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The Effect of Single-Wall Carbon Nanotubes Content on the Properties of Polyurethane Nanocomposite

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This study aimed to investigate the influence of single-wall carbon nanotube (SWCNT) content on the mechanical properties of polyurethane (PU) nanocomposites. The SWCNT content varied from 0 wt% (reference sample) to 2 wt%. Tensile, hardness and Charpy impact tests as well as dynamic mechanical analysis (DMA) were performed. Based on the test results it was observed that an increase in the content of single-wall carbon nanotubes resulted in significant improvements in material strength and stiffness. Furthermore, atomic force microscopy (AFM) was used to examine microsurface topography of the samples and to obtain spectroscopic curves, based on which local elasticity was evaluated. Overall, performed measurements indicate that the incorporation of SWCNTs into PU matrix makes resultant nanocomposite stiffer and more resistant to deformation. The results highlight the potential of SWCNTs as effective reinforcement of polyurethane-based nanocomposites.

Keywords: Polyurethane, Nanocomposites, Single-wall carbon nanotubes, Nanofiller, AFM

1 Introduction

Polyurethanes (PU) are a class of polymers formed through the reaction of diisocyanates with polyols. They are commonly used and important polymers with wide variation of their properties [1]. The properties of the polyurethanes range from elastic to rigid thermoplastic and thermoset materials. Due to the versatile properties of the PU, the resulting material can be tailored according to the needs of a specific application of end products. The diverse applications of the PU include coatings, foams, adhesives and composites due to their excellent abrasion resistance, toughness, chemical and corrosion resistance and a wide range of mechanical strength. As a result of the PU potential and versatility of its properties, these materials are broadly researched [2].

Various nanofillers are used as reinforcement of the PU matrix to enhance its properties. Among others, carbon nanotubes are widely used as a reinforcing filler due to their exceptional mechanical properties, including the high Young's modulus and tensile strength. They also stand out with their thermal and electric properties. The CNTs can be categorized into two primary groups: single-wall carbon nanotubes (SWCNTs) and multi-wall carbon nanotubes (MWCNTs). Similarly to graphene, the CNTs are chemically bound by extremely strong sp^2 bonds, which combined with a natural tendency of the carbon nanotubes to bond with van der Waals forces, provides an opportunity to develop ultra-high-strength, low-weight materials with high electrical and thermal conductivity [3]–[6].

The use of the CNTs as the reinforcing filler in the PU matrix is widely studied. The effective reinforcement and thus resulting enhancement of nanocomposite properties depend on a high aspect ratio, nanofiller alignment, interfacial stress transfer and good dispersion [2], [7]. There are several ways to ensure efficient dispersion of the CNT in the polymer matrix including: melt blending (used for the production of thermoplastic polymer/CNT composites), solution mixing (during which the CNTs are dispersed and polymer dissolved in the same solvent, followed by solvent evaporation) and in situ polymerization (the reaction of the monomers or prepolymer in the presence of dispersed nanotubes) [8]. Additionally, the enhancement of interfacial interactions between the polymer and the CNT improves effective energy transfer within the system. Various methods such as sonication during the mixing stage as well as diverse approaches for chemically functionalizing the surface of the CNTs were investigated to improve interfacial interactions [9], [9].

The studies that investigated mechanical properties of various PU/CNT nanocomposites [10]–[13] report an increase in stiffness, which manifests itself in the higher tensile strength, tensile modulus, hardness and the lower elongation at break, with an increasing CNT content. In several studies [12], [13] it was observed that the increase in strength occurred only up to a certain concentration of the CNTs. However, when the CNT content was further increased, a slight decrease in strength was observed, which could be attributed to the higher concentration of the CNT agglomeration. The studies [14], [15] report improvement in thermomechanical properties of the thermoset PU reinforced with the CNT. The microstructure of PU/CNT nanocomposite have been studied using various techniques such as atomic force microscopy (AFM), scanning electron microscopy, transmission electron microscopy, and X-ray diffraction [16]–[19].

In this study, the thermoset PU nanocomposites reinforced with single-wall carbon nanotubes were subjected to tensile, hardness and Charpy impact tests as well as DMA temperatures and frequency sweeps. The AFM was employed to obtain surface topography and spectroscopic curves were used to study local mechanical properties.

