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The Statistical Evaluation of Epoxy Concrete Heterogeneity

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Abstract

The subject of this paper is the statistical estimation of heterogeneity of epoxy concrete. The study is involved with the potential of its random failures. The main categories and sources of possible uncertainty have been shown. The research concept has been presented, showing the parameters that influence the polymer composite heterogeneity, e.g. composition, or the method of maturing. Experimental results have been analyzed using statistical methods and presented numerically and graphically in the form of histograms and distribution curves. Three types of theoretical distribution — normal, log-normal and Weibull's — have been considered. Conclusions on the variability of the various types of epoxy concrete have been formulated as well as conclusions on the influence of particular factors on this variability. © 1997 Elsevier Science Limited

NOTATION

ah	accelerated hardening
Basalt	basalt aggregate
CQ	crushed quartzite aggregate
d	density (kg m^{-3})
D	maximum diameter of aggregate gravels (mm)
E	modulus of elasticity (GPa)
Granite	granite aggregate
log-n	log-normal distribution
n	safety class
nh	natural hardening
norm	normal distribution
NQ	natural quartzite aggregate
R_c	compressive strength (MPa)
R_f	flexural strength (MPa)
tria	triangular distribution
Weib	Weibull's distribution

INTRODUCTION

Epoxy composites (mortars and concretes) are frequently used as repair materials for building constructions. The main reason for this is the technical performance of epoxy composites (Table 1). However, in building applications, material performance is not the only requirement.

The building material usability is maintained by a balance between performance and reliability. Reliability, in the broad sense, is involved with dependability and with the absence of breakdowns and failures. For engineering analysis it is necessary to define reliability quantitatively as probability. It is the probability that a system will perform its intended function for a specified period of time under a given set of conditions. This definition is associated with several categories of uncertainties:[1]

the uncertainty of the kind and level of various (mechanical, physical and chemical) loadings, involved with the service conditions; it is valid in the same way for the well-known traditional material as it is for the new one;

the uncertainty of the material performance inherent mainly, but not solely, in the 'novelty' of materials.

The fundamental estimation of the composite material heterogeneity is the variability, commonly represented by the variation coefficient:

$$v = \sigma\bar{x}$$

Table 1. Epoxy concrete basic technical characteristic

Main advantages	*Main disadvantages*
1. Good or excellent corrosion resistance	1. Relatively high creep
2. Short hardening time-short time to obtain structural strength	2. Relatively small modulus of elasticity
3. Very good adhesion to various building materials	3. Limited heat resistance
4. High tightness	4. Large thermal expansion
5. High frost-resistance	5. Relatively large setting shrinkage
6. Very good wear resistance concrete	6. Abrasion resistance smaller than ordinary
7. High mechanical strength	7. High cost
8. Colouring ability	8. Special requirements towards production conditions: particularly low level of relative humidity and dry aggregate requirements
9. Minimum requirements in a range of concrete curing	

where σ = standard deviation and $\bar{x}$ = mean value of sample. The main source for this variability is the structural heterogeneity of the composite material:[2]

— heterogeneity of aggregate grains;
— dispersion of polymer binder;
— various situations in the aggregate–polymer binder interface zone, e.g. the difference in the development of adhesion forces or in polymer orientation effects.

At the present stagè of technological development of polymer concretes 'trade-offs' can more often be made according to performance than reliability. It is necessary to stress also that 'trade-offs' are often required between reliability and cost. The criteria on which the 'trade-offs' are based are deeply embedded in the essence of engineering practice which, in the case of polymer concretes, is not very well-established. Proper decisions in this range must be based on relevant information. This is, particularly, the case where an engineer wishes to assign to his decisions a numerically defined probability of them being accurate.

The technological aim of our study is to show how the guaranteed technical features of the epoxy repair composites depend on their heterogeneity. The cognitive aim is to -7ascertain how and why the composite material heterogeneity manifests itself differently in various properties of composite materials. How is this phenomena affected by various material parameters?

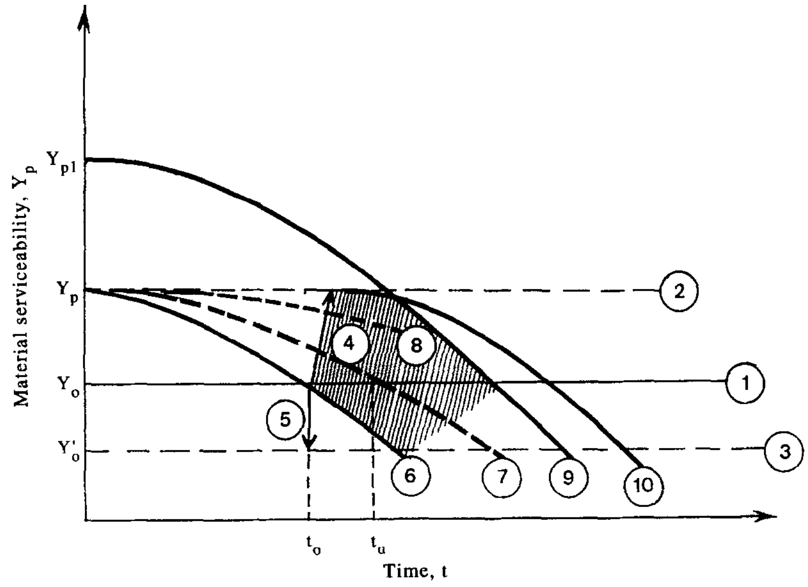

Fig. 1. The change of material serviceability with time: Y_{p1}, Y_{p2}—serviceability of two various materials; Y_o, Y'_o—required level of serviceability; t_u—time of required service; t_0—limiting time (the minimal accepted serviceability level has been attained).

