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Fractal and multifractal characteristics of CVD coatings deposited onto the nitride tool ceramics

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Analysis and modelling

ABSTRACT

Purpose: The goal of this work is the fractal and multifractal characteristics of the TiN+Al_2O_3 and Al_2O_3+TiN coatings obtained in the CVD process on the Si_3N_4 tool ceramics substrate.
Design/methodology/approach: The investigations were carried out of the multi-edge inserts from the Si_3N_4 nitride tool ceramics uncoated and coated with the TiN+Al_2O_3 and Al_2O_3+TiN coatings deposited 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 CVD processes and of the substrate material from the Si_3N_4 nitride 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: Employment of multifractal geometry in materials engineering provides the opportunity to work out more complete, also quantitative, characteristics of properties of the investigated objects. Multifractal analysis makes it possible to characterise in the quantitative way the extent of irregularities of the analysed surface.
Keywords: Computational material science; CVD coatings; Multifractal geometry; AFM

1. Introduction

The Si_3N_4 tool ceramics and sialons belong to materials that have a real possibility to replace steel and sintered carbides in future. Employment of these materials makes high speed machining possible with high feed rates both by turning an by milling [1,2].

Apart from developing the new manufacturing methods or modifying the existing ones, coating techniques are improved for these materials, especially the Si_3N_4 one, with the new hard coatings obtained in the CVD (Chemical Vapour Deposition) and PVD (Physical Vapour Deposition) processes. Tools coated with layers based on carbides, borides, nitrides, and oxides can work at higher machining parameters. The high-temperature CVD making it possible to obtain a combination of the Al_2O_3+TiN layers is one of the most often used methods in coating this ceramics. The thermodynamically stable Al_2O_3 layers feature the diffusion barrier between the tool insert and the chip flowing on it, which results in the insert's wear resistance improvement [3-6].

Employing the fractal models for modelling of structures and processes has become the tool in the theoretical and experimental research in the areas of geology, biology, medicine, astronomy, economy, physics, astrophysics, computer science (mostly for data compression and in computer graphics), and materials engineering. The big-scale matter distributions in the Universe, rock structures, coastline shapes, traces of electrical discharges, short-term changes of prices and stock quotations, and shapes of some cells may be such examples. Employment of fractal geometry in materials engineering provides the opportunity to work out more complete, also quantitative, characteristics of properties of the investigated objects. Fractal and multifractal analyses make it possible to characterise in the quantitative way the extent of irregularities of the analysed surfaces, in case when this value is independent of scale. Better and better service properties of machine elements are obtained by forming the structure and properties of their surface layers. The surface layers display geometrical features, whose description is connected with the following concepts: morphology, topography and surface shape. Carrying out the fractal and multifractal analyses of such layers makes it possible to determine the fundamental parameters describing the surface [7-14].

2. Experimental procedure

The investigations were carried out on the multi-point inserts made from the Si_3N_4 nitride ceramics uncoated and coated in the CVD process with $TiN+Al_2O_3$ and Al_2O_3+TiN coatings.

Phase compositions of the obtained coatings were determined using the Dron 2.0 X-ray diffractometer, using the filtered radiation from the cobalt lamp with the voltage of 40 kV and heater current of 20 mA. The measurements were made in the 2Θ angle ranging from 35 to 95˚.

The microhardness tests of coatings were made on the SHIMADZU DUH 202 ultra microhardness tester. Test conditions were selected so that the required and comparable test results would be obtained for all analyzed coatings. Measurements were made at 0.07 N load, eliminating influence of the substrate on the measurement results.

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. Scanning ranges were 5 and 2 μm respectively.

The detailed methodology of the fractal and multifractal analyses was presented in [7, 8, 15].

3. Results and discussion

Phase compositions of the investigated CVD coatings and of the substrate were examined using the X-ray qualitative phase analysis method. Occurrences of the TiN and Al_2O_3 coating was found in the X-ray diffraction patterns (Fig. 1), and moreover, reflexes occurred coming from the Si_3N_4 nitride ceramics substrate.

It was found out basing on the metallographic examinations on the scanning electron microscope that all coatings developed in the high-temperature CVD process are characterized by the laminar structure. The particular layers of the compound coatings are characterized by tight adhesion to each other and to the substrate from the nitride ceramics (Fig.2).

Characteristic fracture surface of the TiN+ Al_2O_3 coating is presented in Figure 2 and its corresponding surface topography image is presented in Figure 3.

