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Short communication

# Development of ultrananocrystalline diamond (UNCD) coatings for multipurpose mechanical pump seals

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ABSTRACT

The reliability and performance of silicon carbide (SiC) shaft seals on multipurpose mechanical pumps are improved by applying a protective coating of ultrananocrystalline diamond (UNCD). UNCD exhibits extreme hardness (97 GPa), low friction (0.1 in air) and outstanding chemical resistance. Consequently, the application of UNCD coatings to multipurpose mechanical pump seals can reduce frictional energy losses and eliminate the downtime and hazardous emissions from seal failure and leakage. In this study, UNCD films were prepared by microwave plasma chemical vapor deposition utilizing an argon/methane gas mixture. Prior to coating, the SiC seals were subjected to mechanical polishing using different grades of micron-sized diamond powder to produce different starting surfaces with well-controlled surface roughnesses. Following this roughening process, the seals were seeded by mechanical abrasion with diamond nanopowder, and subsequently coated with UNCD. The coated seals were subjected to dynamic wear testing performed at 3600 RPM and 100 psi for up to 10 days during which the seals were periodically removed and inspected. The UNCD-coated seals were examined using Raman microanalysis, scanning electron microscopy, optical profilometry, and adhesion testing before and after the wear testing. These analyses revealed that delamination of the UNCD films was prevented when the initial SiC seal surface had an initial roughness >0.1 μm. In addition, the UNCD surfaces showed no measurable wear as compared to approximately 0.2 μm of wear for the untreated SiC surfaces.



## 1. Introduction

Over the years, great strides have been made in improving the performance and durability of mechanical seals by controlling their structure and mechanical properties. In fact, the failure modes of today's seals are seldom related to the microstructure or mechanical properties of the base seal materials. However, failures due to dry or near-dry running conditions (especially at the start up), severe and un-even loading or load fluctuations during operations, as well as blistering and other near-surface defects and/or deficiencies can still occur and limit the useful lifetimes of seal components used by the sealing industry. Surface defects, such as uneven surface finish, local variations in surface texture and/or mechanical properties, un-removed grinding or polishing furrows, nicks, and dents introduced during manufacturing and/or machining operations, may cause chipping (especially in SiC-based seals) and hence trigger micro-fracture and eventually seal failure. In addition, large variations in contact stresses during operation, as well as very high normal and tangential pressures developing at asperity levels during rotating contacts can give rise to severe temperature spikes and surface distress that can ultimately cause seal failures.

To improve the surface-sensitive properties of mechanical seals, researchers have tried two general approaches in recent years. First, they developed advanced laser machining methods to achieve specific geometric profiles and/or textures on seal faces and second, they have tried a variety of surface engineering or hard coating technologies to improve the surface tribological and mechanical characteristics of seals to prevent wear-related failures. Recent advances in surface metrology, polishing, and handling methods have also reduced the number of surface irregularities and thus reduced the causes of failures resulting from such irregularities.

Among the many types of hard physical- and chemical-vapor deposition (PVD and CVD) coatings, diamond and diamond-like carbon coatings have attracted the most attention for sliding or rotating tribological surfaces in recent years, especially in microelectromechanical systems (MEMS), biomedical, and nano-scale application [1–5]. In particular, diamond coatings were thought to

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be an ideal choice for such applications mainly because of their extreme mechanical hardness, naturally low friction, high thermal conductivity, and excellent chemical inertness. Some of the earliest attempts at producing diamond films on mechanical seal faces go back to the mid 1990s when scientists from Upsala University-Sweden attempted to apply micro-crystalline diamond films on SiC face seals. Their research suggested that diamond films were indeed very promising for mechanical seal applications, but additional polishing steps needed in the case of the intrinsically rough micro-crystalline diamond films was very time consuming and hence costly thus rendering such conventional diamond films essentially useless for commercial scale applications.

