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# Fractal and multifractal characteristics of the PVD and CVD coatings deposited onto compound tool ceramics

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#### 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<sub>2</sub>O<sub>3</sub> coating obtained in the CVD process on the  $Al_2O_3$ +TiC oxide tool ceramics substrate.

**Design/methodology/approach:** The investigations were carried out of the multi-edge inserts from the  $AI_2O_3$ +TiC 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+ $AI_2O_3$  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  $Al_2O_3$ +TiC.

**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; Fractal geometry; Multifractal geometry; AFM

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## METHODOLOGY OF RESEARCH, ANALYSIS AND MODELLING

#### 1. Introduction

Wide choice of coatings available nowadays and technologies for their deposition is an effect of the growing in the last years demand for the state-of-the-art surface modification methods. An increased interest is observed in coatings having joint properties like resistance to tribological wear and corrosion. Tools covered with coatings based on carbides, borides, nitrides, and oxides can work at higher service parameters (temperature, load, etc.). Moreover, the multilayer and multicomponent coatings developed relatively not so long ago make it possible to constitute freely properties of the entire coating as well as of its transition layer, ensuring good adhesion, compensation of the internal stresses, and transmission of the external loads [17-21].

Coatings obtained in the PVD and CVD processes demonstrate many physical properties determined by their chemical composition and structure. They display also the specific geometrical features, whose description is connected with the following concepts: morphology, topography and surface shape. Investigation results indicate to relationships between the coatings surface morphology and the manufacturing technique, and their determining is very important, as the surface morphology has a significant effect on coating properties, like: roughness parameter, friction coefficient, hardness, and wear resistance. Contemporary methods of the coatings surface topography description make it possible to determine relationships among the manufacturing technique parameters, structure, service properties, and their fractal dimension. Therefore, the problem of describing the geometrical features of surfaces of coatings obtained in the PVD and CVD processes is an important issue in surface engineering [3,4,9,10,14, 17-21].

Using the fractal and multifractal models for modelling of structures and processes has become the tool in the theoretical and experimental research in the areas of geology, biology, astronomy, economy, physics, astrophysics, and materials engineering [2,13,15]. The big-scale matter distributions in the Universe, rock structures, coastline shapes, traces of electrical discharges, short-term changes of prices and stock quotations may be such examples. Employment of fractal geometry in case of materials engineering provides the opportunity to work out more complete, also quantitative, characteristics of properties of the investigated objects. Fractal analysis makes it possible to characterise in the quantitative way the extent of irregularities of the analysed surface, when this value is independent of scale. The value of the fractal dimension for the self-similar surfaces, determining the relationship between the surface size and measurement scale is constant in a broad range. Initially, the researchers limited their analyses to determining this parameter only in their investigations of surface properties. However, such attitude turned out to be inadequate in case of test pieces, particular fragments of which have different values of the fractal dimension. Fragments relatively "smooth" and regular may occur next to those being irregular and rough (for which the dimension of the fractal dimension is bigger). Some of their types may occur in the selected fragments only, whereas others may occur on the entire analysed surface in fact. Multifractal analysis makes the quantitative description of such distributions and their comparison possible [4-6, 8-11, 15-16].

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<sub>2</sub>O<sub>3</sub> coating obtained in the CVD process on the  $Al_2O_3$ +TiC oxide tool ceramics substrate.

#### 2. Methodology of research

The investigations were carried out of the multi-edge inserts from the  $Al_2O_3$ +TiC 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, using the filtered radiation of the cobalt anode lamp, powered with 40 kV voltage, at 20 mA heater current. The measurements were made in the 2 $\Theta$  angle range from 35 to 95°.

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. Test conditions were selected so that the comparable results could be obtained for all analysed coatings. The tests were made at load of 0.05 N, making 6 indents for each examined test piece, so that the indent depths would be smaller than 1/10 of the thickness of the deposited coatings, which eliminates to a great extent the substrate influence on the obtained test results.

Measurement of the surface roughness parameter Ra was made on the Taylor-Hobson Sutronic3+ instrument.

Structure of the deposited coatings was observed on transverse sections on the JEOL JCXA 733 transmission electron microscope equipped with the EDS add-on for analysis of the chemical composition. Detection of the secondary electrons at the accelerating voltage of 25 kV was used to obtain the images of fractures.

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. Scanning range was 5, 2, and 1 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 (Fig. 1) [5, 11-12].



