Kaunas University of Technology

Faculty of Mechanical Engineering and Design

Investigation of Electrical Properties of 3D Printed Carbon Fiber Reinforced Composite Structures

Master’s Final Degree Project

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Supervisor

Kaunas, 2021

Kaunas University of Technology

Faculty of Mechanical Engineering and Design

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Industrial Engineering and Management (6211EX018)

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Task of the Master's final degree project

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1. Title of the project

Investigation of Electrical Properties of 3D Printed Carbon Fiber Reinforced Composite Structures

(In English)

3D spausdintų, anglies pluoštu armuotų kompozitinių konstrukcijų elektrinių savybių tyrimas

(In Lithuanian)

2. Aim and tasks of the project

Aim: To investigate electrical properties of 3D printed carbon fiber reinforced composite structures.

Tasks:

1. To develop a methodology to investigate the electrical properties of 3D printed carbon fiber reinforced composite structures;
2. To manufacture specimens using fused deposition modelling technique;
3. Carry out a fatigue experiments with a printed specimen and evaluate how the vibrations affect the electrical properties of the composite structure;
4. To perform a three-point bending tests of the specimens and evaluate the changes in electrical properties of the composite structure.

3. Initial data of the project

N/A

4. Main requirements and conditions

To manufacture specimen using fused deposition modelling technique. Specimens must be made according requirements and recommendations of ‘ASTM D 3039’ standard.

Project author	Darius Kabaila		
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Supervisor	Marius Rimašauskas		
	(Name, Surname)	*(Signature)*	*(Date)*
Head of study field programs	Regita Bendikienė *(Name, Surname)*	*(Signature)*	*(Date)*

Kabaila Darius. Investigation of Electrical Properties of 3D Printed Carbon Fiber Reinforced Composite Structures. Master's Final Degree Project, supervisor Assoc. Prof. Marius Rimašauskas; Faculty of Mechanical Engineering and Design, Kaunas University of Technology.

Study field and area (study field group): Production and Manufacturing Engineering (E10), Engineering Sciences (E).

Keywords: Carbon fiber reinforced composites, composite structures, electrical properties, fatigue experiment, three-point bending experiment.

Kaunas, 2021. 62.

Summary

In this Master’s final project, the electrical properties of 3D printed carbon fiber reinforced composite structures were investigated. In order to investigate the electrical properties of such structures, five carbon fiber reinforced composite specimens were manufactured by fused deposition modeling technique. For composite manufacture, PLA thermoplastic was used as a matrix material and carbon fiber was used a reinforcement material. External forces were applied to the specimens during fatigue and three-point bending experiments in order to investigate how electrical properties of composite structures changes when external forces act on them. The results obtained during the experiments revealed that when the external load was applied to the 3D printed carbon fiber reinforced composite, the overall resistivity of the composite structure always increases. Also, the study revealed that the resistivity of composite structure remains increased, despite the fact that external forces that acted on composite structure were cancelled. More importantly, the study revealed that it is possible to monitor structural damage done to composite structures due to a sudden and sharp increase in electrical resistivity.

Kabaila Darius. 3D spausdintų, anglies pluoštu armuotų kompozitinių konstrukcijų elektrinių savybių tyrimas. Magistro baigiamasis projektas, vadovas doc. Marius Rimašauskas; Kauno technologijos universitetas, Mechanikos inžinerijos ir dizaino fakultetas.

Studijų kryptis ir sritis (studijų krypčių grupė): Gamybos inžinerija (E10), Inžinerijos mokslai (E).

Reikšminiai žodžiai: Anglies pluoštu armuoti kompozitai, kompozitinės struktūros, elektrinės savybės, nuovargio bandymas, trijų taškų lenkimo bandymas.

Kaunas, 2021. 62 p.

Santrauka

Šiame magistriniame darbe buvo tiriamos 3D spausdintų, anglies pluoštu armuotų kompozitinių konstrukcijų elektrinės savybės. Norint ištirti tokių konstrukcijų elektrines savybes, naudojant sulydyto nusodinimo modeliavimo metodiką buvo pagaminti penki anglies pluoštu armuoti kompozitai. Kompozitų gamybai buvo panaudotas PLA termoplastikas, kuris atliko matricos vaidmenį bandinyje, o anglies pluoštas buvo pasirinktas kaip armuojanti medžiaga. Atliekant nuovargio bei trijų taškų lenkimo bandymus, bandiniai buvo apkrauti išorinėmis jėgomis, siekiant išsiaiškinti kaip keičiasi kompozitinių konstrukcijų elektrinės savybės, kai jas veikia išorinės jėgos. Eksperimentų metu gauti rezultatai atskleidė, kad pritaikius išorinę apkrovą 3D spausdintam, anglies pluoštu armuotam kompozitui, visada padidėja bendra kompozitinės struktūros varža. Taip pat, tyrimas atskleidė, kad kompozitinės struktūros varža išlieka padidėjusi, nepaisant to, kad išorinės jėgos, kurios veikė kompozitines struktūras, buvo panaikintos. Dar svarbiau tai, kad tyrimas atskleidė, kad galima aptikti konstrukcinius pažeidimus, padarytus kompozitinėms struktūroms, nes tokiu atveju labai stipriai ir staiga padidėja elektrinė kompozito varža.

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Introduction

With the advent of the fourth industrial revolution in the modern world, extremely large amounts of natural resources are consumed in daily production. The increasing consumption of natural resources is causing a shortage of certain materials and high prices. In order to remain competitive, the manufacturing sector companies are encouraged to search for new alternative materials and new production methods. In a past decade carbon fiber reinforced composites were extensively investigated and used as a replacement of metals. Nowadays, carbon fiber reinforced composites are widely used in civil engineering and high-performance structures, as such composites can be engineered to achieve mass reduction while maintaining strength approaching aerospace-quality aluminum. However, due to the properties of carbon fiber, such composite structures have lower level of damage tolerance and because of that it is needed to monitor the quality of such structures and to detect structural damage in a timely manner. Due to the extremely good electrical properties of carbon fiber, there is a possibility to monitor structural damage in real-time while observing changes in the electrical properties of composite structures. In this way it may be possible to avoid the excessive use of sensors and to use the composite structure itself as a sensor that is able to monitor structural damage emergence. Civil safety was a key factor which led scientists to investigate electrical properties of carbon fiber reinforced composites in order to manufacture composite structures that are able to track its own structural damages.

3D printing is becoming increasingly popular in the industry sector due to the simplicity of the technology and wide range of applications. Meanwhile, the production of carbon fiber reinforced composite structures by 3D printing technology is becoming an increasingly relevant and common case. However, there are very little research regarding electrical properties of 3D printed carbon fiber reinforced composites and the possibility to track structural damage emergence in real-time in such structures.

Aim: To investigate electrical properties of 3D printed carbon fiber reinforced composite structures.

Tasks:

1. To develop a methodology to investigate the electrical properties of 3D printed carbon fiber reinforced composite structures;
2. To manufacture specimens using fused deposition modelling technique;
3. Carry out a fatigue experiments with a printed specimen and evaluate how the vibrations affect the electrical properties of the composite structure;
4. To perform a three-point bending tests of the specimens and evaluate the changes in electrical properties of the composite structure.

1. Literature analysis

1.1. Composite materials

A composite material is a mix of two or more materials which have different chemical and physical properties. During manufacturing process these different materials are combined and they create a material which is dedicated to do a certain job, for example, to become lighter, stronger or even conductive to electricity [1]. Fig. 1 indicates the classification of composite materials.

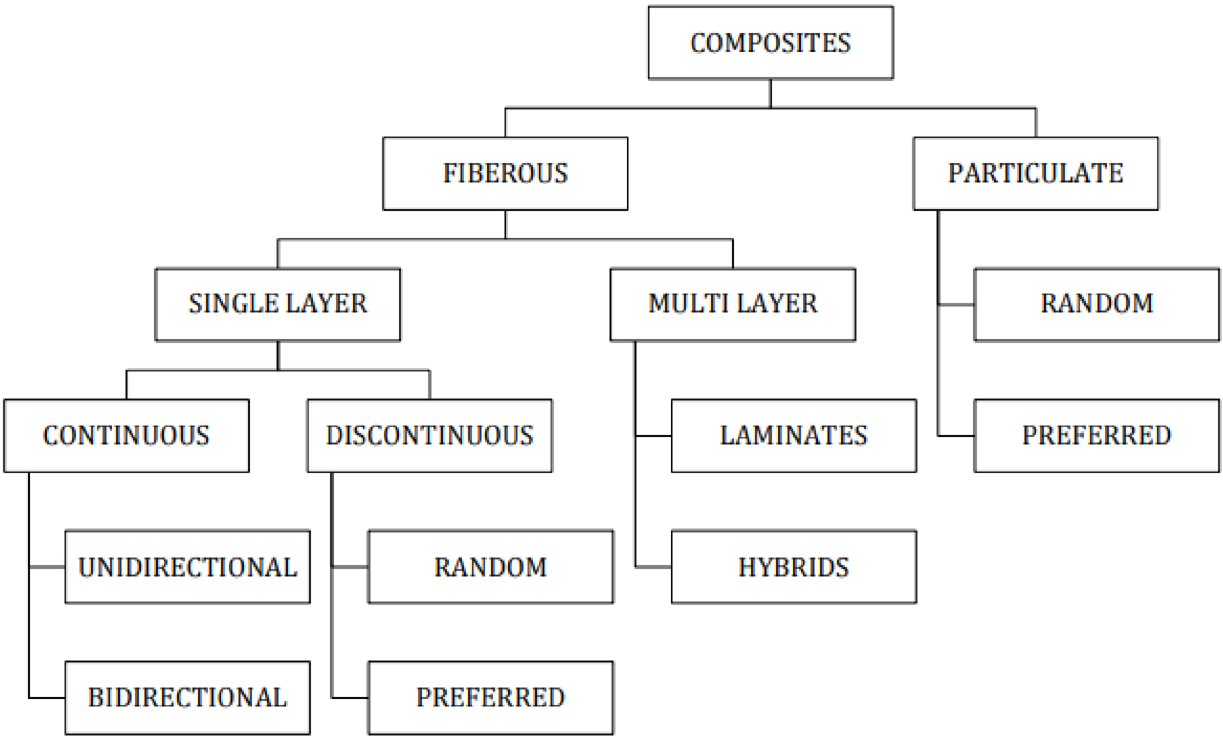

Fig. 1. Composite material classification [2]

From figure 1 it can be seen that composites are classified to two main groups – fiberous and particulate. Fiberous composites are then further grouped as single layer and multilayer composites. Single-layer composites can be made from a lot of layers but only if they have same orientation and properties. Meanwhile, multilayer composites can be made out of several layers and each layer is a single-layer composite with different fiber orientations. Single-layer composites can be divided to continuous composites with long fibers and discontinuous composites with short fibers. Usually discontinuous composites have random orientation, which in many cases can reduce overall mechanical characteristics of the composites such as strength and modulus. On the other hand, composites with random orientation are usually cheaper if compared to continuous composites which have predetermined fiber orientation which can be either unidirectional or bidirectional. Such composites are usually used in spheres where higher strength composites are required [2].

1.2. Structure of carbon fiber reinforced polymer composite

Carbon fiber reinforced polymer composites are composed of polymer resin and carbon fibers. During the manufacturing process carbon fibers are implanted in the polymeric resin and performs reinforcement of the composite [3]. Common structure of carbon fiber reinforced polymer could be seen in fig. 2.

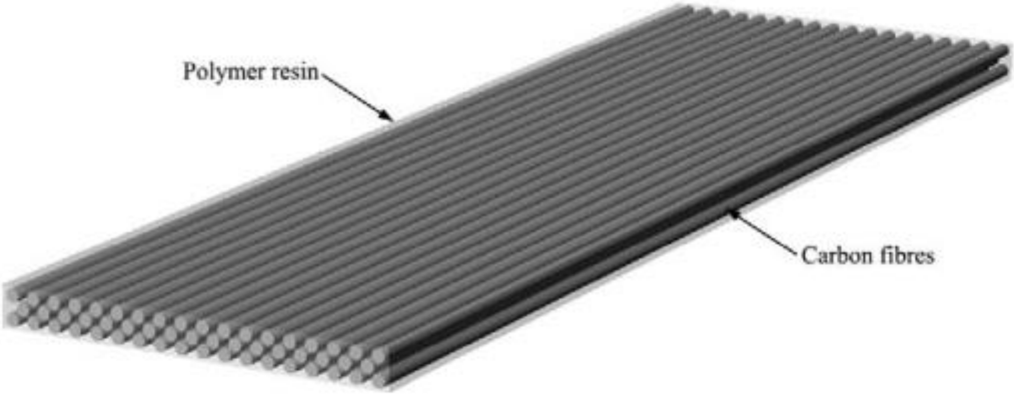

Fig. 2. Structure of carbon fiber reinforced polymer [3]

Carbon fibers consist of at least 90 wt.% and up to 100 wt.% carbon. It is known for great characteristics such as light weight, high strength, resistance to fatigue and high thermal and electrical conductivity. Carbon fibers has low thermal expansion which is why this material significantly increases the stability of the composite if compared to other matrices. As mentioned in previous paragraph carbon fiber composites usually could be grouped to two classes – composites with short fiber and composites with continuous carbon fibers. The latter significantly enhances characteristics of the composites such as electrical conductivity and modulus. Carbon fiber filaments are very thin and usually in composites their thickness is about 0.005 – 0.01 mm.

Meanwhile, polymer resins which are used in composites could be of two different types – thermoplastic resin and thermosetting resin. These resins have different molecular structures therefore their properties are different as well. Thermoplastic resins are related by van der Waals forces. These forces form a linear molecular structure which provides almost non movement restriction for molecular chains and as a result, this thermoplastic resin could be remelted after curing operation. Thermosetting resin are complete opposite material. In thermosetting resins, polymers are chemically bonded, which forms cross-linked molecular structure. This structure significantly restricts the movement of molecular chains and as a result this material becomes insoluble upon the application of heat after curing.

Huge differences between properties of carbon fiber and polymer resin makes carbon fiber reinforced composite a typical orthotropic material. In the fiber direction, carbon fiber reinforced composite displays mechanical properties of the carbon fiber, which is high strength and modulus. Meanwhile, perpendicular to the direction of the carbon fiber, composite displays polymer resin mechanical properties – low strength and modulus [3].

1.3. Electrical conductivity of carbon fiber reinforced polymers

Despite the fact that carbon fibers are good conductors, carbon fiber reinforced polymers show bad electrical conductivity properties. It is due to the polymers which are the major constituents of composites and most of the polymers are highly electrically insulating. Nevertheless, electrical conductivity of such composites could be increased by adding and randomly distributing carbon fibers across insulating polymer matrix. Carbon fiber reinforced polymers could be either electrically insulating or conducting. The electrical conductivity of the composite material depends very much on the direction of the carbon fiber, if the electrical conductivity measurements are made in the direction of the carbon fiber placement, the composite material is extremely electrically conductive, but if the measurements are made at 90 degrees to carbon fiber direction, the composite shows strong dielectric properties [4]. Under ideal conditions, carbon fibers are straight lines and the current flows only through one carbon fiber line and the current cannot jump from one carbon fiber line to another line because of insulating polymer matrix which separates these carbon fiber lines. If ideal conditions would be real, carbon fiber reinforced polymers would be highly insulating to the non-fiber direction. However, it is impossible to reach ideal conditions and electrical conductivity of carbon fiber reinforced composites could never be equal to zero as carbon fibers are not arranged in the straight line but are in a wavy line (fig. 3) [4].

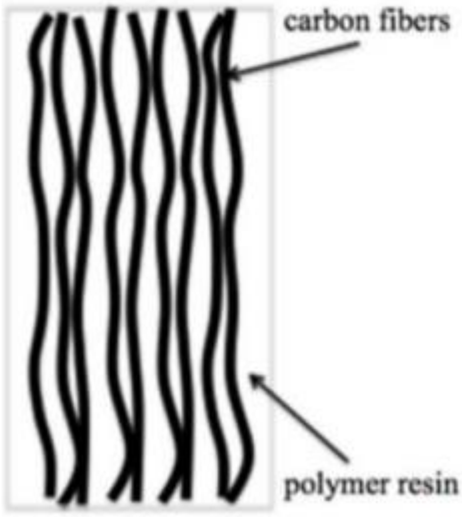

Fig. 3. Carbon fibers in the composite [5]

Conductive paths in the composite material are created when wavy lines of carbon fibers intersect with each other. Increasing the amount of carbon fibers in the carbon fiber reinforced composite will increase the number of conductive paths thus increasing electrical conductivity of the composite [6].

1.4. Damage detection in carbon fiber reinforced composites

Some composite materials have the ability to track their intrinsic properties, such as temperature, strain and even damage. This ability is usually called self-sensing ability. It is achieved by measuring electrical resistivity of the composite. Any mechanical structural changes alter the electrical conductivity of the carbon fiber reinforced polymer. These electrical resistivity changes could be detected by measuring resistivity between two or more electrodes when the electricity flows through carbon fibers which are impregnated in a polymeric material. Carbon fibers in composite structures creates conductive pathways through which electricity can flow and when the amount of carbon fibers increases in the composite material the number of conductive paths increases as well, thus, increases electrical conductivity of the composite material. These conductive paths are broken when damages are made to composite structure thus the resistivity of the carbon fiber reinforced composite increases [5].

The method to detect damages by measuring resistance change is called electrical resistance change method. Using this method to identify damages made to composite structures has several advantages. For example, while using this method it has no impact to static or fatigue strength. Also, it does not increase the load of the composite and displays good monitoring work in stiffness reduction generated by fatigue loads, so damage detection can be monitored without changing the characteristics of the composite. The link between conductivity and filler volume fraction has been experimentally proven. The volume fraction of the filler has a significant effect on the transverse electrical conductivity, which means that it affects the separation detection results when the electrical change method is used [5].

1.5. Electrical resistivity measurements

Over decades huge variety of models and methods were used in order to measure material's electrical resistivity. Plenty factors are known that may affect the choice which measurement method to choose. These factors may be the size and the shape of the specimen, is it in the form of powder pellet, crystallite, thin film or even single crystal. The desired measurement accuracy can also determine which measurement tool and method to choose. In order to measure electrical resistivity of a bulky and powder pellet specimens van der Pauw, Montgomery and Smith methods are usually used. Meanwhile two probes method (voltmeter – ammeter or ohmmeter measurements) may be used for low electrical conductivity specimens and four probes technique may be used for specimens with high electrical conductivity [7]. The electroconductive properties of carbon fiber reinforced composite structures are most commonly measured while using two and four probe techniques which is why only these two measurements methods are further discussed.

1.5.1. Two probe measurement method

Two probe technique is the simplest technique to measure electrical resistivity. During this method it is needed to measure a voltage drop across the specimen. Also, current that flows through the specimen is measured as well [7]. The schematics of two probe measurement could be seen in fig. 4.

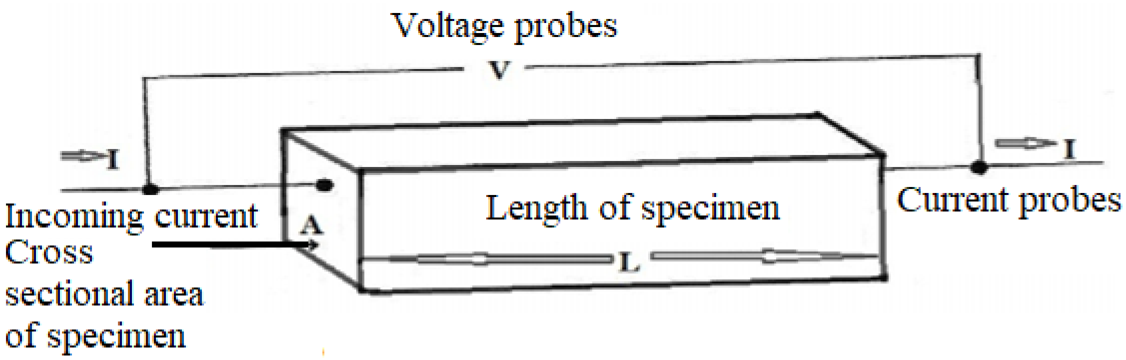

Fig. 4. The schematics of two probe measurement method [7]

When the current and voltage measurements are acquired, it is possible to calculate the bulk electrical resistivity using following formula [7]:

$$\sigma = \frac{V \cdot A}{I \cdot L} \tag{1}$$

Here, V is measured voltage, A is the cross-section area of specimen, I is the current and L is the length of the specimen;

Bulk electrical resistivity measurement unit is called ohm-meter (Ω·m).

