ISSN 0208-7774 TRIBOLOGIA 4/2022 p. 51–60
DOI: 10.5604/01.3001.0016.1609

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# THE PROCEDURE OF SELECTING THE VALUES OF THE WORK CONDITION PARAMETERS OF THE BALL-CRATERING METHOD FOR THE ASSESSMENT OF RESISTANCE TO ABRASIVE WEAR

# PROCEDURA DOBORU WARTOŚCI PARAMETRÓW PRACY METODY BALL-CRATERING DO OCENY ODPORNOŚCI NA ZUŻYCIE ŚCIERNE

**Key words:** abrasive wear, ball-cratering method, research procedure, PVD coating.

**Abstract:** In the example of the CrN coating deposited by the cathodic arc evaporation (CAE) method on two differently prepared substrates, the procedure for selecting the values of the work condition parameters of the ball-cratering method was presented. The procedure was developed thanks to applying an individual experiment design from Taguchi's approach and has not been used to determine the values of the parameters of the operating conditions of the test friction node for tribological tests. The procedure, according to Taguchi, allowed us to determine the values of the parameters for assessing the abrasion resistance of anti-wear coatings, with the use of a much smaller number of tests, compared to the methods used so far and a repetitive wear mechanism.

The starting point of the presented procedure was selecting the area of the permissible values of the operating parameters, in the ball-cratering method, in which optimal values were searched for to minimise the relative error in determining the diameters of wear marks craters. After determining the area of limit values, an experiment plan was generated in which the variables were: load, rotational speed, minimum friction path, and maximum friction path. The results of abrasive wear tests performed with the ball-cratering method, in accordance with the developed test procedure for the CrN coating – were deposited on the substrate after heat treatment and the substrate after the nitriding process had been successfully verified.

**Słowa kluczowe:** zużycie ścierne, metoda ball-cratering, procedura badawcza, powłoka PVD.

**Streszczenie:** Na przykładzie powłoki CrN, osadzonej metodą katodowego odparowania łukiem (CAE) na dwóch różnie przygotowanych podłożach, przedstawiono procedurę doboru wartości parametrów pracy metody ball-cratering. Procedurę opracowano dzięki zastosowaniu indywidualnego planu eksperymentu wynikającego z podejścia Taguchiego dotychczas niestosowanego do wyznaczania wartości parametrów warunków pracy testowego węzła tarcia do badań tribologicznych. Sposób postępowania według Taguchiego pozwolił na wyznaczanie wartości parametrów oceny odporności na zużycie ścierne powłok przeciwzużyciowych, przy zastosowaniu zdecydowanie mniejszej liczby prób w stosunku do dotychczas stosowanych metod oraz powtarzalnym mechanizmie zużywania.

Punktem wyjścia zaprezentowanej procedury był wybór obszaru dopuszczalnych wartości parametrów pracy, w metodzie ball-cratering, w której poszukiwano wartości optymalnych ze względu na minimalizację błędu względnego wyznaczania średnic kraterów śladów zużycia. Po ustaleniu obszaru wartości wielkości dopuszczalnych wygenerowano plan eksperymentu, w którym zmiennymi były: obciążenie, prędkość obrotowa, minimalna droga tarcia i maksymalna droga tarcia. Wyniki badań zużycia ściernego wykonanych metodą ball-cratering, zgodnie z opracowaną procedurą badawczą dla powłoki CrN – osadzonej na podłożu po obróbce cieplnej oraz na podłożu po procesie azotowania, zostały pomyślnie zweryfikowane.

## INTRODUCTION

The increase in the importance of modifying machine parts and tools by using thin, hard coatings makes it necessary to constantly develop techniques for studying their anti-wear properties. The dynamic development of technologies for producing PVD/CVD coatings is not accompanied by an adequately fast development of techniques for studying their

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properties. It particularly applies to tribological characteristics, which, as far as machine parts and tools are concerned, determine to the greatest degree their operating values **[L. 1–5]**. The intensive development in technologies for depositing coatings (multi-layered, gradient, hybrid, etc.) requires the development of a tribological research method, which makes it possible to obtain a fast and precise evaluation of resistance to abrasive wear. The ball-cratering test method of anti-wear coatings, in which loose, hard abrasive transfer into the friction zone is used in order to strengthen the effect of frictional destruction, meets this need. The micro-abrasion test becomes important as a method for the abrasion testing of the surface engineered materials and physical and chemical vapour deposition (PVD/CVD) coatings. Based on the ball-cratering method, a micro-scale abrasive wear test has been used to evaluate the wear resistance of duplex and non-duplex coatings **[L. 6–9]**. The major advantage of this micro-abrasion test is that: it requires a small test area and allows the simultaneous evaluation of the intrinsic wear intensity for both substrate and coating in a coated sample. The diameter of the crater in the substrate and the coating, both parallel and perpendicular to the direction of ball rotation, has been measured. Where the parallel and perpendicular values vary by less than 10%, then the average of these measurements should be used as the size of the crater. Only craters that conform to this condition shall be used to calculate the wear rates. The purposes of the present study were to verify the feasibility and reliability of the application of the design of experiments method – (DOE method) to optimise the methodology of abrasive wear research of a CrN coating. Since there are little data available about the performance of PVD coatings in small-scale abrasion tests, the present work showed the micro-abrasive wear resistance of CrN coating on hardened AISI H13 substrates and composite layer: nitrided layer/CrN coating on AISI H13 too.

