Comparison Between WCC/DLC, CrN/DLC and RF Produced DLC Coatings

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Key Words: Diamond-like carbon coatings; MHz and kHz frequencies; Plasma assisted CVD; Industrial coating equipment

ABSTRACT

Recently DLC coatings have proceeded to large scale industrial usage. The two widely applied coatings are: 1) coatings produced by pure PACVD, utilizing e.g. Radio Frequency (13.56 MHz) plasma, and, 2) coatings produced in PVD equipment, utilizing reactive sputtering and switching over to e.g. Mid-Frequency PACVD for the a-C:H top coating. A comparison is made of typical representatives of the two classes. The mechanical properties like hardness, E-modulus, wear resistance, adhesion and the load carrying capability are compared.

INTRODUCTION

Due to the high potential of diamond-like carbon (DLC) coatings [1,2] in many fields of industrial applications (e.g. automotive components) [3], DLC processes were scaled up to industrial coating equipment.

In this paper we will present a comparison of the tribological properties of three different coating types which were deposited on industrial coating equipment. The first layer system is a metal free hydrogenated carbon film (a-C:H) with a silicon doped (a-C:H:Si) interlayer coating. This coating type was produced by a pure PACVD process by using a radio frequency (13.56 MHz) [4] glow discharge. The second and the third layer system were deposited with different supporting layer systems, a metal containing DLC coating (a-C:H:W) and a Chromium nitride coating (CrN) by using a PVD process. The a-C:H top layers were deposited in the same coating machine in a PACVD process by using a mid-frequency (20-100 kHz) [5] glow discharge. All coating types were deposited in a low temperature deposition process below 200°C.

EXPERIMENT DETAILS

The first layer system a-C:H:Si + a-C:H were produced in an IonBond RF PACVD batch coater (chamber volume 400 l), see Figure 1. The second layer system a-C:H:W + a-C:H and the third layer system CrN + a-C:H were deposited in a PVD + PACVD process by using a Hauzer HTC 1500 (chamber volume 1500 l) and HTC 1200 (chamber volume 800 l) industrial batch coater (see Figure 2 and 3).

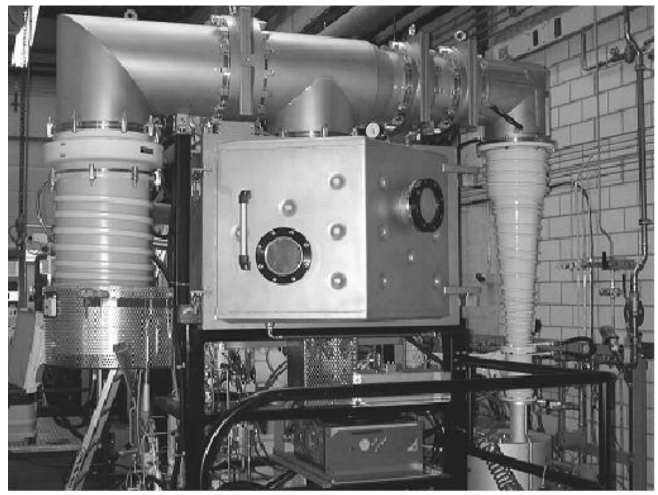

Figure 1. IonBond RF PACVD industrial batch coater.

Figure 2. HTC 1500 PVD/PACVD industrial batch coater.

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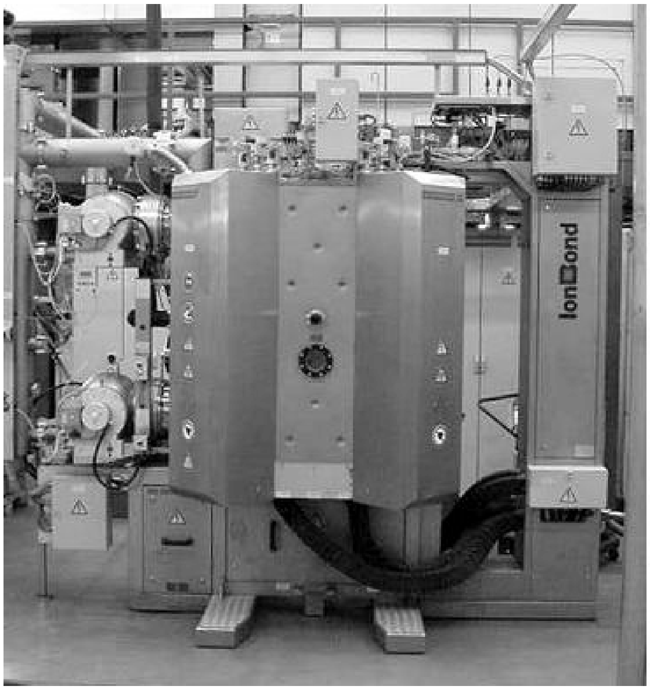

Figure 3. HTC 1200 PVD/PACVD industrial batch coater.

