MECHANICAL PROPERTIES OF POLYPROPYLENE FILLED WITH MAGNESIUM HYDROXIDE

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ABSTRACT

The influence of magnesium hydroxide as filler on the structure and mechanical properties of polymeric composite with polypropylene matrix was studied, with respect to the surface modification of the filler grains, and to the degree of filling. Having combined the factors mentioned above 7 kinds of experimental materials were obtained. The resistance of the materials to impact loading was examined and dynamic fracture toughness K_{Jd} of materials was determined. The microfractography of fracture surface of the samples for testing K_{Jd} were carried out. The experimental data were processed by means of regression analysis. The regression functions describing the dependencies of mechanical properties on temperature and coefficients for these functions were found. Subsequently 3D regression analyses of dependencies of mechanical properties on temperature and amount of filler were done.

KEYWORDS

Mechanical properties, composite, polypropylene, magnesium hydroxide, temperature, impacts properties, fracture toughness, 2D- regression analysis, 3D-regression analysis.

1. INTRODUCTION

Polypropylene (PP) is - due to relatively low cost and universal properties - one of the materials, which are most often used commercially in technical applications **[1]**. PP is also often used as a thermoplastic matrix of composites and its importance has been grown in the last few years **[2]**. Moreover, PP and its composites can be easily recycled **[3]**. Correct choice of composite for specific application might be based on knowledge of the properties of the material and their dependencies on the type of matrix and on the character and amount of the filler. In this contribution the attention is therefore paid to determination and subsequent statistical processing of the values of dynamic fracture toughness of composite with PP matrix filled with magnesium hydroxide.

2. EXPERIMENTAL MATERIALS

2.1 Matrix

Matrix of studied composites is made of copolymer PP SHAC KMT 6100 (produced by Shell International Chemical Co. Ltd.) with density 0,903 g.cm^{-3} and ITT 4,0 dg.min^{-1} (ISO 1133; 2,16 kg /230°C). This material is used for injection moulding of different products with middle toughness **[4].** Distribution of molecular weights is demonstrated on **Fig. 1**.

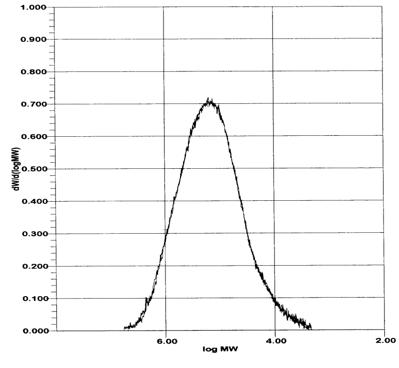
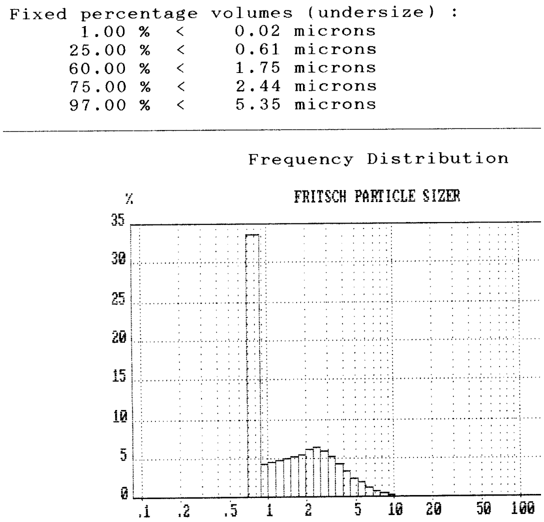


Fig.1: Distribution of molecular weights of copolymer PP SHAC KMT 6100

2.2 Filler



Magnesium hydroxide MAGNIFIN H 10 was used as filler without surface modification, resp. with surface modificated with 2,5w.% of stearin ASTRA.

On electron scanning microscope JEOL JXA-840A was observed that the particles of filler are of shape of thin hexagonal plates.

The result of particle size analysis is shown on **Fig. 2.**

Fig. 2: Results of the particle size analysis

2.3 Resulting composite

Having combined the factors of surface modification of the filler grains and of the degree of filling 7 kinds of experimental materials were obtained (**Tab. 1**). Material **S** is pure PP. Composites **S2**, **S4** and **S6** are of PP + MAGNIFIN H10 with surface modification, types **N2**, **N4** and **N6** are of PP + MAGNIFIN H10 without surface modification. Number in mark of material represents degree of filling in ten %.

