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COMPOSITES THEORY AND PRACTICE

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STATISTICAL VERIFICATION OF STRENGTH PARAMETERS OF FIBROUS COMPOSITES

The paper analyzes the results of static tests conducted on fibrous composites. The strength characteristics were obtained for samples of different measuring length and different reinforcement architecture, produced by vacuum bag moulding and contact moulding using UDO® E-type unidirectional fibreglass fabric as the reinforcement and HAVELpol.2 polyester or LH 160 epoxy as the matrix. Then, the experimental data were analyzed by applying advanced statistical apparatus to verify the assumptions about the normal and logarithmic-normal distributions of strength. The statistical analysis is essential to correctly develop a model to determine the strength characteristics of fibrous composites based on the Markov chain theory.

Keywords: **composite, strength, size effects, statistical analysis**

STATYSTYCZNA WERYFIKACJA WYTRZYMAŁOŚCIOWYCH CHARAKTERYSTYK WŁÓKNISTYCH MATERIAŁÓW KOMPOZYTOWYCH

W pracy przedstawiono wyniki badań statycznych różnych próbek wykonanych z materiałów kompozytowych. Uzyskano charakterystyki wytrzymałościowe dla próbek wykonanych różnymi technologiami, o różnej bazie pomiarowej i odmiennych architekturach wewnętrznych wzmocnienia. Próbki formowano metodą worka próżniowego i metodą kontaktową. Użyto wzmocnienia z tkaniny szklanej jednokierunkowej UDO® typu E z osnową poliestrową (HAVELpol.2) i epoksydową (LH 160). Dokonano analizy otrzymanych wyników. W części drugiej zastosowano zaawansowany aparat statystyczny do weryfikacji przyjętego założenia o normalnym i logarytmiczno-normalnym rozkładzie wytrzymałości badanych próbek. Ta część pracy była niezbędna do zbudowania poprawnego modelu pozwalającego uzyskać charakterystyki wytrzymałościowe kompozytu opartego na teorii łańcuchów Markowa.

Słowa kluczowe: **kompozyt, wytrzymałość, efekt skali, analiza statystyczna**

INTRODUCTION

A frequent problem discussed in the literature on fibrous composites is the large discrepancy between the strength characteristics, which results from the application of different methods of production and analysis as well as the non-uniformity of the material structure and the size effect.

Strength data can only be used in engineering calculations and theoretical analysis if they are stable and repeatable. It is thus vital to apply the latest, most advanced statistical methods able to handle a large number of data. A tool suitable for this purpose is the model based on the Markov chain theory presented in [1]. Problems such as the free edge quality related to the cutting technology, the failure of the sample due to load and the size effect are discussed in [2, 3] and [4], respectively.

To check the universality of the statistical analysis used in this study, the samples differed in their layer arrangement and matrix material. It was possible to assess the effect of the composite architecture on the interlaminar failure [5, 6] resulting from high tangential stresses [7]. By analyzing the bundles of fibres - fibres submerged in the matrix - despite them being of different measuring lengths, we can determine the size effect for different matrix materials [8].

The influence of the process parameters (production and cutting technologies), size effect and sample failure will not be discussed here; they are described in [9, 10]. The strength characteristics obtained in the partial analysis were used as databases to perform statistical studies. The methodology is presented in [11, 12].

MATERIALS AND METHOD

Two types of samples were used. One composite was produced using symmetrically arranged [0/90/0/90] fibreglass fabric (UDO® E type) with an average unit thickness of 0.49 mm as the reinforcement and polyester resin (HAVELpol.2) as the matrix. The composite plates were produced by vacuum moulding at HAVEL Composites. The material was cut into samples along the fibres using a high pressure water jet (approx. 4000 bars) containing fine abrasive material (Garnet #80) at a rate of 1.5 m/min. Two measuring lengths were used: 80 and 150 mm.

The other composite with a non-symmetric structure [0/45/0] was produced by contact moulding using fibreglass fabric (UDO® E type) as the reinforcement and epoxy resin (LH 160) as the matrix. The cutting method was the same and the sample measuring lengths were also 80 and 150 mm.