All coatings were applied on polished 100Cr6 (AISI 51200) material ($R_z < 0.1\mu m$) with a substrate hardness of > 60 HRC.

The properties of the applied layer systems were compared by the following different characterization methods.

The film thickness and the abrasive wear resistance were determined by using a ball cratering system (kaloMAX NT, company BAQ) operating with an alumina suspension. To quantify the wear rate results, the volume of the coating removed by the cratering device was divided by the normal force and the track length of the rotating ball. For the abrasive wear experiments a reasonable unit of wear is 10^{-15} $m^3N^{-1}m^{-1}$.

The adhesion properties were classified by Rockwell C indentation (classification between HF 1 for excellent adhesion and HF 6 for large area delamination) and as well by scratch testing (load 0N-70N, LC2 first delamination).

The coating hardness was determined from measurements with a Fischerscope microhardness tester (H 100, company Fischer) and a nanohardness tester (AFM with a Hysitron® measurement system).

The friction coefficients against 100Cr6 balls (AISI 51200) were measured by using a pin-on-disc tester in an ambient atmosphere (normal load of 3 N, 30 rpm, relative humidity approx. 50%, temperature 19°C).

The structure and the chemical composition of the coatings were examined by using a SEM (Zeiss, Leo) and depth profiles of the multilayer systems were measured by SIMS analysis (Cameca).

RESULTS AND DISCUSSION

In this chapter we will compare the tribological properties, structure and layer design of the three different layer systems:

- coating type no. 1: a-C:H:Si + a-C:H (a-C:H top layer: RF, 13.56 MHz);
- coating type no. 2: Cr + a-C:H:W + a-C:H (a-C:H top layer: MF 20 – 100 kHz);
- and coating type no. 3: Cr + CrN + a-C:H (a-C:H top layer: MF 20 – 100 kHz).

FILM THICKNESS AND ADHESION

The total thickness of coating type no.1 was 2.0 µm with a 0.5 µm thick a-C:H:Si adhesion layer and a silicon free a-C:H top layer (see Figure 4). For the adhesion properties we determined HF2, a good adhesion on the 100Cr6 substrate material (see Figure 5).

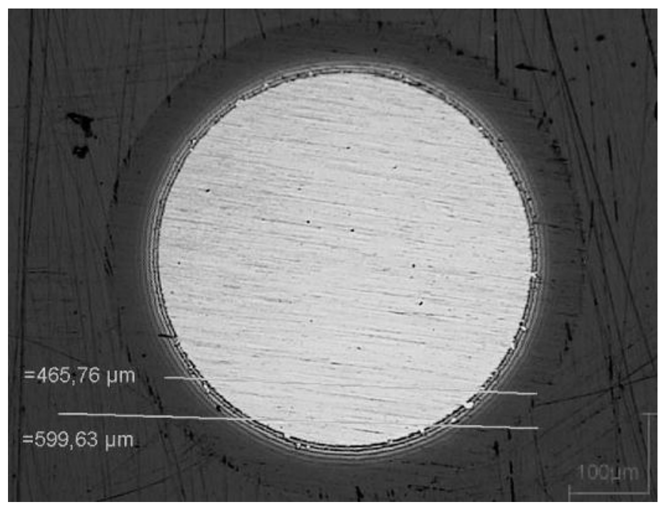

Figure 4. Thickness measurement coating type no. 1 by Calotest.

