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DETERMINATION OF HEAT DEFLECTION TEMPERATURE UNDER A LOAD AND VICAT SOFTENING TEMPERATURE OF POWDER COMPOSITES USED FOR ABLATIVE SHIELDS

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Abstract: This paper presents the results of a study to determine the heat deflection temperature (HDT) and Vicat softening temperature (VST) of polymer matrix powder composites used for ablative shielding. The issue is of particular importance since, during ablative tests, the composite material is partially burnt (ablative layer). However, its remaining parts are additionally heated to a higher temperature, which is consequently associated with a change in the visco-elastic properties that depend, among other things, on the VST and HDT temperatures. In the conducted research, the authors used different mass percentages of powder modifiers (montmorillonite, halloysite, mullite, carbon nanotubes, silicon carbide) matrix base epoxy resin LH 145 Havel with the hardener H147 in the experiments. Apart from observing thermal properties that may change due to a modification of the composition of the composites, the effect of conditioning of the samples on the test results was also noticed. It is preheating a composite at a temperature as low as 55°C was observed to increase the HDT and VST by approximately 20°C and the composite hardness by approximately 3-7%.

Key words: deflection temperature, softening temperature, ablative materials, powder composite

1. INTRODUCTION

One of the most common forms of modifying material properties is the manufacture of composites, which are materials composed of at least two phases. Due to their comprehensive use in a wide range of industries, various improvements are constantly being sought after, including enhancements with regard to mechanical [1-5], ablative [6], or tribological properties [7,8]. The enhancements mentioned above are made either through novel technological concepts of manufacturing composites [9-11] or by including modifiers in the composition of composites.

When classifying modifiers by origin, it is possible to distinguish [12-15] the following:

- natural organic fillers (e.g., wood flour, flax fibers),
- non-organic (e.g., chalk, talc, quartz),
- synthetic (e.g., glass fibers, carbon fibers).

Taking into consideration the form in which the fillers appear, the following can be distinguished:

- powder fillers (spherical, flake, or short-cut fiber form),
- fiber fillers (e.g., glass fibers, boron fibers).

Defining the requirements for material properties is possible after analyzing the operating conditions of components made of a given material. Among the observed external factors affecting the reliability of machine or equipment components is, among others, temperature, which, in the case of polymers, can contribute to a change in the state of aggregation in extreme situations. Therefore, testing newly developed materials for their thermal properties seems appropriate. These, in turn, are determined by the heat distortion temperature under a load (HDT) [16] or Vicat softening temperature (VST) [17] in laboratory conditions.

Numerous attempts have been made in the available literature to improve the properties (especially the mechanical ones) of composite materials by adding modifiers in the form of powders; however, on numerous occasions, they were carried out without determining the influence of the modifier on the thermal characteristics of a composite. The authors have decided to select the best-known powder modifiers used in polymer composites, as well as polymer composites treated as a matrix base, in order to supplement the knowledge base with conclusions formulated on the basis of their own research. The details regarding the selected reinforcement and matrix base are presented in the next section.

2. MATERIALS AND METHODS

Based on the analysis of the state of available expertise on the thermal properties of polymer composites and the fundamentals of experimental planning, the authors have prepared a methodology for experimental research and proposed the research equipment.

The research focused on powder composites made at the laboratory. For this purpose, different types of powder fillers were used (montmorillonite, halloysite, mullite, MWCNT, silicon carbide), described in the following sections, at different mass percentages (1%, 5%, 20%), which were selected as matrix - also presented in the following sections - LH 145 (Havel Composites) with the hardener H147 (Havel Composites).

After 3 and 15 hours, the thermal properties were determined for both non-heated and heated samples in two temperature ranges (55ºC and 80ºC). It is also worth noting that the tests were carried out simultaneously on reference samples without the addition of powder fillers. A thermal test model is shown in Fig 1.

