nitriding by radio-frequency plasma followed by the deposition of single layer (TiN, nc-TiN/a-SiN_x or nc-TiCN/a-SiCN) or multilayer (TiN/nc-TiN/a-SiN_x, TiN/nc-TiCN/a-SiCN) coating systems prepared by plasma enhanced chemical vapor deposition (PECVD). We show that plasma nitriding gives rise to a diffusion layer at the surface due to diffusion of nitrogen and formation of the α -Fe and ϵ -Fe₂N phases, respectively, leading to a surface hardness, H , of 11.7 GPa, compared to $H = 5$ GPa for the untreated steel. Among the TiN, nc-TiN/a-SiN_x and nc-TiCN/a-SiCN coatings, the latter one possesses the highest H value of 42 GPa and the highest H^3/E_r^2 ratio of 0.83 GPa. Particularly, the TiN/nc-TiCN/a-SiCN multilayer coating system exhibits superior tribological properties compared to single layer TiN and multilayer TiN/nc-TiN/a-SiN_x coatings: this includes excellent adhesion, low friction ($C_f = 0.17$) and low wear rate ($K = 1.6 \times 10^{-7} \text{ mm}^3/\text{N m}$). The latter one represents an improvement by a factor of 600 compared to the bare SS410 substrate. The significance of the relationship between the H/E and H^3/E_r^2 ratios and the tribological performance of the nano-composite coatings is discussed.

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1. Introduction

Hard and superhard nanocomposite (nc) coatings with tailored functional properties are suitable for numerous engineering applications ranging from protective coatings for cutting tools and automobile parts to biomedical instrumentation and aircraft engines [1–4]. TiN is commonly used in various tribological applications, but its performance is limited at high temperature (≥ 600 °C). Its thermal stability and tribo-mechanical properties can be substantially improved by the addition of Si, B and C [5–7]. Under appropriate conditions, this leads to the formation of a nc microstructure possessing high H/E (elasticity index or elastic strain to failure) and H^3/E_r^2 (defined as resistance to plastic deformation) ratios compared to simple TiN [7–9].

For tribological coatings, hardness, H , is usually considered the primary property affecting the wear resistance. However, it has recently been shown [10] that the H/E_r (E_r being the reduced Young's modulus) ratio appears to be a more suitable parameter to predict the coatings' wear resistance and to explain the deformation properties of surfaces in contact [11]. This ratio also provides a link between wear resistance (K) and the elastic rebound (R). Recently, Musil et al. pro-

posed the H^3/E_r^2 ratio as a key parameter to predict the tribological behavior [12] as well as the toughness of the coatings [13].

The mechanical and tribological properties of hard and superhard coatings are reduced when applied to low alloy steel (LAS). The latter one exhibits low H and low strength, which limits the load carrying capacity of the coating/substrate system [14,15]. Therefore, interfacial engineering is essential to enhance the tribo-mechanical properties of the coating-substrate system.

Duplex treatment is one of the commonly used methods to improve interfacial adhesion [15]. It consists of two independent steps: surface treatment by the processes such as plasma nitriding, boriding, or carburizing, followed by the deposition of a hard coating [16]. Plasma nitriding is a complex process, where diffusion of nitrogen into steel leads to the formation of a diffusion layer (DL), a compound layer (CL) or both [17]. The CL generally consists of nitride compounds of iron such as Fe₄N, Fe_{2–3}N and CrN, depending on the process parameters, whereas the DL layer is a solid solution of nitrogen in the Fe lattice. The CL increases H of the metal substrate, thus reducing the H difference between the substrate and the coatings; however, it is more brittle and it generally possesses higher roughness compared to the DL. Porosity is also often found in CL, and it possibly leads to reduced adhesion [16,18].

By way of example, Sun et al. deposited TiN on unmodified and nitrided LAS substrates (with and without CL) and showed that the

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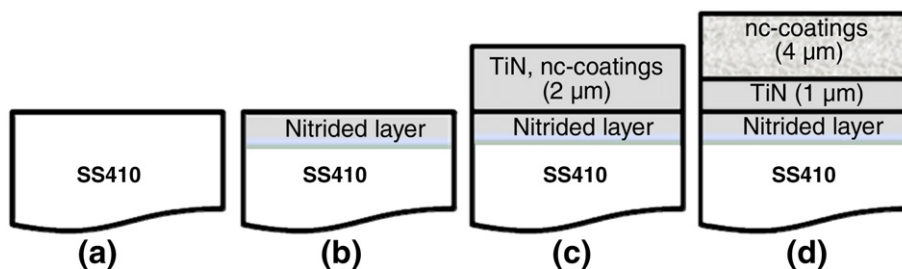


Fig. 1. Schematic representation of interface engineering steps for duplex TiN, nc-TiN/a-SiN_x and nc-TiCN/a-SiCN_x coatings deposited on the SS410 substrate.

nitrided layer consisting of only DL is sufficient to provide satisfactory load bearing capacity and hence improve the tribological performance of the coating–substrate system [16]. Duplex treatments have also been applied to improve adhesion between different steel substrates and the hard coatings [19,20]. Snyders et al. have shown that nitriding of SS316 improved the adhesion between the diamond like carbon (DLC) and the substrate, and it led to an increase of wear resistance by three orders of magnitude [19]. Zukerman et al. have shown that plasma nitriding of PH17 steel improves the adhesion between the substrate and the TiCN coating, and it also significantly enhances the tribological performance [20].

