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Microstructural and tribological characterisation of a nitriding/TiAlN PVD coating duplex treatment applied to M2 High Speed Steel tools

A.F. Rousseau [a,b,c,*], J.G. Partridge [a], E.L.H. Mayes [a], J.T. Toton [a,b,c], M. Kracica [a], D.G. McCulloch [a], E.D. Doyle [a,c]

[a] School of Applied Sciences, RMIT University, GPO Box 2476, Melbourne, VIC 3001, Australia
[b] Defence Materials Technology Centre, Level 2, 24 Wakefield Street, Hawthorn, VIC 3122, Australia
[c] Sutton Tools Pty. Ltd., 378 Settlement Road, Thomastown, VIC 3074, Australia

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ABSTRACT

We describe a duplex nitriding and TiAlN coating process performed with no break in vacuum in an industrial scale deposition chamber. Plasma nitriding at 480 °C resulted in a fracture tough diffusion zone with no evidence of either a compound layer or grain boundary precipitation. Untreated, plasma-nitrided, coating-only and duplex-coated High Speed Steel (HSS) M2 coupons were microstructurally and mechanically characterised whilst similarly treated M2 ¼-inch jobber drills were subjected to accelerated drill testing (using D2 tool steel). In these tests, the duplex treated drills exhibited significantly improved wear performance and tool lives when compared with the other drills. After sectioning and electron microscopy, this extended tool life was attributed to increased toughness of the nitrided cutting edges coupled with improved adhesion at the substrate–TiAlN interface.

1. Introduction

Surface treatments have long been central to improvements in the performance of mechanical components and cutting tools. Amongst these, nitriding is a proven method for increasing the resistance of nitridable alloy steels to plastic deformation, fatigue and wear [1]. Traditionally it is conducted using a ferritic thermochemical method, in which nitrogen diffuses into a steel substrate when treated in an ammonia atmosphere at elevated temperatures (~520 °C). In plasma nitriding, nitrogen molecular gas is ionised in a glow discharge plasma to produce energetic nitrogen ions that enhance substrate diffusion [2]. Protective coating layers, typically composed of metal nitrides, carbides or carbonitrides and deposited by chemical vapour deposition (CVD) or physical vapour deposition (PVD), can also extend the capabilities of substrates. For example, in machining tools tribological interactions occurring at the chip–tool interface [3,4] are shown to be improved with the application of PVD coatings [5,6] however, these improvements are compromised if normal loading is sufficient to cause plastic deformation in the underlying substrate [7–10].

Duplex coatings, consisting of a nitrided steel substrate and a hard PVD coating, can exhibit the high hardness and high wear properties of PVD coatings combined with the enhanced fatigue resistance and load carrying properties of the nitrided substrate. The duplex coating process may be conducted sequentially using separate nitriding and coating apparatus or if it is suitably equipped, within the same vacuum chamber [8,11–16]. PVD equipment has been successfully adapted for plasma nitriding in order to duplex coat nitrided steel with CrAlN in a single, sequential process [7]. The resulting duplex coatings were shown (in scratch and impact fatigue tests) to have superior wear properties to those provided by CrAlN coatings on un-nitrided steel.

This paper reports the properties of M2 HSS coupons and test drills duplex treated by plasma nitriding combined with a commercial PVD TiAlN coating in a single vacuum system designed for industrial applications. TiAlN was selected as an outer coating layer as it is a proven ternary solid solution with good wear resistance, hardness, low friction and elevated operating temperature capability [17–21]. Microstructural analysis combined with mechanical/nano-indentation measurements and accelerated drill testing has been used to characterise the constituent layers and the duplex treatment as a whole.

2. Experimental methods

2.1. Nitriding

M2 HSS coupons (measuring 20 mm and 8 mm in diameter and thickness) and M2 ¼″ jobber drills were heat treated to achieve a nominal Vickers hardness (averaged from 5 indents per coupon) of 850HV30. Cleaning was performed in an alkaline solution with ultra-sonic agitation followed by rinsing in de-ionised water and drying at 110 °C. The tools were then loaded with vertical orientation onto a turntable (diameter 510 mm × 490 mm height) within the (800 mm × 900 mm × 900 mm)

* Corresponding author. Tel.: +61 490127988.
E-mail address: andre-rousseau@hotmail.com (A.F. Rousseau).

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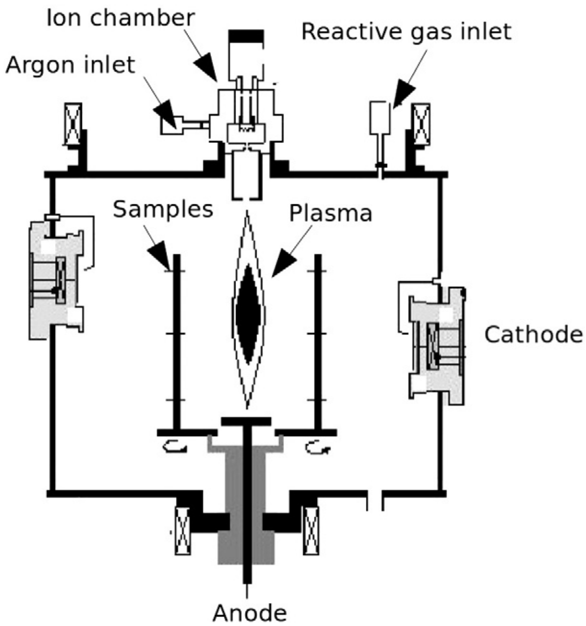

Fig. 1. Schematic of Balzers INNOVA industrial capacity cathodic arc PVD system.

chamber of an industrial capacity Balzers INNOVA cathodic arc PVD system, shown schematically in Fig. 1. The chamber was pumped down to 1×10^{-3} Pa before nitriding was performed in a 16:66:18 hydrogen:nitrogen:argon atmosphere at 1.0 Pa and 480 °C for 30 min. Combined radiative/electron-beam heating was used for nitriding and PVD coating. The plasma current was 150 A and the plasma potential was 58–63 V. A bias potential of −150 V was applied to the coupons and drills.

