

Article

# The Identification of an Adequate Stressing Level to Find the Proper Running-In Conditions of a Lubricated DLC-Metal-System

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Received: 9 July 2020; Accepted: 27 August 2020; Published: 31 August 2020



Abstract: Using a tribometer equipped with a high-resolution wear measurement unit (RNT), the running-in of a diamondlike carbon (DLC) iron spray coating contact was analyzed and optimized. The optimization comprised an initial parameter field with different load and speed levels to find key operation points. These points were used to compose a dedicated running-in parameter field. The analysis underlined the importance of identifying the adequate stressing conditions. With respect to our concept of the running-in corridor, a high-power running-in has to be preferred to obtain a tribological system with low friction, small total wear and wear rate, high system stability, and low sensitivity to external changes.

Keywords: running-in; lubricated sliding; ultra-low wear; parameter field selection

## 1. Introduction

There is no straightforward path to identify running-in procedures in order to ensure excellent tribological performance during the lifetime of mechanical systems such as combustion engines, gear boxes, compressors, or pumps [1–4]. In tribological analysis, troubleshooting, or optimization, the running-in process is frequently either neglected because of its short duration or simply omitted because of its complexity. However, excellent tribological properties are pivotal for energy and CO<sub>2</sub> savings and a long lifetime. It is therefore necessary to analyze and understand the very first minutes or hours of operation to come up with a sound recipe for a controlled running-in. To reach this goal, high-resolution and real-time friction and wear tests have been performed using the lubricated pairing of iron-plated aluminum versus diamondlike carbon.

The technical term "running-in" describes the first stage in the lifetime of a tribological system, and is connected with the expectation that friction and wear rate rapidly develop low and constant values as required for modern lubricated systems. Consequently, a system that shows constant and nondecreasing friction from the beginning, accompanied by linearly increasing wear, does not perform a running-in. In addition, a running-in can be successful or unsuccessful depending on the expectations of the experimentalist. When, for example, gearbox applications are in focus, then a steady coefficient of friction of 0.08 and a wear rate of more than 100 nm/h do not characterize the running-in as successful. The successful running-in has to result in ultra-low wear rates and lowest friction coefficients in order to allow a long lifetime with excellent performance. Moreover, the system has to exhibit low sensitivity and high stability. Whereas friction and wear are common tribological entities, stability and sensitivity must be defined. (1) Stability: When the tribological system after a certain runtime responds with a constant wear rate and constant coefficient of friction, the system can be called stable. Tribological systems can develop different stable states depending on the level of stressing during running-in.



The lower friction and wear rate are, the higher the stability. (2) Sensitivity describes the response of friction and wear to changing boundary conditions, e.g., variations of load and speed but also temperature and viscosity. The system is highly sensitive when the response is intense and vice versa. Further details on stability and sensitivity can be found in [5]. It is therefore desirable to find an adequate sequence of stressing levels—usually, normal forces and sliding velocities—that convey the tribological system to lowest friction and wear rate, high stability, and low sensitivity. However, no straightforward path to find the right running-in conditions exists so far [6]. On the contrary, the daily work of tribologists is mainly characterized by trial and error or the intentional neglect of the running-in [7]. Many tribological systems are analyzed under the assumption that the running-in is over. However, whether the running-in is really over or not remains uncertain. Moreover, when a selected sequence of running-in stressing levels is changed—for instance, simply reversed—then different wear rates and friction coefficients are the result. It is therefore highly important for the evaluation of tribological systems or for system optimizations that the role of running-in is properly understood. Especially, when the impact of nuances-for instance, oil additive variations or roughness modifications-have to be tested, it must be certain that the differences in friction were caused by those variations and not by the effects of a running-in that has not been finished yet.

This contribution presents an analysis of a lubricated DLC-metal-system. Initial experiments with a pin-on-disk tribometer served to identify the critical stressing levels, this means critical normal forces and/or sliding velocities that trigger significant responses of the friction and wear signal. These stressing levels are called key levels and were used to construct a dedicated running-in procedure. The paper will show and discuss differently composed running-in parameter fields and their impact on tribological performance.

#### 2. Experimental Setup

#### 2.1. Radionuclide Tribometry

The experiments were carried out with a pin-on-disk (POD) tribometer, as described in [8]. The measurements were performed using a flat pin made of iron-plated aluminum and a steel disk coated with diamondlike carbon (DLC). The pin assembly comprises a shaft holding a tiltable hemisphere with a flattened spherical sector to realize a self-adjusting leveled contact with the disk. The actual sample, a circular tablet (diameter = 4.9 mm) with iron plating, was attached to the flat side of the hemisphere. With the tribometer, normal forces up to 1.000 N can be applied, corresponding to contact pressures of up to 52 MPa. The sliding velocities can be varied between 0.1 m/s and 5 m/s. The tribometer is equipped with an oil circuit containing a heater. The oil temperature of the fully formulated oil was set to 80 °C. To determine the wear behavior of the system, the pin was marked radioactively to allow the use of a radionuclide wear measuring unit (RNT). RNT is based on counting gamma pulses emitted by wear debris leaving the tribological interface. After proper calibration, wear volumes as low as a few micrograms per liter of oil can be resolved. From the wear volume, a wear rate in nm/h range can be derived. This approach presumes that the density of the worn material is known and that wear proceeds homogeneously across the worn area—in our example, the cross section of the pin. Although this is an approximation, many discussions in the past showed that this value provides a valuable entity to practitioners, e.g., for the design of a journal bearing. Further details of RNT can be found in [9]. The shown RNT measurements were received using a device of Zyklotron AG, Karlsruhe, Germany.

