Evaluation of the friction and wear behavior of a-C:H coatings for lubricant-reduced sheet metal forming

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Abstract. Industrial companies are confronted with increasing demands for sustainability, challenges in the availability of raw materials as well as rising energy and labor costs. Thus, technological measures for improving the ecologic and economic potential of production are required. An important production stage for the manufacturing of metal-based products is sheet metal forming which consists of several production steps. Initially, the semi-finished sheets are lubricated with oil to reduce friction in the actual forming stage and afterwards they have to be cleaned. One approach for the decrease of production costs and increase of sustainability is the avoidance or reduction of oil-based lubricants and hence subsequent cleaning of the sheet metal parts. However, reducing the amount of lubrication leads to higher friction and wear. Consequently, the part quality as well as tool lifetime are significantly decreased. To meet these challenges, surface modifications for the reduction of wear can be applied. In this study, diamond like carbon (DLC) tool coatings were combined with minimum quantities of a solid lubricant on semi-finished parts to analyze the potential of the coatings for beneficial friction conditions. In this context, suitable a-C:H coating systems were needed to be identified. The associated friction and wear behavior was evaluated as a function of the lubricant amount for different variants of semifinished parts. Initially, several a-C:H coatings were generated with varying deposition parameters. Promising variants regarding layer adhesion were analyzed for sheet metal forming in terms of tribological behavior. The resulting coefficients of friction (CoF) and wear resistance were investigated with strip drawing tests. For the comparison of the results over different industrially relevant sheet metals, two aluminum alloys as well as one steel material with different zinc coatings were evaluated. For all materials, the amount of solid lubricant was varied in a range below 0.8 g/m^2 up to a completely dry condition without lubrication.

Introduction

Increasing demands for sustainability and the reduction of dependence on fossil energy are current challenges for the industry. Thus, imports of gas are diversified, and renewable energy is focused for power generation in the European Union [1]. Consequently, energy costs in the industry are increasing. In 2022, the price of electricity in Germany rose by around 50% [2] and the price of gas by 150% [3] compared to 2021. Furthermore, the labor costs are 44% higher than the EU average [4]. To remain competitive as a business location ecologically and economically efficient manufacturing processes are essential. One approach in forming technology is the decrease of lubricant amount to enable a reduction of process steps and resource consumption. Conventional

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steps for processing sheet metal in the automotive sector are lubrication of the sheets, forming, cleaning, joining and coating [5]. By reducing the lubricant amount, the lubrication and cleaning steps become more efficient or can even be omitted. The reduction of lubricant amount can be realized by minimum quantity lubrication or local oiling. However, removing or reducing the lubricant increases friction and wear, which leads to lower component quality and tool lifetime. To meet these challenges, tool modifications can be suitable. These include the application of oxide layers [6], macroscopic [7] and microscopic [8] surface textures as well as DLC coatings [9]. In this study, the tribological behavior of DLC coated tools combined with minimum quantities of solid lubricants on the sheet material are investigated. This should enable the processing of industrial quantities in future applications. For this purpose, a-C:H coatings with and without SiO are deposited with varying coating parameters. Suitable coatings regarding adhesive strength are tested via scratch tests and Rockwell-C indentation tests. Promising coating systems for metal forming are tribologically analyzed with the aid of strip drawing tests. To ensure industry-related conditions three in the automotive sector common materials are used. These include the aluminum alloys AA5182 and AA6014 with the dry lubricant Drylube E1 in quantities of 0.2 to 0.8 g/m^2 . Galvanized DC04 (GI) with and without Prime Lubrication Treatment (PLT) is analyzed as well. This enables the derivation of the required lubricant amount as a function of sheet material and tool coating. The tribological target values are the CoF as well as the surface topography and roughness of the friction jaws and sheets.

