

DIAMOND LIKE CARBON COATINGS—TRIBOLOGICAL POSSIBILITIES AND LIMITATIONS IN APPLICATIONS ON SINTERED SILICON CARBIDE BEARING AND SEAL FACES

by

Michael Fundus
Product Manager
ESK-AC/PD Division

Kempten, Germany
and

Heinrich Knoch
Product Manager
ESK Engineered Ceramics
Norwalk, Connecticut



Michael Fundus is a graduate of the University of Karlsruhe, Germany, with an M.S. degree in Mechanical Engineering. After joining ESK, Mr. Fundus worked four years in the F&E department and customer support group, heading ESK's work on ceramic design and materials and components testing. For another two years, he was responsible for the process technology of sintered silicon carbide components. He is now working as a sales engineer in the marketing and sales group of ESK. Mr. Fundus has authored and coauthored 20 technical and scientific papers, publications, and patents.



Heinrich Knoch is a graduate of the University of Erlangen, Germany, with a Ph.D. degree in Engineering and an M.S. degree in Materials Science. Before joining ESK he spent five years at DLR, the German aerospace research establishment and one year at the Army Materials Technology Lab in Watertown, Massachusetts, doing research on ceramics. At ESK he was working in the engineering and marketing management of ceramic products. Currently, he is a product manager for the American market. Dr. Knoch has authored and coauthored approximately 75 technical and scientific papers, publications, and patents.

ABSTRACT

Promising new developments to master friction and wear under dry run conditions are diamond like carbon coatings. They offer high hardness and wear resistance, combined with a low coefficient of friction. Best coating results are achieved when deposited on sintered silicon carbide surfaces. DLC coatings on SSiC seal and bearing faces have been evaluated. Tests show that with DLC coatings coefficients of friction as low as 0.05 can be achieved in dry run. Loading conditions and design criteria determine the application limits of the coatings, which are outlined. The typical failure mechanisms of coatings are discussed. Application

examples are given for gas seals and pump bearings, where in dry run situations, frictional failure might be a concern. It is also shown that DLC coatings give only excellent results when used on a well designed component under low loads.

INTRODUCTION

Sintered silicon carbide seal faces and slide bearings are successfully used in process pumps dealing with corrosive and abrasive fluids. Universal corrosion resistance, excellent wear resistance, and good thermal shock resistance of this material helped to solve many sealing problems and to reduce downtime and leakage of process pumps. Media lubricated SSiC bearings were essential for the breakthrough and success of zero leakage sealless pumps, important to meet requirements of the Clean Air Act.

The main reason for the success of SSiC is the good tribological properties. Typical friction coefficients of lubricated SSiC pairs range from 0.001 to 0.01 in bearings and up to 0.1 in mechanical seals. Even under marginal lubrication conditions, coefficients of friction from 0.1 to 0.3 can be maintained. The situation is different in really dry "bone-dry" conditions. In SSiC pairs, the coefficient of friction can increase to values of 0.7. Whether this dry run leads to critically high temperatures, thermal shock failures, and accelerated wear and damage (catastrophic failure), very much depends on the loading conditions (PV) and design. Test rig results on correctly designed bearings have shown that SSiC bearings can run dry for several hours without showing signs of damage. In contradiction, bearings can fail within a very short time, if high loads are applied and/or the bearing is running on the edges.

A promising new development to master friction and wear under dry run conditions are diamond like carbon (DLC) coatings. They offer high hardness and wear resistance combined with a low coefficient of friction of < 0.2 . Best coating results are achieved when deposited on SSiC surfaces. It is shown herein that DLC coatings are offering very low friction values, but it is also shown that they are very vulnerable to high loads. All the reported test results have been performed on commercially available coatings from different sources.

DLC COATINGS

Basics

Diamond like carbon coatings (DLC), also sometimes called amorphous hard carbon coatings, can be manufactured using a variety of techniques and, consequently, result in a relatively wide

property range of the coatings. The most common coating methods are chemical vapor deposition (CVD) and plasma-assisted chemical vapor deposition (PCVD). The latter process probably produces the best “tribological” coatings on silicon carbide surfaces. It is the amorphous nature of these films that produces very smooth surfaces, essential for sliding applications where low friction and wear are desirable. Polycrystalline diamond films, in contrast, do not have a smooth surface, and they cannot be used for low friction and low wear applications. Herein, the authors describe only some basic properties of DLC coatings and refer the interested reader to the literature [1, 2, 3, 4].

