

# Selected technical ceramics as nanoreinforcement for polyvinyl butyral/epoxy polymer composites

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## Abstract

In this study, various technical ceramics in the form of nanostructures, known for their exceptional mechanical properties and thermal resistance, were analysed as potential reinforcement of a polymer matrix system. A combined, hybrid composite matrix was considered, consisting of epoxy resin and polyvinyl butyral. The following nanostructures were incorporated into the polymer matrix by ultrasonication and solvent-casting technique: B<sub>4</sub>C, TiB<sub>2</sub>, CNT and INT-WS<sub>2</sub>. Polymer composites were prepared by solvent-casting of neat hybrid polymer matrix and polymer matrix reinforcing with each chosen individual nanostructure from a temporary solvent – ethanol. The characterisation of the obtained polymer composite samples encompassed Fourier transform infrared spectroscopy analysis, differential scanning calorimetry, tensile test, hardness measurement and examination of resistance to cavitation wear.

## 1. Introduction

Composite materials have become more popular than conventional metal materials in the design and construction of vehicles and aircrafts, in the nautical industry, etc. due to their ability to reduce the final weight of the components, resist corrosion and improved fatigue performance. Nowadays, polymer composites are widely used, whether reinforced with some fibres or particulate fillers [1-4]. Thermoset and thermoplastic polymers are typically used as polymer matrices of these composites, and one of the most used polymeric components is epoxy resin, sometimes combined with other polymer materials [4-6]. Designing composite materials for demanding applications, such as nautical or aerospace structures, or specific protective equipment, requires fulfilling specific performance properties such as high static strength, high impact resistance, high fracture

toughness, damage tolerance and resistance to different environmental conditions during exploitation [4,7]. Nanomaterials are currently widely studied and applied as reinforcements in polymer matrices, due to their high surface area, good interaction with the matrix, fewer defects and low volume fraction needed for the desired enhancement. One specific group of materials is becoming more and more attractive as promising nanoreinforcement in novel polymer composites, namely technical or engineering ceramics.

Technical ceramics are materials that possess exceptional mechanical, thermal, electrical and chemical properties. They are made up of inorganic compounds such as oxides, nitrides, carbides and silicates. Although technical ceramics exhibit excellent resistance to compression and even maintain their strength at high temperatures, they have one significant drawback for employment in load bearing and that is their brittleness [8,9]. A potential way to overcome this issue is to reduce the size of these materials, to the nanoscale. When used as nanoreinforcements in



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polymer composites, technical ceramics can significantly improve the mechanical and physical properties of the composite material, i.e. enhance the stiffness, strength, toughness and thermal stability of the material [9-13]. Nanoceramics were applied in armour materials against blunt trauma, even against high-velocity ammunition [8,14,15]. Some of the commonly used technical ceramics as nanoreinforcements in polymer composites include alumina, zirconia, silicon carbide and titanium dioxide [10-13]. The addition of technical ceramics to polymer composites also enhances the resistance of the material to wear and chemical agents, making it suitable for a wide range of applications in industries such as aerospace, automotive, electronics and medical application.

In this study, a combination of epoxy resin with polyvinyl butyral (PVB) was chosen as a hybrid matrix based on the thermosetting and thermoplastic polymer to be reinforced with nanostructures of technical ceramics. Although known for its good adhesion and mechanical properties, epoxy resin is brittle, so in certain applications, it is toughened with the addition of some more ductile polymeric compounds, such as polyurethanes or PVB [6,16].

Several technical ceramics were selected to be considered as nanoreinforcement for matrix: boron carbide ( $B_4C$ ) and titanium diboride ( $TiB_2$ ), as well as carbon nanotubes (CNT) and inorganic nanotubes of tungsten disulfide (INT- $WS_2$ ), which are often considered ceramic materials although they do not belong to the ceramic group of materials.

Tungsten disulfide ( $WS_2$ ) is a layered transition metal dichalcogenide with a lamellar structure, often used as a solid lubricant and anti-wear additive in various engineering applications due to its excellent mechanical, tribological and chemical properties. INT- $WS_2$  exhibits great thermal and mechanical resistance to shock and pressure resistance, and therefore it was applied as reinforcement in some advanced composites for ballistic protection and aircraft engineering [14, 17-19]. Also, carbon nanotubes (CNTs) are not classified as technical ceramics, as they are not inorganic compounds, but a form of carbon. CNTs have a cylindrical shape with a high aspect ratio, and they possess excellent mechanical, electrical and thermal properties. Like technical ceramics, CNTs can be used as reinforcing fillers in polymer composites to improve their stiffness, strength and toughness.

