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On the characterization of polymer concrete fracture surface

Lech Czarnecki [a], Andrzej Garbacz [a,*], Joanna Kurach [b]

[a] Warsaw University of Technology, Institute of Technology and Organization of Building Production, Al. Armii Ludowej 16, 00-637 Warsaw, Poland
[b] Building Research Institute, Filtrowa 1, 00-950 Warsaw, Poland

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Abstract

Fracture surface geometry is often related to material toughness. This approach requires a quantitative description of the fracture surface. The aim of this work was to investigate scale effects on the geometrical features of the fracture surface of polymer concrete (cementless concrete) with different microstructures. Polymer concrete (PC) is a concrete-like composite, in which a resin binder substitutes the cement binder. The fracture surfaces were characterized at different magnifications by: fractal dimension, surface roughness ratio and profile roughness ratio. Relationships between parameters describing the geometry of PC fracture surface were analyzed. The main conclusion is that the geometry of the PC fracture surface depends on the scale of observation. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Polymer concrete; Surface and profile roughness ratio; Fractal dimension; Scale effect

1. Introduction

Polymer concrete (PC) is a concrete-like composite, in which a resin binder, e.g. epoxy resin, substitutes the cement binder [1]. Important applications include repair and anti-corrosion protection of concrete structures (including industrial floors) as well as pre-cast elements, such as manholes, pipes and chemically resistant vessels (e.g. electrolytic cells) [2,3]. Improved mechanical strength and chemical resistance are basic advantages of PC in comparison to ordinary portland cement concrete. According to the general classification of composites [4], both the portland cement concrete and PC can be treated as a particulate composite with two main constituents: a matrix and dispersed particles of strengthening phases. The aggregate in concrete-like composites is mainly added to control the crack propagation and reduce material cost [5]. Fracture surface geometry (roughness and tortuosity) is often related to material toughness [6–8]. This approach requires a quantitative description of the fracture surface. Value of parameters characterizing the PC fracture surface depends not only on the composition and adhesion between binder and aggregate, but also on the scale of observation.

The aim of this work was to investigate scale effects on the geometrical features of the fracture surface of epoxy concrete with different microstructures. The fracture surfaces were characterized at different magnifications by: fractal dimension, surface roughness ratio and profile roughness ratio. Relationships between parameters describing the geometry of PC fracture were analyzed. The main conclusion is that the geometry of the PC fracture surface depends on the scale of observation.

2. The fracture surface characterization

2.1. Parameters of fracture surface characterization

Quantitative fractography, which deals with the description of fracture surfaces, is more advanced in the case of metals and ceramics than in concrete-like composites. However, geometrical and stereological parameters are also of significant importance in concrete-like composites [6–8]. Many stereological parameters are considered useful for characterization of fracture surfaces [9]. The three parameters commonly used are (Fig. 1):

* Corresponding author. Tel.: +48-22-825-76-37; fax: +48-22-825-74-15.

E-mail address: a.garbacz@il.pw.edu.pl (A. Garbacz).

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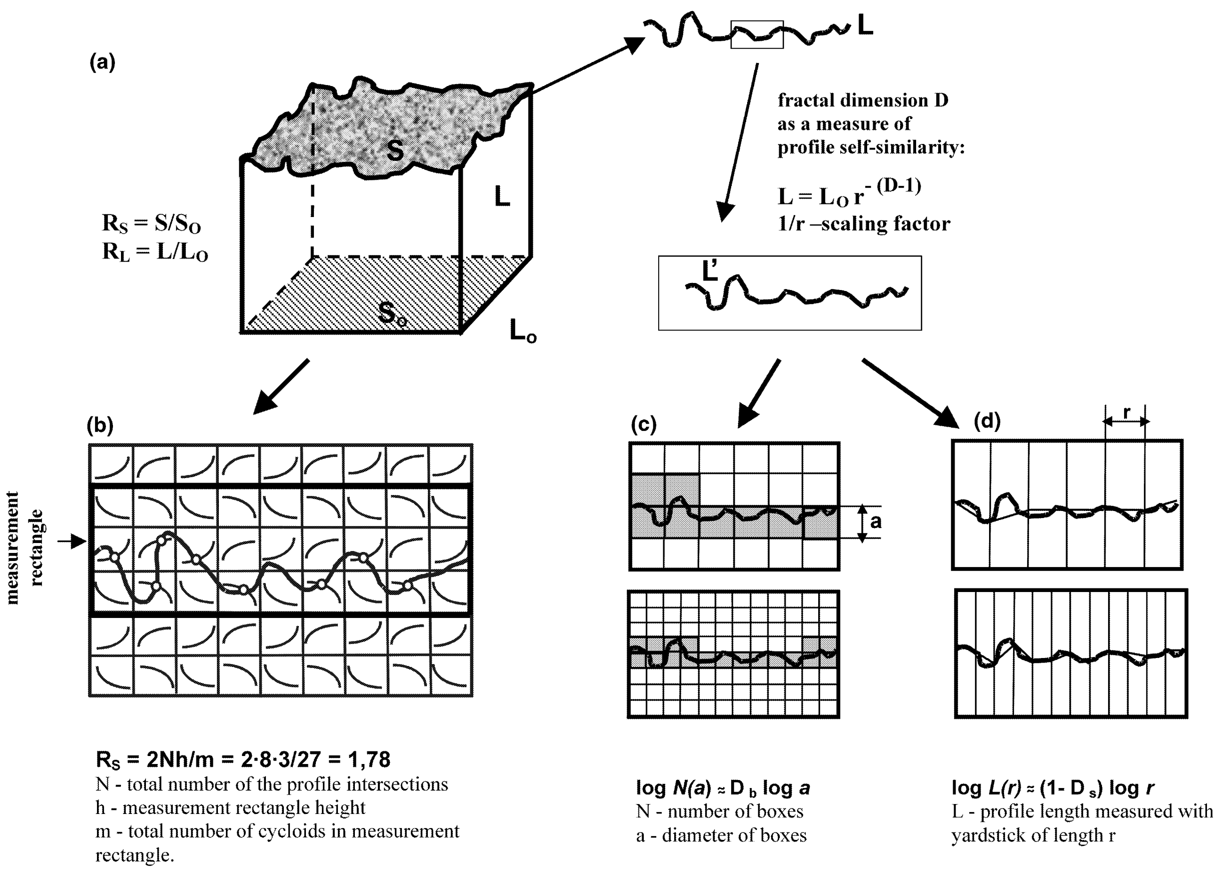

Fig. 1. Example of fracture surface and parameters characterizing its geometry (a) and sketch for the determination of the surface roughness ratio (c) and fractal dimension with the box-counting method (c) and divider method (d) S, S_O – true and projected surface area, respectively, L, L_O – true and projected surface profile length, respectively.