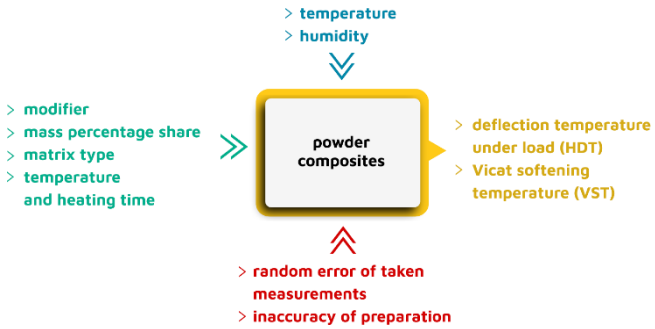

Fig. 1. Scheme of phenomena occurring during sample combustion [6]

The powder modifier, its mass percentage in the composite, the heating temperature, and the heating time were taken as input (independent) factors. The permanent factors included the microclimate in the laboratory rooms, air temperature in the laboratory (22ºC), and technical features of the equipment, while the interferences included random error in the measurements and inaccuracy of preparing the powder composites. The thermal properties of the produced composites, as determined by Vicat and HDT test results, were obtained as input data.

2.1. Powder reinforcements

Several modifiers described in the available literature and exploited to shape the selected properties of composites (including hybrids) were used to produce powder composites. These modifiers, together with their brief characteristics as well as references to wider descriptions, are presented in Tab. 1.

Tab. 1. Characteristics of used powder modifiers

Modifier's name	Characteristics	Manufacturer
montmorillonite (MMT)	It is one of the most popular representatives of the widely used layered silicates, derived from a group of clay minerals (clays), which are the main component of rocks called bentonites. A small additive of MMT into the polymer matrix base of the composite allows for an improvement in the mechanical and thermal properties of the material and contributes to a reduction in its flammability. Sources: [18–23]	Montmorillonite K 10 in powder form, supplied by the American company Sigma-Aldrich
halloysite ($Al_2Si_2O_5(OH)_4 \cdot 2H_2O$)	It is an aluminosilicate consisting of approximately 40% aluminum oxide, approximately 45% silica and water, and traces of metal oxides such as Na_2O, TiO_2, FeO, MgO, Al_2O_3, K_2O, TiO_2, and CaO. Sources: [24–28]	Halloysite Nanoclay in a powder form (nanotubes are 1-3 m long and 30-70 nm in diameter), supplied by the American company Sigma-Aldrich
mullite ($3Al_2O_3 \cdot 2SiO_2$)	It is characterized by high thermal stability, low thermal expansion, thermal conductivity, high resistance to creep and corrosive substances while maintaining adequate strength and resistance to brittle fracture, the ability to attach various cations and the possibility of forming crystals with various contents of silicon and aluminum oxides. Sources: [29–32]	Aluminum Silicate (Mullite) in the form of powder, supplied by Sigma–Aldrich
carbon nanotubes (MWCNTs)	The biaxial nanotube structure is constructed from rolled graphene planes composed of groupings of hexagonal carbon rings. Apart from a high aspect ratio, the nanotubes are characterized by very low density, high rigidity, and thermal resistance, as well as high bending and tensile strength. Sources: [33–37]	Multi walled MWCNT by Bucky USA (5-10 nm in diameter, 30 µm in length)
silicon carbide (SiC)	It is characterized by high thermal and electrical conductivity, high chemical decomposition temperature, good mechanical strength, and high hardness. High resistance to rapid temperature changes (thermal shocks) and oxidation (occurring only above 1,400°C and accompanied by the formation of a protective layer in the form of silicon oxide SiO_2). is also quite relevant. Sources: [38–42]	Carborundum F220 with gradation equal to 53-75 µm

The modifiers were added in specific mass percentages of 1%, 5%, and 20%, respectively. The balance PS2100 R2 Radwag (Radom, Poland) was used to precisely prepare the weighed portions of modifiers.

