The method of tribotesting of PVD coated elements in oscillation motion at high temperature

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Abstract. The development of new types of coatings is realised by many scientific and technological centres in numerous countries. To evaluate new material solutions, researchers use various methods with wide range of tribotesters. Consequently, the obtained results are incomparable. The barrier of new technologies development, especially surface modifications dedicated to work at high temperatures, is a lack of worldwide recognised tribological test methods. This paper presents the method for tribological evaluation of tribosystems with PVD/CVD coated elements, in oscillatory motion and at high temperature (up to 900 °C). The method is realised by using SRV Optimol Instruments Prüftechnik GmbH (Germany) device. The realised verification tests proved the correctness of the elaborated methodology.

1. Introduction

A vast amount of manufacturing processes operate at extreme temperatures and rely on the safety and reliability of components to be capable to cope with harsh environment. Tribological systems working under high operating temperatures (up to 1000 °C), have recently received a great deal of attention, because of the processes (such as oxidation, softening and creep), which although, at room temperature, can be treated as negligible, at elevated temperatures can play a dominant role, especially as their relation with wear is highly complex [1, 2].

Reliable performance of tools and devices working at elevated temperatures requires the use of proper materials with enhanced thermal and wear resistance, as well as the possibility to ensure low energy dissipation by low friction behavior in a wide range of temperature [3, 4].

Currently, one of the leading technologies for depositing the multifunctional coatings is physical vapour deposition (PVD) [5, 6]. Thin hard coatings are used in numerous applications to extend the life of the machine parts [7, 8]. The concept of using PVD coatings is based on previous works, in which it was proved that the presence of Al in the classical TiN and CrN coatings results in the increase in wear resistance and thermal stability at the temperatures up to 1000 °C [9, 10].

The important mechanism worth to be noted here is the formation of highly protective double oxide layer, including a very dense (Cr,Al)₂O₃ on top, providing high wear resistance and friction stability under severe stresses occurring at such temperatures [11].
Nowadays, there is a wide range of either single layered (e.g. TiN, CrN), or multilayered coatings with combined properties of several materials, beginning with TiC/Al₂O₃/TiN in 1982, and also multielement coatings such as TiAlN, BCN, AlCrN and TiAlYN-systems [12]. The modification of coating compositions leads to the extension of their area of application.

Despite the extraordinary wear resistance of the Ti₁₋ₓAlₓN coatings their frictional performance at high temperature is not sufficient, so one of possible ways to provide low friction behavior is the introduction of friction decreasing components to receive structures like: TiAlCrN/TiAlYN or TiAlN+WC/C (a-C:H:W). However, such modifications cause a drop of the temperature resistance by a few hundred degrees Celsius.

The promising results were obtained for TiAlN-based, AlCrTiN-based, TiBN, TiSiN, CrVN and nanocomposite CrAlSiN. In particular, the coatings containing vanadium have been identified as providing low friction at around 600 °C via the in situ formation of V₂O₅.

To evaluate the new material solutions, researchers use various methods and wide range of tribotesters [13 14]. Consequently, the obtained results are difficult to compare [15, 16]. One of the barriers to develop new technologies, especially surface modifications dedicated to work at high temperatures, is a lack of worldwide recognized and standardised tribological test methods [17]. In this paper the authors presents two methods for tribological evaluation of tribosystems with PVD/CVD coated elements in conditions of oscillatory motion and at high temperatures. The first concerns the wear assessment, and the second is focused on friction coefficient measurement at wide range of temperature from room up to 900 °C.

2. Test rig, materials and methods

2.1. Test rig

The wear tests were performed be means of ball-on-disc SRV friction and wear tester (Schwingungs Reibung und Verschleiss [SRV], Optimol Instruments Prüftechnik, Germany) using reciprocating motion of the ball sliding against coated disc (Figure 1). During the run the temperature is measured by the thermocouple situated in the base plate of the test disk.