- profile (linear) roughness ratio, R_L: length of the profile line, L, divided by the projected length of the profile line, L_O:

$$R_L = L/L_O \tag{1}$$

- surface roughness ratio, R_S: true fracture surface area, S, divided by the apparent projected area, S_O:

$$R_S = S/S_O \tag{2}$$

- fractal dimension, D, which has been introduced to materials science by Mandelbrot as a characteristic of rough boundaries of objects. The basic requirement for the fractal boundary is that some structural feature or unit is sequentially repeated at different levels (comp. Fig. 1). It implies that, from a statistical point of view, similar morphology can be observed in a wide range of magnifications of fracture surface, and a measure of this self-similarity is a fractal dimension.

The geometry of the fracture surface is related to the scale of the observation. It implies that the self-similarity of the fracture surface may not be extended over all ranges of magnification. This is important in the case of concrete-like composites where the size of aggregate grains (including microfiller) ranges, practically, from 0.01 to 32 mm.

2.2. Measurement of R_S and R_L

It has been assumed that, for constant sample geometry, the R_S value is related to the energy being dissipated during material decohesion [9]. However, due to technical difficulties with R_S measurements, a profile of perpendicular sections to the fracture surface is frequently examined and the profile roughness ratio, R_L, is calculated from Eq. (1). The estimation of R_L from the profile image is easy for automation with profilometer and image analysis. In the past, much effort has been spent on developing relationships between R_S and R_L [10]. Several linear relationships have been proposed, namely:

- Chermant and Coster [11]:

$$R_S \approx 1.75R_L - 0.75, \tag{3}$$

- Wright and Karlsson [12]:

$$R_S \approx 1.57R_L - 0.57, \tag{4}$$

Table 1
Composition of epoxy concretes tested in this study

Concrete type	PC(I)	PC(II)	PC(III)	PC(IV)	PC(V)	PC(VI)	PC(VII)
Composition (by weight)	Designed flexural strength (MPa)						
	10	10	15	15	20	20	20
Aggregate to binder ratio, A/B	14	14	11	11	7	7	9
Resin binder content (%)	(6.7)	(6.7)	(8.3)	(8.3)	(12.5)	(12.5)	(10)
Sand fraction of aggregate, S/A	0.60	0.60	0.42	0.42	0.36	0.36	0.41
Microfiller fraction of aggregate, M/A	0.04	0.12	0.04	0.12	0.04	0.12	0.18
Average flexural strength (MPa)	12.2	11.7	13.1	17.5	19.5	23.7	20.9
SD (MPa)	(0.5)	(0.8)	(1.4)	(1.9)	(1.9)	(1.9)	(2.0)
Average compressive strength (MPa)	27.6	25.2	38.4	52.6	81.0	86.4	81.5
SD (MPa)	(2.3)	(0.8)	(1.4)	(4.1)	(3.2)	(1.5)	(8.0)

- Underwood [13]:

$$R_S \approx 1.25R_L - 0.27, \quad (5)$$

- Gokhale and Underwood [14]:

$$R_S \approx 1.16R_L. \quad (6)$$

Recent development of stereological methods makes it possible to estimate R_S from fracture profile studies without simplifying assumptions, about the relationship between R_L and R_S. The surface roughness ratio, R_S can be effectively estimated using the method of vertical sections [15,16]. In this method an arbitrary axis is chosen and the examined material specimen is cut parallel to this axis. The sections should be randomly placed and randomly oriented with respect to the specimen geometry (e.g. perpendicular direction to the mean fracture surface), within the restriction of being parallel to the axis. It has been shown that three sets of sections with their normals making of an angle of 120° are enough for fracture surface characterization and R_S estimation [17]. Wojnar (1990) has proposed the procedure of the R_S measurement based on counting the intersection points of the fracture profile with a special grid of cycloids (comp. Fig. 1(b)). The use of cycloids enables to relate the fracture area to the fracture profile in a direct way and this estimation is independent of magnification. The R_S value is calculated from the formula:

$$R_S = 2Nh/m, \quad (7)$$

where N is the total number of the profile intersections with cycloids, h the measurement rectangle height expressed as a multiple of a cycloid length, m the total number of cycloids contained in the measurement rectangle.

3. Measurement of fractal dimension

There are many definitions and different techniques that can be used to estimate the fractal dimension of a fracture surface or profile. The details are given elsewhere (e.g. [6–8]). Although they are all equivalent in the continuous domain, they can differ when the profile is digitized for image analysis [8]. A "box-counting" method and a "divider method" are commonly used for testing the fracture surface of concrete-like composites.

The box-counting method (comp, Fig. 1(c)) consists of generating a square grid of given linear dimension and determining the number of boxes, N_i with dimension a_i, needed to cover the entire profile. The procedure is repeated with progressively smaller box sizes. The fractal dimension is obtained from the slope $\alpha(D_b = -\alpha)$ of the best-fitting line of the bilogarithmic data: $\log(N_i)$ versus log (N_i) for different grid dimensions a_i.

The divider method, also referred to as the "compass" or the "yardstick" method, consists of choosing subsequently smaller yardstick length, r_i, and measuring with this yardstick a total length, L_i, of the entire profile (comp. Fig. 1(d)). The fractal dimension is related to the slope of the log–log plot of L_i versus the measurements step size, r_i. The examined profile is considered a fractal object over all scale of observation, when the result

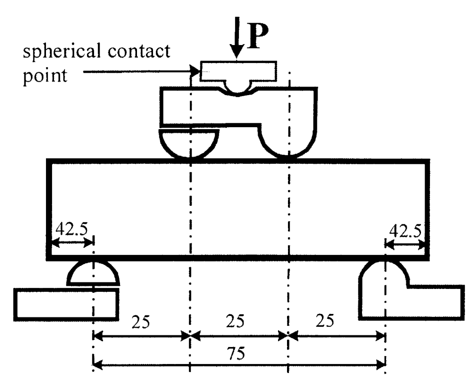

Fig. 2. Sketch of bending test by third-point loading (acc. to the RILEM TC113 procedure [19]).

