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Additive Manufacturing of Carbon Nanotubes Infused Multi-Materials and their Mechanical Characterization

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Abstract

Material extrusion (MEX) is a well-known additive manufacturing (AM) technique used to create 3D objects by extruding semi-molten thermoplastic materials from a heated nozzle or nozzles onto a platform. This technique is commonly used and it is a low-cost fabrication solution for several practitioners. Lately, fiber-reinforced MEX is gaining popularity considering its advantages in light weight and high strength compared to the traditional polymers. Adding Carbon Nanotubes even makes this process more unique and it is a new direction for a number of recent AM studies. This research provides the current findings of the mechanical characterization investigation of such components fabricated by the MEX processes.

Introduction

The filaments used in the MEX process are usually made of polymeric materials such as epoxy, nylon, polycarbonate (PC), polyester, acrylonitrile butadiene styrene (ABS), polylactic acid (PLA), and polyamide (PA) [1]. MEX has several advantages including the ability to produce complex parts, its low cost, minimal material wastage, design flexibility, customization of products for individual consumers, and production of small lots of parts [2].

Investigation of the mechanical behavior of composite materials manufactured by MEX has become significant due to their applications in the structural field [3]. Mechanical testing encompasses a range of loading conditions, such as tension, compression, torsion, bending, or their combinations which are used to assess the performance and durability of materials under cyclic loading [4]. The addition of multiwalled carbon nanotube (MWCNT) is observed as a notable surge in the widespread adoption nowadays due to their remarkable and distinctive properties [5]. It is now known that adding nanoparticles enhances the physical properties of the polymer materials for advanced applications, such as microelectronic, chemical sensors, electromagnetic interference shielding (EMI) [6], and aerospace [7]. This study has examined various mechanical properties, encompassing tensile strength and compression behavior in PLA, PETG, and ABS polymers incorporated with MWCNTs. Figure 1 shows the timeline of integration of MWCNTs to AM processes [8].