1.5.2. Four probe measurement method

Four probe method is used to measure resistivity value of the sample. This method could be applied to measure semi-conductor materials, metallic materials and thin films. This method relies on four probes. Two probes are for current to flow and the other two probes are used to measure voltage drop between them. All four probes have to have good contact with the surface of the specimen and the distance between the probes are usually equal. When measuring the resistivity of the specimen, the voltage drop will occur when the current flows through two outer probes [8]. The schematics of four probe method could be seen in fig. 5.

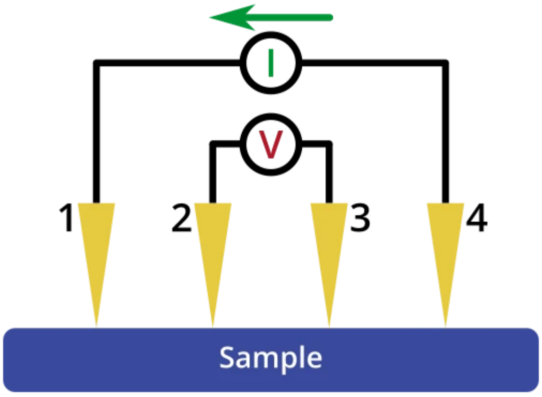

Fig. 5. The schematics of four probe measurement method [9]

As mentioned above, a direct current must be applied to two outer probes, which in this case are 1 and 4 (fig 5). Meanwhile, between inner probes (2 and 3) voltage drop is measured. When the voltage drops and direct current is known it is possible to calculate sheet resistance using following equation [9]:

$$R_s = \frac{\pi}{\ln (2)} \cdot \frac{\Delta V}{I} = 4.53236 \cdot \frac{\Delta V}{I} \tag{2}$$

Here, R_s is sheet resistance, ΔV is voltage change between probes, I is the direct current which is applied;

When the thickness of the component is known, following equation could be used in order to calculate component's resistivity [9]:

$$\rho = R_S \cdot t \tag{3}$$

Here, ρ is the resistivity of material and t is the thickness of the sheet;

1.6. Fused deposition modeling technique

Fused deposition modeling is a part of a rapid prototyping technology during which a thermoplastic filament is melted and then extruded through a nozzle onto a printing bed and in such a way, parts are being manufactured layer by layer. The thermoplastic filament is melted with the help of a liquefier and then the liquefied thermoplastic is being pushed down the nozzle by a solid filament, which is yet to be melted. The liquefier must be heated to the temperature which is greater than the melting point of the thermoplastic. When the nozzle moves along the printing bed, the liquid polymer is extruded and deposited onto the printing bed [10]. The schematics of fused deposition modeling could be seen in fig. 6.

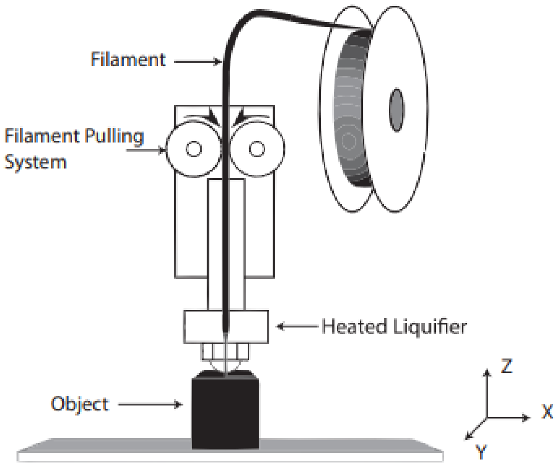

Fig. 6. Schematics of fused deposition modeling technique [10]

Some structures that are manufactured by fused deposition modeling are required to have structural support for any over-hanging faces. This structural support could be either break-away or water-soluble support. When a part which needed some sort of support is manufactured break-away support can be broken from the surface of the part, meanwhile, water-soluble support is separated from the part by immersing the entire structure in a solution that does not interact with the material from which the part was manufactured. Despite the fact that fused deposition modeling is quite a simple technique, it has a control model and the system must combine all printing parameters together, such as, layer thickness, extrusion speed, fan speed, feeding rate, etc. All the parameters are linked with each other as the speed of fused deposition modeling system which is dependent on the gear speed and feeding rate [10]. Meanwhile, feeding rate is dependent on the speed of liquefier at which it can melt the thermoplastic filament and feed it through the nozzle [10]. In order to produce a part using fused deposition modeling technique, a CAD file is needed. Usually, the file must be in *.stl* format which is used to slice the part using slicer software. The CAD file does not give detailed information about the part, it only gives parts’ outline. Meanwhile, slicer software specifies the infill of each layer and the path of the nozzle [10].

In general, fused deposition modeling is a technique that requires little funds while being a very reliable process during which relatively inexpensive materials are used. The technology could be easily used in industry or even home environment as it requires relatively simple installation and provides an opportunity to quickly manufacture parts with thin walls, meanwhile, emitting little waste. However, this technology has some drawbacks as well. The materials which are used in fused deposition modeling technique requires to have low melting temperature, which is why the most popular thermoplastics used are ABS (acrylonitrile butadiene styrene) and PLA (polylactic acid). Nevertheless, fused deposition modeling is a very useful technology for fast prototyping.

1.7. Composite preparation with enhanced mechanical and electrical properties

In many cases composites are expected to have better mechanical and electrical properties than materials they were made from. Such composites may be used as a replacement for metals when it is needed to have lightweight structure or when it is needed to have structure which is not only mechanically strong but also is electrically insulating or conductive. There are many ways to manufacture a composite material but it is crucial to know the best way in order to get a structure with as much as possible enhanced properties. Carbon fiber is used to enhance mechanical and electrical properties of the composite. However, printing orientation of the composite is as important as carbon fiber and combining knowledge about print orientation and carbon fiber ratios in the composite may lead to manufacturing a composite with enhanced electrical and mechanical properties. Sezar K., et al. [11] examined how electrical conductivity and mechanical properties of the samples are changing when different printing orientation and different amount of reinforcing material is applied to composite structures. During experiments researchers made specimens out of ABS (acrylonitrile butadiene styrene) thermoplastic, which was used as a matrix element and carbon fiber MWCNTs which served as a reinforcing material. In order to optimally evaluate mechanical and electrical properties of manufactured composites, twelve specimens were made according 'ASTM D412A' standard. These specimens were divided into two groups according printing orientation. Specimens of the first group were printed in horizontal 45° angle (-45, 45) and the other group of specimens were printed in horizontal concentric orientation (0, 90). Each group had six specimens with different amount of carbon nanotubes – 1, 3, 5, 7 and 10 wt.%. One specimen with 0 wt.% of carbon nanotubes were manufactured to compare how results change when carbon nanotubes are added to the specimen. All specimens were fabricated using 3D printer '3Dison PRO' [11]. The amount of each material that were used to manufacture these specimens are shown in table 1.

Table 1. The quantity of each material used to in specimens [11]

No	ABS (g)	MWCNT (g)	Ratio (wt.%)
1	15	0	0
2	14.85	0.15	1
3	14.55	0.45	3
4	14.25	0.75	5
5	13.95	1.05	7
6	13.50	1.5	10

'Instron' testing machine was used to test mechanical properties of manufactured specimens. When specimens which were made according horizontal concentric orientation (0, 90) were tested the data obtained showed that the tensile strength of the specimen with 1 wt.% of carbon nanotubes was lower than specimen with no carbon nanotubes. However, when specimens with 3, 5, 7 wt.% of carbon nanotubes were tested, the data showed that tensile strength energetically increases when higher amount of carbon nanotubes are applied to specimens. Surprisingly, when specimen with 10 wt.% of MWCNT was tested the results showed that tensile strength was lower by approximately 2 MPa if compared with 7 wt.% specimen. Nevertheless, experimental data showed that specimen with 7 wt.% MWCNT loading tensile strength increased by 28% if compared to blank ABS specimen. Meanwhile, the samples that were printed according raster orientation (-45, 45) showed much worse tensile strength results. The tensile strength of the samples with 1 wt.% and 3 wt.% of MWCNT was very similar to blank ABS specimens' tensile strength. However, specimens with higher amount of carbon nanotubes showed that their tensile strength steadily increases when the amount of carbon nanotubes increases, reaching approximately 48 MPa with 10 wt.% loading. Overall, tensile strength increased by approximately 20% for 10 wt.% when compared to blank ABS specimen. Mechanical test results showed that specimens which were printed according horizontal concentric orientation (0, 90) had higher tensile strength by approximately 20% when compared to specimens which were printed according horizontal 45° raster angle [11]. Mechanical test results could be seen in fig. 7.

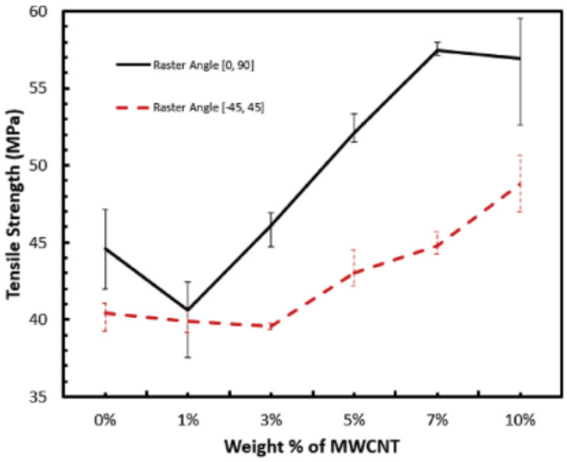

Fig. 7. Mechanical test results of all specimens [11]

After mechanical experiments all specimens were measured using 'Keithley 2400 Source Meter' in order to get their electrical conductivity results. Electrical conductivity results could be seen in fig. 8. Specimens with 0 wt.% and 1 wt.% MWCNT are electrically insulating composites regardless of their printing orientation. However, electrical conductivity results drastically changes for specimen with 3 wt.% of nanotubes which was printed according horizontal concentric orientation, while specimen with the same number of nanotubes and which was manufactured according horizontal 45° orientation still remains nonconductive [11].

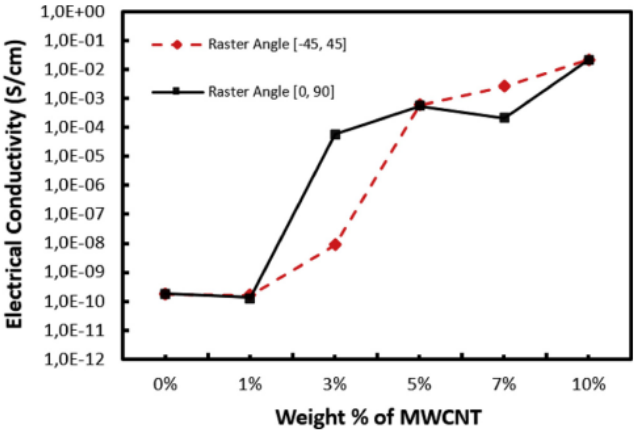

Fig. 8. Electrical conductivity results of the specimens [11]

Specimen with 3 wt.% which was printed in horizontal concentric orientation was more electrically conductive due to filler material which was continuously distributed. Meanwhile, specimen which was manufactured according horizontal 45° raster angle had its filler material distributed in a discontinuous way. Horizontal concentric printing orientation led to accomplish high electrical conductivity while keeping filler material at a very low quantity. Nevertheless, the results that were obtained during further research showed that electrical conductivity is very similar regardless of print orientation. The main difference is that percolation threshold was reached at different amounts of carbon nanotubes used in composites. While printing according to raster angle (0, 90) percolation threshold was reached at 3 wt.% MWCNT and while printing according raster angle (-45, 45) percolation threshold was reached only at 5 wt.% MWCNT. Experiment concludes that while printing according horizontal concentric (0, 90) orientation it is possible to manufacture electrically conductive composite with less carbon nanotubes [11].

In a similar study Fambri L., et al. [12] examined composites which were manufactured using fused deposition modeling technique. The composites were printed in three different orientations – horizontal concentric, horizontal 45° angle and vertical concentric. Same as in previous study composites were manufactured from ABS and carbon nanotubes and their electrical conductivity was measured. However, unlike in the previous article this time researchers examined specimens with different amounts of carbon nanotubes – 1, 2, 4, 6 and 8 wt.% [12]. Printing orientations could be seen in fig. 9.

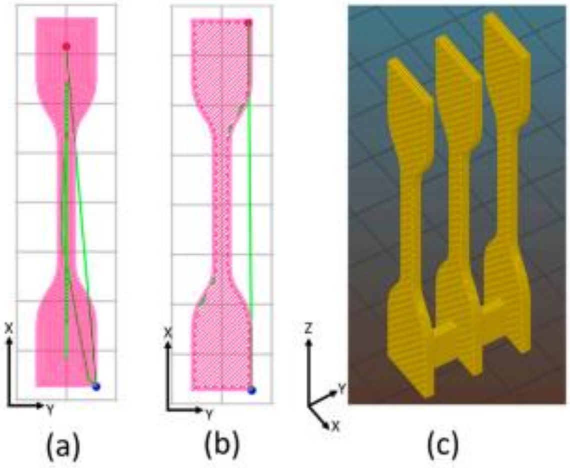

Fig. 9. Printing orientations: a – horizontal concentric, b – horizontal 45°, c – vertical concentric [12]

During this research, filaments were firstly fabricated which consisted of 1, 2, 4, 6 and 8 wt.% carbon nanotubes and their electrical conductivity was measured according four-probe technique. Experimental data showed that filaments which had up to 2 wt.% carbon nanotubes exhibited strong dielectric properties and their electrical resistivity could not be measured by four-probe configuration. However, filaments which had more than 4 wt.% carbon nanotubes showed serious increase in electrical conductivity if to compare with neat ABS thermoplastic which has electrical resistivity equal to 10^{15} Ω × cm. Electrical conductivity results of three different filaments could be seen in fig. 10.

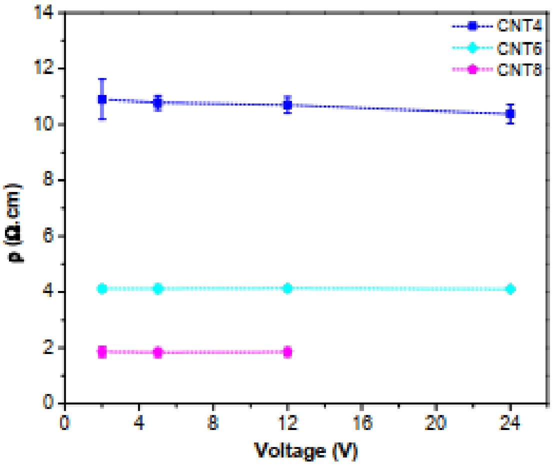

Fig. 10. Electrical conductivity of manufactured filaments [12]

It can be seen that the lowest electrical conductivity showed filament which had 4 wt.% of nanotubes (CNT4) and it was equal to around 11 Ω × cm. Filament with 6 wt.% nanotubes demonstrated much greater electrical conductivity results equal to 4.1 Ω × cm (CNT6). Lastly, filament which had 8 wt.% carbon nanotubes (CNT8) showed the greatest electrical conductivity with voltage set to 12 V. Unfortunately, it was impossible to measure conductivity with voltage set to 24 V due to the fact that the filament started to melt due to resistive heating effect. Since it was impossible to measure real electrical resistivity of CNT8, filament with 6 wt.% of nanotubes was used to manufacture the specimens [12].

Three specimens were manufactured according three different printing orientation and their electrical conductivity was measured according four-probe technique. These specimens were connected to 24 V voltage DC (direct current) power supply ‘IPS303DD’ and the current flow between external electrodes were examines using ‘IDM 67’ multimeter. The electrical conductivity results could be seen in fig. 11.

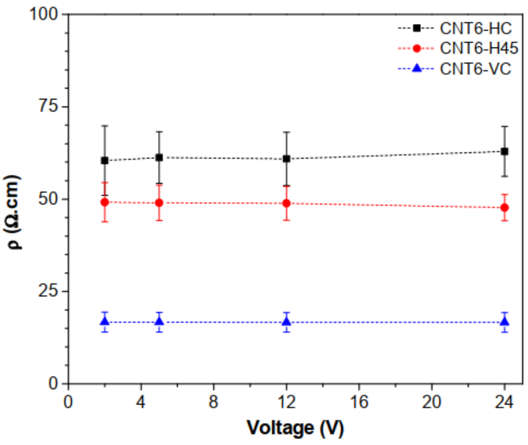

Fig. 11. Electrical conductivity results [12]

Experimental data showed that the specimen which was printed according vertical concentric orientation had the highest electrical conductivity equal to 20 Ω × cm. Meanwhile specimen printed according horizontal concentric orientation had the lowest electrical conductivity equal to 60 Ω × cm. Also, it should be noted that experimental results revealed that filament had approximately 20 times greater electrical conductivity than manufactured specimen. This could be explained by the fact that the gaps between carbon nanotubes are much smaller in filament material than in manufactured specimen and it is much easier to create electroconductive path [12].

To conclude previously reviewed studies, selecting the right amount of carbon fibers and the right printing orientation is of severe importance. This could lead to manufacturing a specimen with the best mechanical and electrical properties while using smaller amount of carbon nanotubes. Both studies found out that specimens with very low amount of carbon nanotubes (3-4 wt.%) became electrically conductive and that selecting the right printing orientation may lead to achieving percolation threshold at a lower amount of carbon nanotubes. The best printing orientations according studies are vertical concentric and horizontal concentric. Specimens manufactured according these orientations will require less carbon nanotubes in order to achieve better mechanical and electrical properties of the composite.

1.8. Electrical conductivity changes during fatigue and bending tests

Wang Z., et al. [13] examined how electrical conductivity is changing in composite structures during bending and fatigue tests. During the experiment, composite structures were manufactured out of PP (polypropylene) and carbon nanotubes (CNT). In order to manufacture the composite, two major processes were used: nanofiller dispersion and injection molding. In order to contain the properties of polypropylene, very low concentration of carbon nanotubes (0.5 wt.%) were mixed with the polypropylene matrix during the dispersion process. For the fatigue test, researchers manufactured a specimen in dimensions as follows – 25 mm in length, 5 mm in width and 2 mm in thickness and they also applied constant amplitude strain equal to 4%. Meanwhile, the overall dimensions of the second specimen for the three-point bending test were – 60 x 10 x 3 mm. During both experiments electrical resistivity was measured using a four-probe method in order to investigate how external mechanical changes alters electrical conductivity of the composite [13]. The experimental schemes are demonstrated in fig. 12.

