Tribology International 57 (2013) 101–106

Contents lists available at SciVerse ScienceDirect

Tribology International

journal homepage: www.elsevier.com/locate/triboint

Sliding wear of CrN, AlCrN and AlTiN coated AISI H13 hot work tool steels in aluminium extrusion

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ARTICLE INFO

Article history:
Received 12 April 2012
Received in revised form
25 July 2012
Accepted 26 July 2012
Available online 3 August 2012

Keywords:
Aluminium extrusion
Tooling
Sliding wear
PVD

ABSTRACT

The frictional forces arising from sliding contact with aluminium produce considerable shear stresses on the CrN coating that degrade its integrity and its adhesion to the substrate tool steel. The CrN coating suffers the most extensive wear damage. There is a net improvement in the sliding wear performance of the AlCrN coating owing to a multilayered structure and a much higher hardness. However, AlTiN outperforms both CrN and AlCrN in sliding contact against aluminium owing to its exceptionally high chemical resistance against aluminium. The superiority of the AlTiN coating is confirmed by wear rates estimations.

1. Introduction

Aluminium extrusion process involves high loads and elevated temperatures whereby a billet is forced through the opening of a die to produce profiles of almost any shape and size [1,2]. The die bearing surface has to withstand substantial mechanical and thermal stresses, abrasion and adhesion as well as chemical attack [3–5]. Sliding wear has been reported to be one of the major failure modes [5]. The die is replaced or reworked once the dimensions and surface finish of the extrudate deteriorate beyond tolerable limits. Hence, extending the service life of extrusion dies is a priority for the economics of this process [4].

The standard die material in aluminium extrusion, AISI H13 hot work tool steel, offers high strength and ductility, good tempering resistance at moderate cost, but fails to combat against sliding wear [6]. Hence, there is a growing interest in using surface engineering techniques to improve the wear resistance of hot work tool steels [7–14]. Thin hard films, coated on tool steels with the Physical Vapour Deposition (PVD) process, offer an attractive combination of high hardness, good wear resistance and chemical stability at high process temperatures [8–10,15–21]. Among the coatings tested, CrN has been popular since the early 1980s [22], owing to its beneficial friction properties and oxidation resistance due to the formation of a passivating surface oxide [4,13,16,23,24]. The more recently commercialized AlCrN demonstrates even superior mechanical properties as well as higher oxidation resistance [25,26]. The aluminium-rich thin hard AlTiN coatings also offer a very attractive combination of hardness and oxidation resistance at elevated temperatures as well as high fatique resistance [27,28]. Hence, it has received a great deal of attention for surface engineering of forming and cutting tools in recent years [29,30]. The present work was undertaken to test the performances of CrN, AlCrN and AlTiN coated AISI H13 hot work tool steel samples under actual aluminium extrusion conditions.

2. Experimental

Wear test samples were machined from a premium grade AISI H13 hot work tool steel, austenized at 1025 °C for 30 min, quenched in circulating air and finally tempered twice at 625 °C for 2 h yielding a hardness of 480 ± 20 HV. The samples thus obtained were polished to a mirror-like finish and cleaned ultrasonically before they were nitrided to a case depth of 150 ± 50 μm. The nitriding operation was carried out in a direct current glow discharge plasma unit, at 520 °C for 12 h in a 100% NH_3 gas atmosphere at total pressure of 100 Pa.

A Novatech Model NVT 12 Cathodic Arc Physical Vapor Deposition (CAPVD) unit was employed for depositing thin hard CrN, CrAlN and AlTiN coatings on tool steel samples. The tool steel substrates were heated and ion-etched with Cr bombardment. The temperature of the substrate samples increased to approximately 450 ± 50 °C due to IR and plasma heating. A CrN bond coat was deposited before the ternary nitrides. 99.99 at% purity Cr and 70/30 at% Al–Cr, 67/33 at% Al–Ti cathodes were used to deposit

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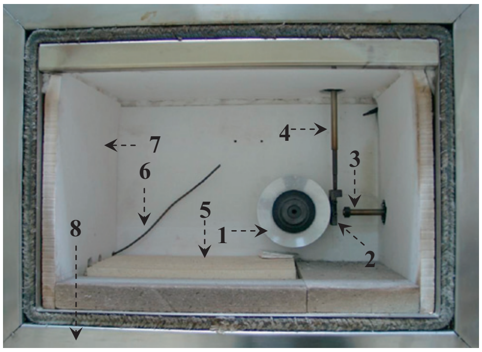
0301-679X/$ - see front matter © 2012 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.triboint.2012.07.023

CrN, AlCrN and AlTiN coatings, respectively. The chamber was evacuated to approximately 10^{-3} Pa. The cathode current was 60–75 A. High purity (99.999%) nitrogen was used at a N_2 pressure of 1 Pa and at a bias voltage of -100 V.

Standard metallographic practices were employed to characterize the CAPVD-coated samples. The coating thickness was measured with a Wirtz–Buehler Calotest ball cratering test unit. A JEOL 6335F model field emission gun scanning electron microscope (FEG-SEM) fitted with an Oxford INCA model energy dispersive X-ray analyzer (EDS), was employed to analyze the morphology and chemistry of the coatings. The phase structure of the surface layers were examined with a Philips PW 3710 grazing incidence X-ray diffractometer equipped with Cu K_α radiation. A scan rate of $0.02^\circ\ s^{-1}$ was used with a grazing incidence of 0.5°. A Fischerscope H100 model Ultra micro hardness tester was used to measure their hardness. 20 mN was applied in 120 steps every 0.5 s with load and depth sensitivities of 0.2 mN and 0.01 nm, respectively. The average of 20 measurements was reported.

