PRODUCTION PROCESS

Analysis of the friction behavior of DLC in warm bulk forming by using the ring compression test

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Abstract The reduction of die wear is an effective way to decrease costs within bulk forming processes. Therefore, specific tool materials and heat treatments as well as special coatings are used to prolong the lifetime of the tools. Diamond-like carbon (DLC) coatings show high hardness and superior frictional behavior. However, these coatings seem to be inappropriate for hot forming due to degradation processes at elevated temperatures. But for warm forming, due to the lower temperature input into the cavity, DLC might be an appropriate coating. Friction influences the shear stresses on the cavity surface and is therefore an important factor for reducing die wear. Hence, the analysis of the frictional behavior of DLC coatings within warm forming by using the ring compression test will be presented within this paper. An amorphous hydrogenated carbon coating and six metallic doped amorphous hydrogenated carbon coatings (Cr, V and W each in two variants) are compared to CrN and no coating. Firstly, nomograms are graphed by the use of finite-element-analysis. Thereafter two test series are carried out varying forming temperature and lubrication. The results show that DLC coatings with and without metallic doping are able to reduce friction in warm forming. Within the investigations, an amorphous hydrogenated carbon doped with 15 % chromium shows the

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R. Weirauch · G. Bräuer IOT - Institut für Oberflächentechnik, Technische Universität Braunschweig, Brunswick, Germany URL: http://www.tu-braunschweig.de/iot lowest friction factor and is able to reduce the friction factor compared to no coating by up to 64 % within warm forming.

Keywords Diamond-like carbon (DLC) \cdot Friction \cdot Warm forming \cdot Warm forging \cdot Bulk forming \cdot Ring compression test

1 Introduction

The quality requirements on parts in the automotive industry increased within the past years and this trend seems to be ongoing. In this industry, the quality of a part is mainly defined by high mechanical properties, low tolerances and good surface qualities [1]. Many components, especially high duty power train parts, are produced by bulk forming. To meet the increased requirements the forming processes are enhanced consequently—for example by lowering the forming temperature to warm bulk forming (600–900 °C) which enables, among others, higher accuracies and better surfaces. But lowering the billet temperature increases the stresses in the die during forming and could affect the lifetime of the die negatively due to increased die wear.

Within bulk forming, die wear mainly determines the profitability of the manufacturing process. A reduction of die wear can be achieved by optimizing the material flow within the forming process, but often this reduction is not practical or not achievable at all. Hence, a reduction in die wear can only be achieved by improving the die material or by using special wear reducing coatings. By the application of special coatings, also a reduction of the friction can be achieved to reduce the occurring shear stresses on the tool surface.

2 State of the art

2.1 Loads in bulk forming processes

Hot bulk forming of steel is mainly carried out at temperatures between 1,000 and 1,250 °C [1]. The main advantage of hot forming at these temperatures is the low flow stress of the material that is formed and high achievable deformation degrees. The required forming force and the stresses in the die decrease with increasing forming temperature, but the high temperature results in high thermal loads in the die as well as in scale on the surface of the billet which facilitates abrasive wear on the cavity. Reducing die wear within hot bulk forming is possible for example by nitrating or using coatings containing boron [1, 2]. The tool making process can also influence the lifetime of the tool in hot forming as well as in cold forming [3, 4], e.g. changing the tool making process from turning to eroding can prolong the lifetime of the tool.

Warm bulk forming of steel between 600 and 900 °C, on the one hand, reduces the thermal load of the die and the formed workpieces show higher accuracy and surface quality compared to hot forming. On the other hand the mechanical loads in the tool increase due to a higher flow stress of the formed material. Whereas the temperature of the cavity surface can reach more than 700 °C within hot forming, the thermal input within warm forming is much lower [1]. Hence, a wear protection coating for warm forming only needs to resist lower temperatures compared to a coating for hot forming. Therefore, new coating materials might be appropriate for warm forming processes—one of them is diamond-like carbon (DLC).