CVD and PCVD coatings contain a relatively high amount of hydrogen (up to 60 percent is possible). The hydrogen, on one hand, helps the stabilization of a short range diamond lattice coordination, which is the cause of the coating’s hardness. It is, however, also the cause for the thermally unstable nature of the coating. There is always a tendency of C atoms to “relax” from a higher energy diamond configuration into a lower energy graphite configuration. (The DLC film changes from “diamond like” to “graphite like.”) This tendency is counteracted by the internal compressive stresses in the coating created by the change of the bonding configuration. This very nature of DLC coatings implies that compressive stresses are unavoidable, and that in effect, these coatings are self stressing. The negative consequence of these stresses lies in the fact that only very thin coatings can be applied, typically one to two μm , before the coating is destroyed by its own internal stresses: spalling, cracking, delamination.

Over the years, research and development on DLC coatings has been concentrated on reducing internal stresses, improving the bond strength of the coating to the substrate, and improving the thermal stability. In order to improve the bond strength of the coating to the substrate, intermediate coatings containing Si and/or Cr atoms have been tried.

Si, Ar, O, and N atoms are built into the coating to influence and improve properties. For example, metal containing hydrogenated carbon films (Me-DLC) show reduced internal stresses and low friction coefficients [1].

The production of high quality coatings typically comprises three steps:

- Cleaning the substrate surface (sliding face of a seal or bearing component) with an Ar plasma.
- Deposition of an intermediate coating.
- Deposition of the DLC coating.

SEM micrographs are shown in Figures 1, 2, and 3 of the surfaces of DLC coatings deposited on a sintered silicon carbide surface. Typical for all deposited DLC films is the orange skin, or cauliflower appearance, of the surface.

Failure Criteria

Application limits and lifetime of DLC coatings are controlled both by the sliding conditions (PV, environment) and by design. Three major causes of coating failure can be determined:

- *Overload*—If the coatings suffer localized overload, the bond strength of the film to the substrate might be exceeded and delamination occurs. Sudden increases of friction are observed. The debris of the delaminated film between the sliding faces will lead to further damage of the coating, so the friction will stay high.
- *High Spot Temperatures Caused by Friction*—At high PV values, hot spots are produced at localized frictional contacts, leading to a thermal degradation and decomposition (diamond graphite) of the coating. Calculations have shown [5] that a sliding velocity of six m/s and a specific load of 0.8 N/mm^2 (110 psi) can result in spot temperatures of 550°C (1000°F) in dry run. This is calculated for a coefficient of friction of 0.05.

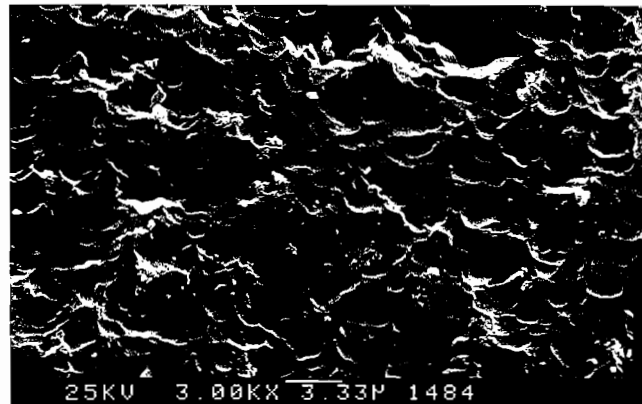
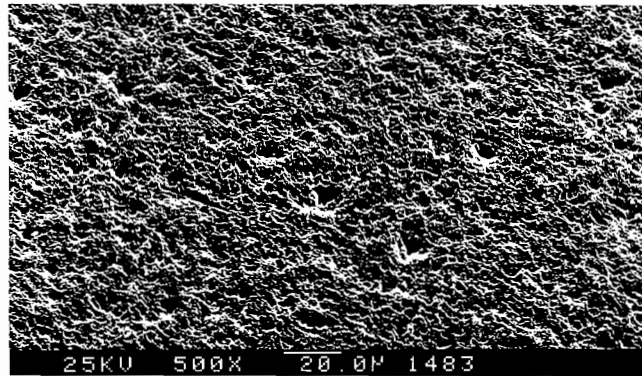


Figure 1. SEM Micrograph of Topography of DLC Coating on SSiC, Coating DLC2.