![Figure 1. The scheme of the SRV machine test chamber: 1 – load rod (F – direction of normal force), 2 – test ball, 3 – test disc, 4 – electrical resistance heater, 5 – test disc holder, 6 – support, 7 – piezoelectric measuring device (S – direction of oscillating motion), 8 – test ball holder.](image-url)
2.2. Specimens
The specimens (discs) were made of K10F sintered carbide (WC/Co) supplied by Sinter Sud. The drawing and photograph are presented in Figure 2. The properties of sintered carbide are presented in Table 1.

![Figure 2](image)

**Figure 2.** The specimen (disc) made of sintered carbide: a) the drawing, b) the photograph.

| Table 1. The properties of K10F sintered carbide (according to the supplier specifications). |
|-----------------------------------------------|
| Parameter                  | Value          |
| Type                        | K10F           |
| Composition, wt%            | 91.5 WC, 8.5 Co|
| Density, g/cm³              | 14.7           |
| Hardness, HRA               | 92.0           |
| Vickers hardness            | 1800 HV30      |
| Grain size, µm              | 0.8            |

2.3. Substrate surface preparation
The samples preparation was divided into the following steps. The first step was to cut the samples from the rod, by means of electrical discharge machine using Cu wire (WEDM), with distilled water as a dielectric.

The next step was to obtain the required thickness of test samples using a grinder equipped with a grinding wheel (1A1 200x200x5x51 D151 K75 MBm) intended for sinters and carbides. The grinding wheel contains diamond particles with a grain size between 126 and 150 µm, at K75 concentration in a binder from a soft resin. During the process a cooling-cutting fluid was used. The grinding was done at the linear speed of 35 m/s.

After obtaining the required thickness and initial roughness, the samples were polished by means of ATM Saphir 550 automatic polisher using the procedure presented in the Table 2.

| Table 2. The parameters used for specimens polishing. |
|-----------------------------------------------|
| Parameter                  | Stage 1 | Stage 2 |
| Polishing plate            | Cameo Gold | Cameo Gold |
| Lubricant                  | BioDiamant 6 Mpe (6 µm diamond grain size) | BioDiamant 3 Mpe (3 µm diamond grain size) |
| Plate speed [rpm]          | 200     | 200     |
| Load per sample [N]        | 15      | 15      |
| Rotation direction         | clockwise | clockwise |
| Time [s]                   | 240     | 240     |
The results of surface roughness ($R_a$ and $R_z$ parameters), obtained after each of performed preparation steps, are presented in the Table 3.

| Surface                     | $R_a$ [µm] | $R_z$ [µm] |
|-----------------------------|------------|------------|
| After grinding              | 0.0318     | 0.2233     |
| After polishing (Stage 1)   | 0.0173     | 0.1412     |
| After second polishing (Stage 2) | 0.0028      | 0.0220     |

2.4. Coatings

The two types of commercially available coatings deposited by PVD (Physical Vapour Deposition) process were tested. The operational temperature upper limit of the AlTiN is 1000 °C and TiAlN coating is 900 °C. The detailed deposition process parameters are not provided by the coating supplier. These coatings maintain hardness even at high temperatures and are resistant to wear and thermal shock. The chemical composition and mechanical properties of the coatings are summarised in Tables 4 and 5.

| Coating type | Concentration [wt%] | Concentration [at.%] |
|--------------|---------------------|----------------------|
|              | Al                  | Ti                   | N        | Al                  | Ti                   | N        |
| AlTiN (A)    | 37.1±0.3            | 40.1±0.2             | 22.3±0.4 | 36.1±0.4            | 22.3±0.1             | 41.7±0.6 |
| TiAlN (B)    | 24.1±0.1            | 55.3±0.1             | 20.4±0.4 | 25.4±0.1            | 32.8±0.1             | 41.7±0.1 |

| Coating type | Hardness HV0.05 | Thickness [µm] | Friction coefficient against dry steel | Surface roughness after deposition $R_a$ [µm] |
|--------------|-----------------|----------------|-------------------------------------|---------------------------------------------|
| AlTiN (A)    | 3000            | 1.9 – 2.0      | 0.35                                | 0.0643                                      |
| TiAlN (B)    | 3300            | 1.4 – 1.5      | 0.30 – 0.35                         | 0.0228                                      |