To our best knowledge, no attempts have been made to deposit superhard nanocomposite coatings such as ternary nc-TiN/a-SiN and quaternary nc-TiCN/a-SiCN on LAS, specifically the martensitic 410 stainless steel (SS410). In our earlier work, we systematically studied the relationship between the microstructure and the basic mechanical and optical properties of nc-TiN/a-SiN and nc-TiCN/a-SiCN superhard films on c-Si substrate [7,21,22]. In the present work, we investigate the tribological properties of such nc coatings on LAS substrate (SS410) while constructing a graded interface according to the film architecture illustrated in Fig. 1.

First, we studied the effect of plasma nitriding of SS410 (Fig. 1b) on the adhesion between the coatings and the substrate. Subsequently, we assured a gradual increase in the hardness by applying an intermediate layer of TiN (1 μm) on top of the plasma nitrided SS410 interface (Fig. 1c), followed by the growth of nc-TiN/a-SiN_x or nc-TiCN/a-SiCN coatings (Fig. 1d). Mechanical and tribological properties of all these combinations of coatings were determined and we discuss the influence of H and of the H/E and H^3/E^2 ratios on the overall coatings' tribological performance.

2. Experimental methodology

2.1. PECVD deposition of nc coatings and interface engineering

Martensitic stainless steel 410 (SS410) and high resistivity crystalline silicon c-Si (100) (as reference) were used as substrates. SS410 plates ($25 \times 25 \times 1$ mm³) were tempered at 1253 K for 30 min, quenched in air and annealed at 823 K for 2 h. Thus prepared substrates were polished, using 1 μm alumina suspension, to a surface roughness of 0.02 μm (R_a), cleaned ultrasonically for 10 minutes in acetone and isopropyl alcohol and mounted on a radio frequency (RF, 13.56 MHz) powered electrode (10 cm in diameter).

The deposition chamber was evacuated to a base pressure of 1.3×10^{-3} Pa using a turbomolecular pump. The substrates were sputter cleaned in argon plasma for 30 min using a bias voltage $V_b = -600$ V, and a substrate temperature (T_s) of 523 K. Nitriding of the substrates was carried out in nitrogen plasma for 3 h at a pressure of 26 Pa, N₂ flow rate of 100 sccm, and three different V_b values of -300 , -450 and -600 V.

After nitriding, TiN, nc-TiN/a-SiN_x or nc-TiCN/a-SiCN coatings were deposited by plasma enhanced chemical vapor deposition (PECVD)

using a mixture of TiCl₄, SiH₄, CH₄, N₂, Ar, and H₂, where the flow rates were individually adjusted using separate mass flow controllers. The total working pressure was maintained at 26.6 Pa, $V_b = -600$ V, and two different T_s values of 673 K or 773 K were tested. The flow rates of TiCl₄, N₂, Ar, and H₂ were maintained at 9, 34, 100 and 70 sccm, respectively, to deposit TiN coatings. Adding 0.6 sccm of silane to the above mixture was used to deposit nc-TiN/a-SiN_x, and adding 0.6 sccm of silane and 16 sccm of methane was used to deposit nc-TiCN/a-SiCN.

2.2. Microstructure and composition characterization

The microstructure of the nitrided top layer and of the coatings was investigated by X-ray diffraction (XRD) using the Philips X'Pert diffractometer, (CuK_α line $\lambda = 0.154$ nm). The grain size was determined from the Debye–Scherrer formula [23]. Cross-sectional morphology of the coatings was observed by scanning electron microscopy (SEM) carried out in the PHILIPS XL30 instrument equipped with an Energy Dispersive Spectrometer (EDS). Compositional depth profiles of the coatings were obtained by elastic recoil detection in time-of-flight regime (ERD-TOF) using a beam of 40 MeV ⁵⁹Co⁸⁺ ions [24].

2.3. Tribo-mechanical characterization

Hardness (H) and reduced Young's modulus (E_r) of the coatings were determined by depth sensing indentation using a Triboindenter (Hysitron Inc.) system equipped with a Berkovich pyramidal tip. The applied loads ranged between 1 and 10 mN. For each sample, H and E_r were obtained from 20 indentations using the method proposed by Oliver and Pharr [25]. Elastic rebound, R , of the coatings was determined from the load–displacement curve. Here, $R = W_e / W_{tot}$, where W_e and W_{tot} are the work of elastic deformation and the total work of indentation, respectively [7]. W_e is defined as the area under the unloading curve, while W_{tot} corresponds to the area under the loading curve.

Adhesion was assessed by the Micro-scratch test (MST, CSEM, Switzerland) measurements using a Rockwell C diamond stylus (200 μm radius), a loading rate of 30 N/min and a scratch length of 10 mm. The normal load at which the failure occurs, the critical load, L_C , was determined by optical microscopy after each scratch test. Three scratches were performed on each coating and an average value of L_C was calculated. During the scratch three values of L_C were evaluated: L_{C1} —appearance of the first cracks, L_{C2} —first chipping of the coating and L_{C3} —delamination of the coating from the substrate.

Table 1

Composition of TiN, nc-TiN/a-SiN_x and nc-TiCN/a-SiCN coatings determined by ERD (c-Si substrate).

Coating	Elemental composition (at.%)						
	Ti	N	Si	C	O	Cl	H
TiN	48.3	46.1	0.2	0.2	0.3	2.7	2.2
nc-TiN/a-SiN _x	47.3	44.0	4.4	0.2	0.3	2.5	1.3
Nc-TiCN/a-SiCN	47.1	27.0	3.9	17.2	0.4	2.4	2.0

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