Material and methods

Coating deposition. To characterize the DLC coatings, they were deposited to the same substrate material as the friction jaws, tool steel 1.2379, hardened to 60 ± 2 HRC appropriate to the following coating process. The specimens were flat round discs with a diameter of 30 mm and a height of 5 mm. Prior to deposition, the surfaces were fine polished to $R_a \approx 0.013$ µm with a 1 µm diamond suspension (Saphir 550.3, ATM Qness GmbH) and then cleaned in an ultrasonic bath in acetone and isopropanol for 10 min each. An industrial-scale PVD coating unit (TT 300 K4, H-O-T Härte- und Oberflächentechnik GmbH & Co. KG) was used for coating production. For this study, two coating systems with hydrogen-containing amorphous carbon (a-C:H) and alternating SiO-doped (a-C:H:SiO) functional layers were deposited under 2-fold substrate rotation. Doping of the SiO content was realized by evaporation of the liquid precursor hexamethyldisiloxane (HMDSO). Before the actual coating process, the coating chamber was evacuated to high vacuum conditions with an initial pressure below 5.0×10^{-4} Pa and subsequently heated to 250 °C for 40 min. Furthermore, the surface of the specimens was plasma etched and cleaned by argon (Ar^+) ion plasma etching for 40 min working with an argon (Ar purity 99.999 %) gas flow of 500 sccm and a bipolar pulsed bias voltage of **−**500 V (pulse frequency 40 kHz, reverse recovery time 5 μs). Subsequently, a sputtering process was carried out for 3 min with closed shutters to remove impurities from the chromium (Cr) and tungsten carbide (WC) targets (both a purity of 99.9 %) with dimension of 267.5×170 mm. The basic coating architecture was characterized by a thin Cr adhesion layer, a graded CrWC intermediate layer and a WC support layer, which were deposited by PVD using unbalanced magnetron (UBM) sputtering in an argon atmosphere. Afterwards, the different functional layers (a-C:H and a-C:H:SiO) were applied in a Plasma Enhanced Chemical Vapor Deposition (PECVD) process using a mixed Acetylene-Ar (C₂H₂-Ar) plasma. The relevant deposition parameters are listed in Table 1. To determine favorable coating parameters, the a-C:H functional layer was deposited on a Box-Behnken DoE, varying the bias voltage (**−**450 V; **−**525 V; **−**600 V), C2H2/Ar-ratio (156/104 sccm; 202/58 sccm; 220/40 sccm) and substrate rotation speed (1 rpm; 3 rpm; 6 rpm). Based on the Box-Behnken DoE, the best coating with respect to adhesion behavior was identified with the parameters (bias voltage **−**450 V, C2H2/Ar-ratio 220/40 sccm, substrate rotation speed 3 rpm). A second full-factorial DoE was defined for doping with SiO. The a-C:H and a-C:H:SiO layers were deposited alternately and both the HMDSO flow (2 sccm;

5 sccm; 8 sccm) and the layer changes (2, 25, 50) were varied. In all cases, the functional layer was deposited with a coating time of 8580 s and a chamber temperature of 100°C, whereby the time of the alternating layers was adjusted accordingly. For the coating of the friction jaws, one coating from each of the two coating systems was selected based on the adhesion tests.

Table 1. Relevant deposition parameters for the DLC coatings considering the basic coating architecture without the functional layer.

Laver	Power	Pulse frequency	Reverse recovery time	Duration	Chamber temperature	Bias voltage	Ar flow
Cr	5.0 kW	70 kHz	4 µs	240 s	140° C	-100 V	180 sccm
CrWC	5.0 0.3 \nearrow 1.2 kW	40 kHz	$5 \text{ }\mu\text{s}$	1260 s	140° C	-100 V	180 sccm
WC	1.2 kW	40 kHz	$5 \mu s$	1080 s	120° C	-100 V	195 sccm

Characterization of the coatings. The indentation hardness of the coatings was measured by means of microhardness measurement according Oliver and Pharr [10] on a nanoindenter (Picodentor HM500, Helmut Fischer GmbH). A test force of 18 mN was used and 15 indentations were evaluated for both coating systems.

Rockwell indentations (HRC method) were generated (DuraJet 10G5, EMCO-TEST Prüfmaschinen) to characterize the coating adhesion. The adhesive strength was then evaluated under a light microscope (DM4000 M, Leica Microsystems) in accordance with DIN 4856. Five Rockwell indentations were analyzed for each coating variant. In addition, the adhesive strength was evaluated using a scratch test in accordance with DIN EN ISO 20502 with a scratch tester (RTG-2, KTmfk) with a normal force increasing linearly to 100 N over a scratch path of 10 mm. The scratch was optically analyzed with the same light microscope in accordance with the standard and the critical load values *Lc1*, *Lc2* and *Lc3* were determined. Five scratch tests were carried out for each layer variant and the resulting load values were averaged. The statistical evaluation between a-C:H and a-C:H:SiO coatings was performed using the Welch Two Sample t-test in R.

