ADHESION AND WEAR PROPERTIES OF HARD COATINGS ON TOOL STEELS

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SUMMARY
During cold forming processes like deep drawing of steel sheets, tools are subjected to various loads. Besides adhesion, abrasion and tribo-oxidation, successive impacts during forming induce Hertzian contact pressures and cause fatigue failure. The aim of this work is thus to evaluate different hard coatings deposited by plasma-assisted chemical vapour deposition (PACVD) onto tool steels with respect to adhesion and wear properties. The adhesion of TiN, Ti(C,N) and Ti(B,N) coatings was investigated using the VDI indentation test and a surface fatigue test, where cyclic loads up to 80 kN are applied to the tool surface by cylindrical indenters. Adhesive wear of the coatings has been characterised using a ball-on-disc tribometer. The abrasion resistance was evaluated using a small-scale abrasive wear test.

Keywords: hard coatings, abrasion, adhesion, fatigue, tool steel

1 INTRODUCTION
Today, hard wear-resistant coatings have found increasing applications in various sectors of industry to protect tools from failure caused by wear and corrosion. These coatings are mainly applied to cutting tools or may also be used for injection moulds; often these tools show a pronounced increase in lifetime after coating [1]. In many metal-forming processes like deep drawing or cold forging, however, dies are used under dynamic elastic conditions and fail after a certain period of service due to fatigue fracture. It has already been proven under near production conditions that PVD (physical vapour deposition) and CVD (chemical vapour deposition) coated sheet-metal working dies show increased lifetimes, as compared to uncoated tool steels [2]. However, there is a need for still improving film adhesion, fatigue limit and wear behaviour for these applications. Therefore, it is necessary to evaluate coatings on tool steels by subjecting them to dynamic loads for characterising their behaviour when in contact to the workpiece. Standard adhesion test methods like scratch and indentation tests involve static or quasistatic elastoplastic loading. To evaluate the fatigue behaviour of a coating/substrate system, a fatigue test technique has to be developed to determine wear caused by dynamic repetitive loading [3]. Coating fatigue failure may not only be adhesive but also cohesive. Adhesive fatigue failure can be seen by interfacial adherence fracture and subsequent macro-delamination, while cohesive fatigue failure causes initial intrinsic coherence release and subsequent chipping. Apart from the fatigue failure, also adhesion and abrasion takes place in many applications like cutting and forming processes [4]. A considerable amount of abrasive wear can be caused when particles which are harder than the tool material are involved. These particles can typically be carbides, oxides or highly strain-hardened fragments like wear debris which are generated during deep-drawing. Adhesive wear and tribo-oxidation may be caused by high contact temperatures.

In the present paper, results on an investigation of TiN, Ti(C,N) and Ti(B,N) coatings deposited onto tool steels using plasma-assisted chemical vapour deposition (PACVD) are presented and discussed. The properties of the coating/substrate interface are characterised with regard to their fatigue behaviour and adhesion. Adhesive wear is characterised by evaluating friction coefficient and by investigating the wear track using scanning electron microscopy (SEM) and optical 3d profilometry. Abrasive wear coefficients are used to quantify the abrasion wear behaviour.

2 EXPERIMENTAL DETAILS
TiN, Ti(C,N) and Ti(B,N) coatings were deposited using a commercial Rübig PACVD system (inner diameter of the deposition chamber: 400 mm, height: 600 mm) [5,6]. The charging plates are connected to a bipolar DC voltage-controlled pulse generator. In addition to plasma heating, heaters are fitted into the chamber walls, and the substrate temperature is measured using two thermocouples. Process parameters, i.e. gas flows, wall heaters, voltage and duration of pulse-on and pulse-off time and total pressure are controlled by a programmable logic controller which is interfaced with a personal computer. In the present study, the coatings were grown using H₂, Ar, N₂, TiCl₄, CH₄ and BCl₃ gases. Typical deposition parameters can be seen in Table 1.

The coatings have been deposited after plasma nitriding without white compound layer [7] onto steel substrates. Coating thickness was adjusted to 2.6 ... 3.5 µm.

Substrates used within this study for fatigue measurements were cylindrical specimens with diameter 40 mm and height 16 mm of hot-working tool steel DIN
1.2344 (AISI H13) which were quenched and tempered to a hardness of about 50 HRC.

<table>
<thead>
<tr>
<th>Deposition parameter</th>
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<tr>
<td>Total pressure [Pa]</td>
<td>50 – 300</td>
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<tr>
<td>Substrate temperature [°C]</td>
<td>480 – 500</td>
</tr>
<tr>
<td>Discharge voltage [-V]</td>
<td>400 – 500</td>
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<tr>
<td>Deposition time [h]</td>
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Table 1: Typical PACVD deposition conditions