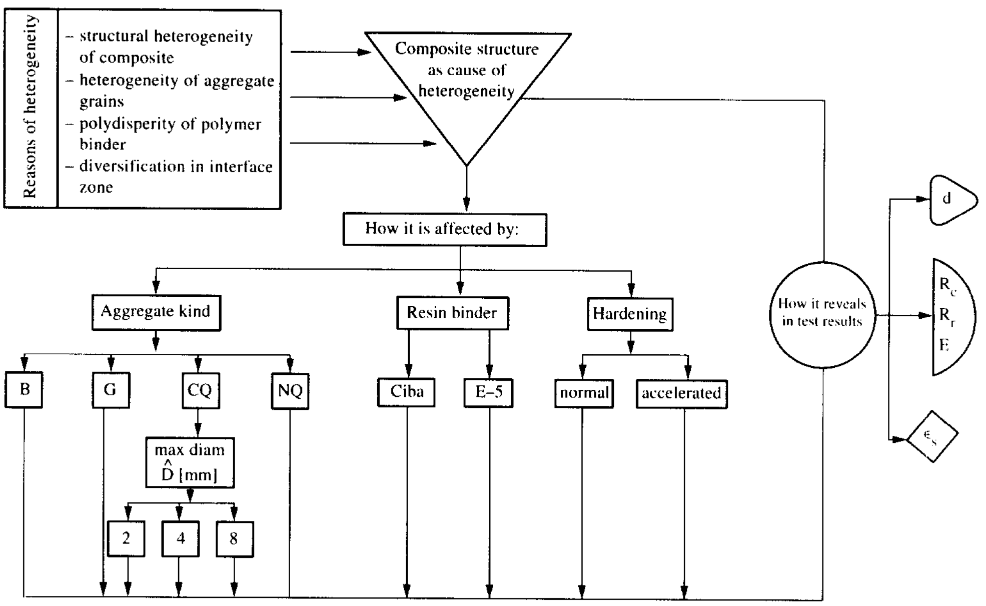

Fig. 2. Research conception: B-basalt; G-granite; CQ-crushed quartzite; NQ-natural quartzite; *d*-density; ε_s-hardening shrinkage; R_c-compressive strength; R_f-flexural strength; *E*-modulus of elasticity.