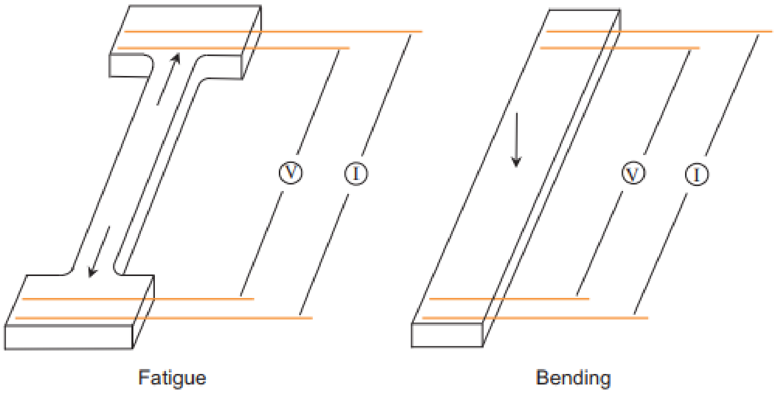

Fig. 12. The schematics of fatigue and bending tests [13]

For the fatigue testing of the specimen cyclic loading was used and the frequency was set to be 1 Hz. The stress that was enforced to the specimen was equal to 0.2 MPa. Fatigue test results could be seen in fig. 13. Since the start of the experiment, when the cyclic loading was applied, it can be seen that electrical resistivity of the specimen kept increasing till the end of the experiment. The electrical conductivity kept decreasing because the structure of internal electrically conductive paths was damaged and this resulted in an increasement of resistivity [13].

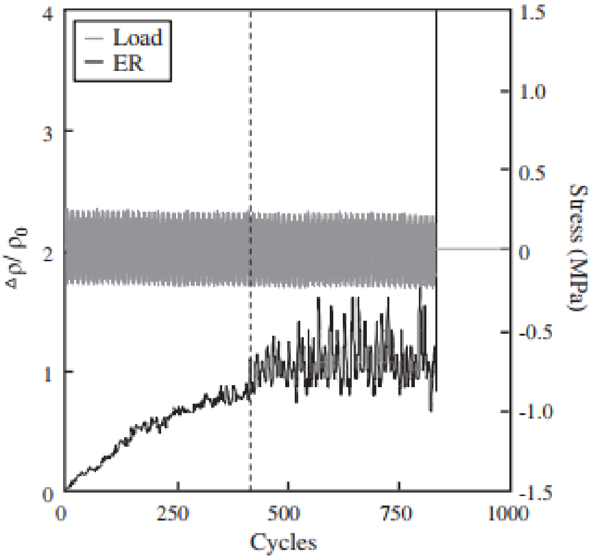

Fig. 13. Experimental results of fatigue test [13]

Since the start of the experiment, electrical resistivity was steadily increasing but after 400 cycles visible microcracking appeared and the resistance curve became highly volatile. The change in resistance calculated after each cycle became larger and more unstable till approximately 800 cycles. Since the start of the experiment and till the end of it. specimen experienced severe fatigue and because of that the specimen was broken. The fact that specimen was broken also could be seen from the fig. 13 when electrical resistivity rose significantly after 800 cycles. The large increase in electrical resistance can be explained by the fact that internal conductive paths were broken.

During another experiment a specimen was tested in three-point bending machine and its electrical conductivity change was measured. This time the force that was applied to the specimen was equal to 10 kPa. The results of the experiment could be seen in fig. 14.

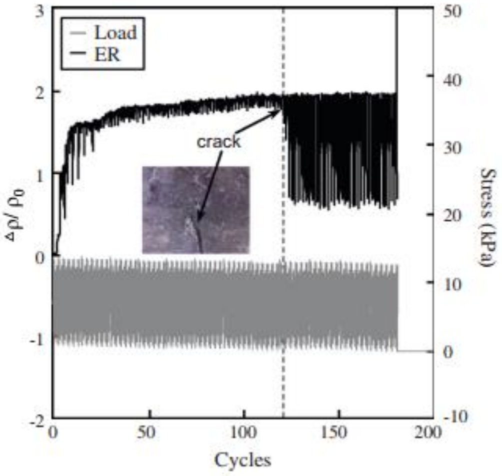

Fig. 14. Three-point bending results [13]

The same as in the previous experiment shortly, after the force was applied, the electrical resistivity increased and kept increasing till approximately 50 cycles then the change of electrical resistivity settled down. However, after 120 cycles, visible microcracking appeared and the electrical resistivity change became unstable till approximately 180 cycles when electrical resistivity suddenly increased. Significant increment of the electrical resistivity could be explained by the fact that the specimen and its conductive paths were broken to pieces [13].

In a similar study Luan C., et al. [14] examined the change in electrical conductivity in composite structures when load is applied on different positions of the composite surface. The purpose of this research work was to investigate if it is possible to track real time conditions of the composite regardless of where the composite structure was loaded. Fused deposition modeling technology was selected to manufacture the specimen for the experiments. The specimen was manufactured out of PLA (polylactic acid) thermoplastic which served as a matrix material and carbon fiber (T300B-3000-40B) which was used as a reinforcing and conductive material and its dimensions was – 300 x 40 x 10 mm. Carbon fiber tow which included 3000 filaments (7 µm each filament) was impregnated eight millimeters below the tension surface. After the specimen was manufactured, researchers prepared special places for the electrodes in order to be able to perform measurements of resistance change during the experiment. Electrodes were attached directly to carbon fiber tow with the help of silver paste. Chloroform was used to get rid of PLA material from the surface of carbon fiber [14]. The gap between the electrodes was equal to 180 mm. The specimen which was prepared for the experiment could be seen in fig. 15.

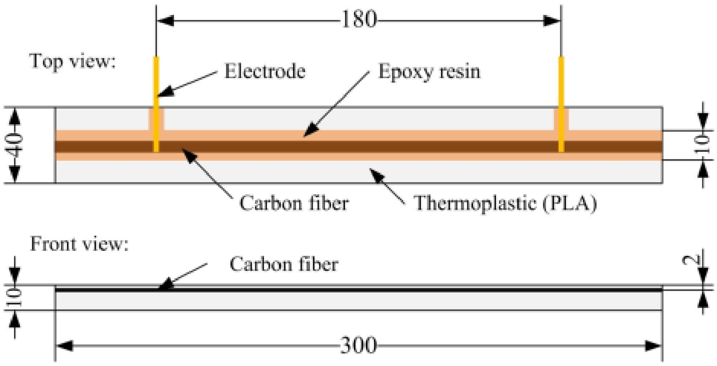

Fig. 15. Manufactured specimen for the experiment [14]

Three-point testing machine ‘WDW-100’ was used during the experiment and load was applied to 7 different positions. During all loading procedures the electrical conductivity was measured according two-probe method. The speed of loading and unloading of the testing machine was programed to be 2 mm/min, the maximum force was set to 110 N while minimal force was equal to 10 N. When the testing machine reached its maximum force, the load was sustained for 10 s and the electrical change was measured using ‘TH2516’ direct current resistance meter during loading and unloading procedures [14]. All loading positions could be seen in fig. 16.

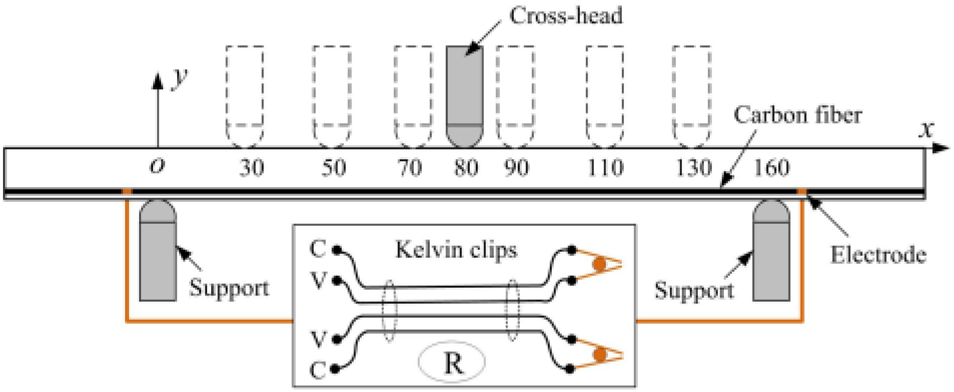

Fig. 16. The structure of the experiment [14]

The whole experiment consisted of a total of seven different loading locations. First of all, force was directed to the center of the specimen which is 80 mm away from the supports and electrical conductivity change was measured. The results of the first loading measurements could be seen in fig. 17.

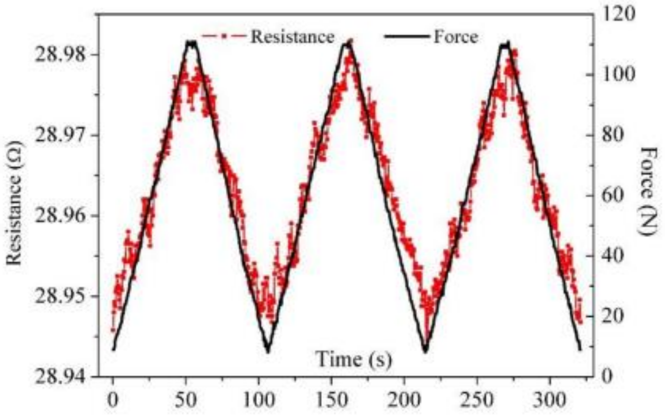

Fig. 17. Electrical conductivity changes during first loading procedure [14]

The experimental data showed that electrical resistivity increases linearly when load that is applied increases and also decreases linearly when the force decreases. When the experiment began, firstly, minimal force was applied which was equal to 10 N and the resistivity of the specimen was approximately 28.945 Ω but when the force was increased to 110 N the resistivity of the specimen raised a little bit and reached 28.975 Ω. The loading and unloading procedure were then repeated several times without any delays between loading and unloading procedures and it proved that resistivity increases when the force applied to the specimen increases. Also, the force which was applied during the experiment did not affect the structure of the specimen in any way because after unloading procedure the resistivity of the specimen dropped to baseline and when the force was applied once more, the resistivity raised to approximately the same reading as before. Overall, the change in electrical resistivity was measured to be approximately 0.03 Ω [14]. After the first loading location was analyzed, load was applied to remaining six positions and the results could be seen in fig. 18.

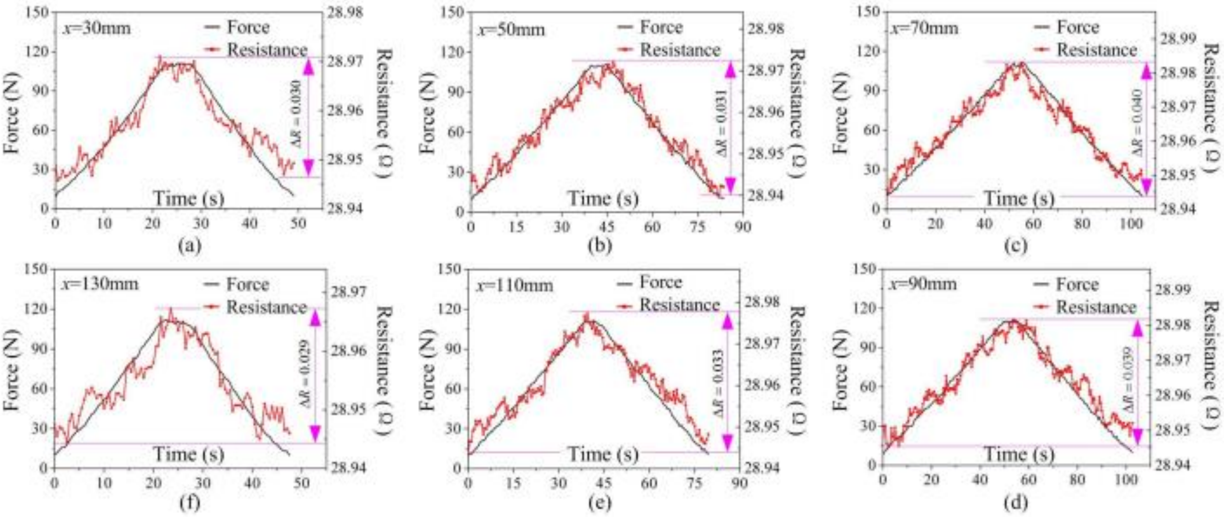

Fig. 18. Experimental results after load were applied to remaining six positions [14]

After the experiment was complete and all data were registered it was clear that there is an obvious correlation between electrical resistivity and external load. When the force was applied to remaining six positions very similar results were obtained – resistivity of the specimen rises when the external force rises. The smallest increase of the resistivity was obtained at a position which was 130 mm away from the first support and the change of the resistivity was equal to 0.029 **Ω**. Meanwhile, the largest increase of the resistivity was measured at a position which was very close to the center between the electrodes – 70 mm away from the first support and the resistivity change was approximately 0.04 **Ω**. Despite the fact that resistivity change is very minimal there is a clear correlation between external load and resistivity regardless of where the specimen is loaded between the electrodes [14].

To summarize two previously reviewed studies it can be said that carbon fibers not only enhance mechanical properties of the composite structures but also creates electrically conductive paths in these composite structures which provides the opportunity to track external loading of the specimen. Both studies approached their experiment in a different way. First study distributed carbon fibers evenly throughout the sample meanwhile in a second study single continuous carbon fiber tow was impregnated into the PLA thermoplastic. Nevertheless, very similar results were obtained. In both studies, the electrical resistivity of the specimen changes during any type of external loading. Both studies proved that load that appeared on the specimen surface could be detected by measuring resistivity of the specimen and in such way external damages of the composites could be monitored.

1.9. Electrical resistivity change of pre-cracked composite structure

Yasuhide S., et al. [15] examined electrical responses of pre-cracked composite structure under external loading. The purpose of the experiment was to investigate how cracks can affect electrical conductivity during tensile tests. The specimen was manufactured using polycarbonate as a matrix thermoplastic and carbon nanotubes as a reinforcing material. The specimen consisted of 2.5 wt.% carbon nanotubes and its dimensions was 180 x 18 x 1 mm. Artificial crack was made with a razor blade when the specimen was manufactured. The crack was 9 mm in depth and 1 mm in width. In order to get valuable electrical resistivity results silver paint was painted on the surface where electrodes were needed. The electrodes were placed 20 mm away from the center of the specimen and overall distance between two electrodes were 40 mm. Before tensile test experiment, nonconductive plates were attached to both composite ends in order to isolate test machine from the specimen. Servo-hydraulic testing machine was used for the experiment which was done in room temperature. The tests were performed in displacement control mode at a cross-sectional speed of 0.6 mm/min. Voltage-current meter was used during the experiment in order to measure the resistance change of the composite structure. The structure of the specimen prepared for the experiment could be seen in fig. 19.

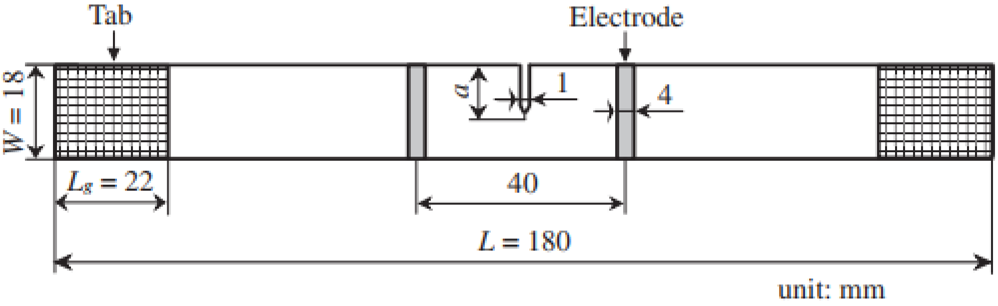

Fig. 19. The structure of the specimen [15]

Firstly, an experiment to find out crack extension amount was made as a function of displacement. The amount of crack extension could be seen in fig. 20. It should be noted that crack extension measurements were made before the load reached its peak value. At the beginning of an experiment, load-displacement curve (P-∂) was linear although it eventually became nonlinear. When displacement value reached approximately 0.8 mm, the crack started to extend and kept increasing till load reached its peak and specimen was broken to pieces. Just before reaching the maximum load, it can be seen that the crack extension is about 1.7 mm while displacement value is approximately 2.1 mm [15].

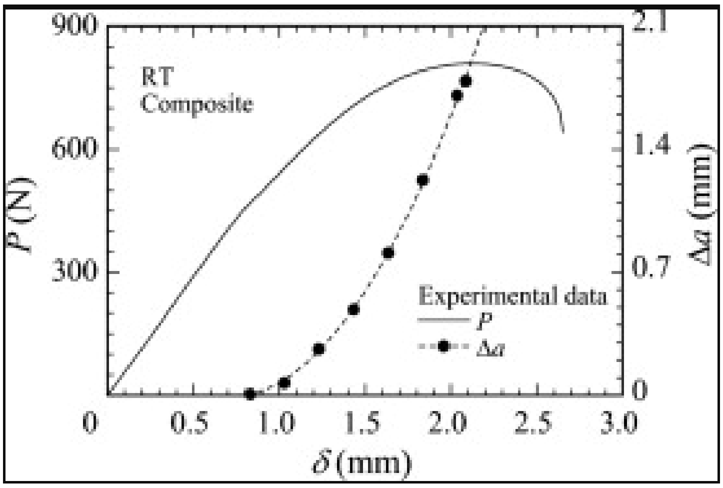

Fig. 20. The amount of crack extension during tensile testing [15]

After crack extension experiment, another experiment was made in order to investigate how much electrical resistivity changes during tensile testing. Before the start of an experiment, the resistivity of the composite was measured and it was equal to approximately 11 kΩ. Noticeable resistance change could be seen when displacement value reached 0.6 mm. At that point the resistivity of the composite started to increase and kept increasing till the end of the experiment when the specimen was broken to pieces. Just before fatal damage to the specimen it can be seen that resistance change reached 0.26 Ω at a displacement equal to 2.3 mm [15]. The results of electrical resistance measurements could be seen in fig. 21.

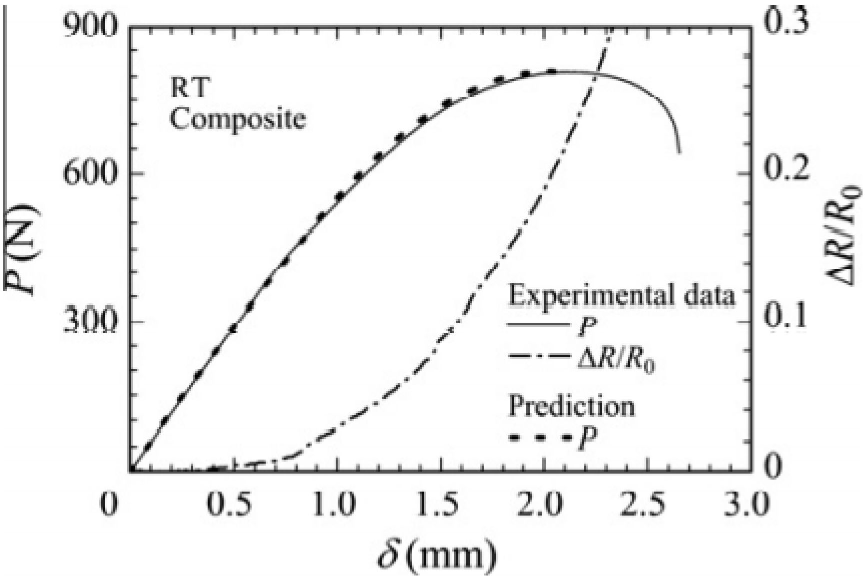

Fig. 21. The results of electrical resistance measurements [15]

Authors of this study reveals a steady relationship between the crack length and the electrical conductivity. Even though cracks appear on the surface of the composite structures they do not fully break electroconductive paths inside the composite and it is still possible to track external loading which occurs on the surface of the specimen. Due to strong bond between cracks and electrical resistivity, it is possible to track whether these cracks, which appeared on the surface of the composite structure, increase or do not change when measuring the resistivity of composite structures [15].

2. Methodology

In order to evaluate the electrical properties of continuous carbon fiber reinforced composite structures printed by a fused deposition modeling device, three-point bending tests and fatigue tests were performed on specimens which were manufactured according 'ASTM D3039' standard. Firstly, before attempting to perform experiments, it was decided that a total of five continuous carbon fiber reinforced composite specimens will be printed.