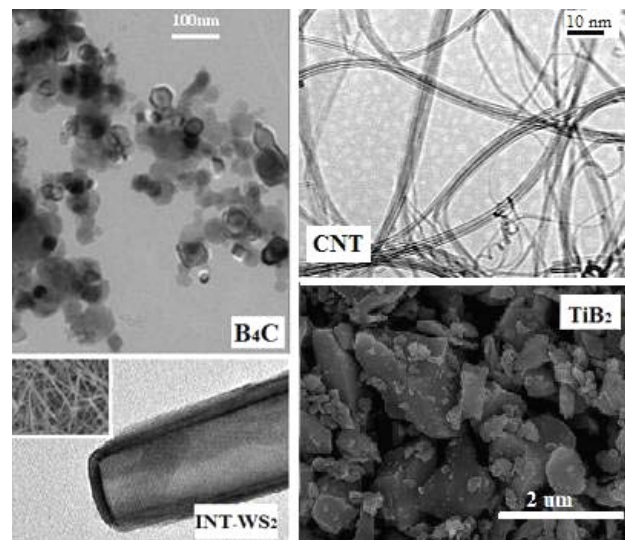
Boron carbide ( $B_4C$ ) and titanium diboride ( $TiB_2$ ) are typical examples of technical ceramics, with

exceptional thermal stability and resistance to oxidation, high hardness and excellent wear resistance. For example,  $B_4C$  is often used in armour due to its high hardness and toughness.  $TiB_2$ , on the other hand, is used as a reinforcing filler in metal and ceramic matrix composites due to its high strength and stiffness [20-25].

## 2. Materials and experimental methods

### 2.1 Composite samples preparation

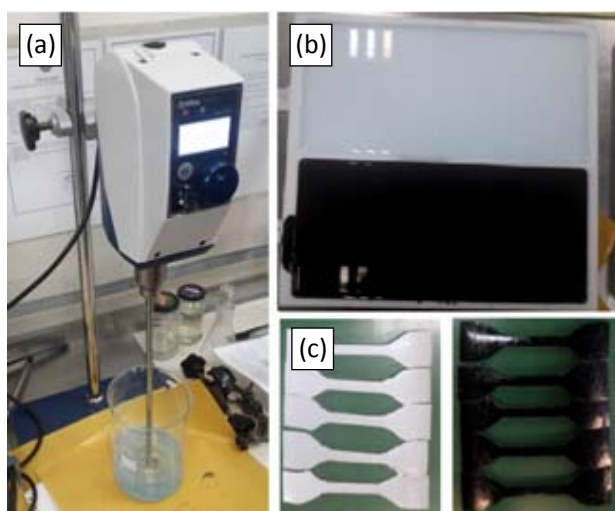
For the preparation of hybrid polymer matrix samples, neat and with the addition of nanoceramic structures, the following materials were used: aero-grade epoxy resin system L385 + 386 (Hexion), mostly used for fibre-reinforced polymer composites intended for aircrafts construction; PVB powder Mowital B 60 H (Kuraray); and ethanol (Uni-Chem). Regarding the used nanostructures, they were provided as follows:  $B_4C$ , diameter 45 – 55 nm (US Research Nanomaterials);  $TiB_2$ , diameter 100 – 200 nm (GetNanoMaterials); CNT (single-wall/double-wall mixture), diameter < 4 nm and length 5 – 30  $\mu m$  (US Research Nanomaterials); and INT- $WS_2$  (multi-wall), diameter 80 – 100 nm and length 10 – 20  $\mu m$  (ApNano Materials). Figure 1 depicts the used nanostructures, in SEM/TEM images provided by the supplier, SpeedUP INTERNATIONAL.