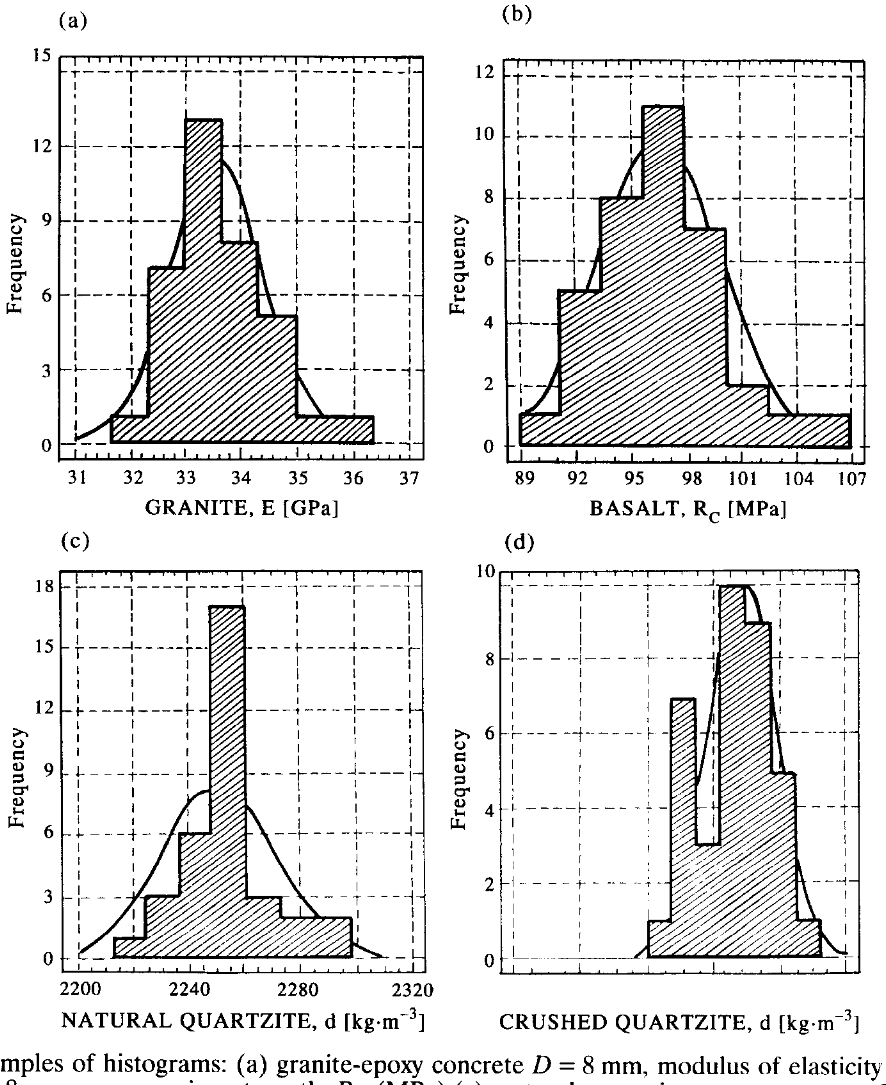

Fig. 3. Various examples of histograms: (a) granite-epoxy concrete $D = 8$ mm, modulus of elasticity *E* (GPa); (b) basalt-epoxy concrete $D = 8$ mm, compressive strength R_c (MPa);(c) natural quartzite-epoxy concrete $D = 8$ mm, density *d* (kg m^{-3});(d) crushed quartzite-epoxy concrete $D = 8$ mm, density *d* (kg m^{-3}).

Table 2. The estimates of the main statistical parameters[4]

Estimator	*Symbol*	*Definition*	
1. Mean value	$\bar{x}$	$\frac{1}{n}\sum_{i=1}^{n} x_i$	*
2. Median	Me	$x_{1/2(n+1)}$ for odd values of n $1/2(x_{1/2n}+x_{1/2n+1})$ for even values of n	
3. Mode	Mo	The most frequent value of the given variability interval	
4. Variance	S_x^2	$\frac{1}{n-1}\sum_{i=1}^{n}(x_i-\bar{x})^2$	
5. Standard deviation	S_x	$\sqrt{S_x^2}$	
6. Variation coefficient	v_x	$\frac{S_x}{\bar{x}}$	
7. Homogeneity coefficient	k	$1-t v_x$	**

*n-sample size.

**t-student's random variable; e.g. $t_{n-1,\alpha/2}$ for confidence level $1-\alpha = 0.95$ and $n = 36$; $t_{35;0.025} = 2.003$; for $n = \infty$; $t_{\infty;0.025} = 1.960$. Note: from $n = 30$, the difference between the distribution can practically be neglected.

APPROACH

Contrary to other applications, in the case of repair there is an extra, mainly sociological, but also technical, effect. The use of polymer composites (Fig. 1) is already a result of construction failure, sometimes even catastrophic failure. As a consequence there is the belief that the repaired construction should survive under the given service conditions satisfactorily. This evidence usually highlights the balance between the willingness to pay the repairing cost and the expected reliability. The general problem of reliability involves time. The properties of all building materials change with time (Fig. 2). In terms of failure rate we are able to consider three general categories of failure: early failure; random failure; and ageing effects.[5] Our study is involved with the potential of random failures.

Table 3. Probability distributions[4,6]

Distribution	*Density function*
Normal	$f(x)=\frac{1}{\sigma\sqrt{2\pi}}\exp\left[-\frac{(x-m)^2}{2\sigma^2}\right]$ m,σ—parameters of distribution m—mathematical expectation σ—standard deviation of the variable x
Log-normal	$f(x)=\frac{1}{x\sigma\sqrt{2\pi}}\exp\left[-\frac{(\ln x-m)^2}{2\sigma^2}\right]$ $x>0, m, \sigma$—parameters of distribution
Weibull's	$f(x)=\frac{\sigma}{\theta}x^{\sigma-1}\exp\left(-\frac{x^{\sigma}}{\theta}\right), x>0$ $\sigma>0$, $\theta>0$—parameters of distribution

RESEARCH CONCEPT

The main problems of the research (Figs 3) were in recognizing how the material heterogeneity is revealed in test results and how test results are affected by various heterogeneity causes:

kind of aggregate (mineral type, grain grade-natural and crushed as well as maximal grain diameter);

way of maturing (natural and accelerated).

Epoxy concretes with four kinds of mineral aggregates: basalt; granite; quartzite crushed; and natural, have been tested. The maximal diameter of aggregate grains were 2, 4 and 8 mm. The samples were tested after the accelerated hardening (1 day/20°C+2 days/60°C+1 day/20°C) and for comparison also after 21 days under normal conditions (20°C, 60% RH). Three categories of material properties have been determined:

classification (physical) parameter—density (d);

mechanical parameters—compressive strength (R_c), flexural strength (R_f) and modulus of elasticity (E);

technological parameter—hardening shrinkage (ε).

