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# A study of the wear performance of duplex treated commercial low-alloy steel against alumina and WC balls



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#### article info abstract

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The objective of this work is to study the improvement of the tribological properties of low alloy steel using a duplex treatment of low pressure carburizing and the deposition of a Cr–(WC–Co) coating by dual RF magnetron sputtering. The treatments result in a 500  $\mu$ m thick carburized layer and a sputtered coating thickness of  $\sim$  2  $\mu$ m. Tribological tests were made with a ball-on-disk tribometer under dry conditions with low load and low speed. The worn surfaces of the disk, the wear counterpart, and resulting debris were analyzed by X-ray diffraction, optical microscopy, optical profilometry, and a scanning electron microscope equipped with an energy-dispersive X-ray spectrometer. The wear performance of the samples was evaluated in terms of wear rates and friction coefficients during the sliding processes against two different counterparts. Experimental results have shown that sliding wear, of the investigated duplex treated low alloy steels, is strongly dependent on the counterface materials. Under testing conditions, the overall wear performance of a Cr–WC coating (Cr/W ratio of 1.12:1) deposited onto the surface of a carburized low alloy steel (0.61 wt.% C; HV =  $654 \pm 5$ ) can be recommended as the best. The duplex-treated samples suffered severe, concentrated wear when against alumina. This wear is characterized by a combination of delamination, mild abrasion and oxidative wear. However, the wear mechanism seems to be oxidative and adhesive when against WC balls.

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# 1. Introduction

As a result of the increased demand for the protection of tools, machine parts, and other devices through surface modification, a wide range of hard coatings and their deposition technology are available. Choosing the proper substrate, coating material and deposition/surface treatment method can dramatically improve the service life of a mechanical component/system, but performance is never the only consideration. Economic and ecological considerations must also be taken into account. For example, changing the substrate material from expensive alloyed steel to cheaper low-alloyed steel can have significant improvements in material availability and reduced production costs [\[1\]](#page--1-0).

Yet, the mechanical and tribological properties of hard coatings may be reduced when applied to low-alloy steels. Therefore, interfacial engineering is necessary to enhance the tribo-mechanical properties of the coating/substrate system. Duplex treatments have been applied to improve adhesion between different steel substrates and various hard coatings, and to enhance the tribological performance [\[2\]](#page--1-0). Duplex

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treatments can be classified as modern technological processes, respecting the environment while ensuring the required properties. Duplex treatments of steel surfaces consist of a thermo-chemical treatment (such as nitriding or carburizing), followed by a coating deposition. The coatings are produced by various techniques, such as chemical vapor deposition (CVD), physical vapor deposition (PVD), plasma assisted CVD (PACVD) or plasma assisted PVD (PAPVD) [\[3,4\].](#page--1-0) Other duplex processes, such as using thermo-reactive diffusion techniques, chromizing, and nitriding, have also been studied in the literature [5–[7\].](#page--1-0) All of these technological processes can be applied to carbides, nitrides, oxides and/or boride hard coatings on various steels and/or other substrates. In comparison to a single process, a duplex treatment can combine the strength and stiffness of the steel substrate with the tribological and/or electrochemical properties of the coating. Kessler et al. [\[8,9\]](#page--1-0) present a series of combined processes, such as heat treat $ment + coating$  and coating  $+$  heat treatment, on different steel substrates. In these papers, they classify the different combinations of coatings/heat treatments and summarize the advantages/disadvantages of each combination.

Many chromium-based coatings have been studied during the last 20 years. For instance, ternary CrXN coatings (where X is a metal, such as Al, Mo, Ti, W…) can be obtained by reactive magnetron sputtering. They are a well-known group of hard and very stable nitride

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coatings. These coatings exhibit high micro-hardness, low thermal conductivity, good wear resistance, and excellent corrosion resistance [\[10,11\].](#page--1-0) Also, the friction and wear of single and multilayer films involving CrN against several materials, remain a subject of interest for several authors. It was established [\[12\]](#page--1-0) that tribological behavior of CrN coating strongly depends on the microstructure and thickness in different tribo-pair systems. More recently, research conducted by [\[13\]](#page--1-0) in the focus to predict the effect of high temperature on CrN coated tools wear performance, confirms the excellent thermal stability and wear resistance of CrN based coating.

Moreover, Cr-based coatings containing carbon are widely used as tribological coating materials in high-temperature applications [14-[16\]](#page--1-0). According to Su et al. [\[17\]](#page--1-0) and Jellad et al. [\[18\],](#page--1-0) sputtered Cr–C films can be of practical interest in abrasive and dry-sliding wear protection applications; in particular,  $Cr_3C_2$  presents excellent strength, hardness, and good corrosion-resistant properties [19–[22\].](#page--1-0)

The previous results on the properties of Cr-based PVD coatings and duplex treatments on steel tools were a good motivation to go further. While forming a good base of knowledge, these previous studies were mostly made for metal machining. Also, no duplex treatments, such as carburizing combined with Cr-based PVD coatings, were studied yet. Then it was obvious that the development of such work on the effect of carburizing and of carburizing  $+$  CrWC PVD coated steel, in the hopes of applying the obtained results for wood cutting tools, would be a significant research advance for the wood industry.