2.2. PVD coating

After nitriding and with no break in vacuum, argon etching was performed using a plasma current of 150 A and a bias of −170 V for 20 min at 0.25 Pa. The central plasma was then extinguished and the coating commenced at 450 °C in a three step process; (1) TiN functional layer (0.8 Pa, 100% N, bias −150 V and 2 Ti targets 180 A, 8 min), (2) TiAlN transition layer (3.2 Pa, 100% N, bias −40 V and 2 Ti targets 200 A plus 2 targets Ti/Al 200 A, 4 min), and (3) TiAlN top layer (3.2 Pa, 100% N, bias −40 V and 4 Ti/Al targets 200 A, 130 min). TiAlN coatings were deposited with a thickness of ~3 μm. TiN and Ti-rich interlayers were incorporated to reduce thermal and lattice mismatch at the substrate/coating interface [14,22].

2.3. Metallography

The nitrided and coated surfaces were imaged in a scanning electron microscope (SEM) and, topographically, using a Veeco Dimension 3100 atomic force microscope (AFM) operating in tapping mode with a <10 nm radius-of curvature Si tip. The coating thickness and nitriding diffusion depths were determined from coupons cross-sectioned with an abrasive cut-off wheel, mounted in a phenolic conductive resin and polished using diamond abrasive laps. The polished cross-sections were etched with 4% Nital for 25 s and then SEM imaged. A Bruker D4 X-ray diffractometer (fitted with graphite-monochromated Cu Kα radiation source (= 1.5406 Å) with a potential of 40 kV and a current of 35 mA) was used to obtain X-ray diffraction measurements (2θ scan range 5–90°, step size 0.02° and a count rate of 0.3 s) from the coatings and nitrided coupons. X-ray photoelectron spectroscopy (XPS) was performed using a Thermo Scientific K-Alpha system equipped with an Al Kα X-ray source (1486.7 eV), takeoff angle of 90°, pass energy of 50 eV and 400 μm diameter analysed area. These measurements revealed changes in chemical bonding occurring after plasma nitriding.

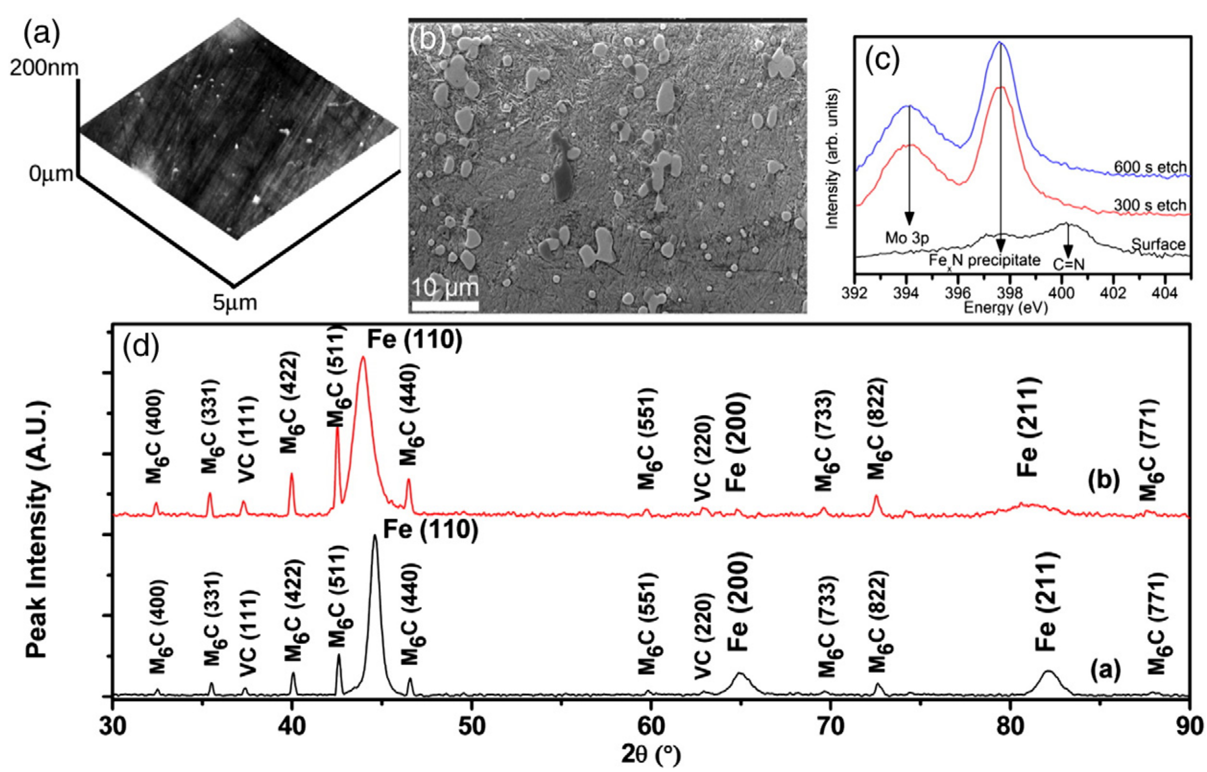

Fig. 2. (a) AFM topographical image, (b) cross-sectional SEM image, (c) XPS N 1s peak and (d) X-ray diffractograms (before and after bright nitriding) measured from a heat-treated and nitrided M2 coupon.