It was found out basing on thickness measurements of coatings that the biggest thickness of 10 μm is displayed by the $TiN+Al_2O_3$ one. It was found out, basing on hardness tests results that the highest hardness of 32.6 GPa is displayed by the nitride ceramics with the $TiN+Al_2O_3$ coating deposited in the CVD process; whereas the smallest hardness of 18.5 GPa was observed for the Si_3N_4 nitride ceramics (Table 1).

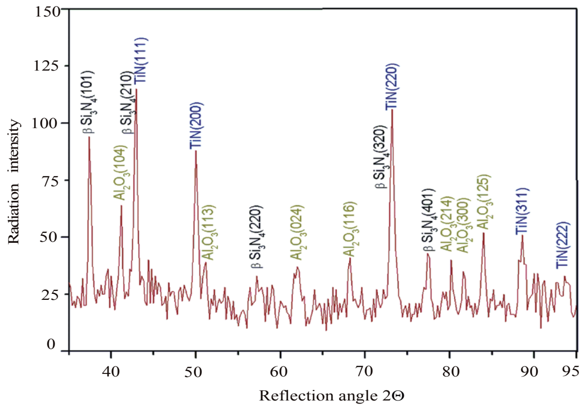

Fig. 1. X-ray diffraction pattern of the $TiN+Al_2O_3$ coating put down onto the substrate from the Si_3N_4 nitride ceramics

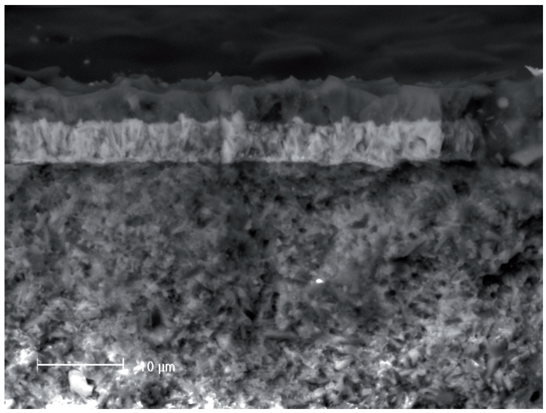

Fig. 2. Fracture surface of the $TiN+Al_2O_3$ coating deposited onto the substrate from the Si_3N_4 nitride ceramics

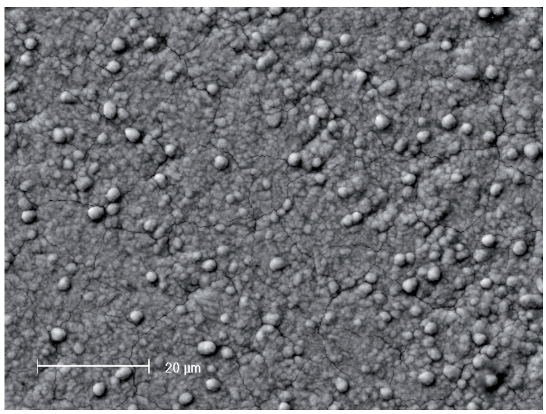

Fig. 3. Surface topography of the Al_2O_3+TiN coating deposited onto the substrate from the Si_3N_4 nitride ceramics