2 Material

Matrix material was PX 521 HT prepared from components ISOCYANATE PX 521HT A and POLYOL PX 521HT B with mixing ratio 100 to 55. The matrix material was reinforced with the single-wall carbon nanotubes. The used SWCNTs had a diameter of 2 nm, a length of 5–20 µm and a purity of 95%. The content of the SWCNT varied from 0 wt% (reference sample without nanotubes) to 2 wt% (Tab. 1). Samples were prepared by vacuum casting:

- The required amount of nanotubes was mixed into the base material using ultrasound. Mixing was carried out in an ultrasonic mixer for 100 minutes.
- The material was degassed in a vacuum chamber for 10 min without stirring.
- This was followed by 2 min of further mixing.
- Then, mixed material was poured into the mould and cured at a temperature of 70 °C for 3 hours.
- Material was cooled and then removed from the mould.

***Tab. 1** Content of SWCNTs in the PU matrix*

Material label	PU-CNT 0	PU-CNT 1	PU-CNT 1.5	PU-CNT 2
SWCNT content (wt%)	0	1.00	1.50	2.00

3 Experimental methods

The following measurements were performed on the polyurethane nanocomposites PU-CNT 1 to PU-CNT 2 as well as the reference material PU-CNT 0:

- The tensile tests were performed in accordance with ISO 527 at the temperature of 20 °C and the loading speed of 100 mm/min. Ten type 5 specimens with the gauge length of 50 mm and the thickness of 2 mm per each material were tested. Tensile strength, tensile modulus and elongation at break were determined.
- The hardness tests were carried out using the Shore D method in accordance with ISO 868 at the laboratory temperature of 20 °C.
- The Charpy tests were conducted in accordance with ISO 179 at the temperature of 20 °C. Charpy notched impact strength, that expresses absorbed impact energy at breaking of a notched specimen, was evaluated. The samples with type A notch were used.
- The frequency and temperature DMA sweeps were performed. Measurements were conducted using plate-shaped samples with the dimensions of 20 mm x 10 mm x 2 mm. During the temperature DMA sweep, the samples were subjected to a tensile loading in the temperature range of -100–240 °C at 1 Hz. The frequency sweep was performed at the temperature of 20 °C and the frequencies varied from 0.01 Hz to 50 Hz.

Furthermore, the atomic force microscopy (AFM) was employed to evaluate topography of the microsurface as well as to evaluate its local elastic properties. The spectroscopic curves of each material were obtained at 10 different places on the surface, based on which local elastic properties of the samples were evaluated. For this evaluation, a significant part of the spectroscopic curve is section, between the point, when the tip of the probe touches the surface to the point where maximal force, that material surface can withstand, is applied by the tip. If the sample surface is stiffer then probe, the slope of this part mostly represents the modulus of the probe and otherwise, if the examined material is softer, slope of the curve allows examination of the surface local Young’s moduli.

Liner approximation of the mentioned part of the spectroscopic curves and Sneddon’s model [20], [21] were used to evaluate measured data and to calculate the ratios of the Young's moduli. Sneddon’s model expresses the dependence between the Young's modulus E and the load gradient dP/dh as:

$$\frac{dP}{dh} = \frac{2A^{1/2}}{\pi^{1/2}} E \; [Nm^{-1}] \tag{1}$$

Where:

A (m^2)…Contact area;

E (Pa)…Combined modulus of elasticity of the probe and the examined surface.

Given that the Young's modulus of the probe is significantly higher compared to examined material, the ratio of Young's moduli of the two samples can be simplified as follows:

$$\frac{E_1}{E_2} = \frac{\frac{dP_1}{dh_1}\frac{\pi^{1/2}}{2A^{1/2}}}{\frac{dP_2}{dh_2}\frac{\pi^{1/2}}{2A^{1/2}}} \Rightarrow \frac{E_1}{E_2} = \frac{\frac{dP_1}{dh_1}}{\frac{dP_2}{dh_2}} \tag{2}$$

The slope of approximated linear function represents load gradient:

$$y = kx + q, \text{ where } k = \frac{dP}{dh}, \tag{3}$$

$$\Rightarrow \frac{E_1}{E_2} = \frac{k_1}{k_2} \rightarrow E_2 = \frac{k_2 E_1}{k_1} \tag{4}$$

Equation (4) was used to compare the samples reinforced with the nanofiller PU-CNT 1 to PU-CNT 2 to the reference sample without the nanofiller PU-CNT 0.