The wear test in the present work employed the common block-on cylinder configuration assembled inside a heating chamber [9]. Hot work tool steel samples, 5 mm × 10 mm × 30 mm, heat treated, nitrided and finally coated with various thin hard coatings on the contact face, served as the die bearing while an aluminium cylinder, ϕ 100 mm × 50 mm, machined from a commercial 6063 alloy billet served as the aluminium extrudate (Fig. 1). The test chamber was heated to 550 °C. A normal force of 60 N was applied to the coated test sample to establish a dry sliding contact with the rotating aluminium cylinder. The sliding speed was $0.52\ ms^{-1}$ and the total sliding distance was 3900 m. The sliding operation was interrupted for 10 s after every 50 s to reproduce the start-and-stop cycle encountered in the industrial aluminium extrusion process when a new billet is loaded to the press. The normal force, the friction force and the temperature were selected with a consideration of the actual extrusion process and were continuously recorded throughout the wear tests. The friction coefficient was estimated from the friction and normal forces acting on the contact face of the samples once steady state conditions were established.

The worn surfaces were investigated with optical microscopy (OM) and field emission scanning electron microscopy (FE-SEM) both on the surface and across the section of tested samples. The aluminium deposits on the wear test samples were removed in a NaOH solution at 80 °C before SEM investigations. The topology of the coated surfaces and of the wear tracks in worn samples was investigated with a VeecoWyko NT1100 3-D model optical profilometer.



1: aluminium cylinder
2: coated hot worktool steel sample
3: normal force
4: friction force
heating element :**5**
thermocouple :**6**
ceramic insulation :**7**
test chamber :**8**

Fig. 1. Testing unit used in the sliding wear tests.

3. Results and discussion

The binary CrN and ternary AlCrN and AlTiN coatings reveal uniform features both over the surface and across the section and a dense structure with no evidence of delamination or structural flaws along the interface (Fig. 2). The former reveals single layer morphology as evidenced by the EDS analysis across the section (Fig. 2a). The thickness and hardness of the CrN coating are 6.3 μm and 1998 ± 57 HV, respectively (Fig. 3). The composition was measured by EDS analysis to be 54 at% Cr and 46 at% N, implying a slightly rich CrN structure as confirmed by the XRD analysis (Fig. 4a). The AlCrN coating, on the other hand, reveals an inner layer that generates only Cr and N signals and a relatively thicker outer layer with additionally Al. The EDS analysis of the outer layer, however, shows that the composition shifts from a Cr-rich to an Al-rich one as we move from the bond coat to the surface, implying essentially a three-layer structure. The gradual change in the chemistry across the top coat is evidenced further by the change in contrast across the coating (Fig. 2b). The composition of the top layer was measured by EDS to be 39 at% Al, 18 at% Cr and 43 at% N, suggesting an Al-rich ternary coating of the type $Al_{0.68}Cr_{0.32}N$. The total thickness of the AlCrN coating is 3.6 μm. Its hardness was measured to be 3514 ± 112 HV (Fig. 3), in reasonable agreement with those reported for AlCrN coatings [31]. Incorporation of Al into the CrN structure has apparently led to a remarkable increase in hardness. The glancing incidence X-ray diffraction spectrum (Fig. 4b) exhibits the NaCl crystal structure. The cubic structure is favoured over the hexagonal variety owing to a relatively higher Al content. The hardness, oxidation resistance and the tribological properties of $Al_xCr_{1-x}N$ coatings have been reported to improve with increasing Al-content provided that the cubic structure is retained, but deteriorate when the hexagonal structure forms at higher ($x>0.75$) aluminium contents [32,33]. Hence, with an Al-rich cubic crystal structure, the present coating has the potential to offer adequate resistance to sliding wear at elevated temperatures.

The section of the surface layer of the AlTiN coated tool steel sample is shown in Fig. 2c. The composition of the outermost layer was estimated by EDS to be 34.2 at% Al, 51.8 at% N, and 14.0 at% Ti, giving a coating of the type $Al_{0.71}Ti_{0.29}N$. The neighbouring layer exhibits additionally a Cr signal suggesting that a transition zone has formed before the top coat (AlTiN) was deposited. Finally, the innermost layer reveals only Cr and N signals and serves to improve the adhesion of the AlTiN coating to the substrate. The total thickness of this multilayered coating was measured to be approximately 3 μm. The hardness measured on the surface was 3291 ± 118 HV (Fig. 3). The structure of the AlTiN coating is also based on the structure of NaCl–AlN with a fraction of the aluminium atoms substituted by titanium atoms (Fig. 4c). The incorporation of aluminium effectively enhances the coating thermal stability and hardness [34]. No evidence for the softer hcp-AlTiN phase is available in the XRD spectra [35].

The surfaces of aluminium cylinders and CAPVD coated hot work tool steel samples slid against each other evidence adhesive wear (Figs. 5 and 6). The aluminium cylinders are heavily deformed by the coated tool steel test blocks confirming the thermoplastic response of aluminium at the present test temperature (Fig. 5). While they look rather similar at overview magnifications, there are marked differences in the features of the wear tracks on the aluminium cylinders. The channel produced by the CrN coated sample (Fig. 5a) is relatively deeper with a rough topology implying a more extensive interaction of the aluminium alloy with the CrN coating and clearly a more extensive displacement of the surface stock. The wear track linked with the AlCrN coating is in a slightly better shape (Fig. 5b). The sliding track on the Al-cylinder tested in contact with the AlTiN