INCREASING THE H13 TOOL STEEL WEAR RESISTANCE BY PLASMA NITRIDING AND MULTILAYER PVD COATING

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The H13 hot-working tool steel is well known for its applications in the metal forming industry. It has been widely used as die material due to its suitable mechanical properties. However, under the extreme working conditions, severe wear always limits the performance of the die as well as the quality of forming. Therefore, strengthening properties of the H13 bulk material and tailoring the surface mechanical properties is of extreme importance. This paper examines and compares the wear behaviour of vacuum heat-treated H13 substrate after plasma nitriding and after Cathodic Arc Physical Vapor Deposition (CA-PVD) coated TiN/TiAlN and Cr/CrN multilayer structures. The wear properties were investigated and compared by ball crater tribometer. The surface hardness of the plasma nitrided and PVD coated multilayers was examined by microhardness tests. The results showed that the wear coefficient of the Cr/CrN coated layer was ten times smaller than the compared TiN/TiAlN and hundred times smaller than the simple plasma nitriding layer.

Keywords: Plasma Nitriding, Cathodic Arc Physical Vapor Deposition, Tribological Properties.

1. Introduction

Steel grades such as H11 and H13 are widely used in the hot-working process wherein the surface temperature is generally above 200°C. These metallurgies are well known for their high strength at elevated temperatures, hot

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wear resistance, good toughness, and good hardenability. In spite of having good hot working capabilities, during industrial production cycles, these steels suffer from surface oxidation, decarbonisation, chemical interactions between oxides, tribological contacts as well as thermal and mechanical loads [1-5].

These tools get loaded by thermal stress as they work in contact with hot material. Although having good wear, corrosion and oxidation resistance, during actual working conditions these tool surfaces get damaged due to wear and cracks formation on the working surface, causing scoring marks and process material sticking to the mould surface. To avoid such problems, advanced surface treatment is required. In the past, several studies have shown the improvement of hot work tools lifetime and performance by surface treatment process [6-9].

These surface treatments are in the form plasma nitriding, PVD coating or combination of both the treatments [10-12]. Several research results have dealt with the tribological performance of the most common TiAlN and CrAlN PVD coatings. These studies show that the performance of coated tools is dependent on several parameters such as substrate chemical composition and microstructure, substrate mechanical properties, deposition method and chemical composition of the deposited layer [12-15].

The investigations of PVD coatings such as CrN and CrAlSiN on steel tool showed an enhancement in the corrosion resistance and its lifetime [16, 17]. The industrial tools such as AISI H11 and H13 were subsequently tested and showed improved performance when coated with CrN, TiAlN or TiN/TiAlN multilayer and plasma nitriding [18-20]. When hot forging tools manufactured with AISI H11 were plasma nitriding and additionally coated with a TiN/TiAlN multilayer, Panjan et. al. confirmed an improved wear resistance and also evaluated the crack propagation during the generation of scratch tracks [21, 22].

The substrate heat treatment is an important factor to attain high strengths and ductility to become compatible with surface nitriding [23]. Several methods are known to investigate the wear resistance and tribological properties of heat-treated and coated tool steels [24-26]. The surface modification by multilayer PVD coatings was reported to be suitable for increasing the wear resistance of the sample surface [27, 28]. In the last few years, the ball-cratering method has been introduced and developed by several researches to determine the wear coefficient [29-32].

The present work deals with the study of the influence on tribological properties of the different duplex multilayer structures of Cr/CrN and TiN/TiAlN with surface modification. In this study, the heat treatment is integrated with nitriding as well as PVD process to improve mechanical properties. The as-obtained wear coefficient using modified ball cratering method developed in Óbuda University, Donát Bánki Faculty of Mechanical and Safety Engineering, Department of Materials Technology (ÓE-BGK-DMT) laboratory [33] and microhardness on the duplex multilayers were compared.

2. Materials and methods

In this work, H13 hot work tool steel was used as a substrate material for the chemical composition described in Table 1.

Each steel substrate was cut and machined from rolled and soft annealed bars with a dimension of Ø15 x 2 mm and subsequently placed in a horizontal vacuum furnace for the preheat treatment.

The samples were preheated up to 650^0 C for 15 minutes with a rate of 30^0 C/min. The substrate was further heated up to 850^0 C with the same rate for 15 minutes.

