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CVD diamond coating of forming dies with a homogenous coating thickness

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Abstract

Ball-on-plate tribometer tests have already shown that polished polycrystalline CVD diamond coatings in dry tribological contact with aluminium lead to low wear rates and a low coefficient of friction of 0.12. The possibility to coat the forming zone of forming dies was investigated in this study to evaluate the applicability of CVD diamond coatings for a tapering process of aluminium rods. The results are examined by laser-scanning confocal microscopy, scanning electron microscopy and cryofractures. CTF12D carbide grade dies with a height of 5 mm and an inner diameter of 7.8 mm could be coated with a laser-based plasma CVD process with a CVD diamond coating with a homogeneous thickness of 2.3 µm. The standard deviation of the coating thickness variation is only 0.1 µm in the entire forming zone. This was achieved by combining an etching process, a masked diamond nucleation, a specially fabricated rotating substrate holder and turning the die upside down after half the coating time so that the plasma flame enters through the other opening of the die.

Keywords: CVD diamond deposition, hard metal, forming die

1 Introduction

Ball-on-plate tribometer tests showed that polished polycrystalline chemical vapor deposition (CVD) diamond coatings in dry tribological contact with aluminium lead to low wear rates and a low coefficient of friction of 0.12 [1]. In order to use diamond in forming technology, a diamond coating is indispensable for economic and production reasons as soon as the forming die exceeds the dimension of an inner diameter of 2 mm [2]. Up to an inner diameter of 2 mm the forming die can be produced out of a single diamond crystal [2]. Schäfer et al. [3] coated silicon nitride drawing dies in a hot-filament CVD process. For diameters above 5 mm they used arrangements where the hot filaments are placed inside the cylindrical parts. Till the waist of the drawing die the coating thickness is quite homogenous between 9.8 µm and 10.3 µm. As soon as the undercut starts the coating thickness decreases rapidly. Towards the waist of the drawing die, the diamond grain size decreases from coarse to fine. The aim of this study was to coat the entire inner wall of a 5 mm high forming die with a CVD diamond coating with a homogenous thickness, using an atmospheric laser-based plasma CVD process.

2 Methods

A laser-based plasma CVD process was used at atmospheric pressure without a vacuum chamber to deposit polycrystalline CVD diamond coatings, which is described in detail in [4]. For the CVD diamond deposition, methane and hydrogen with a total flow of 2 standard liter per minute (slm) in the ratio of 1% was added to the argon plasma flame (26 slm). The deposition was executed at a process temperature of 900 °C.

The forming dies had been manufactured out of K10 hard metal of the type CTF12D consisting out of 94% tungsten carbide and 6% cobalt. The geometry of the forming die is shown in Fig. 1. The dies were etched by Murakami reagent ($K_3Fe(CN)_6 : KOH : H_2O$ = 1:1:10) for 30 minutes and subsequently with Caro's reagent (3 ml 96 wt.% H_2SO_4, 88 ml 40% w/v H_2O_2) for 45 seconds [5]. The diamond nucleation was carried out with a dispersion of 200 ml isopropanol and 210 mg diamond powder with the average crystal size in the range of 0.25 µm to 0.50 µm from the company Microdiamant AG. The substrates were put into the dispersion within an ultrasonic bath for ten minutes and subsequently into isopropanol for three minutes. The areas

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that should not be nucleated were masked before nucleation.

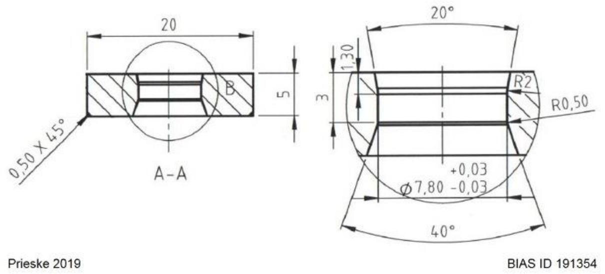

Fig. 1: Technical drawing of the hard metal forming die.

The specially produced substrate holder is shown in Fig. 2. The substrate holder consists of a two-piece brass part to clamp the forming die onto the table. In addition, there are two holes for compressed air cooling and a hole below the die opening which allows the plasma flame to exit at the other end. The ceramic plate underneath serves as thermal insulation. In order to increase the homogeneity in radial direction, the table is freely rotatable during the coating process.

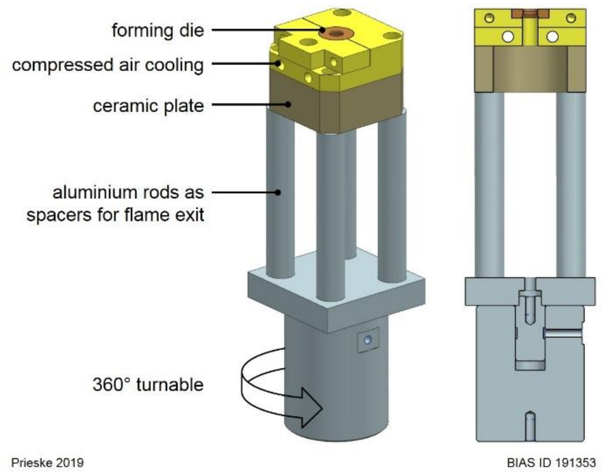

Fig. 2: Substrate holder for the diamond deposition of a forming die.