Sheet materials and lubrication. To enable close to industry conditions three common sheet materials and associated solid lubricants were chosen. These include the two aluminum alloys AA5182 and AA6014 with mill finish texture. For both variants, the lubricant Drylube E1 (Zeller + Gmelin GmbH & Co. KG) was used in varying amounts. Tests without tool coating and with completely decreased semi-finished parts were performed as a reference. Additionally, galvanized DC04 with EDT texture was investigated. In this context, variants with and without Prime Lubrication Treatment (PLT) were analyzed. An overview of the investigated sheet materials, lubrication and tool coatings is given in Table 2.

Sheet material	AA5182	AA6014	DC04		
Surface texture	Mill Finish		EDT		
Lubricant	Drylube E1		Prime Lubrication Treatment (PLT)		
Lubricant amount in g/m	0.0, 0.2, 0.4, 0.8		With/without PLT		
Tool coating	Uncoated, a-C:H, a-C:H:SiO				

Table 2. Variation of sheet material, lubrication and tool coating.

Tribological characterization of DLC coatings. Tribological investigations for different combinations of tool coatings, sheet material and lubricant amount were performed using strip

drawing tests, as shown in Fig. 1. The material of the friction jaws is 1.2379, which is hardened to 60 HRC. They have an ideal contact surface of 100×55 mm² with a bevel.

Fig. 1. a) Setup of strip drawing test and surface analysis of b) friction jaws and c) sheets.

The friction jaws were polished to reach a surface roughness of $R_a = 0.003 \mu m$ before they were tested uncoated or with a-C:H coating and a-C:H:SiO coatings. By coating the jaws, the average surface roughness increases to $R_a = 0.012 \,\mu$ m. To avoid a further expensive process step in industrial application, post-processing of the surfaces was not performed. Sheet strips with dimensions $60 \times 500 \text{ mm}^2$ were then clamped with a contact pressure of 3 MPa between two friction jaws and drawn at a constant speed of 100 mm/s, as shown in Fig. 1 a). The tensile force was measured using a load cell so that the resulting CoF can be determined between a drawing length of 100 mm and 170 mm. For the evaluation of the surface, a laser-scanning microscope (VK-X200, Keyence) was used. The topography and roughness measurements were analyzed transverse to the drawing direction. Five strip drawing tests were performed with one pair of friction jaws and evaluated in terms of CoF. Three surface measurements per friction jaw were carried out for each parameter set. The measurements represent one area with smooth and two areas with rough surfaces. Regarding the sheet surface, the fourth and fifth test was analyzed.

Results and discussion

Mechanical properties of the a-C:H and a-C:H:SiO coatings. The determined indentation hardness for the a-C:H coatings averaged with $H_{IT} = 25.8$ GPa and indentation modulus $E_{IT} = 200$ GPa which is similar or even higher compared to literature [11,12]. For the SiO-doped variants $H_{\text{IT}} = 21.5$ GPa and $E_{\text{IT}} = 187$ GPa are measured which is also higher compared to [13,14]. The SiO-doped coatings therefore achieved slightly lower mechanical properties.

Adhesion strength of the a-C:H and a-C:H:SiO coatings. The adhesive strength of all the coating variants examined using Rockwell indentation could be classified between HF2 and HF4. No systematic differences were identified regarding the used coating parameters. There were also no indications that the SiO-doped coatings exhibited better or worse overall coating adhesion than the pure a-C:H coatings. However, some of the a-C:H coatings exhibited larger contiguous flaking in some variants, presumably due to the slightly higher indentation hardness and thus a brittle fracture of coating flakes. Fig. 2 a) and b) show examples of the best coatings for a-C:H (bias voltage **−**450 V, C2H2/Ar-ratio 220/40 sccm, substrate rotation speed 3 rpm) and a-C:H:SiO (HMDSO flow 2 sccm, 50 layer changes) the corresponding Rockwell indentations. These coating systems achieved an adhesive strength class of HF2, which is comparable to literature [15,16]. The results of the scratch tests are shown in Fig. 2 c). Similar to the Rockwell adhesion strength, there were no differences that can be explained by the coating parameters used for the a-C:H and a-C:H:SiO coatings. On the other hand, the SiO-doped coatings differed significantly from the pure a-C:H coatings regarding the critical loads Lc1 ($p = 5.7e-12$) and Lc3 ($p = 0.0006$). Averaged over all