Material	S	S2	S4	S6	N2	N4	N6
Filler	---	MAGNIFIN H 10 modificated			MAGNIFIN H 10 unmodificated		
[%] of filling	0	20	40	60	20	40	60

Tab. 1: Experimental materials

All kinds of composites were prepared on KO KNEATER BUSS MDK 46 and experimental samples manufactured by means of injection moulding machine Battenfeld BA 750-220 in Polymer Institute Brno, spol. s r.o.,.

Krystalinity of composites was determined by means of DSC (differential scanning calorimetry). Results of DSC analysis and krystalinity values for all experimental material are mentioned in **Tab. 2** and shown on **Fig. 3**.

Material	Heat of fusion $[J.g^{-1}]$	krystalinity [%]
S	76,07	42,74
S2	55,43	31,14
S4	40,07	22,51
S6	25,35	14,24
N2	56,68	31,84
N4	40,33	22,66
N6	25,11	14,11

Tab. 2: Heat of fusion and krystalinity

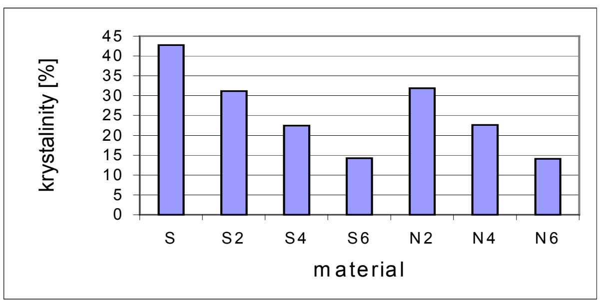


Fig. 3: Krystalinity of materials

3. DETERMINATION OF DYNAMIC FRACTURE TOUGHNESS

The tests of dynamic fracture toughness were carried out on the instrumented Charpy impact tester PSW 300 E/MFL with total energy 150 J. The values of dynamic fracture toughness K_{Id}, resp. values of J-integral J_{Id}, were determined by method **[5]**. The samples 4×10×60 mm were used unnotched for determination of dynamic Young's modulus E_d and dynamic yield stress σ_d, resp. notched with a razor blade up to 4 mm depth for determination of K_{Id}, resp. J_{Id}. The tests were carried out under the temperatures from –30°C to +70°C and speed of loading 0,95 m.s^{-1}. Values K_{Id}, resp. J_{Id}, were converted to equivalent K_{Jd}.

4. MATEMATICAL PROCESSING OF THE EXPERIMENTAL RESULTS

The experimental data were processed by means of statistical methods, especially by regression analysis. The regression functions describing the dependencies of studied mechanical properties on temperature and confidence limits for 95% reliability were found by means of program **TableCurve 2D**. Regression coefficients for these functions and adjusted coefficients of correlation as a measure of availability of regression functions were determined. Subsequently 3D regression analyses of dependencies of mechanical properties on temperature and amount of filler were done by means of program **TableCurve 3D** with results of founded values of regression coefficients and adjusted coefficients of correlation. Founded types of regression functions are mentioned in **Tab. 3**.

Dependence	Type of regression function
$E_d = \mathrm{f}(T)$	$E_d = a\exp(-T/b)$
$\sigma_d = \mathrm{f}(T)$	$\sigma_d = a\exp(-T/b)$
$K_{Jd} = \mathrm{f}(T)$	$K_{Jd} = \exp(a + bT + cT^2)$
$K_{Jd} = \mathrm{f}(T,\%)$	$K_{Jd} = \exp[(a + b.\%) + (c + d.\%)T + (e+f.\%)T^2]$

Tab.3: Founded types of regression functions

5. DISCUSSION

5. 1 Unnotched samples

The regression function describing the dependencies of dynamic Young's modulus E_d and dynamic yield stress σ_d on temperature T was found. Calculated regression coefficients and adjusted coefficient of correlation for all materials are mentioned in **Tab. 4** and **Tab. 5**.

	a	b	adj r^2
S	1436.82190	64.39657	0.911808
N 2	2008.12438	62.00668	0.94696
N 4	2273.03831	73.73931	0.86509
N 6	4241.13107	59.68575	0.84851
S 2	1655.52241	74.98381	0.79086
S 4	1977.36308	77.84081	0.82373
S 6	2403.54145	73.55484	0.78458