Scanning electron microscopy (SEM) (Carl Zeiss Microscopy EVO MA-10), digital microscopy (Keyence VHX-1000) and 3D laser scanning confocal microscopy (Keyence VK 9710) were used to take images of the coating surfaces and measure the coating thicknesses. Cryofractures were prepared to accurately determine the coating thickness. A slit was eroded from both sides towards the middle of the die by electrical discharge machining. Afterwards, the specimens were cooled down by liquid nitrogen and cryofractures were carried out.

3 Results

Fig. 3 shows that without masking during nucleation the flat area at the top of the forming die is coated by a closed CVD diamond coating. The CVD diamond coating at the top side partly detaches or totally delaminates after the deposition process and also leads to a crack formation of the CVD diamond coating in the forming zone (compare Fig. 3 right).

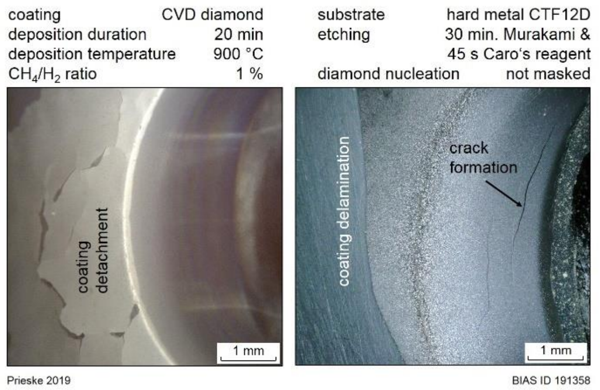

Fig. 3: Microscope images of the CVD diamond coated forming die with usual diamond nucleation.

In the case of masked diamond nucleation, no layer detachment is detected as can be seen in Fig. 4. The closed diamond coating ends at the distance of 28 µm ± 3 µm from the edge of the forming zone.

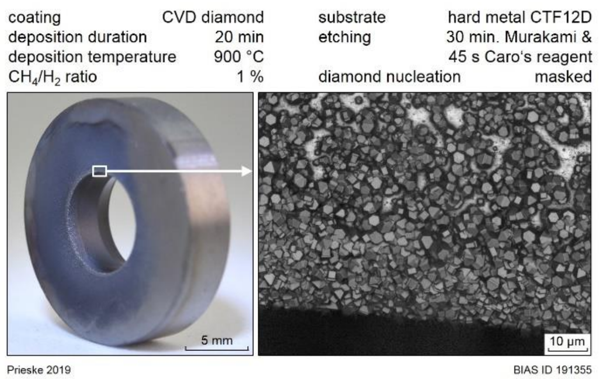

Fig. 4: Photography and microscope image of the CVD diamond coated forming die with masked diamond nucleation.

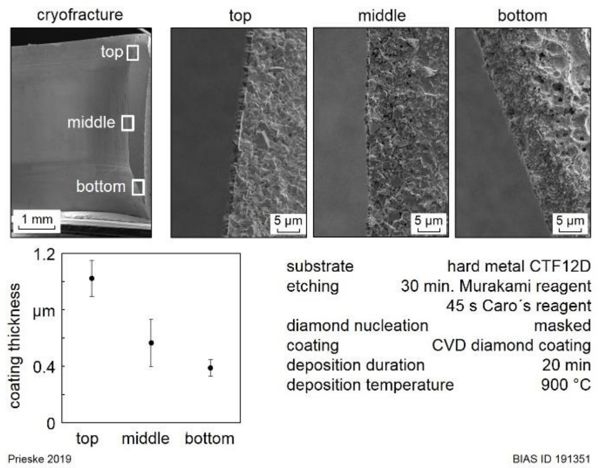

Fig. 5: SEM images of a cryofracture of a CVD diamond coated forming die after deposition through the entrance opening as overview (left) and as detailed images and the coating thickness in axial direction (bottom left).

Fig. 5 shows a cryofracture of a CVD diamond coated forming die. The coating was deposited for twenty minutes through the entrance opening of the forming die. It can be seen that the entire inner wall including undercuts of the forming die is coated. The coating thickness at the entrance opening where the plasma flame enters (Fig. 5 *top* section of forming die) is 1.0 µm ± 0.1 µm. Towards the bottom of the inner wall the coating thickness decreases down to 0.4 µm ± 0.1 µm. That leads to the mean coating thickness of 0.7 µm ± 0.3 µm.