The specimens were printed with fused deposition modeling 3D printer called 'MeCreator2' which was modified in order to be able to print composite structures. All specimens were printed according same printing parameters. However, some specimens had different number of layers. One specimen with two layers was printed for fatigue tests and four specimens were printed for three-point bending tests. Two of them were manufactured to have three layers and the other two had four layers. All specimens had the same layer height which was equal to 0.5 mm. PLA thermoplastic was used as a matrix material for the production of composite structures, and continuous carbon fiber was chosen as a reinforcing material. After the specimens were manufactured, they had to be left on the print bed to cool in order to easier detach them from the printing bed. In order to get valuable results of electrical measurements during fatigue and bending tests it was needed to have very good contact between electrodes and composite structures. In this case, all specimens had to be polished at the locations where electrodes were needed, silver paste and cooper plates were used to enhance contact between composite and electrodes. The schematics of experimental part could be seen in fig. 22.

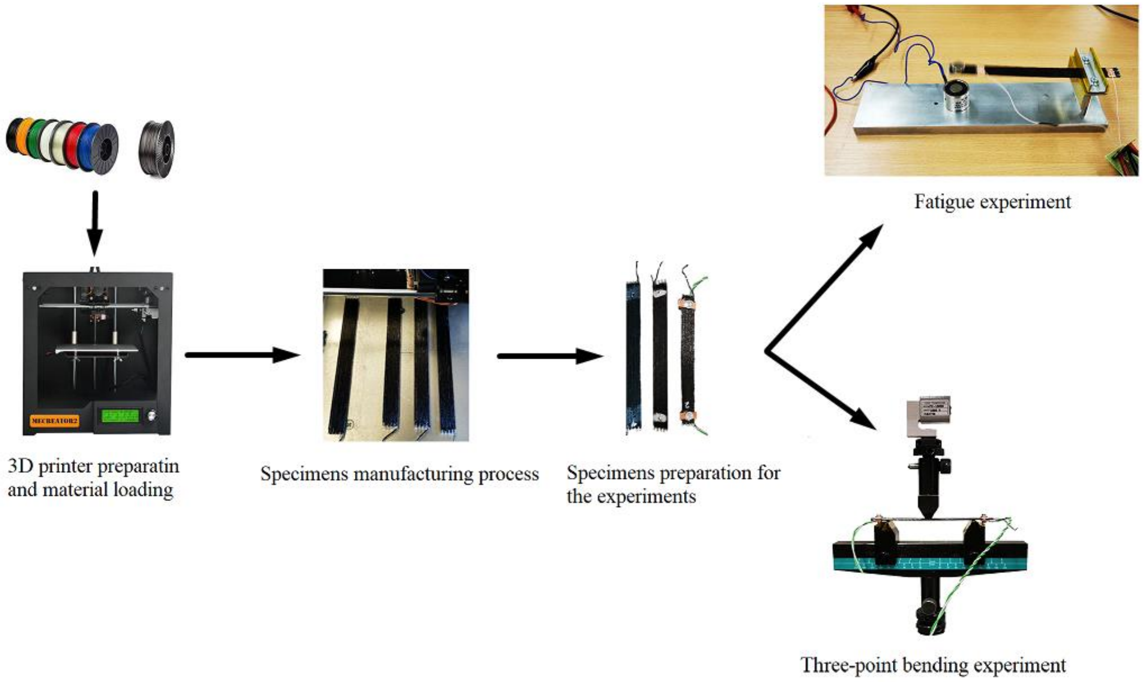

Fig. 22. The schematics of the experimental part

2.1. Materials

Material selection plays a large role when it is needed to get a strong and electrically conductive composite structures. Since fatigue and three-point bending tests will be performed it is crucial to manufacture strong enough composite structures which would withstand incoming loads and at the same time would be electrically conductive in order to get proper electrical measurements. As a reinforcing material I have selected continuous carbon fiber which is well known material that not only enhances mechanical properties but also electrical properties of composite structures. Polylactic acid (PLA) was selected as a matrix material which will hold carbon fiber particles together.

2.1.1. PLA thermoplastic

As a matrix material for composite production, I chose to use polylactic acid (PLA) thermoplastic manufactured by ‘PolyLite™ PLA’. This particular thermoplastic is widely used in 3D print technology for rapid prototyping because it does not emit toxic fumes and is manufactured using renewable resources. The diameter of PLA filament is 1.75 mm (fig. 23).

Fig. 23. Polylactic acid (PLA) thermoplastic

The main purpose of selected thermoplastic is to provide body for carbon fiber in order to prevent carbon fiber from cracking as it is very brittle when exposed to external loading. The main characteristics of PLA thermoplastic could be seen in table 2.

Table 2. Main characteristics of PLA thermoplastic [16]

Solid density, g/cm^3	1.252
Melt density, g/cm^3	1.073
Melting temperature, ℃	165
Heat deflection temperature, ℃	52
Tensile strength, MPa	59
Elongation at break, %	7
Elastic modulus, MPa	1287
Yield strength, MPa	70
Ultimate tensile strength, MPa	73

2.1.2. Continuous carbon fibers

As a reinforcing material in the specimen structures, I chose to use ‘Torayca ®’ manufacturer’s continuous carbon fiber ‘T300B-3000’ which has 3000 carbon fibers in a single tow (fig 24). This carbon fiber is manufactured out of polyacrylonitrile (PAN). This particular material was selected due to its high tensile strength, high electrical conductivity and because it is highly used in the manufacture of low weight structures and similar composite structures.

Fig. 24. ‘T300B-3000’ carbon fiber

Such materials like ‘T300B-3000’ are more and more used due to its light weight and strength reaching aerospace quality aluminum. It has such good qualities like low thermal expansion, which means that this material will not change its dimensions so easily, it is corrosion resistant, has high thermal and electrical conductivity and is lighter than most of the metals. More detailed characterization of chosen continuous carbon fiber could be seen in table 3.

Table 3. Characteristics of ‘T300B-3000’ carbon fiber [17]

Product name	Number of filaments	Tensile strength, MPa	Modulus of elongation, GPa	Elongation, %	Fineness tex, g/1000m	Density, g/cm^3
T300B-3000	3000	3530	230	1.5	198	1.76

2.1.3. Additional materials

During my experiments it is crucial to acquire good electrical measurements in order to evaluate resistivity change in composite structures during external loading. A proper contact between the electrodes and the composite structures helps to better assess the required electrical properties of the product. Silver paste or silver paint are materials that are well-known and used in similar studies that helps to increase contact level between the electrodes and continuous carbon fibers inside composite structures. Such materials provide high electrical conductivity. Thanks to these materials current can easily reach conductive fibers inside composite structures and flow through the structure till it reaches another electrode. In this research silver paint was used to increase contact level between electrodes and composite structure and applied to areas where electrodes are intended to be attached (fig. 25).

Fig. 25. Silver paint [18]

After applying the silver paint, the sample had to be left for a few hours for the paint to dry. Then copper plates were attached on top of the silver paint. Copper plates provides flat surface for electrodes to be attached to and also, they have good electroconductive properties.

2.2. Specimen production

In order to be able to perform the tests, it is necessary to prepare the specimens that will allow to successfully accomplish fatigue and three-point bending tests. It should be mentioned that all specimens were manufactured using fused deposition modeling technique. Firstly, one specimen was manufactured for fatigue experiments in order to investigate if the hypothesis is correct and if it is possible to acquire resistivity change during vibration experiment. When the theory was approved, I proceeded to work on my experiments and printed four specimens for three-point bending experiments. These four specimens were divided to two groups with the essential difference being the number of layers. Two of them had three layers and another two specimens had four layers.

2.2.1. FDM printer ‘MeCreator 2’

A modified fused deposition modeling 3D printer ‘MeCreator 2’ was selected for manufacturing of the specimens. This particular 3D printer is designed by ‘Geeetech’. This 3D printer was designed to print single material at a time so additional changes to print head had to be made in order to print two materials at a time – thermoplastic and continuous carbon fiber. It was selected due to its simplicity to print parts and the possibility to change or adjust some standard elements according needs. ‘MeCreator 2’ provides the opportunity to observe printing operation from all perspectives as it does not have covering sides. Printer could be seen in fig. 26.

Fig. 26. ‘MeCreator 2’ 3D printer [19]

The printer has five main parts:1) printing head, 2) Y axis motor, 3) X axis motor, 4) building platform, 5) Z axis motor. The main 3D printer technical characteristics could be seen in table 4.

Table 4. Main technical characteristics of ‘MeCreator 2’ 3D printer [19]

Build volume	160 x 160 x 160 mm
Printing precision	0.05 mm
Positioning precision	X/Y: 0.05 mm, Z: 0.02 mm
Print speed	60-80 mm/s
Filament diameter	1.75 mm
Nozzle diameter	0.4 mm
Max heated bed temperature	110 ℃
Max extruder temperature	240 ℃
File formats	.STL, 3ds, obj, amf, dae, G-code
Stepper motors	1.8° step with 1/16 micro-stepping
Machine Dimension	320 x 320 x 360 mm
Machine net weight	9.05 kg

2.2.2. Printing process

As previously mentioned, it was decided to manufacture one specimen for fatigue testing and four specimens for three-point bending experiments. The specimen’s which was manufactured for fatigue experiments was designed to be 160 x 15 x 1 mm (length, width and thickness). Meanwhile, specimens manufactured for three-point testing was designed to be 150 x 15 x 2 mm (length, width and thickness). All specimens were designed using ‘SolidWorks 2020’ software and when the design was completed, the design of the designed product was saved as *.stl* format (the format that the printer is capable to process). Stereolithography (STL) file was then uploaded to 3D slicing program called ‘Simplify3D’ and all required print settings have been written. 3D slicing program defines the printing path of the designed specimen, which the program generates when the print parameters are entered.

These print parameters are not only important to generate printing path but also for overall quality of the specimen. The most important parameters are printing speed, infill rate, layer height, extruder temperature, bed temperature, extrusion width and nozzle diameter. The specimen which is ready for printing operation in 3D slicer program could be seen in fig. 27.

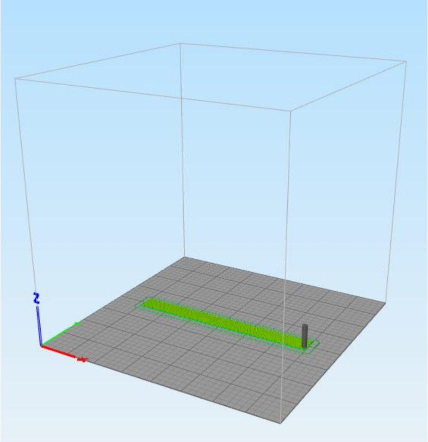

Fig. 27. Specimen prepared for printing operation

As mentioned above, printing parameters are very important in order to manufacture appropriate specimens for the experiments. In this case, parameters were used that were repeatedly discussed and tested in similar studies and it has been experimentally proven that these parameters are suitable for use in the printing of carbon fiber reinforced composites. Some of the most important 3D printing parameters that were used in this study could be seen in table 5 and short description is provided below.

Table 5. Printing parameters

Nozzle diameter	1.5 mm
Extrusion multiplier	0.5
Extrusion width	1.5 mm
Layer height	0.5 mm
Printing speed	3 mm/s
First layer speed	1.2 mm/s
Extruder temperature	210 ℃
Bed temperature	90 ℃
Fan speed	60 %
Internal/external fill pattern	Rectilinear
Infill percentage	100 %

Nozzles with a diameter of up to 0.6 mm are most often used in the thermoplastic printing process, but in this case a much larger nozzle diameter is required because not only PLA is extruded on top of the printing bed but also carbon fiber which is mixed together with PLA. Larger diameter of the nozzle will allow high-quality printing of the composite and the clogging of the nozzle will be avoided. In this case I chose to use 1.5 mm diameter nozzle.

The temperature of the extruder has to be high enough to be able to melt down the thermoplastic, so it should be above 170 °C. However, the temperature of the extruder was set to be much higher than melting point of the thermoplastic due to the fact that at higher temperatures the thermoplastic will melt faster and this will prevent nozzle from clogging. Meanwhile, the temperature of printing bed was set to be higher than usual and equal to 90 °C. The reasons behind this decision is that higher temperature of the bed provides better adhesion between the specimen and printing bed which results in successful specimens production.

Printing speed greatly affects the overall properties of the specimens. In my case, printing speed was set to be 3 mm/s. The selected value is lower than that normally used in thermoplastic printing due to the fact that continuous carbon fiber has to be extruded evenly throughout composite structure. Printing speed of 3 mm/s not only helps to distribute continuous carbon fiber evenly but also prevents the possibility for carbon fiber to break.

Layer height (H) and extrusion width (L) are also very important values for manufacturing of the composite structures. Layer height was set to be 0.5 mm. Layer height is responsible for surface quality and overall esthetics of the specimen. Since in this case esthetics is only optional and is not required the value of layer height was set to be higher than usual which also helps to protect carbon fiber tow from breakage. Meanwhile, extrusion width was set to be 1.5 mm which is higher value than usual. The higher value was chosen due to the fact that two materials are extruded on top of the printing bed and one of them is not even melted. Since two materials are extruded at the same time, they will take up more space when compared with one extruded material at the time so a larger extrusion width is needed in order to prevent printing paths from overlapping with each other. Layer height marked as H and extrusion width marked as L could be seen in fig. 28.

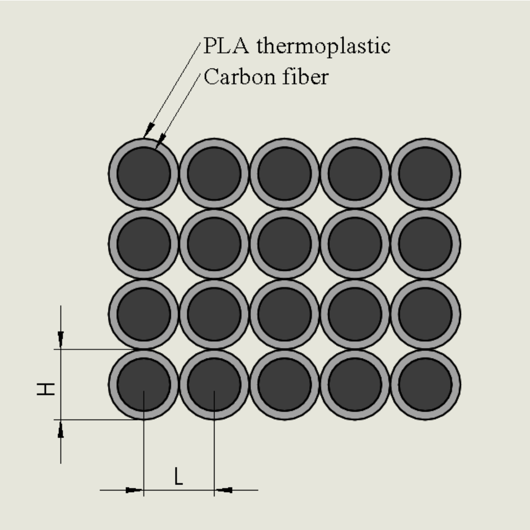

Fig. 28. The cross section of the specimen. H – layer height, L – extrusion width

Once all of the parameters have been described in the 3D slicing program and the G code has been generated, the preparation of the model for printing is completed and the printing operation can already be started. The continuous carbon fiber reinforced composite is produced by inserting fibers into the print head. The thermoplastic and carbon fibers are mixed together inside printing head before extrusion onto the printing bed. In order to manufacture the specimens for experimental part of this study some changes had to be made for printing head of the selected ‘MeCreator 2’ 3D printer. The print head has been redesigned so that it had two input channels (one for thermoplastic and one for carbon fiber) and only one output channel. The schematics of print head and printing process could be seen in fig. 29.

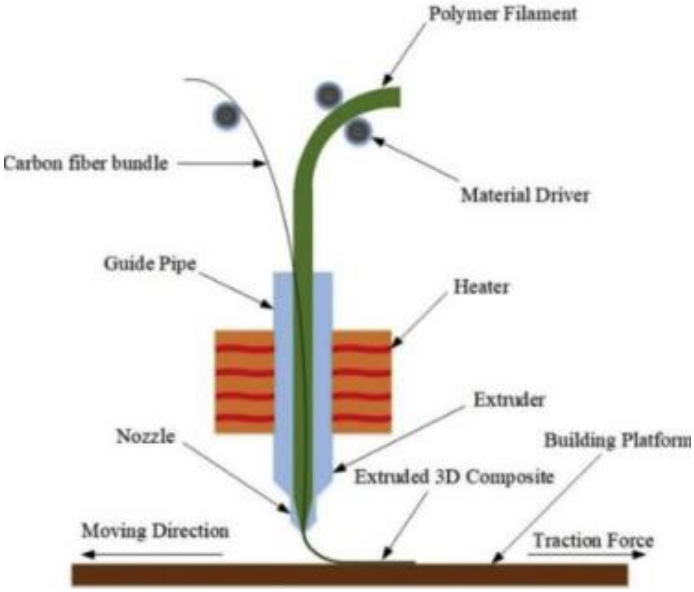

Fig. 29. The schematics of print head and printing process [20]

The 3D printing process is fairly simple. The printer must be properly prepared for printing operation and this include several steps. Firstly, continuous carbon fiber has to be pulled through its input to the guide pipe till it reaches heating element. Important to mention that during this procedure the temperature of the heater must be fairly low in order not to damage continuous carbon fibers. After this procedure, the heater and the printing bed can be heated till they reach operating temperatures of 210 °C and 90 °C , respectively. When all operating temperatures are reached, PLA thermoplastic is pushed through second input channel through guide pipe till it reaches heating element. PLA thermoplastic is melted and mixed with continuous carbon fibers in the extruder. At the meantime, material drivers are pushing solid PLA and continuous carbon fiber inside the extruder and because of excess of material in the extruder, still solid PLA pushes out already melted thermoplastic together with continuous carbon fiber through the nozzle onto the building platform. After the composite material has already been pushed through the nozzle, the rest of the work is done by 3 axis gears (X, Y, Z), controlled by a microcomputer, which moves these three gears according to a predetermined path that has been formed in the 3D slicing program.

It is important to note that when printing specimens, the adhesion between the printing bed and the printed material is often very poor and for this reason the sample does not adhere properly to the printing bed, as a result of which printing operations fail and need to be repeated. In order to prevent failure of printing operation it is necessary to use a special spray, which significantly improves the level of adhesion between the print material and the printing platform. This spray must be sprayed on the printing bed before printing operation. In my case I used '3DLAC' spray which could be seen in fig. 30.

Fig. 30. ‘3DLAC’ spray for better adhesion [21]

Manufactured specimens for three-point bending experiments could be seen in fig. 31. After printing operation of the specimens, they were grouped according number of layers. First group had only one specimen which had only one layer and it was manufactured only for fatigue experiments. Second group had two specimens and each of them had three layers. Third group had also two specimens and each of them had four layers.

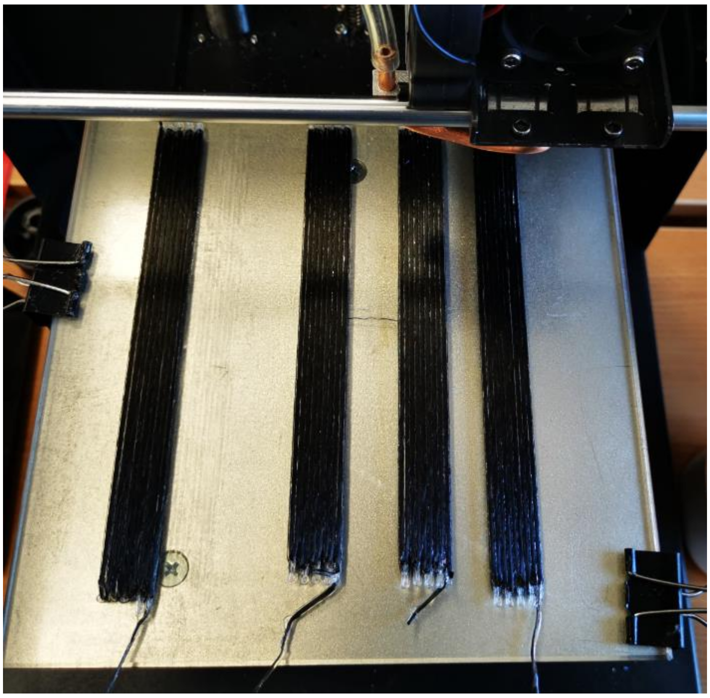

Fig. 31. Manufactured specimens for three-point bending experiments

Theoretically, modified 3D printer ‘MeCreator 2’ is capable to distribute approximately 20 % of continuous carbon fiber during printing operation. Since the amount of continuous carbon fiber plays a large role during electrical resistivity measurements it is important to know the actual amount of continuous carbon fibers which were distributed to the specimen during printing operation. The amount of carbon fibers could be calculated according known ‘fineness tex’ data, which in this case is 198 g/1000m. This numerical value indicates that if a composite would be manufactured while using 1000 m of continuous carbon fibers, the total amount of continuous carbon fiber would be 198

g. Since the number of layers and the number of carbon fiber tows in each layer are known in the composite structures, it is very easy to calculate the actual amount of continuous carbon fiber. The equation to calculate the amount of carbon fiber could be seen below.

$$m_{CF} = \frac{l \cdot n_1 \cdot n_2 \cdot 198}{1000} \tag{4}$$

Here, m_{CF} is the mass of carbon fiber, l is the length of composite structure, n_1 is the number of layers in composite structure and n_2 is the number of carbon fiber tows in a single layer.