4 Results and discussion

The tensile strength, the tensile modulus and the elongation at break were evaluated from the tensile testing and their average values are listed in the Tab. 2. and a graphical comparison of the results is shown in the Fig. 1. The tensile test results show an increment in the tensile strength and the tensile modulus of the PU nanocomposite with the increasing content of the SWCNTs. The tensile strength of the PU-CNT 2 compound with the highest nanofiller content (2.00 wt%) was 8.15% higher compared to the reference material without nanofillers PU-CNT 0 and the tensile modulus increased by 15.19%. The addition of the SWCNTs into PU matrix resulted in the lower values of the elongation at break. Specifically, the PU-CNT 2 sample exhibited an 8.59% decrease in the elongation at break compared to the PU-CNT 0 sample.

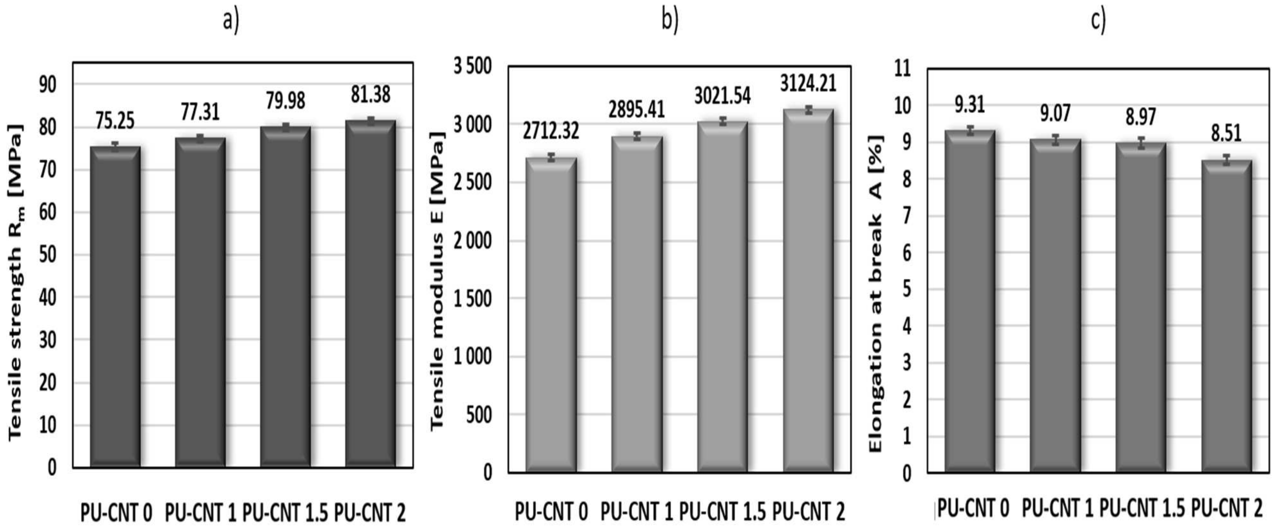

Fig. 1 *The tensile test results a) tensile strength b) tensile modulus c) elongation at break*

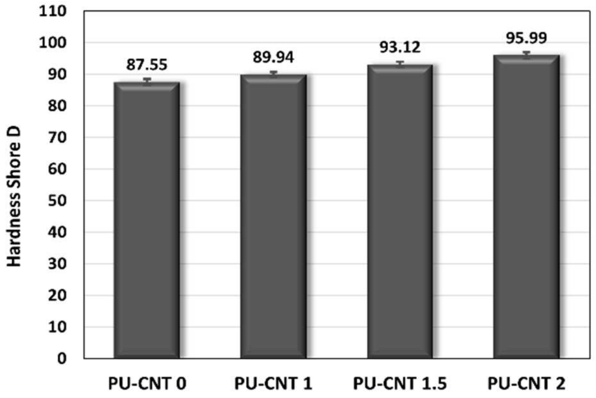

Fig. 2 *The Shore D hardness test results*

The Shore D hardness results are listed in the Tab. 2. and illustrated in the Fig. 2. The incorporation of the SWCNTs into the PU matrix led to the increased values of the Shore D hardness, with the PU-CNT 2 nanocomposite exhibiting a 9.46% higher Shore D hardness compared to the reference material without any nanofiller. This indicates a strengthening effect due to the interfacial interaction between CNTs and the PU matrix, that results in higher resistance of the nanocomposite to indentation and deformation caused by harder bodies.

