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Some Insights into the Scratch Resistance Assessment of Polycrystalline Diamond Coatings

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Abstract

This study explores the scratch resistance and adhesion performance of polycrystalline diamond (PCD) coatings, crucial for their application in demanding industrial environments. Applying instrumented scratch testing, a practical, quick, cheap, and reliable test method, the adhesion strength of PCD coatings on steel substrate is assessed. Recognizing the limitations of the conventional scratch testing using a diamond stylus when applied for PCD coatings, a modification, namely the use of a stainless steel ball as a scratching tool, and its applicability for characterizing the scratch behavior of PCDs are discussed to offer a simpler and more economical way for tribological characterization of diamonds, the hardest, thus the most abrasive material in the nature.

Keywords: polycrystalline diamond, PCD, scratch test, steel ball stylus, critical load

1. Introduction

The ever-changing requirements of aerospace and manufacturing industries have increased the demand for innovative materials with excellent mechanical qualities in recent years. Diamond coatings have received considerable interest for their potential to improve the performance and lifespan of many engineering components. The high hardness of diamond grains in PCD coatings enables efficient cutting, enhancing productivity and reducing machining time. PCD coatings have a low friction coefficient, reducing friction and minimizing heat buildup during machining processes, which improves tool performance and dimensional accuracy. It also exhibits excellent resistance to chemical reactions, making it suitable for machining various materials, including non-ferrous metals, composites, and abrasive materials. The study of polycrystalline diamond coatings stands at the forefront of innovation, promising transformative solutions that align with the evolving needs of modern industries [1,2,3,4,5].

When evaluating whether polycrystalline diamond coatings are suitable for practical applications, their scratch resistance is a crucial criterion. Scratching is a widely observed form of wear, and it is essential to understand their complex reaction to such mechanical loadings to maximize their effectiveness. Various methodologies are employed to assess the adhesion characteristics of coatings [6]. Scratch testing is one of the most effective ways for determining the adherence of a hard and thin layer to a substrate [7,8] due to its reliability and simplicity. This procedure can be easily performed without specific specimen shape or preparation. Adhesion is interpreted when a critical normal load is attained at which the coating fails. Assuming the failure mode is adhesive, the critical load is used to calculate the coating-substrate adhesion strength [9,10].

Scratch testing involves the movement of a conical indenter tip across the coating's surface, resulting in the creation of a groove. This is achieved by applying either a progressive or constant force perpendicular to the surface. The tangential force can be quantified during the test, and the scratch morphology is typically seen immediately or after the test [11]. The analysis of scratch test output data primarily involves identifying the critical load, Lc, at which the adhesion of the film is compromised, i.e., a clean removal of the coating from the substrate occurs [12]. It can be determined from the friction coefficient/loading force vs. scratching distance diagram recorded during the test.

The conventional scratch test – utilizing a Rockwell-C diamond stylus as the indenter – is a frequently employed method for characterizing the quality of hard coatings applied onto predominantly metallic substrates. This is primarily due to the test's relatively stable experimental configuration, straightforward operation, and representative outcomes. Nevertheless, in comparison to alternative mechanical testing techniques [13,14] its applicability has limitations in several circumstances. Failure modes during the testing of hard coatings on soft substrates appear at exceedingly low critical loads, providing challenges in terms of monitoring. In other instances, failure modes may be entirely undetectable. Further complications occur during the testing of ultra-hard diamond layers. The ideal geometry is disrupted due to the rapid abrading of the indenter's tip. Bearing balls were used in the scratch test as an alternative to the Rockwell-shaped indenter. This facilitates the modification of the indenter's material and contact radius, thereby influencing the formation of the tension field in the coating-substrate composite. Sander et al. [15] studied the modified scratch test and applied it on hard layers of CrN and TiN deposited on soft polymeric ASA (acrylonitrile styrene acrylate) substrates as well as on ultra-hard diamond layers on steel substrates. Another study was performed by Buijnsters et al. [16] for adhesion analysis of PCD films on molybdenum.

By synthesizing existing knowledge, this paper aims to study scratch resistance assessment of polycrystalline diamond coatings on steel substrate with a modified test method.

2. Materials and methods

PCD coatings with a uniform thickness of ~ 11 μm were deposited on a steel substrate by chemical vapor deposition (CVD). The instrumented scratch tests were accomplished using a progressive loading in the 2-150 N range using an SP-15 Instrumented scratch tester (producer Sunplant Ltd., Miskolc, Hungary) with d=3 mm diameter steel ball indenters (Figure 1).

Figure 1. The SP-15 instrumented scratch tester

The applied test parameters are given in Table 1.

