Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 17
ISSUE 1-2
July-August
2006

Fractal and multifractal characteristics of coatings deposited on pure oxide ceramics

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Received 15.03.2006; accepted in revised form 30.04.2006

Analysis and modelling

ABSTRACT

Purpose: The goal of this work is the fractal and multifractal characteristics of the TiN and TiN+multiTiAlSiN+TiN coatings obtained in the PVD process, and of the TiN+Al_2O_3 coating obtained in the CVD process on the Al_2O_3+ZrO_2 oxide tool ceramics substrate.

Design/methodology/approach: The investigations were carried out of the multi-edge inserts from the Al_2O_3+ZrO_2 oxide tool ceramics uncoated and coated with the TiN and TiN+multiTiAlSiN+TiN coatings deposited in the cathode arc evaporation CAE PVD process, as well as with the TiN+Al_2O_3 coating obtained in the CVD process. Determining the fractal dimension and the multifractal analysis of the examined coatings were made basing on measurements obtained from the AFM microscope, using the projective covering method.

Findings: Investigations carried out confirm that the fractal dimension and parameters describing the multifractal spectrum shape may be used for characterizing and comparing surfaces of coatings obtained in the PVD and CVD processes and of the substrate material from the Al_2O3+ZrO_2 oxide tool ceramics.

Research limitations/implications: Investigation or relationship between parameters describing the multifractal spectrum and physical properties of the examined materials calls for further analyses.

Originality/value: Investigations carried out confirm that the fractal dimension and parameters describing the multifractal spectrum shape may be used for characterizing and comparing surfaces of coatings obtained in the PVD and CVD processes.

Keywords: Computational Material Science; PVD coatings; Multifractal geometry; AFM

1. Introduction

Fractal and multifractal analysis finds broader and broader application recently in many branches of science, inclusive materials engineering - to describe surface structure. The ongoing research pertains not only to the description of phenomena alone more and more often also to their relationship with properties of materials. In the case of the coatings obtained in the PVD and CVD processes numerous physical qualities depend on the structure and the chemical constitution. The coatings are also characterised by specific geometric qualities to describe which such notions as morphology, topography and shape are used. The results of the research indicate that there is a relation between the morphology of the surface of the coatings and the technology used in the process. It is extremely important to define the kind of relation as the morphology of the surface is crucial for such properties of coatings as: roughness parameter, coefficient of friction, hardness and wear resistance [1-5, 9,10].

2. Experimental procedure

The investigations were carried out of the multi-edge inserts from the Al_2O_3+ZrO_2 oxide tool ceramics uncoated and coated with the TiN and TiN+multiTiAlSiN+TiN coatings deposited in the cathode arc evaporation CAE PVD process, as well as with the $TiN+Al_2O_3$ coating obtained in the CVD process.

Analysis of the phase composition of the obtained coatings was carried out using the Dron-2.0 X-ray diffractometer.

Examinations of the coating thickness were made using the ”kalotest” method, consisting the measurement of the characteristic parameters of the crater developed as a result of wear on the specimen surface made with the steel ball.

The micro hardness tests of the coatings were carried out on the SHIMADZU DUH 202 ultra-microhardness tester. Tests were made with the 0,07N load.

Examinations of the topography of the substrate material surface and of the deposited coatings were made on the scanning electron microscope and using the atomic force microscopy method (AFM) on the Digital Instruments Nanoscope E instrument (Fig. 1a). Scanning range was 10 µm respectively.

Determining the fractal dimension and the multifractal analysis of the examined coatings were made basing on measurements obtained from the AFM microscope, using the projective covering method [2,8].

Basing on the information of the total size of the analysed surface:

$$A(\delta)=\sum_{i=1}^{N(\delta)}A_i(\delta) \tag{1}$$

determined by totalling surfaces of all N(δ) cover boxes in a given scale, needed to cover the analysed set:

$$A_i(\delta)=\frac{1}{2}\{[\delta^2+(h_{ai}-h_{di})^2]^{\frac{1}{2}}\cdot[\delta^2+(h_{di}-h_{ci})^2]^{\frac{1}{2}}$$
$$+[\delta^2+(h_{ai}-h_{bi})^2]^{\frac{1}{2}}\cdot[\delta^2+(h_{bi}-h_{ci})^2]^{\frac{1}{2}}\} \tag{2}$$

it is possible to evaluate the surface fractal dimension D_s using the formula:

$$A(\delta)\propto\delta^{2-Ds} \tag{3}$$

Value of D_s is the real number from the range of [2, 3) and does not depend on the size of the analysed test piece surface size. By evaluating the surface sizes $A_i(\delta)$ of the particular covering boxes it is possible to determine the probability to find a box with a given size:

$$P_i(\delta)=\frac{A_i(\delta)}{A\ (\delta)} \tag{4}$$

The points on bilogarythmic graph (fig. 1b) for fractal surface (described by relationship 3) are arranged along straight line. The slope of this line make possible to determine fractal dimension for analysed surface. If this assumption is not completely true, fractal dimension value is depended on selection of points. Auxiliary graphs (fig. 1c) present changes of fractal dimension value, determined by using two consecutive points. The auxiliary graphs help to select necessary points correctly and to determine selfsimilarity range.

The multifractal analysis consists in partitioning the analysed surface into subsets composed of boxes for which the following relationship is true:

$$P_i(\delta)\propto\delta^{\alpha} \tag{5}$$

where α is the the singularity of the subset of probabilities. The goal of this analysis is evaluation of the size of these subsets. The number of boxes of size δ with the same probability $N_\alpha(\delta)$ included in the subset specified with the value of α is described with function f(α), defined as the multifractal spectrum in the following way (Fig. 1d) [2, 6, 8]:

$$N_\alpha\ (\delta)\propto\delta^{-f(\alpha)} \tag{6}$$

The normalized measure is construed on probability values defined with formula (4):

$$\mu(q,\delta)=\frac{[P_i(\delta)]^q}{\sum_i^{\mathrm{N}(\delta)}[P_i(\delta)]^q} \tag{7}$$

The generalised fractal dimension D(q) assumes the following form with these designations:

$$D(q)=\lim_{\delta\to0}\frac{1}{q-1}\frac{\log Z(q,\delta)}{\log\delta}=\frac{1}{q-1}\lim_{\delta\to0}\frac{\log\sum_{i=1}^{N}[P_i(\delta)]^q}{\log\delta} \tag{8}$$

where the partial function Z(q,δ) (the so-called partition function) is defined in the following way:

$$Z(q,\delta)=\sum_{i=1}^{N(\delta)}[\mu_i(\delta)]^q \tag{9}$$

In the general case exponent q is a real number, determining the order of the moment of the measure. Numerical calculations are carried out on integers, usually from the range of (-100, 100). The formula above cannot be used directly, as it is not possible to determine $P_i(\delta)$ for the arbitrarily small value of δ. The first step of evaluating D_q in the real measurements [8] is determining in what scope of magnifications (for what δ values) the following exponential relationship is true:

$$Z(q,\delta)\propto\delta^{\tau(q)} \tag{10}$$

The auxiliary function τ(q) (convex function) present in formula (10) is connected with the generalised dimension with the following formula:

$$\tau(q)=(q-1)D(q) \tag{11}$$

One can evaluate parameter α using the Legendre’s transform, according to:

$$\alpha(q)=\frac{d\tau(q)}{dq} \tag{12}$$

$$\alpha(q)=\lim_{\delta\to0}\frac{\sum_i\mu_i(q,\delta)\log P_i(\delta)}{\log\delta} \tag{13}$$

and the multifractal spectrum f(α):

$$f(\alpha)=q\alpha(q)-\tau(q) \tag{14}$$

$$f(\alpha)=\lim_{\delta\to 0}\frac{\sum_i \mu_i(q,\delta)\log\mu_i(q,\delta)}{\log\delta} \quad (15)$$

To make characterising and comparison of the obtained multifractal spectra possible their width - Δα is determined:

$$\Delta\alpha=\alpha_{\max}-\alpha_{\min} \quad (16)$$

as well as of the spectrum arms' heights difference Δf:

$$\Delta f=f(\alpha_{\min})-f(\alpha_{\max}) \quad (17)$$

As $\delta \ll 1$, then α_{min} represents the highest probability ($P_{max} \sim \delta^{\alpha min}$; according to formula (6)); whereas α_{max} represents the lowest probability ($P_{min} \sim \delta^{\alpha max}$) Therefore, the Δα value (span of the multifractal spectrum arms) may feature a measure of variability of probabilities ($P_{max}/P_{min} \sim \delta^{-\Delta\alpha}$) and indirectly also of the range of variability of the cover boxes sizes $A_i(\delta)$ for the particular fragments. Results of both the computer simulation [7] and of the multifractal analysis of the surface topography obtained from the AFM microscope described in the literature [6] suggest that the spectrum breadth is connected with roughness of coatings. Parameters $f(\alpha_{max})$ and $f(\alpha_{min})$ reflect the numbers of boxes with the maximum ($N_{Pmax}(\delta)=N_{\alpha min} \sim \delta^{-f(\alpha min)}$) and minimum ($N_{Pmin}(\delta)=N_{\alpha max} \sim \delta^{-f(\alpha max)}$) probability values respectively. Value $\Delta f=f(\alpha_{min})- f(\alpha_{max})$ is a measure of the ratio of the number of boxes with the highest probability to the number of boxes with the lowest probability ($N_{Pmax}(\delta)/N_{Pmin}(\delta)=\delta^{-\Delta f}$). In case $\Delta f>0$ then fragments described by the high probability value predominate; whereas, in case $\Delta f<0$ then fragments described by the low probability value predominate [6].

Moreover, measurements carried out using the AFM atomic force microscope made it also possible to determine parameter R characterising the analysed test piece surface roughness for the analysed scanning ranges, which was evaluated according to [7] using the formula:

$$R=\left[\frac{\sum_i (h_i-H)^2}{N_s}\right]^{\frac{1}{2}} \quad (18)$$

and

$$H=\sum_i h_i / N_s \quad (19)$$

where R- roughness, h_i – test piece height at point; N_s – number of measurement points, H - average test piece height.

3. Results and discussion

It was found out basing on the X-ray qualitative phase analysis that according to the assumptions the TiN and TiN+ multiTiAlSiN+TiN coatings were deposited on the investigated Al_2O_3+ZrO_2 oxide tool ceramics in the cathode arc evaporation CAE PVD process and the TiN+Al_2O_3 one obtained in the CVD process respectively.

Table 1.
Results of the mechanical properties tests of the analysed materials

Examined material	Thickness [μm]	Microhardness [GPa]	Roughness [R]
Substrate Al_2O_3+ZrO_2	-	18,5	0,12
TiN (PVD)	1	23	0,17
TiN+multiTiAlSiN +TiN (PVD)	2,5	40	0,27
TiN+Al_2O_3 (CVD)	6	34	0,51

It was found out, basing on the coating thickness measurement results that the TiN+Al_2O_3 coating obtained in the CVD process demonstrates the biggest thickness of 6 μm; whereas the smallest thickness of 1,0 μm displays the TiN coating obtained in the PVD process.

The microhardness tests carried out revealed that the uncoated Al_2O_3+ZrO_2 substrate has hardness equal to 18,5 $HV_{0,07}$ [GPa]. Deposition of the TiN, TiN+TiAlSiN+TiN and TiN+Al_2O_3 coatings onto the substrate causes the significant surface layer hardness increase within the range of 23-40 [GPa]. The highest hardness of 40 [GPa] is displayed by the TiN+multiTiAlSiN+TiN coating; whereas, the lowest hardness of 23 [GPa] is displayed by the TiN coating.

Table 1 presents results of thickness measurements, microhardness tests, and roughness measurements specified by parameter R and determined according to (18) of the analysed coatings put down onto the oxide tool ceramics, as well as of the microhardness and roughness of the substrate material.

Depositing the TiN, TiN+multiTiAlSiN+TiN and TiN+Al_2O_3 coatings in the PVD and CVD processes onto the analysed substrate causes the fractal dimension value growth (Table 2).

One can state, basing on the obtained results that the width and difference of heights of the multifractal spectrum arms correlate with roughness of the investigated surface determined by parameter R. Multifractal spectrum refers to surfaces characteristic of a lower parameter R value. These materials also have the highest (in terms of their absolute value) negative difference of the multifractal spectrum arms heights. The TiN+Al_2O_3 coating described by the highest value of the surface fractal dimension and roughness R has the broadest spectrum and the smallest difference of the multifractal spectrum arms heights (Table 2).

4. Conclusions

The paper presents investigation results obtained from the AFM microscope of the Al_2O_3+ZrO_2 tool ceramics uncoated and coated with the TiN and TiN+multiTiAlSiN+TiN coatings deposited in the CVD process. Values of the surface fractal dimension and the multi-fractal spectra were determined using the projective covering method.