#### 2.2. Samples and Lubricant

Steel disks (100Cr6, 1.3505) were polished using a diamond tape finish to a roughness  $R_a$  of 0.2 µm. A DLC coating was applied using Laser-Arc (Fraunhofer IWS, Germany), and afterwards smoothed by a brushing process to a roughness  $R_a$  of 0.06 µm. Iron-plated pins were received from a cylinder liner with a thermal spray coating. The thermal spray coating had a thickness of approximately 600 µm.

The pairing was lubricated with a fully formulated engine oil, Fuchs Titan 0W20. The 2.71 oil within the circuit were applied directly to the disk.

#### 2.3. Test Procedure

The initial test procedure followed the approach suggested by Richard Stribeck, which means  $\mu = f(Hearsey \ parameter) = f(\eta v/p)$ . The tribological system was subjected to a sequence of different contact pressures p and sliding velocities v. Viscosity  $\eta$  was kept constant throughout the experiment. In total, four initial contact pressures aiming at mixed lubrication of 26, 38, 48, 52 MPa, based on normal forces ranging between 500 N and 1000 N, were passed. During each load level, five different speeds were applied and kept constant for four hours. Timing is crucial for the evaluation of friction and wear response. In order to pass the Stribeck curve with decreasing Hersey parameter, the contact pressures were increased as shown below, while the sliding velocities were decreased during each load level. For each load level, the initial sliding velocity was adjusted to keep the v/p ratio constant to prevent the system from seizure. Table 1 shows a summary of all experimental data.

Entity	Unit	Value
lubricant		Fuchs engine oil SAE 0W20
samples		pin: iron-coated aluminum/disk: DLC (ta-C)
oil temperature	°C	80
velocities	m/s	0.5–4.2
applied load	Ν	500/700/900/980
track radius	mm	32/39/46
pin diameter	mm	4.9
nominal pressure	MPa	26/38/48/52
level duration	h	4

Table 1. Summary of experimental conditions.

#### 3. Results

#### 3.1. Initial Parameter Field

The design and outcome of the initial parameter field experiment is shown in Figure 1. The coefficient of friction started at about 0.06, which is lower when compared to lubricated metal–metal contacts. At each increase of load every twenty hours, the v/p ratio was kept constant to prevent a shift towards boundary lubrication. At each onset of the five speed levels, friction increased. These are the common Stribeck effects. In contrast, within each level of constant sliding, velocity friction decreased. This decrease can be attributed to materials effects, known as third-body formation [10].

With respect to total wear, a steadily increasing signal was received. The slope of the wear curve was interpreted as wear rate (w = dw/dt). The highest wear rate was obtained at the initial stressing level. Throughout the first load level, friction vividly responded at each velocity level. This was the topographic running-in caused by flattening asperities. The topographic running-in was followed by the tribo-chemical running-in to form the third body. Besides the first load level, high wear rates appeared during the last load level.



**Figure 1.** Initial parameter field displaying coefficients of friction (green), total wear (black), and linearized wear rate (yellow). Straight red lines inside the wear curve were introduced to show sections with constant wear rate. The slope of these lines corresponds to the numbers indicated by the yellow curve. In addition, the lower diagram shows the stressing conditions. The contact pressures are shown in red, the sliding velocities in blue color.

#### 3.2. Derivation of Optimized Parameter Field—Approach I

The strategy to obtain the optimized parameter field was based on the evaluation of the tribological entities: friction, wear, stability, and sensitivity. With respect to the definition provided above, the system shown in Figure 1 is not stable, since wear rate changes at and within each load level. In addition, the system is highly sensitive, since wear and particularly friction change at each alteration of stressing conditions. With the given definitions in mind, the key levels were recruited within the area of the final load level to concentrate on conditions that support third-body generation. Here, we found the highest wear rate, since the initial wear rate during topographical running-in was not considered. In addition, friction showed large fluctuations (high sensitivity) and the strongest decreases of the entire parameter field. Stability was low, as indicated by the lack of degression of the wear rate.

Figure 2 shows the new parameter field derived from identified key levels. Instead of the initial 20 levels shown in Figure 1, just ten levels were used here and the total duration was reduced to 50 h. The contact pressures ranged between 25 MPa and 50 MPa as before. It is obvious that this parameter field did not lead to the desired effects. On the contrary, friction, total wear, and wear rate showed higher values than before. Total wear of 760 nm was accumulated already after 50 h. During the first experiment, it took 80 h.



