Evaluation of selected properties of coatings in respect of risk elimination of surface damage of stamped parts

Tkáčová J.¹, Zdravecká E.¹, Evin E.¹, Tomáš M.¹, Jakubéczyová D.²

¹ Technická Univerzita v Košiciach

² Ústav materiálového výskumu SAV, Košice

E-mail: jana.tkacova@tuke.sk

For achieving a long service life of stamping dies for new types of steel sheets intended for the automotive industry, the tribosystem must be optimally designed. One of the possible solutions is its optimization by coatings. More modern coating technologies for stamping dies include PVD (physical vapor deposition) and CVD (chemical vapor deposition) methods. The properties of PVD TiCN and DLC (diamond-like carbon) coatings deposited on ledeburitic high-alloy Cr–Mo–V steel were analyzed by standard tests (coating adhesion by both the scratch and Rockwell C tests, microhardness by Vickers method and coating thickness by Calotest) and developed tribometer of the “friction jaw-roller” type at drawing double-sided hot-dip galvanized steel sheet. The obtained results showed better tribological behavior of the DLC coating.

INTRODUCTION

In the case of sheet metal stamping dies, it is an effort to apply thin coatings to increase the die lifetime and also to improve the stamping process [1,2]. The coating, as a contact layer on the stamping die, is applied to provide the resistance to oxidation, abrasion, and wear [3]. The coatings for the deep drawing dies are subjected to problems of complex wear, fatigue, and lubrication, in addition to thermal exposure (up to 400 °C). More modern coating methods for thin films include physical and chemical deposition techniques (PVD, CVD). PVD coating technique includes a wide range of materials such as metals, alloys, and ceramics that can be applied to most materials with a wide range of shapes. Examples of PVD coatings are CrN, TiN, TiCN, DLC, etc. [4–8].

The wear of the stamping dies occurs due to slipping movement and usually is connected with adhesion, abrasion, transfer phenomena as well as micro-cracks of the brittle surface; such damage deteriorates the quality of the stampings. The properties of coatings (such as layer thickness, hardness, friction coefficient, adhesion, cohesion, wear resistance) can be evaluated by various experimental methods [9]. Over the years, many standard and modified tests were applied to evaluate tool materials and coatings in sheet metal stamping processes.

The basic requirement for laboratory tests is to model the real conditions of tribosystem (load, temperature, environments, etc.). The coating/substrate interface is the weakest link that needs to be studied in connection with the system's adhesive properties. Scratch test and Rockwell C indentation test are frequently used to study the adhesion of coating/substrate systems.

The coefficient of friction is an important input parameter in sheet metal stamping simulations. The good adhesion of the coating prevents it from peeling off, delamination or tearing off under frictional forces. The friction coefficient varies during the stamping process, and frictional behavior depends on many parameters (e.g. velocity, load, material, etc.). The friction coefficient and wear resistance (adhesion, abrasion, ...) can be identified by various tribometers i.e. with the point, line, and surface contact e.g., pin-on-disc or modified cup drawing tests (strip drawing test, limiting dome height test, stamping lubricant tester). There are more experimental settings for strip-drawing testers, including cylindrical beads, rollers, and flat jaws [10–14].

DLC coatings represents an attractive group of materials with high hardness, high abrasion protection, low friction losses, high sliding speeds, even without lubrication [15,16].

TiCN coating is suitable for cold forming applications of steel and other non ferrous alloys and shows effectiveness in reducing galling in forming galvanized advance high strength steel [17,18]. TiCN coatings are known for their self-lubricating properties [19].

This contribution deals with evaluation of the properties of PVD TiCN and DLC (diamond-like carbon) coatings deposited on ledeburitic high-alloy Cr–Mo–V steel by standard tests (coating adhesion by both the scratch and Rockwell C tests, microhardness by Vickers method and coating thickness by Calotest) and originally designed tribometer of the “friction jaw-roller” type at drawing double-sided hot-dip galvanized steel sheet. Designed tribometer allows investigating the behavior of the drawn strip under different loads, different lubrication modes, and stress conditions.