Fig. 1. Projective covering method (division of the projection plane by means of the square net along with the projection onto the analysed surface and magnification of one element of the covering projective surface along with the projection onto the part of the analysed surface)

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

$$A(\delta) = \sum_{i=1}^{N(\delta)} A_i(\delta)$$
<sup>(1)</sup>

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

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

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

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

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:

$$P_i(\delta) = \frac{A_i(\delta)}{A(\delta)}$$
<sup>(4)</sup>

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  $\alpha$  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  $\delta$  with the same probability  $N_{\alpha}(\delta)$  included in the subset specified with the value of  $\alpha$  is described with function  $f(\alpha)$ , defined as the multifractal spectrum in the following way (Fig. 2) [6]:

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



Fig. 2. Example of the multifractal spectrum

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

$$\mu(q,\delta) = \frac{[P_i(\delta)]^q}{\sum_{i}^{N(\delta)} [P_i(\delta)]^q}$$
(7)

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

$$D(q) = \lim_{\delta \to 0} \frac{1}{q-1} \frac{\log Z(q,\delta)}{\log \delta} = \frac{1}{q-1} \lim_{\delta \to 0} \frac{\log \sum_{i=1}^{N} [P_i(\delta)]^q}{\log \delta}$$
(8)

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

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

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  $\delta$ . The first step of evaluating  $D_q$  in the real measurements [5] is determining in what scope of magnifications (for what  $\delta$  values) the following exponential relationship is true:

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

The auxiliary function  $\tau(q)$  (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  $\alpha$  using the Legendre's transform, according to:

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

$$\alpha(q) = \lim_{\delta \to 0} \frac{\sum_{i} \mu_i(q, \delta) \log P_i(\delta)}{\log \delta}$$
(13)

and the multifractal spectrum  $f(\alpha)$ :

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

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

To make characterising and comparison of the obtained multifractal spectra possible [11] their width -  $\Delta \alpha$  is determined:

$$\Delta \alpha = \alpha_{\rm max} - \alpha_{\rm min} \tag{16}$$

as well as of the spectrum arms' heights difference  $\Delta f$ :

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

As  $\delta \leq 1$ , then  $\alpha_{\min}$  represents the highest probability  $(P_{max} \sim \delta^{\alpha min}; according to formula (6)); whereas \alpha_{max} represents the$ lowest probability ( $P_{min} \sim \delta^{\alpha max}$ ) Therefore, the  $\Delta \alpha$  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 (Chaudhari et al., 2004) and of the multifractal analysis of the surface topography obtained from the AFM microscope described in the literature (Hui-Sheng et al., 2002) 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 [7].

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 [1] using the formula:

$$R = \left[\frac{\sum_{i} (h_{i} - H)^{2}}{N_{s}}\right]^{\frac{1}{2}}$$
(18)

and

$$H = \sum_{i} h_i / N_s \tag{19}$$

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

### **3. Results**

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  $Al_2O_3$ +TiC oxide tool ceramics in the cathode arc evaporation CAE PVD process and the TiN+ $Al_2O_3$  one obtained in the CVD process respectively (Fig. 3).

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  $\mu$ m; whereas the smallest thickness of 1.0  $\mu$ m displays the TiN coating obtained in the PVD process.

FIC (200) ALO.(104) 250 FIC(111) Al<sub>2</sub>O<sub>3</sub>(113) Al<sub>2</sub>O<sub>3</sub>(116) 200 TIC(220) Intensity, Imp/s Al,O,(110) 150 AI, O, (3 00 FIC(311) Al.,O. (024) ALO. (214) Al<sub>2</sub>O<sub>3</sub>(1010) FIC (222) 100 50 0 35 55 65 45 75 85 95 Reflection angle, 20 b) Al<sub>2</sub>O<sub>3</sub>(104) 250 Al,O,(116 200 Intensity, Imp/s 150 Al<sub>2</sub>O<sub>3</sub>(024) O.(1010) N.O.(214) C,N)(311 o(300 N(220) 100 50 0 35 45 55 65 75 85 95 Reflection angle, 20

Fig. 3. X-ray diffraction patterns of the of the a) oxide tool ceramics, b) oxide tool ceramics with the TiN+Al<sub>2</sub>O<sub>3</sub> coating

Table 1.

a)

	Results of the r	nechanical p	oroperties t	tests of the	analysed	materials
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Examined material	Thickness, μm	Microhardness, GPa	Roughness, R <sub>a</sub>
Substrate Al <sub>2</sub> O <sub>3</sub> +TiC	-	19	0.17
TiN (PVD)	1	33	0.21
TiN+multiTiAlSi N+TiN (PVD)	2	40	0.27
TiN+Al <sub>2</sub> O <sub>3</sub> (CVD)	6	38	0.29



b)



Fig. 4. a) Fracture surface; b) topography of the surface of  $TiN+Al_2O_3$  coating deposited onto  $Al_2O_3+TiC$  ceramics

The microhardness tests carried out revealed that the uncoated  $Al_2O_3$ +TiC substrate has hardness equal to 19 HV<sub>0.07</sub> [GPa]. Deposition of the TiN+TiAlSiN+TiN coating onto the substrate causes the significant surface layer hardness increase within the range of 33-40 HV<sub>0.07</sub> [GPa]. The highest hardness of 40 HV<sub>0.07</sub> [GPa] is displayed by the TiN+multiTiAlSiN+TiN coating; whereas, the lowest hardness of 33 HV<sub>0.07</sub> [GPa] is displayed by the TiN coating.