2.2. Section title Resin-based matrix

Due to the wide range of applications of powder modifiers in polymer compositions and the demand of various industries for improved composite properties, it was decided to use polymer resins as a matrix base for the manufactured powder composites.

LH 145 resin is a material characterized by relatively low viscosity and high resistance to crystallization at low temperatures.

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It shows good resistance to chemical agents. Produced by the Czech company Havel Composites, the resin is made from bi-phenol and epichlorohydrin. Its main application recommended by the manufacturer is the production of boats, tools, and automotive parts. Its low viscosity and chemical neutrality are decisive for a convenient application and good compatibility with fiber reinforcements made of aramid, carbon, or glass. In order to initiate the resin curing process, the authors used the H 147 hardener proposed by the manufacturer in the recommended 100:25 weight ratio. The detailed information on LH 145 is provided in Tab. 2.

Tab. 2. LH 145 resin characteristics [43]

Properties	Values
Density at 25°C	1.15 g/cm3
Flash-point (PMCC)	149°C
Viscosity at 25°C	0.7 Pa·s
Epoxy value	170-179 g
Temperature stability when curing at 25°C (for at least 2 days)	50-60°C
Temperature stability when curing at 50-60°C (for 3 hrs)	80-90°C

2.3. Sample heating

Each sample of prepared samples, differing both in the type of the used reinforcement, its mass percentage, and the matrix, was divided into sets. The first one contained samples that had not undergone heating. Other sets were divided into those heated at 55°C (3 hrs and 15 hrs) and 80°C (15 hrs). The samples were heated in a WKL 64 Weisstechnik climate chamber (Weiss Umwetltechnik GmbH, Heuchelheim, Germany) presented in Fig. 2. By controlling the heating process, the machine maintained a set temperature with an accuracy of ±0.1°C.

A summary Table was prepared, focusing on the distinguishing features of a given sample (see Table 3). It included the following data: sample number, number of samples, type of resin and hardener, name of additive and its mass content, temperature, and curing time, all collected in one place. The specified division and nomenclature will function unchanged throughout the remainder of this work.

Tab. 3. Table showing manufacture parameters of individual series of measurement samples

Sample	Number of samples	Resin	Hardener	Additive	Content of the additive [%]	Heating temperature [°C]	Heating time [hrs]
0/ 0W	3	LH 145	H 147	-	-	-/55	-/3
1/ 1W				Montmorillonite	5		
2/ 2W				Montmorillonite	20		
3/ 3W				Halloysite	5		
4/ 4W				Halloysite	20		
5/ 5W				Mullite	5		
6/ 6W				Mullite	20		
7/ 7W				MWCNT	1		
8/ 8W				MWCNT	5		
9/ 9W				Silicon carbide	5		
10/ 10W				Silicon carbide	20		

2.4. Determining the deflection temperature (HDT) under load

The prepared samples were subjected to HDT testing on an Instron HV6X HDT test stand (Pianezza, Italy) powered by Instron Bluehill HV software (Bluehill version 1.9, Instron Corporation, Canton, MA, USA). It was used to configure parameters concerning the test method, such as the test type (HDT), the norm under which the test was conducted [44], variant, heating speed, initial temperature, and conditioning time understood as the time of immersion of the samples in oil prior to launching the examination (see Tab. 4).

Tab. 4. HDT test parameters

Instron Bluehill HV programme parameters	Adopted boundary conditions
variant of research	A – 1.8 MPa
rate of oil temperature rise	120°C/h
initial temperature	25°C
time of conditioning	5 min

Time plays an important role in the reliability of the results, as it allows the temperature of the moulding and the oil to level off before the test begins. The next step was to place the samples in the measuring stations, keeping their recommended central position. Special care was taken to ensure that the temperature gauges were positioned at a short distance from the examined profile. The dimensions of each test sample were then entered into the Bluehill programme. This way, the software calculated the load to be applied so as to obtain the bending stress value.