Pioneering research work on diamond films at Argonne National Laboratory for more than two decades resulted in the discovery of an ultrananocrystalline diamond (UNCD), a material consisting of 2–5 nm diamond grains separated by 0.4 nm wide grain boundaries comprised of $sp^3$ and $sp^2$ bonded carbon. Thin films of UNCD posses extreme hardness (97 GPa), high fracture strength (5.4 GPa) and Young's modulus (990 GPa) as well as a super-smooth surface finish (i.e., less than 20 nm RMS roughness) in addition to impressive friction and wear properties. Systematic tribological studies on UNCD-coated Si and SiC substrates revealed that their friction coefficients were indeed very impressive (i.e., ranging from 0.03 to 0.1, depending on environment and counterface materials) and the amount of wear was un-measurable even after very long sliding tests [6,7]. These films were also deposited on a few small diameter (~5 cm in diameter) SiC face seals and limited testing was performed to evaluate their performance and durability in a seal test machine [8]. Microscopic and surface profilometric inspection of the coated seal faces did not reveal any measurable wear even after 21 days of testing. Based on these very promising initial results, we have undertaken a more systematic study to ascertain the true potential of UNCD as a wear protective coating on a large number of SiC seals. For these seals, a variety of seeding methods was used to enhance film-to-substrate adhesion which is very critical for durability and performance improvements under high-load, high-speed sliding conditions.

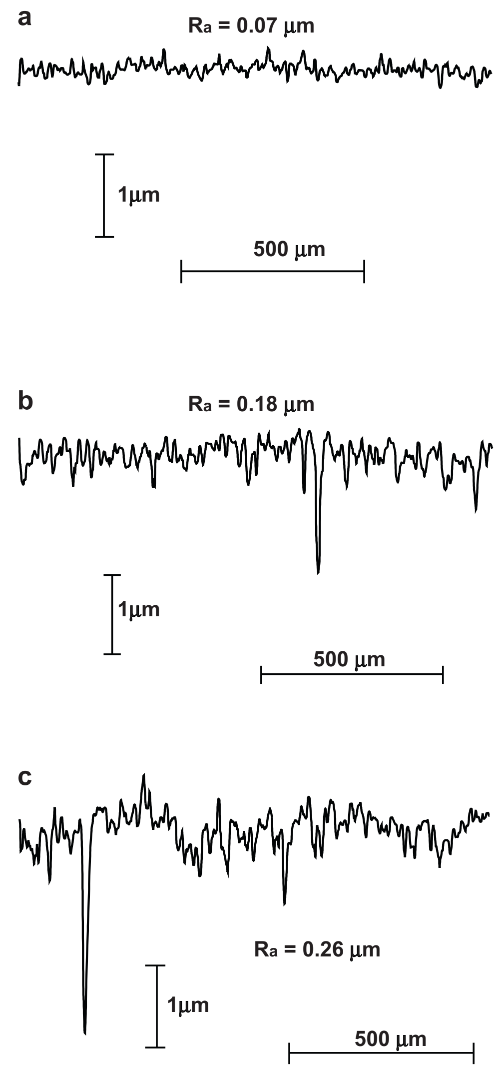


**Fig. 1.** Initial seal surface profiles measured using optical profilometry after polishing using 6 µm (a), 12 µm (b), and 30 µm (c) diamond powder.

## 2. Experimental procedure

Deposition of the UNCD films onto the SiC seals was performed using an Iplas (Stuttgart, Germany) Cyrranus I large area microwave plasma chemical vapor deposition reactor. Prior to seeding, the SiC seals were polished with diamond powder of various sizes (6, 12, and 30 µm diameter) to prepare surfaces with different initial RMS roughnesses as described in Table 1. The profiles of the different treated surfaces are shown in Fig. 1. The objective of this procedure was to investigate the effect of different initial roughhouses on the adhesion, friction, and wear of the resulting UNCD coatings. After this surface roughening procedure, the SiC seals were seeded by polishing the front surface using diamond nanopowder to ensure uniform nucleation of the UNCD resulting in smooth, continuous films. After this seeding treatment, the seals were ultrasonically cleaned in methanol, rinsed with methanol, and then dried in flowing nitrogen. The SiC seals were then mounted in a graphite fixture with a recessed groove such that the top surface of the seal was flush with the top surface of the fixture. This mounting method guaranteed that the spherical microwave plasma discharge encountered a flat surface without any sharp edges and ensured a uniform UNCD coating across the SiC surface. The UNCD coating procedure used a 98% Ar/2% $CH_4$ gas mixture, a temperature of 900 °C, and 1 kW of microwave power for 4 h. This procedure deposited a UNCD film with a thickness of ~1 µm.