Basing on roughness examinations it was found out that deposition of the TiN, TiN+multiTiAlSiN+TiN, and TiN+Al<sub>2</sub>O<sub>3</sub> coatings onto the Al<sub>2</sub>O<sub>3</sub>+TiC oxide tool ceramics results in roughness increase up to 0.21-0.29  $\mu$ m. Table 1 presents results of thickness measurements, microhardness tests, and roughness measurements of the analysed coatings put down onto the oxide tool ceramics, as well as of the microhardness and roughness of the substrate material.

It was found out, basing on the metallographic examinations of fractures made on the scanning electron microscope, that the TiN, TiN+multiTiAlSiN+TiN and TiN+Al<sub>2</sub>O<sub>3</sub> coatings were deposited uniformly onto the substrate from the Al<sub>2</sub>O<sub>3</sub>+TiC oxide tool ceramics.

All these deposited coatings are characterised by a compact structure with no visible pores and cracks and by tight adherence to the substrate material (Fig. 4). Figure 5 presents the images of the exemplary substrate material surface topography and of the analysed coatings, obtained using the AFM atomic force microscopy, which were the basis for determining their fractal dimension using the projective covering method and for determining their multifractal spectra [7].

Roughness measurements results of the analysed surfaces specified by parameter R and determined according to (18) are presented in Table 2. It was found out basing on the investigations that the surfaces of the  $Al_2O_3$ +TiC oxide tool ceramics - uncoated and coated with the TiN coating, are characteristic of a low value of this parameter being within a range of 0.01-0.04 µm, depending on the scanning range. Depositing the TiN+  $Al_2O_3$  coatings in the CVD process onto the analysed tool ceramics results the parameter R to increase to the value of 0.50-0.57 µm; whereas the TiN+multiTiAlSiN+TiN coatings obtained in the PVD process display roughness demonstrate roughness values from a broad range of 0.15-0.44, which probably suggests the significant inhomogeneity of surfaces of these coatings, resulting from their fabricating technology.

Table 2.

Values of parameter R, determined basing on the AFM measurements, depending on scanning range

	Scanning range, nm		
Examined material	1000	2000	5000
Substrate	0.02	0.03	0.04
TiN	0.01	0.03	0.04
TiN+multiTiAlSiN+TiN	0.15	0.18	0.44
TiN+Al <sub>2</sub> O <sub>3</sub>	0.57	0.50	0.54

Table 3.

Values of the fractal dimension Ds of the analysed surface, depending on scanning range

	Scanning range, nm			
Examined material	1000	2000	5000	
Substrata	$2.017 \pm$	$2.02 \pm$	$2.017 \pm$	
Substrate	0.003	0.003	0.003	
TiN	$2.002 \pm$	$2.002 \pm$	$2.010 \pm$	
1118	0.001	0.001	0.003	
TiN+multiTi A 1SiN+TiN	$2.288 \pm$	$2.128 \pm$	$2.097 \pm$	
	0.028	0.031	0.026	
	$2.074 \pm$	$2.074 \pm$	$2.063 \pm$	
$1 \text{IIN} + \text{Al}_2 \text{O}_3$	0.013	0.041	0.033	

The obtained roughness measurement results of the analysed surfaces defined by parameter R correlate with the roughness measurement results defined by parameter  $R_a$  and differences in the obtained values result from differences of the measurement range, measurement technique employed, and its accuracy.