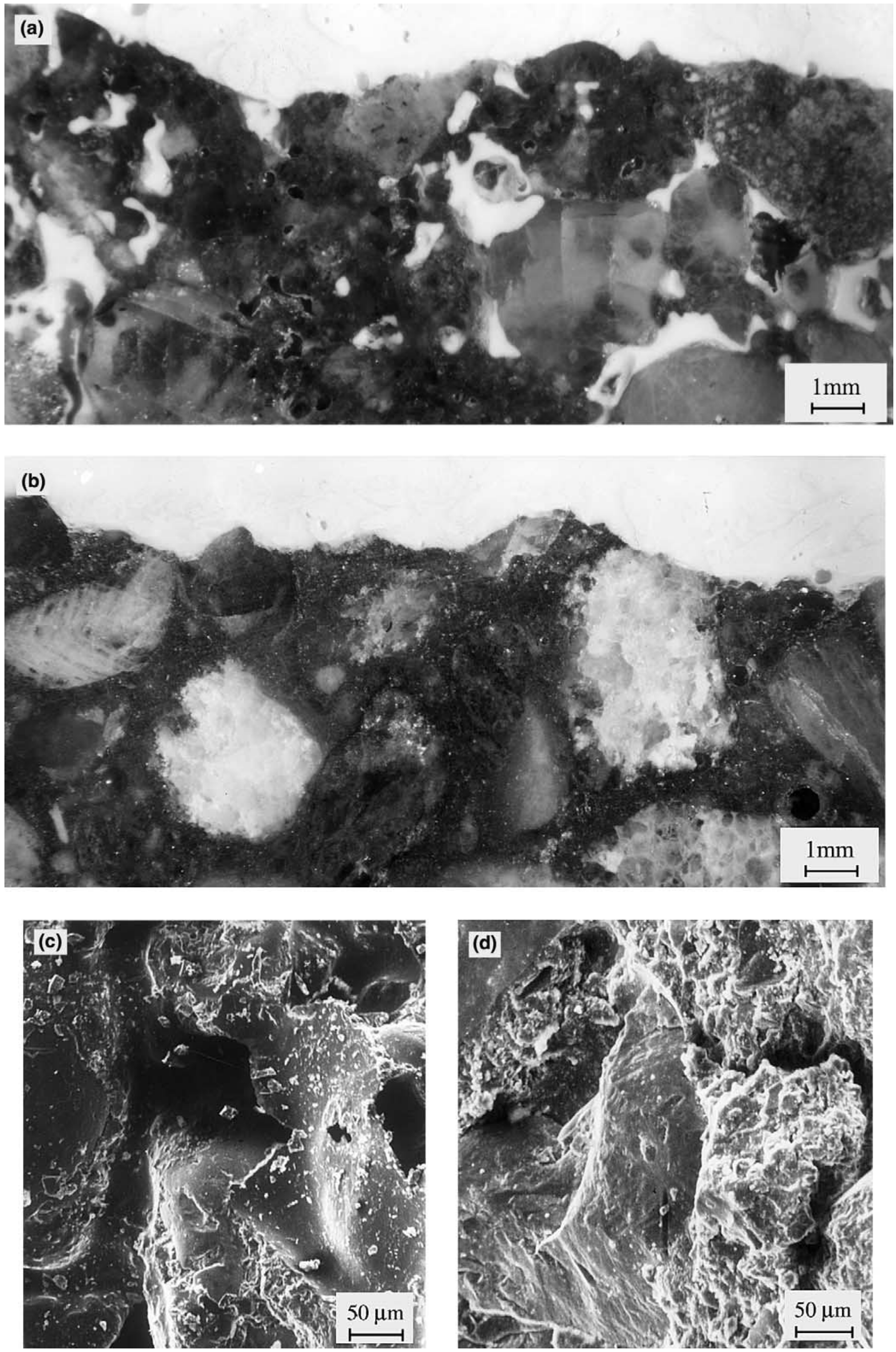

Fig. 3. Example of fracture surface profile at magnification 10× for epoxy concretes: (a) PC(II) – $A/B = 14,\ S/A = 0.6,\ M/A = 0,12$ and (b) PC(VI) – $A/B = 7,\ S/A = 0.6,\ M/A = 0.12$ and (b) PC(VI) – $A/B = 7,\ S/A = 0.36,\ M/A = 0.12$; (b)–(d) fracture surface at magnification 160× (SEM) for PC(II) and PC(VI) concrete, respectively.

obtained from plot: $\log(L_i) - \log(r_i)$ is approximately a straight line with a negative slope: $\alpha = 1 - D$. The total profile length, L_i is often exchanged by its relative measure: R_L or R_S. In this case the fractal dimension is related to the slope bilogarithmic data: $\log(R_L) - \log(r)$ or $\log(R_S) - \log(r)$, respectively.

Table 2
Observed geometrical properties of fracture surface of the epoxy concretes tested

Concrete type	PC(I)	PC(II)	PC(III)	PC(IV)	PC(V)	PC(VI)	PC(VII)
Profile roughness ratio, R_L							
At magn.							
10 ×	1816	1915	1891	1681	1457	1390	1443
25 ×	1737	1766	1758	1650	1427	1365	1385
63 ×	1644	1573	1601	1569	1440	1359	1419
160 ×	1636	1573	1633	1638	1528	1563	1457
400 ×	1543	1487	1608	1610	1488	1528	1548
Surface roughness ratio, R_S							
At magn.							
10 ×	2213	2386	2324	2011	1607	1708	1660
25 ×	2101	2173	2144	1972	1571	1665	1598
63 ×	1985	1903	1927	1869	1559	1685	1654
160 ×	1949	1889	1984	1954	1851	1819	1712
400 ×	1834	1743	1924	1942	1815	1760	1819
Fractal dimension, D_b							
At magn.							
10 ×	1063	1060	1070	1070	1078	1057	1060
25 ×	1034	1034	1036	1048	1031	1034	1035
63 ×	1028	1025	1022	1023	1023	1026	1020
160 ×	1024	1021	1025	1031	1022	1029	1022
400 ×	1023	1026	1023	1026	1027	1025	1033