These calculations took into account the pin's weight, which informed the operator which weights to put on a tripod. The dimensions were also used to determine an individual standard deflection value for the subsequent profiles.

At the end of the preparatory procedure, automatic temperature control of the heating bath was started, which was then stabilized at 25°C. After starting the test program from the computer program panel, the heating and cooling systems of the stand were checked for several minutes; the temperature was stabilized, and the specimens were loaded and immersed in the heating liquid. This stage was followed by the main part of the test, during which each station separately recorded deformations occurring in the

sample and the temperature rising at a constant rate around it. The test ended once the last section reached the standard deflection. Next, cooling was activated to reduce the temperature of the liquid to its initial value.

2.5. Determination of Vicat softening temperature (VST)

Vicat softening temperature tests were performed on an Instron HV6X stand (Pianezza, Italy). It holds a dedicated indenter and a clip that keeps the sample in position during the test. As in the previous test, the Instron Bluehill HV software (Bluehill version 1.9, Instron Corporation, Canton, MA, USA) was used to configure the test method parameters, such as the test type (VST), standard [45] load variant and heating rate, the initial temperature and the conditioning time, understood as the period for which the sample is immersed in oil prior to the test (see Tab. 5).

Tab. 5. VST test parameters

Instron Bluehill HV programme parameters	Adopted boundary conditions
load variant	A120 - load 10 N
heating speed	120°C/ h
initial temperature	25°C
time of conditioning	5 min

The samples were then placed sequentially in the measuring stations, maintaining a recommended position, which ensures a minimum distance of 3 mm between the indenter tip and the edge of the section. Due to the constant load value, the total mass acting on the indenter of each station was the same.

At the end of the preparatory procedure, the automatic temperature control of the heating bath was started, which from now on was stabilized at 25°C. After activating the software, the heating and cooling systems of the station were checked for several minutes; the temperature was stabilized, and the samples were loaded and immersed in the heating liquid. This stage was followed by the main part of the test, during which each station separately recorded the movement of the indenter and the temperature rising at a constant rate around it. The test was terminated once a 1 mm cavity was reached in the last of the profiles or when the test was manually aborted. After this time, cooling was activated to reduce the temperature of the liquid to its initial value.

In accordance with the previously described methodology for determining the Vicat softening temperature, all the measurement series were tested in accordance with method A120. However, it occurred that not all of the tests that were conducted resulted in the penetration of the indenter tip in the material being required to be 1 mm by the norm. The problem mainly affected the profiles subjected to the heating process and made it impossible to determine the sought VST temperature (in accordance with the norm). It was, therefore, decided that for each such series, an agreed softening temperature of the composite material would be determined. The method of "tangents' intersection - increase and stabilization - of the pin's indentation in the material, in the function of temperature" was used in order to determine the temperature of the intersection of two lines tangent to the curve of the experimental course in the function of temperature.

2.6. Additional tests and measurements

Apart from the tests aimed at determining the thermal properties of powder composites, the authors additionally decided to

- determine their density values,
- determine their hardness values,
- measure the indentations that occurred during the Vicat test.

In order to provide a more complete presentation of the Vicat study, imaging of the resulting indentation after the study is also presented. The equipment used for this purpose, along with information on the standards underlying the research activities, are presented in Tab. 6.

Tab. 6. Tests and measurements

Type	Device	Standard
determination of values densities	Mettler Toledo XSR 205 analytical balance (Mettler Toledo, Zürich, Switzerland)	ASTM D 792 [46]
determination of values hardness	Digi Test II modular hardness tester (Bareiss, Germany) Shore D/C/DO module	ISO 48 [47]
Measurement of the indentations that occurred during the Vicat test	MicroProf 100 (FRT GmbH, Bergisch Gladbach, Germany)	not applicable

3. RESULTS AND DISCUSSION

3.1. Density testing

The density of the composite materials was tested using a Mettler Toledo XSR 205 analytical balance with the necessary accessories. The values are listed below in Table 7.