The properties of an elementary fibre bundle were determined using samples in the form of fibre bundles separated from the fabric (UDO® ES) stabilized with polyester resin (with measuring lengths of 120 and 250 mm) and epoxy resin (with measuring lengths of 120, 250 and 450 mm). The specimens had shoulders at both ends to enable firm gripping by a universal testing machine. Table 1 contains all the data concerning the technologies of sample production.

TABLE 1. **Process parameters for two different methods of moulding of polymer-matrix composite**
TABELA 1. **Parametry procesu dla dwóch różnych metod formowania kompozytu polimerowego**

Technology (symbol - layer arrangement)	Fibre-glass fabric	Resin	Hardener	Moulding time	Gel time	Additional heating
vacuum bag moulding (HPOL - [0/90/0/90])	Udo UD ES 500/300 51.0%	HAVEL pol.2	2% Butanox M50	approx. 24 h	30 min (T=18÷20°C)	50°C (6 h)
manual moulding (HEPOXY - [0/45/0])		LH 160	25% H 147	24 h	60 min (T=22÷23°C)	60°C (16 h)

The dimensions of the specimens were: length - 250 and 180 mm; width - 25 mm and thickness - ranging from 1.5 to 1.6 mm and from 1.92 to 1.98 mm for the symmetric and non-symmetric laminates, respectively, according to the DIN-EN ISO 527 standard.

The samples were denoted by 'HPOL_A(B)-xxxx' and "HPOL-C1(C2)-xxxx", where 'HPOL' stands for a polyester laminate with a layer arrangement of [0/90/0/90] and different measuring lengths (L_{BP}): A = 120 mm and B = 250 mm, while "HPOL-C1(C2)-xxxx" represents fibre bundles submerged in resin with measuring lengths - Cl = 120 mm, C2 = 250 mm - with the sample number 'xxxx'), and 'HEPOXY_A(B)-xxxx' and "HEPOXY-C(C1;C2;C3)-xxxx", where 'HEPOXY' is an epoxy-matrix composite laminate with a layer arrangement of [0/45/0] with different measuring lengths: A = 120 mm and B = 250 mm, and "HEPOXY_C(C1;C2;C3)-xxxx" with different L_{BP} of the fibre bundles: Cl = 120 mm, C2 = 250 mm and C3 = 450mm and the sample number 'xxxx'.

The static tensile strength tests were conducted on the composites and bundles of fibres embedded in resin using MTS 5T 50 kN and Zwck/Roell TC-FR2.5TN.DO9 machines at the laboratory of the Institute of Polymer Mechanics of the Riga Technical University, Latvia. The MTS 5T 50 kN testing machine was equipped with an MTS Flex Test SE. The loads were measured using HBM 1-XY91-6/350 rosettes (consisting of two perpendicular tensometric sensors) and single HBM 1-XY91-6/350 gauges all with the same measuring length of 6 mm and a nominal electrical resistance of 350 Ohms.

A strain rosette and a strain gauge were placed at the opposite ends of each sample. The strain, load and displacement were registered by means of an HBM Spider 8 equipped with a Catman controller. The tests were performed at a head displacement rate of 2 and 1 mm/min, depending on the sample measuring length. The axial stress was calculated as a ratio of the force applied to the average cross-sectional area of the sample, whereas the Poisson ratio (μ_{xy}) was determined from the lateral strain/axial strain curve. The average static strength ($\sigma_{stat.}$) of the polymer-matrix composite determined in a static tensile test was used to plot the S-N curve.

RESULTS AND DISCUSSION

From the experimental analyses found in the literature, it is clear that there is a large scatter of strength characteristics of fibrous composites. The processing of experimental results is essential, mainly to determine and check the hypotheses of statistical distributions of static strength (S_{stat}) (the mean and standard deviation) for bundles of fibres submerged in resin and composite samples with different L_{BP}. The results are shown in Tables 2 and 3.

In this study, it is assumed that the distributions of strength are normal and logarithmic-normal. This approach requires analytical justification. The statistical analysis was performed using the OSPPT criterion [13, 14]. For the level of significance $\alpha = 0.05$, the methodology was not rejected because the OSPPt statistics did not exceed the criterion of the Calfa hypothesis (OSPPt < Calfa). The analysis results are shown in Figure 1, which is a graphical representation of the data in Tables 2 and 3.