Figure 2. Parameter field based on key levels.

#### 3.3. Derivation of Optimized Parameter Field—Approach II

Based on the findings above, the identified key levels were kept, but their sequence was reversed to start the parameter field at high stressing level, see Figure 3. This kind of approach is based on the concept of third body generation. It is well-known that third-body generation requires an activation energy in order to initiate plastic flow and mechanical intermixing. Both processes are necessary to obtain the appropriate shear and strengthening horizon, as shown in [11], to realize low friction and wear rate. It is therefore recommended to start a running-in parameter field with high stressing levels. As before, the initial stressing step—initiating the topographic running-in—yielded the highest wear rate of about 140 nm/h when compared to the first (20 nm/h) and second parameter field (70 nm/h). After the fourth load level, wear rate dropped to values of around 10 nm/h and later to less than 5 nm/h. Changes in sliding velocity did not affect the course of the wear curve. Even the drastic increase in contact pressure at 15 h (returning to the initial stress level) did not destabilize the system. The increase in wear rate was very low, indicating decreased sensitivity and high system stability. Compared to the other parameter fields, friction showed significantly less fluctuations. After 15 h, load and speed were kept constant to monitor the evolution of the system. Throughout this, stressing level friction kept decreasing up to a runtime of about 50 h.



Figure 3. Reversed parameter field derived from key levels.

#### 4. Discussion

As mentioned above, the running-in can generally be subdivided into a topographic and a tribo-chemical phase, Figure 4. Whereas the topographic phase is usually a matter of minutes, the tribo-chemical phase can last hours, days, or even longer, depending on the initial friction power density the system is subjected to. During the topographic phase, wear rates comparable to systems showing abrasive or adhesive wear—i.e., in  $\mu$ m/h range—can occur. When those high wear rates prevail, the system will fail due to seizure.

During the tribo-chemical phase, the system changes with respect to topography, near-surface chemical composition, and microstructure [12]. This process is called third-body formation and concerns mostly the metal part of the tribological system analyzed here. DLC undergoes similar changes, however, due to their localization very close to the surface, these changes are extremely hard to detect. The topographical, structural, and chemical changes of the near-surface materials will be described in another contribution.

The tribo-chemical phase is mainly controlled by the deposited amount of friction power during the very first moments of runtime. As our analysis of the running-in corridor demonstrated, a certain activation energy is necessary to trigger third-body formation [8]. This crucial energy input takes place during high-power initial stressing, however, it is retarded or blocked when the initial stressing is too low. As a curtailment of this statement, it has to be pointed out that high-power running-ins can be

applied to tribological systems that have received an adjusted conditioning, e.g., by a surface finish with adapted coolant liquid. This is usually an evolutionary process. When surface treatment is not possible or not optimized, then the running-in serves as a conditioning process and should be handled carefully. In many cases, a stepwise arrangement of the initial stressing levels is necessary [13].



**Figure 4.** Summary of findings. The response of the system for the case of low-power stressing (red) and high-power stressing (blue) is shown. The running-in can be divided into a topographical and a tribo-chemical phase.

The identification of key levels is successfully accomplished; for friction, wear, stability, and sensitivity, real-time data are available for evaluation. However, in most cases, continuously recorded wear data are not at hand. The evaluation of friction data alone is not constructive, since friction and wear seldom run in a proportional way. Both entities usually possess different time constants, as demonstrated in Figure 3. At about 42 h, the wear rate stays constant, while friction is still decreasing. In addition, there are tribological states where friction is low but wear is exceedingly high, as shown in [14] for the case of a lubricated metal–metal system with presence of soot in the oil. As a consequence, tribological optimization by running-in control has to be accompanied by in-depth analysis of wear.

## 5. Conclusions

The following approach to receive a running-in with the conditions described above can be deduced:

- With an unknown or altered tribological system, an initial stressing field composed of different load and sliding velocity levels has to be passed. The parameter field should comprise stressing conditions as in real applications. The parameter field should be passed with increasing loads. The *v*/*p* ratio has to be controlled to avoid overstressing of the tribological system.
- From stressing levels yielding significant changes in friction and wear rate as well as in system stability and sensitivity, key stressing levels can be identified.
- Using the key levels, a new parameter field can be derived and should be arranged in a way that the high-power key levels start the sequence of stressing states.
- In case this parameter field does not result in ultra-low wear rates and friction coefficients, an additional parameter field should be run to redefine the key levels.
- If additional changes in wear rates and friction coefficient occur after returning to the initial stress level, a redefinition of the key levels is required.

**Author Contributions:** Conceptualization, M.S.; formal analysis, J.F.; funding acquisition, M.S.; investigation, J.F.; methodology, M.S.; project administration, J.F.; writing—original draft, J.F. and M.S. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by Bundesministerium für Wirtschaft und Energie grant number 03ET1609E.

Conflicts of Interest: The authors declare no conflict of interest.

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