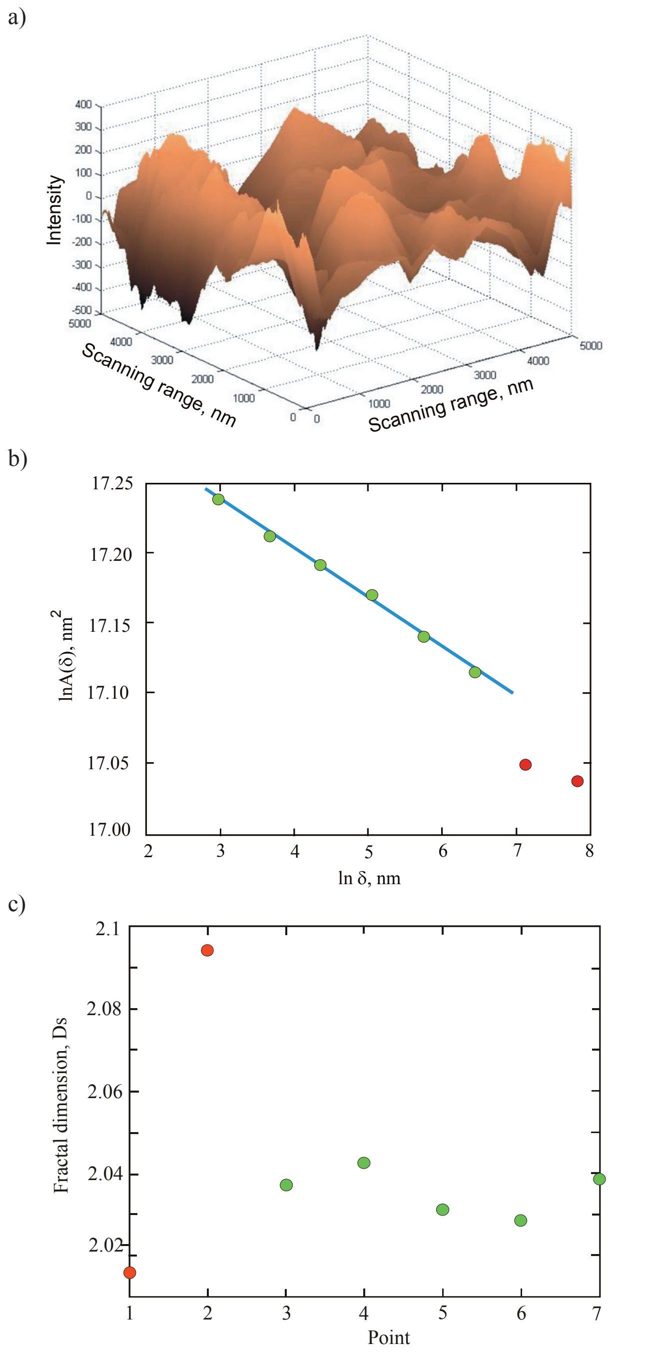

Fig. 4. a) Image of surface topography of the Si_3N_4 nitride ceramics with the TiN+Al_2O_3 coating obtained in the CVD process, b) bilogarithmic relationship of the approximated analysed surface of the Si_3N_4 nitride ceramics with the TiN+Al_2O_3 coating depending on the mesh side size used to its determining, and c) its corresponding auxiliary plot (scanning range 5000 nm)

Fractal dimension of the substrate material and of the TiN+Al_2O_3 and Al_2O_3+TiN coatings deposited onto the Si_3N_4 nitride ceramics

Table 1
Summary of the investigation results of the coated Si_3N_4 nitride ceramics

Coating	Coating thickness, μm	Hardness, GPa
TiN+Al_2O_3	10.0	32.6
Al_2O_3+TiN	2.6	26.3
uncoated Si_3N_4	-	18.5

was determined using the projective covering method [15]. Topography images of the analysed coatings' surfaces and of the substrate material, obtained using the AFM atomic force microscopy, were used for its calculation and saved in the text file as 512x512 measurement points (Fig. 4a). The determined A(δ) values are presented in bilogarithmic plots (Fig. 4b) and the auxiliary plots were made which show changes of the fractal dimension value, determined basing on two consecutive points of the bilogarithmic diagram make their correct selection easier (Fig. 4c).

Based on the multifractal analysis spectra of the generalised fractal dimensions were determined for all scanning ranges (Fig. 5) and their corresponding multifractal spectra (Fig. 6) of the analysed coatings and substrate material. Measurements carried out using the AFM atomic force microscope made it also possible to determine parameter R characterising the analysed surface roughness according to [15].

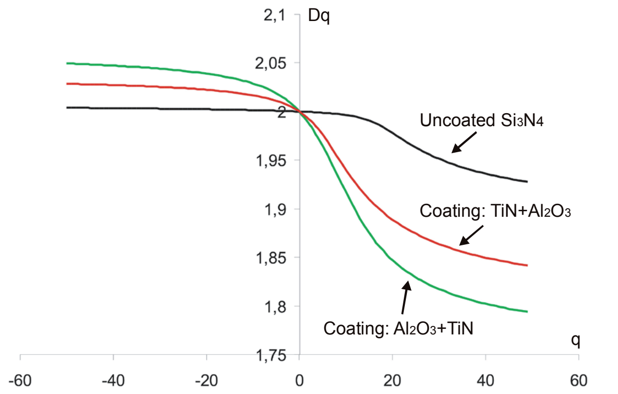

Fig. 5. Spectra of the generalized fractal dimensions of the analysed coatings and substrate material (scanning range 5000 nm)