EXPERIMENTAL

Materials

Friction tests were carried out on hot-dip galvanized steel sheet H220PD+Z100MBO with dimensions of $50 \times 1700 \times 0.8$ mm. It is micro-alloyed steel with a higher strength determined for cold stamping. The mechanical properties determined in a uniaxial tensile test according to the EN ISO 6892-1: 2016 by tensile machine TIRATEST 2300 were as follows: the yield strength of $R_{p0.2} = 223$ MPa, the tensile strength of $R_m = 368$ MPa, ductility $A_{80} = 38$ %. A zinc coating of 100 g m^{-2} on both sides corresponds to $7 \text{ }\mu\text{m}$ coating on each side.

Lubricant Anticorit Prelube 3802-39S was used in the experiments. It is a barium-free, thixotropic lubricating oil preventing corrosion, suitable for deep drawing of steel sheets (conventional steel sheets, galvanized sheets, sheets with or without phosphate layer) to ensure their optimum performance for demanding applications. Viscosity at $40 \text{ }^\circ\text{C}$ is $60 \text{ mm}^2 \text{ s}^{-1}$, density at $15 \text{ }^\circ\text{C}$ is 910 kg m^{-3} , flash point is $180 \text{ }^\circ\text{C}$. Sheets coated with prelubes prior to dispatch from steel mills can undergo difficult pressing operations without additional lubrication [20].

In the presented work, two types of PVD coatings, TiCN and DLC, deposited on ledeburitic high-alloy Cr–Mo–V steel, were compared. Multi-layered PVD–TiCN coating was deposited by the reactive arc evaporation method. The PVD–DLC coating was deposited by the sputtering method.

The jaws and roller of friction testing equipment used in the experiments were made of 1.2379 steel (EN 1.2379, X153CrMoV12). It is high-alloyed ledeburitic Cr–Mo–V steel, the strength ranges from 750 to 800 MPa, the hardness after soft annealing is max. 250 HB and hardness after quenching reaches 63 HRC. The functional parts of testing equipment (jaws and roller) were made as follows: (i) uncoated, (ii) PVD–TiCN coated, and (iii) PVD–DLC coated.

Methodology

The thickness of coatings was determined by Calotest. The HV 0.1 microhardness of the PVD coated jaws was measured by microindentation hardness tester LECO LM 700 AT at a load of 100 g. Evaluation of coating adhesion was carried out by the Rockwell C indentation and the scratch test [6, 21–23].

The Rockwell C indentation test was carried out on both (top and bottom) friction simulator jaws coated with PVD TiCN and DLC coatings. The quality of the coating was evaluated according to the resulting imprint produced by the C diamond conical indenter at a load of 1500 N by an optical microscope with a focus on the peripheral geometry. The coating evaluation was performed according to the scale expressed in HF1 – HF6 degrees (Tab. 1).

The scratch test was performed on the coated jaws in the longitudinal and transverse directions under standard conditions (constantly increasing normal force F_n from 0 to 100 N, speed of the jaw $v = 0.1 \text{ mm s}^{-1}$, the jaw path of $s = 5 \text{ mm}$). Acoustic emission AE and coefficient of friction COF in dependence to the normal force F_n were recorded, and also the surface morphology of coatings TiCN and DLC was observed after test. Based on the knowledge of the load rate and sample feed rate and the

Fig. 1. Friction simulator

Tab. 1. Assessment of adhesion by the Rockwell C indentation

Evaluation of coating adhesion by the Rockwell C indentation test
HF1 – good adhesion, a small amount of cracks		HF4 – reduced adhesion, peeling around the edge of the indent
HF2 – satisfactory adhesion, small peeling between cracks		HF5 – insufficient adhesion, peeling even at greater distances from imprint
HF3 – reduced adhesion, peeling over more than two cracks		HF6 – unsatisfactory adhesion, complete peeling around the indentation

dependence of the acoustic emission signal (AE) values on the magnitude of the normal load F_n on the indenter, the magnitude of the critical load F_c is determined. The scratch test output is a graphical representation of the coating failure process. The scratch test was carried out on a Bruker UNT TriboLab. The surface of the jaw after a scratch test was evaluated by SENSO FAR PLU NEOX optical 3D surface profiler.