Fig. 5. Image of surface topography a) oxide tool ceramics b) oxide tool ceramics with the  $TiN+Al_2O_3$  coating along with the bilogarithmic relationship of the approximated analysed surface from the mesh side size used to its determining (scanning range: 2  $\mu$ m)



Fig. 6. Spectra of the generalized fractal dimensions and their respective multifractal spectra of the analysed coatings and substrate material; scanning range:  $2 \ \mu m$ 

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Examined material	Scanning range, nm	$\alpha_{min}$	$f(\alpha_{min})$	$\alpha_{max}$	$f(\alpha_{max})$	Δα	$\Delta f$
	1000	1.87	0.15	2.01	1.87	0.14	-1.72
Substrate	2000	1.81	0.18	2.01	1.87	0.20	-1.69
=	5000	1.88	0.15	2.00	1.92	0.12	-1.77
TiN TiN+multiTiAlSiN+ TiN	1000	1.99	0.19	2.00	1.99	0.00	-1.80
	2000	1.92	0.22	2.01	1.98	0.09	-1.76
	5000	1.90	0.20	2.00	1.93	0.10	-1.73
	1000	1.60	0.17	2.26	0.78	0.66	-0.61
	2000	1.70	0.10	2.14	1.25	0.44	-1.15
	5000	1.62	0.07	2.12	1.36	0.50	-1.29
TiN+Al <sub>2</sub> O <sub>3</sub>	1000	1.44	0.01	2.21	1.28	0.77	-1.27
	2000	1.60	0.19	2.35	0.75	0.75	-0.56
	5000	1.64	0.24	2.15	1.27	0.50	-1.03

Table 4.					
Results of the	multifractal	analysis	of the	investigated	materials

It was found out basing on the investigations of Al<sub>2</sub>O<sub>3</sub>+TiC oxide tool ceramics - uncoated and coated with the TiN coating 1 µm thick, that it demonstrates the relatively low value of the fractal dimension (<2.02), regardless of the scanning range. A low value of this dimension for the TiN coated ceramics results probably from the fact that the TiN coating surface topography is determined by the shape of the substrate material in case of such coating thickness. Depositing the TiN+multiTiAlSiN+TiN and TiN+Al<sub>2</sub>O<sub>3</sub> coatings in the PVD and CVD processes onto the analysed substrate with the thickness values of 2 and 6 µm causes the fractal dimension value growth (Table 3). In case of the TiN+Al<sub>2</sub>O<sub>3</sub> coatings the fractal dimension value is in a narrow range of 2.063-2.074. It was found out in case of the TiN+multiTiAlSiN+TiN that the fractal dimension for the consecutive, smaller and smaller scanning ranges, keeps growing from the value of 2.097 to 2.228. This may be explained with the shape of the surface topography of the analysed TiN+multiTiAlSiN+TiN coatings - observations carried out reveal occurrence of the clearly visible unevenness and irregularities with small amplitude, which are neglected at the small measurement accuracy; however their effect grows systematically when the scanning range gets smaller and measurement accuracy grows. The observed tendency may be also incidental and result for the inhomogeneity of the analysed test piece surface only. One should stress that for all examined test pieces the bilogarithmic diagrams are linear, which confirms the fractal nature of the analysed surfaces. Only in case of the TiN+Al2O3 coating data points representing the initial partitions are distributed in a non-linear way (or: do not display the linear arrangement) which results from the high roughness of these surfaces.

Table 4 presents the multifractal analysis results, whereas Fig. 6 presents spectra of the fractal dimensions and their equivalent multifractal spectra. 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. A more narrow range of the multifractal spectrum refers to surfaces characteristic

of a lower parameter R value (substrate and TiN coating) for all scanning ranges. These materials also have the highest (in terms of their absolute value) negative difference of the multifractal spectrum arms heights. The TiN+Al<sub>2</sub>O<sub>3</sub> coating described by the highest value of the surface fractal dimension and roughness R has the broadest spectrum arms heights. The multifractal spectrum of the TiN+multiTiAlSiN+TiN coating surface is described by the intermediate values.

## 4. Conclusions

The paper presents investigation results obtained from the AFM microscope of the Al<sub>2</sub>O<sub>3</sub>+TiC tool ceramics uncoated and coated with the TiN and TiN+multiTiAlSiN+TiN coatings deposited in the PVD process, as well as with the TiN+Al<sub>2</sub>O<sub>3</sub> coating obtained in the CVD process. Values of the surface fractal dimension and the multifractal spectra were determined using the projective covering method. This value is independent from the scanning range, except for the TiN+multiTiAlSiN+TiN coating, which confirms their fractal nature. Variability of the fractal dimension value depending on scale for the TiN+multiTiAlSiN+TiN may be caused the presence of clear, yet very fine unevenness and/or inhomogeneity of the coating, which is suggested by analysis of parameter R 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 and of the substrate material from the  $Al_2O_3$ +TiC oxide tool ceramics.

The fractal dimension has a relatively clear interpretation - it is a measure of irregularity and degree of complexity of the surface shape, whereas interpretation of the multifractal spectrum is not unequivocal. Investigation or relationship between parameters describing the multifractal spectrum and physical properties of the examined materials calls for further analyses.

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