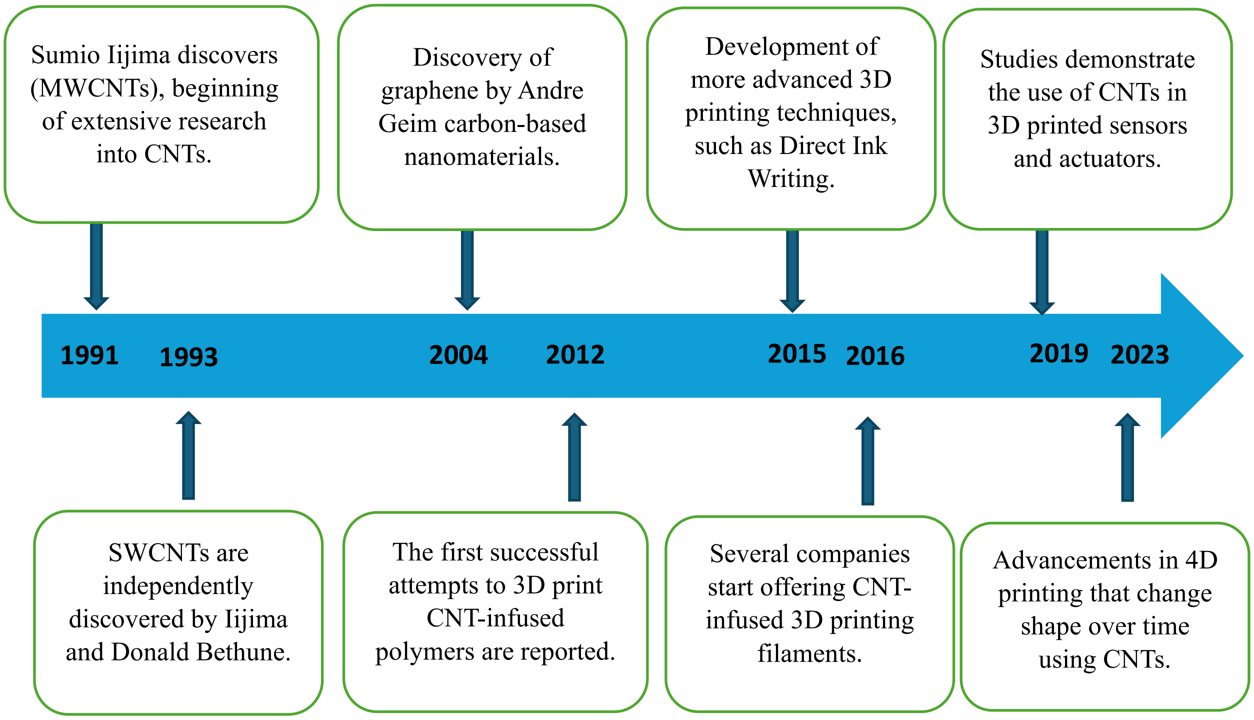


Figure1: Timeline of integration of MWCNTs in 3D printing.

Problem Statement

The incorporation of carbon nanotubes into multi-materials via AM processes poses significant challenges. These challenges include ensuring uniform dispersion of MWCNTs within the matrix and achieving strong interfacial bonding between the MWCNTs and the base materials [9]. Achieving high print quality and maintaining the structural integrity of MWCNT-infused multi-materials is complex due to the varying properties of different material issues such as material compatibility, print defects, and anisotropy in printed structures need to be addressed [10].

While MWCNTs are known for their unique mechanical properties, effectively transferring these properties to multi-material composites through AM remains challenging [11]. Overall, the degree to which MWCNT infusion can enhance properties such as tensile strength, hardness, and impact resistance in 3D-printed multi-materials should be investigated [12] [13].

Significance of the Study

MWCNTs are renowned for their good mechanical, thermal, and electrical properties. Integrating MWCNTs into multi-material composites through AM can lead to the development of materials with significantly enhanced properties [14]. This study contributes to the advancement of material science by exploring innovative ways to leverage the unique characteristics of MWCNTs. The infusion of MWCNTs into multi-materials has the potential to drastically improve mechanical properties such as tensile strength, hardness, and impact resistance [15]. This study is aimed to provide valuable insights into the degree of enhancement that can be achieved, leading to the development of stronger, more durable materials that can outperform conventional composites and used in various applications [16]. Table 1 shows the latest AM research studies and their findings related to MWCNTs.

Literature Review

In the last few years, several research studies have been presented about the enhancements made on the knowledge blocks of additively manufactured MWCNT components. Table 1 summaries just few of them.

Table 1: Latest AM research studies and their findings related to MWCNTs

Key Article Information	Core Finding of Article
Contemporary review on carbon nanotube (MWCNT) composites and their impact on multifarious applications [17] Sherma et al. 2023, Polymers	Demonstrated enhanced mechanical properties and electrical conductivity in MWCNT-reinforced polymer composites. Optimized dispersion techniques led to significant improvements in tensile strength and thermal stability.
Study of zno-MWCNT nanocomposites in high-pressure conditions [18] Cursaru et al. 2023, Materials Today	Developed multifunctional structures with integrated MWCNTs for applications in sensors and actuators. Achieved uniform MWCNT distribution and strong interfacial bonding, resulting in improved performance.
Improved bond strength, reduced porosity and enhanced mechanical properties of 3D-printed polyetherimide composites by carbon nanotubes Chen et al. 2022, Composites Science and Technology [19]	Investigated the effect of MWCNT concentration on the mechanical properties of 3D printed polymers. Found that an optimal MWCNT loading of 1.5% by weight provided the best balance between strength and printability.
Fabrication of high-performance MWCNT reinforced polymer composite for additive manufacturing by phase inversion technique Parnian et al. 2022 Polymers [20]	Demonstrated the feasibility of direct ink writing for fabricating MWCNT-infused ceramic components. Achieved enhanced fracture toughness and thermal shock resistance.

4D-printed low-voltage electroactive polymers modeling and fabrication Luo B et al. 2022, Nature Communications [21]	Explored 4D printing techniques with MWCNT-embedded materials, enabling shape transformation and self-healing properties. Highlighted potential applications in adaptive and responsive systems
Electrical conductivity of MWCNT/polymer composites: 3D printing, measurements and modeling Mora et al. 2021, Composites [22]	Showed that incorporating MWCNTs significantly enhanced the electrical conductivity of 3D printed composites, making them suitable for printed electronics and conductive pathways.