Tab. 6. Mass, density, and volume values of individual resins and additives, the filling levels expressed in mass and volume, as well as the density of the composite

Sample	m_{res} [g]	ρ_{res} [g/cm^3]	V_{res} [cm^3]	m_{add} [g]	ρ_{add} [g/cm^3]	V_{add} [cm^3]	V_{all} [cm^3]	in masses [%]	by volume [%]	ρ_{comp} [g/cm^3]
0	50	1.15	43.48	0	-	-	43.48	0	0	1.15
1	50	1.15	43.48	2.6	2.5	1.04	44.52	5	2	1.178
2	50	1.15	43.48	15	2.5	6	49.48	20	12	1.299
3	50	1.15	43.48	2.6	2.53	1.03	44.51	5	2	1.181
4	50	1.15	43.48	15	2.53	5.93	49.41	20	12	1.308
5	50	1.15	43.48	2.6	3.03	0.86	44.34	5	2	1.185
6	50	1.15	43.48	15	3.03	4.95	48.43	20	10	1.347
7	50	1.15	43.48	0.6	0.08	7.31	50.79	1	14	1.154
8	50	1.15	43.48	2.6	0.08	31.69	75.17	5	42	1.16
9	50	1.15	43.48	2.6	3.15	0.83	44.30	5	2	1.194
10	50	1.15	43.48	15	3.15	4.76	48.24	20	10	1.352

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3.2. HDT testing

It was decided to present the test results in the form of a clear graphical representation of the deflection as a function of temperature. For scientific purposes, there is only an example of such a measurement (see Fig. 2 and 3. and Tab. 8 and 9). Each of the colored lines reflects successive measuring stations and, thus, successive samples. As the resin without additives is a kind of reference for other composites with fillers, a black line was additionally placed (as a reference line for other materials). Below the graph, there is a table summarizing the most important material parameters and test results for each measurement series. The next part of the graph shows how the heating temperature of the material before the test influenced the change in HDT and how this temperature changed for different resins.

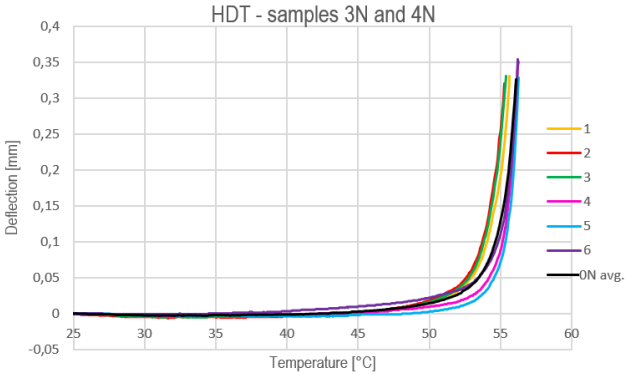

Fig. 2. Examples of HDT temperature measurements in a graphical form combined with tabular values from this graph for composite without heating

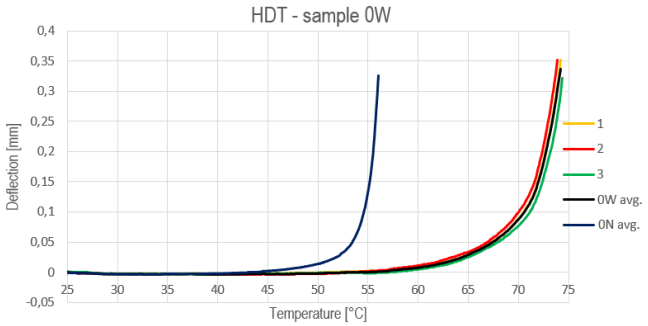

Fig. 3. Examples of HDT temperature measurements in a graphical form combined with tabular values from this graph for heated composite

Tab. 8. Results of HDT experimental tests for exemplary composite samples without heating