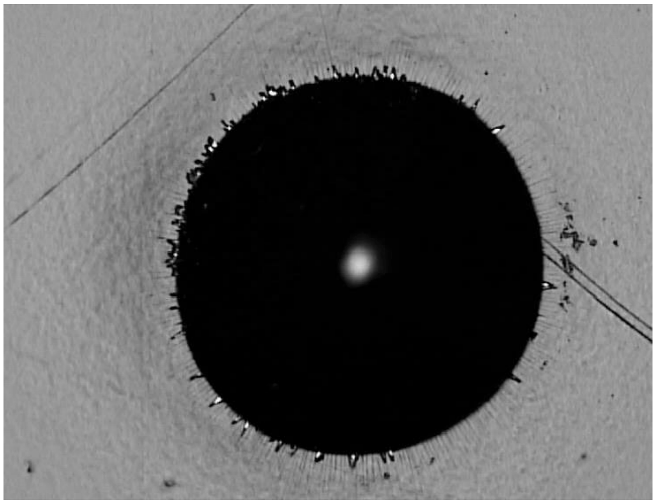

Figure 5. Adhesion test coating type no. 1 by Rockwell C indentation.

For layer system no. 2 we had measured a total thickness of 2.5 µm. 1.0 µm for the Cr + a-C:H:W supporting layer system and 1.5 µm for the metal free a-C:H top layer (see Figure 6). For the adhesion properties we determined HF1, an excellent adhesion on the 100Cr6 substrate material (see Figure 7).

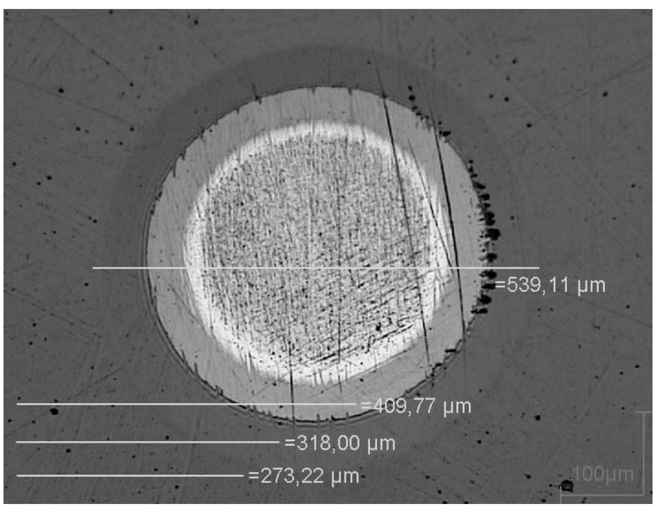

Figure 6. Thickness measurement coating type no. 2 by Calo-test.

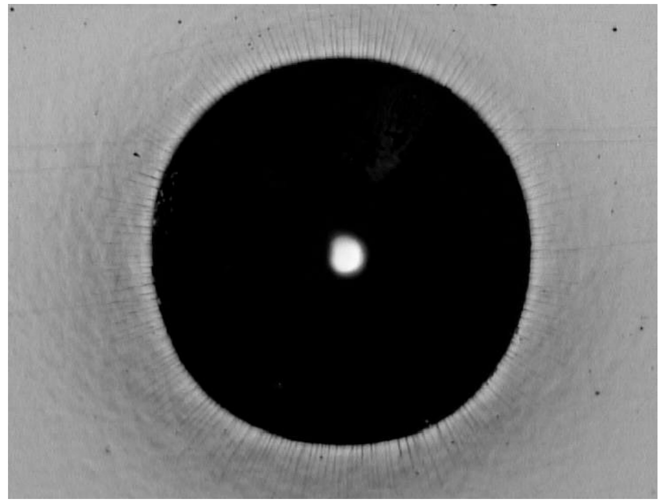

Figure 7. Adhesion test coating type no. 2 by Rockwell C indentation.

The total thickness of coating no. 3 was 3.0 µm. 1.5 µm for the Cr + CrN supporting layer system and 1.5 µm for the metal free a-C:H top layer (see Figure 8). For the adhesion properties we determined HF1, an excellent adhesion on the 100Cr6 substrate material (see Figure 9).

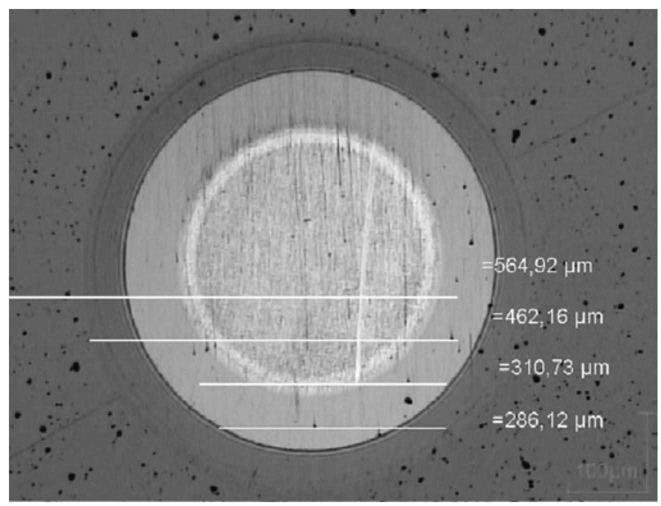

Figure 8. Thickness measurement coating type no. 3 by Calo-test.