**Table 1**
Seal surface roughness as a function of diamond particle size used for surface polishing.

| Group of tested, coated SiC seals | 1st group | 2nd group | 3rd group |
|---|---|---|---|
| Size of diamond particles used for seal polishing before UNCD deposition (µm) | 6 | 12 | 30 |
| $R_a$ (µm) | 0.05–0.07 | 0.12–0.28 | 0.2–0.3 |
| $R_p$ (µm) | 0.65–0.82 | 1.04–2.84 | 1.32–3.37 |
| $R_v$ (µm) | (−1.18)–(−0.8) | (−5.12)–(−2.83) | (−3.09)–(−1.89) |
| PV (µm) | 1.48–2.36 | 3.86–7.14 | 4.08–5.62 |
| $R_q$ (µm) | 0.07 | 0.18–0.45 | 0.26–0.41 |
| $R_{sk}$ | 0.05–0.26 | (−3.69)–(−2.34) | (−1.15)–(−0.26) |
| $R_{ku}$ | 1.14–2.78 | 10.92–31.43 | 0.11–4.59 |

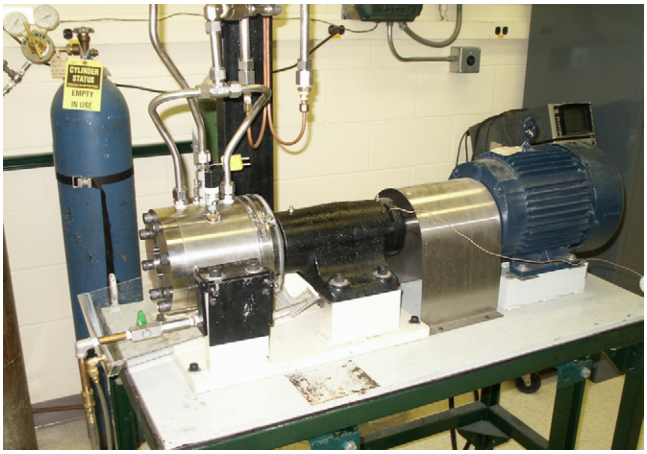

**Fig. 2.** Photograph of dynamic seal test machine.

Adhesion testing was performed using a Quad Group Sebastian Five pull-test machine. Tapered aluminum pins with a head diameter of 2.69 mm were bonded to the UNCD film using an epoxy adhesive that was cured for 30 min at 150 °C. During the adhesion testing procedure, the load on the pull-test machine was increased until either the epoxy failed or the UNCD film was removed from the SiC seal. Following the adhesion testing, both the surface of the SiC seal as well as the underside of the Al pin were inspected using a micro-Raman system (Renishaw Ramascope, 633 nm laser). Delamination of the film was confirmed by the presence of UNCD Raman features on the underside of the Al pin or by SiC features on the seal surface.

The UNCD coated and reference or uncoated seals were tested in a dynamic seal test machine which closely simulated the real operating conditions of an actual water pump. A schematic of this test machine and its main components are shown in Figs. 2 and 3. Two pairs of reaction-bonded SiC mechanical face seals (UNCD coated and reference uncoated) were mounted on the rotating shaft of the test machine, which passed through a chamber filled with water. The chamber pressure could be raised up to 400 psi (2.8 MPa) by passing compressed air through an air cylinder attached to the seal tester. Both face seals (coated and uncoated) were slid under the same conditions against the fixed graphite seal rings. This provides an equal speed-loading condition for both SiC seals for correct wear resistance comparison.

The main rotating shaft of the test machine with both SiC seals and graphite rings is presented in Fig. 4. The seal test system featured a water-level and temperature monitor to provide feedback signals for system shut-down in case of leakage and water level reduction below the minimum required for seal operation. This shut-off capability avoids seal damage that would occur under dry running conditions. In our case, all the tests were carried out using the following conditions: initial run-in was performed at 20, 40, 60, and 80 psi water pressure during 8 h at each pressure. The main part of the test was performed at 100 psi (0.7 MPa) water pressure for 50 h with an axis constant rotating speed 3600 rpm. These test parameters represent typical operating conditions for pumps used in industrial applications. UNCD coated SiC seals from each of the 3 groups (see Fig. 1 and Table 1) with different roughnesses were tested three times to provide reliable wear data. Some additional tests with seals from the 2nd and 3rd roughness groups were carried out for 21 days (more than 150 h) to test the long-term reliability of the UNCD-coated seals. After each test at different water pressure during the running-in period and periodically during the main tests at 100 psi water pressure the test was interrupted and the SiC seals and graphite counterface rings were removed for wear measurement and wear surface examination.