Table 1

Chemical composition of the used steel substrate in weight percentage (%)

Element	C	Si	Mn	Cr	Mo	V
Concentration, [%]	**0.40**	0.25	0.45	5.25	2.31	0.65

After preheating treatment samples were heated for austenitizing at 1030°C with a heating rate of 15 °C/min for 50 minutes. The austenitized material was then followed by a quenching process at 80°C in a nitrogen atmosphere at high pressure of 10^6 Pa. The heating parameters for the above process are presented in Table 2. The substrates were then polished to a roughness parameter Ra of ~0.01 μm.

Table 2

Heating Parameters

Treatments	Parameters
Austenitization temperature	1030^0C/50 min
Cooling media	Nitrogen Gas
Tempering temperature	550^0C, 580^0C, 540^0C
Plasma Nitriding	520 ^{0}C/24hrs

To improve the hardness, the quenched and tempered samples were subsequently plasma nitriding: first the furnace chamber was cleaned at 480 ^{0}C using N_2:H_2 gas mixture (nitrogen 1 l/h, hydrogen 40 l/h, argon 5 l/h) for 2 hours, second step heating to 520 ^{0}C for plasma nitriding and holding it for 24 hours in mix gas 120 l/h hydrogen and 40 l/h nitrogen (pressure of 2.7 mbar and voltage of

500 V) and after it was cooling in the furnace to 180 °C performed under the same atmosphere.

Duplex multilayer coatings (Cr/CrN and TiN/TiAlN) were further deposited on nitrided substrates using CA-PVD technique. Prior to deposition, all the substrates were cleaned with an ultrasonic cleaning machine, including a hot air drier. Chromium or aluminium/titanium materials were placed as targets on the inner sides of the chamber walls to deposit multilayer coating respectively, onto the substrate. The Ar and N_2 gases were supplied into the chamber, and the gas pressure was maintained at 1 Pa. The coating deposition was carried out at 400^0 C with -80 V bias and 60 A arc current for 120 min. After all treatments surface microhardness for the specimens were evaluated by a Vickers microhardness tester (Buhler 1105). Several measurements were taken for each test and the mean value was utilized.

The wear coefficient for the as-obtained coatings was measured using ball cratering tribometer (Fig. 2) (ÓE-BGK-DMT laboratory). All the samples and the tester ball were cleaned with ethanol and dried with compressed air before testing. The test was performed with 10 mm Al_2O_3 ball running against the coating at room temperature (21 ± 1 °C) and relative humidity of 52%. Diamond abrasive slurry was drip-fed onto the contact surfaces. The worn crater diameters were measured and used to evaluate the substrate as well as the coating wear coefficient using a supported optical microscope (Software: Perthometer Concept Version 6.32-3). The average surface roughness measured on the surface of the nitrided samples (Ra ≈ 0.04 μm). The surface roughness was measured by surface roughness tester (Mahr) (Fig. 1).

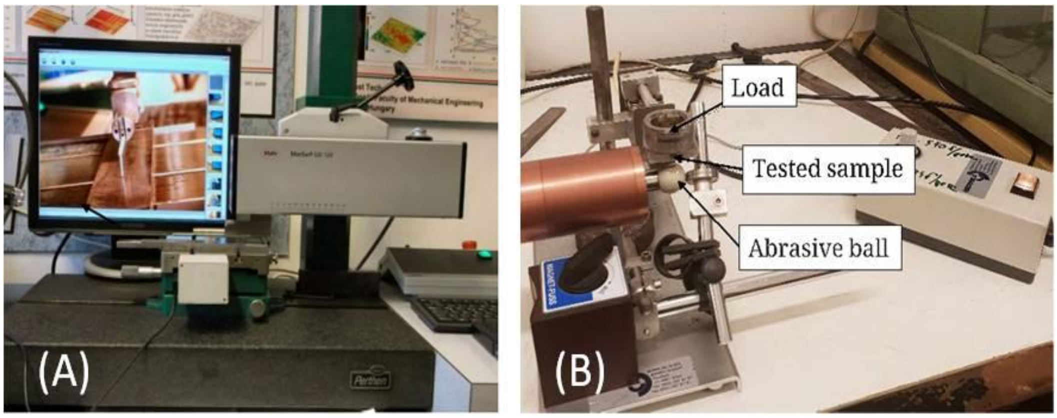

Fig. 1. (A) Surface roughness tester, (B) Ball crater tribometer

3. Experimental results and discussion

3.1. Microhardness

A wide range of microhardness has been produced from 549 to 2756 HV depending not only on PVD process condition but also on surface modification condition. The bar graph in Fig. 2 summarizes the microhardness of the treated samples with different processing.

The measurements revealed that the quenched and plasma nitriding substrates had a hardness value are between 900 HV_1 and 1200 HV_1 respectively. The hardness of the Cr/CrN and TiN/TiAlN multilayer coatings was observed to be effectively increased by the subsequent PVD process.

