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Filled epoxy composites based on polyfraction microcalcite

Наполненные эпоксидные композиты на основе полифракционного микрокальцита

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Ключевые слова: эпоксидные композиты; наполнитель; микромрамор; степень наполнения; предел прочности при сжатии; предел прочности на растяжение при изгибе; изолинии; треугольные диаграммы Гиббса-Розебома

Abstract. The effect of the polyfractional micromarble introduction on the change in the physical and mechanical characteristics of polymer composites based on low-viscosity epoxy binders has been studied. Evaluation of changes in compressive strength and tensile strength in bending of epoxy composites samples containing micromarble of three different fractions, depending on the degree of filling, was carried out. Using the method of least squares, mathematical dependences of the epoxy composites strength characteristics on the type of used micromarble and the degree of filling have been obtained. The analysis of isolines and Gibbs-Rosebom triangular diagrams allowed to determine the composition of the polyfractional filler, the composites on the basis of which have the greatest strength characteristics. Polymer composite containing a mixture of micromarble of different fractions in the amount of 80 % of the maximum filling level and having an increased compressive strength (up to 40 %) and close to the limit tensile strength during bending (decrease by no more than 10 %) compared to the unfilled composition epoxy polymer was obtained. The mechanical properties and the high filling degree of the obtained composite indicate the possibility of using micromarble as an effective filler for epoxy polymers, which is also confirmed by the achieved substantial economic effect.

Аннотация. Исследовано влияние введения полифракционного микромрамора на изменение физико-механических характеристик полимерных композитов на основе низковязких эпоксидных связующих. Проведена оценка изменения предела прочности при сжатии и на растяжение при изгибе образцов эпоксидных композитов, содержащих микромрамор трех различных фракций, в зависимости от степени наполнения. С использованием метода наименьших квадратов получены математические зависимости прочностных характеристик эпоксидных композитов от вида используемого микромрамора и степени наполнения. Анализ изолиний и треугольных диаграмм Гиббса-Розебома позволил определить состав полифракционного наполнителя, композиты на основе которого, обладают наибольшими прочностными характеристиками. Получен полимерный композит, содержащий смесь микромрамора различных фракций в количестве 80 % от предельного уровня наполнения и обладающий повышенным пределом прочности при сжатии (до 40 %) и близким по значению пределом прочности на растяжение при изгибе (снижение не более 10 %) по

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сравнению с ненаполненным составом эпоксидного полимера. Механические свойства и высокая степень наполнения полученного композита свидетельствуют о возможности использования микромрамора в качестве эффективного наполнителя для эпоксидных полимеров, что также подтверждается достигаемым существенным экономическим эффектом.

1. Introduction

Nowadays, polymeric materials are widely applied in various industries, which is caused by a set of unique properties and continuously extending product range [1–6]. In the building industry, thermoset polymers on the basis of low-molecular epoxy and diane resins with the average molecular weight of 350-600 are the most widespread [7–11]. High resistance to the majority of corrosive media allows to apply them as protective coats and impregnations almost anywhere [12–16]. However, wider application is significantly restricted by high cost and insufficient preservation of properties under the influence of solar radiation [17–22]. To date, the main way of reducing the cost price of protective coats is reduction of binders consumption by supplementing their composition with various disperse fillers [23–29].

It is considered that fillers were initially added to rubbers for increase of their durability in dozen times [27]. Then fillers were added to thermoset materials too. Today numerous kinds of fillers of various purposes are used in different industries and enable to modify mechanical, technological, and decorative characteristics of composites. Marshallit, chalk, portland cement, diatomite, and dolomite [11–13, 27–29] are applied as fillers at manufacture of polymeric composites of building purposes. However, scientists and manufacturers are searching for new effective fillers with controllable chemical and fractional composition. In the recent years, microcalcite (calcium carbonate, micromarble) is widely applied for manufacture of polymeric composites. This material is obtained by mechanical crushing of waste left after processing of white marble with further separation [27]. Constant physical and chemical properties, granulometric composition, and high degree of whiteness made microcalcite very widespread in manufacture of plastic, building materials, and papers [27, 30]. These properties combined with high resistance to corrosive media, atmospheric factors, and ultra-violet radiation make microcalcite very promising filler for building polymeric protective and decorative coats.