The amount of continuous carbon fiber in all specimens are calculated and displayed in table 6. Also, other measurements such as specimen's length, width, thickness, weight were also performed and could be seen in the same table below.

Table 6. The data of measured specimens

Specimen's number	Length, mm	Width, mm	Thickness, mm	Carbon fiber mass, g	Total mass, g	Carbon fiber percentage, %
1 layer						
1	158.8	12.9	0.4	0.28	1.37	20.43
3 layers						
1	152.4	14.4	1.8	0.81	3.96	20.45
2	151.2	14.9	1.9	0.8	3.89	20.56
4 layers						
1	148.5	15.2	2.8	1.05	5.19	20.23
2	149.9	14.9	2.7	1.06	5.14	20.62

As can be seen from table 6, the actual overall dimensions of all the printed specimens are slightly different from how they were designed in 'SolidWorks'. This could be explained by the fact that while printing two materials at the same time there are more process instability, as many variables occur during printing procedure. Such variables could be unstable 3D printer work, insufficiently uniform temperature of printing bed and heater, uneven continuous carbon fiber and PLA thermoplastic feed into the printing head. However, these deviations will have no effect at all during further experiments of electrical resistivity measurements during vibration and three-point bending tests. More importantly, the amount of continuous carbon fiber content remained roughly the same as planned in all cases. This amount ranges from 20.23 % to 20.62 %, however, it will have no effect on further researches.

2.3. Fatigue experiments

The fatigue experiments are performed on a specimen which was manufactured according 'ASTM D3039'. These particular experiments were performed in order to investigate whether it is generally possible to determine the change in electrical resistivity during fatigue testing. For these specific experiments a simple electromagnet was used which caused the specimen to vibrate. In this section, two experiments of a different nature were performed. During first experiment, carbon fiber reinforced composite was exposed to vibrations for approximately 23 hours. During this experiment, an attempt was made to elucidate how resistivity of a composite structure changes when it is exposed to a uniform load for a long period of time. Meanwhile, during second experiment vibrations were cyclically induced to the specimen. During second experiment, an attempt was made to investigate how the electrical resistivity of a carbon fiber reinforced composite changes when it is cyclically exposed to vibrations. A more detailed description of the equipment used and the course of the experiments are given in the sections below.

2.3.1. Equipment used

In order to perform the fatigue experiments properly, during which the change in resistance could be measured, it was necessary to use some electronical equipment. As mentioned above the vibrations of the specimen are caused by a 12 V electromagnet. In order to make it work, it was necessary to use such electronic devices as power amplifier ‘LV102’, function generator ‘Agilent 33220A’ manufactured by ‘Keysight Technologies’, power supply ‘EX355P’ manufactured by ‘AIM-TTI INDUSTRIES’, oscilloscope ‘PicoScope 4000 Series’ and a personal computer to record all measured data. The technical parameters of all the equipment used are given in the tables below.

Table 7. Technical parameters of power supply ‘EX355P’ [22]

Parameter	Value
Number of channels	1
Output voltage	0 – 35 V DC
Output current	0 – 5 A
Output voltage resolution	100 mV
Output current resolution	10 mA
Dimensions	140 x 160 x 295 mm

Table 8. Technical parameters of power amplifier [23]

Parameter	Value
Frequency range	3 – 40000 Hz
Power	50 W
Input resistance	100 kΩ
Power voltage	220 V
Dimensions	334 x 130 x 221 mm

Table 9. Technical parameters of function generator ‘Agilent 33220A’ [24]

Parameter	Value
Waveforms	Sine, square, ramp, triangle, pulse, noise, DC
Number of channels	1
Frequency range	1 μHz to 20 MHz
Amplitude range	10 mV_{pp} to 10 V_{pp}
Accuracy	20 ns
Dimensions	261 x 103 x 303 mm

Important to mention, that several additional equipment was needed to execute these experiments. Self-made fatigue experiment stand had to be made which consisted of several aluminum plates, spacers, fasteners, the previously mentioned electromagnet and simple magnet which was glued on the edge of the composite. The schematics of vibration stand could be seen in fig. 32.

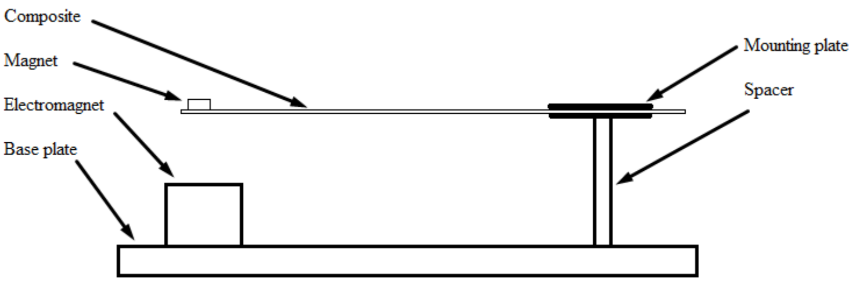

Fig. 32. The schematics of vibration stand

Also, electronic caliper was used to measure the distance between the two attached electrodes.

2.3.2. The procedure of fatigue experiments

First of all, it was necessary to make a stand where the specimen and the electromagnet could be attached. The electromagnet had to be attached directly below one of the specimen edge's, which means that the composite had to be lifted above base the plate with the help of spacers and attached using mounting plates (fig. 33). The distance between the composite structure and the electromagnet had to be large enough that during the experiment, when the electromagnet was switched on, it did not attract the magnet which was attached on top of the composite. It was experimentally determined that when the electromagnet is turned on and vibration of the specimen is induced, a distance of 30 mm between the composite and the electromagnet is sufficient to prevent the magnet on the composite from being attracted. Meanwhile, two electrodes spaced 120 mm apart each other were attached on top of the composite in order to measure the voltage change. Electrical measurements were performed according a two-probe technique.

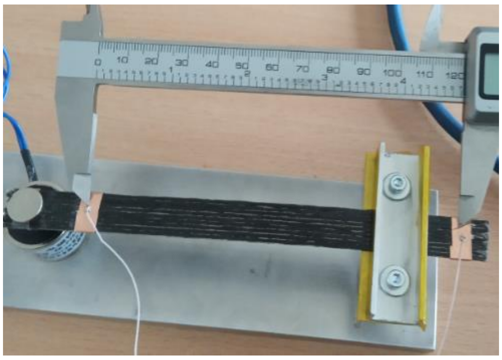

Fig. 33. Vibration's experiment stand

As mentioned in a previous subsection, during experiment power supply, function generator, oscilloscope and power amplifier were used. The schematics of how the devices were connected to each other could be seen in fig. 34.

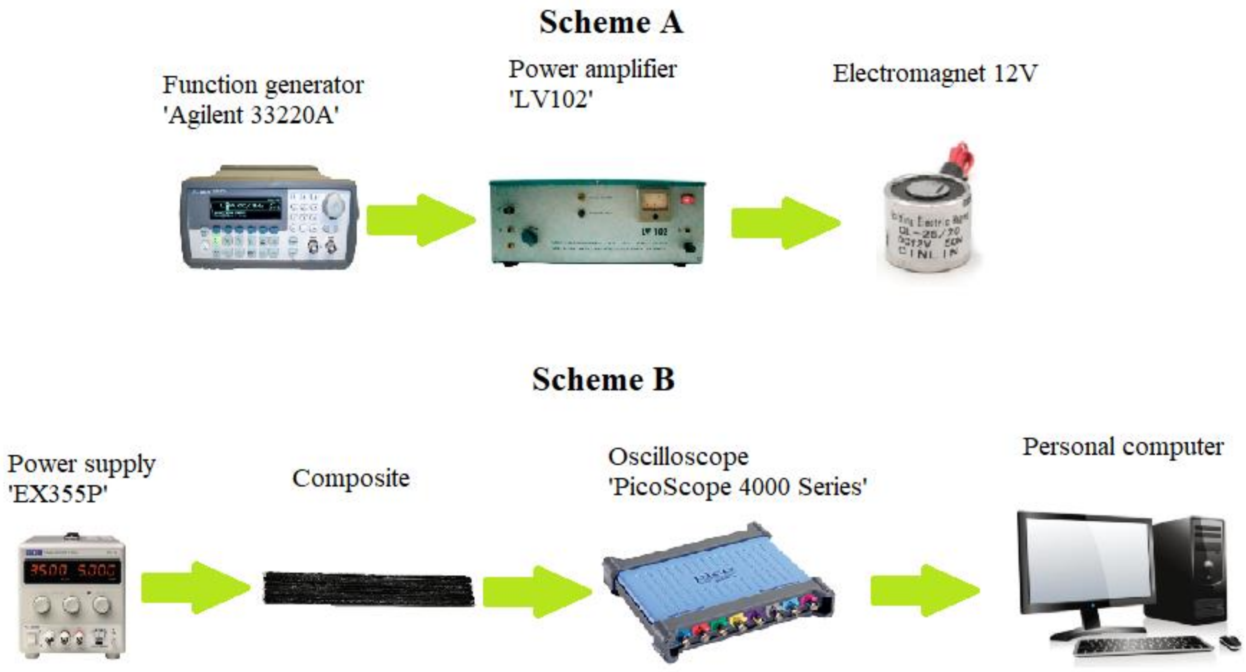

Fig. 34. The schematics of vibration experiment: A – vibration induction, B – electrical measurements;

This experiment essentially consisted of two procedures. The first procedure was to cause the specimen to vibrate and the second procedure – measure voltage changes during experiments and after measurements were taken – calculate resistivity change. The function generator device was set to generate a sinusoidal signal with an amplitude equal to 4.2 V, while the frequency was set at 20 Hz. Then, function generator was connected to power amplifier in order to boost the voltage. Power amplifier was used because during the experiment and it was investigated that the amplitude of the 4.2 V was not sufficient to cause large enough vibrations. It was measured that the power amplifier increased the voltage from 4.2 V to 13.2 V. Despite the fact that manufacturer of the electromagnet states that the maximum working voltage is 12 V, this electromagnet performed really well at 13.2 V and did not fail during the experiments. As mentioned above, the signal was transmitted to the electromagnet at a frequency of 20 hertz. This means that the electromagnet turns on and off 20 times in one second. Meanwhile, when the electromagnet was active, it pulls the magnet attached to the composite, causing the composite to bend toward the electromagnet, and when the voltage was lost, the magnet tries to return to its original position while returning the composite to its former position. However, this magnet was not able to do so because immediately after the electromagnet was turned off, it was reactivated and began to pull the magnet again. This time, the composite was subjected to inertial forces generated during the previous cycle, which made it much easier to tilt the composite towards the electromagnet. In subsequent cycles, the amplitude of oscillation of the composite only increases until it reached its maximum value.

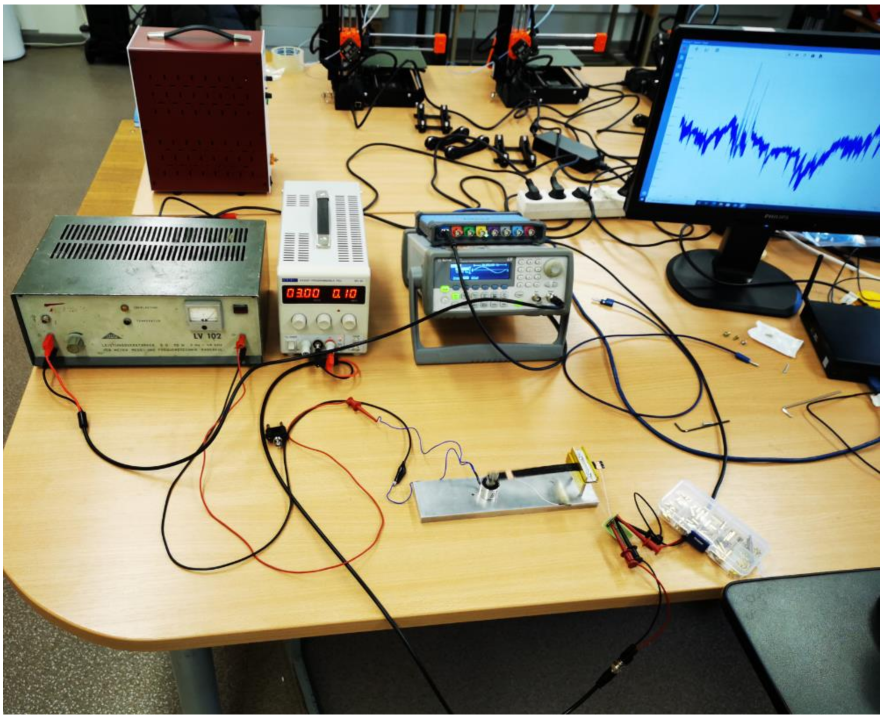

Fig. 35. Fatigue test run

In fig. 35 the progress of the fatigue test and the equipment used could be seen. When vibrations were induced to the specimen, it was necessary to measure the voltage change in order to investigate how resistivity was changing when the vibrations were applied to the specimen. As mentioned above, the voltage change will be measured using two probe method and the distance between the probes were equal to 120 mm. In order to measure voltage change in the composite structure during fatigue testing, the current is needed to be applied to the specimen. In this specific case, ‘EX355P’ power supply was used. The power supply was connected to the composite structure and configured in a way that during the experiment the current which flows through the specimen was equal to 0.1 A and the voltage was 3 V. Data recording is also required to see how much the voltage changes with the help of oscilloscope. The wires of the oscilloscope were connected to the specimen, meanwhile, oscilloscope was connected to the personal computer. In order to record experimental data ‘PicoLog’ software was used and it was configured in a way that the sampling interval was equal to 100 ms and the input range would be ±2 V.

The main purpose was to investigate if the resistivity is changing when the load is applied to the specimen. As mentioned above, this part consists of two experiments. The first experiment was performed to investigate how the electrical resistivity changes when the specimen is exposed to vibrations for a long period of time. During this experiment vibrations were induced in the specimen and maintained for approximately 23 hours. When the voltage change curve was obtained, the resistivity change of carbon fiber reinforced composite was calculated. The second experiment was held in order to investigate how voltage changes when the vibrations are cyclically induced to the specimen. To achieve this goal, it was decided that in order to obtain solid test results, the experiment should take approximately one hour. The voltage was measured throughout the experiment, but the vibrations were induced at intervals. In total, the fatigue test consisted of 14 intervals of 4 minutes each and the duration of the experiment was 56 minutes. During one interval, the specimen was subjected to vibrations lasting 3 minutes, followed by a one-minute pause, at the end of which the next interval began. A pause was made to assess whether the voltage returned to the initial position after the vibrations of the specimen were stopped. The experimental data obtained and their analysis are discussed in the next chapter.

2.4. Three-point bending experiment

After evaluating voltage change in the specimen during fatigue testing, I have decided to perform a three-point bending test in order to evaluate how the resistance changes during bending tests. A more detailed description of the experiment regarding equipment used and overall procedure of the experiment could be found in the following subsections.

2.4.1. Equipment used

Mechanical bending experiments were performed using a universal bending machine 'H25KT' manufactured by 'Tinius Olsen' (fig. 36) whose maximum load force is equal to 25 kN.

Fig. 36. Universal bending machine 'H25KT'

This universal machine is capable to perform not only bending tests but also tensile tests and compression tests. However, only bending tests were performed and for this reason three-point bending stand and 1000 N force sensor was used. The bending machine can be programmed according to the desired principle of operation using the software 'Horizon' which was installed on the personal computer. Technical specification of universal mechanical testing machine could be seen in table 10.

Table 10. Technical specification of 'Tinius Olsen H25KT' [25]

Model	Maximum load capacity, N	Maximum crosshead travel, mm	Accuracy, %	Speed range, mm/min	Distance between columns, mm
H25KT	25000	1100	0.5	0.001 – 500	405

Unlike in the previous experiment, during three-point bending test it was decided to measure the resistivity directly rather than to measure the change in voltage in order to obtain more accurate measurement results. For this reason, source meter '2614B' was used manufactured by 'KEITHLEY' in order to measure resistivity during the experiment. Technical specification of the source meter used could be seen in table 11.

Table 11. Technical specification of '2614B' source meter [26]

Series	2600
Number of channels	2
Source voltage range	±200 mV – ±200 V
Source current range	±100 nA – ±10 A
Output power	60 W
Resistance measurement range	500 nΩ – 100 TΩ

2.4.2. The procedure of three-point bending experiments

As previously mentioned, four specimens were tested and their resistivity measured during three-point bending experiments. These specimens were grouped to two groups according to the number of their layers. Despite the difference in the specimens, they were prepared uniformly for the three-point bending experiments. First of all, holes with a diameter of 3 mm were drilled at both ends of the specimens. The distance between two holes was approximately 130 mm. These holes were needed so that the copper plates to which the electrodes were soldered could be easily attached with a screw and a nut. After drilling the holes in the specimens, it was necessary to sand the area around the holes with a file in order to remove the electrically non-conductive PLA thermoplastic from the composite surface. When the latter procedure was performed the carbon fiber was raised to the surface, thus, achieving a direct contact between the carbon fiber and the copper plates. After sanding operation silver paste was painted on top of the composite surface around the area where holes were drilled. Silver paste significantly improves adhesion between the composite structure and copper plates. The resistivity measurements were taken according to four probe technique. To ensure high quality resistivity measurements it was needed to use four copper plates and four wires in total. These four wires were soldered to two copper plates (two wires each) and then with the help of other two copper plates and tightening elements were attached to the specimen. The specimen's preparation procedures could be seen in fig. 37.

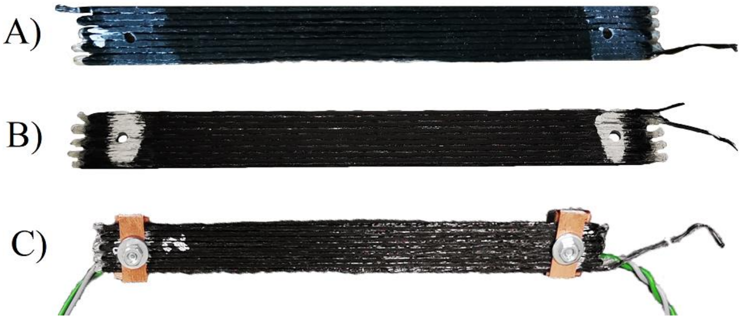

Fig. 37. Specimen's preparation procedure for three-point bending experiment: a) polished specimen with drilled holes; b) specimen with silver paint applied around holes; c) electrodes and copper plates attached to the specimen

After preparing the specimens for the three-point bending experiments, it was decided that all specimens would be bent at the centerline. Meanwhile, it was decided to set the distance between the supports at 110 mm and the electrodes were attached behind those roller supports. Since roller supports have direct contact with the composite structure, it is likely that these metal roller supports will have a significant effect on the resistance measurements. To avoid this, these supports and the bending roller had to be isolated from the composite by gluing a ‘kapton’ tape on them, which is extremely good dielectric. The positioning of the specimen on the three-point bending stand could be seen in fig. 38.