The Charpy impact test results for each material are also listed in the Tab. 2. and a visual comparison is shown in the Fig. 3. Results indicate that the addition of the SWCNT fillers improves the impact resistance of polyurethane nanocomposites. The Charpy notched impact strength measured at 20 °C increased with the increasing content of the SWCNTs and a 9.61% increment was observed, when comparing PU-CNT 0 to PU-CNT 2. This can be attributed to the CNTs ability to act as barriers, that effectively dissipate and absorb energy during impacts, which helps impede crack propagation and enhances the material's ability to withstand impacts without failure.

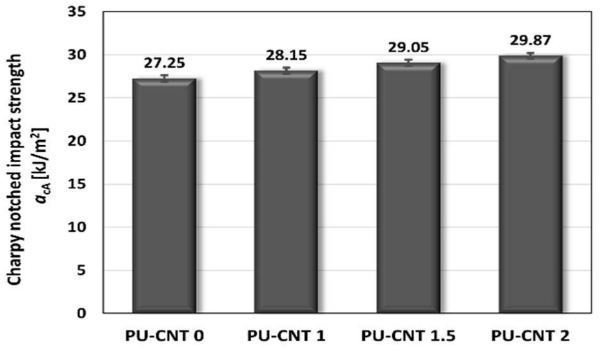

Fig. 3 *The Charpy impact test results (temperature of 20 °C)*

Tab. 2 *The results of tensile tests, hardness tests and Charpy impacts tests*

Material label	PU-CNT 0	PU-CNT 1	PU-CNT 1.5	PU-CNT 2
Tensile strength R_m [MPa]	75.25 ±0.88	77.31 ±0.75	79.98 ±0.79	81.38 ±0.78
Elongation at break A [%]	9.31 ±0.11	9.07 ±0.12	8.97 ±0.13	8.51 ±0.11
Tensile modulus E [MPa]	2712.32 ±25.21	2895.41 ±24.32	3021.54 ±26.32	3124.21 ±25.44
Hardness Shore D	87.55 ±0.91	89.94 ±0.84	93.12 ±0.85	95.99 ±0.97
Charpy notched impact strength a_{cA} [kJ/m²]	27.25 ±0.36	28.15 ±0.35	29.05 ±0.36	29.87 ±0.34

The CNTs possess exceptional mechanical properties, characterized by their high strength and stiffness. When integrated into polyurethane, CNTs reinforce the polymer matrix, resulting in enhancements in hardness, strength, and notch toughness of the resulting nanocomposite. Consequently, the nanocomposite becomes more stiffer and less susceptible to deformation due to the formation of bonds between the CNTs and the polymer matrix, that establish a supporting network within the material, enhancing load transfer and ultimately leading to the improved mechanical properties. The ultimate mechanical enhancement in the nanofiller-reinforced polymer composites can only be attained when the nanofiller is uniformly dispersed throughout the matrix, and the external load is efficiently transferred through a robust interfacial interaction between the filler and the matrix.

Dynamic Mechanical Analysis (DMA) is a technique used to study the viscoelastic properties of materials as a function of temperature, frequency or time. It provides insight into the behaviour of materials under dynamic mechanical load conditions. The storage modulus (E') is a measure of a material's ability to store elastic energy and it represents the stiffness of the material. The loss modulus (E'') is a measure of energy dissipation in the material. It represents the viscoelastic nature of the material and is associated with its ability to absorb and dissipate energy. The measured temperature dependencies of storage modulus E', loss modulus E'' and tangent of the phase angle *tan δ* are displayed in the Fig. 4–6 together with their frequency dependencies.