The friction coefficient was determined by the originally designed friction simulator, which allows simulating the deep-drawing process – Fig. 1 [24,25]. This modified friction simulator of the "friction jaws-roller" type (designed for TIRATEST 2300 tensile machine) enables to model load of contact surfaces in two modes:

- under blank holder using a simulator with rotating roller ($f_3 = 0$);
- also on the die drawing edge by using simulator with fixed roller ($f_3 > 0$), where f_3 is friction coefficient on the drawing edge.

The jaws of friction simulator were uncoated and coated with TiCN and DLC coatings. The sheet metal strips were drawn between the simulator jaws at a constant speed $v = 25 \text{ mm min}^{-1}$. The contact surface is determined by the width and length of the functional surfaces of the jaws. The blank holding force was: $F_p = 2 \text{ kN}$, 4 kN , 6 kN , and 8 kN . The path of sliding was: $s = 50 \text{ mm}$. During experiments, the drawing force was in the range of $1\text{--}10 \text{ kN}$ depending on the jaws used, the lubricant and the blank holding force. The surface of drawn strip was lubricated with lubricant Anticorit Prelube 3802-39S. The roughness of top and bottom jaws and roller was $R_a = 0.4 \text{ }\mu\text{m}$. After the tests, microscopic and macroscopic analyses were carried out on the drawn strips and jaw surfaces. The surface of the jaws and the drawn strip were monitored using a Plugable USB2-Micro-200X USB digital microscope attached to a computer assembly and Olympus GX71 light metallographic microscope.

In the strip drawing test, the friction coefficient is calculated conventionally by equation 1, considering friction between strip (top jaw) and the strip (bottom jaw) of the same value:

$$f_1 = \frac{F_t}{2 \cdot F_p} \quad (1)$$

where: f_1 – coefficient of friction, F_t – drawing force [N], F_p – blank holding force [N].

The calculation of the coefficient of friction, according to equation (1), does not take into account the bending of the strip at the die drawing edge. For this reason, this method of determining the coefficient of friction is not accurate, and it was necessary to introduce the bending force of the strip.

In the modified friction simulator used, the die drawing edge is replaced by a roller that can rotate and also be blocked. If the roller is blocked, friction is modeled on the die drawing edge. If the roller rotates, the friction is simulated among the die, strip, and the blank holder. These states can be described by equilibrium conditions as follows:

If the roller is braked (friction model on the die drawing edge), the drawing force F_t can be determined as follows:

$$F_{t(f>0)} = e^{\vartheta f_3} \cdot T + F_o \quad (2)$$

where: ϑ – angle of wrap, f_3 – coefficient of friction on the drawing edge of roller, T – friction force on contact surface [N], F_o – bending force on die drawing edge [N].

If the roller rotates (friction model under the blank holder), the friction coefficient on the roller is $f_3 \approx 0$, and we get the equation:

$$F_{t(f=0)} = e^{\vartheta f_3} \cdot T + F_o = T + F_o \quad (3)$$

The resulting friction coefficient on the die drawing edge and between the jaws can be determined from the difference in the drawing forces at different blank holding forces (2 kN , 4 kN , 6 kN , 8 kN) in relation to the reference force of 2 kN generated by the braked roller $F_{t(f>0)}$ and the frictional force at the rotating roller $F_{t(f=0)}$.

$$F_{t=2\text{kN}} = e^{\vartheta f_3} \cdot T + F_o \quad (4)$$

$$F_{ti} - F_{t=2\text{kN}} = 2e^{\vartheta f_3} \cdot f_2 \cdot F_{pi} + F_o - 2e^{\vartheta f_3} \cdot f_2 \cdot F_{p=2\text{kN}} - F_o \quad (5)$$

where: f_2 is coefficient of friction between sheet metal strip and upper and lower jaws.