Four series of experimental tests (four samples each) on the given polymer concrete have been carried out for estimation of param-

Table 4. Values of the estimates of the statistical parameters of investigated properties of epoxy concretes (D_{max} = 8 mm; accelerated hardening)

Estimator	R_c *(MPa)*	R_f *(MPa)*	*E (GPa)*	*d* *(kg m^{-3})*	ε_s *(%)*
Aggregate	Basalt				
$\bar{x}$	96.8	35.3	39.8	2370	0.0048
Me	97.0	35.4	39.5	2370	0.0043
Mo	97.4	34.2	41.1	2370	0.0040
S_x^2	10.47	10.72	4.88	368	$1.27\cdot10^{-6}$
S_x	3.24	3.27	2.21	19.2	0.00113
v_x	0.03	0.09	0.06	0.01	0.23
k	0.94	0.82	0.88	0.98	0.53
Aggregate	Granite				
$\bar{x}$	80.8	29.9	33.6	2257	0.0063
Me	81.4	30.1	33.5	2260	0.0060
Mo	74.4	30.1	33.1	2260	0.0080
S_x^2	39.57	1.48	0.70	323	$2.18\cdot10^{-6}$
S_x	6.29	1.22	0.48	18.0	0.00147
v_x	0.08	0.04	0.02	0.01	0.24k
k	0.84	0.92	0.96	0.98	0.51

Statistical symbols—see Table 2; others—see Notation list.

eters of random variable distribution of each feature. The hypotheses concerning homogeneity of variances and the equivalence of means in each series have been verified by Hartley's test as well as Snedecor's F test at various confidence levels. The estimates of the main statistical parameters (Table 2) have been calculated. The comparison of empirical and theoretical distributions has been tested by means of the Kolmogorov-Smirnov test[6] for level of fit. Three types of theoretical distribution (Table 3) have been considered:

normal, as the most common and typical for the estimation of technical properties;

log-normal, as often used for analysis of variables, which can only take on values above zero;

Weibull's, as often used for estimations of the mechanical properties of polymer composites.

RESULTS AND DISCUSSION

Experiments have been performed according to the program presented in the preceding section. In each four experimental series for the given polymer concrete no reason has been found to contest the homogeneity of variances and equivalence of means for the significance level

Table 5. Values of the estimates of the statistical parameters of investigated properties of epoxy concretes (D_{max} = 8 mm; accelerated hardening)

Estimator	R_c *(MPa)*	R_f *(MPa)*	*E* *(GPa)*	*d* *(kg m^{-3})*	ε_s *(%)*
Aggregate	Crushed	Quartzite			
$\bar{x}$	89.5	31.4	40.9	2237	0.0054
Me	88.6	30.8	41.35	2240	0.0058
Mo	88.6	30.6	41.8	2250	0.0060
S_x^2	18.67	1.74	5.59	428	$1.14\cdot10^{-6}$
S_x	4.32	1.32	2.36	20.7	0.00107
v_x	0.05	0.04	0.06	0.01	0.20
k	0.90	0.92	0.88	0.98	0.59
Aggregate	Natural	Quartz	Gravel		
$\bar{x}$	85.1	31.1	40.3	2251	0.0044
Me	85.35	31.0	40.45	2250	0.0045
Mo	86.7	31.5	41.2	2260	0.0045
S_x^2	21.66	2.86	5.61	422	$1.42\cdot10^{-6}$
S_x	4.65	1.69	2.37	20.5	0.00376
v_x	0.05	0.05	0.06	0.01	0.09
k	0.90	0.90	0.88	0.98	0.82

Statistical symbols—see Table 2; others—see Notation list.

Table 6. Values of the estimates of the statistical parameters of investigated properties of epoxy concretes (aggregate: quartzite; epoxy resin: Ciba-Geigy Araldit $D_{\mathrm{max}} = 2.4$ mm, hardening under normal conditions)

Estimator	R_c *(MPa)*	R_f *(MPa)*	E *(GPa)*	d *(kg m^{-3})*
D_{max}	2 mm			
$\bar{x}$	93.5	41.1	38.0	2010
Me	95.15	40.75	37.8	2010
Mo	97.1	40.2	37.9	1990
S_x^2	15.94	1.32	0.90	850
S_x	3.99	1.15	0.95	29.1
v_x	0.04	0.03	0.02	0.01
k	0.91	0.94	0.96	0.98
D_{max}	4 mm			
$\bar{x}$	86.1	35.3	41.4	2180
Me	86.4	35.2	41.2	2180
Mo	76.8	34.6	41.0	2180
S_x^2	68.42	3.4	0.17	177
S_x	8.27	1.84	0.41	13.3
v_x	0.10	0.05	0.01	0.01
k	0.80	0.90	0.98	0.98

Statistical symbols—see Table 2; others—see Notation list.

$\alpha = 0{\cdot}10$. Therefore, values of estimates of the statistical parameters have been computed for all 36 samples of the given polymer concretes (Tables 4). A considerable argument (Table 8) in favour of the examined distribution being normal (Figs 3–8) or log-normal has been found. It is also in agreement with some literature data.[3]

The technical feature which has shown (Tables 4 and 5) the highest scattering (material homogeneity coefficient, $k = 0{\cdot}50$–$0{\cdot}80$) is the curing shrinkage. It means that under given conditions it is very difficult to consider this technical feature as the particular material characteristic. Undoubtedly the precision of the test measurements should be improved in this case. On the base of the present results the aggregate has been categorized according to diminishing scattering of the curing shrinkage of polymer concrete as follows: granite; basalt; crushed quartzite; and natural quartzite.