coating systems, the value for the critical load Lc1 was 15.0 N for the a-C:H coatings and 10.9 N for the SiO-doped coatings. At 43.8 N, Lc3 for the SiO-doped coatings was also higher than the value of 37.4 N for the pure a-C:H coatings. In contrast, no difference was determined for the critical load Lc2 ($p = 0.95$); the critical load was 25.6 N in both cases. The increased resistance in the scratch test is assumed to be due to the higher indentation hardness of the a-C:H coatings. In comparison to literature values, it must be considered that these can vary considerably depending on the exact coating system and can depend on the device used and the person evaluating it. However, the values calculated are quite common for a-C:H coating systems [16].

The coatings tested showed a coating adhesion typical for a-C:H coatings in this application [15] with the best variants of HF2 in the Rockwell test. As the coating adhesion seemed to be most important for the chosen application, the best a-C:H and a-C:H:SiO coatings in terms of adhesion were selected for further deposition on the friction jaws.

Fig. 2. Exemplary Rockwell indentations of the best a) a-C:H (HF2) and b) a-C:H:SiO (HF2) coatings and c) results of the scratch tests with n = 13 for a-C:H and n = 9 for a-C:H:SiO.

Characterization of friction. As a reference, tests without tool coating and lubrication were performed for both aluminum alloys. As expected, the CoF showed high values of $\mu = 0.41$ for AA5182 and μ = 0.55 for AA6014. Due to high abrasion together with adhesion and thus plastic deformations of the strips, only one valid test could be performed for AA6014. Analogous to the studies of Krachenfels et al. [17], AA6014 seemed to exhibit a higher adhesion tendency under dry conditions. According to Fig. 3 a significant decrease of the CoF could already be reached by the smallest lubricant amount of 0.2 g/m² Drylube E1. The CoF was then reduced to $\mu = 0.11$ for AA5182 and μ = 0.12 for AA6014. With increasing lubrication, the friction reduced continuously down to μ = 0.03 for AA5182. Maximum values for CoF were detected for a-C:H:SiO coatings and AA6014 sheets, which might be explained by the higher surface roughness of the semifinished parts, as shown in Fig. 6. Consequently, the higher roughness of the tools and the sheets led to an enhanced interlocking of the surfaces and thus higher CoF. For galvanized DC04 the friction was significantly lower under dry conditions than for both aluminum alloys. In the case of uncoated friction jaws, the CoF was $\mu = 0.12$. This might be attributed to the zinc coating that was already observed by Krachenfels et al. [17]. They assumed that the hexagonal densely packed lattice structure of zinc exhibited a lower adhesion tendency than the cubic face-centered lattice structure of aluminum. Friction jaws with the a-C:H coating led to a higher CoF of $\mu = 0.16$, which might also be caused by the higher surface roughness. SiO-doped coatings showed a similar friction level as uncoated tools with a CoF of $\mu = 0.12$. Unlike the aluminum alloys, the SiO-doped coatings seemed to reduce friction for DC04. Semi-finished parts with PLT exhibited a slightly lower CoF in general. The lowest value of $\mu = 0.10$ was reached for a-C:H:SiO coatings.

Characterization of the friction jaw surfaces. Initially, the characterization of the topography of the friction jaws was performed. These are shown in Fig. 4 as a function of tool coating and the lowest and highest lubricant amount. a-C:H coated friction jaws had surface defects. Strong adhesion was observed for AA5182 with 0.2 g/m^2 Drylube E1 and uncoated tools. It seemed that there was still an insufficient surface separation. Otherwise, there was no visible wear for AA5182.