The Taguchi optimisation methods were used in order to devise and verify the methodology of the research. The Taguchi methods are applied in many fields of science; among others, they are used to optimise the parameters of coating deposits **[L. 10–13]** and to determine the lifespan of tools operated in specific conditions **[L. 14]**. The design of the experiment (DOE) method is a technique to optimise a complex process. The Taguchi method has been thoroughly described by Sahoo **[L. 15]** and other works **[L. 12–16]**.

## MATERIALS AND METHODS

### Ball-cratering method

The research method applied to determine the abrasion resistance of the coatings by a micro-abrasion wear test is based on the ball-cratering method (using T-20 tester, designed and manufactured in the Łukasiewicz – ITEE, Radom, Poland), **Figure 1**.

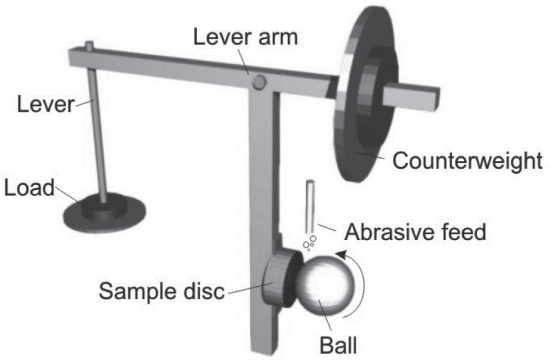

**Fig. 1. Schematic layout illustrating the geometry of the micro-abrasion apparatus [L. 20]**

Rys. 1. Schemat zestawu badawczego do badania zużycia ściernego powłok **[L. 20]**

The test ball used in these experiments was an AISI 52100 (Dejay Distribution Limited, UK) bearing ball with a diameter of 25.4 mm, hardness of 58.6 HRC, and surface roughness Ra = 0.177 µm. The abrasive slurry used in the study was composed of a suspension of SiC particles (Grade F1200-C6, Washington and Mills Abrasives, Manchester, UK) in distilled water (abrasive concentration of 20% v/v). The mean size of the SiC particles was 4.0 µm (**Figure 2**). Before starting the test, the samples were cleaned in the ultrasonic cleaning machine for 10 minutes.

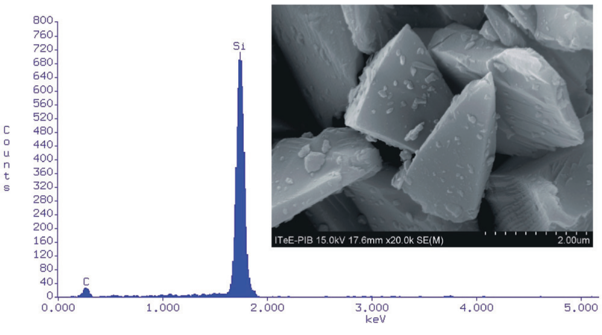

**Fig. 2. SEM image of SiC abrasive particles together with EDS spectrum [L. 20]**

Rys. 2. Obraz SEM i widmo promieniowania rentgenowskiego EDS ścierniwa SiC **[L. 20]**

## Coatings

The duplex CrN and non-duplex CrN coating were deposited using cathodic arc evaporation (CAE) on hardened and tempered AISI H13 (55 HRC) steel discs dia. 25.4 x 10 mm thick. Prior to the process surface of the samples was polished and chemically cleaned using an ultrasonic cleaner. The duplex CrN and non-duplex CrN coatings were produced in a CDS-Standard CAE system manufactured by the Ł-ITEE Radom, Poland. During the ion nitriding, special OES (optical emission spectroscopy) was used for setting the $N^+/H^+$ ratio. Ion nitriding was performed in an 80 % $H_2$ – 20 % $N_2$ glow discharge. The nitriding temperature was around 520°C. After the ion nitriding process surface of the samples was mirror polished. After evacuating to a pressure of $2x10^{-5}$ mbar, the CrN coatings were deposited onto the planetary rotating substrate holders at a typical nitrogen pressure of $3.5x10^{-2}$ mbar. Before initiating coating deposition, samples were heated up by the radiation of IR heaters and the bombardment with Cr ions, and the coating temperature did not exceed 400°C. The main process parameters during ion nitriding and coating deposition are shown in **Tab. 1**.