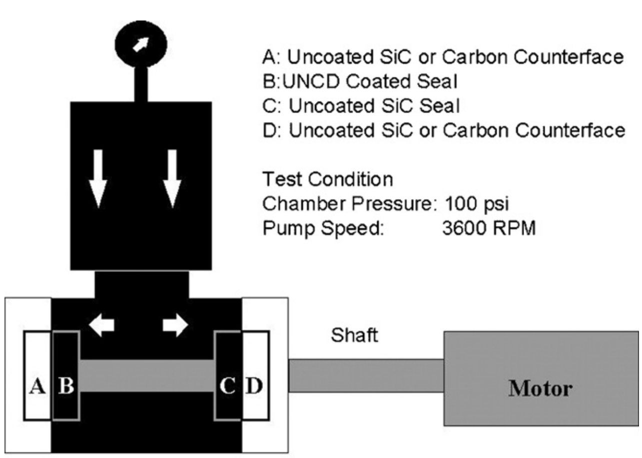


**Fig. 3.** Schematic diagram of seal testing machine.

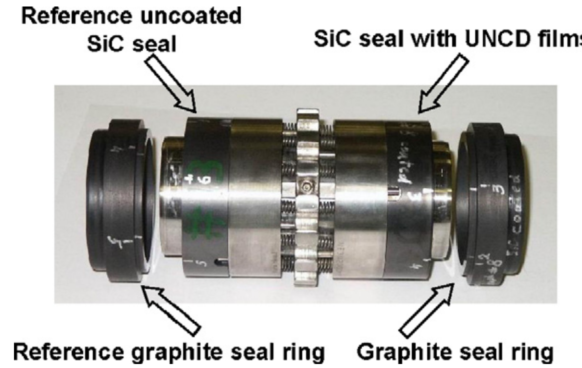


**Fig. 4.** Main shaft of the seal testing machine with UNCD-coated seal (right) and reference uncoated SiC seal (left) running against fixed graphite rings (both sides).

The wear of the SiC seals was estimated by measuring the wear track depth using an optical profilometer based on the phase-shift principle. The profiles were scanned perpendicularly to the sliding direction. Optical profilometry also permits examination of the morphology and wear damage of the worn surfaces. The average wear was quantified using thickness measurements taken at 6 points with equal distance between points in the central part of the sliding surface. The SiC seals were also examined after wear testing using scanning electron microscopy (SEM). These measurements used a Hitachi S4700 SEM with a field emission gun electron source and both secondary electron and backscattered electron (BSE) detection. Additional measurements were performed on the SiC seals after wear testing using a Renishaw Ramascope micro-Raman system.

## 3. Results and discussion

No measurable linear wear was revealed by surface profilometry on seals of the 2nd and 3rd groups (see Table 1) after each step of testing during the running-in period during which the water pressure was increased from 20 to 80 psi as well as during the main testing at 100 psi water pressure for 50 h. In fact, the wear tracks in the tested seals look identical to the unworn surface by optical microscopy. The scratches and grooves introduced by the polishing process are still visible on the wear track and resemble the initial, unworn surface. An optical image of the wear track on the seal of the 2nd group is shown in Fig. 5. Some graphitic debris particle transfer from the counterface seal rings is observed near the inner edge of the wear track (left side of Fig. 5). The wear track profiles