**Figure 1.** SEM/TEM images of the used nanostructures provided by the supplier

As may be observed, the used nanostructures do not have uniform particle sizes, and some of them are agglomerated ( $B_4C$  and  $TiB_2$ ). This is why the selected nanostructures were first dispersed in ethanol, using an ultrasonic homogeniser Sonopuls

HD 4100 (BANDELIN electronic). The device has a 13 mm sonotrode TS 113 made of the titanium alloy TiAl6V4, with a maximum amplitude of 82  $\mu\text{m}$ . The ultrasonication was done for 30 minutes, at 60 W and 20 kHz. This first step (ultrasonication), is the preparation of the particles for incorporation into the polymer matrix. It is necessary to prevent agglomeration of the nanostructures in the composite. The second step is mixing of the particles dispersion with the polymers (PVB and epoxy). This dispersion was mixed with epoxy L385, PVB was added in small portions and dissolved in this mixture while still mixing on a mechanical stirrer and finally, epoxy hardener 386 was added. The third step is the casting of the obtained mixture (polymer solution with the incorporated particles) into the non-adhesive moulds made of PTFE (Teflon). The last step is solvent evaporation overnight, leaving the solid polymer reinforced with the dispersed particles in it. From the obtained cast plates (thick films) of polymer composites, the properly standardised specimens were taken for tensile test, as shown in Figure 2. The ratio between the epoxy and PVB was 95:5 wt. %. The selected nanostructures were added in different concentrations, based on available data from the literature and some preliminary research: 0.5 wt. %  $\text{B}_4\text{C}$ , 1 wt. %  $\text{TiB}_2$ , 0.5 wt. % CNT and 0.5 wt. % INT- $\text{WS}_2$  [17,20,26].



**Figure 2.** Preparation of the cast polymer matrix samples: (a) ultrasonication, (b) cast polymer solutions in PTFE moulds (white: neat epoxy/PVB, black: epoxy/PVB/ $\text{B}_4\text{C}$ ) and (c) cut specimens for tensile test

## 2.2 Characterisation techniques and mechanical testing

In order to verify the chemical reaction between the two combined polymers and to

examine if there was any chemical interaction with the added nanostructures, Fourier-transform infrared spectroscopy (FTIR) analysis was carried out. Nicolet iS10 spectrometer (Thermo Fisher Scientific) was used, with the possibility of sampling by attenuated total reflectance (ATR).

Differential scanning calorimetry (DSC) was performed to determine the glass transition temperatures ( $T_g$ ) of the prepared composites, using a DSC Q20 device (TA Instruments), with the data acquisition program Universal V4.7A. DSC provides information on the thermal properties of solid materials such as the melting and degradation temperatures, glass transition temperature of polymers and other thermal characteristics of the materials. The  $T_g$  is read as the point of a slight change in the slope of the DSC thermogram and is often considered a measurement of the thermal stability of a polymer. The DSC measurements were performed under a nitrogen flow of 50 ml/min in the temperature range from 0–200  $^{\circ}\text{C}$ . The samples were first heated at a rate of 10  $^{\circ}\text{C}/\text{min}$ , then cooled at the same rate and heated again. Two cycles of heating were performed to note the eventual difference after the polymer relaxation, i.e. after the first heating-cooling cycle, since the first heating scan in DSC is mostly used for the removal of residual solvents and to erase the thermal history of the polymer.

Neat and nanoreinforced polymer composite samples were examined regarding the tensile strength, using the universal testing machine Instron 1122 (average sample width 9.9 mm and thickness 0.8 mm). The maximum load was 100 N and the speed was 5 mm/min. The polymer composites were cut on a custom-made vertical cutting tool with a cutter in the shape of specimens for tensile test, according to appropriate standard ASTM D638-03.

The hardness was measured using a Zorn Stendall DDR device, in Shore D scale for plastics. For each composite 5 specimens were tested.

Resistance of the polymer composites to cavitation wear was examined using the above-described ultrasonic processor Sonopuls HD 4100 (BANDELIN electronic). The square-shaped specimens were cut from the polymer composite plates and exposed to ultrasonic cavitation in distilled water, during 4 periods of 30 minutes, at room temperature (22–24  $^{\circ}\text{C}$ ). The ultrasonicator was set to 20 kHz and 60 W of power. After each cycle of 30 min of cavitation, samples were dried to a constant mass and the weight loss was

tracked. The appearance of the samples was observed with Smart 5MP Pro (Delta Optical) optical inspection microscope.

### 3. Results and discussion

#### 3.1 FTIR results

The registered FTIR spectra for the examined polymer composites are given in Figure 3. The characteristic peaks originating from the constituents of the prepared composites are given in Table 1.