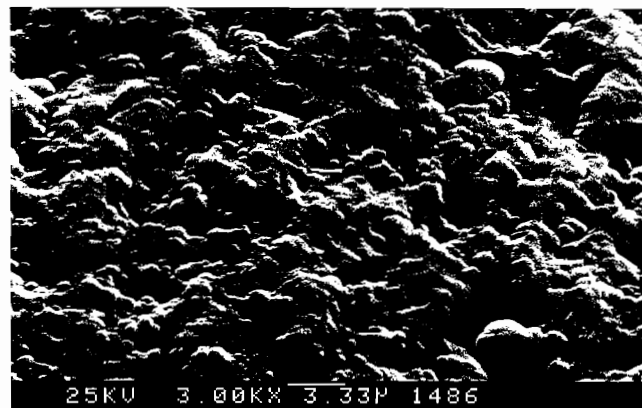
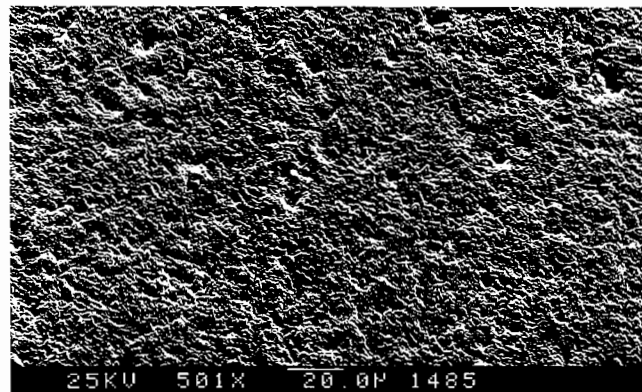


Figure 2. SEM Micrograph of Topography of DLC Coating on SSiC, Coating DLC4.

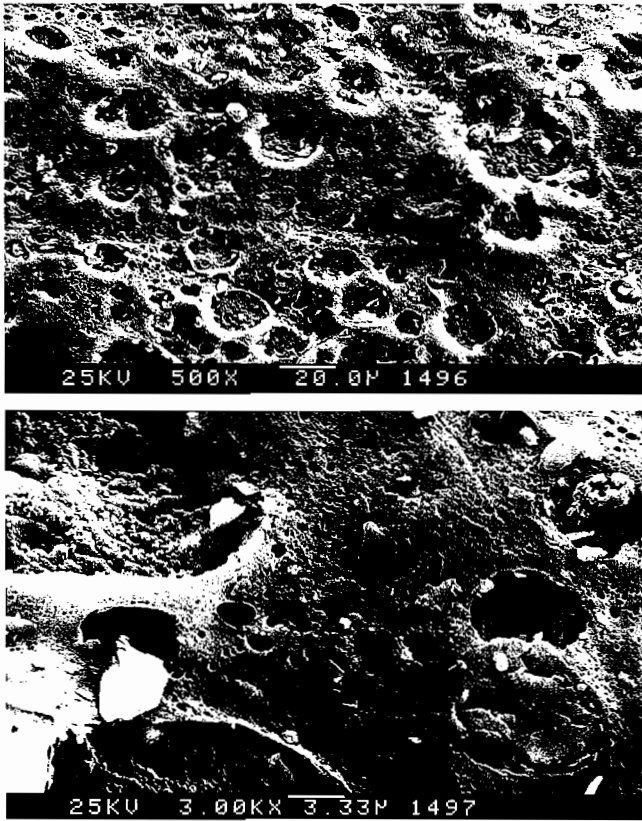


Figure 3. SEM Micrograph of Topography of DLC Coating on SSiC, Coating DLC5.

• **Regular Wear**—Dependent on DLC film properties and sliding conditions a certain wear rate will be reached, which after a certain time, will wear out the film and no coating will be left on the surface.

Dry Run Tests

Testing was done on a test rig designed for dry running. Ring-shaped samples were run axially against each other, similar to the faces of a mechanical seal. The flatness of the sliding faces was always better than one μm to assure full face contact. The test rig allowed control of speed and load. The friction was measured at the stationary ring, using a load cell measuring the friction induced torque. All tests were performed under normal laboratory conditions. The sliding velocity was 1.3 m/s. The nominal load was 0.2 N/mm^2 (28 psi).

When the tests were begun, during the running in period, effective loads of up to $> 2 \text{ N/mm}^2$ (280 psi) could be achieved, which leveled after running in, to the nominal value. Coefficient of friction and sample temperature were monitored during the tests.

Out of a multitude of coating samples tested, the results of five typical coatings are presented. All of these coatings were tested against themselves and against sintered silicon carbide surfaces. The surface roughness of the virgin samples, both silicon carbide and DLC coating, was $0.2 \pm 0.1 \mu\text{m}$.