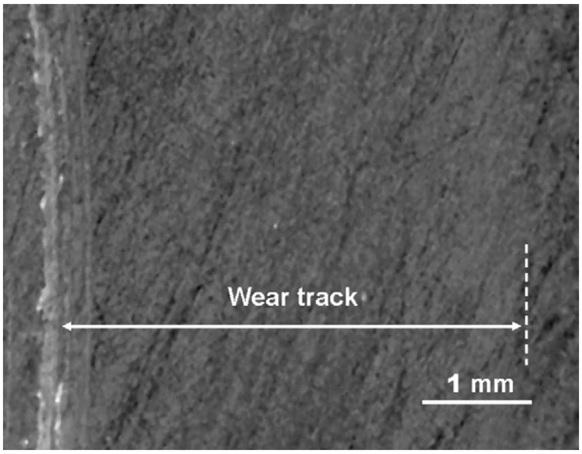


**Fig. 5.** Wear track on UNCD-coated seal of the second group.

of the seals from the 2nd and 3rd groups recorded perpendicularly to the rotational direction are presented in Fig. 6a and b. It is clearly seen that there is no downward shift in the wear track as compared to the unworn surface which confirms the absence of linear wear. Note that the roughness of the wear track is almost the same as on the unworn surface. In contrast, significant wear is observed on the reference seals which were not coated with UNCD films. A significant decrease in thickness is observed in the profilometer traces measured on these seals after only the running in period at low water pressure (20 and 40 psi) as shown by the optical profilometer measurements in Fig. 7. The linear wear value of the uncoated SiC seal is more than 1 μm according to numerical data of the wear track profile (Fig. 7b). Furthermore, a large accumulation of transferred graphite and wear debris is visible near inner edge of the wear track in 3D image (Fig. 7a) and in wear track profile (Fig. 7b).

These results clearly demonstrate the advantages of UNCD coatings for improving the wear resistance of the SiC seals. However, wear behavior of the 1st group of UNCD coated seals ($R_a$ = 0.05–0.07 μm, polished by 6 μm diamond powder) differs considerably from the other two seal groups prepared to have a higher initial surface roughness. Delamination of the UNCD films is observed even after running-in at 20 psi water pressure during 8 h for the seals in the 1st group. Furthermore, the delaminated area increased in extent as the wear test progressed for longer times and higher water pressures. Fig. 8a and b shows profilometer data for the wear track following the UNCD film delamination. The wear track can be divided into three zones:

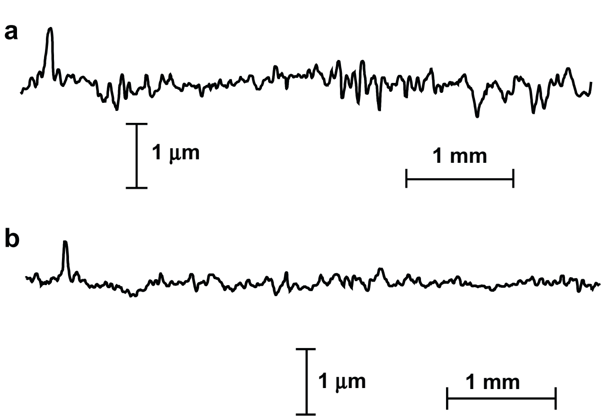


**Fig. 6.** Wear track profiles on UNCD-coated seals of the 2nd group (a) and 3rd group (b).

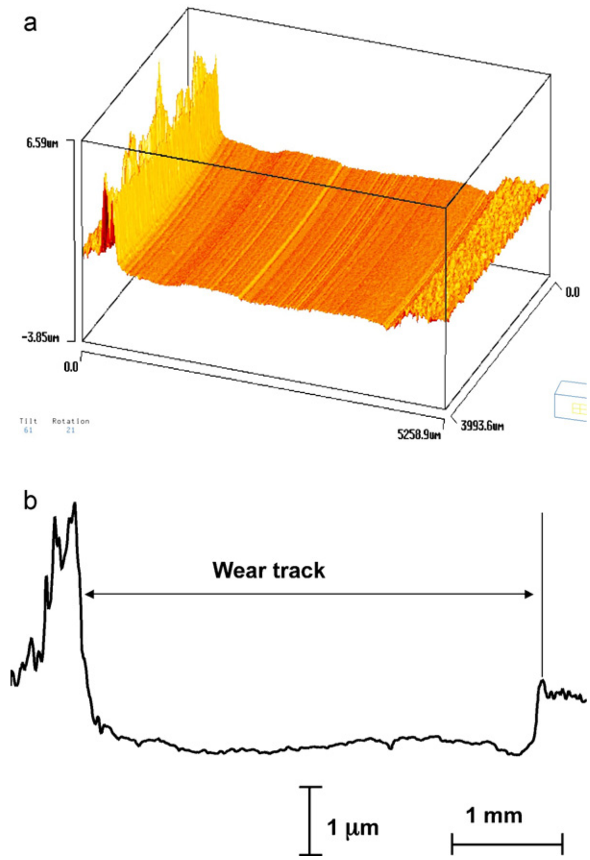


**Fig. 7.** Three-dimensional image (a) and two-dimensional profile (b) of the wear track on a reference uncoated SiC seal after testing measured using optical profilometry.