Materials and Methods

To investigate the properties of MWCNT infused polymers, we have experimented the diverse group of materials which involves electrostatic discharge Multi-Walled Carbon Nanotubes (ESD MWCNT) infused PLA, ESD MWCNT infused PETG, ESD MWCNT infused ABS. To find out mechanical characterization of this samples, tensile testing is carried out [23]. Although MWCNTs are promising materials in various fields due to their exceptional properties, impurities present in MWCNT samples can significantly affect their performance and properties [24].

Scanning electron microscopy (SEM) was employed to investigate the microstructure and surface morphology of MWCNT-infused filaments. This analysis provided detailed insights into the dispersion of MWCNTs within the polymer matrices, revealing the degree of uniformity and identifying the presence of any agglomerates. Additionally, SEM allowed for a thorough evaluation of the filaments' surface characteristics, offering crucial information about the material's structural integrity and overall quality [25].

Hence, accurate determination of purity is crucial for ensuring the quality and reliability of MWCNT-based applications. The parameters used for printing the specimens for MWCNT PLA, MWCNT PETG, and MWCNT ABS are given in Table 2.

Table 2: Printing parameters of specimens

Parameters	Values for PLA	Values for PETG	Values for ABS
Nozzle temperature (°C)	210 and 240	230 and 260	210 and 220
Bed temperature (°C)	60	75	90
Infill density (%)	100	100	100
Infill pattern	Line (0/90)	Line (0/90)	Line (0/90)
Layer width (mm)	0.35	0.35	0.35
Layer height (mm)	0.2 and 0.3	0.2 and 0.3	0.2 and 0.3
Printing speed (mm/sec)	15	15	15

Specimen fabrication and preparation

For the preparation of tensile test specimens, ASTM standard E606M [26] was followed. The dimensions of the prepared specimens are illustrated in Figure 2. Each specimen measures 135 mm in length, 20 mm in width, and 5 mm in thickness, with a curvature radius at the gauge length of 10 mm. A single nozzle single-material 3D printer called PRUSA 3.9. is used to fabricate multi-material structures. The printer operates on the principle of the MEX technique and allows for the simultaneous extrusion of single materials through a single nozzle [27]. The filaments used had a diameter of 1.75 mm, with MWCNT PLA, MWCNT PETG, MWCNT ABS being Black in color and printed in standard room temperature specimen configuration is shown in figure 2 [28]. For this experiment one iteration of specimen printing is done and SEM results are achieved after the tensile testing of specimen. XZ specimens are printed in vertical direction with 100 % infill density.

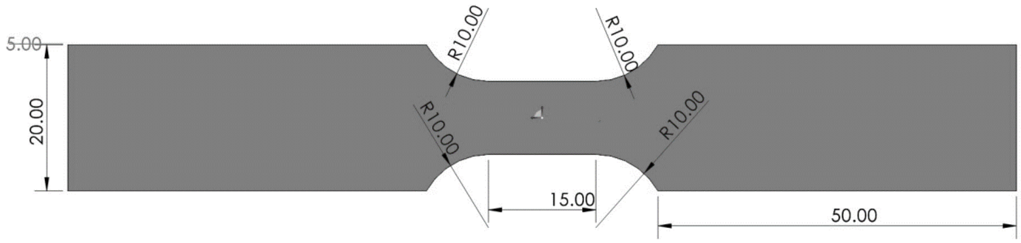


Figure 2: Dimensional details of the fatigue test specimen as per E606M

In the present study, total 51 sample were printed with XY, XZ variation two nozzle variations of 0.4 and 0.8 mm and varying layer thickness of 0.2 and 0.3 mm. This gradient transition aimed to enhance the robustness of the material interface.