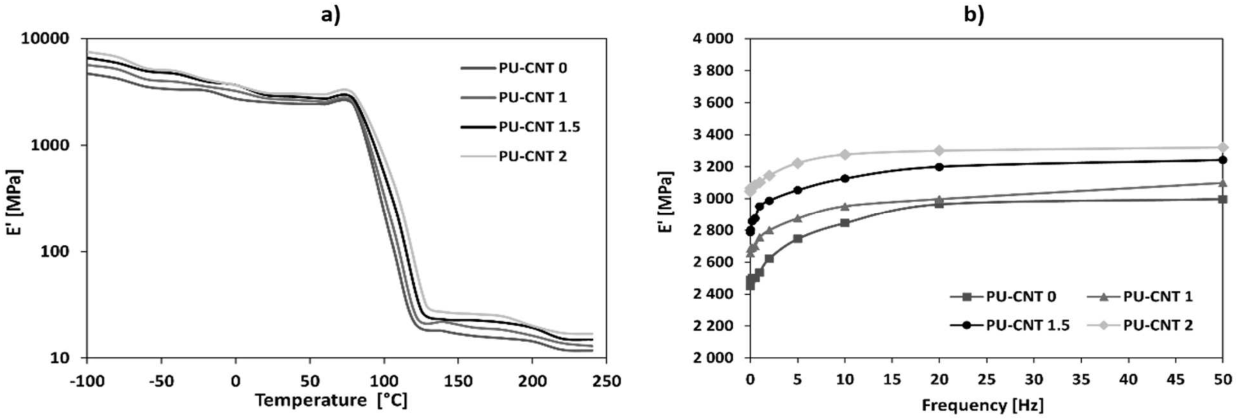

Fig. 4 *The DMA results – the storage modulus E': a) temperature sweep (-100–240 °C) at frequency of 1 Hz; b) frequency sweep (0.1–50 Hz) at temperature of 20 °C*

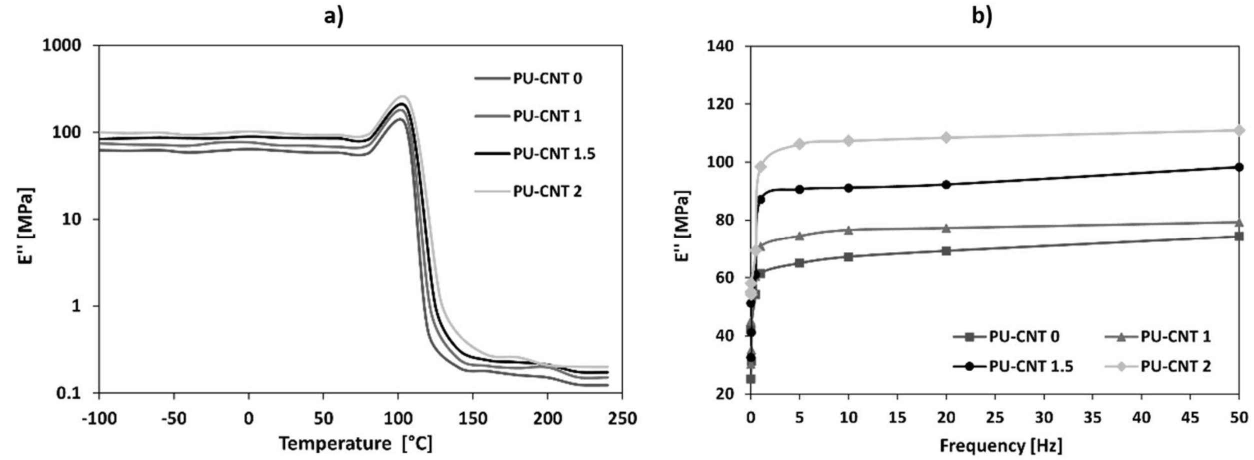

Fig. 5 *The DMA results – the loss modulus E'': a) temperature sweep (-100–240 °C) at frequency of 1 Hz; b) frequency sweep (0.1–50 Hz) at temperature of 20 °C*