From one hand, increase of disperse filler share in polymer coat composition leads to considerable reduction of its cost price due to smaller consumption of binder. On the other hand, this results in significant worsening of mixture workability [30–32]. As a result, the optimal filler content for ensuring the best possible economic effect should be researched and determined. As a rule, application of plasticizers and solvents for obtaining a better plasticity of the filled mixtures is not expedient, as it leads to significant reduction of crack resistance, worsening of strength and adhesive properties of polymeric coats [16, 23, 28]. Considering constantly growing range of low-molecular epoxy and diane resins, authors believe this problem can be solved by application of low-viscosity binders, which, in its turn, requires conducting additional researches for analyzing the principles of their interaction with various fillers.

There are ways for obtaining filled coats of building products and structures, which properties are distributed according to functional requirements by control of polymeric binder of viscosity, as well as filling degree and fraction composition of the filler [26, 33]. Approach applied fir forming non-uniform distribution of polymeric coat properties by cross section height based on theoretical and experimental researches of restricted sedimentation processes with the help of multispeed continuum mechanics has shown obviously promising outlook for development of filled composite compositions.

The purpose of carried out researches was the development of structures filled with epoxy composites on the basis of low-viscosity binders and polyfractional microcalcite featuring high physical and mechanical properties. The following tasks have been solved for achieving the set objective: 1) influence of fractional composition of microcalcite and filling degrees on the change of mechanical properties of polymeric composites was defined; 2) polynomial equation ratios were determined with the help of planning methods and mathematical analysis of experimental researches and the graphic dependences reflecting the influence of filling degree and fractional composition of microcalcite on filled composite properties were built; 3) compositions on the basis of which the composites feature the best complex of properties were determined.

2. Materials and Methods

Surface properties of composites based on two-component epoxy compound of cold setting Etal-27NT/12NT which composition was supplemented with microcalcite of various fractions were researched within the scope of experiment:

1) MKM1 (V_1) – coarse fraction (0.5÷1 mm);

2) MKM2 (V_2) – medium fraction (0.2÷0.5 mm);

3) MKM3 (V_3) – fine fraction (less than 0.2 mm).

Degree of composite filling varied in the range from 40 to 80% of the limit filler content (Table 1). $V_1 + V_2 + V_3 = 1$ condition was observed for all compositions at experiment planning. Experimental research plan in coded values is presented in Table 2.

Change of ultimate compression and tensile strength when bending bar samples having the size of 20×20×70 mm was determined at the experiment. Samples filled with epoxy composites were produced by casting a mix of filler and Etal-27NT/12NT compound (manufacturer - JSC “ENPC EPITAL”). Samples solidified on air within 24 hours with further additional solidification within 6 hours at temperature of 80 °C.

Table 1. Level of variable factor varying

In 100 mass fractions of epoxy binder					
Mass content of fillers			Maximal content of fillersin a mix, gram		
–1	0	+1	MKM1 (V_1)	MKM2 (V_2)	MKM3 (V_3)
40 %	60 %	80 %	300	250	200

Table 2. Experiment plan in coded values

Test number	Values of factors under study			
	fraction in filler mix			Degree of filling (X)
	MKM1 (V_1)	MKM2 (V_2)	MKM3 (V_3)	
1	1	0	0	-1
2	0	1	0	-1
3	0	0	1	-1
4	0.5	0.5	0	-1
5	0.5	0	0.5	-1
6	0	0.5	0.5	-1
7	1	0	0	0
8	0	1	0	0
9	0	0	1	0
10	0.5	0.5	0	0
11	0.5	0	0.5	0
12	0	0.5	0.5	0
13	1	0	0	+1
14	0	1	0	+1
15	0	0	1	+1
16	0.5	0.5	0	+1
17	0.5	0	0.5	+1
18	0	0.5	0.5	+1