TABLE 2. **Statistical analysis of static strength of polyester-matrix composite and fibre bundle samples with different L_{BP}**

TABELA 2. **Statystyczna analiza wytrzymałości statycznej wiązek włókien i próbek KW na osnowie poliestrowej z różną L_{BP}**

Strength of composite components in samples with different L_{BP}	OSPPt	Calfa	Mean	Standard deviation	Number of samples
Polyester-matrix composite					
Logarithmic-normal distribution of $S_{stat.}$ [MPa]					
S_{HPOL_C1}	0.14341	0.25531	6.5849	0.19681	33
S_{HPOL_C2}	0.25403	0.29532	6.4669	0.17618	29
S_{HPOL_A}	0.15542	0.29650	6.0005	0.09145	23
S_{HPOL_B}	0.16584	0.32713	6.0861	0.082331	18
Normal distribution of $S_{stat.}$ [MPa]					
S_{HPOL_C1}	0.2006	0.25518	737.7397	146.2042	33
S_{HPOL_C2}	0.22296	0.29463	652.6013	110.0079	29
S_{HPOL_A}	0.16221	0.29316	405.2347	37.0302	23
S_{HPOL_B}	0.14487	0.32676	441.0872	35.9297	18

* $S_{HPOL\_A,\ HPOL\_C2}$ - strength of composite samples (HPOL_A) and fibre bundles submerged in resin (HPOL_C2) with L_{BP}=250mm;
**$S_{HPOL\_B,\ HPOL\_C1}$ - strength of composite samples (HPOL_B) and fibre bundles submerged in resin (HPOL_C1) with L_{BP}=120mm

TABLE 3. **Statistical analysis of static strength of epoxy-matrix composite and fibre bundle samples with different L_{BP}**

TABELA 3. **Statystyczna analiza wytrzymałości statycznej wiązek włókien i próbek KW na osnowie epoksydowej z różną L_{BP}**

Strength of composite components in samples with different L_{BP}	OSPPt	Calfa	Mean	Standard deviation	Number of samples
Epoxy-matrix laminate composite					
Logarithmic-normal distribution of $S_{stat.}$ [MPa]					
S_{HEPOXY_C1}	0.27439	0.31162	6.59	0.14987	20
S_{HEPOXY_C2}	0.18322	0.26505	6.5439	0.13653	31
S_{HEPOXY_C3}	0.24046	0.34601	6.4111	0.076333	15
S_{HEPOXY_A}	0.21924	0.29487	6.0758	0.065701	23
S_{HEPOXY_B}	0.14194	0.30321	6.1891	0.059936	21
Normal distribution of $S_{stat.}$ [MPa]					
S_{HEPOXY_C1}	0.33237	0.33977	573.0166	89.3891	20
S_{HEPOXY_C2}	0.23027	0.26217	621.3026	86.9879	31
S_{HEPOXY_C3}	0.25157	0.34707	610.2328	46.797	15
S_{HEPOXY_A}	0.20613	0.30016	436.0709	28.3748	23
S_{HEPOXY_B}	0.1553	0.30878	487.5612	29.4538	21

* $S_{HPOL\_A,\ HPOL\_C2}$ - strength of composite samples (HPOL_A) and fibre bundles submerged in resin (HPOL_C2) with L_{BP} = 250 mm
** $S_{HPOL\_B,\ HPOL\_C1}$ - strength of composite samples (HPOL_B) and fibre bundles submerged in resin (HPOL_C1) with L_{BP} = 120 mm
*** S_{HPOL_C3} - strength of fibre bundles submerged in resin (HPOL_C3) with L_{BP} = 450 mm

a)

b)