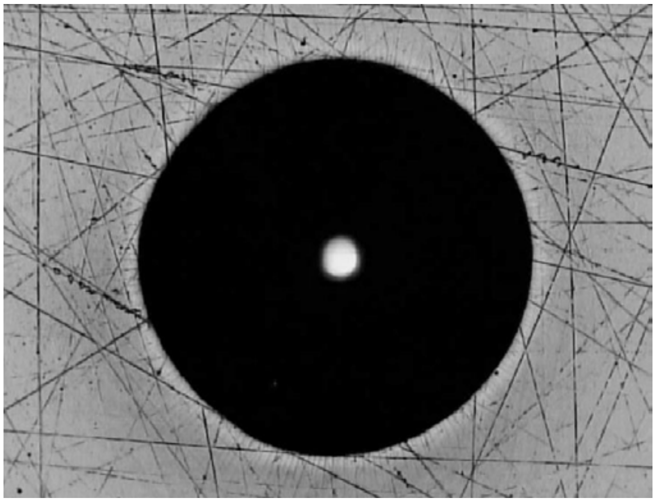

Figure 9. Adhesion test coating type no. 3 by Rockwell C indentation.

SEM ANALYSIS

Figure 10 shows the typical amorphous structure of a DLC coating (type no. 1) produced by using a PACVD glow discharge.

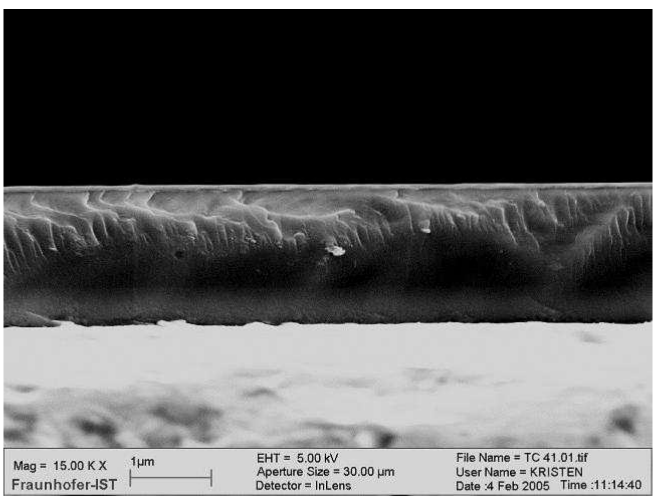

Figure 10. Cross section fraction of coating type no. 1.

The cross section fraction of coating type no. 2 shows the columnar structure of a metal containing DLC coating produced in a PVD process and the amorphous structure of a metal free DLC coating deposited by using a PACVD glow discharge (see Figure 11).

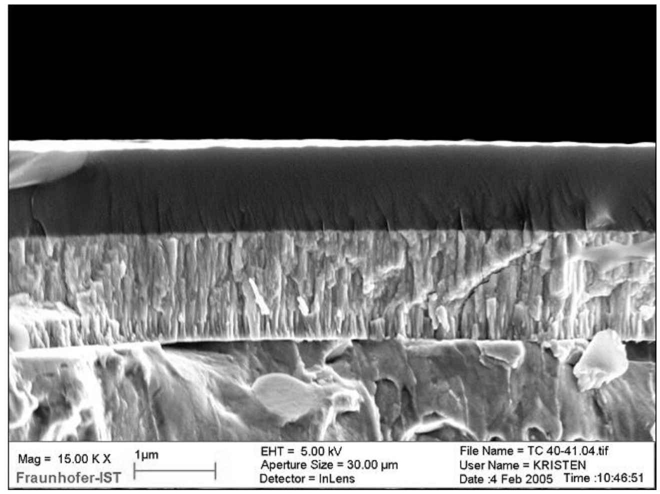

Figure 11. Cross section fraction of coating type no. 2.

