The influence of the powder additive upon selected mechanical properties of a composite

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Abstract. Five series of samples were prepared, each batch consisting of 20 epoxy-resin items, CES R70 with CES H72 hardener and 4 types of powder additives: montmorillonite (MMT), Expancell d25 Microsphere (microsphere), nanocarbon (NanoC) and silicon carbide (SiC). All the additives were added in quantities corresponding to 15% of the total volume share of the material. The test samples for tensile test and three-point transverse bend test were made in accordance with the standards: PN-EN ISO 527-1 and PN-EN ISO 178. The addition of powder materials into the resin affected the change of the examined parameters when investigating the mechanical properties. The static tensile test proved the supposition about changing the strength of the material. It also confirmed the change in fragility of the prepared samples in relation to the base material. Similar conclusions may be drawn from the development of the research findings of the materials with regard to bending and hardness of the produced samples.

1. Introduction
A combination of high material strength and stiffness with low weight is the main advantage of fibre-reinforced composites, which are being increasingly used in various industries, including aviation (lifting of the aircraft and helicopter), motoring (carbon-fibre and honeycomb composite structure in Formula One), recreation (snowboards, fishing rods, boats, etc.) and many other (such as wind energy) [1,2]. The properties of glass-reinforced and carbon-reinforced fibres are commonly known [3]. One of the most important investigations aimed at determining the usefulness of a given material for air industry applications is to specify its strength characteristics. Moreover, research is being conducted to determine the strength properties of resins used in aerospace technology [4,5], bearing in mind high fragility of the matrix and low binding between the matrix and the base-material [6,7]. In order to increase the mechanical and thermal strength of laminates even further, there are additional reinforcements in the form of particles [8,9,10]. In the available literature, numerous studies address the issue of the material parameters of modified composites, both for fibre reinforcement [11] and powder reinforcement, especially when using carbon nanotubes [11,12,13,14,15], SiC [16,17] and MMT applications [18,19]. The study measures the strength properties of powder composites (including dispersion-strengthened composites) with a 15% volume share of the additive in order to determine the effect of particular additives upon the change of composite ultimate tensile strength and bending strength. The article presents partial findings of research into mechanical properties of composites which exert an influence on the wear process of composites in friction couples, due to the
fact that in aviation it is essential to keep the highest level of safety, also including material strength, combined with other properties which may be mutually exclusive, e.g. abrasive resistance [example 16] or ablative properties of materials [18].

2. Experimental

Tests were carried out on the strength of composite powder in accordance with the PN-EN ISO 527-1 and PN-EN ISO 178 norms in order to determine the strength properties during tensile and bending tests of the examined composite, depending on the type of the used powder additive. The investigations were conducted on the Zwick Roell Z5.0 machine used for static tests. Furthermore, imaging of the fracture structure of the damaged material was performed on a Hitachi electron microscope TM3030Plus, also measuring the toughness of the examined materials by means of the durometer Shore/IRHD DIGI Test II manufactured by Bareiss.

2.1. Test materials

The strength tests were conducted on a powder composite. Composite samples were prepared on the basis of the CES R70 matrix resin, cured with CES H72 hardener, in proportions 100:54 wt. (recommended by the manufacturer). The powder fillers were: montmorillonite (MMT), Expancell d25 Microsphere (microsphere), nanocarbon (NanoC) and silicon carbide (SiC). The amount of a powder additive in each sample equalled 15 \% of the composite volume. The curing occurred at ambient temperature. The author prepared 20-sample batches for research with every additive as well as base samples without any additive, both for static tensile and bending tests. The samples for experiments were created by casting resin with an additive into special silicone moulds, whose dimensions are specified in figures 1 and 2.

![Figure 1. Universal static moulding - tensile test.](image1)

![Figure 2. Universal static moulding - bending test.](image2)

2.2. Geometry of samples used to research

The samples (mouldings) were prepared as flat samples with necking (figure 1). The following dimensions of samples were adopted: thickness 4.0\pm0.2 mm, width of the measurement part 10\pm0.2 mm and overall length above 150 mm. In the case of mouldings made with resin and an additive, the measuring part equalled 80\pm2 mm.

Next, the author prepared samples for testing in a shape illustrated in figure 2. The dimensions of samples prepared for the examination of bending, rectangular-shaped, have been listed in table 1.

| Mould sizes                     |                 |
|---------------------------------|-----------------|
| l – specimen length             | 80 mm           |
| b – specimen width              | 10 mm           |
| h – specimen thickness          | 60 mm           |
| L – distance between supports   | 64 mm           |
| R1 – punch radius               | 5 mm            |
| R2 – support radius             | 5 mm            |
After conducting mechanical investigations (tensile and bending tests), selected samples from each batch underwent testing on a durometer in order to determine the hardness by Shore (D-scale) method. Also the morphology of the surface of the fracture of the examined materials was featured with an electron microscopy.

