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#### Full Length Article

# Complex fine-scale diffusion coating formed at low temperature on high-speed steel substrate



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#### 1. Introduction

Surface properties of tool materials, particularly of high-speed steels (HSSs), can be improved using surface engineering [1,2] using techniques such as chemical vapour deposition [3–6], physical vapour deposition [7–10], the diffusion-based thermo-chemical treatments [11–15] and other methods [16–18]. Surface hardened tools where the surface microstructure is optimised in the surface layer can serve well in conditions where abrasive or/and adhesion wear are the prime concerns [19].

The thermo-chemical treatments modify the chemical composition of the surface due to thermal diffusion of carbon, nitrogen, boron, and other elements, which results in a hard surface that is metallurgically bonded to a tough core [20]. Numerous experiments [21–26] have established that boriding, because of the high hardness of the formed borides, provides superior wear resistance of steel products. Moreover, the boride layer retains its high hardness at elevated temperatures [11] thereby enhancing tool life when exposed to abrasion at high temperatures during certain cutting operations [1,22,27]. Enhanced corrosion-erosion resistance and oxidation resistance at elevated temperature are other sig-

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#### ABSTRACT

A complex B-C-N diffusion coating was produced at 580 °C for 1 h on AISI M35 steel substrate and compared with a reference coating formed at 880 °C for 2.5 h. The surface and the cross-sections of the samples were subjected to detailed characterisation. The surface roughness, hardness, residual stresses and adhesion of the coatings were also evaluated together with cutting tests using drills on coated and uncoated samples while monitoring cutting force and torque. The surface of the steel treated at 580 °C revealed Fe<sub>2</sub>B, boron nitride and boron iron carbide, but FeB was noted to be absent. The 580 °C coating had the fine-scale microstructure, which resulted in the excellent adhesion and enhanced wear resistance, relative to reference samples that contained coarse borides. The results established that a complex fine-scale diffusion coating enhanced the wear resistance and reduces the cutting force and torque during drilling, thereby increasing the drill life by a factor of 2.2.

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nificant advantages of the borided steels, as reported by [22,27]. However, conventional boriding treatments are carried out at relatively high processing temperatures in the range of 850-1000 °C [11,27] that coarsen the boride phases with adopt a typical sawtooth morphology [21], which makes the coating brittle and hence sensitive to dynamic loading [28]. According to Campos-Silva et al. [29] the boride layer may also contain residual stress that affects its fracture toughness. The magnitude of these residual stresses increases with the increasing temperature during the treatment. Heavy cracking of worn surfaces of the borided steels during sliding at elevated temperature was also attributed to thermal stresses at the contact region [30]. Furthermore, in our previous study [31] hard and brittle B<sub>4</sub>C phase was revealed in the coarse microstructure of a double phase (FeB/Fe<sub>2</sub>B) boride layer on the tool steel.

With high-speed steels, a more compact boride layer with less of the saw-tooth morphology usually forms on the borided substrate [32–35]. Nevertheless, the presence of residual stresses, due to differences in expansion coefficients of the borides and steel, resulted in cracking on the growing interfaces during treatment [35], especially at high temperature ( $1050 \,^\circ$ C) and long soaking time (8 h) [33]. During turning of AISI 1018 steel the failure and subsequent removal of the boride layer with thicknesses 18 and 40 µm was observed at a nose of the cutting tool made of AISI M2 HSS [34]. Therefore, it can reasonably be concluded that relatively thick boride coatings developed at conventional boriding tempera-







tures are not suitable for cutting tools made of highly alloyed HSSs. One solution is to use a low-temperature treatment but to diffuse simultaneously several elements into the surface, including boron, at which a fine-scale coating thus can be formed. Final heat treatments are then unnecessary so a low temperature thermo-chemical treatment can be simply combined with tempering of HSSs. The purpose of the present work was to characterise the microstructure and the properties of a complex B-C-N diffusion coating formed on AISI M35 HSS substrate at the low temperature.