For ball-on-disc tests and abrasion measurements specimens with diameter 30 mm and height 10 mm of high speed steel DIN 1.3343 (AISI M2) were used which were quenched and tempered to a hardness of about 63 HRC. Observations of the surface condition after the fatigue and wear tests were carried out using scanning electron microscopy (SEM; Cambridge Instruments Stereoscan 360). Coating characterisation was performed with respect to thickness (spherical abrasion test), Vickers microhardness and adhesion (VDI Rockwell indentation). The abrasion resistance was evaluated employing a small-scale abrasive wear test (CSEM CaloWear) where a steel sphere is rotating against the coated sample in the presence of an aqueous suspension of abrasive SiC particles. The diameter of the resulting wear crater was determined as a function of the sliding distance by means of a calibrated optical microscope and served as the basis for calculating the abrasive wear coefficient [8]. A ball-on-disc test (CSEM High-Temperature Tribometer) was used to evaluate adhesion and tribo-oxidation between coated tool steel discs and 100Cr6 steel balls (ball radius: 6 mm). The experiments were conducted in air at a temperature of about 22 °C and a humidity of about 40 %. A load of 10 N was applied to the 100Cr6 ball. The sliding speed and the radius of the wear track were 10 cm/s and 7 mm, respectively. An optical 3d profiling system (Wyko NT1000) was used to investigate wear tracks on the coatings and the steel balls. In order to investigate the fatigue behaviour of coated steel specimens under repetitive loads, a hydraulically driven push-pull testing machine with loads up to 80 kN was employed [10]. A cemented carbide cylinder with diameter 10 mm was used to apply the load to the coated samples investigated. The sinusoidal force, the frequency and the number of compressive cycles are registered and regulated employing a personal computer. In this study, each sample was subjected to $2 \cdot 10^5$ load cycles using a frequency of 20 Hz. The experiments were carried out using a relatively high compressive load which was sinusoidally varied between 80 and 2 kN. After each test, the cemented carbide cylinder was twisted to a new track on the surface to avoid flattening.

3 RESULTS AND DISCUSSION

3.1 Adhesion and fatigue behaviour

Results obtained by the VDI indentation test are presented to illustrate the different adhesion behaviour of the coating/substrate systems investigated. The indentation was performed using a Rockwell C tester and the damage of the coatings was classified from HF 1 to HF 6 according to DIN report 39 [9]. The results of this indentation test are shown in Table 2 together with the chemical composition and the coating thickness. All three coatings investigated have comparable thicknesses. The TiN coating deposited onto the high speed steel substrate shows some areas of adhesive delamination around the indentation cavity and the adhesion is thus classified as HF 3. As compared to un nitrided high speed steel samples [10], an improvement in adhesion for TiN coatings was reached using substrate nitriding as pre-treatment. The results of the VDI indentation test for Ti(C,N) and Ti(B,N) coatings are also shown in Table 2. Ti(C,N) shows an improvement in adhesion behaviour which might be attributed to the lower elastic modulus of Ti(C,N) coatings with respect to TiN [10]. The adhesion behaviour of Ti(B,N) is comparable to the TiN coating and is also classified as HF 3.

The VDI indentation tests only describe the static adhesion behaviour. To describe the fatigue damage after a certain number of load cycles, additional fatigue tests were performed. Figure 1 shows SEM micrographs of the damage condition of the three coatings investigated after $2 \cdot 10^5$ cycles at a load of 80 kN. TiN exhibits cracks parallel to the contact zone. Around these cracks the coating was squeezed but no exfoliation of the coating from the steel substrate was determined (see Figure 1a). Nitriding prior to deposition has been shown to yield a superior fatigue behaviour [10] which is in good agreement with the VDI indentation tests. The fatigue behaviour of Ti(C,N) and Ti(B,N) coatings on hot-working steel is shown by the SEM micrographs in Figure 1b and Figure 1c. The Ti(C,N) coating has a superior fatigue behaviour. There are only cracks parallel to the contact zone but no exfoliation was determined after $2 \cdot 10^5$ cycles. This behaviour is in good agreement with the results of the VDI indentation tests (see Table 2) where the Ti(C,N) coating yields a higher adhesion strength quality number than TiN. Ti(B,N) shows a larger area of damage around the parallel cracks compared to TiN and small areas of the coating are spalled off.

3.2 Adhesive and abrasive wear behaviour

For utilising coatings to protect tools from early failure, not only good adhesion at the interface but also good wear resistance is required. Therefore, the tribological properties of the coatings have been investigated, i.e. the frictional behaviour as well as adhesive and abrasive wear. Hardness and abrasion coefficients of the coatings investigated are listed in Table 2.

The friction behaviour of the three different coating systems is shown in Figure 2. The friction curve for TiN shows a characteristic behaviour typical for ball-on-disc testing. After a short running-in period with a friction coefficient of about 0.7, the friction coefficient drops down to a value of 0.17. In this running-in period, the friction is mainly a result of ploughing and polishing of the surfaces of the two partners by asperities [11]. After about 80 m of sliding distance, the friction coefficient reaches a steady-state value of about 0.17 where it
remains constant during the whole test period. It should be noted here that this value is significantly lower than those reported in the literature (e.g., 0.5 ... 0.85 for CVD TiN [12]) and those obtained in the authors laboratory for sputtered TiN coatings, which are in the range of 0.6 ... 0.8. The reasons for these low friction coefficients are not clear up to now, probably the chlorine content of the coatings (see Table 2) affects the contact conditions in pin-on-disc testing.