4. Experimental

4.1. Material

Commercial epoxy resin was used as the binder. Natural quartz aggregate (fractions: 0.1/0.5, 1/2, 2.5/4 mm) was used as the coarse aggregate and a microsilica as the microfiller. To design a composition of the tested epoxy concretes the material model of epoxy concrete and material optimization approach was used [18]. Seven types of epoxy concrete (Table 1) were obtained for three values of target flexural strength: 10 MPa for PC(I) and PC(II), 15 MPa for PC(III) and PC(IV) and 20 MPa for PC(V)–PC(VII). They differed in the total aggregate to binder ratio (by weight), A/B, sand to aggregate ratio, S/A, and microfiller to aggregate ratio, M/A. The composition of PC(V) series was selected to achieve a value of flexural strength similar to that of PC(V) and PC(VI), but with the highest possible M/A value for workability of resin mix. The standard unnotched beam-samples ($40 \times 40 \times 160$ mm^3) were subjected to bending test (see Table 1) by the third-point loading following the RILEM TC113 procedure [19] after 14 days of hardening. The bending test load set-up is shown in Fig. 2. Additionally, compressive strength for all tested types of PC was determined.

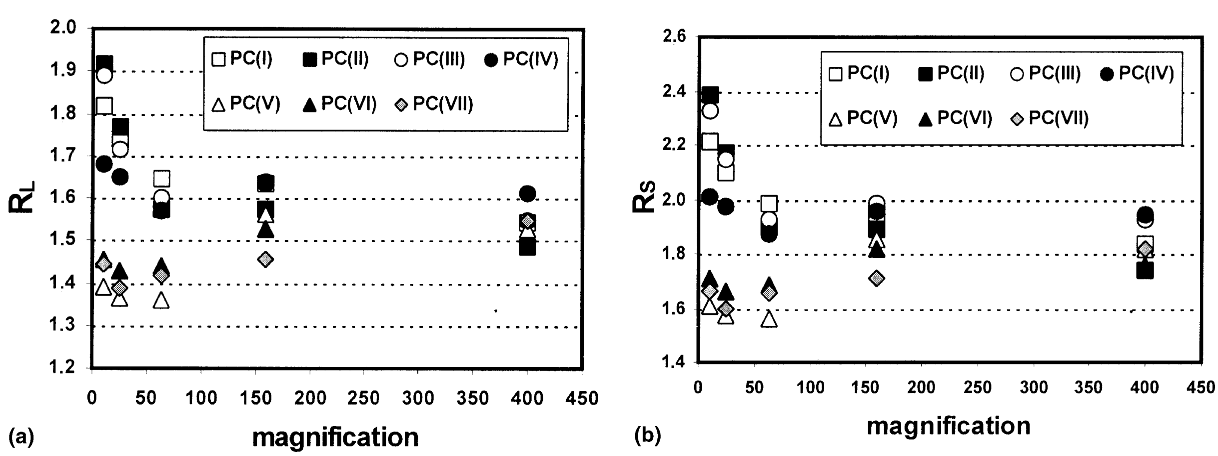

Fig. 4. Profile roughness R_L (a) and surface roughness ratio R_S (b) of epoxy concretes at different magnifications.

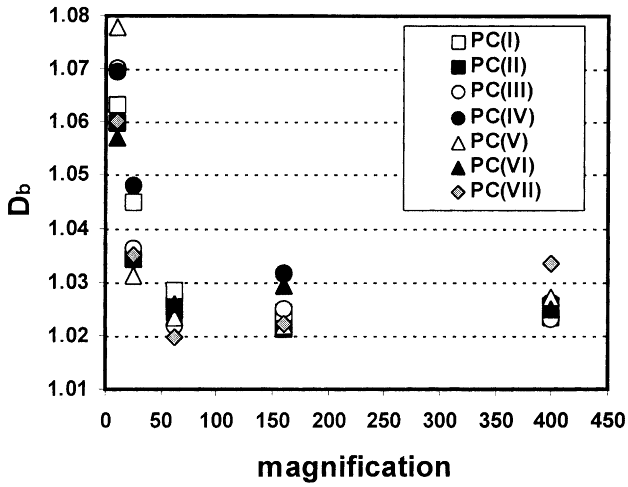

Fig. 5. Box-counting fractal dimension, D_b of epoxy concretes at different magnifications.

4.2. Method

The fracture surface of each epoxy concrete type, created during bending test, was examined at magnifications of: 10×, 25×, 63×, 160× and 400×, respectively. The profiles of fracture surfaces (Fig. 3) were obtained according to the method of vertical section. In this work, the fractured samples were cut along sections parallel to the sample axis. The fracture surface was covered with an epoxy coating pigmented with titanium white and perpendicular sections were polished for a better contrast of the profile line. The profile line image was obtained with a optical microscope. The entire profile line – 40 mm was examined at each magnification. All profiles were digitized with a resolution of 512 × 512 pixels and subjected to computer image analysis. The thinning procedure was applied to achieve the profile line with unit width of 1 pixel.

In the case of the R_L value, the total profile length was measured by a direct counting of pixels. The length of projected profile line, L_O, in pixels was the same ($L_O = 480$ pixels) at each magnification. The profile roughness ratio, R_L, was calculated from Eq. (1). The measurement of the R_S value was based on counting the intersection points of the digitized fracture profile with a grid of cycloids. The R_S value was calculated from Eq. (7). In this study, the box-counting method was applied and the fractal dimension was related to the slope of the line representing: $\log(N_i)$ versus $\log(a_i)$.