In order to create more transparent graphs, the author adopted labels of the examined samples in accordance with those listed in table 2.

Table 2. Nomenclature of samples for examinations with powder additives.

| Composite                        | Tensile test | Bending test |
|----------------------------------|--------------|--------------|
| CES R70 + CES H72                | R1           | Z1           |
| CES R70 + CES H72 + microsphere  | R2           | Z2           |
| CES R70 + CES H72 + MMT          | R3           | Z3           |
| CES R70 + CES H72 + SiC          | R4           | Z4           |
| CES R70 + CES H72 + nanocarbon   | R5           | Z5           |

3. Results and discussion

3.1. Tensile test

The first type of the experimental studies of the powder-strengthened composites under scrutiny was a static tensile test. Figures 3 and 4 show an exemplary line chart of the obtained graphs with one exemplary course of elongation. The composites with the addition of MMT and SiC have the properties of brittle materials (destruction occurs approximately after 1.1 – 1.2% of stretching), whereas the resin and composites with the addition of nanocarbon and microspheres have the qualities of plastic materials.

![Figure 3. Exemplary characteristics of composite stretching.](image)

![Figure 4. Distribution of tensile strength of particular composites.](image)

Table 3. Results of a statistical analysis of failure strain in samples during elongation [MPa].

|        | average | variance | standard deviation | standard error | skewness | kurtosis |
|--------|---------|----------|--------------------|----------------|----------|----------|
| **R1** | 44.230  | 44.650   | 4.140              | 2.034          | 0.455    | -0.200   |
| **R2** | 36.600  | 36.900   | 6.800              | 2.607          | 0.583    | -0.739   |
| **R3** | 28.570  | 29.700   | 20.270             | 4.503          | 1.007    | -0.291   |
| **R4** | 20.895  | 20.850   | 6.230              | 2.495          | 0.558    | 0.528    |
| **R5** | 42.456  | 43.700   | 16.730             | 4.091          | 0.915    | -1.169   |

It is possible to observe a significant drop in strength of a composite material after the addition of powder, apart from carbon nanotubes, whose value is approximately equal to the value obtained for a
composite without the additive. The basic values resulting from a statistical analysis have been presented in table 3.

3.2. Bending test
The second type of the experimental studies of the powder-strengthened composites under scrutiny was a bending test. In the figures below (figures 5 and 6), there is an exemplary graph of the obtained courses for bended composites. The composite with the addition of SiC became damaged after the bending of approximately 1.8 mm, which corresponds to its brittle properties. On the other hand, the composites with the addition of microspheres were damaged after the bending of 13 mm on average (figure 5).

![Figure 5](image) Exemplary characteristics of composite bending.

![Figure 6](image) Distribution of bending strength of particular composites.

Similarly, as in earlier research, there is a statistical analysis for all examined materials with regard to the bending strength parameter, with the results presented in table 4. It is possible to observe a significant drop in strength of the composite material after the addition of powder, apart from carbon nanotubes, whose value is even higher than that of the epoxy resin without the additive.

| Table 4. Results of a statistical analysis of failure strain in samples during bending [MPa]. |
|---|---|---|---|---|---|---|
| | average | variance | standard deviation | standard error | skewness | kurtosis |
| **Z1** | 53.635 | 53.65 | 12.05 | 3.472 | 0.776 | -0.047 |
| **Z2** | 48.830 | 49.20 | 3.50 | 1.870 | 0.418 | -0.587 |
| **Z3** | 35.555 | 35.65 | 9.48 | 3.079 | 0.689 | -0.283 |
| **Z4** | 29.010 | 29.05 | 3.41 | 1.846 | 0.413 | 0.421 |
| **Z5** | 56.776 | 58.75 | 20.12 | 4.486 | 1.003 | -0.403 |

3.3. Measurement of hardness
After conducting mechanical research, the author measured hardness of a composite material. The results are listed in table 5, in which there are hardness values and the most important findings of a simple statistical analysis of the obtained results. It is possible to observe very similar values of hardness for Z1, Z3 and Z5 composites. It undoubtedly influenced a uniform distribution of the additive in the resin with MMT additives and nanocarbons, which are characterized with small dimensions. Moreover, Z4 (composite with the addition of SiC) achieved much higher hardness values due to high SiC hardness (when it exceeds 9 points on Mohs scale, it is used as an abrasive material). On the other hand, Z2 (the composite with the addition of a microsphere) has low hardness and low density, which resulted in a considerable drop in the value of hardness of the whole composite.