Tab. 4: Regression coefficients and adjusted coefficient of correlation for $E_d = \mathrm{f}(T)$

	a	*b*	adj r^2
S	71.08703	81.20594	0.89358
N 2	73.54473	83.92646	0.88024
N 4	68.67519	109.46136	0.83596
N 6	63.33667	153.29412	0.64589
S 2	63.83133	100.16735	0.72448
S 4	63.46544	89.16599	0.82953
S 6	58.09111	77.71935	0.76327

Tab. 5: Regression coefficients and adjusted coefficient of correlation for σ_d = f (T)

Generated values of E_d and σ_d were computed from regression functions. These values were further used for determination of K_{Id}, resp. J_{Id}.

5. 2 Notched samples

The regression function describing the dependence of dynamic fracture toughness K_{Jd} on temperature T was found. Calculated regression coefficients and adjusted coefficient of correlation for every material are mentioned in **Tab. 6**.

	a	*b*	*c*	adj r^2
S	0,80973	-0,00079699	8,7165EXP-5	0,00477
N 2	0,89827	-0,00013760	0,00052143	0,74906
N 4	1,07379	-0,00303021	0,00021573	0,22620
N 6	0,67958	-0,00553639	2,21114EXP-5	0,33126
S 2	1,31028	0,00453915	-0,00016316	0,48283
S 4	1,33184	0,00813519	-0,00021748	0,69132
S 6	0,55458	-0,00688093	0,00010351	0,25315

Tab. 6: Regression coefficients and adjusted coefficient of correlation for K_{Jd} = f (T)

Convenience of founded regression function to experimental values of K_{Jd} for material a) **S**, b) **N4**, c) **S4** d) **S6** follows from **Fig. 5**. It is clear, that appreciation of the influence of type and amount of filler on K_{Jd} is not very simple. The main problem seems to be wide dispersion of experimental data, which is probably caused by the fact, that K_{Jd} is not directly measured, but computed from much input. Values of K_{Jd} then can be influenced by statistical character of these individual inputs. But it can be seen that founded regression function agrees with experimental data.