2.2 Diamond-like carbon coatings

DLC is very hard and shows low adhesive behavior against many materials. In former times it was mainly used for high technology applications (e.g. magnetic storage media) but now it is gaining more and more interest within common production e.g. engine components or medical applications [5]. Investigations showed that the friction coefficient of DLC against steel is about 0.2 at normal atmosphere [6]. Other coatings like TiN show values of about 0.5. But since the friction behavior of DLC is strongly depending on environmental boundary conditions, within warm forming other frictional behavior may be resulting [6, 7].

Hydrogen-free amorphous carbon (a-C) consists of carbon which is hybridized in sp^2 - and sp^3 -hybridisation [8]. The sp^2 -hybridisation is responsible for the typical lubrication behavior as known from graphite, whereas the sp^3 -hybridization results in a high hardness which is known

from diamonds. Hence, hardness and frictional behavior of the coating are determined by the ratio of sp^2 - and sp^3 -hybridisation [9, 10]. Most industrial DLC-coatings also contain hydrogen (H) which stabilizes the amorphous network of the coatings (hydrogenated amorphous carbon—a-C:H) [6, 8]. Within this paper, the notation DLC will be used only for a-C:H as well as the metallic doped variants.

The main disadvantage of DLC-coatings is their sensitivity to environmental influences such as surrounding atmosphere, humidity and temperature [6]. These influences can increase graphitization and oxidation of the coating. Graphitization means the conversion from sp^3 - to sp^2 -hybridisation which results in reduced hardness. Oxidation occurs when oxygen atoms from the atmosphere bond carbon atoms from the coating which will also result in reduced hardness. To reduce the sensitivity against environmental influences, DLC coatings can be doped by elements like tungsten (W), titanium (Ti), vanadium (V) or silicon (Si).

The application of DLC for forming processes was investigated by several works using sheet and bulk forming processes. Iriyama et al. [11] and Horiuchi et al. [12] analyzed forming of DLC coated magnesium and aluminum sheet and could thereby reduce the needed forming forces. Within sheet metal forming of high strength steel DLC coated tools showed low friction, but unsatisfactory wear behavior [13]. Investigations of DLC coated tools in bulk forming processes have mainly been performed for light metals like magnesium and aluminum alloys, since forming of those alloys is performed at lower temperatures than hot and warm bulk forming of steel. Wank et al. [14] proved the ability of DLC tool coatings for cold forming of aluminum although the resulting forming forces and material take up could not be reduced within their investigations. Matsumoto and Osakada [15] proved that DLC coated tools show less friction in warm bulk forming of magnesium by using the ring compression test. Reisel et al. [16] investigated the capability of DLC as wear protective coating in bulk forming processes of steel up to 500 °C using a forced-in test. The results show that DLC and silicon doped DLC are able to reduce wear on the tool. Further investigations of Reisel et al. [17] using a compression spin test even showed advantages of DLC and silicon doped DLC at temperatures between 500 and 700 °C. Former investigations of the authors could also prove the ability of several DLC doped coatings and not doped DLC to protect the forming tool [18, 19] but since forming processes are strongly influenced by the frictional behavior a detailed investigation on this issue will extend the knowledge on wear performance of DLC. Therefore, in this paper the friction behavior of several DLC coatings will be analyzed by using the ring compression test.





2.3 Ring compression test for measuring the friction factor

The ring compression test recommended by Male and Cockcroft [20] as well as Burgdorf [21] is one of the most common ways to determine the friction behavior for bulk forming processes. To perform the ring compression test only a small press is needed and the friction factors are analyzed by measuring the diameters of the rings. Additionally, nomograms are needed which are usually compiled with finite-element-analysis (FEA). For example Noh et al. [22] show very detailed FEA on the geometry changes during the ring compression test.