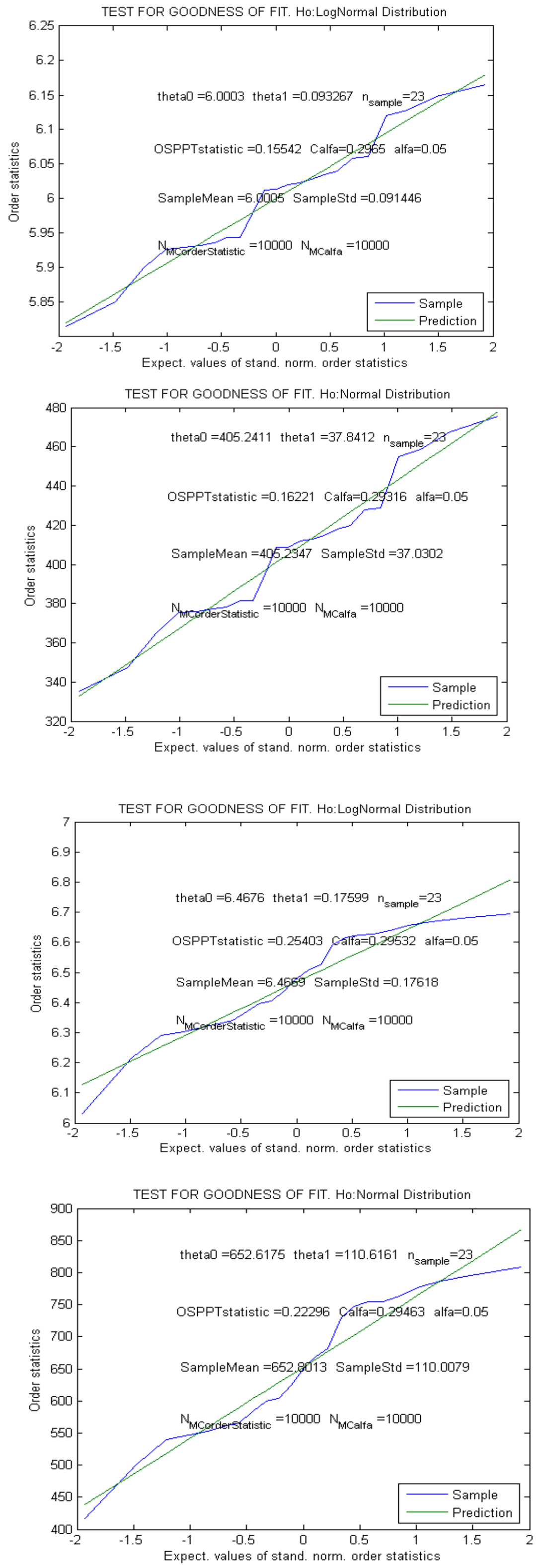

Fig. 1. Verification of hypotheses of normal and logarithmic-normal distributions of strength for (a) polyester-matrix composite samples and (b) fibre bundles submerged in polyester resin; L_{BP} = = 250 mm

Rys. 1. Sprawdzenie hipotezy normalnego i logarytmiczno-normalnego rozkładu wytrzymałości: próbek (a) i wiązek włókien usztywnionych w żywicy (b) na osnowie poliestrowej z LBP = 250 mm

The graphical representation of the changes in strength for composites or fibre bundles in the function of empirical density $\hat{P}_i$ enables us to assess the rate and range of these changes. The value of $\hat{P}_i$ [14] was determined from the following relationship:

$$\hat{P}_i = \frac{i - 1/3}{n + 1/3} \quad (1)$$

where:
i - order number of expected value in an ordered set of samples
n - total set size.

Figure 2 shows the relationship $\hat{P}_i$–ln(S) for a symmetric polyester-matrix composite and fibre bundles. It is clear that the strength of the fibre bundles submerged in resin is much higher (up to 80%) than that of the symmetric laminate composite.

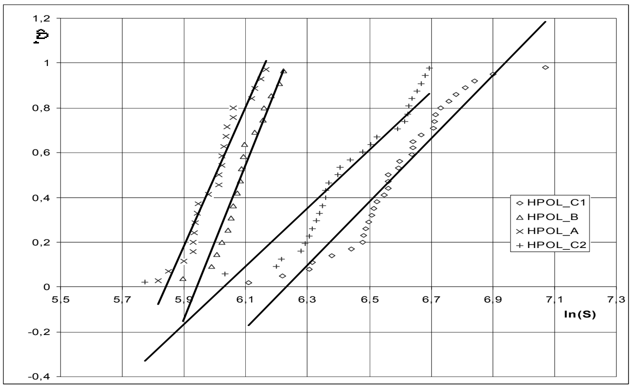

Fig. 2. Comparing experimental data with statistical analysis data for HPOL composite and fibre bundle samples

Rys. 2. Porównanie eksperymentalnych rezultatów z danymi uzyskanymi z analizy statystycznej dla kompozytu i wiązki włókien (próbki HPOL)

As can be seen, the range of strength for the fibre bundle samples is much larger than that of the polyester composite. This is due to the fact that it is never possible to produce two identical active fibre bundles. Each bundle has a different number of broken fibres, which do not transmit load.