This paper will study the wear behavior of Cr–(WC–Co) films synthesized by dual RF magnetron sputtering on low-alloy steel. Results are presented as follows:

- 1) Optimization of the carburizing of the steel substrate and the necessary deposition conditions of Cr–(WC–Co) coatings for their application on wood machining tools.
- 2) Investigation of the wear resistance of the Cr–WC–Co coatings against different counterparts  $(Al<sub>2</sub>O<sub>3</sub>$  and WC balls).

# 2. Materials and methods

## 2.1. Material

The substrate material considered in this study is a commercial lowalloy steel, DIN 18CrMo<sub>4</sub> (Mat. No. 1.7243). The chemical composition of the substrate is shown in Table 1. This steel has high mechanical strength, high fatigue resistance, and a low price. Indeed, with a carburizing cycle followed by quenching and annealing, this material presents a high superficial hardness with a good ductility at its core (bulk material). This material is commonly employed for machine devices working under surface wear and alternating shock conditions.

## 2.2. Low-pressure carburizing (LPC) treatments

The samples were cut cylindrical ( $\varnothing$  20  $\times$  5 mm height) with an aim for perfect parallelism on the surface. The resulting pieces were mechanically polished with P800 paper, ultrasonically cleaned with acetone, and finally introduced into a single-chamber industrial vacuum furnace "BMI" for carburizing. The chamber is pumped down to 10 Pa. This is followed by heating up to 900 °C (Fig. 1). The substrates were carburized with alternating boost/diffusion stages. The carburizing gas was ethylene  $(C_2H_4)$  under 1 kPa. The length of the "boost-diffusion" carburizing step for two different carburizing mixtures is optimally









Fig. 1. Heat treatment diagram applied to carburize the  $18CrMo<sub>4</sub>$  steel substrates.

selected on the basis of prior experimental data [\[23\].](#page--1-0) The total time intervals of the boost/diffusion stages were 120 min and 105 min for the first one (carburizing C1) and the second (carburizing C2), respectively.

After the carburizing process, samples are in-situ quenched in a high pressure  $N_2$  environment (500 kPa), and subsequently annealed at 200 °C for 120 min. The samples were cooled down in a 100 kPa  $N_2$  atmosphere. These carburized steels were characterized by scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDS, JEOL JSM-5900LV), and optical microscopy (OM; Olympus VANOX-T). Vickers micro-hardness tests were performed to assess the role of the treatment on the modification of mechanical properties (LECO M-400- G micro hardness tester, 100 g applied load). The carbon content at the surface of samples was measured with the use of a LECO CS-200 Analyzer.

# 2.3. Deposition of coating

Cr–(WC–Co) coatings were prepared by reactive dual RF magnetron sputtering in a modified commercial system (Nordiko 3500, 13.56 MHz). Substrate to target distance was 80 mm and the residual pressure was  $6 \times 10^{-5}$  Pa. The 4-in targets were Cr (Neyco, purity of 99.95%) and WC–Co (Ampere, purity of 99.5%); the cemented WC target contained 6 wt.% Co. The substrates were chemically cleaned, followed by a 12 kV DC glow discharge pulsed plasma (25 ms on/50 ms off) for 5 min in an Argon atmosphere at 2 Pa. Substrates were heated to 300 °C to improve both adhesion and mechanical properties of the coatings. The Cr–(WC–Co) coatings were deposited at an argon working pressure of 1 Pa, the cemented-carbide target bias was −600 V, and the Cr target bias was  $-300$  V for the F101 coating and  $-900$  V for the F102 coating.

The coatings' structures were characterized by grazing incidence XRD (Philips X'pert with  $\lambda_{\text{Cu-K}\alpha} = 0.15406$  nm), SEM observations and EDS microanalyses. The micro hardness of the layers was investigated by nanoindentation, with an indentation load ranging from 0 to 10 mN (MTS Nano-indenter XP, Berkovich indenter).

## 2.4. Wear tests

The wear resistance of the treatments has been studied with a pinon-disk tribometer (CSM HT1000) and the Tribox 4.1.1 software under dry-sliding conditions at room temperature. The two standards for the pin-on-disk test are DIN 50324 and ASTM G 99–95a [\[24\]](#page--1-0). The counterparts were alumina (hardness  $H = 16.14$  GPa; arithmetic average  $(R_a)$  and the root mean squared (RMS) surface roughness were  $178 \pm 0.03$  (nm) and  $256 \pm 0.03$  (nm), respectively) and WC–6% Co balls (H = 15 GPa;  $R_a$  = 125 (nm) and RMS = 173  $\pm$  0.03 (nm)), supplied by CSM Instruments. These materials have a higher hardness than steel. Abrasion and shocks are expected during wood machining process thus they are quite suitable to test the wear resistance of coatings in this study. In addition, alumina is widely used as a counter-body because this system (alumina vs. hard coating based CrN) has already been proven to show higher wear resistance and significant differences in material behavior during sliding. This may be related to the high oxidation resistance and high surface chemical inertness of the  $Al_2O_3$  ball [\[25\].](#page--1-0)

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