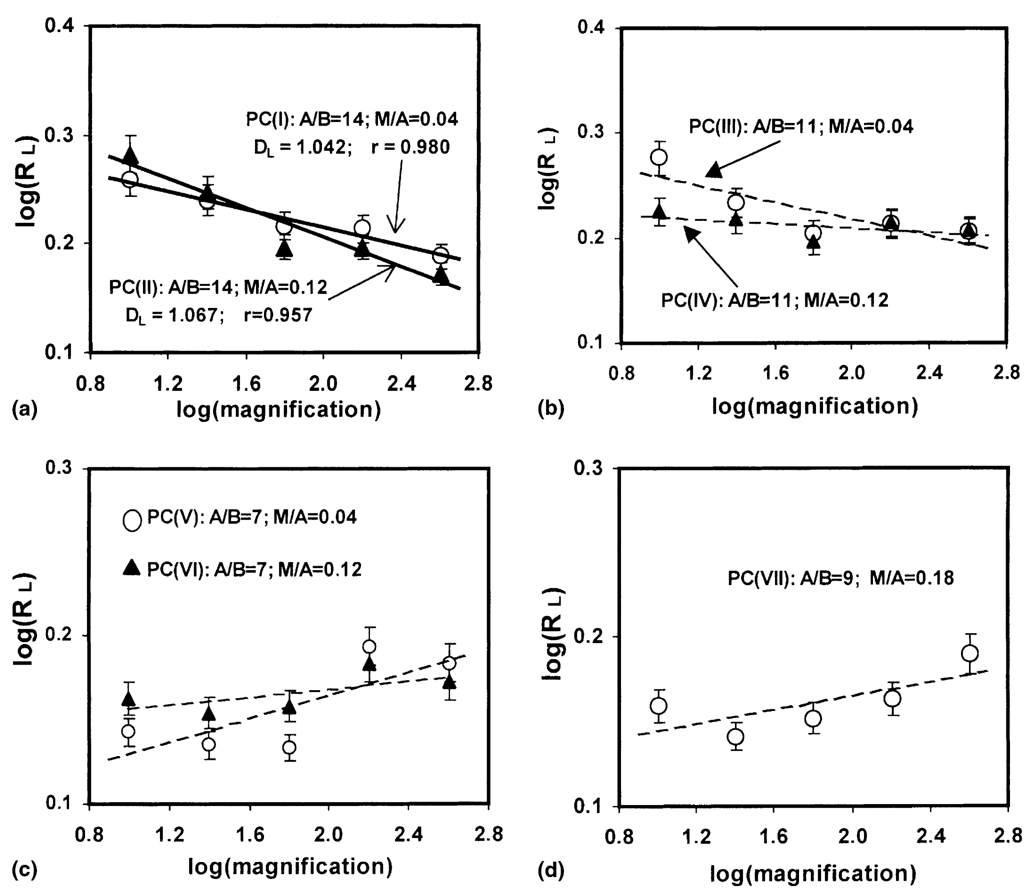

Fig. 6. Estimates of fractal dimension D_L from $\log(R_L)$ – log(magnification) curves.

5. Results and discussion

5.1. Scale effect

The tested epoxy concretes differed in the geometrical features of their fracture surfaces (Table 2). In general, it can be concluded that R_S and R_L values decreased (Fig. 4) with an increase of the resin binder (A/B) and a decrease of microfiller (M/A) content. The differences in microstructure of particular epoxy concrete types also caused changes in R_S and R_L values with different magnifications. The R_S and R_L decreased with an increase in magnification for concrete of PC(I)–PC(IV)-type with $A/B > 9$, and slightly increased for concretes wit higher content for resin binder $(A/B \leqslant 9)$: PC(V)–PC(VII). This suggests that for PC with higher resin binder content the microsilica significantly affect the geometrical features of the resin binder zone of fracture surface, when the profile is examined at higher magnifications. The R_S values for particular PC types had the highest variability at magnifications up to $63\times$. As the magnification was increased the differences in R_S were decreased and at magnification of $400\times$ the R_S values were practically same for each type of PC. A similar scale effect was observed for the R_L values.

The fractal dimension, D_b, determined with the box-counting method, decreased with an increase in magnification for all tested epoxy concretes (Fig. 5). As the magnification was increased the difference in the D_b values, for particular PC series, decreased. For magnifications higher than $63\times$ the fractal dimension was practically constant and independent of the PC microstructure. The value of $D_b = 1.02$ obtained for higher magnifications is close to the topological dimension for line $D = 1$. This indicates that the resin binder zone causes profile "smoothing", when it is examined at higher magnification.

The R_S and R_L values obtained at different magnifications can be used to estimate the fractal dimension from the log–log plot of R_L (or R_S) versus magnification. This method corresponds to the divider method. PC fracture surface is a fractal object over the whole range of magnifications when the data: log(R_L) – log(magnification) can be approximated by straight line with negative slope and the fractal dimension obtained can be treated as the average fractal dimension for the range of magnifications used. The results obtained from the log–log plot of R_L versus magnification (Fig. 6) suggest that the PC with $A/B > 11$, with the highest roughness of a fracture surface, demonstrates the self-similarity property and can be treated as the fractal object within the range of magnifications applied in this study. The value of fractal dimension D_L was close to the value of fractal dimension obtained from the box-counting method, D_b: $D_L = 1.04, D_b = 1.06$ for PC(I) at magn. $25\times$ and $D_L = 1.07, D_b = 1.06$ for PC(II) at magn. $10\times$, respectively. The difference in fractal dimension value, which equals ± 0.02, results from different estimation methods applied in this study (comp. also e.g. [7,8]). An increase in the resin binder and microfiller content also caused an increase in the scattering of data points from the linear approximation and led to a positive slope of log–log plot. In this case the self-similarity property cannot be observed for a wide range of magnifications. It means that the PC is the fractal object in a narrow range of magnifications. The results obtained indicate that PC fracture surface can be treated as a fractal object over the wide range of magnifications (10–400$\times$) for limited resin and microfiller contents. This implies that a thickness of resin envelope around aggregate grains is a decisive factor for self-similarity of PC within the wide range of observation levels. As the thickness of resin envelope increases the geometry of fracture surface is significantly modified by new microstructure elements (microfiller grains) which appeared at higher magnifications. In general, the resin content in the commercial PC ranges from 8% ($A/B \approx 11.5$) to 15% ($A/B \approx 5.5$). This suggests that commercial PC cannot be considered a fractal object. The investigation of relationship between the fracture surface geometry and toughness for different PC microstructures should be carried out at the same magnification.