An example of the powerful potential of DLC coatings to decrease friction under dry run is shown in Figures 4 and 5. After running in, the plain sintered silicon carbide surfaces reach a steady state coefficient of friction of < 0.7 . In contrast, DLC coated samples start at coefficient of friction values of < 0.2 and reach steady state values of 0.05. In the same time, the frictional heat leads to temperatures of $> 220^\circ\text{C}$ (430°F) in the silicon carbide

samples, whereas the reduced friction of the DLC coated samples leads to temperatures of only 40°C (100°F).

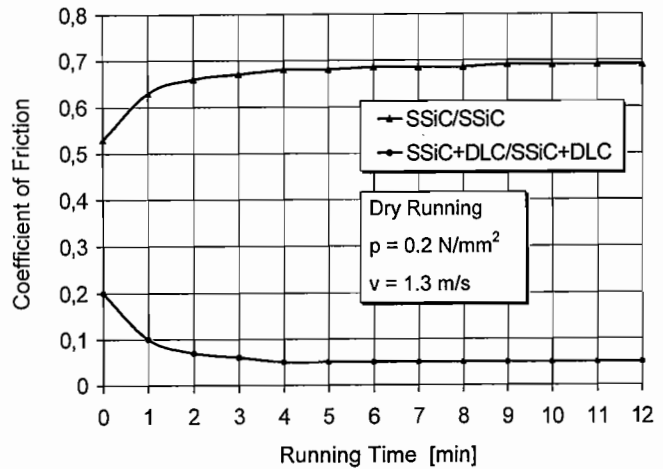


Figure 4. Coefficients of Friction under Dry Running Conditions of DLC Coated and Uncoated SSiC Pairs.

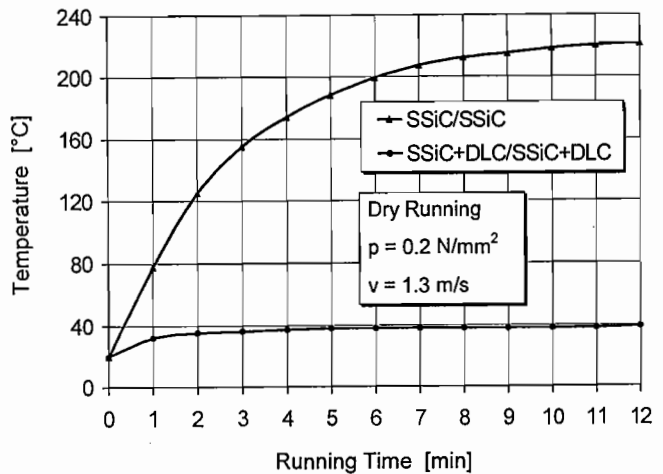


Figure 5. Frictional Temperatures of DLC Coated and Uncoated SSiC Pairs.

For a more complete characterization of the coatings, some further property data is shown in Table 1, including hardness, Young's modulus, thickness of the film, and the critical load in the sclerometer test.

Table 1. DLC Coating Property Data in Comparison to SSiC.

		SSiC	DLC1	DLC2	DLC3	DLC4	DLC5
Hardness	[GPa]	28	30	32	28	20	12
Young's Modulus	[GPa]	420	260	290	190	195	140
Thickness Interlayer/Coating	[μm]		0/1.7	1.0/1.8	1.2/2.4	0/2.5	0.4/1.9
Critical Load Sclerometer Test	[N]		1-5	20-30	15-25	40-70	40-65

With each of the coatings, four tests were done with both slide faces DLC coated—equal pairs, and four tests with one face DLC coated, the other slide face uncoated silicon carbide—mixed pairs. The obtained results are shown in Figure 6, indicating the

minimum and maximum dry running times that could be achieved with a certain coating/pair. The measured lifetimes range from minutes to several hours.