Fig. 4. Surface topography of the tested friction jaws as a function of tool coating and lubrication for a) AA5182, b) AA6014 and c) DC04.

AA6014 exhibited a similar effect, whereas there was only a quiet small area with adhesion at the lowest lubricant amount. Tests with DC04 GI and uncoated friction jaws indicated a slightly increased surface roughness. This might be caused by the direct metallic contact without intermediate medium. Since there were almost no visible signs of roughening, the surface roughness is also evaluated, as shown in Fig. 5.

Fig. 5. Surface roughness of the tested friction jaws as a function of tool coating and lubrication for a) AA5182, b) AA6014 and c) DC04.

To derive correlations between the testing conditions and the tool surface a comparison of the roughness with the initial state was necessary. Uncoated friction jaws had an average roughness of $R_a = 0.003$ µm. a-C:H-coated tools showed a roughness of $R_a = 0.009$ µm and $R_a = 0.012$ µm with SiO. For uncoated friction jaws and without lubrication the tests with aluminum led to a high surface roughness of $R_a = 0.127 \mu m$ for AA5182 and $R_a = 0.474 \mu m$ for AA6014 due to strong adhesion. The higher adhesion tendency of AA6014 under dry conditions was clearly visible. By the application of lubricants, a significant decrease of adhesion was achieved. Analogous to Fig. 4a) high surface roughness of $R_a = 0.028 \mu m$ was measured for AA5182 with 0.2 g/m² lubrication. When increasing the lubricant amount to 0.4 $g/m²$ the surface roughness was on a similar level as in the initial state. A sufficient separation of the surfaces seemed to be reached. Using the a-C:H-coated friction jaws, the surface roughness decreased from $R_a = 0.009 \mu m$ to $R_a = 0.005$ µm and showed no significant change with increasing lubricant quantity. Possibly a smoothing of the friction jaws occurred, which was independent of the lubricant amount. The surface roughness of the SiO-doped a-C:H coatings was similar to the initial state. Thus, the lowest lubricant amount led to acceptable surface protection in context of the strip drawing test. A slight increase in surface roughness was observed for AA6014 with uncoated friction jaws. In contrast to AA5182, almost no adhesion was visible at the smallest amount of lubricant, as shown in Fig. 4. According to Zhao et al. [18] AA6014 had a low tendency to build loose wear particles in contrast to AA5182. This might be one reason why the adhesion tendency was lower for AA6014 under lubricated condition. Only slight differences could be found for a-C:H-coated friction jaws compared to the initial state. However, the surface roughness tended to be lower after the strip drawing tests. Due to the high hardness of the coatings only marginal flattening occurred. For a-C:H:SiO coatings no trend was visible for different lubricant amounts. The surface roughness was at the level of the initial state except for 0.4 g/m^2 . Hence there was no significant influence for AA5182 as well, SiO-doped a-C:H layers seemed to be a suitable option to reduce surface roughening for both aluminum alloys. Tests with DC04 without tool coating and lubrication led to an increase of surface roughness up to $R_a = 0.011 \,\mu\text{m}$. This effect could be decreased by the application of PLT on the semi-finished parts, which resulted in values of $R_a = 0.006 \,\mu\text{m}$. Compared to the roughness of the friction jaws for both aluminum alloys under dry conditions DC04 caused a low surface roughening. As already discussed for the CoF the zinc coating might lead to low friction and wear. Variants with coated friction jaws exhibited no significant change in surface roughness. In combination with the low CoF for a-C:H:SiO coatings, provided suitable surface protection of the friction jaws for galvanized DC04.