6. Relationship R_S versus R_L

Different relationships have been suggested for different materials. Accordingly, specific relationships were

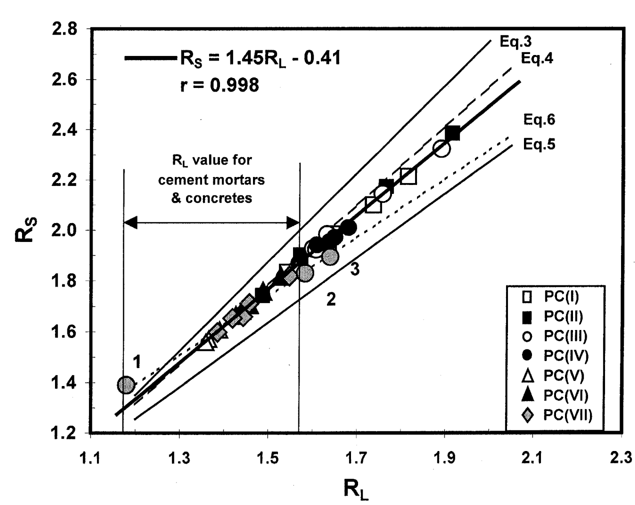

Fig. 7. Surface roughness ratio R_S against profile roughness ratio R_L for epoxy concretes tested at different magnifications; (• – are the results for cement mortar (1) and vinylester concrete (2.3) at magnification $10\times$ as for [19]).

also suggested for some types of construction materials. For example, to estimate the R_S value for cement concretes and mortars Eq. (6) is often used [6].

The relationship between R_S and R_L for the tested epoxy concretes (Fig. 7) can be approximated, over the whole range of magnifications, by linear dependence

$$R_S \approx 1.45R_L - 0.41 \text{ regression coeff. } r = 0.996. \qquad (8)$$

This equation is close to that provided by Wright and Karlsson (Eq. (5)). It also confirmed the previous estimation of relationship R_S versus R_L for vinylester concrete [20] (see Fig. 6). It should be pointed out that the formula often used for cement concrete (Eq. (6)) can underestimate the value of the R_S in the case of polymer concretes.

6.1. Relationship D_b versus R_S and R_L

The results obtained were also analyzed to establish a possible relationship of the box-counting fractal dimension, D_b, versus R_S and R_L at different magnifications (Fig. 8). Statistically significant regression equations (reg. coeff. >0.94) were obtained for PC with low contents of resin binder ($A/B > 11$) and microsilica ($M/A = 0.04$). An increase in resin and microsilica contents caused a decreasing significance of the relationship D_b, versus R_L and R_S. It indicates that the fractal dimension, D_b, R_L and R_S are, in general, independent parameters and characterize the fracture surface profile in different ways. It is important to point out that the changes in PC microstructure affect the R_S and R_L values more than the fractal dimension, D_b.

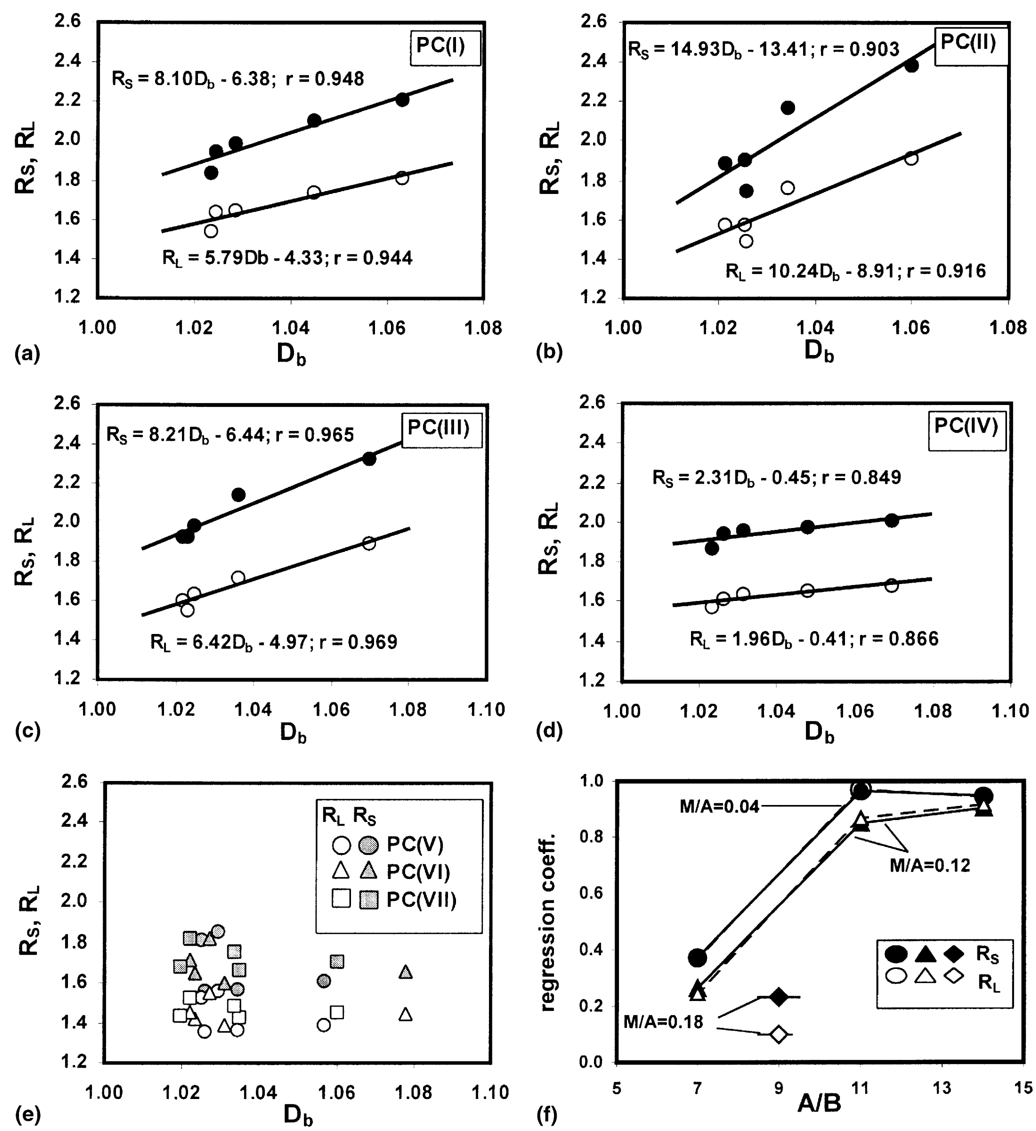

Fig. 8. Profile roughness ratio R_L and surface roughness ratio R_S versus fractal dimension D_b for tested epoxy concretes.