**Table 1. Parameters of the ion nitriding process and coating deposition process**
Tabela 1. Parametry procesów: azotowania i osadzania powłoki

| Process | Pressure | Atmosphere | Potential | Evaporator current | Ubias | Time | Thickness |
|---|---|---|---|---|---|---|---|
| | p[mbar] | [%] | p(N/N$^+$H) | I [A] | U[V] | t[min] | g[μm] |
| Ion nitriding | 4.3 | $N_2$ 20%<br>$H_2$ 80% | 8.0% | - | -600 | 420 | 70 |
| Coating deposition | $3.5x10^{-2}$ | $N_2$ 100% | - | 4x70 | -150 | 90 | 2.8 |

## Analysis of the coatings

Coating adhesion was evaluated using scratch adhesion tests. Scratch adhesion tests were carried out using CSM Instruments Revetest produced by CSM, Peseux, Switzerland equipment with a standard Rockwell C diamond indenter, 10 mm/min table speed, load increase rate of 100 N /min, and a scratch length of 10 and 20 mm. In order to evaluate the adhesion, an LC3 load was used at which the coating was completely removed from the substrate across the whole width of the scratch track. The surface roughness of non-duplex and duplex samples was also measured using the Taylor Hobson Form Talysurf PGI 830 profilometer, Leicester, UK. Nano hardness of the coatings was obtained with a CSM Instruments NHT NanoHardnessTester, Peseux, Switzerland, set to a max depth of 10% of coating thickness, using a Berkovich indenter. In order to determine the hardness of the nitrided layers, hardness measurements were made at a load of 0.3 N using a Future-Tech FV-7 Vickers hardness tester in Tokyo, Japan. Non-duplex and duplex CrN coating roughness, hardness, and adhesion are shown in **Table 2**.

**Table 2. Summary of roughness, hardness, and adhesion ($L_{C3}$) for non-duplex and duplex CrN coatings**
Table 2. Właściwości powłoki CrN: chropowatość, twardość, adhezja

| Sample | Properties of nitrided layer | Properties of coatings | | |
|---|---|---|---|---|
| | Hardness | Roughness | Hardness | Adhesion |
| | HV | Ra [μm] | HV | $Lc_3$ [N] |
| Non – duplex CrN | – | 0.37 | 1720 ±146 | 64 |
| Duplex CrN | 800 | 0.35 | 1860 ±250 | 156 |

The following test apparatus will be used to analyse wear and observe the surface of the border areas of the coating – substrate.

The optical microscope will be used to observe the wear traces and measure the obtained wear scars. The image acquisition and analysis system