In Figure 12 the SEM picture shows the columnar structure of a CrN coating and the amorphous structure of a pure DLC coating on top of the CrN coating.

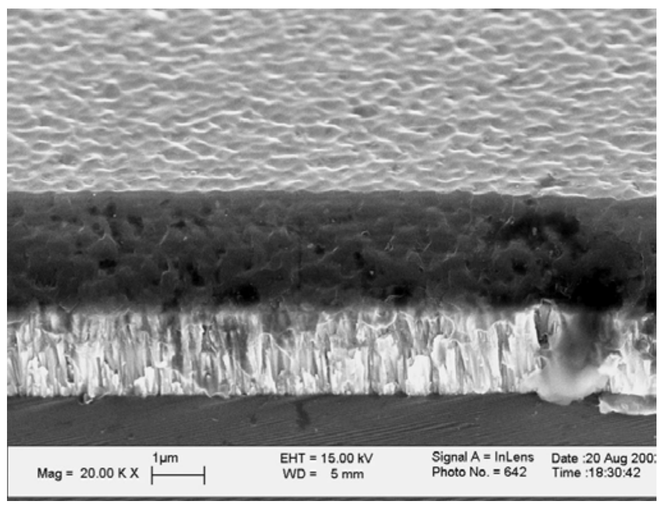

Figure 12. Cross section fraction of coating type no. 3.

HARDNESS MEASUREMENTS

The hardness values of the amorphous metal free a-C:H top layers were measured by two different methods. They were determined from measurements with a Fischerscope microhardness tester (H 100, company Fischer, Load 20 mN) and a nanohardness tester (AFM with a Hysitron® measurement system).

The results from the micro- and nano-hardness measurements are compared in Figures 13 and 14. The reference material was a metal containing a-C:H:W coating with a nanohardness of 11 GPa and a Vickers (micro) hardness of 1.135 kg/mm^2 calculated from the universal hardness HU_{plast}. It is demonstrated that the hardness values of the a-C:H top coatings are typically by a factor of 2 higher compared to the a-C:H:W coating independent from the used glow discharge frequency (MF or RF).

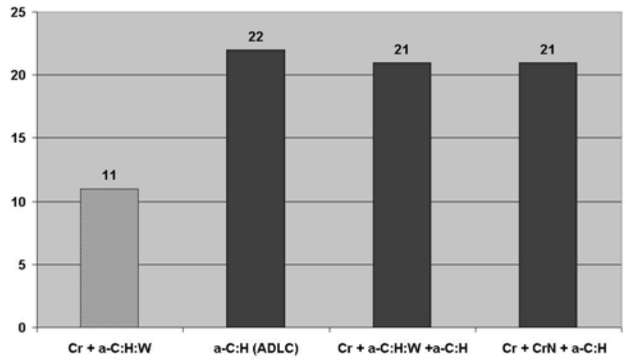

Figure 13. Comparison of the nano hardness values [GPa] of different DLC coatings.

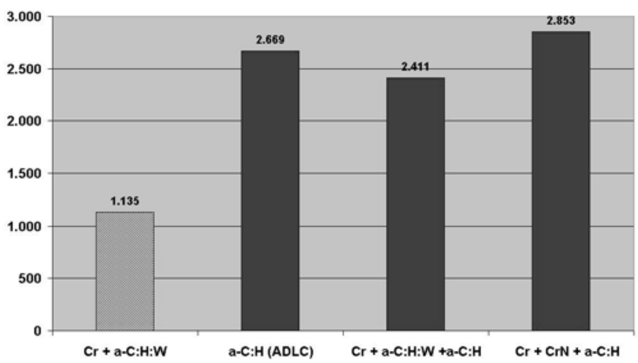

Figure 14. Comparison of the Vickers hardness values [kg 1 mm^2] of different DLC coatings.

ABRASIVE WEAR PROPERTIES

The results of the abrasive wear measurements are shown in Figure 15. The abrasive wear rates of the different a-C:H top layers are the same independent from the used glow discharge frequency (MF or RF). The abrasive wear volumes of all measured a-C:H top layers are 0.7 x 10^{-15} m^3/Nm and by a factor of 6 lower compared with the metal containing a-C:H:W reference coating.

