

# FRACTAL GEOMETRY CHARACTERIZATION OF FRACTURE PROFILES OF POLYMERIC MATERIALS\*

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The concept of fractals was used in the analysis of the fracture surfaces of two different polymeric materials. Polymer networks obtained from three popular dental dimethacrylate monomers: Bis-GMA, TEGDMA and UDMA as well as two copolymers of these monomers were analysed. Dense polymer membranes with dispersed magnetic powder (magnetic membranes) for air separation were also investigated. In both cases, profiles of fractures were described by a modified fractal dimension. It is based on scaling the length of a profile with the size of a measuring step. The modified fractal dimension is considered a diagnostic tool for structure-morphology analysis of fracture path and roughness parameter. A quantitative description of fracture surface is an important link in the investigation of the properties of materials and mechanisms of their decohesion.

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## 1. Introduction

The world in which we live has various abstract equivalents. Euclidean space plays a special role among them as the first estimation but also for historical reasons. One of its features is the possibility of measuring distance. While examining features of distance in Euclidean space it was noticed that some of them are the consequence of others. They are intuitively obvious

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and it is possible to formulate them in a particularly simple way. Many theorems of Euclidean geometry can be proven only on the basis of these fundamental features of the notion of distance. In such a case, a more general notion of space has been introduced. The space where the distance of a pair of points is a primary notion and the properties of distance in Euclidean space are considered to be axioms. This leads to metric spaces. They are a starting point for further generalization of the concept of spaces. We have the possibility of providing a more precise description of the world around us. Quantitative characterization of the set is required to define appropriate measures. One of such measurements is the fractal dimension. Since Mandelbrot's publications [1, 2], fractal geometry has been extensively applied to characterize roughness of fracture surfaces and correlate it with mechanical properties [3–5]. The concept of fractals was used in the analysis of the fracture surfaces profiles to determine whether fracture surfaces are fractal in their nature, and whether the fractal character can be related to either mechanical properties or the microstructure of the specimens. Many issues in materials sciences like percolation [6], diffusion [7] or roughness [8] have been studied through fractal analyses.

## 2. Fractal dimension

A fractal dimension is an index used to characterize self-similarity sets by means of quantifying their complexity as a ratio of the change in detail in relation to the change in scale. Mandelbrot's paper [9], where he cited previous work by L. Fry Richardson, described the counter-intuitive notion that the length of the measured coastline changes with the length of the measuring stick used. In terms of that notion, the fractal dimension of the coastline quantifies how the number of scaled measuring sticks required to measure the coastline changes with the scale applied to the stick. There are several formal mathematical definitions of fractal dimension that are built on this basic concept of the change in detail connected with the change in scale.

Fractal analysis based on fractal dimension  $D_F$  and general fractal dimension  $D_q$  is a useful and popular tool for quantifying the surface morphology of various materials [10–12]. The fracture topography of a polymer may also be analysed by observing its surface shape, as a fracture profile. A profile line can be extracted as a line through an area and expressed as a single numeric fractal parameter of the modified fractal dimension ( $D_\beta$ ).

The profile line of the fracture was characterized by means of the modified fractal dimension ( $D_\beta$ ) [5]. The profile lines were subjected to the analysis, in which the length of each fracture profile was measured with a varying step  $\eta$  (the length of the step in each following operation was reduced by half), see Fig. 1. The power relationship between the length of profile ( $L$ ),

the size of measuring step ( $\eta$ ), and the development of the line profile ( $R_L$ ) was used for this purpose

$$R_{L(\eta)} = R_{L(0)} \eta^{-(D_\beta-1)}. \quad (1)$$

In logarithmic scale, the following equation was used for calculating  $D_\beta$

$$\log R_{L(\eta)} = \log R_{L(0)} - (D_\beta - 1) \log \eta. \quad (2)$$

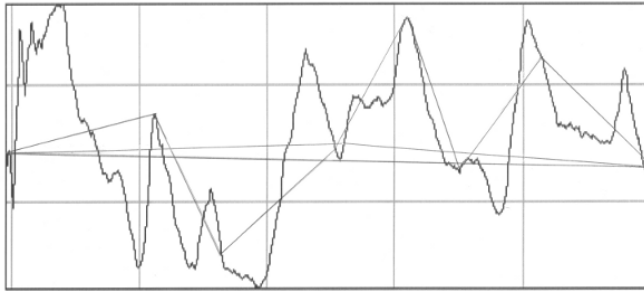


Fig. 1. The modified fractal dimension ( $D_\beta$ ) method.