The observed hardness of the Cr/CrN multilayer coating was 2756 HV which is remarkably higher than not only for quenched and nitride samples but also for TiN/TiAlN multilayer coating.

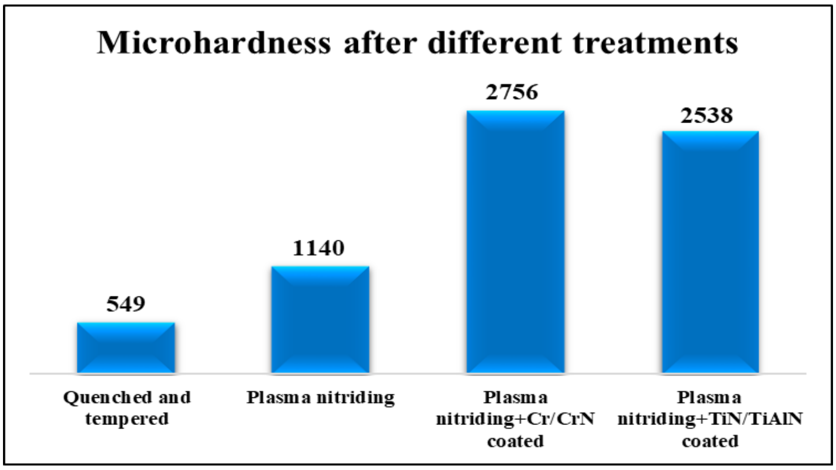

Fig. 2. Microhardness after different treatments

3.2. Thickness and wear coefficient measurements

The thickness of the coating layer was measured using optical microscopy. Fig. 3A and 3B shows the cross-section of the Plasma nitrided + Cr/CrN and Plasma nitrided + TiN/TiAlN multilayer coatings. The coating layer thickness of Cr/CrNand TiN/TiAlNwas found to be 1.77 µm and 1.57 µm respectively. The thicknesses of different surface layers are shown in Table 3.

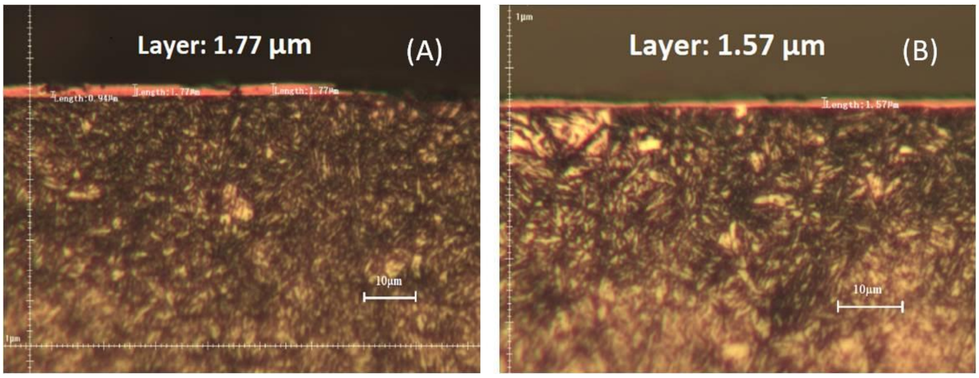

Fig. 3. (A) Cross-section of the coating layer of Cr/CrN sample, (B) Cross-section of the coating layer of TiN/AlTiNsample

Table 3

The surface layers thickness of the different surface treated samples

Surface treatments	Layer thickness (µm)
Quenched and tempered Plasma nitriding	**200**
Plasma nitriding + Cr/CrN	**200+1.77**
Plasma nitriding + TiN/TiAlN	**200+1.57**

The micro-abrasive wear test has been applied in the study of the abrasive wear of all the treated samples using the ball crater tribometer. The wear behaviour of the material with different processing is analyzed based on the dimensions of the wear crater.

The wear coefficient was determined using the average crater radius value. The microscopic images of the obtained craters are shown in Fig. 4. It is observed that the plasma nitriding + TiN/TiAlN coating surface layer frayed during the test and exposed the nitriding zone inside the wear ring as shown in Fig. 4(D).

The wear coefficient K (mm^3/Nm) was calculated using the following Archard equation.

$$K = \frac{V_V}{SN} \tag{1}$$

where S is the wearing length, N is the normal load and V_V wear volume which is given by

$$S = f2\pi Rt \tag{2}$$

$$V_v = \frac{h\pi}{6}\left(\frac{3}{4}(2r)^2 + h^2\right) \tag{3}$$

where R is the ball radius, f is the number of rotations of the ball per min and h is the ball radius and the depth of the were crater respectively which is denoted by

$$h = R - \sqrt{R^2 - r^2} \tag{4}$$

where ‘r’ is the crater radius.