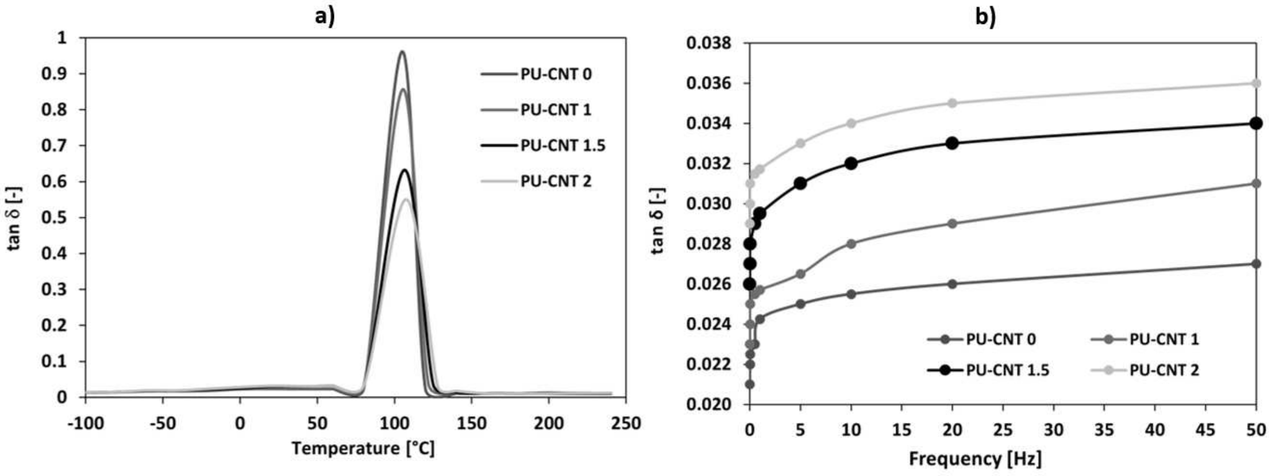

Fig. 6 *The DMA results – the tangent of phase angle tan δ: a) temperature sweep (-100–240 °C) at frequency of 1 Hz; b) frequency sweep (0.1–50 Hz) at temperature of 20 °C*

The SWCNTs affected the viscoelastic behaviour of the polyurethane nanocomposites and their inclusion into the PU matrix resulted in an increase in both the elastic and the viscous modulus. The storage modulus of the polyurethane matrix was enhanced by the addition of the CNTs due to the restriction of polymer chain movement, resulting from interactions between the carbon fillers and the polymer chains. The interfacial friction between the SWCNTs and the polymer matrix contributed to enhanced energy dissipation, leading to the higher values of the loss modulus. The tangent of phase angle *tan δ*, which represents the damping behaviour of the material, is given by the ratio of the loss modulus to the storage modulus. The higher *tan δ* values indicate superior energy dissipation and greater damping ability. The incorporation of the SWCNT fillers resulted in a reduction and slight shift of the peak in temperature dependence of the *tan δ*. This peak signifies the glass transition temperature (*Tg*) of the material. For the unfilled polyurethane, the peak value of the *tan δ* occurs around 105 °C. The addition of the carbon fillers slightly shifted the *tan δ* peaks, thereby altering the *Tg* value of the nanocomposite (PU-CNT 1 – 105.5 °C, PU-CNT 1.5 – 106.5 °C, and PU-CNT 2 – 107.5 °C). The glass transition affects the temperature-dependent viscoelastic properties, with a sharp drop in the elastic modulus from the values in scale of GPa in the glassy state to values in MPa scale in the elastic state. Similar shift in the *Tg* of the CNT – PU nanocomposites was also observed in [22] and [23]. A slight increase in the E' was also observed at subzero temperatures. The DMA frequency sweep revealed an increase in the storage modulus, the loss modulus, and the *tan δ* with both the increasing frequency of the applied tensile load and the amount of the added SWCNTs.

A NT-206 atomic force microscope was used to evaluate the PU samples. Examples of surface topography are displayed in the Fig. 7 and Fig. 8. For each sample characterised by different percentage of the SWCNT nanofiller, ten spectroscopic curves were created at different surface locations. The section of this curves was linearly approximated as shown in the example in the Fig. 9. The local elasticity was evaluated based on the slope of the linear function, which values for individual samples are listed in the Tab. 3. The higher absolute values of the slope were observed with the increasing SWCNT content as well as a larger variance of the values. Presence of the SWCNT caused higher differences in the local values of the Young's moduli. The average values of the slopes were used to express ratios of the reinforced PU to the reference sample PU-CNT 0 (Tab. 4).

Tab. 3 *The slope of the linearly approximated part of the spectroscopic curve*

Measurement	**PU-CNT 0**	**PU-CNT 1**	**PU-CNT 1.5**	**PU-CNT 2**
1	-1.6931	-1.8071	-1.9084	-1.9525
2	-1.6975	-1.8102	-1.8895	-1.9421
3	-1.6921	-1.8122	-1.9075	-1.9478
4	-1.6905	-1.8054	-1.8901	-1.9498
5	-1.6912	-1.8002	-1.8901	-1.9585
6	-1.6921	-1.8145	-1.8905	-1.9587
7	-1.6929	-1.8175	-1.9024	-1.9504
8	-1.6929	-1.8022	-1.8931	-1.9489
9	-1.6902	-1.8065	-1.8887	-1.9501
10	-1.6987	-1.8012	-1.8901	-1.9452
Average value of the slope k	**- 1.6931 ±0.0009**	**- 1.8077 ±0.0018**	**- 1.8950 ±0.0025**	**- 1.9504 ±0.0016**

Tab. 4 *The ratios of Young's moduli of the PU-CNT 1, PU-CNT 1.5 and PU-CNT 2 to the reference sample PU-CNT 0*