### 3. Materials

The first object of the research was dense polymer membranes with dispersed magnetic powder, called magnetic membranes [13]. They are used for air separation. Magnetic membranes were made by pouring the 3 ethylcellulose (EC) solution with a dispersed magnetic neodymium (Nd) or ferrite (Fe) powder (of appropriate granulation 20–32  $\mu\text{m}$  or 32–50  $\mu\text{m}$ ), into a Petri dish and then evaporating the casting solvents in external field of a coil (a stable magnetic field with a range of induction 0–40 T) for 24 h. The obtained membranes were removed from a Petri dish (with some distilled water) and dried in 40°C. For membranes with dispersed magnetic powder, the permeation measurements were carried out before and after magnetization in a field magnet. The magnetic induction of this field was approximately of 2.5 T. Figure 2 shows the structure of the membrane. Surfaces of membranes were observed by optical microscopy (Olympus BX51M). This picture shows images of membranes with different kinds and granulation of magnetic powder. The texture created by magnetic powder was observed with 40 magnification and analysed by fractal formalism.

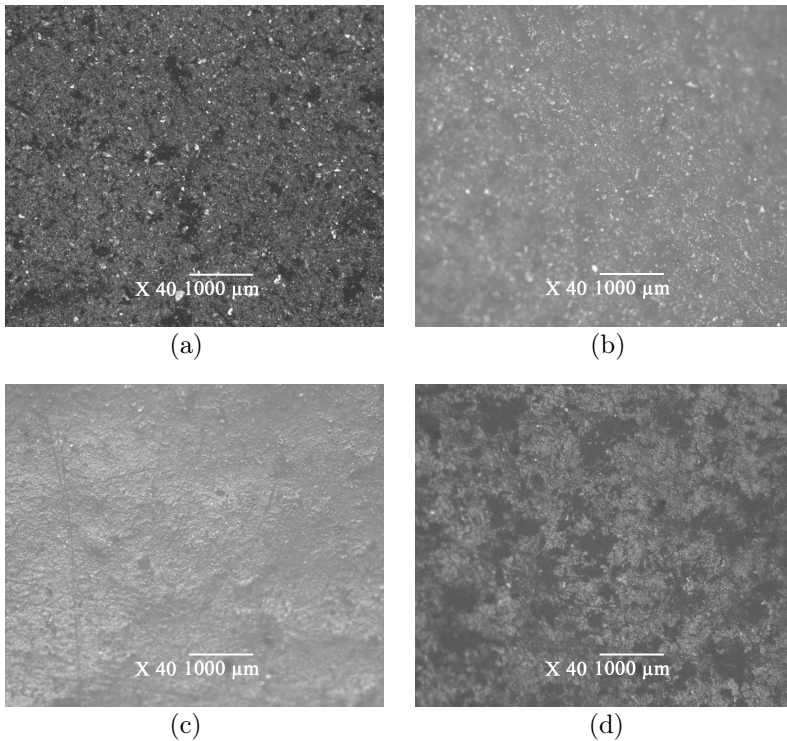


Fig. 2. The texture created by magnetic powder was observed with 40 magnification. (a) EC with neodymium powder of granulation 20–32 micrometers; (b) EC with neodymium powder of granulation 32–50 micrometers; (c) EC with ferrite powder of granulation 20–32 micrometers; (d) EC with ferrite powder of granulation 32–50 micrometers.

As the second object of research, five series of polymer networks were analysed: three homopolymers of Bis-GMA, TEGDMA and UDMA, and two copolymers of these monomers [14], see Fig. 3. The polymers were obtained by visible light photopolymerization, with the addition of 0.4 wt% of CQ (camphorquinone) and 1 wt% of DMAEMA (N,Ndimethylaminoethyl methacrylate) being the most common photoinitiator system used in photoactivated polymer-based dental materials [14]. The amount of monomers was selected to fill the Petri dishes of 12 cm in diameter and obtain the thickness of polymer samples equal to 4 mm. The samples were irradiated with a mercury lamp (FAMED-1, model L-6/58, Poland, power 375 W) set at a distance of 15 cm for 30 min.