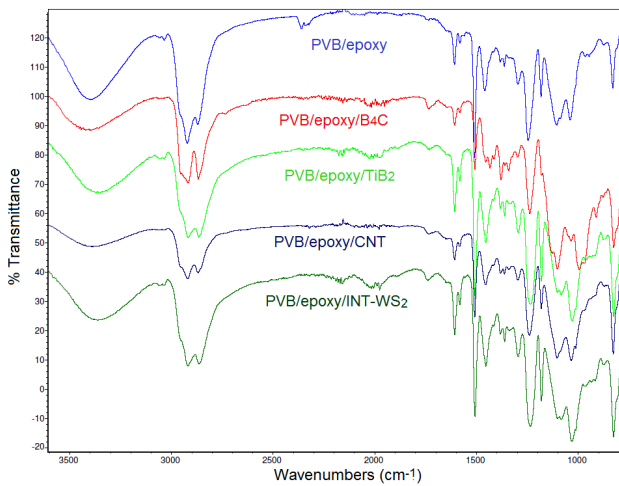


Figure 3. FTIR curves

Table 1. FTIR results – registered characteristic peaks

Sample constituent	Peak, cm <sup>-1</sup>	Functional group
PVB	3350 – 3450	broad peak OH
	2850 and 3000	-CH <sub>3</sub> , -CH <sub>2</sub> , -CH
	1400 and 1280	alkynes
	1740 – 1750	C=O
	1200	esters
	1100	C-O-C
Epoxy resin	950	acetals
	~ 920	vibrations of the epoxy ring
	1670	vibrations of the aldehydes

Comparing the FTIR curves for the neat sample with those for the nanoreinforced samples, there is an evident difference in the intensity of the -OH peak. Its intensity has been reduced due to a more complete curing reaction between PVB and epoxy resin that took place in the samples with the nanoreinforcements – the presence of these particles, especially CNT and B<sub>4</sub>C, has improved

mobility of the molecular chains and made it easier for the functional groups to interact on the interphase zone on the surface of the particles. Besides this effect, it may be observed that the FTIR curves are very similar for all the examined polymer matrix samples, so it may be concluded that the incorporated nanostructures are chemically inert, meaning that their reinforcing effect relies on their physical interaction with the matrix and is a consequence of their mechanical resistance.

#### 3.2 DSC results

The values of the glass transition temperatures were read from the registered DSC thermograms, as depicted on the example of sample PVB/epoxy/B<sub>4</sub>C in Figure 4. For the examined polymer composites the registered T<sub>g</sub> values are given in Table 2.

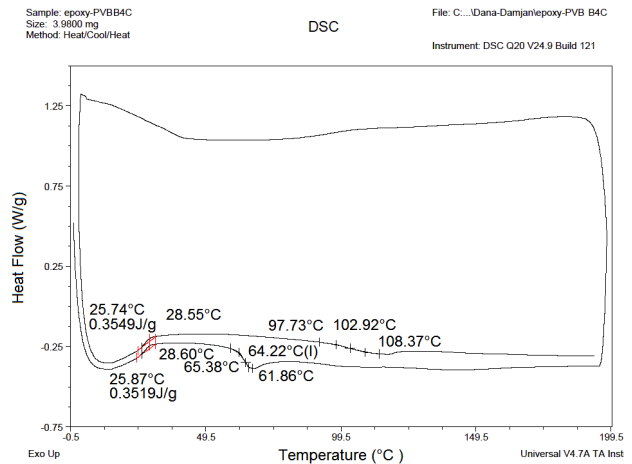


Figure 4. DSC curves for PVB/epoxy/B<sub>4</sub>C

Table 2. The T<sub>g</sub> values obtained by DSC

Sample	First heating T <sub>g</sub> , °C	Second heating T <sub>g</sub> , °C
PVB/epoxy	55.40	93.72
PVB/epoxy/B <sub>4</sub> C	64.22	102.92
PVB/epoxy/TiB <sub>2</sub>	47.63 – 52.20	78.74
PVB/epoxy/CNT	62.58 – 72.29	102.50
PVB/epoxy/INT-WS <sub>2</sub>	48.62 – 52.14	86.10