Polynomial dependence of the following kind was used for description of experimental research results:

$$Y = b_1 \cdot V_1 + b_2 \cdot V_2 + b_3 \cdot V_3 + b_{12} \cdot V_1 \cdot V_2 + b_{13} \cdot V_1 \cdot V_3 + b_{23} \cdot V_2 \cdot V_3 + d_1 \cdot V_1 \cdot X + \\ + d_2 \cdot V_2 \cdot X + d_3 \cdot V_3 \cdot X + d_{12} \cdot V_1 \cdot V_2 \cdot X + d_{13} \cdot V_1 \cdot V_3 \cdot X + d_{23} \cdot V_2 \cdot V_3 \cdot X + \\ + k_1 \cdot V_1 \cdot X^2 + k_2 \cdot V_2 \cdot X^2 + k_3 \cdot V_3 \cdot X^2 + k_{12} \cdot V_1 \cdot V_2 \cdot X^2 + k_{13} \cdot V_1 \cdot V_3 \cdot X^2 + k_{23} \cdot V_2 \cdot V_3 \cdot X^2 \quad (1)$$

Polynomial equation ratios were defined using the method of the least squares, which consists in the selection of the equation coefficients, for which the squares of deviations sum of the calculated values from the experimental ones is minimal [34]. According to the experiment plan, the plan matrix $[X]$ and the outputs column vector $[Y]$ were compiled. Using matrix operations, unknown coefficients column vector $[B]$ was found from the solutions of linear algebraic expressions system:

$$[B] = ([X^T] \cdot [X])^{-1} \cdot [X^T] \cdot [Y]. \quad (2)$$

Adequacy of mathematical models was verified with Fisher criterion. Numerical values of polynomial equation ratios are specified in Table 3.

Table 3. Ratios of polynomial equation describing the change of ultimate compression and bending strength depending on varied factors

Polynomial equation ratios (1)																
b_1	b_2	b_3	b_{12}	b_{13}	b_{23}	d_1	d_2	d_3	d_{12}	d_{13}	d_{23}	k_1	k_2	k_3	k_{12}	k_{13}	k_{23}
Ultimate compression strength, MPa																	
56.6	62.3	67.4	-5.16	3.72	8.06	0.87	1.33	2.74	-0.99	6.62	5.08	-1.36	-3.58	-0.20	21.01	8.48	2.28
Ultimate bending tensile strength, MPa																	
46.8	59.2	48.9	-3.77	80.1	40.9	-20.0	-19.9	-5.32	19.4	41.8	28.0	8.05	0.76	0.81	-7.97	-31.5	-4.39

3. Results and Discussion

Isolines of ultimate compression and tensile strength change in bending (Figures 1, 2) depending on ratio of two fractions in micromarble at various degrees of filling were formed on the basis of obtained polynomial equations.

All studied compositions feature increased strength at compression with the increase of degree of filling, which is caused by two factors, one of which underlies the principle of filling and consists in greater strength at compression of mineral filler compared to polymer's strength. The second factor relates to composition changes in the polymeric matrix on the border between filler and binder. During sedimentation, filler particles aim to reduce their surface energy and are grouped as cluster formations that have new quality properties. Structural order of polymeric matrix can be observed inside the cluster, in a polymer layer adjoining filler particles' surface. As a result, higher density leads to significant strengthening of the boundary layer [27, 31, 32]. Binder in the cluster is in a film state, and beyond the cluster - in a bulk state. Increase in the degree of filling leads to greater amount of clusters, which raises film phase content in a composite.

On the basis of results of the carried out research, it was found out that the greatest compression strength values (Figure 1, c) are observed in composites filled with a combination of medium and fine fractions of micromarble (MKM2+MKM3) in the ratio of 0.2:0.8 at maximum degree of filling. Similar values were obtained for combination of MKM1+MKM3 (Figure 1, b). Thus, increase of fine fraction micromarble share in the composition of binary filler leads to significant increase of strength, which we believe is caused by greater density of a structure and greater total area of the boundary layer between the filler particles and the binder and, as consequence, greater volume content of film phase in a composite. The obtained data can be compared to results of researches presented in works [31, 32, 35, 36] and describing polymer property change depending on filler's degree of filling and degree of dispersion.