The density has demonstrated a very uniform distribution pattern ($k \approx 0{\cdot}98$). The distribution curves are not too sensitive to the kind of resin or hardening condition (Fig. 4), as well as to the kind of aggregate (compare with Fig. 8(d)), but

Table 7. Values of the estimates of the statistical parameters of investigated properties of epoxy concretes (aggregate: quartzite; epoxy resin: Epidian-5 PL, $D_{\mathrm{max}} = 2.8$ mm, hardening under normal conditions)

Estimator	R_c *(MPa)*	R_f *(MPa)*	E *(GPa)*	d *(kg m^{-3})*
D_{max}	4 mm			
$\bar{x}$	78.4	35.8	53.0	2192
Me	76.9	35.9	53.3	2190
Mo	87.6	35.1	53.8	2210
S_x^2	56.24	4.12	0.88	227
S_x	7.50	2.03	0.94	15.1
v_x	0.09	0.06	0.02	0.01
k	0.81	0.88	0.96	0.98
D_{max}	8 mm			
$\bar{x}$	89.2	38.2	55.1	2235
Me	89.9	37.95	55.1	2240
Mo	91.2	37.7	54.7	2230
S_x^2	91.2	1.95	3.24	317
S_x	9.55	1.40	1.80	17.8
v_x	0.10	0.04	0.03	0.01
k	0.80	0.92	0.94	0.98

Statistical symbols—see Table 2; others—see Notation list.

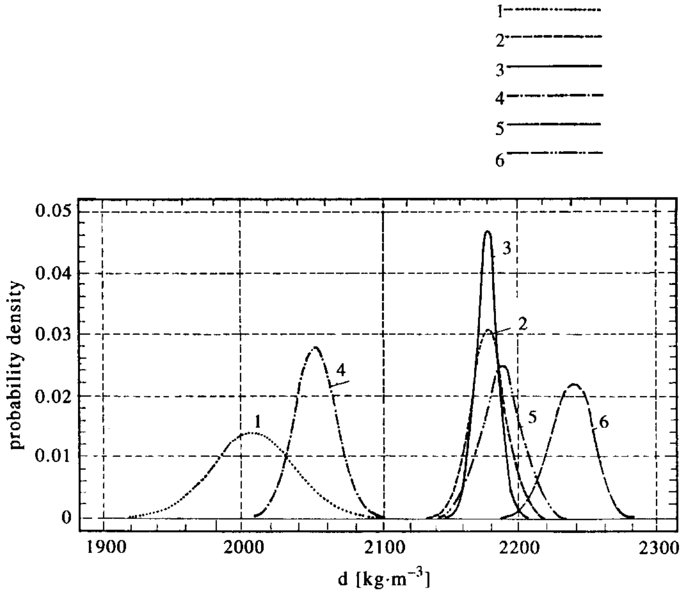

Fig. 4. Normal distribution of density of epoxy resin concretes: 1. E-5, $D = 2$ mm, normal hardening, 2. E-5, $D = 4$ mm, normal hardening, 3. E-5, $D = 2$ mm, accelerating hardening, 4. E-5, $D = 4$ mm, accelerating hardening, 5. CIBA, $D = 4$ mm, normal hardening and 6. CIBA, $D = 8$ mm, normal hardening.

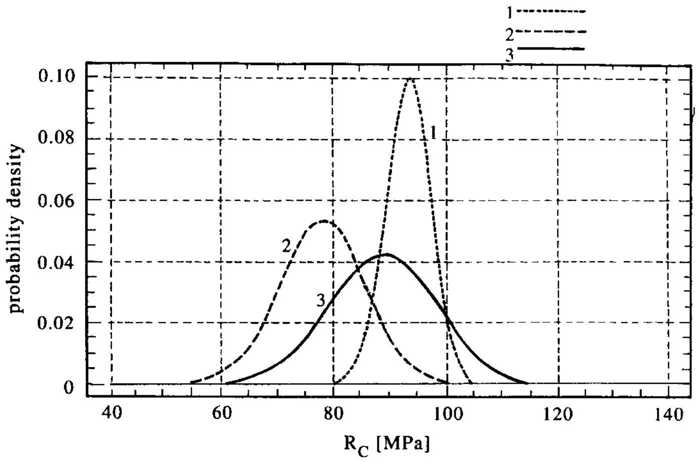

Fig. 5. Normal distribution of compressive strength of epoxy resin concretes: 1. E-5, $D = 2$ mm, normal hardening, 2. CIBA, $D = 4$ mm, normal hardening and 3. CIBA, $D = 8$ mm normal hardening.

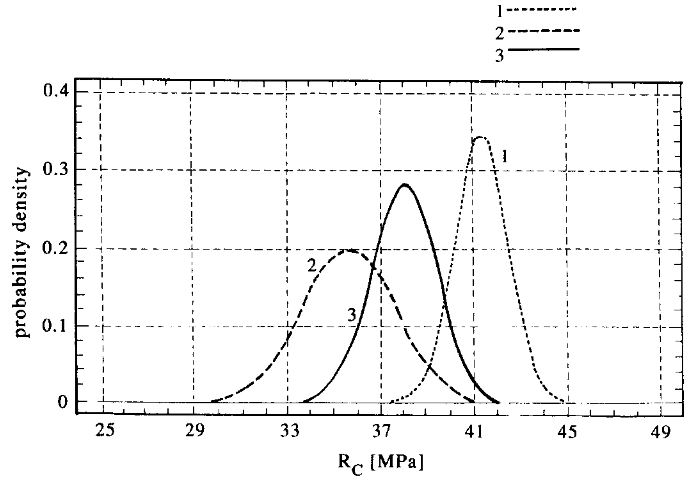

Fig. 6. Normal distribution of flexural strength of epoxy resin concretes: 1. E-5, $D = 2$ mm, normal hardening, 2. CIBA, $D = 4$ mm, normal hardening and 3. CIBA, $D = 8$ mm normal hardening.