(1) Intact zone: This zone is still covered by the intact UNCD film and the morphology does not differ from the unworn surface area. The average profile line of this zone is the same as in the unworn surface out of wear track and thus linear wear is absent.
(2) Delamination zone: The depth of this zone, according to the profile, is about 1 μm which is equal to the thickness of the UNCD coating. Furthermore, there are abrupt transitions where the height changes suddenly by ∼1 μm. These observations suggest delamination of the UNCD film. It is likely that the cause of the delamination is insufficient adhesion between UNCD films and SiC substrate.
(3) Debris zone: This zone covered by transferred graphite from the counterface. The transferred graphite sometime accumulates in the form of longitudinal ridges on the wear track, and sometime the graphite fills areas where the UNCD film has delaminated.

No water leakage was observed during the entire tests for both the UNCD-coated and the uncoated seals. As described earlier, no measurable wear was revealed for UNCD-coated SiC seals from the 2nd and 3rd groups. The most intense wear for the uncoated reference seals was observed during the running-in period. After reaching a wear track depth of ∼1 μm, the wear rate decreased considerably and to below 0.1 μm per 50 h of tests at 100 psi (0.7 MPa) water pressure. The decrease in average thickness of the graphite rings tested in contact with the reference uncoated SiC seals was no more than 10 μm and was observed only during the running-in period. The thickness of the graphite rings remained constant during the steady-state tests. Similar behavior was observed for the graphite rings running against all three groups of the UNCD-coated seals, however, the decrease

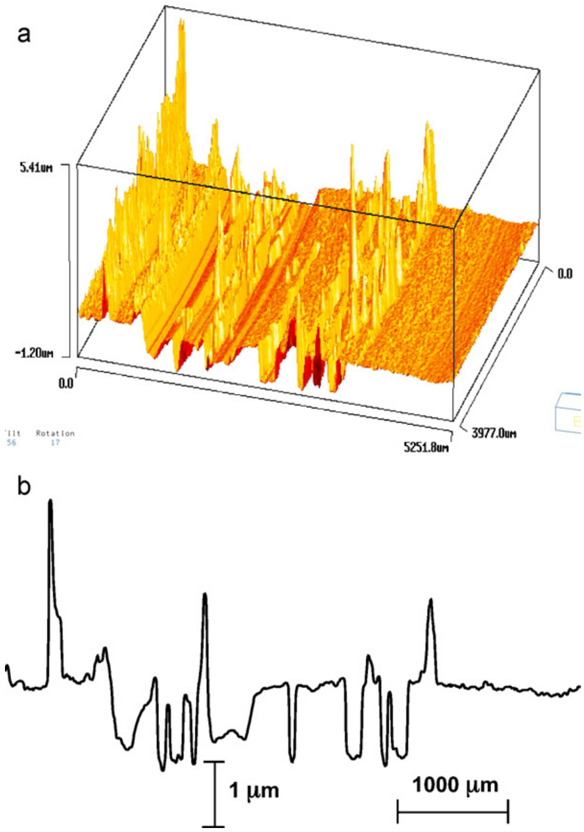


**Fig. 8.** Three-dimensional image (a) and two-dimensional profile (b) of the wear track on a UNCD-coated seal from the 1st group (minimum roughness) after testing measured using optical profilometry.

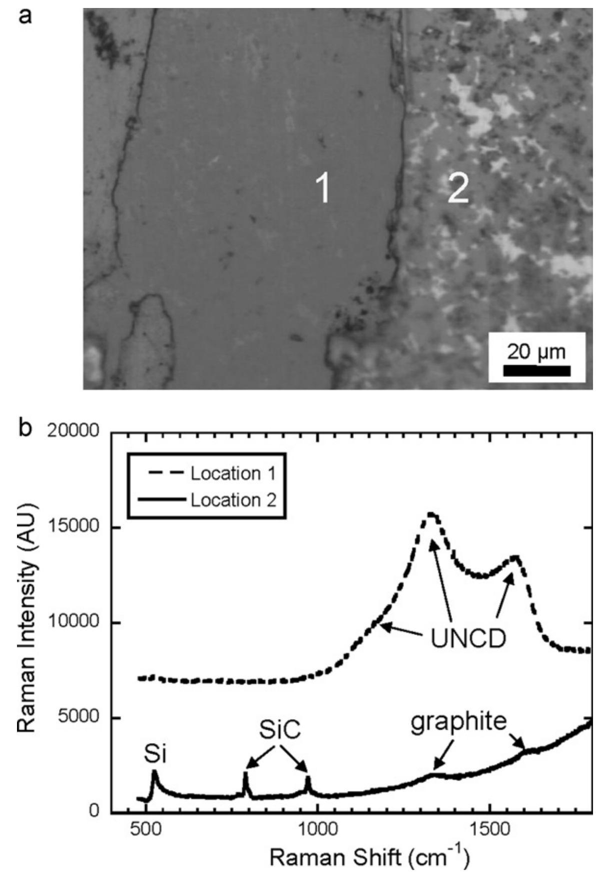


**Fig. 9.** Optical image (a) and Raman spectra (b) from a UNCD-coated seal of the 1st group after testing. Raman spectra are recorded from the numbered locations in the optical image.