Table 1. Test parameters of the progressive loading scratch test

Test parameter	**Nomination**	**Value**
Minimum load	$F_{n, min}$	2 N
Maximum load	$F_{n, max}$	150 N
Load gradient	dF/ds	15 N/mm
Scratch length	L	10 mm
Scratching velocity	v	5 mm/min
Ball material	Steel, AISI 420; Q+T; d =3 mm	

The need for the repeated replacement of the damaged Rockwell diamond stylus after each measurement, as used in a standard scratch test, is accompanied by extremely high costs in the testing of polycrystalline diamond coatings. Therefore, we worked out another procedure using a stainless steel ball stylus to reduce these costs. The usual Rockwell-C diamond stylus applied during the conventional standard scratch test and the newly designed, custom-made scratch tool used in the current investigations are shown in Figure 2.

a) *b)*

Figure 2. The standard Rockwell diamond stylus (a) and the novel steel ball stylus designed and applied in the experimental work

This method is much cheaper and allows us to replace the probe with a new, intact piece for each test at a reasonable cost.

2.1 Scratch tests with progressive load using a unique tool

When using the new steel ball stylus for the scratch test, the following aspects must be taken into consideration:

- For repeatable measurements and reliable results, the steel ball stylus must be replaced in each test.
- Commercially available styluses can’t be disassembled. Therefore, a purposefully constructed tool holder for the ball stylus has been designed and manufactured at the Institute of Materials Science and Technology of UM.
- The design requirements for the ball holder were as follows:
  - quick replacement of the steel ball must be assured;
  - safe fixing the ball in the holder, preventing rotation during scratching is essential;
  - the dimensions of the tool shank should match the joining dimensions of the available scratch tester.

The essential features of the modified test method are the following:

- Using a ball-type stylus, this test represents a different contact geometry, contact pressure, and loading condition than the traditional one.
- Therefore, the results measured this way cannot be compared with those derived from the usual standard test method.
- However, it can serve as a quick and cheaper test for evaluating the effect of specific technological or geometrical parameters on the production of PCD coatings.

2.2 Test samples

Due to the high costs of the diamond coatings, the first trial tests reported here were performed on samples used previously for other mechanical tests by our German cooperative research partner at the Fridrich Alexander University, Erlangen-Nürnberg. The “recycled” samples, with visible damages from different investigations before the scratch test, are shown in Figure 3.

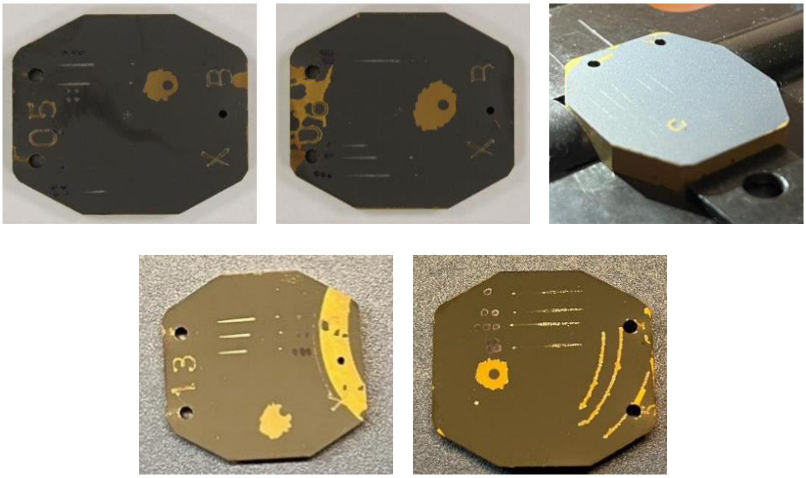


Figure 3. The recycled samples used in the trial scratch tests

The substrate material was an X46Cr13 (1.4034) martensitic stainless steel. The surface of the substrate was produced by ultrasonic vibration superimposed machining” (UVSM) process [17] simulating the particle-blasted pretreated surfaces used widely in the industry. The simulated particle blasted surface was characterized by the wavelength of the periodic topography λ (µm) and the structure height A (µm), which were approximated by the mean distance between roughness peaks, Rsm (µm), and the mean maximum roughness, Rz (µm) of the machined surface. The details on the sample processing and characterization are found in the work [17].

The coating has a double-layer architecture, with a 5 µm TiBN interlayer between the substrate and the 11 µm thick PCD top layer. TiBN was produced by CVD, and the polycrystalline diamond layer was made by HF-CVD with a coating temperature of 865 °C.

Five samples were tested, and five measurements were accomplished on each sample. The structural parameters of the samples are shown in (table 2). It should be mentioned that the coating roughness indicated in the table is not identical to the substrate roughness, but in most cases, they were closely similar.

Table 2. The main parameters of the coating process and surface roughness of the coatings

Sample number	**Machined wavelength, λ [µm]**	**Structural height, A_{us} [µm]**	**Roughness of the coatings**	
			R_a [µm]	**R_z [µm]**
DB05	25	5	1.11	4.98
DB06	25	5	0.96	4.17
DB08	35	5	1.84	12.03
DB12	35	5	1.96	12.67
DB13	25	5	1.13	2.61

3. Results

The scratch diagrams (Figure 4) present the connections of friction coefficient vs. scratching distance and loading force vs. scratching distance. From the character of the two representative friction coefficient curves displayed in the diagram, it is established that the applied F= 150 N force did not cause delamination due to good adhesion of the coating. Therefore, it is impossible to derive the critical force from the tests. However, the friction coefficient showed some correlation with the coating roughness, i.e., if the Ra value is increasing, the coefficient of friction – calculated as an average recorded on the last 10% of the scratch length – is also growing. The measured friction coefficient was μ=0.33 and 0.37 for the DB06 and DB08 coatings, respectively, while their Ra values were 0.96 and 1.84, respectively.