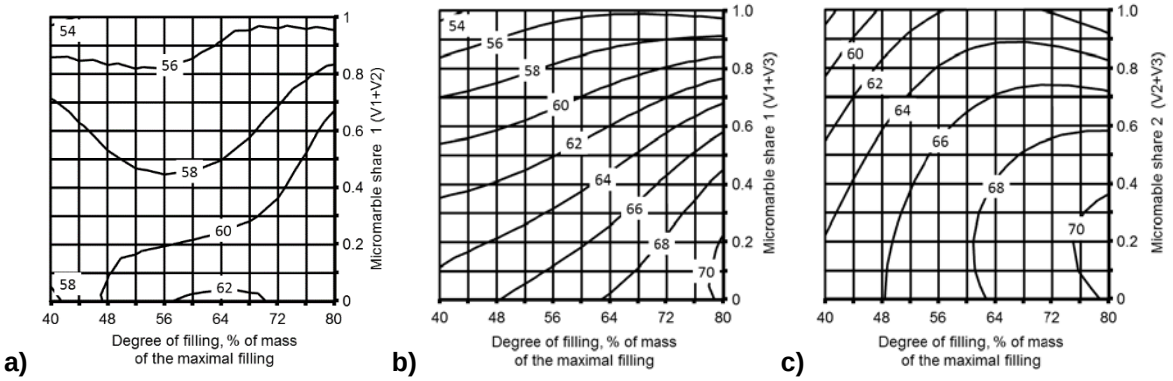

Figure 1. Isolines of epoxy composite ultimate strength change at compression (MPa) depending on degree of filling and micromarble fractions ratio: a) coarse (MKM1) and medium (MKM2); b) coarse (MKM1) and fine (MKM3); c) medium (MKM2) and fine (MKM3)

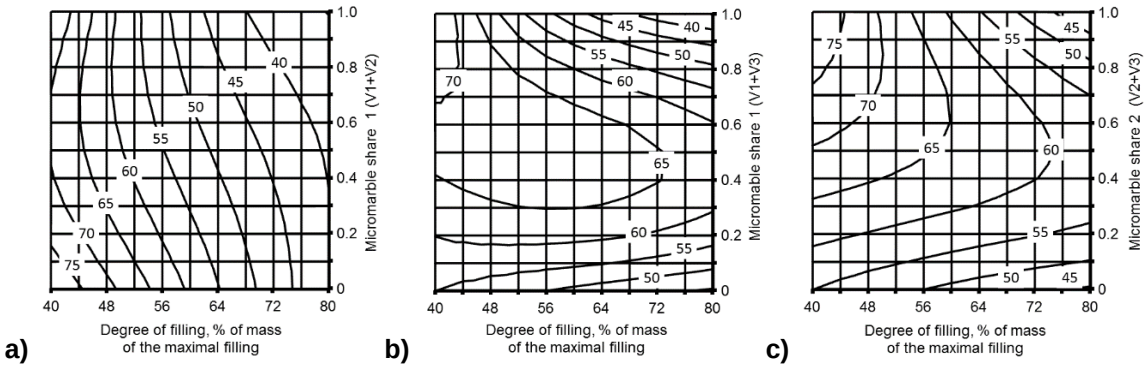

Figure 2. Isolines of epoxy composite ultimate bending tensile strength change depending on degree of filling and micromarble fractions ratio: a) coarse (MKM1) and medium (MKM2); b) coarse (MKM1) and fine (MKM3); c) medium (MKM2) and fine (MKM3)

It is known [31, 32] that increase of the total filler surface area leads to strengthening of the composite at compressing load impact only to a certain extent. This effect is caused by increase in polymer consumption for wetting of filler's grains, which results in formation of unit from them that are not wetted with the binder and are encapsulated in it. Compounds obtained from combination of micromarble of fine fraction and insignificant (up to 20 % of the total filler's mass) content of medium and coarse fractions at the maximal degree of filling feature higher compression strength than the composites that are filled only with micromarble of MKM3. Increase of strength when a polyfractional filler is used is also caused by the fact that with the greater density of a structure packing, where fine fraction micromarble particles fill the space between grains of the coarse filler ejecting binder's volume phase.