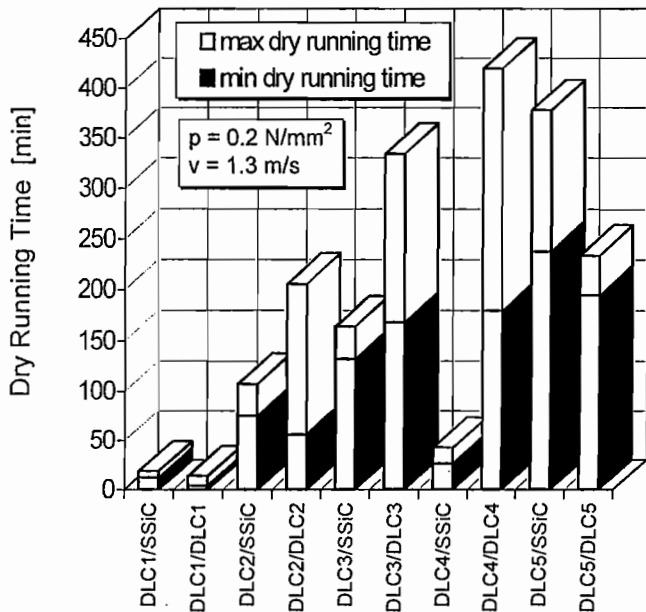


Figure 6. Minimum and Maximum Lifetime of Different DLC Coatings in Dry Run Testing.

DLC1 is of high hardness, however, as a consequence of very poor bond strength of the coating to the substrate, there is only very short life, both against itself as against SSiC. The failure mechanism is delamination of the film.

DLC2 is of the same coating type as DLC1, shown in similar values of Young’s modulus and hardness. In addition to DLC1, it contains an intermediate layer of one μm thickness to improve its bond strength to the substrate.

DLC3 has slightly thicker layers of both intermediate and outer coating. Young’s modulus and hardness are lower. This change resulted again in increased wear life. With DLC2 and DLC3 it can also be seen that the longer life can be achieved with equally coated pairs. However, in mixed pairs, the scatter of results is much less.

DLC4 is different in structure and hardness when compared with DLC1, 2, and 3. In spite of not containing an intermediate layer, the bond strength is very good. The coating achieves excellent life when run against itself. Against SSiC, it wears out quickly, because its hardness is too low.

DLC5 is a multilayer coating. Despite its low hardness, this coating exhibited good dry running performance and little wear, both with equal and mixed pairs. The coating has a very good bond strength.

Additional tests were performed to evaluate the performance limits of the coatings under higher loads. With $v = 1.3 \text{ m/s}$ and a nominal load of one N/mm^2 (140 psi), the achieved lifetimes under dry run conditions ranged from seconds to 20 min max.

APPLICATION EXAMPLES

Gas Seals

Centrifugal compressors can be sealed by means of oil or gas lubricated seals. The trend goes to gas lubricated seals, because they offer contact free operation with minimum wear of the seal faces, minimum power dissipation, and minimum leakage. In addition, the required environmental control systems are easier to

install and lighter than those for oil seals. This leads to an investment and operating cost advantage of dry gas seals over wet systems.

In dry gas seals, one of the faces contains grooves that allow gas to penetrate between the seal faces. When the seal rotates, these grooves pump gas between the faces, which creates a lift off pressure and causes the faces to separate. Under operating conditions, a defined gap setting is reached, which also determines a defined leakage [6].

For the reliable performance of dry gas seals, it is essential that the very narrow gap between rotor and stator remains geometrically stable in operation, and it is also essential that there is no wear on the faces after startoffs and shutdowns, when the faces come into solid contact. These demands ask for “stiff” and wear resistant seal face materials.

It has been shown that carbon graphite materials can be used for dry gas seals [6], however, due to the compliant nature of these materials, the achievable gas pressure limit to be sealed is low, <10MPa (1400 psi). In order to seal pressures > 10 MPa (1400 psi) in one stage, materials with higher stiffness are required. These materials should also have a good dry running capability.

Seal faces of choice are sintered silicon carbide for high stiffness, and the same material with a DLC coating to lower the coefficient of friction in dry run and improve the overall dry run performance.

Comparative tests were performed with sintered SiC rings, with and without DLC coated faces. Test conditions used [7] are shown in Table 2. Three typical operating phases of dry gas seals were simulated: start/stop, operation in the vicinity of the lift off speed, and nominal operating conditions.

Table 2. Operating Conditions of Gas Seal Tests [7].

	Medium	Air
Pressure	[MPa]	0-12
Speed	[rpm]	100-20000
Peripheral Speed	[m/s]	0-105
Max. Temperature	[°C]	180

In addition to the results [7] listed in Table 3, there is another convincing result: the break away torque of the DLC coated silicon carbide surfaces is only 50 percent of that of the uncoated material, Figure 7. The film reduces the coefficient of friction and, after the test runs, there was no visible damage of the faces. In contrast, the uncoated surfaces did reveal some scarring and damage after the applied testing, Table 3.