Characterization of the sheet surfaces. The sheets were evaluated in terms of topography and surface roughness, according to Fig. 6. In initial state the surface roughness of the sheet material was between $R_a = 0.168 \mu m$ and 0.309 μ m. Because of adhesion, for uncoated friction jaws and the lowest lubricant amount the roughness of the AA5182 sheets increased up to 0.715 µm. By increasing the lubricant amount, the sheet roughness decreased. For 0.8 g/m^2 Drylube E1 AA5182 showed only weak adhesion in the topography images and a roughness of $R_a = 0.240 \mu m$. Tests with a-C:H coatings tended to higher values than in initial state, but the difference was not significant. However, roughness peaks seemed to be slightly removed. This was also observed for SiO-doped coatings but with a higher surface roughness of $R_a = 0223 \mu m$ than in initial state. The higher roughness of the friction jaws might be the reason for this. For AA6014 a significantly higher surface roughness was measured in the initial state with $R_a = 0.309 \,\mu m$. No significant change in roughness was observed for uncoated friction jaws with a value of $R_a = 0.335 \mu m$. The surface roughness of coated friction jaws was below the initial value, which meant that the higher hardness of the a-C:H coatings results in smoothing due to abrasion or plastic deformation. This was also indicated by the lack of smoothing on the friction jaws in contrast to AA5182. The analyzed AA6014 sheets with SiO-doped coatings on the tools showed small grooves and adhesion as well as an increased roughness. A higher lubricant amount tended to a lower surface roughness on the sheets. In case of DC04 GI, uncoated friction jaws caused a significant increase in surface roughness from $R_a = 0.198 \mu m$ in initial state to $R_a = 0.331 \mu m$. This could be also seen in the topography images and might be explained by adhesion. By the application of PLT on the semifinished parts, this effect decreased and resulted in smaller values for the surface roughness. a-C:H coated friction jaws might induce strong abrasion and adhesion. It seemed as if the roughness peaks were removed and then accumulated in the roughness valleys and welded together. It is likely that this was due to the zinc layer, which was removed by the harder a-C:H coatings. This effect decreased by the application of PLT on the sheets, which might be explained by the better separation of the surfaces. Furthermore, PLT could impeded the adhesion of already abraded particles. Using a-C:H:SiO coatings small grooves appeared on the surface of DC04 sheets without PLT. Additionally, zinc abrasion and adhesion were significantly reduced compared to the coatings without SiO. When PLT was applied to the sheets, the formation of grooves was inhibited, and the topography was similar to the tests without tool coating. Thus, slight adhesion occurred on the surface. The elimination of excessive abrasion and adhesion could cause the significant lower CoF compared to a-C:H:SiO coatings. In general, the coating of the sheet material with PLT led to less abrasion and adhesion. Therefore, it was well suited as surface protection. Regarding the sheet surfaces, SiO-doped a-C:H coatings were most suitable for galvanized DC04 sheets.

Fig. 6. Surface topography and surface roughness Ra of the tested sheets as a function of tool coating and lubrication for a) AA5182, b) AA6014 and c) DC04.

Summary

In general, the reduction of lubricant amount leads to an increase of friction and wear in metal forming. To decrease this influence, a-C:H coatings with and without SiO doping were applied to tool surfaces. Using Rockwell C indentation, the adhesive strength class HF2 was achieved for both coatings systems. In scratch tests, the selected a-C:H coating showed higher critical loads *Lc1* and *Lc3* compared to a-C:H:SiO. In addition to the tool coatings, dry lubricants were applied to the semi-finished products in small quantities to enable tool lifetimes suitable for large-scale production and thus industrial applications. The selected coatings and lubricant variants were then tested regarding their tribological behavior for forming technology in a strip drawing test. Topography and roughness measurements were used to analyze the surface of friction jaws and semi-finished products. As a reference for the effectiveness of the coatings, all tests were also carried out with uncoated friction jaws. It was found that the application of lubricant resulted in a strong reduction of the CoF for both aluminum alloys tested. The CoF decreased further as the amount of lubricant increased. The use of coated friction jaws led to an increase in friction, which could be due to the higher surface roughness. With DC04, the lubricant only caused a slight reduction in the CoF. However, the values were significantly lower than for aluminum, possibly

due to the zinc coating. The a-C:H coating increased friction; with SiO doping, the CoF was like that of an uncoated friction jaw. Adhesion on uncoated friction jaws was observed for all materials. a-C:H-coated tools tended to be slightly smoothed, while no significant change in roughness was measured for SiO-doped friction jaws. Similar tendencies were found for the sheet surfaces. In general, adhesion and thus the amount of lubricant could be reduced by coating the friction jaws. When doped with SiO, abrasion was also reduced. Consequently, this variant appeared to be the most suitable for all tested materials.

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