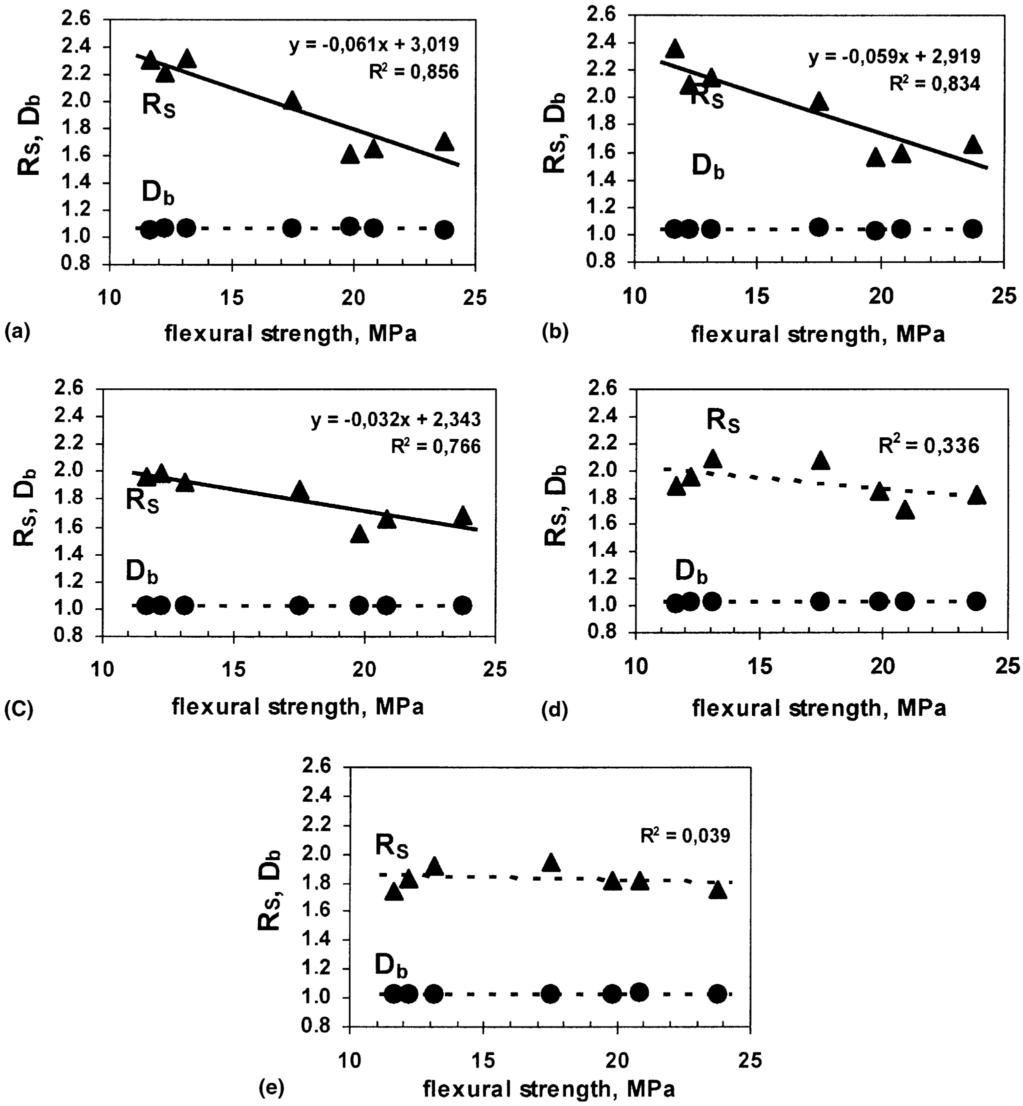

Fig. 9. Surface roughness ratio R_S and fractal dimension D_b versus flexural strength for tested epoxy concretes.

6.2. Relationships mechanical properties versus R_S and D_b

The relationships between mechanical properties (compressive and flexural strength) and parameters, R_S, and D_b, describing PC fracture surface at different magnifications were analyzed. The statistical analysis of the results showed that the relationships between mechanical properties and box-counting fractal dimension, D_b, for all tested magnification were not statistically significant. However, the relationships R_S versus compressive and flexural strength (Figs. 9 and 10) were statistically significant in the limited range of magnifications. For further consideration a determination coefficient, $R^2 > 0.5$ was treated as statistically significant. Following the above approach the limit of magnification towards fractographical analysis of PC was established (Fig. 11). The results obtained indicate that the relationships between mechanical properties and geometry of PC fracture surface might be statistically if the parameters describing the PC fracture surface were obtained at magnification lower than $120\times$ (see. Fig. 9(a)–(c) and Fig. 10(a)–(c)). Although there is no direct relationship between flexural strength and material toughness it can be expected that the above limit of magnification is also valid for the relationship between fracture roughness parameters and PC toughness. This also confirms that surface roughness ratio, R_S, is more useful for characterization of PC fracture surface in terms of its relation to toughness.