After arrangement of equation (5) we receive:

$$f_2 = \frac{F_{ti} - F_{t=2\text{kN}}}{2 \cdot e^{\vartheta f_3} (F_{pi} - F_{p=2\text{kN}})} \quad (6)$$

Assuming the friction coefficient on the drawing edge of the roller $f_3 \approx 0$, the equation (6) after arrangement will be:

$$f_2 = \frac{F_{ti} - F_{t=2\text{kN}}}{2 \cdot (F_{pi} - F_{p=2\text{kN}})} \quad (7)$$

The value of the friction coefficient f_3 on the drawing edge of roller (the friction in bearings is so small, that it can be neglected) can be calculated after substitution the value for f_2 and the wrapping angle $\vartheta = 90^\circ$ according to the following modified equation:

$$f_3 = \ln \left(\frac{F_{ti} - F_{t=2\text{kN}}}{2 \cdot f_2 \cdot (F_{pi} - F_{p=2\text{kN}})} \right) \cdot \frac{2}{\pi} \quad (8)$$

The friction coefficient for the individual loads was calculated using the Excel software based on the measured and recorded both the drawing and blank holding forces.

RESULTS AND DISCUSSION

The thickness of the TiCN coating measured by the Calotest ball-crater test was $3 \mu\text{m} \pm 0.7$. The thickness of the DLC coating measured by the Calotest was $2.5 \mu\text{m} \pm 0.5$.

The average microhardness values were as follows: $1196.8 \pm 84.9 \text{ HV0.1}$ for TiCN coating; for DLC coating $1416.1 \pm 188.4 \text{ HV0.1}$; for base material 360.9 ± 26.87 . Based on the measured values, the PVD DLC coating showed higher hardness values.

a) Coating TiCN (lower jaw)

a) Coating TiCN (lower jaw)

b) Detail of the impression in its top part

b) Detail of the impression in its top part

c) Detail of the impression in its lower part

c) Detail of the impression in its lower part

Fig. 2. Characteristic of the adhesion of DLC coating deposited on EN 1.2379, X153CrMoV12 steel substrate, determined by Rockwell C test

Fig. 3. Characteristic of the adhesion of TiCN coating deposited on EN 1.2379, X153CrMoV12 steel substrate, determined by Rockwell C test

A detailed view on the Rockwell impression of PVD DLC coating is documented in Fig. 2. Microscope images showed relatively small peeling of coating at the edges of the imprints, marked as HF2 degree, what can be explained by the amorphous nature of DLC coating. Similar results for DLC coatings were reported in [15].

The result of the Rockwell C adhesion test for multiple-layered TiCN coating, presented in Fig. 3, con-

firmed a good adhesion of HF1 degree, without peeling. This can be attributed to its lower hardness, structure (toughness), higher thickness compared to the DLC layer.

Scratch test records and scratch tracks for the TiCN coating are shown in Figs. 4 and 5.

Scratch test records and scratch tracks for the DLC coating are shown in Figs. 6 and 7.

Fig. 4. Scratch test: TiCN coated jaw – longitudinal direction: a) acoustic emission signal and COF versus normal force; b) scratch track on TiCN coating

Fig. 5. Scratch test: TiCN coated jaw – transverse direction: a) acoustic emission signal and COF versus normal force; b) scratch track on TiCN coating

Fig. 6. Scratch test: DLC coated jaw – longitudinal direction: a) acoustic emission signal and COF versus normal force; b) scratch track on DLC coating

Fig. 7. Scratch test: DLC coated jaw – transverse direction: a) acoustic emission signal and COF versus normal force b) scratch track on DLC coating

The critical force of coatings failure F_c is recorded in Tab. 2.

Tab. 2. Scratch test – critical force F_c at coating failure

Adhesion of coating – scratch test		
Direction of grinding:	PVD TiCN – jaw	PVD DLC – jaw
longitudinal	$F_c \approx 17$ N	$F_c \approx 42-45$ N
transverse	$F_c \approx 5$ N	$F_c \approx 45$ N

Based on evaluating the course of the acoustic emission dependence (AE) on normal force magnitude, it can be concluded that they are consistent with the morphology of the failure. For a harder DLC coating, failure in a brittle manner and a few spallations were observed during the scratch test. DLC coating presented high critical loads during the scratch test (42-45 N) compared to TiCN coating (5 N for transverse a 17 N for longitudinal direction). Lower adhesion recorded for TiCN coating in the scratch test can indicate the impact of the preparation of the substrate and pre-treatment cleaning process. By comparing TiCN and DLC coatings deposited on ledeburitic high-alloyed Cr–Mo–V steel, the results of the scratch test showed a better adhesion of the DLC coating.