Experimental Setup

In this study, uniaxial tensile were performed on plastic specimens using a test resources 810E4 [29] load frame equipped with a 15 kN load cell, in accordance with the ASTM E606M standard. The Newton Test ware [30] interface was used to control and input all testing parameters, including frequency, amplitude, and average load. A PID controller was utilized to maintain the desired stress level throughout the tests [28]. The experimental test setup is illustrated in figure 3.

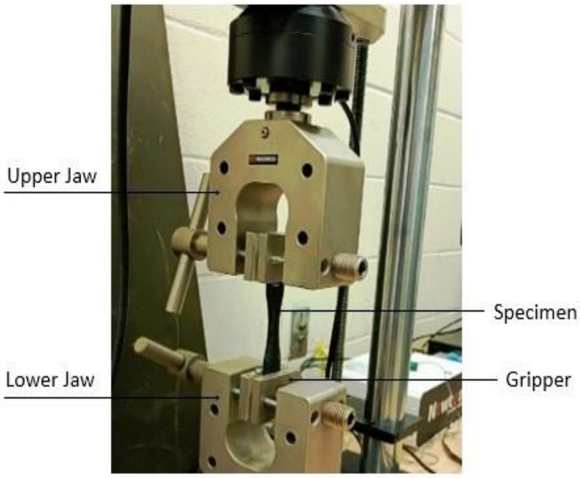


Figure 3: Test setup

Results and Discussions

A. Analysis of Tensile Behavior

The focus of this test was to determine mechanical characterization of MWCNT PLA, MWCNT PETG, and MWCNT ABS specimens and how interstation of MWCNTs affects the mechanical behavior and properties [31]. The MWCNT-reinforced PLA sample in the XY orientation, printed with a 0.8 mm nozzle at a temperature of 240°C, achieved the maximum tensile strength of 56.16 MPa, as shown in figure 4. In contrast, the XZ-oriented PLA sample, also printed with a 0.8 mm nozzle at 240°C and a layer thickness of 0.2 mm, exhibited the lowest tensile strength of 22.1 MPa, as illustrated in figure 4.

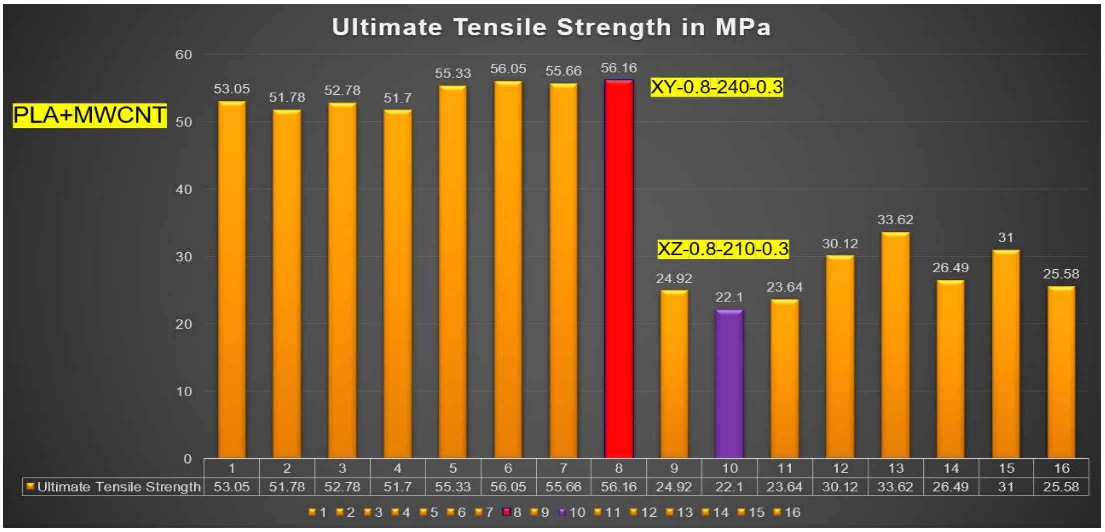


Figure 4: Tensile strength for PLA+MWCNT

The MWCNT-reinforced PETG sample printed in the XY orientation using a 0.8 mm nozzle at a temperature of 260°C exhibited the highest tensile strength of 41.83 MPa, as depicted in figure 5. In contrast, the XZ-oriented PETG sample, printed with a 0.4 mm nozzle at 230°C and a layer thickness of 0.2 mm, demonstrated the lowest tensile strength. For the MWCNT-reinforced ABS sample, the highest tensile strength of 30.88 MPa was observed in the XY orientation when printed with a 0.8 mm nozzle at 220°C. However, the XZ-oriented ABS sample printed with a 0.4 mm nozzle at 240°C and a layer thickness of 0.2 mm exhibited the lowest tensile strength, measuring 9.8 MPa.