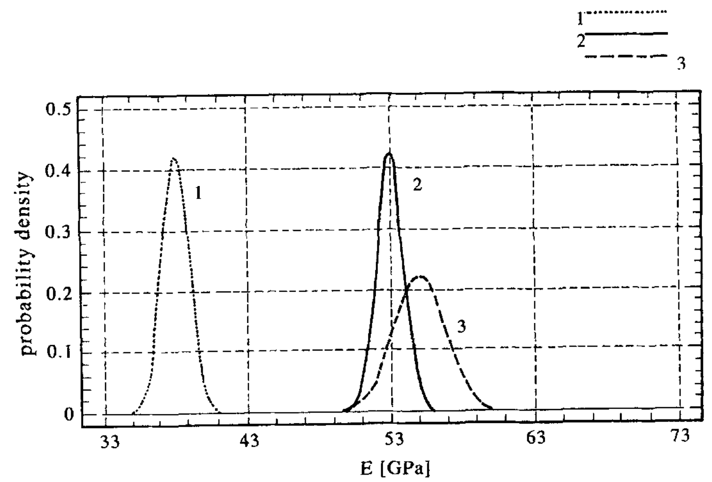

Fig. 7. Normal distribution of modulus of elasticity of epoxy resin concretes: 1. E-5, $D = 2$ mm, normal hardening, 2. CIBA, $D = 4$ mm, normal hardening and 3. CIBA, $D = 8$ mm normal hardening.

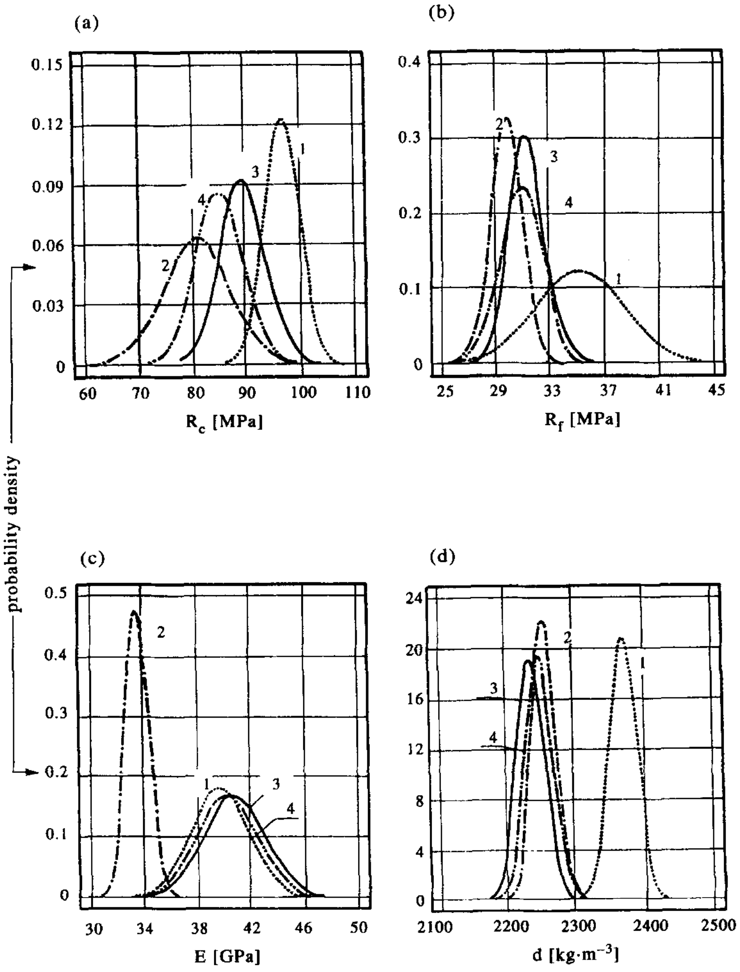

Fig. 8. Normal distribution of compressive strength (a), flexural strength (b), modulus of elasticity (c) and density (d) of epoxy resin concretes ($D = 8$ mm, accelerated hardening): 1: basalt, 2: granite, 3: crushed quartzite and 4: natural quartzite.