Measurement no.	Color	Sample	Load [g]	Deflection std.	Deflection value [mm]	Temp. HDT [°C]
1	Yellow	3 N	315	Achieved	0.33	55.6
2	Red	3 N	335	Achieved	0.32	55.3
3	Green	3 N	329	Achieved	0.33	55.4
4	Pink	4 N	287	Achieved	0.35	56.3
5	Blue	4 N	338	Achieved	0.31	56.2
6	Violet	4 N	291	Achieved	0.34	56.2
0N average						56.0

Tab. 9. Results of HDT experimental tests for exemplary heated composite samples

1	Color	Sample	Load [g]	Deflection std.	Deflection value [mm]	Temp. HDT [°C]
1	Yellow	0 W	289	Achieved	0.35	74.2
2	Red	0 W	287	Achieved	0.35	73.9
3	Green	0 W	334	Achieved	0.31	74.4
0W average	Black	0 W	303	-	0.337	74.2
0N average	Black	0 N	329	-	0.327	56.0

After experimental testing of all the manufactured samples, the average HDT temperature for all the examined composites has been summarized graphically (see Fig. 4).

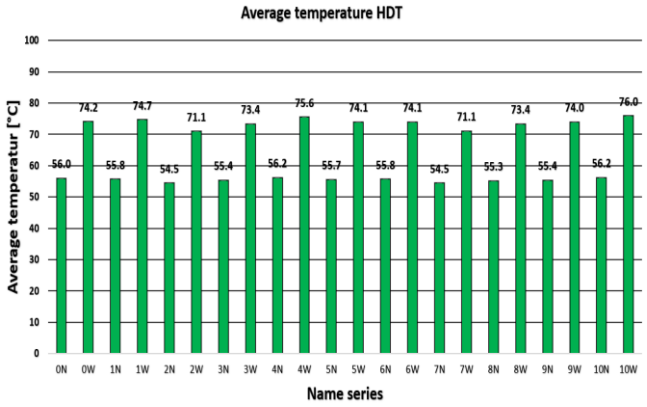

Fig.4. Summary results of average HDT temperature values

3.3. Vicat testing and post-test surface profilometry

In accordance with the previously described methodology for determining the Vicat softening temperature, all the measurement samples were tested in accordance with method A120 (Figure 5 and Table 10). However, it turned out that not all of the tests that were conducted resulted in the penetration of the indenter tip in the material, which was required to be 1 mm by the norm. The problem mainly affected the profiles subjected to the heating process and made it impossible to determine the sought VST temperature (in accordance with the norm). It was, therefore, decided that for each such series, an agreed softening temperature of the composite material would be determined. The method of "tangents' intersection - linear increase and stabilization - of the pin's indentation in the material, in the function of temperature" was used in order to determine the temperature of the intersection of two lines tangent to the curve of the experimental course in the function of temperature (Fig. 6 and Tab. 11). The first line described is the tangent to the linear increase in indentation, while

the second is the tangent to the stabilized value of indentation. Thus, the conventional softening temperature of the material was determined, and then it was entered in the column "Temp. VST" in the summary Tables. In order to easily distinguish it from the VST temperature value (in accordance with the standard), a green font was used (see Tab. 11).

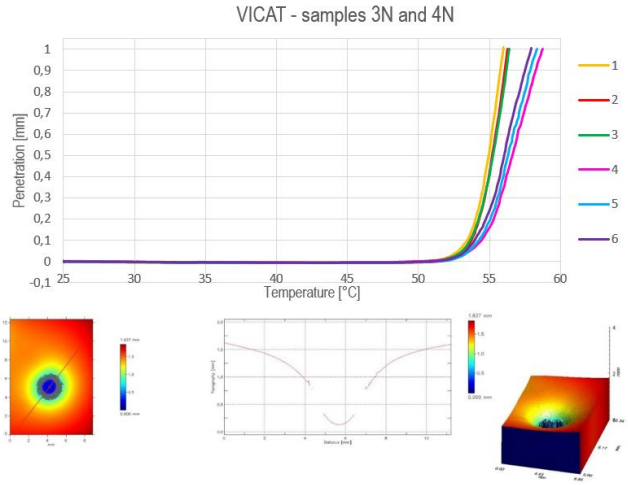

Fig. 5. Examples of Vicat temperature measurements in a graphic form, combined with tabular values for this graph composite without heating