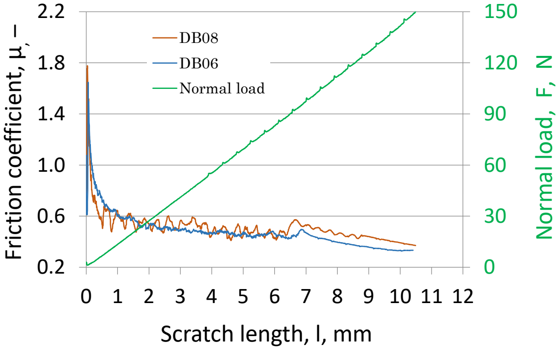


Figure 4. Scratch diagram

It should be underlined that these results have been derived from trial tests made on specimens previously used for other measurements. Therefore, the observations presented here are unsuitable for drawing well-founded conclusions or generalizations.

Scratch grooves on the DLC coating are difficult to reveal by optical microscopy. Still, transfer film of the stylus, i.e., steel ball origin along the scratch groove and some indication of the termination of the coating integrity could be observed in Figure 5 for the DB05 (Ra=1.11 µm) and DB 06 (Ra=0.96 µm) coatings.

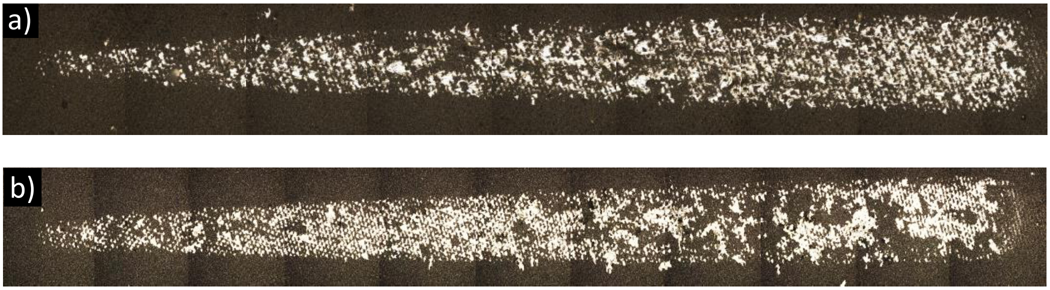
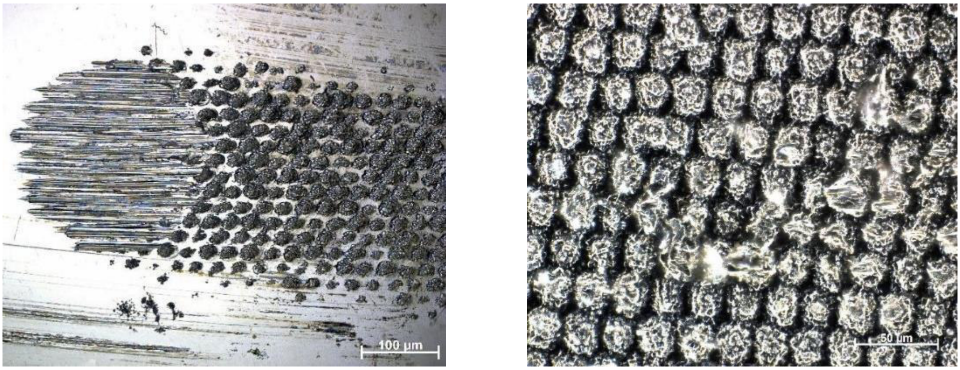


Figure 5. Scratch grooves obtained on the DB05 (a) and DB06 (b) samples

The OM photographs of the wear scar on the stainless steel ball show a high amount of adhered transfer film originating from the diamond with a surprisingly ordered pattern (Figure/a) resembling the surface texture of the diamond coating (Figure 6/b).



a) *b)*

Figure 6. Optical micrograph of the wear scar on the steel-ball stylus with a high amount of adhered debris (a) and the surface texture of the initial PCD coating (b) show morphological similarity in the ordered location of diamond grains

4. Conclusion

An instrumented scratch test using a stainless steel ball stylus has been applied to investigate PCD diamond coatings. Trial tests were performed to justify the applicability of the test for polycrystalline diamond coatings. The main result of the research work is to demonstrate that scratch tests using a steel ball as a stylus can provide meaningful information on the scratch resistance of the PCD coatings under investigation.

The observed phenomena, such as the monotonically decreasing shape of the friction coefficient vs. scratch length curves or the relationship between the friction coefficient and the roughness of the PCD coating, are not yet suitable for general conclusions but must be confirmed and validated by further large-scale and systematic investigations. However, the information gained from the tests performed provides a sufficient basis for continuing the modified scratch tests presented here, representing a promising, cost-effective test procedure compared to conventional standard scratch tests using a diamond cone-stylus.

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