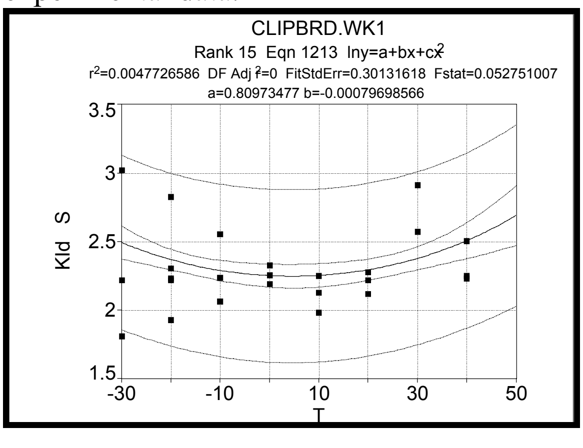


Fig. 5 a) K_{Jd} = f(T) for material **S**

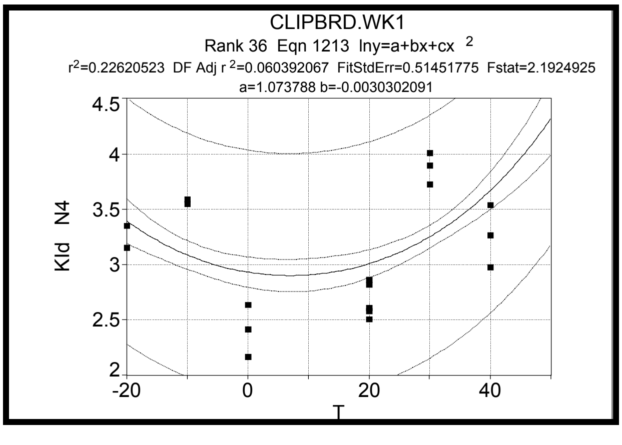


Fig. 5 b) K_{Jd} = f(T) for material **N4**

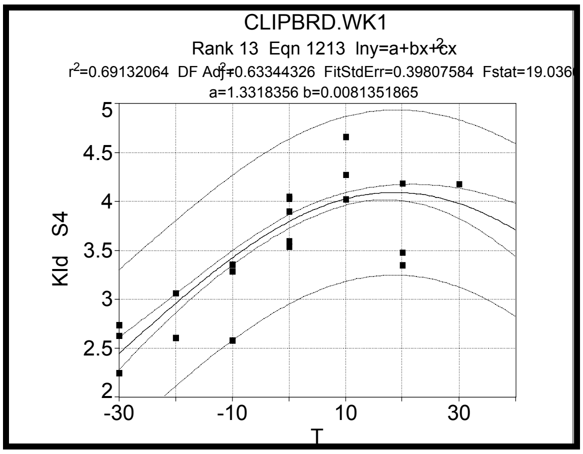


Fig. 5 c) K_{Jd} = f(T) for material **S4**

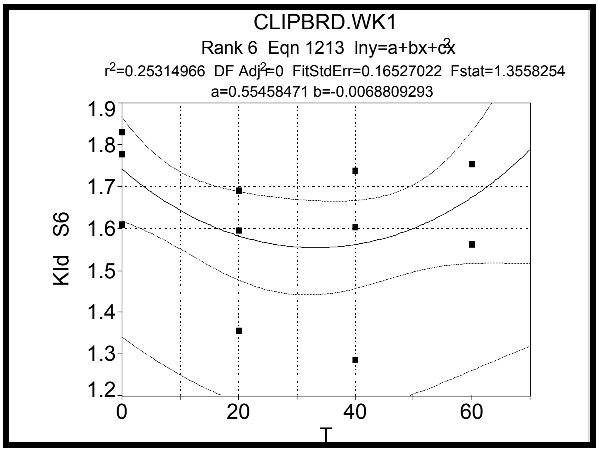


Fig. 5 d) K_{Jd} = f(T) for material **S6**

Subsequently another analysis was done – values K_{Jd} generated from regression functions for composites filled with increasing amount of one type of filler were compared (**Fig. 6 a,b**).

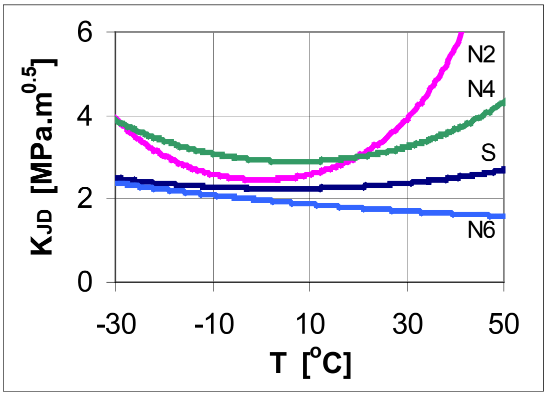


Fig. 6 a) Generated K_{Jd} for increasing amount of unmodified filler

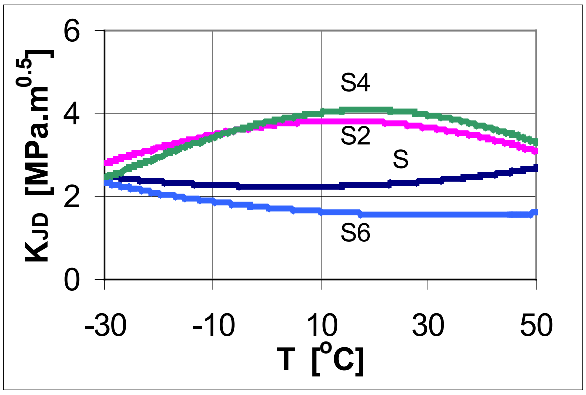


Fig. 6 b) Generated K_{Jd} for increasing amount of modified filler

Further, K_{Jd} generated for the same amount of different kind of filler was compared (**Fig.7a,b,c).**

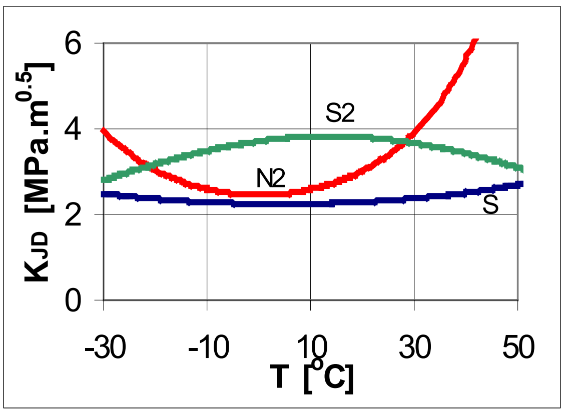


Fig. 7 a) Generated K_{Jd} for 20% of unmodified (N2) and modified (S2) filler.