These coatings are used in hot forging and extrusion industries to protect dies and moulds operating at high temperatures. They are also applied to internal combustion engine components (e.g. nozzle needles and control valves of fuel injection systems). The coatings were deposited on the samples made of K10F sintered carbide.

2.5. Wear test procedure

The test parameters for wear assessment are summarized in Table 6. The parameters were selected in such a way, as to obtain measurable wear during the test but also to prevent the coating from being rubbed through (based on the experience gathered during previously conducted research).

On a single disc, 5 test runs are carried out in the places indicated in Figure 3. The test runs are performed in the order of increasing temperature. The volumetric wear was determined by means of Talysurf CCI – Lite Non-contact 3D Profiler.
Table 6. Test parameters for wear measurements.

| Parameter             | Value                                      |
|-----------------------|--------------------------------------------|
| Temperature, °C       | 25 (RT), 400, 600, 750, 900               |
| Temperature gradient, °C/s | 1                                      |
| Time of temperature stabilisation, s | 1200                                   |
| Load, N               | 5                                         |
| Stroke, μm            | 1000                                      |
| Frequency, Hz         | 10                                        |
| Run time, s           | 300                                       |
| Ball material         | Si₃N₄ (grade G20)                         |
| Ball diameter, mm     | 10                                        |
| No. of repetitions    | min. 3                                    |

Figure 3. The location of wear scars on the disc.

In high-temperature tests, the method of heating the samples is important. Its full cycle consists of: heating from room temperature to the set point (e.g. 600 °C) at a constant rate of 1 °C/s, stabilization of the set temperature, and the 300 seconds test run. An example of the wear test step is shown in Figure 4.

Figure 4. The example of the test procedure for wear measurement (at 600 °C) 1 - the beginning of the test, 1-2 – heating from RT up to 600 °C with 1 °C/sec. increase rate, 2-4 – stabilization at 600 °C, 3-4 – 300 seconds run, 4 – the end of the test.

After the test run, the wear scar is measured using a 3D profilograph. The run is treated as valid if the coating is not rubbed through.
2.6. The friction coefficient measurement procedure

The test parameters for the friction tests are summarized in Table 7.

| Parameter                          | Value                                      |
|------------------------------------|--------------------------------------------|
| Temperature, °C                    | 25 (RT), 100, 200, 300, 400, 500, 600, 700, 800, 900 |
| Temperature gradient, °C/s         | 1                                          |
| Time of temperature stabilisation, s | 300                                        |
| Load, N                            | 20                                         |
| Stroke, µm                         | 1000                                       |
| Frequency, Hz                      | 10                                         |
| Run time, s                        | 10                                         |
| Ball material                      | Si₃N₃                                      |
| Ball diameter, mm                  | 10                                         |
| No. of repetitions                 | min. 3                                     |

The procedure for measuring the friction coefficient is shown in Figure 5a. The detailed course of the test for the 700 °C cycle is shown in Figure 5b. The friction coefficient is calculated as the arithmetic mean of the maximum quantities in the cycle.

**Figure 5.** The friction coefficient measurement procedure: a) temperature curve with vertical lines representing each of measurement cycles, b) the detailed section of test between 600 and 800 °C: 1-2 – heating from 600 °C up to 700 °C with 1 °C/s rate; 2-4 – stabilization at 700°C; 3-4 – 10 s measurement cycle.
Aforementioned approach makes it possible to conduct one test run on a single disc, covering the whole selected temperature range: from RT up to 900 °C. Due to the fact that the friction process always takes place in the same area of the specimen, the test run time was reduced to 10 seconds. Between the test runs, heating and temperature stabilization of the sample is applied. After reaching the set temperature, another test run is carried out (in the same place, but at a higher temperature).