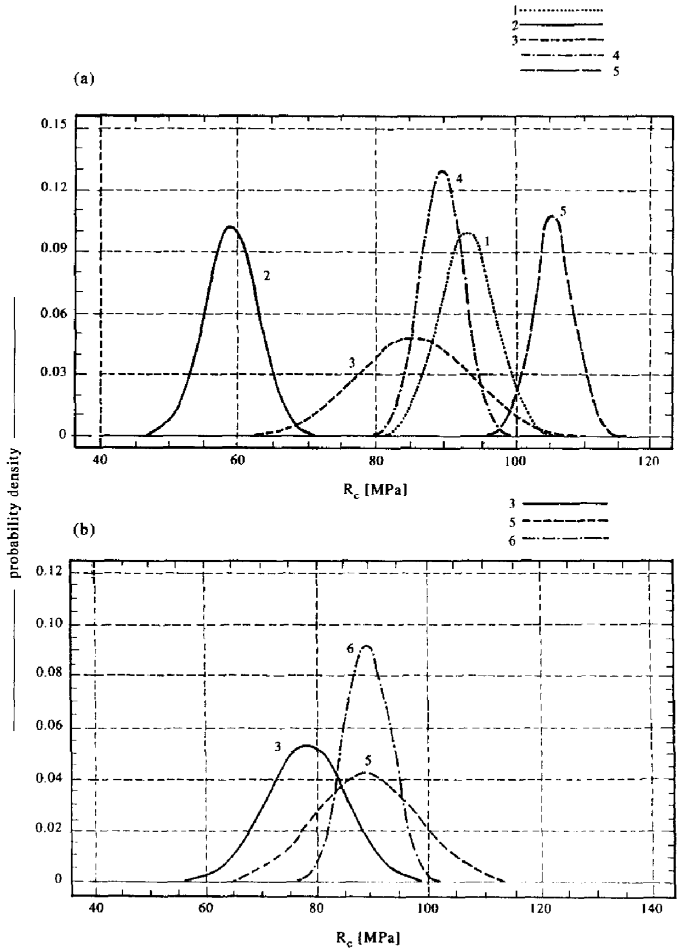

Fig. 9. Normal distribution of compressive strength of epoxy resin (a: E-5, b: CIBA) concretes hardened in various conditions: $D = 2$ mm, normal hardening, 2. $D = 2$ mm, accelerated hardening, 3. $D = 4$ mm, normal hardening, 4. $D = 4$ mm accelerated hardening, 5. $D = 8$ mm, normal hardening and 6. $D = 8$ mm, accelerated hardening.

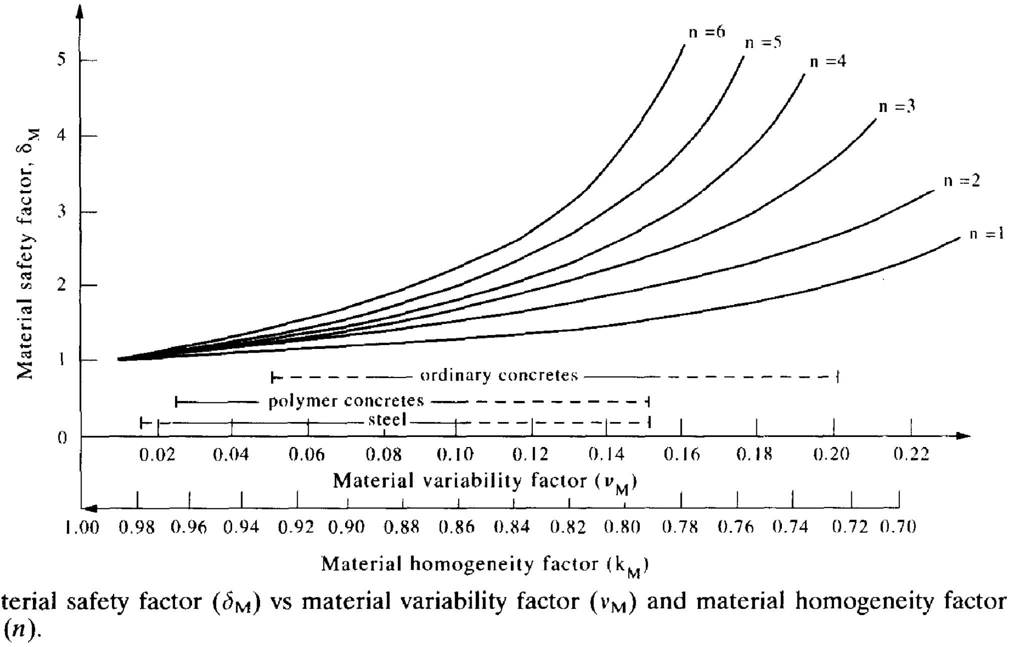

Fig. 10. Material safety factor (δ_M) vs material variability factor (ν_M) and material homogeneity factor (k_M) for various safety classes (n).

Table 8. Results of the Kolmogorov-Smirnov's tests for goodness of fit. The confidence levels$^{1)}$ of the conformability between empirical and theoretical distributions