The concept of the ring compression test for determining the friction factor is based on the geometrical change of the inner and the outer diameter of a ring during the forming process. The geometrical changes are depending on the friction conditions of the interface between the upper and lower plate and the ring (Fig. 1). A high friction factor will lead to a decrease of the inner diameter, whereas a low friction factor will lead to a smaller decrease or will even result in an increase of the inner diameter.

3 Simulation of the ring compression test

To compile the needed nomograms to determine the friction factor the ring compression test is simulated using SimuFact Forming. The friction factor model is used since within bulk forming processes it is applicable and common to use in FEA in industry and science [1, 22]. The friction factor m is varied between 0.1 and 1 with steps of 0.1 (0.1, 0.2, ..., 1.0) and additionally a friction factor of 0.01 is used to show behavior for very low friction. The rings have the dimensions shown in Fig. 1 and the mesh size of the ring is set to 1 mm. The material data of steel 1.7225 is taken from the software library and the temperature of the rings is set to 600 and 900 °C (upper and lower limits of warm bulk forming), whereas the temperature of the plates is set to 200 °C. The simulations are performed to a maximum height reduction of 55 %.

For a good comparison between the friction factors, nomograms are desirable which show a high and constant sensitivity towards changes of the friction factor. A high sensitivity results in a wide spread of the nomogram. Noh et al. [22] have made very profound investigations using FEA to get the most appropriate nomogram for the evaluation of friction using ring compression test. Noh et al. suggest not to use the most common analysis of the inner diameter d_{i1}, if friction factors of more than 0.3 occur since d_{o2} shows better linearity and sensitivity. Additionally, Noh et al. suggest using curves which plot the change of the contact surface. Within the presented paper, only the dimensional change of the inner diameter di1 will be considered because within the measurements of the experimental trials it is found that this value shows the best sensitivity on changes of the friction factor (chapter "5.1 Identification of the most appropriate diameter"). Therefore nomograms are plotted for the inner diameter d_{i1} by calculating $-\Delta d_{i1}$ at height reductions of $-\Delta h = 10, 20,$ 30, 40, 50 and 55 % (Fig. 2).

4 Experimental tests

4.1 Tested DLC coatings

The bulging plates to form the rings are made of die tool steel 1.2343 with a hardness of 48 HRC. The coated surface is an area of 60 mm diameter which is suitable for forming the rings. Seven DLC-coatings are used for the investigations—additionally a CrN-coated and a not coated plate are used as references. The CrN coating shows equal percentages of chromium and nitrogen. In Table 1 the references and the DLC-coatings used are shown and for DLC-coatings the type of doping is given. The right column shows the abbreviations of the coating which will be used in the following. These abbreviations correspond with [8].

Fig. 2 Nomograms on the basis of the inner diameter d_{i1} for $\theta_{ring} = 600$ and 900 °C



 Table 1 Coatings chosen for investigations of frictional behavior

Туре	Doping	Notation
Not coated	-	Not coated
CrN	50 % of chromium, 50 % of nitrogen	CrN
DLC	None	a-C:H
	15 % of chromium	a-C:H:Cr(15)
	40 % of chromium	a-C:H:Cr(40)
	10 % of vanadium	a-C:H:V(10)
	25 % of vanadium	a-C:H:V(25)
	15 % of tungsten	a-C:H:W(15)
	30 % of tungsten	a-C:H:W(30)

The doping elements chromium (Cr), vanadium (V) and tungsten (W) are chosen because in previous investigations these coatings showed appropriate properties for warm bulk forming processes [18]

4.2 Trial plan and setup

In the test series the ring material is 1.7225 and the rings are lathed to the dimensions $30 \times 15 \times 10$ mm as shown in Fig. 1. A height reduction of $-\Delta h = 50$ % is chosen. The forming temperatures of the rings are oriented to the lower and upper end of warm forming of steel (600 and 900 °C) and tests are performed using water and graphite lubrication as well as no lubrication. In the following, every combination of used coating, temperature and lubricant is named parameter-combination.