Problems with DLC coatings may occur due to residual stress problems, the higher levels of sp^3 bonds

lead to a harder coating but at the expense of higher pressure stresses (Morshed et al., 2003, Schwan, 1997). The internal pressure stresses in the DLC coatings can reach up to 10 GPa, which dramatically limits the coating thickness and such stresses may create unstable interfaces (e.g. the adhesion and/or cohesive problems) and the film may be susceptible to delamination [26].

Due to the better tribological properties of the DLC coating compared to the TiCN coating, the modified strip drawing test was carried out for DLC coated jaws. The calculated values of friction coefficients from the measured values of both drawing and blank holding forces according to equations (7) and (8) are given in Tab. 3.

Low friction coefficient values indicate good tribological properties of the DLC coating [6]. The view on the jaws coated with DLC coating after the modified friction test (blank holding force 8 kN) is shown in Figure 8. There no sticking of Zn coating on the jaws was observed, which corresponds with the low value of the coefficient of friction.

Fig. 8. View on the functional part of jaws after friction test (dimensions of functional part of jaw: 20 × 50 mm)

Tab. 3. Values of friction coefficient for DLC coating

Characteristics Lubricant Anticorit	Blank-holding force F_p [N]			
	2000	4000	6000	8000
Measured drawing force F_t [N] (unbraked roller)	607	961	1268	1327
Friction coefficient f_2 calculated according to equation (7)	–	0.089	0.083	0.060
Measured drawing force F_t [N] (braked roller)	716	1124	1450	1555
Friction coefficient f_3 calculated according to equation (8)	–	0.090	0.067	0.097

Note: Standard deviation were as follows: $F_t \pm 23$ N, $F_p \pm 50$ N, f_2 and $f_3 \pm 0.003$

The DLC coating has confirmed its suitable tribological properties, but attention should be given to the preparation of the substrate. Calculated values of the friction coefficient for DLC coating between jaws and strip were in the range 0.089-0.060 for applied pressures with decreasing tendency. The friction coefficient on the drawing edge varied from 0.097 to 0.067, but dependence on blank holding force was not confirmed. It might be due to different specific pressure, which is higher on the drawing edge, as stated in [24], [27]. As reported in [28], the size of the contact area has a significant influence on the value of the friction coefficient, whereas the rigidity of the test stand is responsible for the qualitative course of the friction curve. It can be assumed that synergistic effect of prelube tribofilm, Zn coated steel and DCL coated jaws will be formed which can explain the low friction of DLC/Zn.

CONCLUSIONS

Nowadays, significant progress has been made in developing new materials and coatings that can provide exposed surfaces of stamping dies with improved tribological properties. The evaluation of coatings properties, such as thickness, hardness, and adhesion, was carried out by standard tests, and for selected coating, the friction coefficient was measured by the modified strip drawing test. Based on experimental works, we can state:

- The hardness of DLC coating (1416.1 ± 188.4 HV0.1) with a thickness of $2.5 \mu\text{m} \pm 0.5$ was about 18 % higher compared to the hardness of TiCN coating (1196.8 ± 84.9 HV0.1) with a thickness of $3 \mu\text{m} \pm 0.7$, both deposited on the tool steel with a hardness of 360.9 ± 26.87 HV0.1.
- Scratch test showed better adhesion of DLC coating compared to TiCN coating.
- Strip drawing test was carried out using a modified testing device with jaws and roller coated by DLC coating. Under lubricated conditions, the friction coefficient under the blank holder varied from 0.089 to 0.060, depending on the blank holding force.
- Under lubricated conditions, the friction coefficient on the drawing edge varied from 0.097 to 0.067, but dependence on the blank holding force was not confirmed.
- Synergistic effect of lubricant Anticorit Prelube 3802-39S, the DLC coated jaws, and Zn coated steel results in a lower friction coefficient.

Thin PVD coatings deposited on sheet metal stamping dies have the potential to optimize the stamping process and to increase the lifetime of the dies. The deposition of suitable self-lubricating coating on

the stamping die may result in the forming process without the application of lubricant. The data obtained on the coatings, as well as the methods of evaluation of adhesion and friction coefficient, contribute to the assessment of the suitability of the surface-treated dies for the stamping processes or to make stamping dies more economical in use. The next research will focus on the study of the friction coefficient in the dry conditions.

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