These findings highlight the critical role of printing parameters and orientation in optimizing the mechanical properties of 3D-printed MWCNT composite materials. For all the specimen printing we have used filament from 3DXTECH [32].

The deviation in tensile strength across the multiple printed samples was carefully analyzed to assess the consistency and reliability of the results. For the PLA MWCNT samples, the tensile strength showed a standard deviation of ±2.5 MPa. The PETG MWCNT samples exhibited a slightly higher deviation, with a standard deviation of ±3.1 MPa. In contrast, the ABS MWCNT samples had the highest variation, with a standard deviation of ±4.2 MPa. A smaller nozzle diameter might cause shear forces that help in better dispersion of MWCNTs, while a larger nozzle might not achieve the same level of dispersion, affecting the mechanical properties [33]. The nozzle diameter directly affects the flow of material and the resolution of the printed layers. A smaller nozzle diameter allows for finer resolution, which can improve the bonding between layers, leading to better layer adhesion and potentially higher tensile strength.

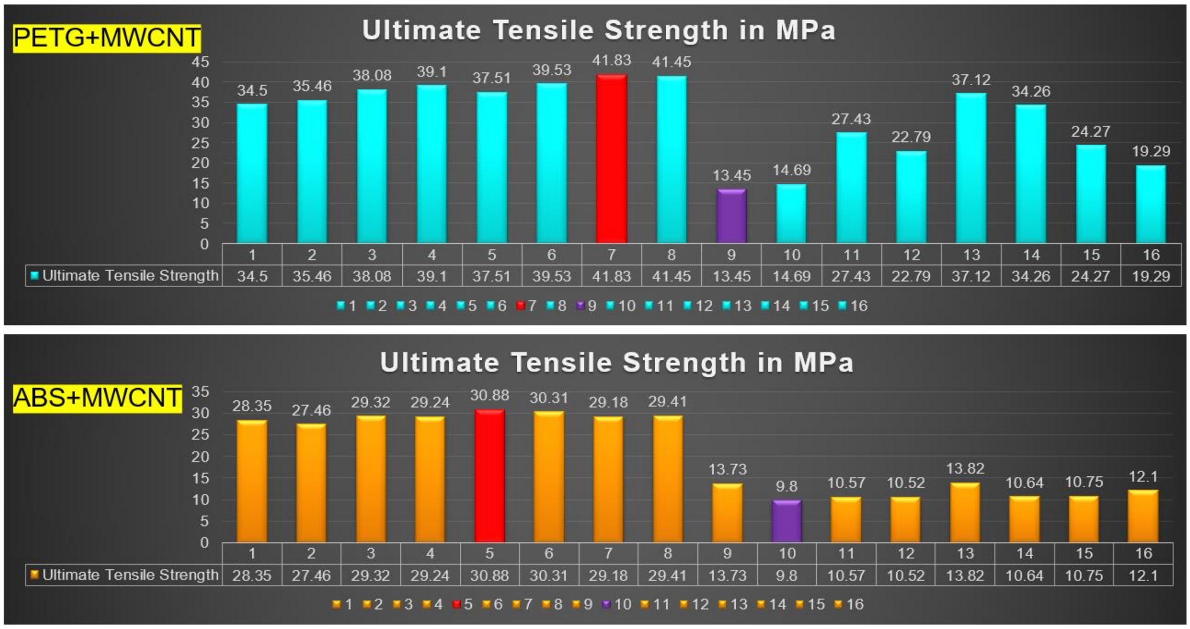


Figure 5: Tensile strength for PETG+MWCNT and ABS+MWCNT

Extrusion temperature affects the viscosity of the polymer matrix and the flow of the material. If the temperature is too low, the material may not flow properly, leading to poor layer adhesion and weaker tensile strength. On the other hand, too high of a temperature can degrade the material or MWCNTs, reducing their reinforcing effect.