in average thickness of the graphite rings fell in the range of 10–40 μm.

To investigate the delamination zone, Raman spectra were recorded from the SiC seals in the 1st group following wear testing. Fig. 9a is an optical microscope image showing the edge of the delaminated zone between the intact UNCD film (region 1) and the delaminated area (region 2). Fig. 9b presents Raman spectra recorded from regions 1 and 2. The Raman spectrum from region 1 shows only features characteristic of UNCD at 1330 and 1770 cm$^{-1}$ with a shoulder at 1120 cm$^{-1}$. In contrast, the Raman spectrum recorded from the delaminated area in region 2 shows the characteristic silicon peak at 524 cm$^{-1}$ and silicon carbide peaks at 791 and 973 cm$^{-1}$. These features in the Raman spectrum from region 2 are identical to the spectral features recorded from an uncoated SiC seal. In addition, the region 2 Raman spectrum exhibits small peaks from graphitic carbon at 1350 and 1600 cm$^{-1}$. This graphite probably results from abrasion and transfer from the graphite counterface during the wear testing.

These Raman results confirm that UNCD films delamination has occurred due to rupture of the interface between UNCD films and SiC substrate indicating that the cause of the delamination is insufficient adhesion. Raman spectroscopy performed on the seals from groups 2 to 3 following wear testing revealed only UNCD spectral features and showed no peaks associated with the SiC substrate. These findings agree with the optical profilometry measurements that no delamination occurred from these surfaces.

SEM measurements were also performed on the group 1 SiC seals following the wear testing. Fig. 10 shows a low resolution SEM image acquired with the backscattered electron (BSE) detector showing the entire wear track. The inner edge of the SiC seal is visible in the top right corner of the image. The delaminated area (marked SiC) is clearly seen as the white regions in Fig. 10 because the higher average atomic number ($Z = 10$) of the SiC generates a higher intensity of backscattered electrons than the UNCD ($Z = 6$). Higher resolution SEM images from the same region of the group 1 SiC seal were acquired using secondary electron detection (SE, Fig. 11a) as well as BSE (Fig. 11b). By comparing the images acquired with the SE and BSE detectors, it is evident that in addition to the intact UNCD and SiC from the delaminated areas, there are regions of graphite as marked in Fig. 11. The graphite shows up at a similar intensity to the UNCD in the BSE image because both materials are pure carbon. In the SE image, however, the higher conductivity