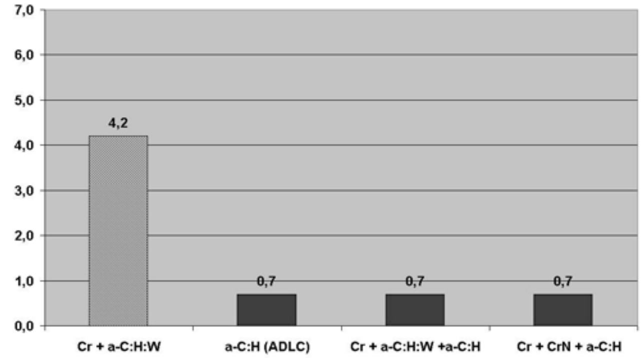

Figure 15. Comparison of the abrasive wear resistance [10^{-15} m^3/Nm] of different DLC coatings.

LOAD CARRYING CAPABILITY

The load carrying capability of the three different coating systems were measured by scratch test (increasing load 0 - 70N) and compared with a Cr + a-C:H layer system (Cr adhesion layer) as a reference material. As shown in Figure 16, it is possible to increase the load carrying capability of the metal free DLC top layer by 40% by using a metal containing DLC supporting layer (coating type no. 2) compared with a Cr + a-C:H coating system. By using a CrN supporting layer it is possible to improve the load carrying capability of the a-C:H top layer by 80% compared with the Cr + a-C:H coating system.

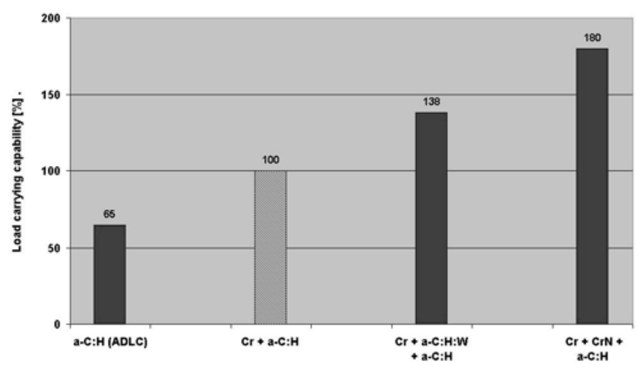

Figure 16. Comparision of the load carrying capability of different DLC coating systems.

SUMMARY AND CONCLUSION

In this paper the properties of three different metal free hydrogenated DLC coatings produced in industrial coating equipment were compared. The structure and the tribological properties of the different layer systems were compared depending on the applied supporting layer system and the used glow discharge frequency (RF or MF). The tribological properties are summarized in Figure 17.

Film	a-C:H (ADLC) PACVD	Cr + a-C:H PVD/PACVD	a-C:H:W + a-C:H PVD/PACVD	CrN + a-C:H PVD/PACVD
Vickers Hardness ($HV_{0,02}$) [kg/mm^2]	2.669 ± 270	2.646 ± 190	2.411 ± 308	2853 ± 190
Nanoindentor (AFM) [GPa]	22 ± 2	21 ± 2	21 ± 1	21 ± 1
E-modulus (AFM) [Gpa]	200 ± 15	188 ± 15	187 ± 15	185 ± 15
Scratch Test Critical Load (LC2)	65%	100%	138%	180%
Abrasiv wear [10^{-15} m^3/mN]	0.7 ± 0.1	0.7 ± 0.1	0.7 ± 0.1	0.7 ± 0.1
a-C:H thickness [µm]	1.5 ± 0.1	1.5 ± 0.1	1.5 ± 0.1	1.5 ± 0.1
Friction coeff. (dry vs. steel)	0.2 ± 0.05	0.2 ± 0.05	0.2 ± 0.05	0.15 ± 0.05

Figure 17. Overview of the tribological properties.

Hardness friction and wear properties of the deposited RF- and MF-DLC top layers are similar. A clear difference between the three applied coating systems was found concerning the load carrying capability (llc) depending on the used supporting layer system. We found a minimum (65%) load carrying capability for coating type no.1, a medium (138%) load carrying capability for coating type no. 2 and a maximum (180%) load carrying capability for coating type no. 3.

- Minimum llc: coating type no. 1: a-C:H:Si + a-C:H (ADLC)
- Medium llc: coating type no. 2: Cr + a-C:H:W + a-C:H (factor 2 higher)
- Maximum llc:coating type no. 3:Cr + CrN + a-C:H (factor 3 higher)

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