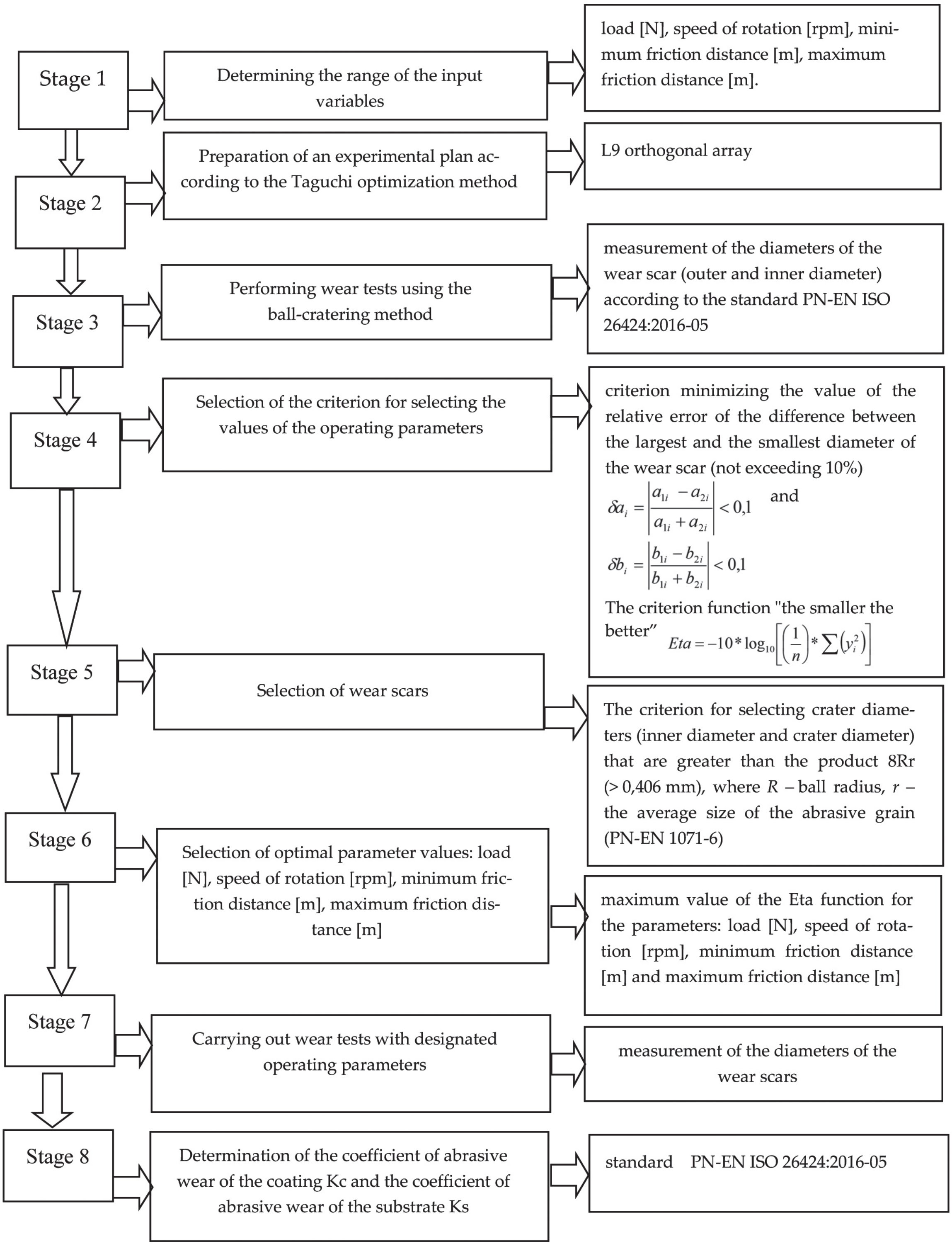

**Fig. 3. Scheme of proceedings in the developed method of testing the resistance to abrasive wear of anti-wear coatings [L. 20]**

Rys. 3. Schemat postępowania w opracowanej metodzie badania odporności na zużycie ścierne powłok przeciwzużyciowych **[L. 20]**

of the microscope used consists of a measuring microscope model MM-40 by Nikon and the MultiScan system v.8.08 by Computer Scanning Systems, Tokyo, Japan.

The field-emission scanning electron microscope SEM, the Hitachi SU-70 model Tokyo, Japan, equipped with Thermo Scientific's EDS energy dispersive X-ray spectroscopy Madison, Wisconsin, USA, will be used to observe the surface of wear scars and characterise the wear mechanisms, as well as to determine the elemental composition and mapping, i.e., elemental distribution on the tested surface.

The Talysurf CCI interferometric microscope by Taylor Hobson (White Light Interferometry WLI) Leicester, UK, is a measuring system for measuring the geometric structure of a surface (roughness, waviness and shape) by an optical method. It uses the phenomenon of white light interference to carry out an analysis of the geometric structure of the surface by contactless scanning of the surface.

### *The procedure for the selection of parameters of working conditions*

The procedure for the selection of parameters under working conditions in the ball-cratering method, developed and proposed in the work, has been presented in eight stages.

The first stage concerns the determination of the range of input variables: load, rotational speed, and above all, the friction path, as they significantly affect the obtained wear trace, which was found on the basis of own research carried out using the method of planning the experiment developed by Taguchi.

The second stage involves building an orthogonal table L9, consisting of nine test trials for four variables: load, rotational speed, minimum distance d1, and maximum distance d4. Each variable has three ranges of values; hence the experimental design is L9 ($3^4$). It significantly reduces the number of experiments performed, which should be performed at 81, and repeats each attempt three times over 250 **[L. 17, 18]**.

The third stage includes carrying out ball-cratering wear tests with the designated ranges of input variables.

Stage four and stage five are concerned with the selection of craters. After the wear tests are carried out, the obtained wear traces should be selected in accordance with the selected criterion for the selection of the values of the operating parameters "the smaller – the better", the criterion minimising the value of the relative error of the difference between the largest and the smallest wear traces, not exceeding 10%.

The fifth stage concerns the selection of the inner diameter (a1i, a2i) of the crater, which is greater than the product of 8Rr, where R – sphere radius, r – average abrasive grain size according to the standard **[L. 19]**.