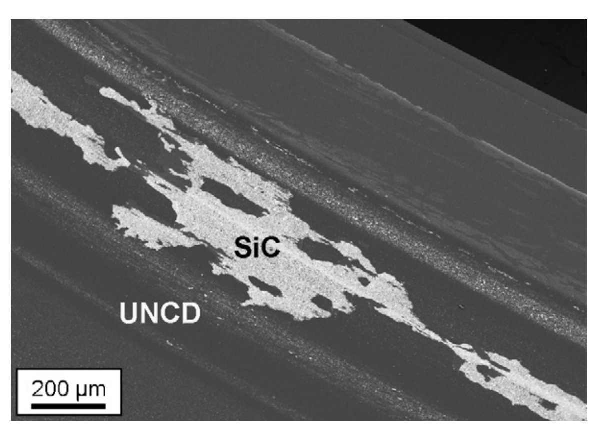


**Fig. 10.** BSE image of UNCD-coated SiC seal from the 1st group after testing.

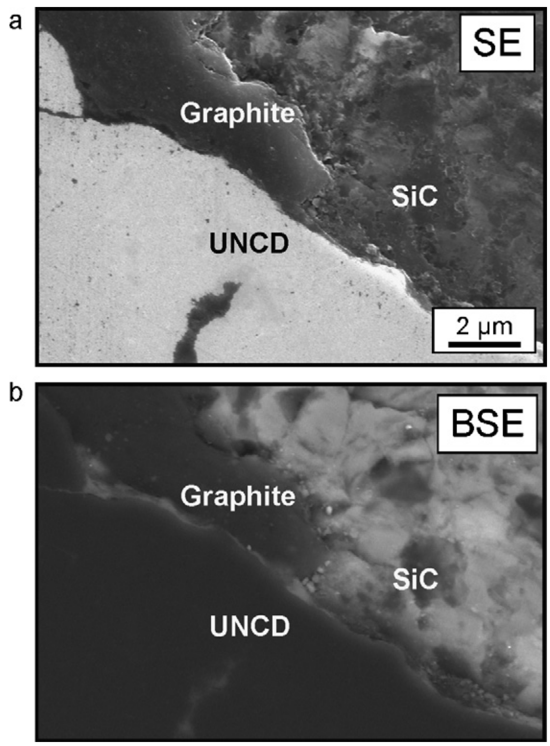


**Fig. 11.** SE image (a) and BSE image (b) of UNCD-coated SiC seal from the 1st group after testing.

graphite appears darker than the UNCD because the UNCD charges slightly under illumination from the SEM probe beam. The presence of graphite in the SEM images agrees with the Raman results, and suggests that transfer has occurred from the graphite counterface during the wear testing.

To quantify the adhesion of the UNCD films to the SiC seal surfaces, pin-pull adhesion testing was performed on the UNCD coated SiC seals following the wear testing. The adhesion tests were performed using regions of the UNCD coating that were outside of the wear track so were unaffected by any possible damage introduced during the wear testing process. For the group 1 seals, delamination of the UNCD films was observed to occur at ~8000 psi. Delamination was evident by visual inspection of the SiC seal surface as well as the pull pin, which both revealed that the UNCD film had broken free from the SiC and was attached to the pin. This result was confirmed using Raman spectroscopy which showed that the material transferred to the pin was UNCD, while the delaminated area on the seal surface showed silicon and SiC spectral features. Similar adhesion measurements performed on the group 2 and group 3 seals did not show any delamination. No UNCD was removed from these rougher SiC surfaces even at the maximum testing force of ~12,000 psi.

Our investigations clearly showed that the delamination of coating depends on the adhesion between the UNCD coatings and the SiC substrate. Generally speaking, adhesion is the most challenging problem for coatings intended to reduce friction and wear. All of the advantageous properties of the coating are jeopardized by poor adhesion, and consequently a great deal of research is devoted to increasing the adhesion strength of the interface between the coating and the substrate. This adhesion strength is affected by a number of parameters including the coating thickness which affects residual stress, the choice of substrate material and underlayer, the preparation of the substrate material prior to coating, etc. In this work we have demonstrated that the relatively simple method of roughening the SiC substrate by polishing with diamond powder dramatically improves the adhesion of the UNCD film. Presumably the effect of the diamond polishing is to increase the interfacial surface area between the SiC and the UNCD so that even if the specific adhesion per unit area remains the same the overall adhesion increases with increasing roughness of the SiC surface. We should note that increasing the roughness of the SiC may have a detrimental effect if this also increases the roughness of the final seal surface after UNCD coating since this may induce abrasive wear of the mating counterface. However, we did not observe significant wear of the contacting graphite rings in our study.

## 4. Conclusions

The effect of initial surface roughness of SiC seals on the adhesion of UNCD films and the wear performance of these UNCD-coated seals has been investigated. Our results demonstrate that increased roughness of the working surface of the SiC mechanical seals significantly improves the adhesion of the UNCD film. This led to negligible wear of the UNCD coated seals in real working sliding conditions. Our results clearly demonstrate the utility of UNCD films on SiC seals for improved wear resistance. We obtained the same positive results in the case of adhesion measurement as in wear resistance assessment after tribotests for UNCD-coated SiC seals of the second and third groups (polished by 12 and 30 μm diamond powder, respectively). No delamination was observed using pin-pull adhesion measurement up to the instrument limit (>12,000 psi) and practically no wear was revealed after tribotests. This is in contrast with UNCD-coated seals of the first group (polished by 6 μm diamond powder) for which delamination was revealed in both adhesion measurements and in profilometry performed after the tribotests. UNCD coated SiC seals of the second and third groups have advantages in wear resistance against references uncoated seals. Some increased wear of the graphite counterface rings during the running-in period is eliminated during the final steady test period for all of the seals.

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