The glass transition does not occur suddenly, at a strictly determined point, it usually happens in a temperature range, as shown in Table 2. Above the glass transition temperature, the polymer chains have high mobility. At temperatures above T<sub>g</sub>, the chains may have enough energy to form ordered arrangements and undergo crystallisation, an exothermic process, so a certain amount of heat is

released. This is why in the second heating often less heat is needed to keep the heating rate of the sample pan the same as that of the reference pan [27,28]. This results in different values of  $T_g$  in the second recorded heat flow. The selected nanoreinforcements are known for their great heat resistance, but for different interactions with the matrix. This might explain the increase in  $T_g$  values of the samples with  $B_4C$  and CNT, and the decrease with INT- $WS_2$  and  $TiB_2$ , compared to the neat matrix sample. Besides the intrinsic heat resistance of the incorporated nanoreinforcements, the two polymers have a more complete curing reaction with incorporated nanostructures of  $B_4C$  and CNT, as proven by FTIR, which also contributes to the increased glass transition temperatures.

### 3.3 Hardness of polymer nanocomposites

For the prepared neat and reinforced polymer matrix samples, mean values of hardness are given in Table 3.

**Table 3.** Shore D hardness of the polymer composites

Sample	Shore D hardness	Standard deviation
PVB/epoxy	82.00	7.56
PVB/epoxy/ $B_4C$	90.88	2.17
PVB/epoxy/ $TiB_2$	96.14	1.07
PVB/epoxy/CNT	94.17	3.66
PVB/epoxy/INT- $WS_2$	97.50	0.84

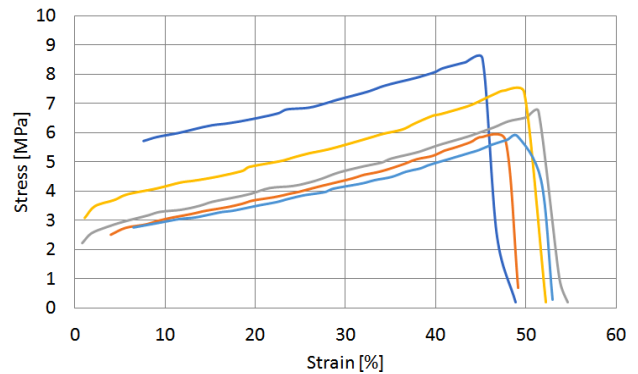
There is an evident increase in the Shore D hardness for all the samples with nanostructures of the technical ceramics. The most prominent increase was achieved with INT- $WS_2$  (~ 19 %) and  $TiB_2$  (~ 17 %). Most probably, this is a consequence of the dispersion of individual reinforcement in the matrix, their physical interaction with the matrix, the shape and size of reinforcement and the size of their active surface.

### 3.4 Tensile strength of polymer nanocomposites

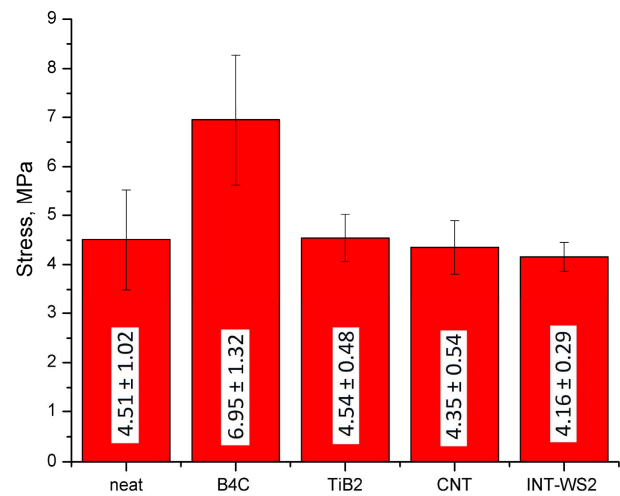
From the tensile test, the force-displacement curves were obtained, which were transformed into stress-strain dependencies, as for the example of a polymer matrix reinforced with  $B_4C$  (Fig. 5).

As it may be seen, there are certain deviations between the curves, characteristic for such composites, but the trend and the maximum values are very similar for all the 5 tested specimens of the matrix reinforced with  $B_4C$ .