The accumulated tests and field experience confirm a very good behavior of gas seals with carbon graphite faces for pressures of <10MPa (1400 psi) only. For high pressure gas seals, $p > 10 \text{ MPa}$ (1400 psi) per stage, materials with high stiffness should be used. The dry running capability of DLC coatings, together with the high stiffness of silicon carbide, can produce reliably working gas seals for high pressures and temperatures of 300°C (570°F).

Pump Bearings

Dry running performance of sintered silicon carbide bearings in sealless pumps, both mag drives and canned motor pumps, has raised some controversial discussions over the past few years.

There is one party who claims (and they prove it) that their pumps, equipped with long-lasting wear resistant silicon carbide bearings, are running dry without any problem for a shorter or

Table 3. Seal Face Appearance of Sintered Silicon Carbide Rings with and without DLC Coatings after Gas Seal Tests [7].

Test conditions	Face Appearance	
	Rotary SSiC	Stationary SSiC
Start/Stop 50 cycles at 0, .2, .5, 1, 3, 5 MPa Gas Pressure	wider wear scar at the ID narrow wear scar at the OD	seal face roughened and scored
Endurance Run at 0 MPa Gas Pressure 100 h 100 rpm	wider wear scar at the ID narrow wear scar at the OD	seal face roughened and scored
Endurance Run at 12 MPa Gas Pressure 100 h 20,000 rpm	---	---
	Rotary SSiC with DLC Coating	Stationary SSiC with DLC Coating
Start/Stop 50 cycles at 0, .2, .5, 1, 3, 5 MPa Gas Pressure	hardly visible contacts at ID	like new
Endurance run at 0 MPa Gas Pressure 100 h 100 rpm	few shiny spots at ID	like new
Endurance Run at 12 MPa Gas Pressure 100 h 20,000 rpm	like new	like new

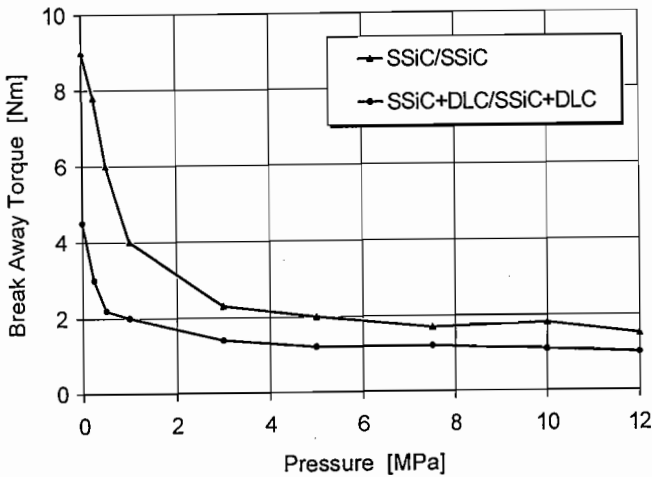


Figure 7. Break Away Torque as a Function of Gas Pressure of DLC Coated and Uncoated SSiC Gas Seal Rings [6].

longer period of time, and there is the other party who claims (and they prove it) that their experience with dry running silicon carbide bearings is nothing less than catastrophic failure.

How can this discrepancy be explained? Can DLC coatings help?

The frictional energy that is dissipated into a bearing face is directly proportional to the coefficient of friction, the sliding speed, and the applied load. Even if the applied loads are small (related to the bearing size), there can be very high effective loads if the bearing faces contact each other along a line, or even worse, at a point. If this is happening in a bone-dry situation where there is no liquid left to cool or to lubricate, then the silicon carbide coefficient of friction of dry run (~ 0.7) produces very high temperatures along the line or at the point of contact. This can lead to localized material damage, which immediately spreads into the bearing face and destroys it.

The first straightforward strategy to avoid point or line loading situations is to apply the floating or compliant design principles for the bearing together with a closely tolerated machining finish. The mastering of this task results in the “shorter or longer” survival times under dry run. In other words: if the bearing design is such that the total load is low and load concentrations do not occur, then sudden temperature increases will not happen.

The next strategy to keep the frictional energy low is to apply DLC coatings. The frictional energies shown in Table 4 are generated in sintered silicon carbide bearings of plastic lined mag drive pumps with and without DLC coating [8]. As can be seen, the frictional energy generated with coating is less than 10 percent of the energy generated without. Accordingly, the temperature of the bearing stays low and there is no danger of temperature related bearing failure, Figure 8.

Table 4. Frictional Energies Generated in Sintered Silicon Carbide Bearings of Plastic Lined Mag Drive Pumps with and without DLC Coating under Dry Running Conditions (n = 2900 rpm) [8].