Reducing the measuring length by half for the same series of samples, i.e. from 150 to 80 mm, caused an increase in the average strength of 10% (Table 2, column 4). Defects are more likely to occur in longer samples than in shorter ones. Similarly, the average strength of the fibre bundles embedded in resin increased by 12% (Table 2). This is linked to the so-called size effect. Another observation is that the deviations of strength from the regression line are larger for the fibre bundle samples than for the composite samples, which has been explained above.

There are certain differences in the relationship $\hat{P}_i$–ln(S) for the samples of the non-symmetric epoxy-matrix composite and the fiber bundles submerged in the same resin, as illustrated in Figure 3. It can be concluded that, like for the polyester matrix, the strength of the non-symmetric composite samples is about 30 and 15% smaller than that of the fibre bundles for samples with a measuring length of 250 and 120 mm, respectively. The ranges of scatter of the results for the fibre bundles and the non-symmetric composite with an epoxy resin matrix are similar to those obtained for the symmetric polyester-matrix composite. Figure 3 shows that the regression line for the HEPOXY_C3 samples does not coincide with the regression line for the HEPOXY_C1 and HEPOXY_C2 samples. No explanation has been found for these differences, except for the size effect and the production technology. The instability of changes in the strength characteristics testifies that the manual moulding method (the non-symmetric epoxy-matrix composite) does not guarantee repeatability, which is not the case with the strength of the samples produced by vacuum moulding (Fig. 2).

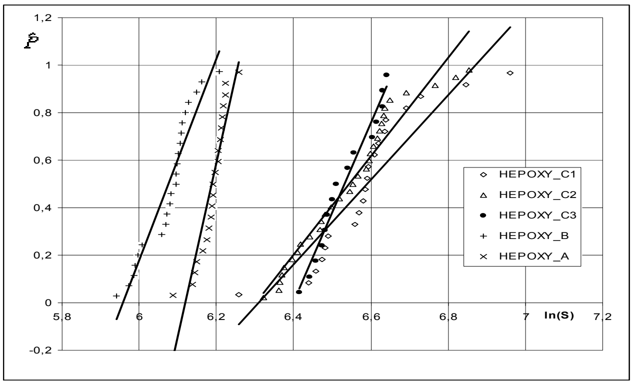

Fig. 3. Comparing experimental data with statistical analysis data for HEPOXY composite and fibre bundle samples

Rys. 3. Porównanie eksperymentalnych rezultatów z danymi uzyskanymi z analizy statystycznej dla kompozytu i wiązki włókien (próbki HEPOXY)

The quality of the composite production technology is assessed in accordance with the literature data and the weakest link theory based on the Weibull distribution function [15]:

$$F(x) = \exp[-(\frac{x - x_u}{x_0})^m] \quad (2)$$

or as the probability of survival $P_s(\sigma)$ described by the function:

$$P_s(\sigma) = \exp[-V(\frac{\sigma - \sigma_u}{\sigma_0})^m] \quad (3)$$

where: x_u - positional parameter; x_0 - scale parameter; σ_0 - characteristic strength being the value of strength per unit volume; σ_u - threshold value below which failure does not occur; m - shape parameter (so-called Weibull modulus) determined by the relationship $m \cong 1.2\frac{\bar{x}}{\bar{\sigma}}$ (there are many statistical procedures for determining this module); $\bar{\sigma}$ - standard deviation; $\bar{x}$ - arithmetic mean for a given population of samples.

From the experiments and the information included in [11-12, 16], we can conclude that the strength characteristics of laminate composites should be analyzed using hypotheses with normal and logarithmic-normal distributions. The distribution of the experimental data for the symmetric polyester-matrix composite is shown in Figure 4. The upper and lower confidence limits as well as the average confidence are indicated. From this Figure it is clear that for the shorter measuring length (L_{BP} = 120 mm), the average strength of the composite is not affected by a slight change in the sample thickness. However, for the almost 50% longer samples, where the volume is higher, even a very small change in the thickness may influence the sample strength. The longer the sample is, the more imperfections it contains, and the more likely it is to fail. The change in strength was not greater than 15%.