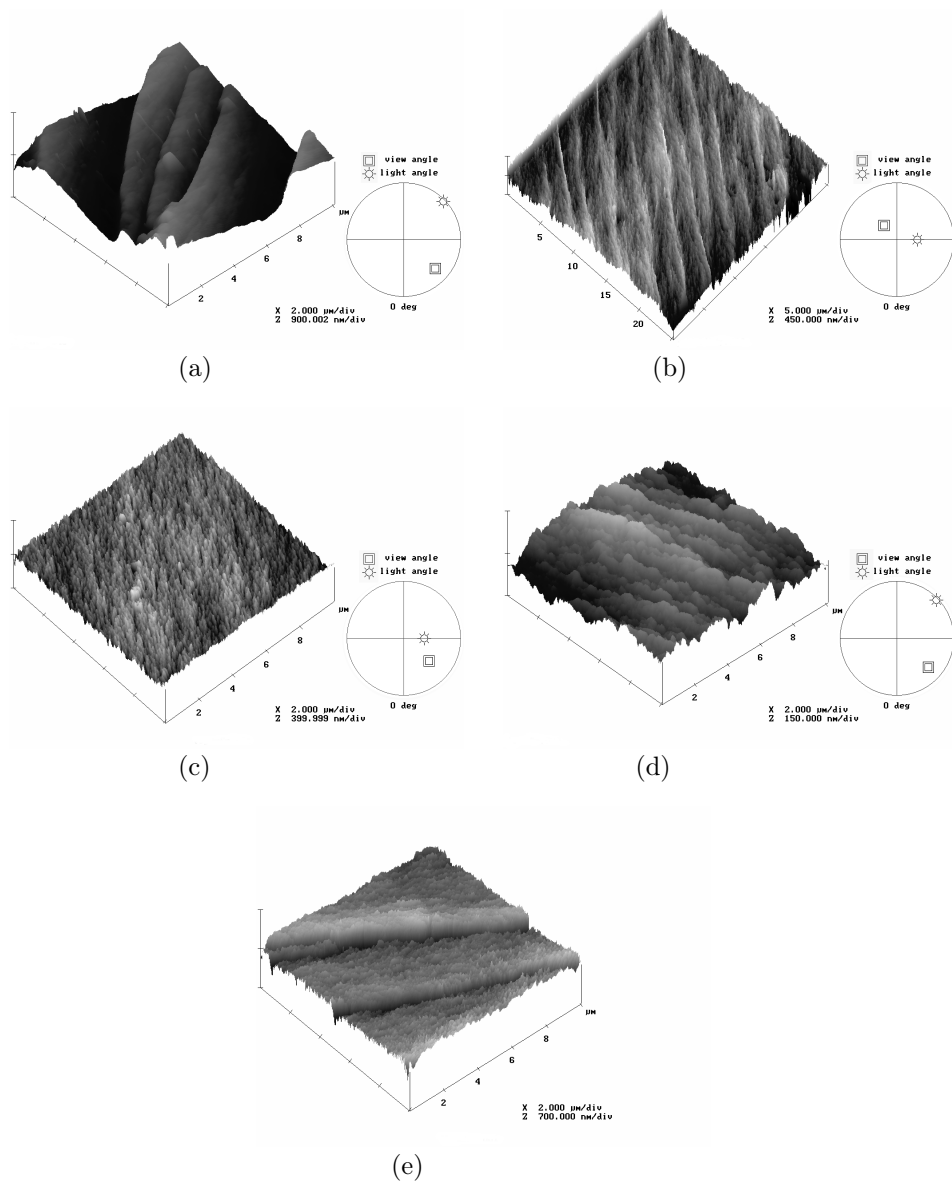


Fig. 3. Atomic force microscopy images of fractured surfaces. (a) 2,2-bis-[4-(2-hydroxy-3-methacroyloxypropoxy)phenyl]propane (Bis-GMA); (b) urethane-dimethacrylates (UDMA); (c) triethylene glycol dimethacrylate (TEGDMA); (d) GDMA-TEGDMA; (E) TGDMA-TEGDMA-UDMA.

#### 4. Results and discussion

Figure 4 presents sample profile lines of the fracture of the analysed magnetic membranes.