For carrying out the investigations the rings are heated up to the required temperature in an electric furnace for about 20 min. The ring temperature is observed by an infrared thermometer. Thereafter, the rings are taken out of the furnace and are formed by accordingly coated bulging plates assembled in a mechanical screw press of Müller-Weingarten. To have a high accuracy in reaching the height, the way of the upper die is limited by blockers which are placed next to the bulging plates. When using water and graphite as lubricant, it is brushed on the plates manually. For each parameter-combination four rings are compressed.

4.3 Procedure of the determination of the friction factor

After forming, the rings are measured at the four characteristic diameters $(d_{i1}, d_{i2}, d_{o1}, d_{o2})$ by the use of an electronic sliding caliper with an accuracy of 0.01 mm. These diameters are measured four times at each ring by an angle of 45° and the average value of the four measurements at the four rings for every parameter-combination is calculated. By using Eqs. 1 and 2, the reduction of height $(-\Delta h)$ and the reduction of the diameters $(-\Delta d)$ are calculated for each parameter-combination (see Fig. 1 for symbols).

$$-\Delta h = \frac{10\text{mm} - h}{10\text{mm}} \quad \text{[referring to 21]} \tag{1}$$

$$-\Delta d = \frac{d_0 - d}{d_0} \quad [referring \text{ to } 21] \tag{2}$$

Equation 2 is adequate for the outer and inner diameters as d₀ denotes 30 mm for the outer and 15 mm for the inner diameters. Since there is only one initial value of the height (10 mm) this value is displayed in the equation. There is a need to calculate $-\Delta h$ since the actual values may differ slightly from the appointed value of 50 %. Hence, every parameter combination has four coordinates ([$-\Delta h$; $-\Delta d_{i1}$]; [$-\Delta h$; $-\Delta d_{i2}$]; [$-\Delta h$; $-\Delta d_{o1}$]; [$-\Delta h$; $-\Delta d_{o2}$]). These coordinates are plotted into nomograms which are simulated at the appropriate temperature (600 and 900 °C) as described in Sect. 3. Thereafter, the friction factor is determined by comparing on the position of the coordinates in the nomogram.

5 Results of the experimental tests

5.1 Identification of the most appropriate diameter

In literature, different opinions exist on the most appropriate diameter to investigate the friction by using the ring compression test [22, 23]. To examine which of the four diameters (d_{11} , d_{12} , d_{01} , d_{02}) shows the best sensitivity on





changes in friction behavior, the mean values of all diameters are measured and calculated within all experimental trials. Thereafter, the values are plotted as a function of $-\Delta h$. In Fig. 3 all calculated values for the change of the diameters at $\theta_{ring} = 600$ °C (lubricated and not lubricated) are displayed. A suitable indicator to characterize changes in friction behavior is determined by a preferably large distance to its baseline value—which is 0 % for all—and a large range at a given change of friction factors. Since the friction factors observed by the given number of coatings within this analysis is fixed, the largest range can easily be identified with the largest range in $-\Delta d$. The range of the values is shown in the brackets in Fig. 3.

The trend of the both outer and the both inner diameters look quite similar but an offset is noticeable. It is obvious that the outer diameters d_{o1} , and d_{o2} show only a very small range of changes in their diameter and therefore both are not suitable to notice differences in friction behavior. The inner diameters both seem to be more appropriate since their values have ranges of 27.6 % (d_{i2}) and 31.2 % (d_{i1}). Hence, d_{i1} shows the largest range of values and also shows the largest change to its baseline value-therefore d_{i1} seems to be most appropriate. At a ring temperature of 900 °C, di1 also shows largest range and largest distance to the baseline value. Another advantage of d_{i1} compared to d_{i2} is that it is much easier to measure when using a sliding caliper and the influence of the gager is smaller than for d_{i2} which at some specimens is hard to detect due to the shallow transition from the abutting faces to the rounding.