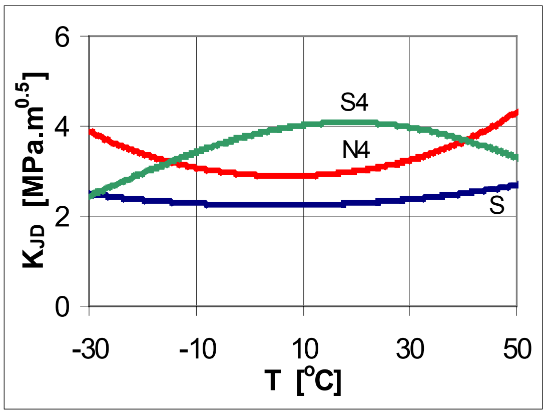


Fig. 7 b) Generated K_{Jd} for 40% of unmodified (N4) and modified (S4) filler

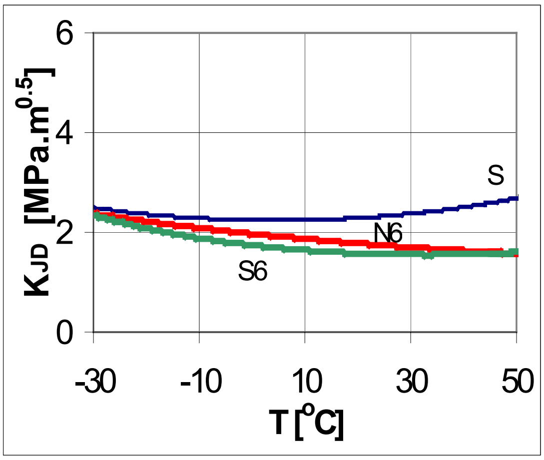


Fig. 7 c) Generated K_{Jd} for 60% of unmodified (N6) and modified (S6) filler.

It can be seen, that:

- 20% and especially 40% of both unmodified (**N2**,**N4**) and modified (**S2**, **S4**) filler increase K_{Jd} of composites above the value of pure PP (**S**); the influence of unmodified filler is not as expressive as the influence of modified filler;
- Curves S2 and S4 exhibit a maximum, which represents the optimal working temperature for the composite; curves N2 and N4 exhibit a minimum,
- 60% of both kinds of filler decrease K_{Jd} of composites under the values of pure PP.

Complex influence of type and amount of filler on K_{Jd} values was studied by means of 3D regression analysis. Regression function $K_{Jd} = \exp[(a + b.\ \%) + (c + d.\ \%)\ T + (e + f.\ \%)\ T^2]$ was determined as the best and regression coefficients were computed for unmodified filler (**N**), resp. for modified filler (**S**). Results are shown in **Tab. 7** and in **Fig. 8 a,b**.

	a	*b*	*c*	*d*	*e*	*f*	adj r^2
N	0.81758	0.01960	0.0004958	-	3.6276e-06	-1.2784e-	0.68339
S	0.81069	0.03767	0.0005252	-	-3.3508e-	-8.5032e-	0.77893

Tab. 7: Regression coefficients and adjusted coefficient of correlation for $K_{Jd} = f(T, \%)$