	Norm	log n	Weib	Norm	log n	Weib
		Basalt; 8 mm; ah*			Granite; 8 mm; ah***	
R_c	0.99	0.99	0.99	0.99	0.99	0.99
R_f	0.99	0.99	0.99	0.53	0.46	0.99
E	0.99	0.99	0.36	0.46	0.46	0.43
d	0.15	0.16	nc	0.16	0.16	nc
		CQ; 8 mm; ah***			NQ; 8 mm; ah***	
R_c	0.40	0.48	0.99	0.99	0.99	0.99
R_f	0.12	0.13	0.39	0.99	0.99	0.52
E	0.28	0.22	0.16	0.32	0.24	0.31
d	0.25	0.24	nc	0.44	0.41	nc
		CQ; 2 mm; nh**			CQ; 4 mm; nh**	
R_c	0.99	0.99	0.99	0.99	0.99	0.99
R_f	0.99	0.99	0.49	0.99	0.99	0.99
E	0.99	0.99	0.47	0.99	0.99	nc
d	0.99	0.99	nc	0.99	0.99	nc
		CQ; 4 mm; nh***			CQ; 8 mm; nh***	
R_c	0.99	0.99	0.99	0.99	0.99	0.51
R_f	0.99	0.99	0.99	0.99	0.99	0.99
E	0.99	0.99	0.99	0.99	0.99	0.99
d	0.99	0.99	nc	0.99	0.99	nc

nc-not calculated (unable to calculate-division by 0).
*The confidence level > 0.1 is usually accepted in engineering considerations.
**Epoxy binder—Epidian-5, PL.
***Epoxy binder—Ciba-Geigy, Araldit.

depend mainly on the maximal diameter (D) of the aggregate grain. In the case of finer fillers the distributions are broader. The density depends on porous content; under the given preparation condition it is involved mainly with the filler grading.

The maximal diameter of the aggregate grain is also the factor which affected, in a significant way, the mechanical properties, e.g. compressive strength (Fig. 5), flexural strength (Fig. 6) and modulus of elasticity (Fig. 7). The narrowest distribution curve is obtained for the smallest filler grain diameter. It can be assumed that in the case of load dependent properties the heterogeneity of properties is to some extent 'parallel' to macrostructural heterogeneity of the material.

The width of the distribution of load independent and load dependent properties changes also regarding the kind (basalt, granite, crushed quartzite and natural quartzite) of aggregate. The distribution of various properties of the given polymer concretes is manifested in various ways, e.g. for basalt epoxy concrete the compressive strength distribution is narrow but the distribution of flexural strength or modulus of elasticity is broad. From a practical point of view it means that estimation of the material inhomogeneity should be done with reference to the given properties (Fig. 8.).

The conditions of hardening also affected the load dependent properties. As a rule, the width of distribution is broader after normal hardening than after accelerated hardening (temperature treatment). In our experimental program the kind of resin has only a slight influence on the shape of distribution curves (Fig. 9.).

For the ideal homogenous material the variation and homogeneity coefficients should be equal: $\gamma = 0$ and $k = 1$, accordingly. For engineering materials the evaluation criteria could be defined ($\alpha = 0{\cdot}05$, $n = 35$) as follows:

$\gamma \leq 0{\cdot}04$	$k \geq 0{\cdot}92$	very good
$0{\cdot}04 < \gamma \leq 0{\cdot}06$	$0{\cdot}92 > k \geq 0{\cdot}87$	good
$0{\cdot}06 < \gamma \leq 0{\cdot}10$	$0{\cdot}87 > k \geq 0{\cdot}80$	sufficient
$\gamma > 0{\cdot}10$	$k < 0{\cdot}80$	insufficient

Taking into consideration the technical responsibility involved with the repair job the criteria mentioned above are, relatively, twice more stringent than for the ordinary concrete.[7] Nevertheless the tested composites can be classified often as 'good' or even 'very good' and only rarely as 'satisfactory' towards various properties. This gives an estimation of a suit-

Table 9. Safety classes n accepted for various cases of material reliability

Group	*Subgroup*		
	A	B	C
I	$n = 1$	$n = 2$	$n = 3$
II	$n = 4$	$n = 5$	$n = 6$
	Explanation		
I	Possible failure does not cause the change of the usefulness of the element and only decreases the user comfort and aesthetics		
II	Possible failure causes the element not to comply with the usability state and is perilous for the user		
A	Repair is easy and non-expensive and can be done during routine maintenance		
B	Repair is possible but difficult and expensive		
C	Repair is impossible; element should be replaced as a whole or in a part		

able 'material reliability' — the material safety factor on the given safety level and/or guarantee value. The relationship between the safety factor and the material variability factor (Fig. 10) depends on the values of accepted safety class. The safety class n is defined as follows: $-\log(1-P)$ or $P = 1-0{\cdot}1^n$, where P is the desired (assumed) certainty of the non-failed work of the element; for $n = 1$, $P = 0{\cdot}9$ and for $n = 6$, $P = 0{\cdot}999999$. Acceptance of the safety class depends on the effects caused by possible failure of the element as well as on the possibility and cost of its repair (Table 9). The material variability of epoxy concrete and mortars used in repair could be evaluated as equal to, or better than, ordinary concrete, and similar to steel (compare with Fig. 10). However, it should be stressed that all results presented above are dealing with 'lab concrete'. The situation would be different/more severe 'on-site' in the case of 'real concrete'. Some indications in this field could be found in the British Standard 089;1981.[9]

CONCLUDING REMARKS

A polymer composite is inhomogeneous by definition. As a result the composite properties are not represented by 'sharp' values and it is necessary to use a statistical approach. The test results can be organized into suitable frequency histograms and on this basis the statistical distribution of technical characteristics can be presented. Regarding the potential variability, various epoxy concretes and mortars could be frequently evaluated as 'good' or even 'very good'. However, 'proper' estimations should be done for each property of significance in the given application.

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