Pump size	Frictional Energy [J/s]	
	SSiC bearings without coating coefficient of friction 0.6	SSiC bearings with DLC coating coefficient of friction 0.04
1	98.8	8.6
2	198.9	13.3
3	345.2	23.0
4	575.8	38.4

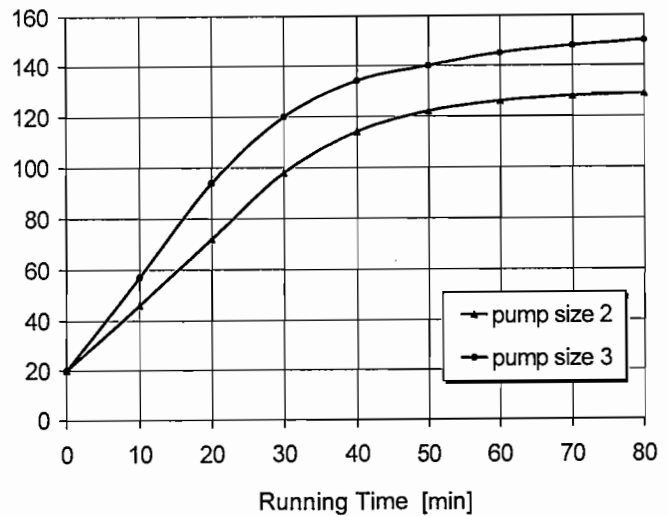


Figure 8. Typical Bearing Temperatures under Dry Running Conditions for Two Pump Sizes [8].

For real world applications of a pump, it is essential that the DLC coating has a long life. Wearing out of the coating must be avoided. It would be absolutely critical if a mag drive pump used to empty a tank would fail suddenly after some service life, because the coating wore out and the bearings did not work under marginal lubrication conditions. As was shown in the dry run tests, the wear life of DLC films depends on the load. Under high loads the coatings fail quickly. This means that the benefits of a DLC film in a long service life can only be exploited if high loads can be avoided. Good design avoiding line and point contacts is the prerequisite to achieve this. It needs a good bearing design and a DLC coating on the bearing faces to successfully master the dry running performance of SiC bearings in pumps.

DISCUSSION

The investigation of the tribological properties of DLC coatings on sintered silicon carbide surfaces clearly shows that the coefficient of friction in dry running can be lowered to values of ~ 0.05, which is otherwise only achieved under lubricated conditions. This low coefficient of friction is achieved both for

DLC films running against themselves and for DLC coatings running against sintered silicon carbide.

The low friction values decrease the generation of frictional heat on sliding couples. The probability of creating hot spots, which cause damage to the sliding faces, is minimized.

The hardness and wear resistance of these films and their corrosion resistance offers the opportunity to apply long lasting low friction coatings on seal and bearing faces. One could be tempted to announce: the dry running of sintered silicon carbide bearing and seal faces is no problem anymore.

However, this is wishful thinking. As is shown in Figure 6, the life of DLC coatings is limited, and it is controlled by the load applied to the coating. Above loads of $\sim 1 \text{ N/mm}^2$ (140 psi), the lifetime of the film is very short and is limited to minutes.

It must also be stated that at higher loads, when at point contacts the generated contact stress exceeds the bond strength of the coating, the overload damages and destroys the coating. There are coatings with higher bond strength and higher contact strength, but the problem remains. In most cases, longer maximum lifetimes in dry run are achieved when both sliding partners are coated. When coatings are running against SSiC, similar minimum dry running times are achieved (Figure 6). Since the minimum life must be looked at as the design criteria, it is often sufficient and economically favorable to coat only one surface.

In order to utilize the low friction benefits of DLC coatings on SiC sliding faces in dry run, the effective loads on the sliding faces must be low (less than 0.2 N/mm^2 (28 psi)), and load concentrations must be avoided. These are requirements that are met (or missed) by design principles and by machining/finishing operations. If these basic requirements (compliance, roundness, flatness) are ignored, then there will be load concentrations on the seal or bearing faces in operation, which will quickly destroy the low friction film. A DLC film is, by no means, a cure for poor component manufacturing or false design. It is, for that reason, the absolutely false approach to apply a DLC coating in sliding systems—seal or bearing—if dry run and solid state friction occurs as a consequence of overload that squeezes out the hydrodynamic lubricant film between the faces and forces solid contact.