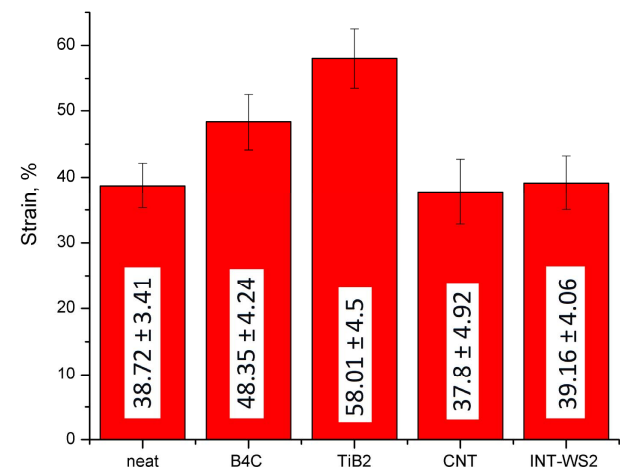
Similar curves were obtained for all the samples, and the tensile strength parameters were read and compared in Figures 6 and 7.



**Figure 5.** Stress-strain curves for the examined specimens of PVB/epoxy/ $B_4C$  (five specimens tested)



**Figure 6.** Maximum tensile stress for the tested polymer composites



**Figure 7.** Maximum tensile strain for the tested polymer composites

From the bar charts, it is evident that the tensile strength was significantly increased for  $B_4C$ , by 54 %. Also,  $B_4C$  and  $TiB_2$  have shown significantly higher maximum strain values compared to other samples. For other

nanoceramics, there were no significant improvements. This is most probably the consequence of poor interaction between the incorporated nanostructures with the polymers in the matrix. The composites consist of a polymeric continuous phase and the nanoparticulate filler as a discontinuous phase, and it is known that the dispersed nanostructures can alter the properties of the continuous phase due to their size and geometry, which may change the complexity of the polymer phase from the physical and chemical aspect [29]. This can also cause a certain decrease in the polymer matrix tensile strength. However, it is interesting to observe the difference in the elongation of the samples during the tensile test (Table 4). The neat polymer matrix has the most prominent elongation, while the samples with nanoreinforcements show lower displacements. This is because the nanostructures behave like discontinuities in the material, while neat polymer presents a continuous phase with a better possibility of polymer chains to reorganise while exposed to tensile load, i.e. provide higher ductility. Also,  $B_4C$  shows high elongation, due to good interaction of the particles with the matrix.

**Table 4.** Displacements during a tensile test of the polymer composites

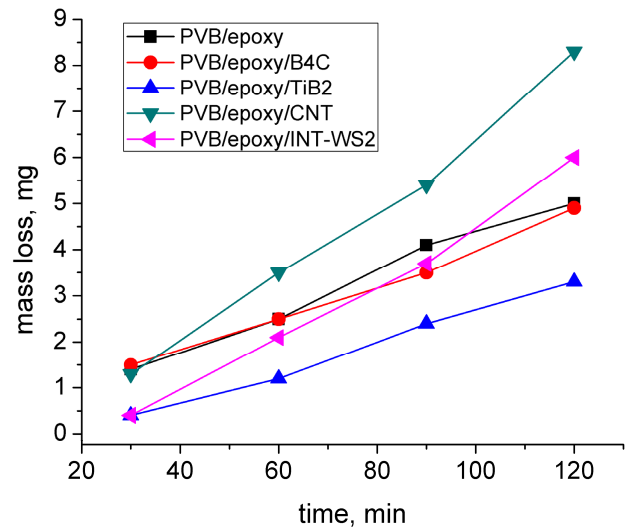
Sample	Displacement, mm	Standard deviation, mm
PVB/epoxy	9.97	0.84
PVB/epoxy/ $B_4C$	8.35	1.50
PVB/epoxy/ $TiB_2$	4.41	0.23
PVB/epoxy/CNT	6.53	0.88
PVB/epoxy/INT- $WS_2$	6.87	1.61

### 3.5 Resistance to cavitation wear

The mass loss of the polymer composite samples after each cycle of ultrasonic cavitation is given in Table 5 and the cumulative curves of the mass loss are given in Figure 8.