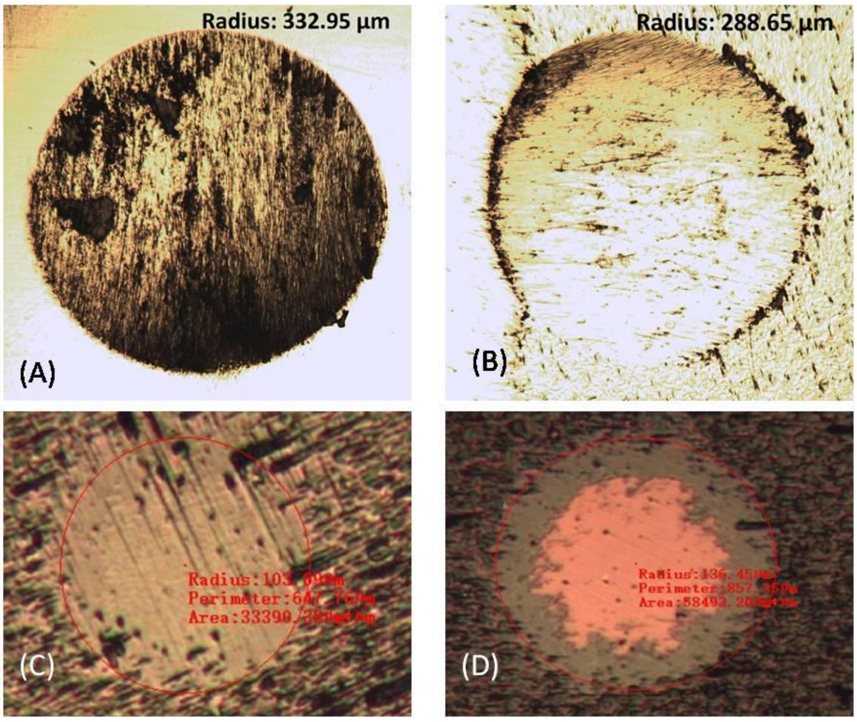

Fig. 4. Microscopic image of wear crater for (A) Quenched and tempered substrate, (B) Plasma nitriding substrate, (C) Plasma nitriding + Cr/CrN coating and (D) Plasma nitriding + TiN/TiAlN coating

Table 4 gives the summary of the properties investigated for all the samples under consideration such as surface layer thickness, average Vickers hardness value and the wear coefficient. From the table, it can be seen that the Cr/CrN multilayer coating deposited on plasma nitriding substrate have shown an improved wear coefficient (4.23×10^{-11} mm^3/Nm) as compared to TiN/TiAlN multilayer coating as well as the bare plasma nitriding substrate.

Table 4

Properties of all samples after different treatments

Sr. No.	Heat and surface treatment	Treated layer thickness (µm)	Hardness, HV	Wear coefficient, K (mm^3/Nm)	Ra (µm)

1	Quenched and tempered	0	549	6.32×10^{-9}	0.01
2	Plasma nitriding	200	1140	1.95×10^{-9}	0.04
3	Plasma nitriding + Cr/CrN coated	200 + 1.7	2756	4.23×10^{-11}	0.17
4	Plasma nitriding + TiN/TiAlN coated	200 + 1.6	2538	7.57×10^{-10}	0.18

3. Conclusions

In this study, the duplex treatment is implemented to improve tribological properties. The average roughness of bare plasma nitriding substrate and duplex multilayer deposited using CA-PVD is higher than the hardened steel. The hardness values of Cr/CrN and TiN/TiAlN multilayer coatings deposited using PVD are significantly higher as compared to the bare plasma nitride and hardened steel sample substrate. It was observed that the surface hardness, surface roughness, surface layer chemical composition has a greater influence on the wear properties. The wear coefficient of the Cr/CrN and TiN/TiAlN duplex multilayer coating deposited on the plasma nitriding substrates using CA-PVD was found to be significantly improved. However, Cr/CrN displayed lower wear coefficient as compared to TiN/AlTiN multi-layer. In conclusion, the plasma nitriding + Cr/CrN multilayer surface-coated samples can be suggested to increase the life-time for the H13 tool steel. In order to understand the cause of the damage to TiN/TiAlN multilayer during the wear coefficient testing process, more tests need to be performed to conclude it.

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