PU-CNT 1	**PU-CNT 1.5**	**PU-CNT 2**
$\boldsymbol{E_{PU-CNT\,1} = \frac{k_{PU-CNT\,1}E_{PU-CNT\,0}}{k_{PU-CNT\,0}}}$	$\boldsymbol{E_{PU-CNT\,1.5} = \frac{k_{PU-CNT\,1.5}E_{PU-CNT\,0}}{k_{PU-CNT\,0}}}$	$\boldsymbol{E_{PU-CNT\,2} = \frac{k_{PU-CNT\,2}E_{PU-CNT\,0}}{k_{PU-CNT\,0}}}$
$\boldsymbol{E_{PU-CNT\,1} = 1.0677E_{PU-CNT\,0}}$	$\boldsymbol{E_{PU-CNT\,1.5} = 1.1192E_{PU-CNT\,0}}$	$\boldsymbol{E_{PU-CNT\,2} = 1.1520E_{CPU-CNT\,0}}$

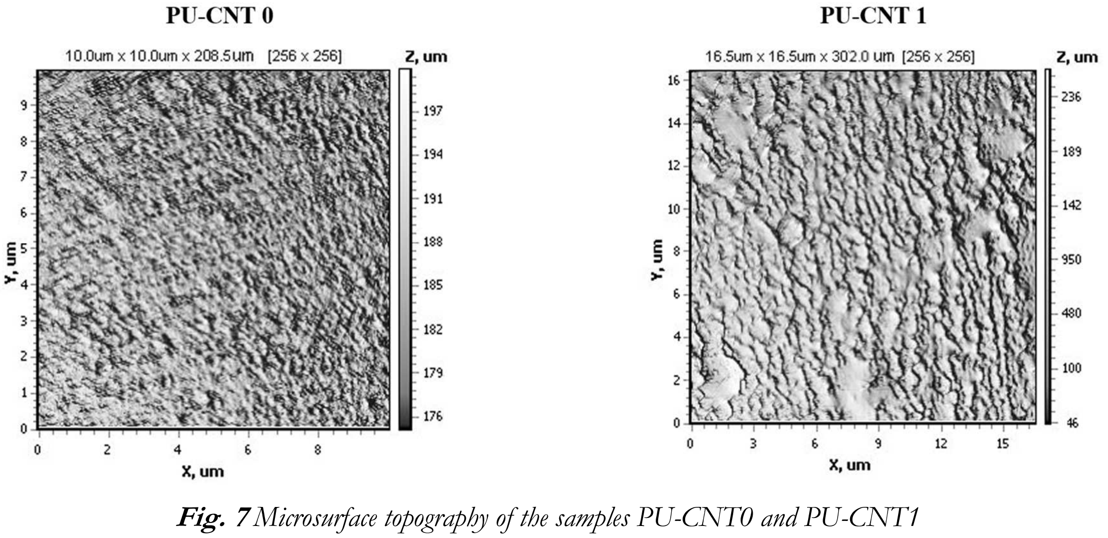

Fig. 7 *Microsurface topography of the samples PU-CNT0 and PU-CNT1*

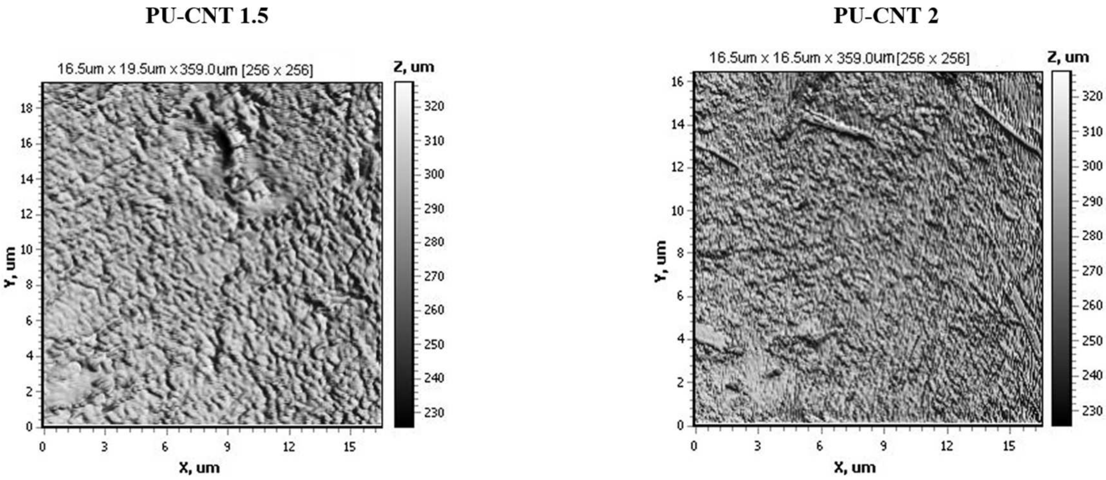
PU-CNT 1.5 **PU-CNT 2**

Fig. 8 Microsurface topography of the samples PU-CNT 1.5 and PU-CNT 2

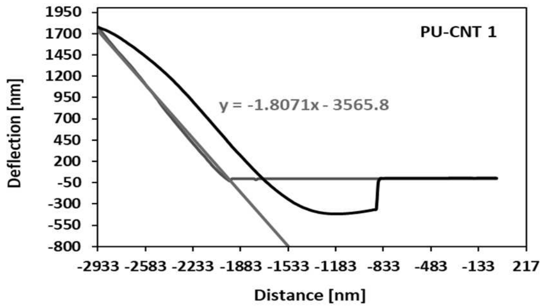

Fig. 9 Example of spectroscopic curve (PU-CNT 1)

Based on the comparison of the average slope values it can be concluded that the nanocomposite PU-CNT 2 has the highest value of the Young's modulus. The modulus of the material PU-CNT 0 without the SWCNT is the lowest, which correspond with the tensile test results. To determined particular values of the Young's modulus of the nanocomposites, the *E* of the reference sample CNT-PU 0 measured by the tensile testing was used (2712.32 MPa). The calculated values of the *E* are listed in the Tab. 5.

Tab. 5 Comparison of Young's moduli measured by tensile test and the AFM

Material	AFM results Young‘s modulus E [MPa]	Tensile test Young‘s modulus E [MPa]
PU-CNT 1	2895.91	2895.41
PU-CNT 1.5	3035.76	3021.54
PU-CNT 2	3124.51	3124.21

Good agreement was observed between the values measured by the tensile tests and the values obtained from the spectroscopic curves as the highest difference was less than 0.5% (PU-CNT 1.5).

Using the AFM, no significant agglomeration of the SWCNT was observed, however for further research more thorough investigation of the dispersion and orientation of the SWCNT would be useful using a microscope with higher resolution. Based on such results, mixing process could be optimized to further improve the nanocomposite properties.

5 Conclusion