The sixth stage includes the construction of the Eta function, the maxima of which are determined by the optimal values of the operating parameters of the variables: load, rotational speed, and the minimum distance and maximum distance.

The seventh stage concerns the performance of wear tests with the parameter values indicated from the Eta curves. In accordance with the requirements of the Taguchi method, the determined values from the Eta, the function should be subject to empirical verification confirming the obtained results or indicating the necessity to repeat the optimisation process with other parameter values.

The last, eighth step of the procedure is determining the abrasion resistance of the tested coating, defined by the coating wear factor Kc.

The diagram of the developed procedure of conduct in the proposed method of selecting operating conditions parameters is shown in **Fig. 3**.

## RESULTS AND DISCUSSION

Experiments were carried out using the combination of tribological test parameters based on L9 Taguchi orthogonal design with three test parameters: load, speed of rotation of the ball, and sliding distance.

The research plan consisted of nine experiments with such variables as load, rotation speed, and minimum and maximum distance. Each of the research runs was repeated three times. One research plan was prepared for CrN coating and nitrided layer/CrN. **Tables 3** and **4** are shown the test parameters and the scheme of the test conditions according to the Taguchi approach.

**Table 3. Experimental L9 orthogonal array**
Table 3. Plan ortogonalny L9

| Experiment no. | Load | Speed of rotation | Distance min | Distance max |
|---|---|---|---|---|
| 1. | 1 | 1 | 1 | 1 |
| 2. | 1 | 2 | 2 | 2 |
| 3. | 1 | 3 | 3 | 3 |
| 4. | 2 | 3 | 1 | 2 |
| 5. | 2 | 1 | 2 | 3 |
| 6. | 2 | 2 | 3 | 1 |
| 7. | 3 | 2 | 1 | 3 |
| 8. | 3 | 3 | 2 | 1 |
| 9. | 3 | 1 | 3 | 2 |

**Table 4. Test conditions for the CrN and the composite layer: nitrided layer/CrN coating**
Tabela 4. Parametry planu eksperymentu dla powłoki CrN i warstwa azotowana/CrN

| Specimen no. | Experimental parameters for the CrN and nitrided layer/CrN | | | | | |
|---|---|---|---|---|---|---|
| | Load [N] | Speed of rotation [rpm] | Distance 1 [m] | Distance 2 [m] | Distance 3 [m] | Distance 4 [m] |
| 1. | 0.1 | 38 | 4 | 8 | 12 | 16 |
| 2. | 0.1 | 80 | 8 | 24 | 40 | 56 |
| 3. | 0.1 | 150 | 12 | 40 | 68 | 96 |
| 4. | 0.2 | 150 | 4 | 21 | 39 | 56 |
| 5. | 0.2 | 38 | 8 | 37 | 67 | 96 |
| 6. | 0.2 | 80 | 12 | 14 | 15 | 16 |
| 7. | 0.4 | 80 | 4 | 35 | 65 | 96 |
| 8. | 0.4 | 150 | 8 | 11 | 13 | 16 |
| 9. | 0.4 | 38 | 12 | 27 | 41 | 56 |

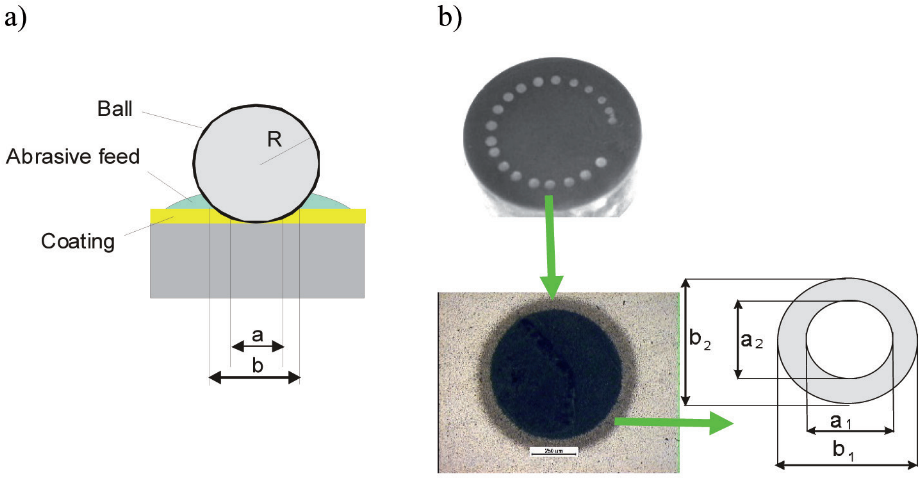

**Fig. 4. Micro-abrasion tests: a) diagram, b) traces of crater-shaped wear after wear tests on the PVD coating [L. 20]**
Rys. 4. Testy mikrościerania: a) schemat, b) ślady zużycia po badaniach ściernych **[L. 20]**

The diameters, **a** of the craters in the substrate (inner diameter) and **b** in the coating (outer diameter) of the worn disc (**Figure 4**), are both parallel and perpendicular to the direction of ball rotation and were measured after the wear test.