From the analysis of isolines of bending tensile strength, presented in Figure 2, it has been found out that composites containing micromarble of medium (MKM2) and coarse fractions (MKM1) features essential reduction of bending tensile strength – from 77 to 38 MPa at increase of the filling degree (Figure 2,a). High bending tensile strength obtained for these compounds with 40% degree of filling can be explained, first of all, by lack of filler's particles in the stretched section zone due to their complete setting in the process of sedimentation. In compounds having fine fraction micromarble (MKM3) of more than 80 % in MKM1+MKM3 combination, bending tensile strength with increased degree of filling almost does not change, which is proved by almost horizontal arrangement of isolines (Figure 2,b). In this case, the highest tensile strength at the maximal degree of filling was observed at equal shares of coarse and fine fillers (63 MPa).

According to [36], internal stresses do not depend on filler's dispersion; though, deformation energy caused by residual stresses is higher around coarse particles, which results in the fact that composites filled with micromarble of coarse (MKM1) and medium (MKM2) fractions have smaller strength at high degrees of filling than compounds containing micromarble of fine fraction (MKM3). Tests of composites filled with micromarble of fine fraction together with coarse or medium fractions led to a wide range of tensile strength values in bending, varying values depending on the share of each fraction (Figure 2,b,c). The best results were observed at equal ratio of shares; in this case, bending tensile strength does not exceed 10 % for the degree of filling from 40 to 80 %.

Three-component Gibbs-Roseboom diagrams based on simplexes in a form of regular triangles were used for a visual presentation of change in properties of filled epoxy composite materials at varying ratio of three micromarble fractions (MKM1, MKM2, MKM3). Figures 3 and 4 present triangular Gibbs-Roseboom diagrams of compression and tensile strength changes in bending plotted with Statistica 10.0.1011 software.

On the basis of triangular diagrams presented in Figure 3 it can be seen that the greatest compression strength values for all degrees of filling is observed with maximal content of MKM3 micromarble and combination of MKM2+MKM3 fillers, while MKM2 share does not exceed 25–40 %. Samples containing micromarble of coarse and medium fractions show that the optimal content of MKM2 makes about 60 % with the degree of filling of 40 and 80 % (Figure 3, a, c). The maximal compression strength has been observed in the compound filled with a combination of MKM2+MKM3 fillers in the ratio of 0.2:0.8 with the degree of filling of 80 %. Increase of micromarble coarse fraction in all investigated compounds led to considerable decrease of compression strength. The most obvious change of compression strength has been observed at variation of fine and coarse fractions of microcalcite, while

change of micromarble medium share in polyfractional filler has essentially smaller impact, especially when the degree of filling makes 60 % (Figure 3,b).

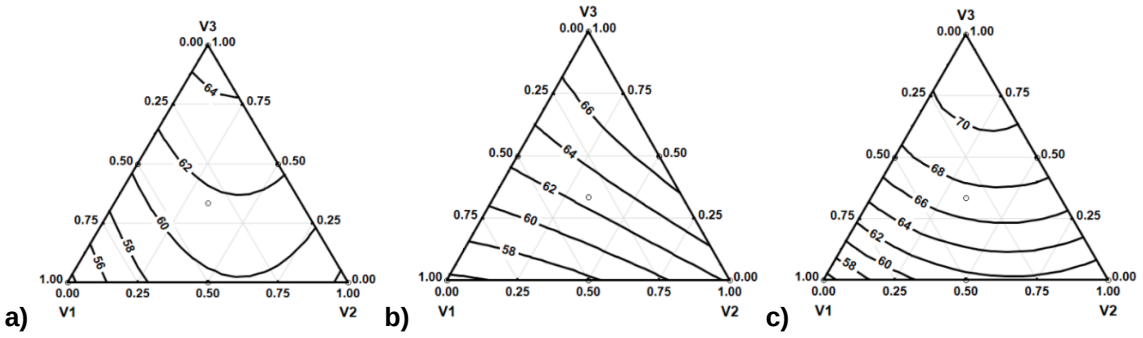

Figure 3. Triangular Gibbs-Roseboom diagrams of compression strength of filled epoxy composite materials (MPa) with degree of filling: a) 40%; b) 60%; c) 80% (MKM1 (V1) – coarse fraction (0.5÷1 mm); MKM2 (V2) – medium fraction (0.2÷0.5 mm); MKM3 (V3) – fine fraction (less than 0.2 mm))