Fig. 6 shows the result when the coating is deposited through both openings. To do this, the CVD diamond deposition through the entrance opening is stopped after twenty minutes and the forming tool is re-clamped upside down in the substrate holder before the re-start of the process. The coating is then deposited for an additional twenty minutes by entering through the exit opening. This procedure leads to the mean coating thickness of 2.3 µm ± 0.1 µm.

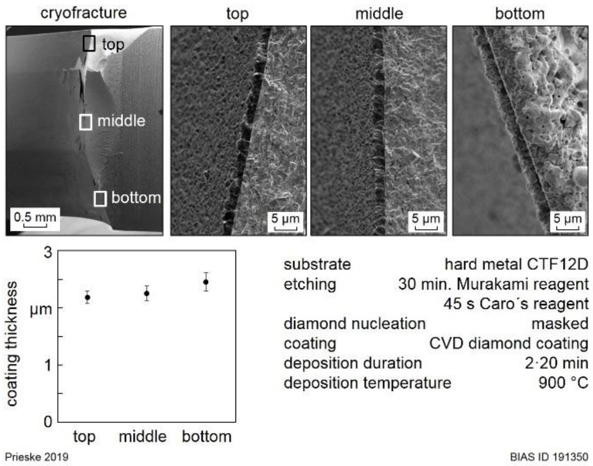

Fig. 6: SEM images of a cryofracture of a CVD diamond coated forming die after deposition through the entrance and the exit opening as overview (top left) and as detailed images (top right) and the measured coating thicknesses in axial direction (bottom left).

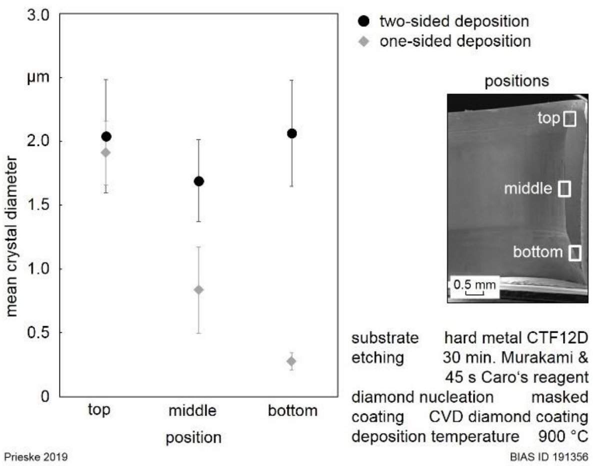

Fig. 7: Comparison of the mean diamond crystal diameter at different positions of the inner wall after one- and two-sided deposition.

The distribution of the diamond crystal size along the inner wall varies depending on whether a one-sided or two-sided deposition was applied, as can be seen in Fig. 7. The procedure of the one-sided deposition shows a decreasing crystal size towards the bottom of the forming die. In case of the two-sided deposition, the crystal size at the top and bottom of the die is approximately the same. At the middle position, the diamond crystal size is 0.4 µm smaller than at the top and bottom.

4 Discussion

In Fig. 3 can be seen that a closed CVD diamond coating on the top flat area of the forming die leads to a delamination of the CVD diamond coating, which also leads to a spallation and crack formation at the inner wall of the forming die. Xu et al. [6] deposited CVD-diamond coatings on cemented carbide substrates with smooth (Rq 0.04 µm) as well as blasted (Rq 0.27 µm) surfaces using a Cr-CrN interlayer system. In Rockwell C indentation tests, the diamond coating without surface pre-treatment showed significantly poorer attachment (delamination at the interface to the interlayer); it was suggested that increased surface roughness prior to CVD-diamond coating enhances layer adhesion [6]. These results go hand in hand with the investigations of Lee et al. [7] on CVD diamond deposition on laser induced micro-rough surfaces on cemented carbide substrates. They concluded that a more intensive mechanical interlocking and a mechanically graded transition from the substrate material to the CVD diamond coating can significantly reduce compressive stresses in the transition area compared to a smoother substrate surface. In this study, this leads to the conclusion that the low roughness at the top area of the forming die is the cause of the delamination. The problem can be solved by masking that area during diamond nucleation, which results in a not closed diamond coating. In this way, no residual stresses can build up which lead to the layer flaking off.

Fig. 5 shows that the growth rate decreases with increasing distance between the position to be coated and the plasma source. According to Corat et al. [8], the distance between substrate and plasma source at a fixed substrate temperature has a significant influence on the deposition rate, since at a greater distance the CH_3 molecules produced are already partially converted back to CH_4 molecules. At the distance of 7 mm, they detected a relative CH_3 concentration three times higher than at the distance of 11 mm. To overcome that problem in this study, the forming die was turned upside down in the middle of the deposition process, which resulted in the homogenous coating thickness of 2.3 µm ± 0.1 µm and the crystal diameter of 1.9 µm ± 0.2 µm.

5 Conclusion

The entire inner wall of a 5 mm high forming die including undercuts was coated with a CVD diamond coating with homogeneous layer thickness and crystal diameter with a standard deviation of 0.1 µm and 0.2 µm, respectively.

Acknowledgements

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