In order to observe the structure of the fracture of the examined material, the author made an imagery of the surface by means of electron microscopy and the results are presented below for a selected sample from each measurement on completion of fracture testing.
Table 5. Results of a statistical analysis on sample hardness after strength tests [-].

|       | average | variance | standard deviation | standard error | skewness | kurtosis |
|-------|---------|----------|--------------------|---------------|----------|----------|
| Z1    | 77.66   | 0.955    | 0.977              | 0.219         | -1.041   | -0.148   |
| Z2    | 54.44   | 1.070    | 1.034              | 0.231         | 0.172    | 0.306    |
| Z3    | 78.33   | 2.590    | 1.609              | 0.360         | -0.623   | -1.266   |
| Z4    | 86.04   | 1.140    | 1.067              | 0.239         | -0.141   | -0.787   |
| Z5    | 77.70   | 0.490    | 0.700              | 0.157         | -0.248   | -1.285   |

All the obtained research findings do not follow normal distribution (proved by skewness and kurtosis results, see tables 3-5). Therefore, the significance of the influence of the fillers upon the examined properties were conducted by means of a non-parametric Mann-Whitney U test to compare outcomes between two independent samples. The Mann-Whitney U test is based on calculating U statistics with the total number of observations \( n \leq 20 \). The hypothesis of changing the significance is fulfilled only when \( U \) statistics is smaller than the critical \( U_{kr} \) value read from tables, adequate to the assumed confidence level \( \alpha (\alpha < 0.05) \) and the number of groups \( n_1 \) and \( n_2 \). Table 6 lists the findings of the confidence test with regard to the impact of the filler on the composite properties against pure resin.

Table 6. U Mann-Whitney test results significant impact of filler if \( p<0.05 \).

| Property          | Microsphere | MMT         | SiC        | nanocarbon |
|-------------------|-------------|-------------|------------|------------|
|                   | U   | p     | U   | p     | U   | p   | U   | p     |
| hardness          | 0.00000 | 0.000000 | 130.0000 | 0.060112 | 0.00 | 0.000000 | 188.5000 | 0.766046 |
| Tensile strength  | 2.00000 | 0.000000 | 0.0000  | 0.000000 | 0.00 | 0.000000 | 155.0000 | 0.228695 |
| Bending strength  | 44.00000 | 0.000026  | 0.0000  | 0.000000 | 0.00 | 0.000000 | 124.0000 | 0.041124 |

Due to the use of an Hitachi electron microscope, it was possible to capture the image of structures and surfaces of examined conducting and non-conducting specimens, without the necessity to make initial processing and applying additional elements. The article presents only representative images obtained during the examinations of composite materials with additives. In the conducted examinations, the author used the magnification of 50 to 200 (depending on the used additive in photos at applied magnifications). In order to illustrate a composite, the following observation mode was used: secondary electron (SE) to depict the surface morphology (figures 7, 8, 9) and backscattered electron (BSE) to show the compositional information in the sample (figures 10 and 11), in which the used additives are visible.

Figure 7. CES R70 +CES H72 magnification 50x SE.

Figure 8. CES R70 +CES H72 + microsphere magnification 50x SE.
4. Conclusions
When analysing the above results of strength tests of powder composites and observations on an electron microscope, it is possible to formulate the following conclusions:

- The addition of powders to resin significantly changes the strength of the composite both during tensile testing and bending. Only the composite with the addition of nanocarbon showed increased strength to bending, approximately equal to 56.8 MPa, i.e. 5.8% than resin. The remaining composites show a drop in strength to bending even by 45.9%.
- The composites with the addition of SiC and MMT (figure 3) are characterized by a higher Young’s modulus, at the level of 2.1 and 2.3 GPa. The lower Young’s modulus can be observed in the composite with nanospheres, at the level of 1.6 GPa, i.e. 6% less than resin.
- In case of SiC nanofillers and the microspheres, the influence of the fillers’ hardness on composite hardness was observed. In relation to resin hardness, the hardness of the SiC composite grew by approximately 10.8%. In case of the composites with nanospheres, the hardness fell by approximately 29.9%.
- Only the addition of carbon nanotubes did not diminish tensile strength or bending strength. Besides, it made the materials more brittle (apart from the composite containing microspheres). The changes are statistically significant.
- The use of various types of additives changes the internal structure of a composite, as observed on an electron microscope. It clearly affects the strength properties of materials with the powder additives. Nanocarbon, as an additive, significantly influences the change in the mechanical properties only in the three point bending test.

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