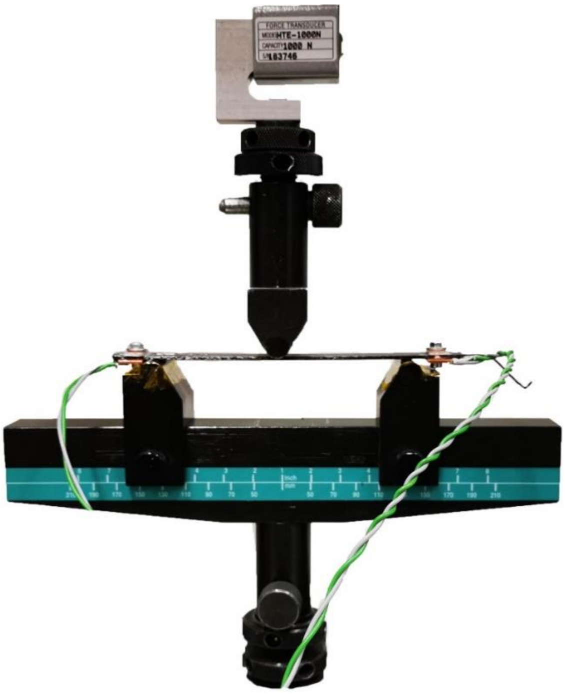

Fig. 38. Specimen positioned in three-point bending machine

The bending machine had to be calibrated before each bending experiment in order to get valuable resistivity change results. A calibration process is quite simple, it was necessary to lower the bending roller to the composite so that it gently rested on the composite surface. In this way the position of the machine in relation to the composite was set to zero, and the bending takes place until a predetermined displacement value was reached or until the specimen breaks. The fact of a specimen break was recorded by a highly sensitive force sensor which detects a sudden change in the resistance force between the sample and the force sensor. Meanwhile, source meter ‘2614B’ records resistivity during the experiments.

The three-point bending experiments consisted of several different tests. First of all, an attempt was made to investigate whether the resistivity changes when the specimen was bended to the same position several times in a row. For this test the specimen which had 3 layers and was marked as a second specimen was chosen. During this experiment, it was decided to measure mean and standard deviation of the resistivity after each bending cycle and after the bending machine returned to its original position. For this reason, source meter which measures the resistivity, had to be programmed so that it would record 30 readings over the period of time and calculate the mean and standard deviation of the resistivity. Meanwhile, the bending machine was programmed to bend at a speed of 10 mm/min until a displacement of 5 mm was reached. Before the start of the test, the initial values of the mean and standard deviation of the resistivity were obtained, and these values were subsequently obtained immediately after the specimen was tilted and when the specimen was returned to its initial state. Overall, 5 cycles of the same displacement were made. Later, the bending machine was programmed to bend at a speed of 10 mm/min until a displacement of 7.5 mm was reached. However, these tests did not show a significant change in resistivity, so this test was not performed with the remaining specimens. The test results are illustrated and discussed in the following chapter. The progress of three-point bending experiment could be seen in fig. 39.

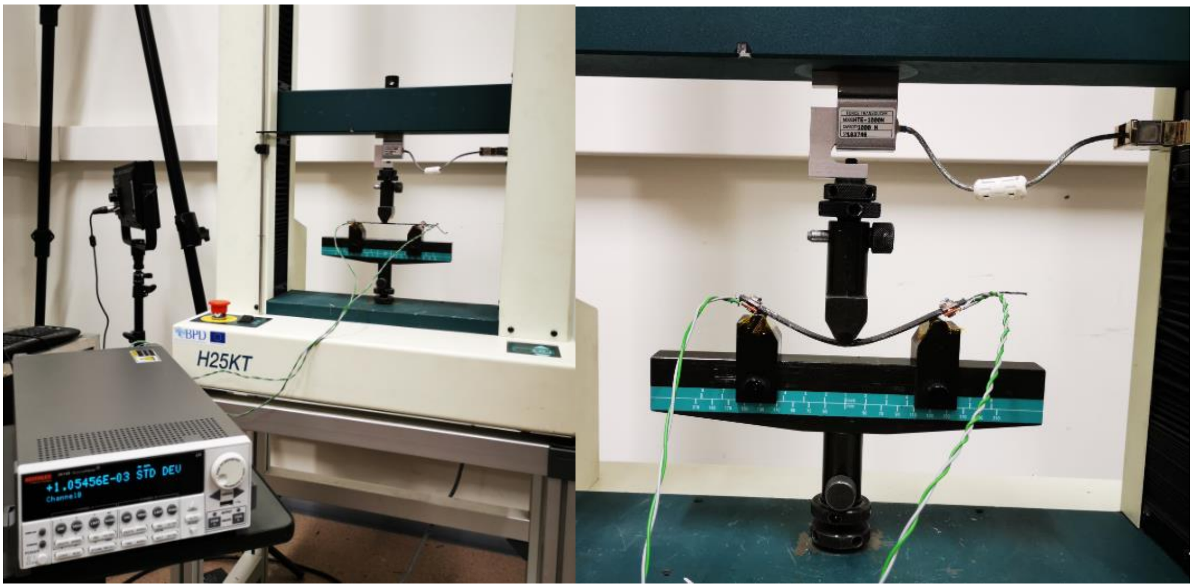

Fig. 39. Three-point bending experiment

The second experiment was performed differently. Source meter was programmed to record 115 readings per minute while the bending machine was programmed to finish bending cycle in 1 minute. This experiment was divided into several bending cycles during which different displacement values were achieved. The specimens were bent until they reached the 5 mm, 7.5 mm, 10 mm, 15 mm, 20 mm and 25 mm displacement mark or until they were broken, except for the specimen used in the previous test, the first bending displacement cycle of which in this experiment was 7,5 mm. During every cycle 115 readings of resistivity measurements were recorded but only first, the last and every 10th reading were taken into consideration in order to avoid data redundancy. The results of this experiment could be found in a following chapter.

3. Experimental results and their analysis

In order to evaluate the electrical properties of carbon fiber reinforced composites several mechanical experiments were needed. Specimens manufactured in accordance of ‘ASTM D3039’ standard were tested during fatigue and three-point bending experiments. Experimental results and their analysis could be found in a section below.

3.1. The results of electrical measurements of fatigue experiments

As mentioned earlier, fatigue experiments consists of two different experiments. During first experiment vibrations were induced in the specimen which lasted for approximately 23 hours and during this experiment voltage change was measured. Meanwhile during the second fatigue experiment vibrations were induced to the specimen in 14 intervals which lasted 4 minutes each. These 4 minutes consisted of 3 minutes during which the sample was subjected to vibrations and a pause of 1 minute. Each interval started with a vibration and ended with a pause and during the experiment the voltage change was measured.

3.1.1. The results of electrical measurements of first experiment

The results of the first fatigue experiment are visible in figure 40. During this particular experiment vibrations were induced to the specimen for approximately 23 hours. The results showed that when vibrations were introduced to the specimen the voltage rose significantly this could be explained by the fact that external load that was applied to the specimen affected the conductive carbon fiber paths, resulting in an increase in voltage.

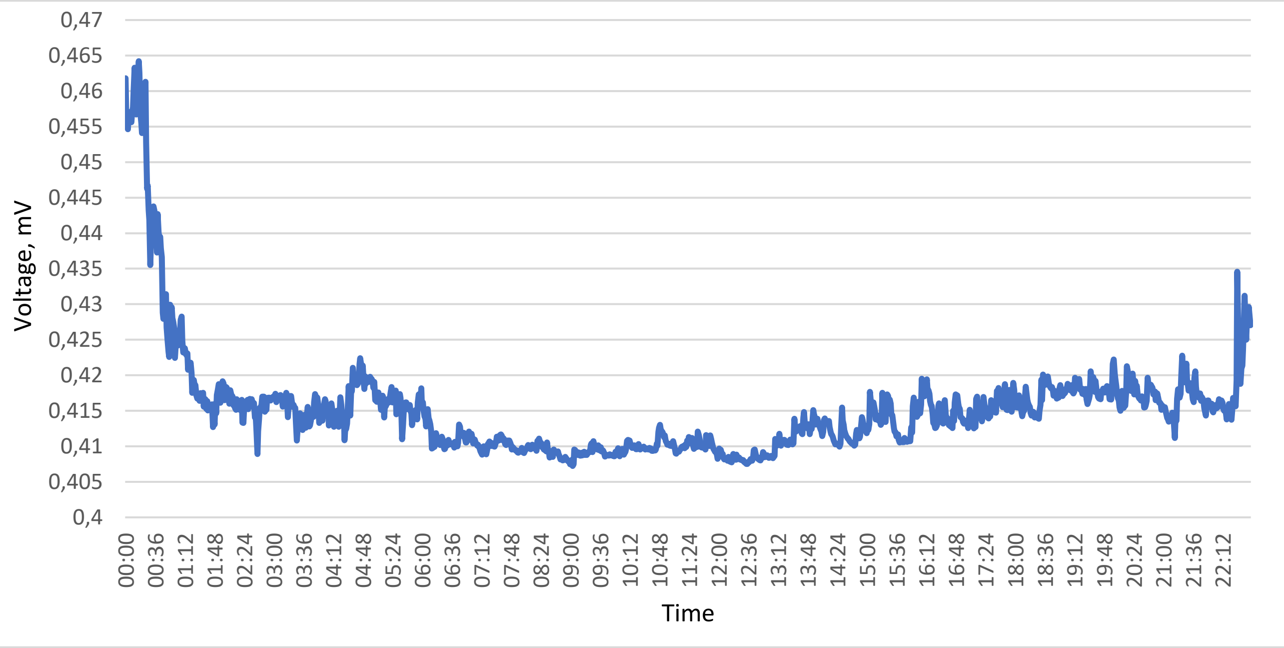

Fig. 40. Voltage change during first fatigue experiment

When the voltage change curve was obtained it was necessary to calculate resistivity change from known values in order to evaluate how resistivity was changing throughout the experiment. Since, the voltage and current are known it is possible to calculate the resistivity of the composite structure using equation stated below [27]:

$$R = \frac{U}{I} \tag{5}$$

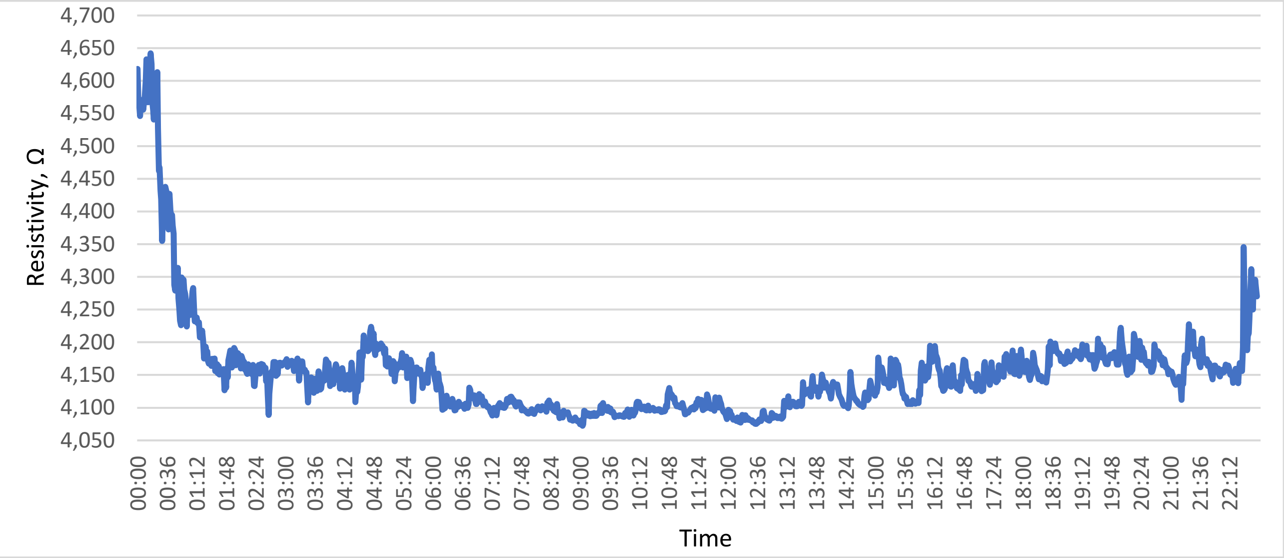

Fig. 41. Resistivity change during first fatigue experiment

Fig. 41 demonstrates the resistivity change curve. This curve is very similar to voltage change curve and it is because resistivity directly depends on the voltage change. As can be seen from the graph the resistivity of the composite structure at the beginning of the experiment is approximately 4.6 Ω. However, such increased resistivity persists for a short time and after about half an hour into the experiment, it begins to fall down. Therefore, the results of this experiment can be divided into 3 stages. The first stage could be called the resistivity 'settling down' stage. During this stage, the composite's resistivity drops to about 4.15 Ω and stabilizes within one hour and 40 minutes from the start of the experiment. This stage can be explained by the fact that the carbon fibers that are in composite structure got stretched out and adapted to the new conditions so as to minimize the load resistance. Therefore, after a while, the current began to more easily find electrically conductive paths inside composite structure, which led to a voltage and, consequently, resistivity drop. The second stage lasted much longer – about 12 hours which ends approximately 13 hours and 30 minutes after the start of the experiment. During the second stage, a slight but consistent decrease in resistance is observed. However, no significant change in resistivity could be noticed. 13 hours and 30 minutes after the start of the experiment it can be noticed that the resistivity of the composite gradually starts to increase. From this point the third stage begins. A noticeable increase in the resistance of the carbon fiber reinforced composite may be related to the fatigue of the internal particles of the composite structure. This may mean that the composite structure began to feel slight signs of fatigue, which led to an increase in structural resistivity. However, this fatigue can be considered to be negligibly small, as no significant change in electrical resistivity was observed during the continuation of the experiment, and no signs of fatigue could be seen on the surface of the composite structure at the end of the experiment. Nevertheless, it can be said that if the experiment is extended for more than 23 hours, the electrical resistivity would continue to increase and perhaps after some time it would be possible to detect obvious fatigue signs on the surface of composite structure.

3.1.2. The results of electrical measurements of second experiment

Fig. 42 demonstrates the results of second fatigue experiment. From obtained data, it can be concluded that the activation of the electromagnet slightly changes the voltage. At the beginning of each interval, when the electromagnet is activated, the voltage rises, but it is worth noting that the voltage is not constant throughout the period of 3 minutes and it fluctuates. When the electromagnet stops working and the vibrations are cancelled the voltage drops by approximately 0.03 V at all intervals. During each interval when the vibrations are induced to the specimen the voltage fluctuates regularly, but on average remains about 0.04 V higher than during the pause period. From the data obtained, it can be stated that each experimental interval is different, and the voltage never returns to the same position at the end of the pause as before. However, several features specific to each interval are noticeable such as at the very start of each interval voltage always rises and when the vibrations are cancelled voltage always falls down.

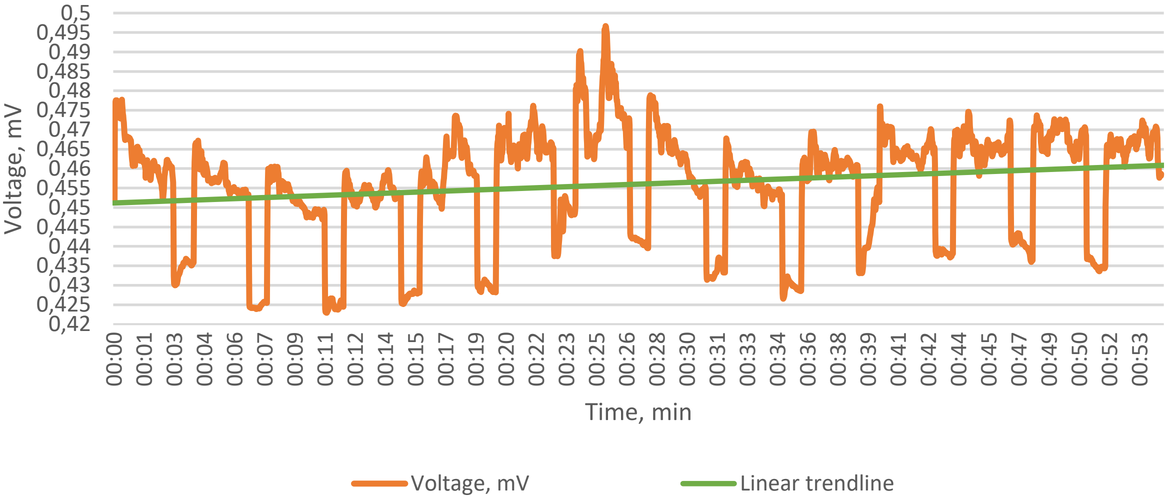

Fig. 42. The voltage results obtained during fatigue experiment

After the evaluation of the voltage changes during the experiment, it was decided to calculate how the resistivity changes. The results which were obtained are presented in fig. 43. The resistivity was calculated using the voltage values obtained during the experiment and knowing the value of the current which is supplied by the power supply.

As can be seen from fig. 43 the resistivity fluctuates throughout the experiment, regardless of whether the vibrations were induced to the specimen or when the specimen was in a pause mode. When the vibrations were induced in the specimen at the beginning of each interval the resistivity raised significantly by approximately 0.25 Ω and always decreased when the vibrations were cancelled. The highest resistivity was obtained during 24-27 minutes interval reaching its peak at 4.95 Ω. Such significant change in resistivity could be due to contact instability, noise or even human error. Meanwhile, the lowest resistivity was obtained during the 8-11 minutes interval when it decreased to all time low value at 4.25 Ω.

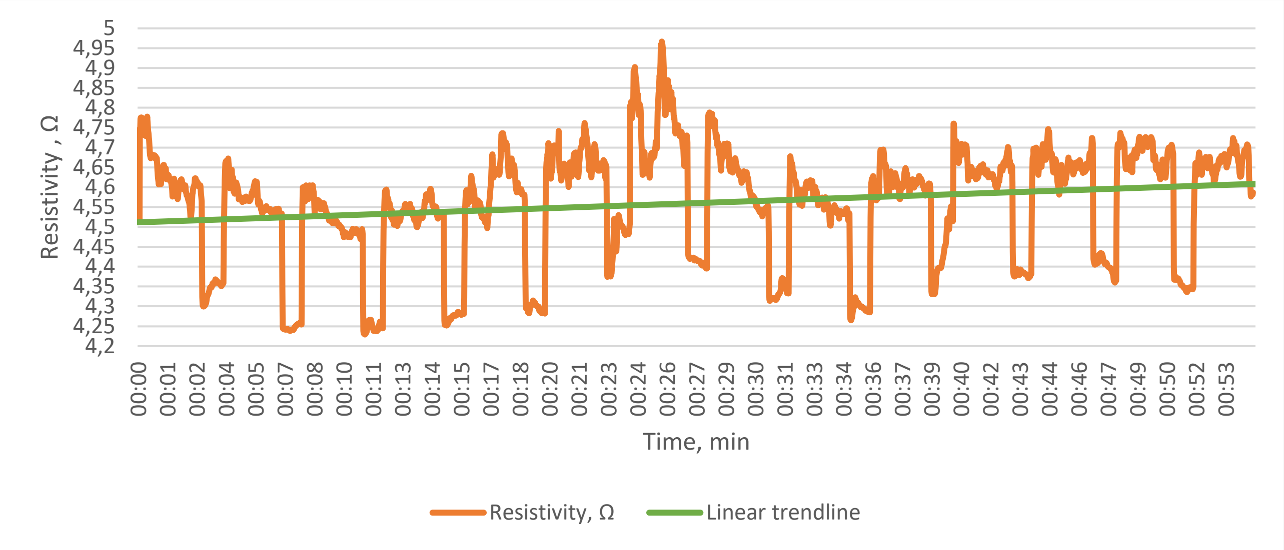

Fig. 43. Resistivity change during fatigue experiment

Since the results for each interval are unpredictably different, it is very difficult to evaluate change in resistivity throughout the experiment so the specimens have to be tested during three-point bending tests to better analyze the load dependence of the resistivity change. However, looking at the trend line it could be seen that the resistivity tends to increase. The resistivity has risen from 4.5 Ω to 4.6 Ω since the start of the experiment. Despite the instability of the results obtained, the fatigue experiments proved that it is possible to detect load emergence in 3D printed carbon fiber reinforced composites by measuring specimen's voltage.