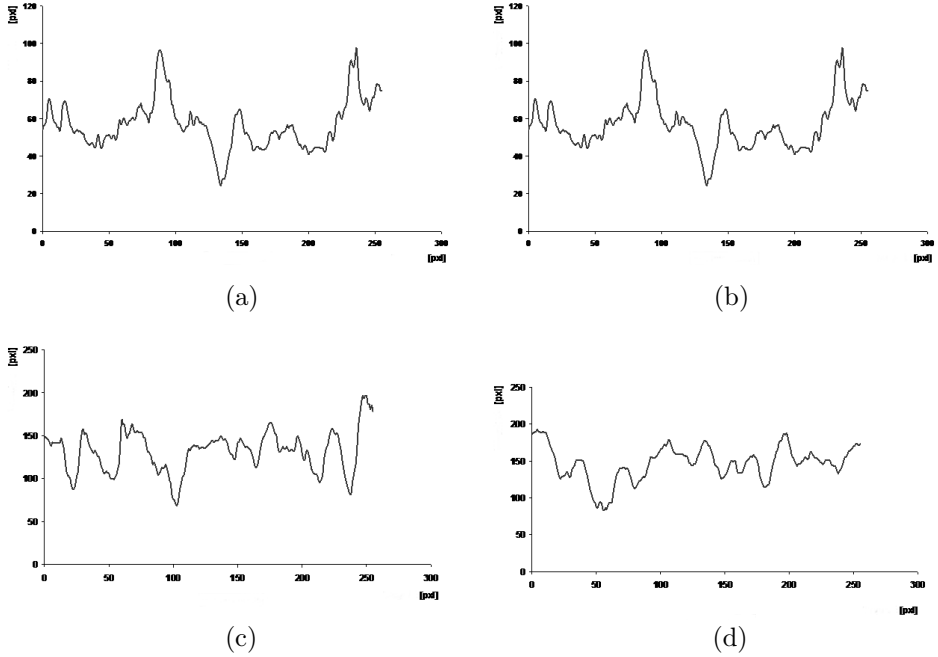


Fig. 4. The profile lines of the fracture of magnetic membranes. (a) EC with neodymium powder of granulation 20–32  $\mu\text{m}$ ; (b) EC with neodymium powder of granulation 32–50  $\mu\text{m}$ ; (c) EC with ferrite powder of granulation 20–32  $\mu\text{m}$ ; (d) EC with ferrite powder of granulation 32–50  $\mu\text{m}$ .

Table I provides results of fractographic and fractal analysis for magnetic membranes.

TABLE I

Fractal dimension and fractographic parameters for membranes.

	$D_\beta$	$R_p$	$R_z$
EC + Fe (20–32 $\mu\text{m}$ )	$1.52 \pm 0.01$	$71 \pm 1$	$140.09 \pm 0.25$
EC + Fe (32–50 $\mu\text{m}$ )	$1.42 \pm 0.01$	$98 \pm 1$	$156.96 \pm 0.25$
EC + Nd (20–32 $\mu\text{m}$ )	$1.44 \pm 0.01$	$110 \pm 1$	$177.54 \pm 0.25$
EC + Nd (32–50 $\mu\text{m}$ )	$1.36 \pm 0.01$	$118 \pm 1$	$193.04 \pm 0.25$

As the basic fractographic parameters, we selected shape factor  $R_p$  and surface roughness  $R_z$ . The shape factor is calculated taking into account the slenderness of the peaks and can be used to determine the degree of stress concentration. The surface roughness is a measure of the texture of a surface. It is quantified by the vertical deviations of a real surface from its ideal form. In both cases, the increase in the fractal dimension reduces fractographic values.

Figure 5 presents sample profile lines of the fracture of the analysed dental materials.