**Results and discussion**

In order to verify the developed research methodology, tests of wear resistance and friction coefficient were carried out for two selected coatings dedicated to high-temperature applications.

2.7. **Wear test results**

The experiments on coatings have shown a considerable dependency between tribological characteristics and temperature. In most cases the increase in temperature increases the wear. However this rule is not valid for coating intended for high temperature applications. For coating denoted as “B” the minimum wear was observed at 600 °C, while the wear of the coating denoted as “A” remained high.

![Figure 6. The results of discs volumetric wear at selected temperatures.](image)

The developed method made it possible to precisely determine the differences between the wear characteristics of the A and B coatings. Despite the fact that both coatings are dedicated to high-temperature applications, their wear characteristics differ depending on the temperature. For coating A the wear at 600 and 750 °C was similar to the wear at room temperature (25 °C). The minimum wear was recorded for 400 and 900 °C. However, the result obtained for the temperature of 900 °C should be rejected, because the coating was completely removed during the test – the maximum depth of wear track, measured with the profilograph, exceeded 3 µm, which means that the ball rubbed through the coating and went into the substrate material.

At room temperature, the wear of the coating B was only slightly lower than that of the coating A. Research using the developed method allowed to find differences between these coatings at 600 °C and 750 °C. Contrary to the combination with coating A, for the combination with coating B, at 600 °C and 750 °C, the volumetric wear was several times lower than the one measured at room temperature.

The appearance of wear scars on the coated discs is shown in Figure 7. The maximum depth of wear scars is summarized in Table 8.
Figure 7. 3D images of wear scars on the coated discs.

Table 8. The maximum depth of wear scars measured at various temperatures.

| Temperature [°C] | Coating A [µm] | Coating B [µm] |
|------------------|----------------|----------------|
| RT               | 1.6            | 1.1            |
| 400              | 2.2            | 1.6            |
| 600              | 2.2            | 1.5            |
| 750              | 2.2            | 1.1            |
| 900              | 3.1            | 1.5            |

The test conditions are set between two boarders: obtaining the wear high enough to be clearly measurable (while maintaining a satisfactory repeatability), and avoiding the total removal of the investigated coating. For the adopted parameters, the maximum depth of the wear scar for coating A slightly exceeds the thickness, but microscopic observations showed that the abrasion occurs only on a small part of the friction mark, which is considered as acceptable. However, for the B coating, no total removal of the coating was found in the entire test temperature range. Increasing the severity of the test conditions (time, frequency, load) would lead to the coating removal in each test run, which would be disqualifying for the test method. On the other hand, reducing the value of the test parameters would result in obtaining unmeasurable wear.

Summarizing, it can be stated that the results of the verification tests proved the correctness of the adopted test method for the assessment of the wear resistance of elements covered with coatings intended for operation at high temperatures.
2.8. Friction coefficient test results
The results of friction coefficient at selected temperatures are presented in Figure 8.

![Figure 8. The results of friction coefficient at different temperatures.](image)

At room temperature, the friction coefficients of the coatings A and B do not differ significantly. Only for the test temperature of 400 °C, the friction coefficient of the coating A is higher than for the coating B. The obtained results showed that using the developed methodology, it is possible to precisely measure the coefficient of friction in the entire temperature range from RT to 900 °C. The method has several significant positive attributes. The method is well repeatable as the obtained results are characterized by a scatter lower than 10%. It is also relatively low time and cost consuming. 3 sets of samples are sufficient for the entire measuring cycle, which can be conducted with a time schedule of approximately three hours.

3. Conclusion
The results of the verification tests proved the correctness of the adopted test method to assess the wear resistance and friction coefficient of the elements covered with coatings intended for operation at high temperatures.

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