To conclude both fatigue experiments, it can be stated that it is possible to detect changes in resistivity in a carbon fiber reinforced composite which is under external loading. The first experiment, which lasted 23 hours proved that after the internal carbon fibers adapt to the load acting on them and the electrical resistivity has stabilized, it may be possible to detect the beginning of structural fatigue. Meanwhile, second experiment proved that the resistivity of the composite structure during vibration period differs from the resistivity measured during calm state of the composite structure. This experiment proved that it is possible to detect external load appearance on composite structure by measuring composite's resistivity.

3.2. The results of electrical measurements of three-point bending experiments

3.2.1. The results of electrical measurements of first experiment

As previously mentioned, there were several three-point bending experiments. First experiment was performed to investigate how the resistivity of 3D printed carbon fiber reinforced composite changes when the specimen is repeatedly bent to the same displacement value. For this experiment, second specimen which had 3 layers was chosen and it was bent 10 times in total. Five times the specimen was bent at a speed of 10 mm/min till it reached 5 mm displacement value. Then the specimen was bent another five times at a speed of 10 mm/min till it reached 7.5 mm displacement value. The resistivity was measured when the specimen was bent and when returned to its natural position. Important to mention that the resistivity of the specimen before bending experiment was equal to 1.715 Ω. The results of first three-point bending experiment could be seen in table 12.

Table 12. The results of the first bending experiment

Bending No.	Maximum force reached, N	Mean resistivity value after bend procedure, Ω	Average, Ω	Standard deviation	Mean resistivity value when returned starting position, Ω	Average, Ω	Standard deviation
Displacement value: 5 mm							
1	18.48	1.721	1.724	$1.707\cdot 10^{-3}$	1.73	1.727	$1.516\cdot 10^{-3}$
2	18.68	1.728		$1.469\cdot 10^{-3}$	1.727		$1.728\cdot 10^{-3}$
3	18.43	1.724		$1.825\cdot 10^{-3}$	1.724		$1.674\cdot 10^{-3}$
4	18.4	1.725		$1.541\cdot 10^{-3}$	1.728		$1.684\cdot 10^{-3}$
5	18.3	1.723		$1.753\cdot 10^{-3}$	1.726		$1.695\cdot 10^{-3}$
Displacement value: 7.5 mm							
1	26.5	1.729	1.728	$1.557\cdot 10^{-3}$	1.728	1.729	$1.702\cdot 10^{-3}$
2	26.03	1.726		$1.262\cdot 10^{-3}$	1.731		$1.454\cdot 10^{-3}$
3	25.67	1.728		$1.997\cdot 10^{-3}$	1.731		$1.521\cdot 10^{-3}$
4	25.36	1.729		$1.255\cdot 10^{-3}$	1.729		$1.279\cdot 10^{-3}$
5	25.53	1.726		$1.528\cdot 10^{-3}$	1.728		$1.477\cdot 10^{-3}$

As can be seen from table 12 when the specimen was bent 5 times in a row till displacement value of 5 mm was reached, its resistance force to the bending roller fluctuated from 18.3 N to 18.68 N. Meanwhile, the mean resistivity value of the bent specimen fluctuated between 1.721 Ω and 1.728 Ω. While doing first test the highest mean resistivity value was measured during the second bending operation and it was equal to 1.728 Ω and the lowest value was obtained during first bending operation and it was equal to 1.721 Ω. When the bending roller returned to initial position the mean resistivity values were obtained as well. The mean resistivity values which were obtained after bending roller returned to its initial position in mostly all cases were slightly higher than the values which were obtained immediately after bending roller reached predetermined displacement value of 5 mm. These resistivity values fluctuated between 1.724 Ω and 1.73 Ω and on average were 0.003 Ω higher than those values which were obtained after bending operation. Second test results during which the specimen was bent 5 times until displacement value of 7.5 mm was reached revealed an increase in mean resistivity value when compared to results obtained during first test results. This time the resistance force was obtained fluctuating between 25.36 N and 26.5 N and the mean resistivity of bent specimen fluctuated between 1.726 Ω and 1.729 Ω and on average were 0.004 Ω higher than during the first test. During second test mean resistivity was also measured after bending roller returned to its initial position. As in the previous test, the mean resistivity was slightly higher than the values obtained immediately after bending operation and on average were higher by 0.001 Ω. Important to mention that in order to justify the validity of values obtained, standard deviation were also measured. During this experiment standard deviation was not higher than $1.997\cdot 10^{-3}$ which indicates that measured experimental values were very close to mean resistivity values.

This experiment showed that repeatedly performing the same bending operation under the same conditions as previously determined, different mean resistivity values are measured. This could be due to contact instability between copper electrodes and specimen, the inaccuracy of the measurement devices or bending machine inaccuracy. From the data obtained during this experiment, it would be very difficult to evaluate and specifically determine the electroconductive properties of carbon fiber reinforced composites, so a more detailed study is needed. However, this first experiment allows to make a hypothesis that perhaps the resistivity of the specimen fluctuates within certain limits, and while increasing the displacement value during three-point bending experiment the resistivity of the specimen tends to slightly increase. And when the bending roller is returned to its original position, the resistance often does not stabilize, but remains increased or even in most cases slightly higher than during the bending procedure.

3.2.2. The results of electrical measurements of second experiment

Fig. 44 demonstrates the bending results of the first, four-layer specimen and how the bending force changes throughout the experiment when the displacement value increases.

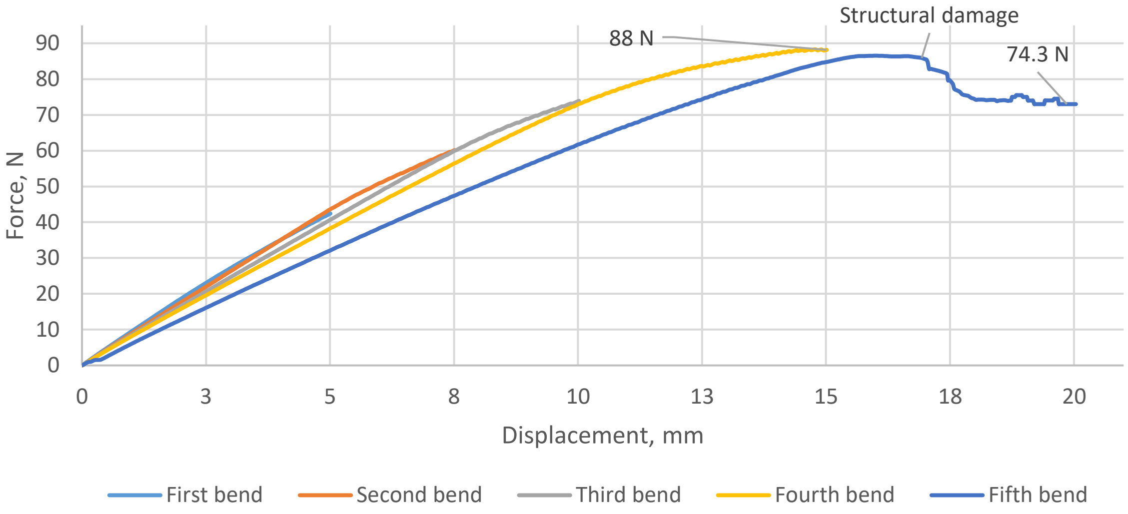

Fig. 44. The dependency of the bending force on displacement of the first specimen with four layers

During first bend test the specimen was bent till bending roller reached 5 mm displacement value, second bend was made until 7.5 mm displacement value, third – 10 mm displacement value, fourth – 15 mm displacement value and fifth – 20 mm displacement value. The bending force was created when the specimen tried to resist the force acting on it, which in this case was bending roller and it was registered with the help of force sensor which was connected to the bending roller. As can be seen from figure 44 the maximum bending force was reached during fourth bending procedure, when the specimen was bent until 15 mm displacement value and the bending force value was equal to 88 N. However, during fifth bending operation the maximum resistance force was equal to 86 N when 17.8 mm displacement value was reached. After that moment a sudden decrease in resistance force was observed which indicates the breakage of the matrix material of the specimen. Important to mention that the specimen did not fully break due to the fact that carbon fiber tows kept holding composite in one peace and created a resistance force to the bending roller.

The resistivity measurement results of all bending operations of the first four-layered specimens could be seen in figure 45. Before the first 5 mm displacement bend, the resistivity of the specimen was measured to be 1.612 Ω. Meanwhile, when the specimen was bent to 5 mm displacement value, the resistivity of the specimen increased to 1.615 Ω. However, when the bending roller returned to initial position the resistivity of the specimen did not return to initial position and remained the same as when the specimen was bent up to 5 mm displacement value. A steady increase in specimen's resistivity was also observed during the second bend operation and when the predetermined displacement value was reached it was equal to 1.617 Ω. However, unlike the previous bending operation, when the bending roller was returned to its original position, the resistivity also returned to previously obtained value (1.615 Ω). During third bending operation the resistivity of the composite structure kept steadily increasing and at a 10 mm displacement value the resistivity was equal to 1.62 Ω. More pronounced changes in resistivity of the specimen began when the specimen was bent to the 15 mm displacement value, when 1.63 Ω resistivity was measured and in general the resistance increased by 0.01 Ω. The biggest changes in the resistance of the specimen were obtained during the fifth bending operation, when the specimen was bent to 20 mm displacement value. From the very beginning of the bending procedure, a strongly increased resistivity of the specimen was obtained and equal to 1.641 Ω. However, the resistance kept fluctuating until 15 mm displacement value was reached, when the resistance of the composite structure significantly increased to 1.695 Ω when 16.5 mm displacement value was reached and at that point the maximum bending force of 86 N was measured. From that point the bending force kept decreasing while the resistivity of the specimen kept increasing and at the end of the experiment the bending force was equal to 74.3 N and the resistivity of the composite structure was equal to 1.705 Ω. Overall, the resistivity of the specimen increased by nearly 0.1 Ω. Important to mention that the structural damage of the specimen could be registered not only from the fall of the bending force but also from the sudden and sharp resistivity increasement.

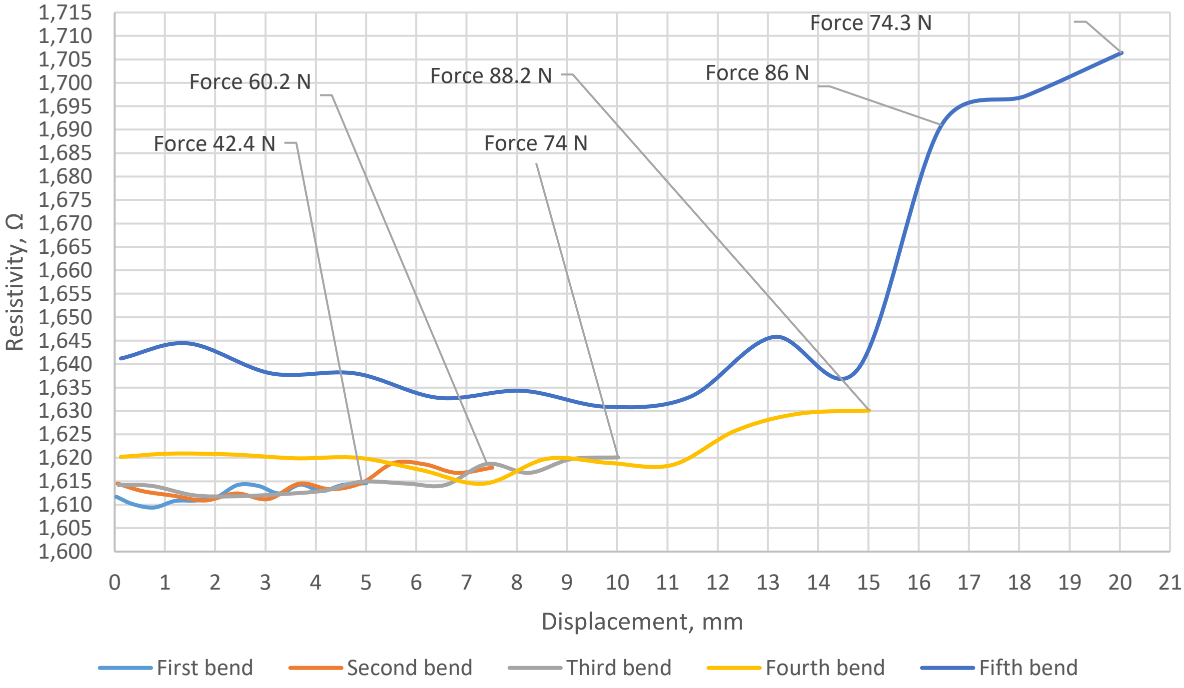

Fig. 45. The experimental results of the first specimen with four layers

Fig. 46 demonstrates the dependence of the bending forces of the second 4-layer specimen on the displacement. As for previous specimen, five bending procedures were performed, with the same parameters. During this experimental part, same as in previous, the specimen broke down during fifth bending procedure, when the displacement value was set to be the highest at 20 mm and the speed was set at 20 mm/min. However, unlike in the previous test, the bending force values were obtained much higher. At the end of fourth bending procedure when the displacement value of 15 mm was reached, the bending force was measured to be 87 N which is only lower by 1 N than in previous test. However, during fifth bending procedure the bending force increased significantly and reached its peak at 120.5 N at 19.19 mm displacement value, which is almost by 34 N higher than during previous test. Important to mention that first breakage sign could be seen when 15 mm displacement value was reached. At that moment the resistance force dropped by approximately 5 N and some of the matrix layers may have been broken, however, carbon fiber tows continued to resist the bending roller and because of that the bending force kept increasing. Right after the maximum bending force was reached, the second breakage could be indicated and during remaining time of the experiment the bending force fell to 115.3 N at the end of the experiment.

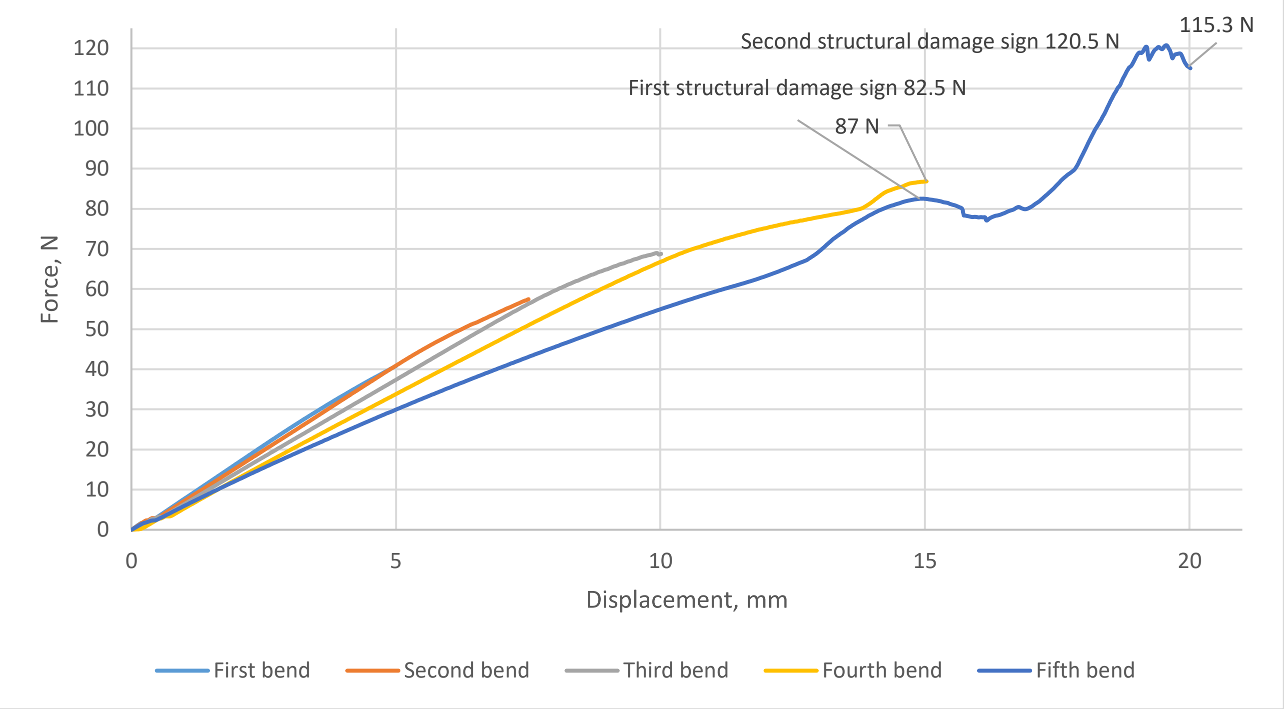

Fig. 46. Second specimen's with four layers bending force dependency on displacement

The resistivity measurements during specimen's three-point bending test could be seen in fig. 47. Before starting the experiment specimen's electrical resistivity was measured to be 1.456 Ω. During first bending procedure the resistivity of the carbon fiber reinforced composite fluctuated but when the bending roller reached predetermined value of 5 mm the resistivity of the specimen increased by 0.001 Ω and was equal to 1.457 Ω. Before second bending procedure the resistivity was approximately the same as before starting the first bending procedure, however, this time the resistivity of the composite structure increased to 1.458 Ω. More significant results were obtained during fourth bending procedure when resistivity of the composite structure increased by approximately 0.08 Ω if compared with the resistance measured at the beginning of the fourth bending procedure. The biggest changes in composite's resistivity were obtained during fifth bending procedure. During this bending procedure the resistivity of the composite remained approximately the same (fluctuated between 1.465 Ω and 1.469 Ω) till displacement value of 12 mm was reached. However, dramatic changes in resistivity measurements began when the displacement value of the bending roller exceeded 12 mm until it reached a displacement value of 15 mm. During this period the resistivity of carbon fiber reinforced structure increased by approximately 0.013 Ω. From 15 mm displacement value to 16 mm displacement value, it was not possible to see a significant change in resistivity, although the resistance increased, but slightly. However, as the bending roller continued to move to predetermined displacement value more dramatic increase in resistivity measurements were obtained. From 16 mm displacement value until the end of the experiment when the displacement value of 20 mm was reached the resistivity of the composite structure rose by 0.032 Ω and reached all time high value of 1.514 Ω. Overall, during these composite's bending sessions the resistivity of the composite increased by nearly 0.061 Ω.