#### 2. Experimental methods

Commercially available AISI M35 HSS in the form of round bar was used as a substrate to be coated. A nominal chemical composition of the steel was as follows, in wt.%: 0.80-0.90C, max 0.45 Mn, max 0.45 Si, 3.8-4.6 Cr, 5.50-7.00 W, 1.50-2.20 V, 4.50-5.50 Mo, 4,3-5.20 Co, max 0.035P, max 0.055S, and Fe as balance. After full heat treatments, which involved quenching from 1230 °C in oil and triple tempering at 570 °C for 1 h for each cycle, the hardness of the AISI M35 HSS was 65 HRC. The steel samples (12 mm in diameter and 5 mm in height) were metalographically prepared using a Buehler EcoMet 250 with a Buehler AutoMet 250 head (SiC emery paper up to 1200-grit size and Buehler MetaDi diamond suspension with particle size 9, 6 and  $3 \mu m$ ) and finally a Vibromet 2 with a Buehler ChemoMet (suspension Buehler MasterPrep with  $Al_2O_3$  particle size 0.05  $\mu$ m). Commercially manufactured twist drills (6 mm in diameter) made of AISI M35 HSS were selected for cutting tests.

To produce complex B-C-N diffusion coatings, the samples and drills were packed in the powder mixture containing boron carbide, quartz sand, carbonitrided iron powder, sodium fluoride, potassium ferrocyanide, dispersed turf, and polymineral clay and sealed in a steel container, which then was placed into a chamber of an electric resistance furnace with air atmosphere. A low temperature treatment was carried out at 580 °C for 1 h to develop a thin coating and considerably thicker reference coating was produced at 880 °C for 2.5 h. When the treatments were complete, the container was removed from the furnace and slowly cooled in air to an ambient temperature.

The phase composition at the surface of the coatings and residual stresses in the coating and substrate were determined by XRD analysis using an Empyrean PANalytical diffractometer with a Co Ka radiation (a wavelength of 1.7902 Å) operated at 40 mA and 40 kV. The residual stresses were analysed with point focus X-ray tube using polycapillary ( $2 \times 2$  mm) in the incident beam. The plane Fe<sub>2</sub>B (332) at  $2\Theta$  = 117.6° (CoK $\alpha$ 1 radiation) and Fe (211) at  $2\Theta$  = 156.6° (CrK $\alpha$ 1 radiation) were used to determine the residual stresses by the sin2 $\Psi$  method, which is described elsewhere [36]. The diffracted beam path contained a scintillating detector with a soller slit 0.04 rad, and parallel plate collimator 0.27°.

The surface morphology and the cross-sectional microstructure of the studied samples were examined employing a JEOL JSM-7600F scanning electron microscope (SEM), equipped with an Oxford Instruments energy dispersion spectroscopy (EDS) facility. Surface roughness of the samples was evaluated according to standard STN ISO 4287:1999 using a SURFCOM 5000 profilometer.

Nanohardness was measured by NHT<sup>2</sup> Anton Paar nanomechanical tester with the Berkovich type indenter according to the Oliver–Pharr method. The maximal load was 5 mN (15 mN) and the loading and unloading rate was 15.00 mN/min. For each sample, 9 nanohardness tests were conducted and the averaged value was taken as the result. Vickers microhardness tests were carried out using a BUEHLER Indentament 1105 with the maximal load 9.8 N (HV<sub>1</sub>) and 0.098 N (HV<sub>0.01</sub>) according to the standard EN ISO 6507.



**Fig. 1.** (a) Compressed air supply during drilling and (b) controlling the wear land width using a DinoLite Pro digital microscope.

For each sample, 10 measurements were done and the averaged value was taken as the result.

For Rockwell adhesion test a Rockwell "C"-type diamond cone indenter with an applied load of 150 kg was used. Obtained indents were examined using SEM.

The cutting tests were carried out on a Dugard Eagle 1000 CNC vertical machining centre via drilling the EN 10083 C45 steel plate with 225 HB maximum hardness in the annealed state. Three drills were tested for each set of uncoated and coated tools. The cutting conditions were: cutting speed  $v_c = 20 \text{ m/min}$ ; feed  $v_f = 100 \text{ mm/min}$ ; revolutions n = 1200 rpm. Compressed air was used as a coolant, which simultaneously was served to evacuate the chips from the cutting zone (Fig. 1a). The rate of flank wear of the drills with time was controlled by a digital microscope Dino-Lite Pro, which was connected to the computer (Fig. 1b). The flank wear of the drills, calculated as the average values of wear land width measured at flank surfaces of both the cutting edges, was recorded at regular intervals during the tests using software program DinoCapture and then automatically plotted as a function of time in the form of wear curves. Wear land of 0.25 mm in width at the flank surface of the cutting edge was determined to be a critical tool wear criterion. To evaluate the cutting force and the torque during drilling a KISTLER 9257B dynamometer connected via an amplifier to a computer with a DynoWare software was used. This software automatically processed the measured data for cutting force and torque and provided their storage and real time visualDownload English Version:

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