5.2 Determination of friction factors

5.2.1 Lubricated ring compression tests

The coordinates for the coatings of lubricated ring compression at 600 °C are plotted in Fig. 4. The rings are formed to about 52 % height reduction which is a little more than targeted. For having a detailed view on the differences of the values, the nomogram is enlarged to the section of the coordinates.

The plate coated with CrN shows the lowest friction factor at 600 °C (m = 0.31). The values of the uncoated plate, a-C:H and a-C:H:Cr(40) show quite similar friction factors ranging from 0.36 to 0.37. Both coatings doped with vanadium show friction factors of about 0.40. Only the values of the tungsten doped DLCs are higher than 0.40.

At a ring temperature of $\theta_{ring} = 900$ °C (Fig. 5) the friction factor of the CrN-coating is much higher and the second highest of all (m = 0.54)—only the value of the not coated specimen shows a higher friction factor (m = 0.57). The friction factor of a-C:H:Cr(40) also shows a very high increase compared to its value at 600 °C. The lowest friction factor can be identified for a-C:H:Cr(15) with m = 0.34.

All determined friction factors for the lubricated trials are summarized in Fig. 6. The bars in the figure depict the mean values and the appropriate standard deviations (SD) are shown by the black lines. The figure outlines that most coatings show a temperature dependency that leads to increasing mean values of the friction factors when increasing the ring temperature. Especially the coatings





Fig. 5 Measured mean values for reduction in height $-\Delta h$ and inner diameter $-\Delta d_{i1}$ for all coatings plotted in a nomogram $(\theta_{ring} = 900 \text{ °C})$

CrN, a-C:H, a-C:H:Cr(40), a-C:H:W(30), and the uncoated plate show a significant increase, since the ranges of the SD are not tangent to each other. The vanadium doped DLCs show a small increase in their mean values. Conversely, the friction factors of a-C:H:Cr(15) and a:C:H:W(15) seem to decrease with increasing ring temperature—but the ranges of the SD show that this decrease is not significant.

Taking into account both temperatures, a-C:H:Cr(15) shows the lowest friction factors of all coatings. Looking at the SD, the results at 900 $^{\circ}$ C show a little smaller SD than

at 600 °C, although the mean values are higher in most cases. Altogether, the mean values of the friction factors for all coatings and both temperatures range between 0.31 and 0.58.

5.2.2 Not lubricated ring compression tests

Figure 7 summarizes the determined friction factors for ring compression tests performed without lubrication which are as expected higher than with lubrication. The





Fig. 7 Summary of calculated friction factors in not lubricated ring compression test (mean value and standard deviation)

lowest friction factor at $\theta_{ring} = 600$ °C is observed for a-C:H:Cr(15) (m = 0.49), whereas the higher chromium doped coating shows the second highest friction factor. A quite similar correlation can be seen for the tungsten doped coatings. The vanadium doped coatings do not show this distinctive behavior as the difference between the friction factors of the doping percentages is only about 0.02.

These tendencies seem to be valid also at $\theta_{\rm ring} = 900$ °C. The difference between the low and high percentage of doping DLC with chromium results in the lowest (m = 0.36) and the second highest friction factors (m = 0.78)—which is quite similarly observed at the trials with lubrication. For the tungsten doped coatings this tendency is less distinctive and the vanadium doped coatings show quite the same friction behavior at both doping percentages. Considering the ring temperature, it is obvious that for all doped DLCs the mean values of the friction factor are decreasing by increasing the ring temperature. But looking at the SD, only at a-C:H:Cr(15) and a-C:H:W(30) this trend is significant. Also the friction factor of the CrN and a-C:H coated plates seem to be slightly decreasing by increasing the ring temperature, whereas the not coated plate shows an intense increase from m = 0.67 to 1.00. Altogether, the values of m without lubrication range from m = 0.36 to 1.00 which is a much wider range than in the lubricated ring compression tests.