On the basis of the experiments performed using the Taguchi statistic program, the parameters at which abrasion of the coating occurs (with the best wear traces in terms of shape and the thickness of the ring between the abrasion and the substrate) were determined. On the basis of the Eta function (**Fig. 5**), the values of the maxima were read, which are the optimal values of the working conditions for the CrN coating. The necessary condition was to obtain the smallest possible relative error of the difference between the largest and the smallest wear mark diameters, with the appropriate dimension of the internal diameter, denoting abrasion of the coating.

According to Taguchi's approach, the optimal values of the working conditions parameters in the case of the CrN coating are respectively for the parameter: load 0.2 N, rotational speed 150 rpm, minimum friction distance d1 – 12 m, and maximum friction distance d4 – 96 m, while the intermediate roads were: d2 – 40 m and d3 – 68 m.

As a result of the verification tests carried out with the optimal operating parameters determined from the Eta function: load 0.2 N, rotational speed 150 rpm, the values of the four friction paths: 12 m, 40 m, 68 m, 96 m (minimum distance d1, maximum d4 together with intermediate routes d2, and d3),

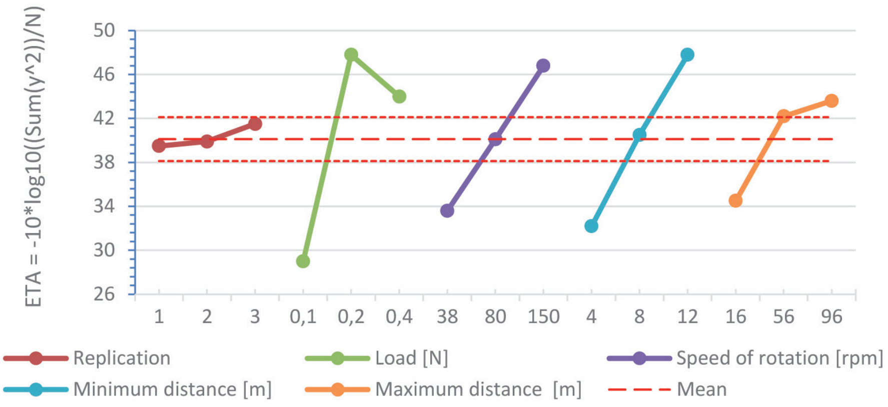

**Fig. 5. Chart of the average values of the criterion function Eta for the CrN coating**

Rys. 5. Wykres średnich wartości funkcji kryterium Eta dla powłoki CrN

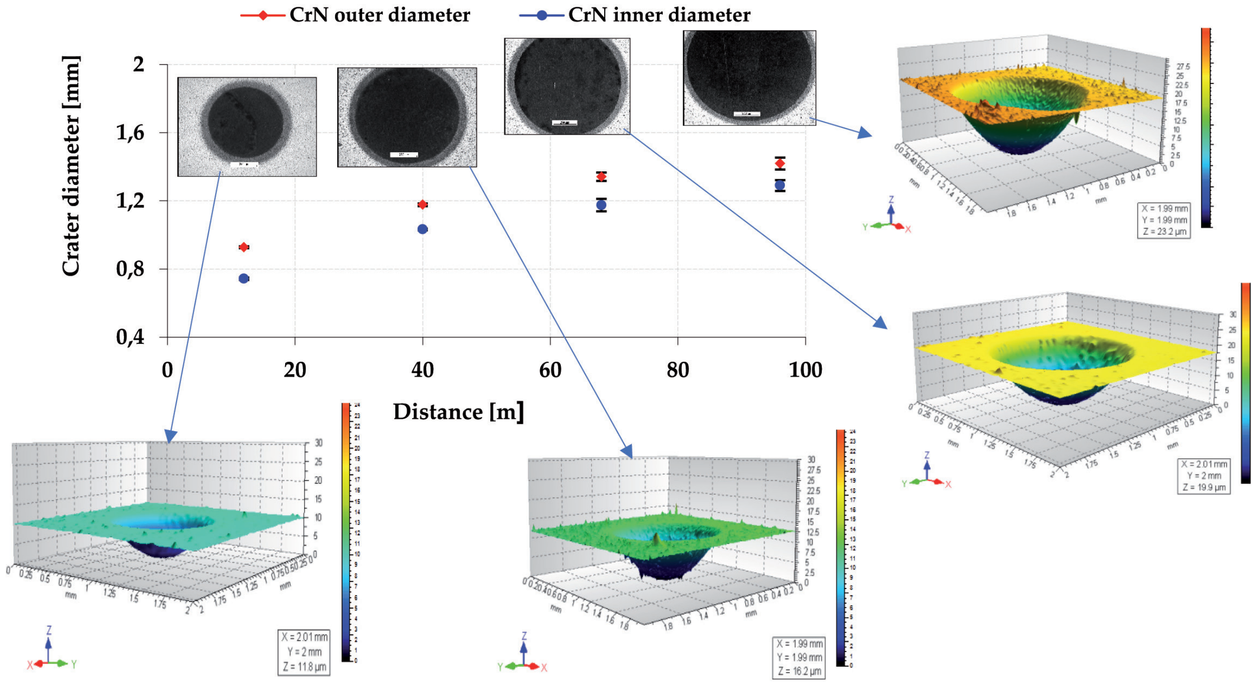

**Fig. 6. The effect of sliding distance on the diameters of the crater in the coating and the substrate with the standard deviation of the CrN coating**

Rys. 6. Wpływ drogi tarcia na wartości średnic otrzymanych kraterów po badaniach zużyciowych dla powłoki CrN

four wear scars were obtained (**Figure 6**). The craters met the conditions described in the standard, i.e., the shape and size of the internal diameter; thus, they can participate in the determination (Kc) of the CrN abrasion coefficient.