Nozzle diameter, extrusion temperature, and build orientation play critical roles in determining the maximum tensile strength of MWCNT specimens by influencing layer adhesion, MWCNT dispersion, and alignment, as well as the overall mechanical properties of the printed material. This becomes particularly valuable in the context of AM, specifically MEX processes, where MWCNT components can be intricately fabricated on the XY plane [34]. This breakthrough holds the transformative promise for diverse applications, such as aerospace, automotive, and biomedical fields. Components with varying strengths and stiffness profiles can be strategically designed to optimize performance, minimize weight, and enhance structural integrity. As a result, these findings contribute significantly to the advancement of customizable and high-performance material systems. Ultimately fostering innovation and practical implementation of MWCNT based polymers in various engineering sectors.

B. Analysis of Scanning electron Microscope

In this study, SEM was employed to analyze the microstructure and surface morphology of MWCNT infused filaments fabricated from three different polymer matrices: MWCNT PLA, MWCNT PETG, and MWCNT ABS. The SEM analysis aimed to investigate the dispersion of MWCNT within the polymer matrix, the presence of agglomerates, and the overall surface characteristics of the filaments. SEM results are achieved after the tensile testing of specimen.

The SEM table is configured to change the stationary position of the specimen holder in any of three orthogonal linear directions and an angular direction. The rotatable iron optics are configured to emit an iron beam towards a predetermined location on the specimen from any of the one or more iron sources at any angle around an axis that is orthogonal to a horizontal surface of the table . The imaging device is configured to generate an image of the specimen including the predetermined location, thereby enabling real-time monitoring of the milling or machining process [7].

C. Experimental Details

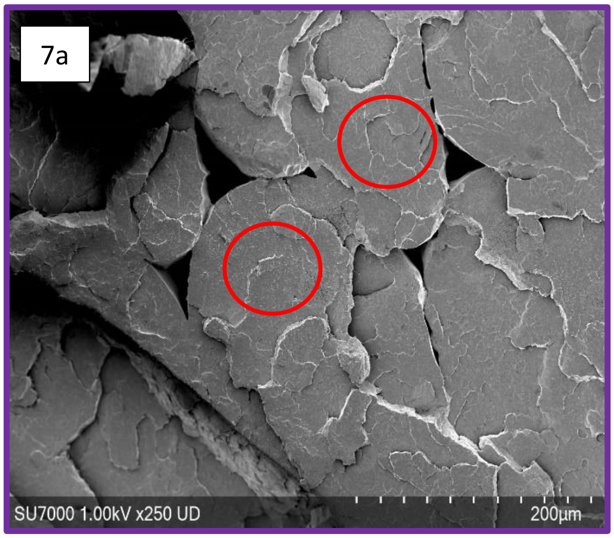
MWCNT infused filaments of PLA, PETG, and ABS were prepared using a melt blending technique. Samples were prepared by cutting the tensile stress specimen into small sections and mounting them onto SEM stubs using conductive adhesive. The samples were sputter-coated with a thin layer of gold to enhance conductivity and minimize charging effects during imaging. SEM analysis was conducted using a high-resolution field emission SEM shown in figure 6 [35].