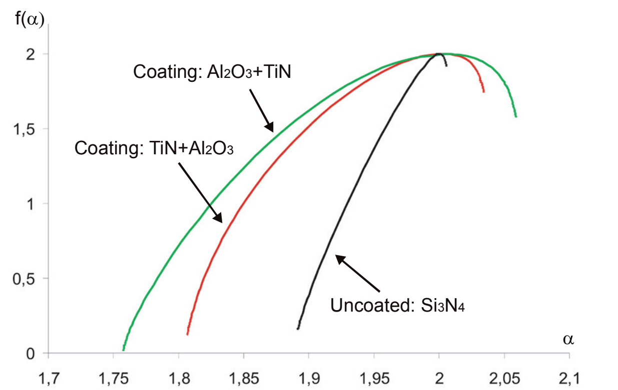

Fig. 6. Multifractal spectra of the analysed coatings and substrate material (scanning range 5000 nm)

Table 2.
The detailed results of the fractal and multifractal analysis and of the roughness parameter R

Material type	Coating	α_{min}	$f(\alpha_{min})$	α_{max}	$f(\alpha_{max})$	$\Delta\alpha$	Δf	D_s	Roughness, μm
Si_3N_4 nitride ceramic (Scanning range 5000 nm)		1.8916	0.1564	2.0056	1.9161	0.114	1.7597	2.0134	0.31
	$TiN+Al_2O_3$	1.8066	0.1195	2.0343	1.7417	0.2277	1.6222	2.0353	0.89
	Al_2O_3+TiN	1.7575	0.0124	2.0589	1.5731	0.3014	1.5607	2.1101	0.98
Si_3N_4 nitride ceramic (Scanning range 2000 nm)		1.9630	0.1564	2.0035	1.9443	0.0405	1.7879	2.0083	0.13
	$TiN+Al_2O_3$	1.7769	0.3329	2.0215	1.8141	0.2446	1.4812	2.0111	0.36
	Al_2O_3+TiN	1.7663	0.0728	2.0592	1.5961	0.2929	1.5233	2.0257	0.50

It was found out based on the investigations carried out that the lowest fractal dimension Ds value and the narrowest multifractal spectrum $\Delta\alpha$ width displays the Si_3N_4 nitride ceramics substrate material, regardless from the scanning range. Deposition of the $TiN+Al_2O_3$ and Al_2O_3+TiN coatings on the Si_3N_4 nitride ceramics results in the increase of the fractal dimension Ds value and in the increase of the $\Delta\alpha$ multifractal spectrum from Ds = 2.013 and $\Delta\alpha$ = 0.114 to Ds = 2.035 and $\Delta\alpha$ = 0.227, and Ds = 2.11 and $\Delta\alpha$ = 0.301 respectively for the scanning range of 5μm and from Ds = 2.008 and $\Delta\alpha$ = 0.04 to Ds = 2.011 and $\Delta\alpha$ = 0.244, and Ds = 2.025 and $\Delta\alpha$ = 0.292 respectively for the scanning range of 2μm.

It was found out based on the obtained roughness R values that development of the $TiN+Al_2O_3$ and Al_2O_3+TiN coatings on the Si_3N_4 nitride ceramics results in the increase of the same parameter value in reference to the substrate material. The detailed fractal and multifractal analysis summary results and the obtained R parameter results are presented in Table 2.

4. Conclusions

Examinations of the phase composition confirmed that, according to the assumptions, the $TiN+Al_2O_3$ and Al_2O_3+TiN coatings were developed on the Si_3N_4 nitride ceramics.

Observations of the fractures on the scanning electron microscope revealed that the coatings were deposited uniformly and that they adhere tightly to the substrate from the Si_3N_4 nitride ceramics and that they are characteristic for the structure with no cracks, pores, and discontinuities.

It was found out based on the investigations carried out that depositing the $TiN+Al_2O_3$ and Al_2O_3+TiN coatings onto the Si_3N_4 nitride ceramics results in hardness growth on an average by 80% and 40% respectively.

It was found out based on the fractal and multifractal analyses in case of the analysed test pieces that depositing the $TiN+Al_2O_3$ and Al_2O_3+TiN coatings results in the increase of the Ds and $\Delta\alpha$ parameters in respect to the substrate material. Moreover, based on the roughness parameter R determined for the analysed test pieces it was found out that the fractal dimension Ds and $\Delta\alpha$ parameter values grow along with the R parameter growth.

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