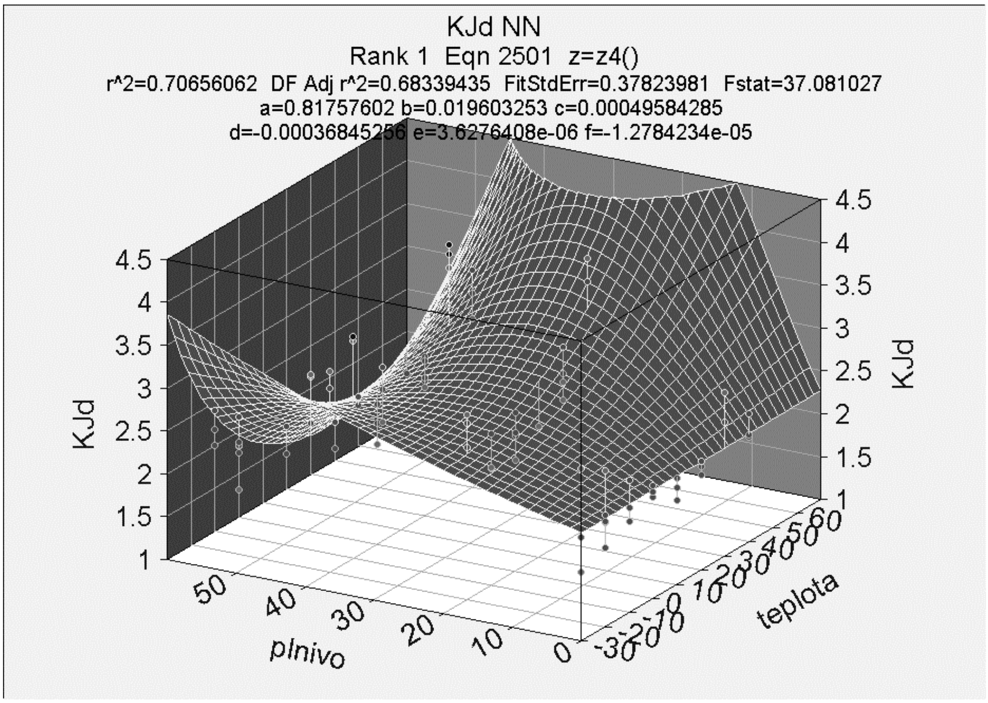


Fig. 8 a: Generated $K_{Jd} = f\ (T, \%)$ for unmodified filler (**N**)

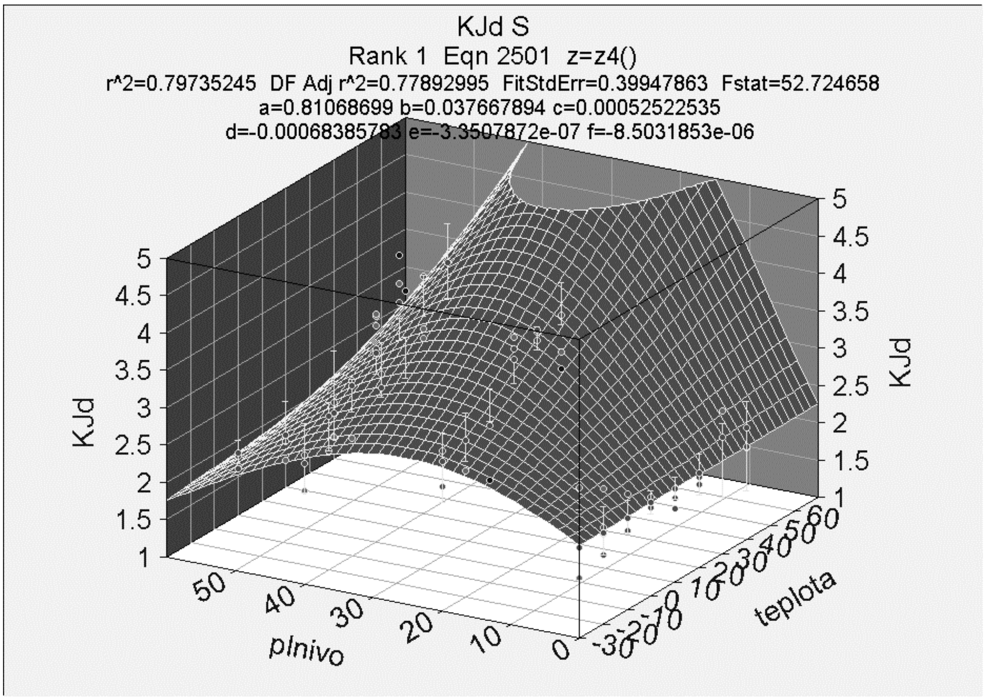


Fig. 8 b: Generated $K_{Jd} = f\ (T, \%)$ for modified filler (**S**)

6. SUMMARY

The results of testing of dynamic fracture toughness K_{Id}, resp. values J_{Id} confirm positive influence of 20 % and especially 40 % of MAGNIFIN H10 with surface modification on the mechanical properties of the composite with PP matrix. 60% of the mentioned filler decrease toughness of composites in similar manner as 60% of MAGNIFIN H10 with unmodified surface.

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