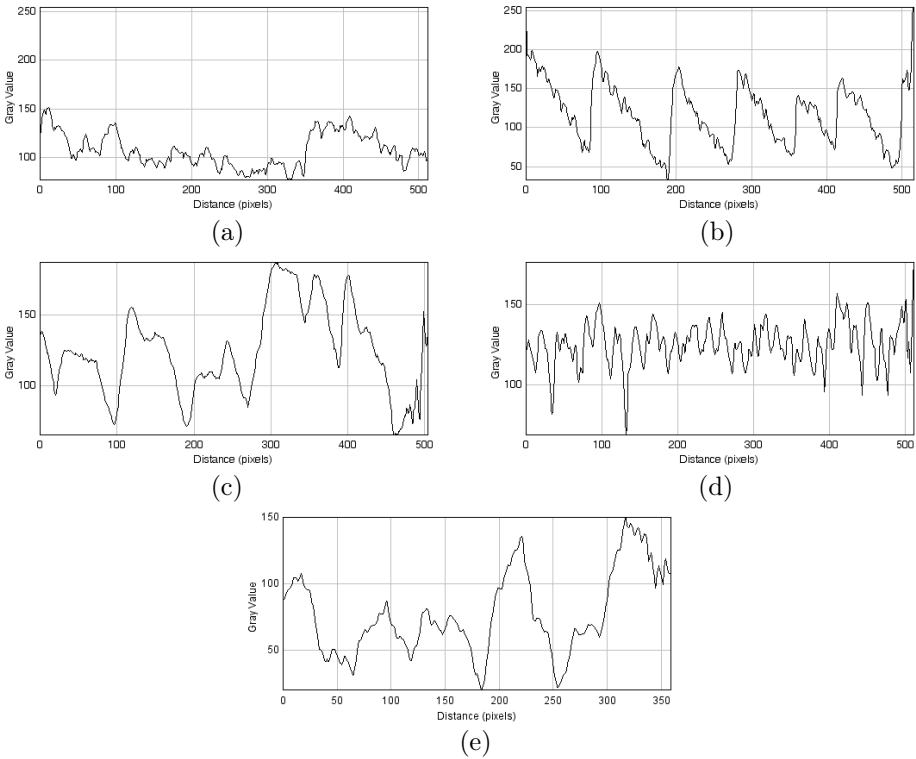


Fig. 5. The profile lines of the fracture of dental materials. (a) 2,2-bis-[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl]propane (Bis-GMA); (b) urethane-dimethacrylates (UDMA); (c) triethylene glycol dimethacrylate (TEGDMA); (d) GDMA-TEGDMA; (E) TGDMA-TEGDMA-UDMA.

One of the important properties of dental materials is hardness. It has been determined for the analysed materials. The ball indentation hardness (HB) was determined in accordance with ISO 2039-1:2004, on test specimens with a 4 mm height and a 12 mm diameter, using VEB Werkstoffprüfmaschi-

nen apparatus. HB was calculated according to

$$HB = \frac{F_r}{\pi d h_r}, \quad (3)$$

$$F_r = F_m \frac{0.21}{h - h_r + 0.21}, \quad (4)$$

where  $F_r$  is the reduced test load,  $h_r$  — the reduced depth of impression ( $h_r = 0.25$  mm),  $d$  — the diameter of the ball indenter ( $d = 5$  mm),  $F_m$  — the test load on the indenter ( $F_m = 490$  N),  $h$  — the depth of impression.

The values of  $D_\beta$  were found to be in the range of 1.11–1.88, thus confirming the fractal character of surface profiles [15]. Their complexity increased in the following order: poly(Bis-GMA), poly(Bis-GMA-co-TEGDMA), poly(UDMA), poly(TEGDMA), poly(Bis-GMA-co-TEGDMA-co-UDMA).

The result does not seem to depend on the presence of hydrogen bonds in the system, but rather the elasticity of the monomer molecule. The monomer molecule of a greater stiffness results in the regular line of profile for the polymer, approaching a straight line. The averaged results showed that, amongst all investigated profiles, those obtained for fractured poly(Bis-GMA) were the most regular. This can be attributed to the change in the fracture mode depending on the mass distribution. The polymer hardness increased as the  $D_\beta$  increased. The correlation between  $D$  and hardness was found and  $R^2 = 0.897$  (Fig. 6). It is calculated for images obtained at  $5 \mu\text{m} \times 5 \mu\text{m}$  scanning area. The investigated polymer networks were found to possess the fractal characteristics. The findings clearly showed a highly complex morphology of fractal surfaces, which results from molecular organization formed through the polymerization.

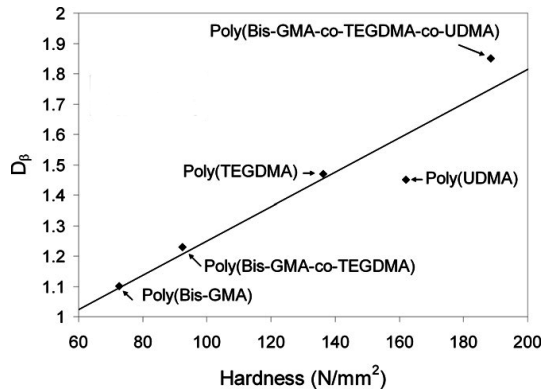


Fig. 6. The relationship between  $D_\beta$  (for  $5 \mu\text{m} \times 5 \mu\text{m}$  scanning area) and hardness of dimethacrylate polymer networks.



## 5. Concluding remarks

The morphology of fracture surfaces is strongly dependent on the material, its fracture mechanism and the scale of observation. Fractal geometry is a significant tool for understanding the properties of surface of polymeric materials and the fracture process mechanism. The self-similarity of the observed surfaces proves that mechanisms involved in the fracture process are scale independent. Among many methods for determining the fractal geometry, the analysis of the fracture surface profile lines has been selected in this work. It has been found out that it is possible to correlate fractographic and fractal parameters.

The investigated polymer networks were found to possess the fractal characteristics. Hardness of these materials had a correlation with  $D_\beta$  and increased with the increase of  $D_\beta$  value. This can be attributed to the change in the fracture mode depending on the mass distribution. Moreover, it can be observed even in one dimensional analysis. However, the role of fracture morphology and fractal parameters and their relations with the material properties are still not well understood and this issue is worth further research.

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