7. Conclusions

The parameters describing the geometry of the polymer concrete fracture surface namely: the surface

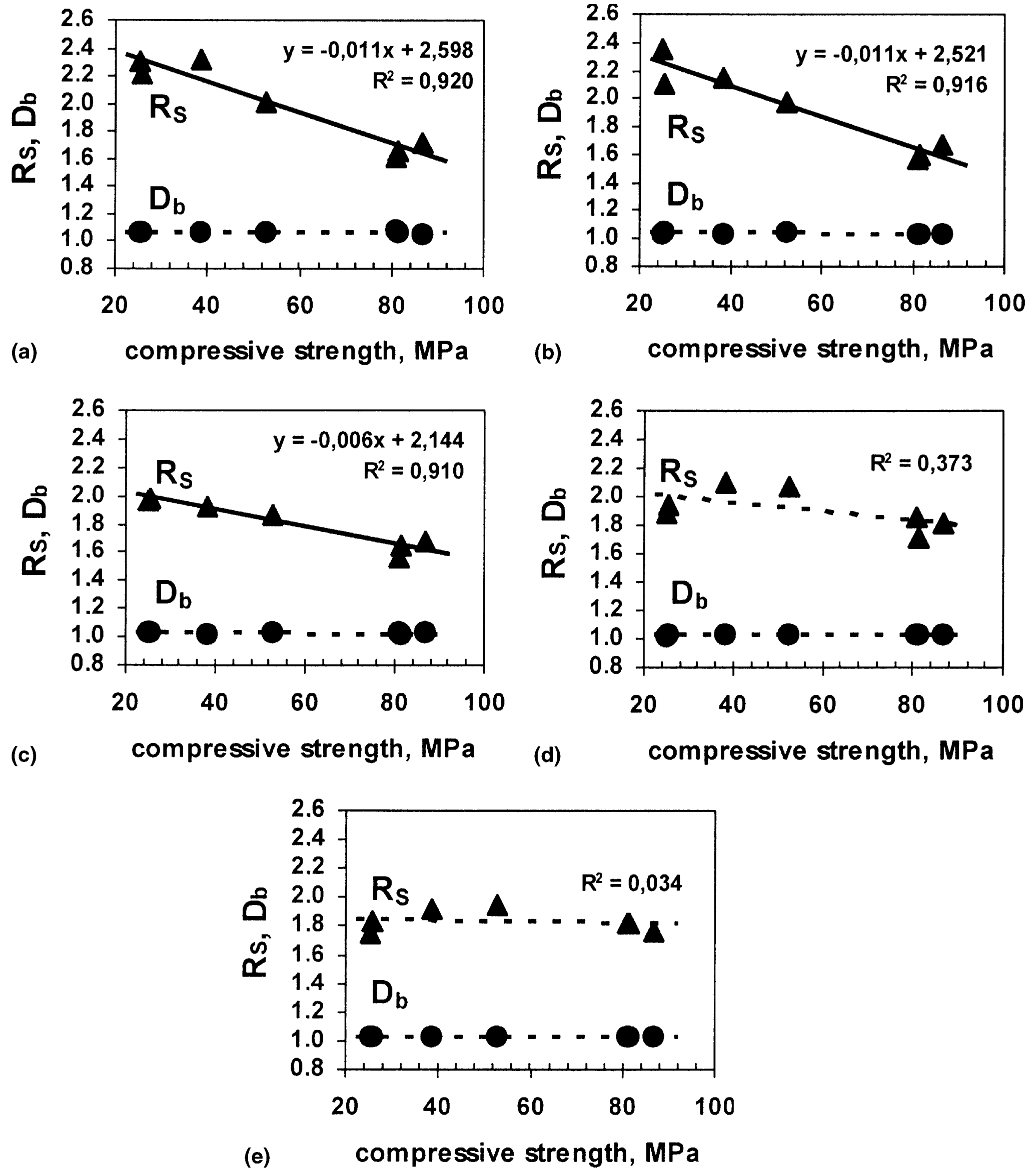

Fig. 10. Surface roughness ratio R_S and fractal dimension D_b versus compressive strength for tested epoxy concretes.

roughness ratio, R_S, profile roughness ratio, R_L, and fractal dimension, D_b, were determined for a wide range of magnifications:10–400 ×. Based on the test results, the following conclusions can be drawn:

1. The parameters characterizing the fracture surface of polymer concrete depend on the scale of observation. The box-counting fractal dimension, D_b, decreased with an increase in magnification.. The value of D_b was practically constant and independent of PC microstructure at magnification higher than 63 ×. The R_S and R_L decreased with an increase in magnification for PC with low content of resin binder. An increase in resin binder and microsilica content caused an increase in R_S and R_L values with an increase in magnification.
2. Estimating the fractal dimensions from log–log plot of R_L versus magnification (divider method) indicates that self-similarity was observed over the range of magnifications used only for PC with low content of resin binder ($A/B > 11$). As the resin content increased the self-similarity property was observed in narrow range of magnifications with the given value of fractal dimension. Microfiller significantly changed the geometry of fractal surface at resin binder zone when the profile is examined at higher magnifications.
3. The relationship between R_S and R_L for the tested polymer concretes can be approximated for wide range of magnifications by the linear equation: $R_S = 1.45R_L - 0.41$.

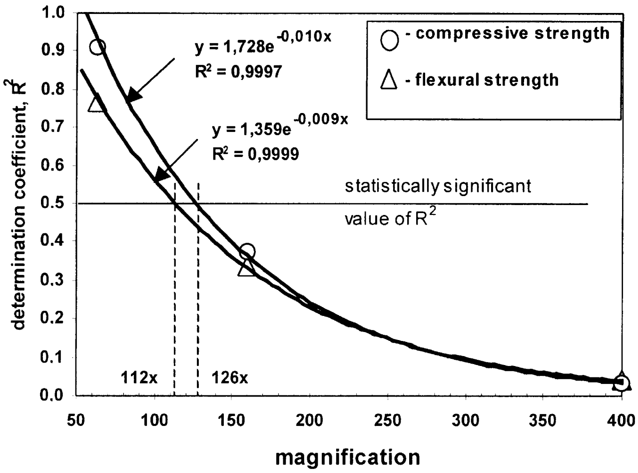

Fig. 11. Determination coefficients versus magnification determined for relationships: surface roughness ratio R_S and fractal dimension D_b versus flexural and compressive strength.

4. Statistically significant regression equation function ($r > 0.94$) for the relationship, D_b versus R_S and R_L, estimated at different magnifications, was obtained for PC with low content of resin binder ($A/B > 11$) and microsilica ($M/A = 0.04$). As the resin binder and microsilica content was increased the correlation coefficient decreased.
5. Statistically significant regression equation functions for the relationship between mechanical properties (compressive and flexural strength) versus R_S were obtained for magnification lower than 120 ×. The results indicate that relationships between mechanical properties and fractal dimension, D_b, were not statistically significant within range of magnifications used. This suggests that for investigating the relationship between fracture surface roughness of PC and its toughness, the fracture surface should be described by the fracture surface ratio, R_S, determined at magnification lower than 120 ×.

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