The results of the wear scars measurements were presented in the form of a graph of the value of the outer diameter and the value of the inner diameter as a function of the distance (**Figure 6**). By analysing the curves, it was observed that the thickness of the visible ring, formed as a result of abrasion of the coating, is stable and does not change along the road section of 12 m – 96 m.

Then, measurements of the depth of the craters were made using a profilometer. The obtained test results are shown in **Figure 6**. A rubbing penetration into the crater increases with the increasing length of the friction distance.

In order to calculate the value of the coating coefficient Kc, the values of the four friction distance were used, by means of which the linear curve was drawn, and the values of the coefficient of the abrasive wear rate of coating $K_{c\ CrN} = 21.0$ [$10^{-13}m^3N^{-1}m^{-1}$] and the coefficient of the abrasive wear rate of substrate $K_s = 8.0$ [$10^{-13}m^3N^{-1}m^{-1}$] were determined.

Following the procedure, the CrN coating/nitrided layer tests were carried out. On the basis of the conducted experiments, the Eta function was built in accordance with Taguchi's requirements, and the maxima of the function for the CrN coating/nitrided layer were determined (**Fig. 7**). Then, the parameter values for the load, speed, and minimum and maximum friction distance were read and verified.

**Figure 7** shows the maxima of the Eta function for the nitrided layer/CrN coating verification test. In accordance with Taguchi's approach, the following values of operating parameters were selected: load 0.4 N, rotational speed 150 rpm, minimum distance 12 m and maximum distance 96 m, and intermediate roads 40 m and 68 m.

Wear tests were carried out with the operating parameters determined from the maximums of the Eta function. The results of measurements of the outer and inner diameters of the craters of the measured diameters are shown in **Figure 8**.

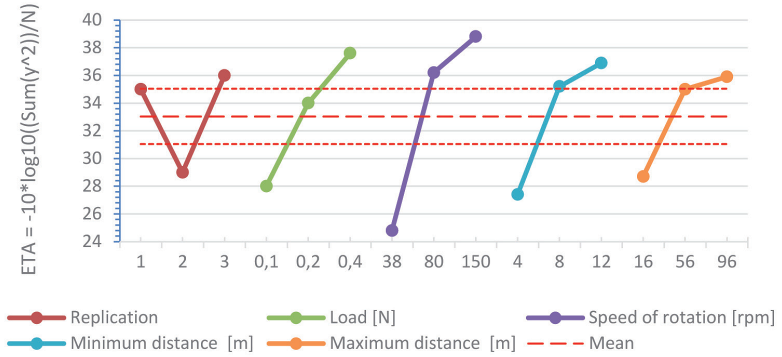

**Fig. 7. Chart of the average values of the criterion function ETA for the nitrided layer/CrN coating**
Rys. 7. Wykres średnich wartości funkcji kryterium ETA dla warstwy azotowanej/powłoki CrN