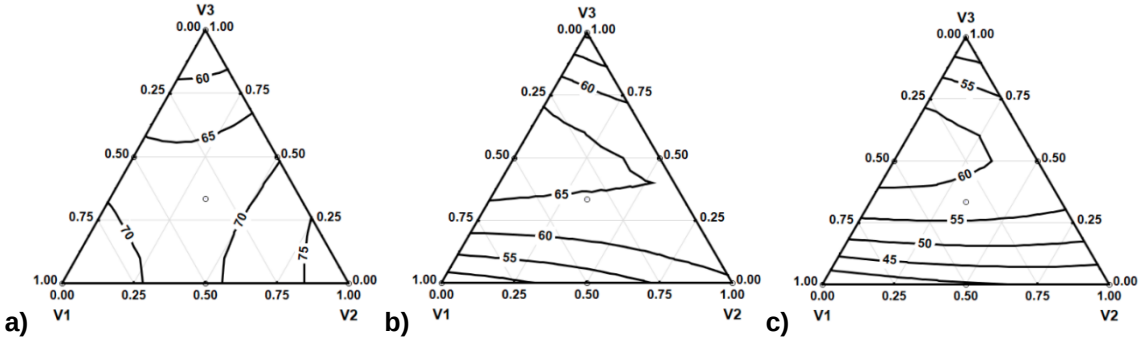

Figure 4. Triangular Gibbs-Roseboom diagrams of tensile strength of filled epoxy composite materials(MPa) with degree of filling: a) 40%; b) 60%; c) 80% (MKM1 (V1) – coarse fraction (0.5÷1 mm); MKM2 (V2) – medium fraction (0.2÷0.5 mm); MKM3 (V3) – fine fraction (less than0.2 mm))

The greatest bending tensile strength value has been observed in a compound with maximal share of micromarble of medium fraction at 40% degrees of filling (Figure 4,a). Generally, increase of degree of filling to 60 and 80% leads to reduction of bending tensile strength; thus, the optimal combination of strength and economic indicators is observed in composites filled with a combination of MKM1 + MKM3 micromarble. With these degrees of filling, medium fraction micromarble in polyfractional filler has minimal impact on strength properties.

4. Conclusions

Analysis of research results allowed us to draw the following conclusions:

1. Ultimate compression strength properties of epoxy composites can be enhanced from 10 to 40 % depending on dispersion and fractional composition of the added micromarble, as well as on the filling degree. The greatest ultimate strength reaching 70 MPa can be attained with application of the binary filler with increased calcium carbonate content of MSM3 fraction; the composite filled with microcalcite of fine and average fraction with 0.8:0.2 ratio and filling degree of 80 % features the optimal properties.

2. It has been determined that compounds with the filling degree of 40 % containing microcalcite of coarse or average fraction feature the greatest ultimate tensile strength, which is caused by forming a layer consisting mainly from unfilled epoxy polymer in a stretched area of the composite. Generally, increase of the filling degree leads to reduction of ultimate tensile bending strength. When polyfractional combinations of fillers containing micromarble with the fraction size of less than 0.2 mm and 0.5÷1 mm or 0.2÷0.5 mm tensile bending strength decreases does not exceed 10% for compounds with the filling degree of 80 % compared to unfilled compound (64 MPa), which proves economic feasibility of highly filled composite application.

3. Analysis of mathematical models of filled composite properties change depending on dispersion and fractional composition of the filler and filling degree presented in the form of isolines and triangular Gibbs-Rosenboom diagrams enabled us to reveal a compound featuring the best combination of economic and performance properties. Obtained composite contains a combination of MKM1+MKM3 fillers in the

ratio of 0.2:0.8 and has almost maximal compression and tensile strength in bending with the filling degree of 80 %.

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