Figure 1: SEM micrographs of the fatigue failure after 2 \( \times 10^5 \) cycles using a load of 80 kN and a test frequency of 20 Hz. (a) TiN, (b) Ti(C,N) and (c) Ti(B,N)

Figure 3 shows an optical image of a wear track on a TiN coating obtained by 3d profilometry. The wear track on this coating shows a characteristic profile. In the centre the roughness is higher than at the border zones of the track. Both sides of the wear track show deeper areas with smoother surfaces as compared to the centre. The deeper areas could be attributed to the formation of wear debris during the sliding contact and, consequently, increasing abrasive wear. The friction curve of the Ti(C,N) coating shows a behaviour which is similar to TiN (see Figure 2). After a running-in period at about 0.7 and a sliding distance of 200 m, the friction coefficient decreases to 0.2 ... 0.25. This value keeps constant during the whole test period. In this section small fluctuations of the friction coefficient can be seen, probably caused by a higher density of wear debris as a result of the higher coating hardness (compare Table 2). One reason for the longer running-in period could be the higher hardness of the Ti(C,N) in comparison to the TiN coating, so that longer sliding distances are necessary to polish initially rough surfaces. The running-in period of the Ti(B,N) coating is only about 35 m which could be related to the lower initial roughness of this coating as compared to TiN and Ti(C,N) [13]. Then, the friction coefficient drops to a steady-state value of about 0.23 without significant fluctuations.

Figure 2: Friction coefficients of TiN, Ti(C,N) and Ti(B,N) coatings against 100Cr6 balls at a load of 10 N and a sliding speed of 10 cm/s

Figure 3: Optical 3d image of a wear track on a TiN coating

A typical surface of the 100Cr6 ball after sliding contact is shown in Figure 4. The sliding direction can clearly be detected. On both sides of the wear track, the surface is smoother as in the centre. This is in agreement with
the 3d image of the TiN coating (compare Figure 3). The abrasive wear coefficients of the three different coating systems were determined using a small-scale abrasion test and an abrasive SiC slurry. The values of the coefficients are listed in Table 2. For TiN, the abrasion coefficient shows a value of \(1.33 \times 10^{-12} \text{ m}^2/\text{N}\) and is almost twice as high as for Ti(B,N). The abrasion coefficient of Ti(C,N) is compared to the two other coatings extremely low. There is an obvious correlation between the abrasion coefficients and the coating hardness [11] (see Table 2).

Figure 4: Optical 3d image of a 100Cr6 ball after sliding against a TiN coating

4 CONCLUSIONS

TiN, Ti(C,N) and Ti(B,N) coatings have been investigated with regard to their adhesion, fatigue and tribological behaviour. Observations of the coating surfaces after fatigue and adhesion tests show a good correlation between adhesion and fatigue behaviour. Substrate nitriding as pre-treatment improves the adhesion behaviour significantly. Ti(C,N) shows an improvement in adhesion and fatigue behaviour which might be attributed to the lower elastic modulus with respect to TiN. All three coatings investigated show similar friction curves. After a running-in period they reach a steady-state value with extremely low friction coefficients of about 0.2. The duration of the running-in period might be a result of coating hardness and initial roughness. The abrasion measurements show the expected correlation with the hardness. The abrasion coefficient of the superhard Ti(C,N) coating (>5000 HV0.01) is compared to the two other coatings superior.

Although in this study the Ti(C,N) coating yielded the best values in the tribological tests applied, the real application performance of coatings is also determined by other parameters like toughness or oxidation resistance. Consequently, also TiN as well as Ti(B,N) coatings deposited by PACVD have been proven to increase the lifetime of tools in a variety of applications.

5 ACKNOWLEDGEMENTS

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6 REFERENCES


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<tr>
<th>Coating</th>
<th>Thickness [µm]</th>
<th>Chemical composition [at.-%]</th>
<th>Hardness [HV0.01]</th>
<th>Adhesion</th>
<th>Abrasion coefficient [m²/N]</th>
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<tr>
<td>TiN</td>
<td>3.5</td>
<td>46% Ti, 50.5% N, 3.5% Cl</td>
<td>2700</td>
<td>HF 3</td>
<td>1.33 \times 10^{-12}</td>
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<tr>
<td>Ti(C,N)</td>
<td>2.6</td>
<td>43% Ti, 25% C, 29.2% N, 2.8% Cl</td>
<td>&gt; 5000</td>
<td>HF 2 . . . HF 3</td>
<td>0.08 \times 10^{-12}</td>
</tr>
<tr>
<td>Ti(B,N)</td>
<td>3.4</td>
<td>42.8% Ti, 7.3% B, 44.1% N, 5.8% Cl</td>
<td>4500</td>
<td>HF 3</td>
<td>0.68 \times 10^{-12}</td>
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Table 2: Summary of thickness, chemical composition, hardness, adhesion and abrasion coefficient determined for the coatings investigated.