Tab. 10. Results of Vicat experimental tests for exemplary composite samples without heating

Measurement no.	Color	Sample	Penetration (in accordance with the norm)	Depth [mm]	Temp. VST [°C]
1	Yellow	3 N	Achieved	1.000	56
2	Red	3 N	Achieved	1.000	56.3
3	Green	3N	Achieved	1.000	56.4
4	Pink	4N	Achieved	1.000	58.7
5	Blue	4N	Achieved	1.000	58.3
6	Violet	4 N	Achieved	1.000	57.9
Average					56.5

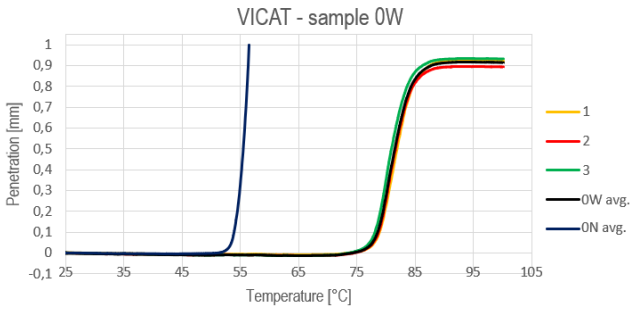

Fig. 6. Examples of Vicat temperature measurements in a graphic form, combined with tabular values for this graph: part A - composite without heating; part B - heated composite

Tab. 11. Results of Vicat experimental tests for exemplary heated composite samples

Measurement no.	Color	Sample	Penetration (in accordance with the norm)	Depth [mm]	Temp. VST [°C]
1	Yellow	0W	Not achieved	0.928	- ; 84.9
2	Red	0W	Not achieved	0.895	- ; 84.3
3	Green	0W	Not achieved	0.936	- ; 84.0
Average	Black	0 W	-	0.920	- ; 84.4
Average	Black	0 N	-	1.000	56.5

After experimental testing of all the manufactured samples, a summary of the results of the average VST temperature for all the examined composites has been presented graphically (see Fig. 7).

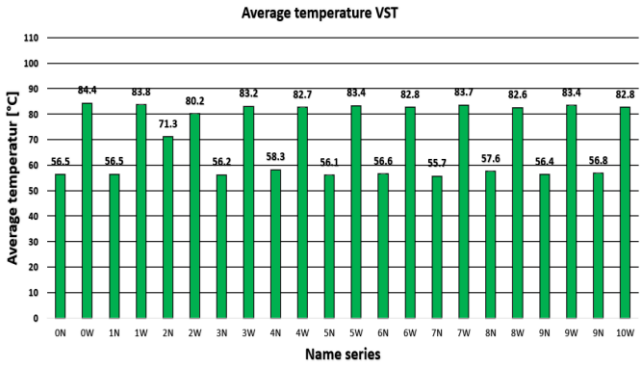

Fig. 7. Summary results of average VST temperature values by Vicat

3.4. Hardness tests

The tests were carried out 10 times for each profile (one from a given sample), taking care to test at different points away from the edges of the profile. The experimentally obtained hardness values of the tested materials are presented in a graphic form in Figure 8 and Table 12. The authors used colors to distinguish between the heated and non-heated samples. The sets of profiles cured at room temperature are green. The yellow color indicates the samples heated at 55°C. Samples 9 and 10 were measured from both sides in order to control the hardness of the material due to the high density of the powder additive, which resulted in higher sedimentation in the composite; higher hardness was obtained from the bottom side of the sample. Below the graph is a clear summary table with the results of the average hardness and standard deviation of each sample.