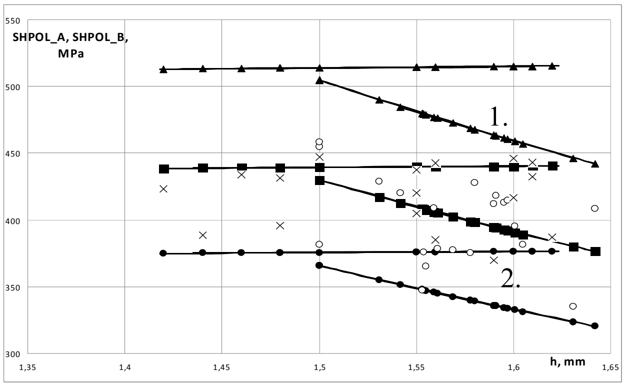

Fig. 4. Confidence limits with average strength of polyester-matrix fibrous composite for (1) L_{BP} = 120 mm and (2) L_{BP} = 250 mm

Fig. 4. Granice ufności średniej wytrzymałości KW na osnowie poliestrowej z L_{BP}: 120 mm (1) i 250 mm (2)

The correctness of the proposed confidence intervals for the average strength of the polyester- and epoxy-matrix composites for samples with different L_{BP} was verified using the Kolmogorov-Smirnov criterion (Table 4), that is from a distribution characterized by a maximum difference in the experimental [17, 18] F*(x) and theoretical F(x) quantities of the distribution function [9, 13]:

$$D_n = \sup\left|F^*(x) - F(x)\right| = \max\left|D_n^+, D_n^-\right| \qquad (4)$$

where: $D_n^+ = \max\limits_{1<i<n}(\frac{i}{n} - F_i)$ - upper limit; $D_n^- = \max\limits_{1<i<n}(F_i - \frac{i-1}{n})$ - lower limit; *ni* - number of meanings in the set; F(x) - distribution function.

If the obtained value of D* (D - see Table 4, column 4) does not satisfy any of the conditions given in [13], then the distribution assumed for *n* experimental data is not correct. In the case considered, the assumed condition was satisfied.

CONCLUSIONS

- It has been found that the strength and scatter of the strength for fibre bundles submerged in polymer resin are much larger than those obtained for the polymer-matrix composite.
- It has been confirmed that the hypotheses with normal and logarithmic-normal distributions of normal stresses (OSPPT criterion for level of confidence α = 0.05) are correct for the samples of fibre bundles embedded in resin and the composite samples with different L_{BP}.
- Using the proposed statistical analysis, we can observe changes in the static strength, including phenomena resulting from a change in the measuring length (size effect, failure).
- The instability of changes in the strength characteristics for samples produced by contact moulding indicates that this manual moulding method (non-symmetric composite) does not guarantee repeatability of results, which is not the case when vacuum moulding is used.
- The statistical method of analysis used in this study requires further verification, which should involve the diversification and extension of databases (more experimental data), taking account of the quality and architecture of the material; the method may then be useful when developing models for laminate composites according to the Markov chain theory.

TABLE 4. **Parameters of statistical analysis of average strength of polyester- and epoxy-matrix fibrous composites determined for samples with different L_{BP}**

TABELA 4. **Parametry obróbki statystycznej średniej wytrzymałości KW na osnowie poliestrowej i epoksydowej z różnymi** L_{BP}

	Strength of samples with different L_{BP}	Number of samples n	D*	Mean	Dispersion	Standard deviation	Kolmogorov-Smirnov criterion [13]
$S_{stat.}$ of polyester-matrix laminate composite							
1	S_{HPOL_A}	23	0.125871	6.0005	0.007999	0.09145	0.604629 < 0.99
2	S_{HPOL_B}	18	0.112625	6.0861	0.006402	0.082331	0.469043 < 0.99
$S_{stat.}$ of epoxy-matrix laminate composite							
3	S_{HEPOXY_A}	23	0.113879	6.0758	0.003421	0.065701	0.516735 < 0.99
4	S_{HEPOXY_B}	21	0.135569	6.1891	0.004986	0.059936	0.60825 < 0.99

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