Polyurethane is an incredibly versatile polymer with numerous applications, including its use as a matrix in nanocomposites. The addition of the carbon nanotubes (CNTs) to polyurethane can significantly improve the mechanical properties of the resulting nanocomposite. In this study, the incorporation of the SWCNTs into the PX 521 HT polyurethane matrix resulted in a desirable stiffening effect, which was manifested by an enhancement of the tensile modulus, the tensile strength, the hardness, and the Charpy notch impact strength and a decrease in the elongation at break. The DMA data provided insight into the viscoelastic behaviour of the PU nanocomposites, which further supports the findings from the tensile tests. Both the loss and the storage moduli increased with the increasing CNT concentration, the tangent of the phase angle decreased and the glass transition temperature slightly shifted towards higher temperatures.

The AFM was employed to investigate microsurface topography of the examined materials. Furthermore, the spectroscopic curves were obtained to investigate local elastic properties. Higher variance in the local values of the slope of their relevant section was noted in the samples reinforced with the SWCNT, suggesting existence of phases with different stiffness. The improvement in the polymer properties arises from the interphase formed between the nanofillers and polymer matrix, with different properties from those of the matrix [24].

Several critical factors significantly impact the mechanical properties of the nanocomposites, that include achieving a good dispersion of the CNTs within the polyurethane matrix, establishing strong interfacial interactions between the polymer matrix and the CNTs, and ensuring proper alignment of the CNTs within the polyurethane matrix. To achieve optimal dispersion and interfacial adhesion, thereby maximizing the benefits of utilizing CNTs in polyurethane nanocomposites, it is imperative to employ appropriate processing techniques. Therefore, an additional research could be useful to explore techniques such as chemical modification, functionalization or surface treatment of the CNTs to further improve the properties of the tested PU nanocomposites.

In conclusion, the addition of the CNTs to the polyurethane matrix enhances the mechanical properties of the resulting material, rendering polyurethane-based nanocomposites suitable for applications that demand high mechanical performance. These applications include structural components, automotive parts and sports equipment, among others.

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