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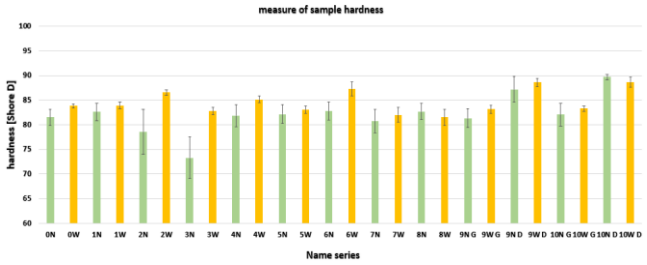

Fig. 8. Hardness tests graph showing the average hardness value of each sample (N - not heated. W - heated. G- top. D - down) together with a summary table

Tab. 12. Mean values and standard deviation of experimental tests of composite hardness

Sample	0N	0W	1N	1W	2N	2 W	3N	3 W	4 N
$\bar{x}$	81.5	83.8	82.6	83.9	78.6	86.6	73.3	82.8	81.9
σ	1.58	0.46	1.78	0.71	4.52	0.60	4.20	0.81	2.28
Sample	**4 W**	**5 N**	**5 W**	**6 N**	**6 W**	**7 N**	**7 W**	**8 N**	**8 W**
$\bar{x}$	85.2	82.1	83.0	82.8	87.3	80.8	82.0	82.7	81.5
σ	0.63	1.89	0.75	1.82	1.42	2.39	1.45	1.61	1.62
Sample	**9N G**	**9W G**	**9N D**	**9W D**	**10N G**	**10W G**	**10N D**	**10W D**	
$\bar{x}$	81.4	83.2	87.2	88.6	82.1	83.3	89.7	88.7	
σ	1.93	0.81	2.55	0.82	2.29	0.54	0.00	1.03	

4. CONCLUSIONS

The authors attempted to determine the properties defining the softening and deflection temperatures of powder composites, which are often disregarded in scientific publications. They also placed a great deal of emphasis on examining the selected mechanical properties. Due to the two-track nature of the research, the conclusions have also been split.

HDT testing

- Non-heated samples - the additives that increased the temperature of deflection under a load are as follows: halloysite (20 wt.%) and silicon carbide (20 wt.%) increased the temperature of deflection by 0.2°C compared to the reference samples. Other powder additives decreased the examined temperature (in the case of montmorillonite by 20% and in the case of MWCNT by 1%, even by 1.5 °C).
- Heated samples - additives that increased the temperature of deflection under a load include montmorillonite (5% of content increased the deflection temperature by 0.5°C compared to the reference samples); halloysite (20 % of content increased the deflection temperature by 1.4°C compared to the reference samples) and silicon carbide (20 % of the content increased the deflection temperature by 1.8°C compared to the reference samples, with the highest HDT). Other powder additives had a lowering effect on the examined temperature. The lowest values were found in filled profiles: MMT (20 %) of content and MWCNT (1 % of content, by 3.2 °C).

VST testing

- Non-heated samples - additives that increase the softening temperature include MMT (20 % content of the highest VST, which was equal to 71.3 °C, halloysite (20 % content) an increase up to 58.3 °C; mullite (20 % content) an increase by 0.1 °C, MWCNT (5 % content) an increase up to 57.6 and silicon carbide (20 % of content and two types of filling when tested on the bottom side). Other powder additives had a lowering effect on the examined temperature. The lowest values were found in profiles filled with MWCNT (1 %) of content, with a VST value of 55.7).
- Heated samples - none of the heated profiles with LH 145 resin matrix base reached the standard-defined cavity required for VST temperature determination; however, each of the powder fillers lowered the conventional softening temperature. The composite achieved the value closest to the pure heated resin with an addition of MMT (5% of content). The lowest agreed VST was obtained for a composite with the same additive but with a higher fill level (20%).

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