**Table 5.** Cavitation wear resistance – mass loss

Sample	Mass loss, mg			
	$\Delta m_1$	$\Delta m_2$	$\Delta m_3$	$\Delta m_4$
PVB/epoxy	1.4	1.1	1.6	0.9
PVB/epoxy/ $B_4C$	1.5	1.0	1.0	1.4
PVB/epoxy/ $TiB_2$	0.4	0.8	1.2	0.9
PVB/epoxy/CNT	1.3	2.2	1.9	2.9
PVB/epoxy/INT- $WS_2$	0.4	1.7	1.6	2.3

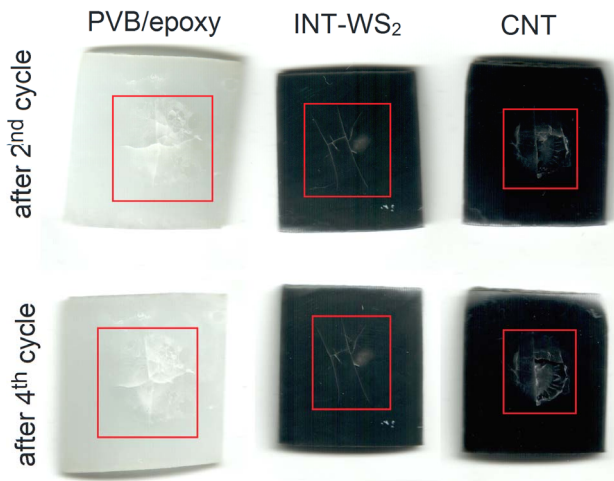


**Figure 8.** Cavitation wear curves for the tested polymer composites

Polymer composite samples had the following total mass losses: neat matrix 5 mg, composite with  $B_4C$  4.9 mg, composite with  $TiB_2$  3.3 mg, composite with CNT 8.3 mg and composite with INT- $WS_2$  6.0 mg.

All the samples have a gradual mass loss with a similar trend of cavitation wear curves. Composites with CNT and INT- $WS_2$  have a higher slope of the cavitation wear curves, which means that the ultrasonic cavitation wear takes more material from the surface, and this process happens faster in time. On the other hand, samples reinforced with ceramic nanoparticles  $B_4C$  and  $TiB_2$  have lower slopes, which means that the wear process goes slower. It may be noted that only  $TiB_2$  has significantly lower total mass loss compared to the neat polymer. In cases of other nanostructures, total mass loss was higher or equal to a neat matrix. The  $TiB_2$  was also the reinforcement which improved the hardness of the polymer matrix. Although it was expected that other structures that induced higher hardness, like INT- $WS_2$ , also improve the cavitation wear resistance, as in similar studies [30,31]. However, in this study that was not the case. If the reinforcement particles are too large or agglomerated, they can cause flaws in the matrix which weaken the polymer. Also, the reason for this might be poor interaction between polymer matrix and nanostructures, and possibly could be enhanced in further research by the addition of appropriate coupling agent for functionalisation of the nanostructures.

The appearance of selected samples after the test of cavitation wear is shown in Figure 9.



**Figure 9.** Polymer composite samples after ultrasonic cavitation wear test

On all the tested samples, in the beginning, only small cracks have been observed, which merged into bigger damaged areas as the cavitation cycles went on. It seems like the neat matrix PVB/epoxy has the widest area of damage, while this area is smaller for nanoreinforced samples, where, probably, the incorporated nanostructures have reduced the propagation of cracks. Due to the high content of brittle epoxy resin, the failure on the surface of the polymer composites reveals a brittle fracture. A similar mechanism was observed for this type of polymer composite in literature [30].

#### 4. Conclusion

A new hybrid polymer matrix system consisting of epoxy/PVB reinforced with technical ceramics nanostructures  $B_4C$ ,  $TiB_2$  and two kinds of non-ceramic reinforcements, CNT and INT- $WS_2$  was developed. FTIR analysis has revealed that the presence of nanostructures induced a more efficient curing reaction between the epoxy and PVB component, but that the nanoreinforcements do not interact chemically with the matrix. DSC analysis has demonstrated that the present nanoceramics significantly improve the thermal resistance of the polymer matrix, increasing  $T_g$  up to 30 °C. Hardness was most improved with the addition of INT- $WS_2$ , by 19 %. Tensile strength was increased for  $B_4C$  addition by 54 %. Cavitation wear resistance was examined by the application of ultrasound and has revealed that only the addition of  $TiB_2$  has improved the resistance to this kind of wear. Most probably the cause of individual effects that the incorporated nanostructures had, originates from their shape and size, as well as their interaction with the polymer matrix system. The new reinforced polymer composites might find

a wide range of potential applications: as a polymer matrix for lamination of advanced laminated composites for the automotive, naval and aerospace industries, in civil engineering, for coatings, for sports equipment, etc.

#### Acknowledgement

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