The applicable loads in dry run must be compared to the load-bearing capability of sliding faces supported by a hydrodynamic lubricant film: 0.2 N/mm^2 (28 psi) is high for dry running DLC surfaces, 10 N/mm^2 (1400 psi) is quite normal for hydrodynamically supported SiC tilt pad bearing faces. This comparison gives an important advice: although DLC coatings help to lower friction in dry run, always design for lubrication and try to prevent dry run. If a lubricant film is breaking down as a consequence of overload, this very load cannot be supported in dry run by a DLC film either. This is very important to be considered in a "real" pump application. As long as the pump is in hydraulic operation, i.e., as long as the hydraulic operating conditions determine the load on the bearings (including pressure pulsations and vibrations), it must always be kept in mind that the load-bearing capability of a hydrodynamic lubricant film is much larger than the load-bearing capability of a DLC film: one to two orders of magnitude. Consequently, the handling of hydraulic loads has to be tackled by means of hydrodynamics (bearing design). DLC films offer their advantages under dry circumstances and low loads only.

If the loading conditions of a seal face or a bearing face are "in favor" of a DLC coating, then applications can be mastered that are otherwise not possible, or improvements of applications can be achieved. The reliability/safety in dry run situations will be improved.

Application examples have been given previously. If the SiC bearing of a plastic pump running dry is developing too much heat, then a DLC coating can lower the frictional energy, so that the bearing remains relatively cool, even running dry. A straightforward application of such coated bearings could be in self priming

pumps and in vertical pumps, where the bearings have to start running dry.

Another example for the benefits of a DLC coating is its application on seal faces of high performance gas seals. Seal faces of gas seals are accurately designed and closely tolerated. Normally operated, the seal faces liftoff when running and they only contact each other on shutdowns and start offs. It must be secured that no wear occurs on the contacting faces. In this situation, DLC coatings offer an extra reliability/safety factor.

DLC films must be looked at as means to improve the performance of bearing or seal faces in dry run situations and to make them more reliable. However, they must be used properly. They are of no use if the loading conditions cannot be controlled.

It is highly recommended to proof test in special cases, when it is not absolutely sure whether a DLC coating can master a certain task.

CONCLUSION

The results show that DLC coatings offer a good potential to improve the reliability of sintered SiC components in seal and slide bearing applications when dry run situations must be handled.

The limitations of these coatings in terms of tolerable load and temperature must be taken into consideration. Another important point is to choose the right DLC quality, among the different coatings available on the market.

A good appropriate design for sintered SiC bearings or seal faces is the key for reliable performance. DLC coatings can push the dry run limits. They cannot, however, guarantee unlimited dry run performance, and they especially cannot make up for inappropriate design, which results in point or line load of the bearing/seal faces.

REFERENCES

1. Grill, A., "Review of the Tribology of Diamond-Like Carbon," *Wear*, 168, pp. 143-153 (1993).
2. Erdemir, A. and Fenske, G. R., "Tribological Performance of Diamond and Diamondlike Carbon Films at Elevated Temperatures," STLE Annual Meeting, Cincinnati, Ohio (1996).
3. Oguri, K. and Arai, T., "Low Friction Coatings of Diamond-Like Carbon with Silicon Prepared by Plasma-Assisted Chemical Vapor Deposition," *Journal of Materials Research*, 5, (11), pp. 2567-2571 (1990).
4. Gardos, M. N., "Tribology and Wear Behavior of Diamond," *Synthetic Diamond: Emerging CVD Science and Technology*, New York, New York: John Wiley & Sons, Inc., pp. 419-503 (1994).
5. Berndt, F. and Ziegler, G., "Beschichtung von SiC mit Diamantähnlichem Kohlenstoff für Tribologische Anwendungen," *Abschlußbericht Forschungsvorhaben B2BA 170991*, Bayern, Lehrstuhl Keramik und Verbundwerkstoffe, Universität Bayreuth (1994).
6. Gesche, A., Nosowicz, J., and Zeus, D., "Dry Gas Seals for Low and High Pressure," *Advancements in Bearing and Seal Technologies*, Calgary, Canada (1994).
7. Nosowicz, J., Schicktan, R., Schrüfer, A., and Zeus, D., "Beschichtung von SiC mit Diamantähnlichem Kohlenstoff für Tribologische Anwendungen," *Abschlußbericht Forschungsvorhaben B21010B*, Bayern, F. Burgmann Dichtungswerke GmbH & Co (1994).
8. Mersch, A., "Gleitlager aus Siliciumcarbid mit Optimierter Trockenlauffähigkeit für Kunststoffausgekleidete Magnetkupplungspumpen," *Pumpen und Kompressoren*, 2, pp. 88-92 (1996).