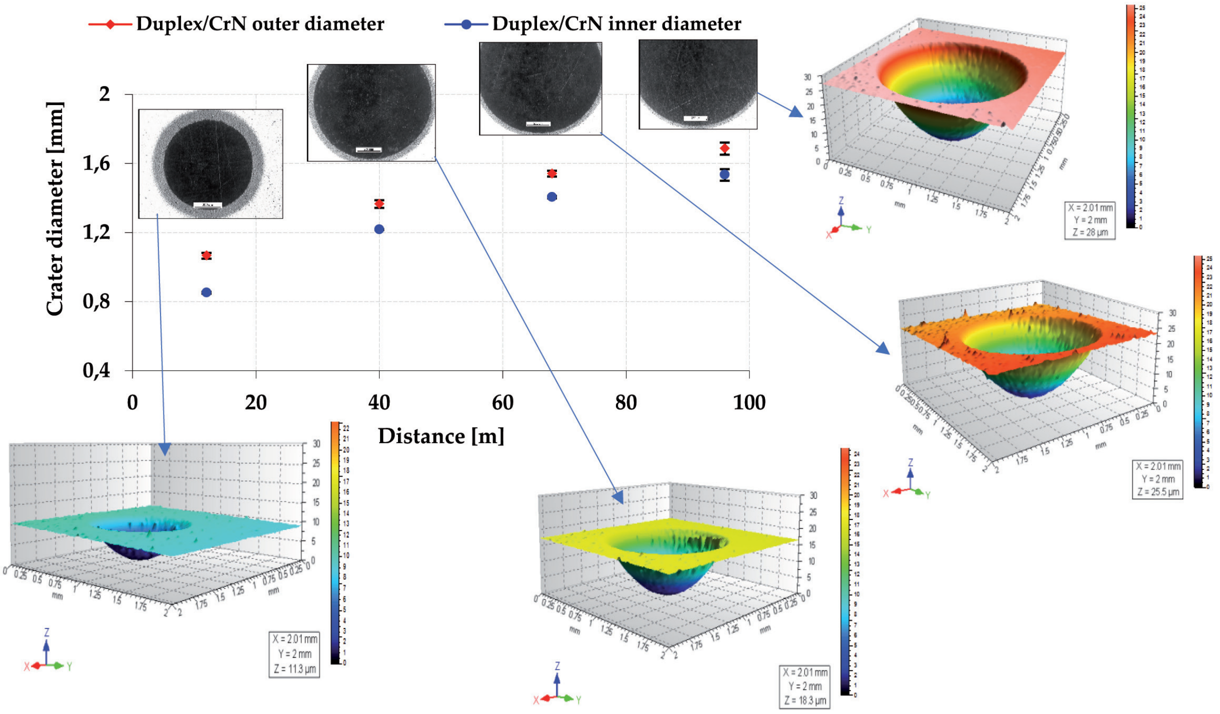

**Fig. 8. The effect of sliding distance on the diameters of the crater in the substrate with the standard deviation of the nitride layer/CrN coating**
Rys. 8. Wpływ drogi tarcia na wartości średnic otrzymanych kraterów po badaniach zużyciowych dla warstwy azotowanej/powłoki CrN

By analysing the curves of the inner and outer diameters shown in **Figure 8**, a stable and uniform difference in the values of both diameters was observed, which allowed distinguishing the thickness of the ring in the designated range of friction paths. This is also confirmed by the analysis of optical photos, on the basis of which it was found that the craters meet the shape conditions and are allowed to agree to the view on the correctness of the determined operating parameters. The crater depths were measured, and the results of the wear trace profiles are shown in **Figure 8**. When analysing the graphs, it was confirmed that the longer the path, the deeper the crater has the wear trace. The results obtained for both diameters (outer and inner) for the tested coatings show a tendency to increase the diameters' size by increasing the sliding distance. The repeatability of micro-abrasion wear tests performed has been found to be very good.

The last stage, in accordance with the developed procedure, was the determination of the coefficient of the abrasive wear rate of the substrate $K_s$ = 8.0 [$10^{-13}m^3N^{-1}m^{-1}$] and the coefficient of the abrasive wear rate of the nitrided layer/CrN coating $K_c$ nitrided layer/CrN = 18.0 [$10^{-13}m^3N^{-1}m^{-1}$].

## CONCLUSIONS

The main purpose of the article was to present the developed procedure for selecting the values of the operating parameters of the ball-cratering method for assessing resistance to abrasive wear using the Taguchi method, a statistical optimisation method. In this study, the feasibility and reliability of the procedure have been verified in the investigation of the resistance to abrasive wear for the CrN coating and nitrided layer/CrN coating by using the ball-cratering method. The results of the presented study indicate that the Taguchi optimisation method can be used to select the testing conditions for a ball-cratering abrasive wear test of PVD coatings. According to the presented procedure, the wear abrasive tests showed very good repeatability to obtain wear scars.

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