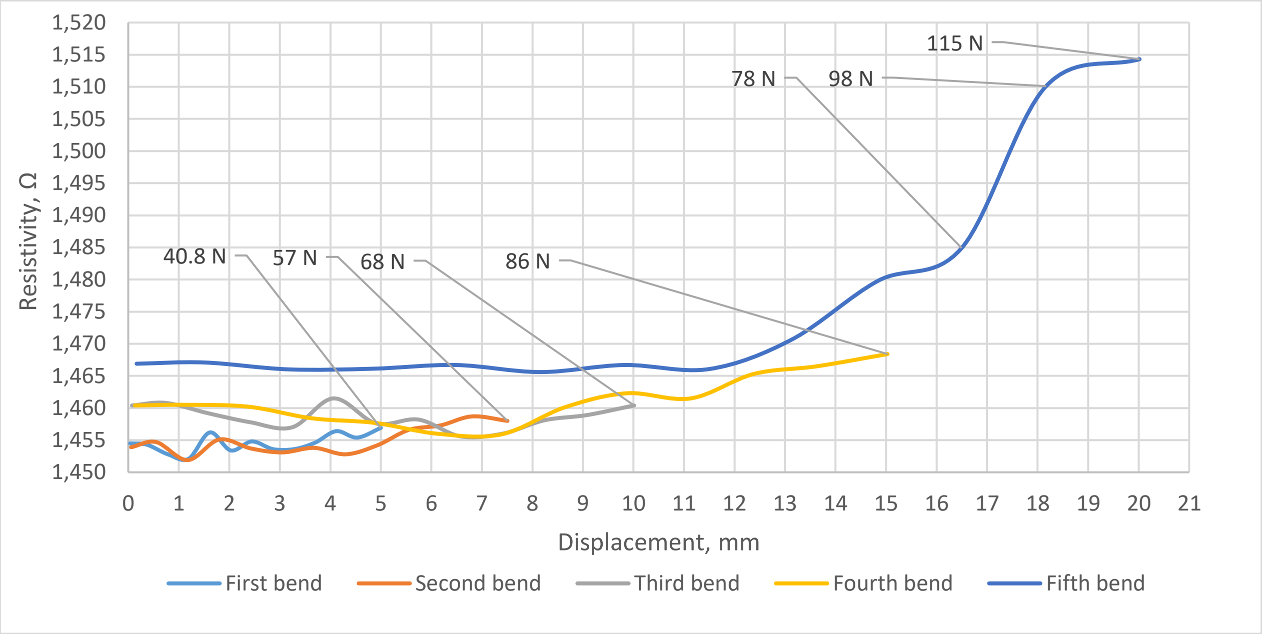

Fig. 47. Resistivity measurements of second four-layered composite

Fig. 48 demonstrates the bending force dependency on displacement value of first specimen with three layers. As can be seen from the figure 48 up until fourth bending procedure experimental results were normal, meaning, the bending force increased when displacement value of the bending roller increased. However, changes appeared during fourth bending procedure when the bending roller reached 18.5 mm displacement value and the bending force was equal to 47 N. At that point the bending force fell by 2.5 N to 44.5 N. This fall is an indication that carbon fiber reinforced composite received structural damage. The second proof that the specimen suffered a structural damage could be seen during fifth bending procedure. At the start of the fifth bending procedure the bending force did not increase until 1.5 mm displacement value was reached. This is due to the fact that the specimen was arched and the bending roller did not reach the specimen up until 1.5 mm displacement was reached. However, the specimen did not break to pieces and it continued to resist the bending roller. Slightly bigger structural damage could be indicated when 24.2 mm displacement value was reached. The bending force fell from 59.3 N to 53.8 N.

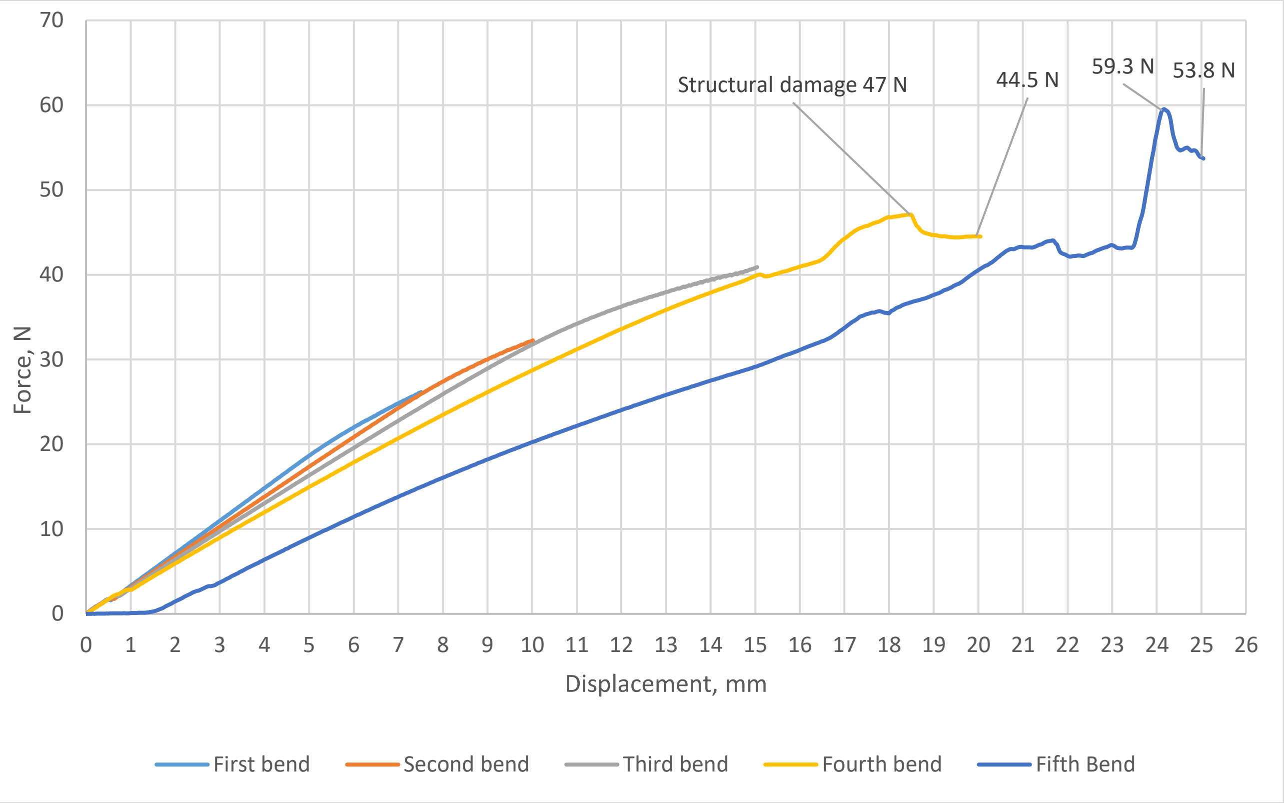

Fig. 48. First specimen's with three layers bending force dependency on displacement

Fig. 49 indicates resistivity measurements of first specimen's with three layers during three-point bending experiment. Before the experiment, the resistivity of the specimen was measured and it was equal to 1.776 Ω. Throughout first bending procedure the resistivity of the specimen fluctuated but at the end of the first bend it remained elevated and equal to 1.78 Ω. More significant changes were visible during fourth bending procedure when the specimen was bent until 20 mm displacement value was reached. At the beginning of the fourth bending procedure specimen's resistivity was approximately 1.789 Ω and it increased to 1.83 Ω at the end of the procedure. The biggest resistivity changes were obtained during fifth bending procedure when the specimen was bent until 25 mm displacement value was reached. Significant changes in resistivity began after bending roller passed 20 mm displacement value. From that moment the resistivity of the composite structure rose significantly by 0.048 Ω and reached all-time peak at 1.853 Ω at the end of the experiment. Overall, the resistivity of this specific composite structure increased from 1.776 Ω to 1.853 Ω.

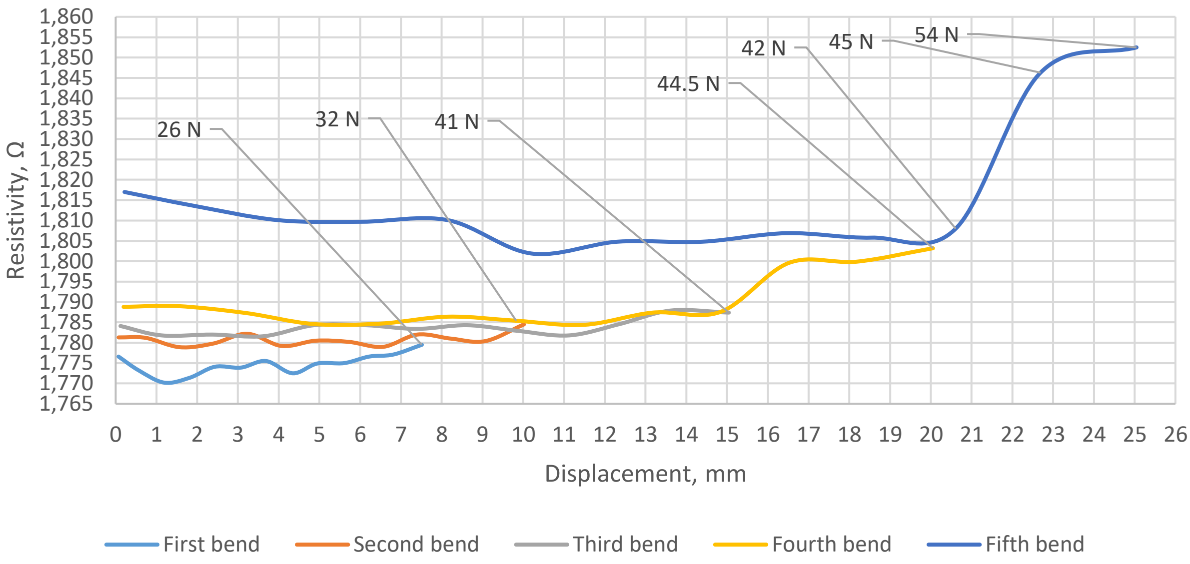

Fig. 49. Resistivity measurements of first three-layered composite structure

Fig. 50 demonstrates second three-layered specimen's bending force dependency on displacement. As in a previous same group specimen's testing during first three bending operations the bending force increased linearly when the displacement value increased. However, during fourth bending procedure structural damage appearance to the composite could be observed when 19,8 mm displacement value was reached and bending force was equal to 45 N. At that point the resistance fell by approximately 1.5 N. The fact that the composite is damaged could be seen during fifth bending procedure. Same as before, carbon fiber tows continued to create a resistive force to the bending roller which is why the bending force increased to 47.5 N at the end of the experiment.

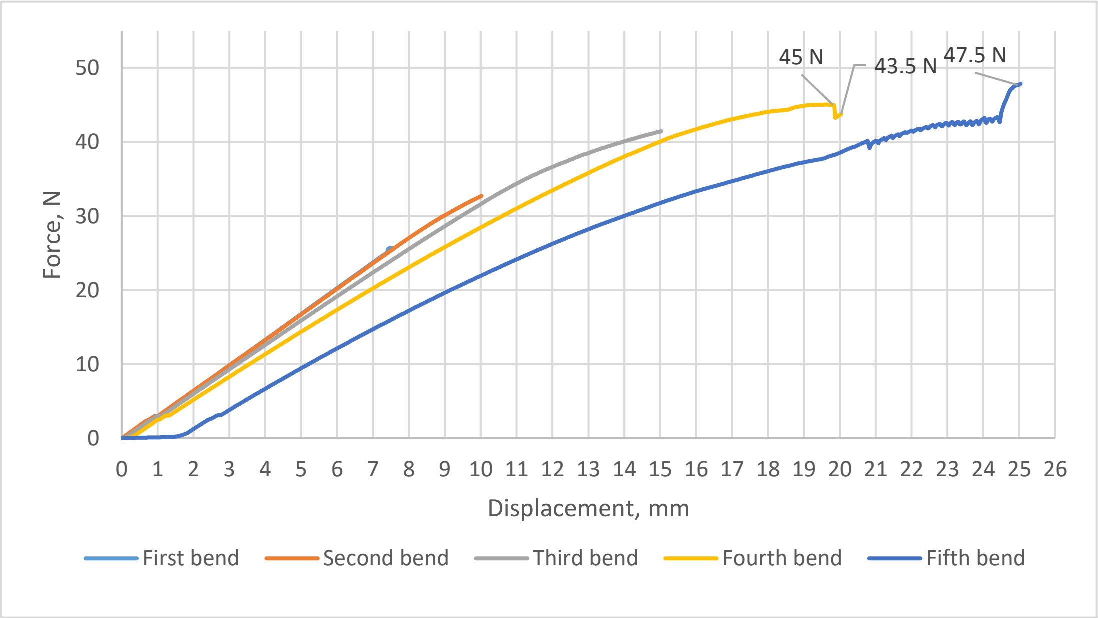

Fig. 50. Second specimen's with three layers resistance force dependency on displacement

Fig. 51 indicates resistivity measurements of second three-layered composite structure during three-point bending testing. Before bending specimen's resistivity was measured and it was approximately

1.715 Ω and at the end of first bending procedure when the specimen was bent until 7.5 mm displacement value, the resistivity of the composite structure increased to 1.717 Ω. As in all previous experiments more significant changes began during fourth bending operation. During fourth bending procedure the resistivity of the composite structure increased by 0.013 Ω and was equal to 1.749 Ω at the end of the fourth bending procedure. The biggest resistivity changes were obtained during fifth bending procedure. The resistivity of the specimen increased to 1.795 Ω at the end of the experiment. Overall, throughout the experiment the resistivity of carbon fiber reinforced composite increased by approximately 0.08 Ω.

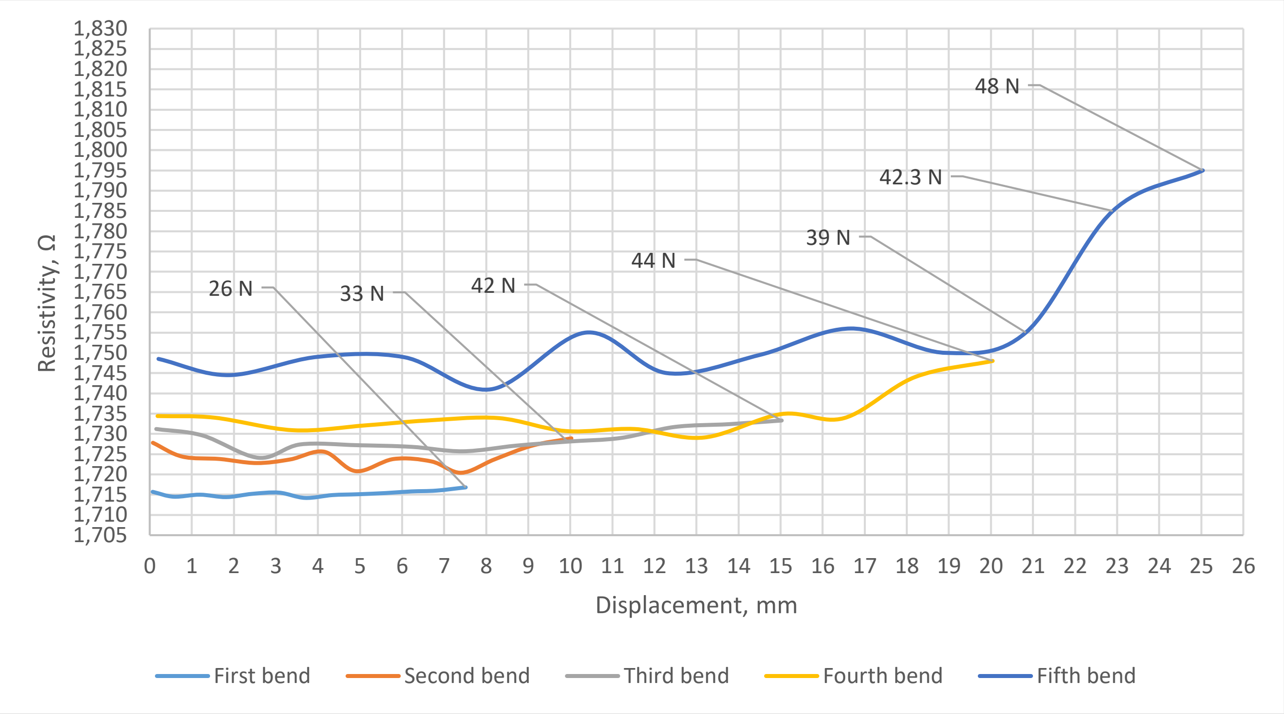

Fig. 51. Resistivity measurements of second three-layered composite structure

To conclude obtained electrical resistance measurement results when the specimens were exposed to external forces, it can be noted that positive experimental data were obtained and the experiments made could be marked as successful. During all experiments it were noticed that when the specimens were exposed to any kind of external forces the resistivity of the carbon fiber reinforced composites always changes. When performing fatigue experiments, a significant increase in the electrical resistance of the composite structure was observed when vibrations were induced and a significant decrease in electrical resistance was observed when the vibrations were cancelled. When analyzing the results of the fatigue experiment a tendentious increase in resistance was observed at each interval of the fatigue experiment which allowed to make a hypothesis that when the composite is exposed to external forces the overall resistivity of the structure increases and remains elevated despite the fact that the force that acted on the composite was canceled. The hypothesis was proofed during three-point bending experiments. During three-point bending experiment four specimens were tested and their electrical resistivity was measured. In almost all cases, the electrical resistance of the composite structures was obtained higher when the bending roller had reached the maximum set displacement value compared to the initial electrical resistance value at the beginning of each bending procedure. Also, in almost all cases the electrical resistivity of the composite structures remained elevated when the bending roller was returned to initial position. This is due to the fact that when the specimens are bent, inner carbon fibers are distorted and deformed, meaning the conductive carbon fiber paths are also deformed and because of that the current which flows inside composite’s structure has to find other, more complex conductive paths and because of that the overall resistivity of the composite structure rises. Despite the fact that the electrical resistance of carbon fiber reinforced composites fluctuates unpredictably, the overall resistivity tends to increase as the value of the displacement increases. More importantly, experimental data revealed that it is possible to detect structural damage to the composite structure when measuring composite’s resistivity. In the event of structural damage, the resistivity of the composite structure significantly increases in all cases.

Conclusions

1. In this final work, the methodology for investigating the electroconductive properties of 3D printed carbon fiber reinforced composite structures was analyzed and described in detail. Composite structures must be properly processed and additional materials has to be used to obtain the most accurate electrical measurements in order to monitor structural damage done to the composite structures. The new methodology was tested during three-point bending and fatigue experiments. The experimental data proved the reliability of the new developed methodology to detect structural damage of composite structures.
2. Five composite specimens were manufactured by fused deposition modelling technique according ‘ASTM D3039’ standard. The specimens were made of PLA thermoplastic, which acted as a matrix, and the reinforcing material was selected to be carbon fiber, which accounted for 20 wt.% of the specimen. First specimen was manufactured to have two layers and it was assigned for the fatigue tests. Second and third specimens were manufactured for three-point bending experiments and they consisted of 3 layers. Meanwhile, fourth and fifth specimens had 4 layers and they were also assigned for three-point bending experiments in order to better analyze the electrical properties.
3. Two fatigue experiments of a different kind were performed. During first fatigue experiment, vibrations were induced to the specimen for approximately 23 hours. The experimental data showed that when the specimen was exposed to a constant external force for a long time, its resistance decreased and stabilized over a long period of time. The resistivity of the composite structure decreased by 0.53 Ω after 12 hours into the experiment. Fatigue experiment also showed that it may be possible to observe the primary signs of fatigue due to increasement of the resistivity of the composite structure. Meanwhile, the second fatigue experiment showed a strong resistivity dependency on external load. During almost all intervals of the experiment the resistivity of the composite increased by approximately 0.3 Ω when the vibrations were induced to the specimen. Also, the resistivity of the composite always dropped down when the vibrations were cancelled by approximately 0.3 Ω almost in all cases.
4. During three-point bending experiments four specimens were tested and their electrical resistivity was measured. Experimental data showed that during bending procedure the resistance of the composite structure tends to increase while fluctuating, when the displacement of the bending roller is increasing. In almost all cases, the resistivity of the composite structure at the end of bending procedure was obtained higher than at the beginning of bending procedure. The largest resistance changes in all composite structures were obtained during the last bending procedures, when the specimens were bent to fracture. The biggest resistivity changes were obtained in the first, four-layered specimen as it increased by 0.096 Ω at the end of experiment. Three-point bending experiment proved the strong resistivity dependency on external loading. More importantly, experimental data revealed that it is possible to detect severe structural damage to the composite structure when measuring composite’s resistivity. In the event of structural damage, the resistivity of the composite structure significantly increases in all cases.

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