Table 2.
Fractal and multifractal characteristics analyses coatings

Examined material	D_s	α_{min}	$f(\alpha_{min})$	α_{max}	$f(\alpha_{max})$	Δα	Δf
Substrate	2,018	1,88	0,43	2,01	1,87	0,13	-1,44
TiN	2,031	1,81	0,04	2,02	1,79	0,21	-1,75
TiN+multiTiAlSiN+TiN	2,035	1,81	0,47	2,03	1,71	0,22	-1,24
TiN+Al_2O_3	2,078	1,72	0,41	2,08	1,51	0,36	-1,1

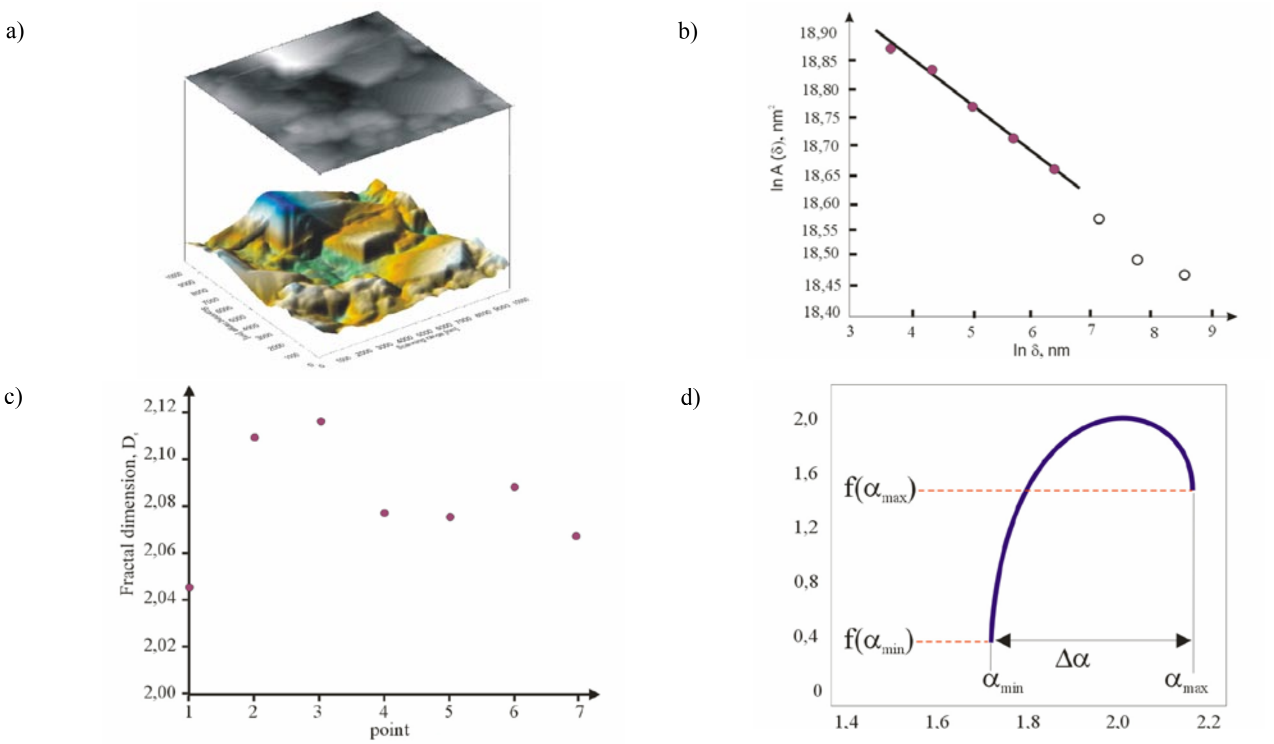

Fig 1. Fractal and multifractal characteristic TiN+Al_2O_3 coatings a) Topography of the analysed surface (AFM) b) Bilogarithmic relationship of the approximated analysed surface from the mesh side size used to its determining c) Auxiliary diagram making it possible to determine correctly the fractal dimension of the analysed surface d) Multifractal spectrum of the analysed surface

Investigations carried out confirm that the fractal dimension and parameters describing the multifractal spectrum shape may be used for characterizing and comparing surfaces of coatings obtained in the PVD and CVD processes and of the substrate material from the Al_2O_3+ZrO_2 oxide tool ceramics.

Acknowledgements

Investigations were financed in part within the framework of the research project KBN 3 T08C 019 28 headed by Dr. W. Kwaśny.

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