5.3 Conclusions

The presented results underline the ability of adjusting the properties—in this paper shown for the frictional behavior—of DLC by doping with metallic elements. The frictional behavior can be adjusted by the selection of the element itself as well as by the percentage of doping. Especially, the results of Cr and W doped DLC without lubrication show that the friction factor can be varied by the percentage of doping—a higher percentage of Cr and W has a negative influence on the friction behavior.

It is observed that most DLC coatings show lower friction factors than CrN or not coated plates when forming without lubrication—only a-C:H:Cr(40) reaches high friction factors at both temperatures. Using a-C:H:Cr(15) a reduction of the friction factor of 48 % compared to CrN and even 64 % compared to a not coated plate could be achieved at a ring temperature of 900 °C. Whereas the not coated plate shows a severe increase, all DLC coatings show a more or less significantly lower friction factor at 900 °C than at 600 °C. Lower friction indicates that there

is a higher percentage of sp^2 hybridized carbon in the coating when forming at 900 °C compared to forming at 600 °C. A higher percentage of sp^2 may be a result of graphitization and oxidation which is known to be affected by the temperature—high temperatures facilitate graphitization and oxidation. But since the percentage of sp^2 increases, the percentage of sp^3 will decrease and this may have a negative influence on the wear properties of the coating, since the sp^3 -hybridized carbon is responsible for the hardness of the DLC coating. This assumption is underlined by the investigations of Steiner et al. [24] who showed that wear increases by increasing the load on the DLC coating-as expected. But it also leads to lower friction. Former investigations of the authors on the temperature effect of DLC using different forming temperatures also emphasize the assumption of the conversion from sp^3 - to sp^2 -hybridization at elevated forming temperatures resulting in worse wear behavior [19].

When using lubrication this dependency of the temperature on the friction inverses for the mean values of all DLCs but a-C:H:Cr(15) and a-C:HW(15). Anyway, considering their standard deviations, those decreases are not significant. The lubrication may protect the coating from graphitization and oxidation at elevated temperatures. On the one hand, the lubrication protects the DLC coating from direct contact to the formed ring and thereby from its highly reactive surface during forming. On the other hand, the lubrication may also reduce the temperature input from the ring into the coating.

Altogether, at a forming temperature of 600 °C in lubricated condition DLC could not show favorable friction properties compared to no coating or CrN. At 900 °C all DLCs show better friction behavior than no coating and CrN. The DLC coating with the best friction behavior [a-C:H:Cr(15)] could reduce the friction factor compared to CrN by 37 % and compared to no coating by 41 %. Hence, DLC coatings could prove their ability to reduce friction in warm forming processes both with and without using lubrication.

6 Summary and outlook

Within the presented paper rings of steel 1.7225 were compressed on plates coated with several types of hydrogenated amorphous carbon coatings as well as CrN and not coated plates as references. These tests were made at ring temperatures of 600 and 900 °C with and without lubrication. A first investigation of the values showed that the inner diameter d_{i1} is most suitable to notice changes in friction and was used for further investigations. For the determination of the friction factor, nomograms have been compiled with the help of FEA. The coordinates ($-\Delta h$; $-\Delta d_{i1})$ were plotted into the nomograms and the friction factors were determined.

The results show higher friction factors for the not lubricated ring compression tests than for the lubricated tests—as expected. Most of the analyzed DLC coatings show lower friction factors than CrN and not coated references—especially when not using lubrication. The DLC which showed the lowest friction factors of all (a-C:H:Cr(15)) could reduce the friction factor compared to no coating by up to 64 %. Even when using lubrication, all DLCs show lower friction factors than both references at a forming temperature of 900 °C. Hence, the analyzed DLCs could prove their ability to reduce friction in warm forming.

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Conflict of interest The authors declare that they have no conflict of interest.

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