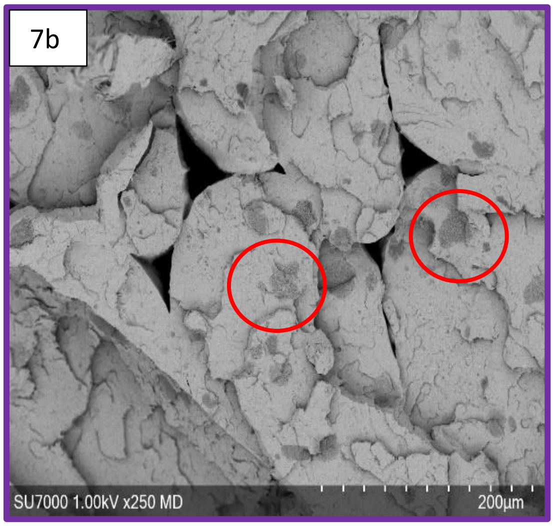
Figure 6: Test setup on SEM

D. Results and Discussion of SEM

D. 1. Dispersion of MWCNT

SEM images revealed the dispersion of MWCNT within the polymer matrix for all three filaments. In MWCNT PLA, a relatively homogeneous dispersion of MWCNT was observed, with individual nanotubes well-dispersed and distributed throughout the matrix as shown in figure 7. MWCNT PETG exhibited a similar dispersion pattern, with MWCNT evenly distributed within the polymer matrix, indicating good compatibility between MWCNT and PETG. In MWCNT ABS, although MWCNT dispersion was observed, some regions showed agglomeration, suggesting potential challenges in achieving uniform dispersion within the ABS matrix.





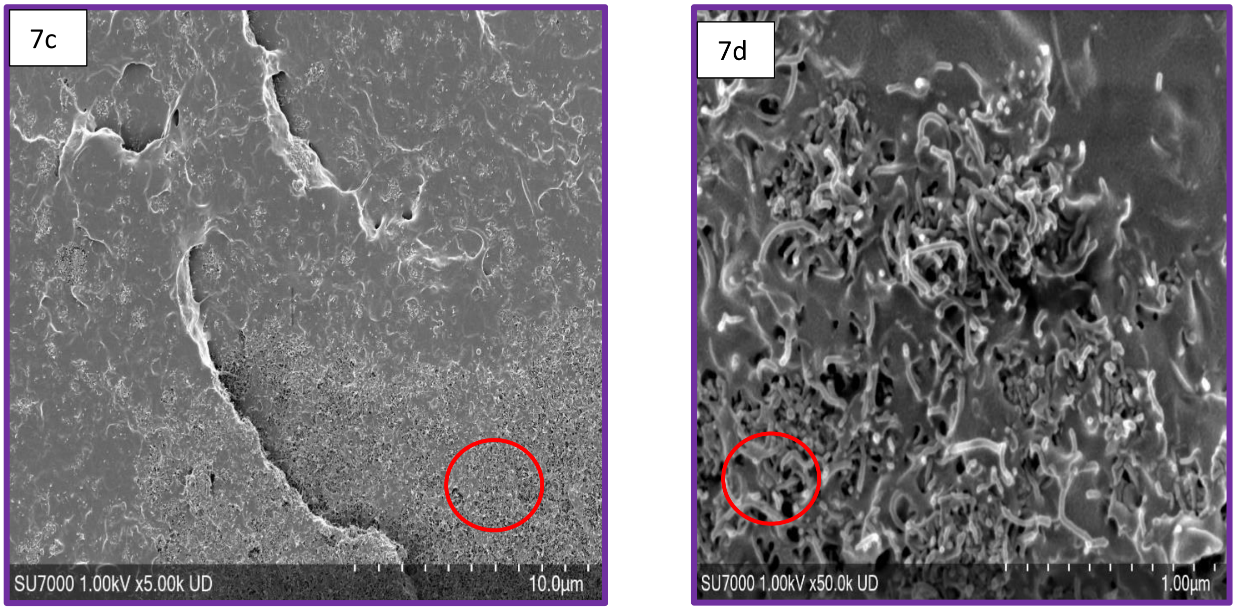


Figure 7: 7a) PLA virgin material micrograph 7b) PLA infused MWCNT micrograph 7c) surface topology of MWCNT concentration at crack 7d) Randomly placed MWCNTs nano filler material

D.2. Agglomeration Behavior

Agglomeration of MWCNT was evident in certain regions of the MWCNT ABS filament, indicating poor dispersion and possible interfacial interactions between MWCNT and ABS. Agglomerates were relatively smaller and less frequent in MWCNT PLA and MWCNT PETG filaments as shown in figure 8, suggesting better dispersion and compatibility with these polymer matrices.

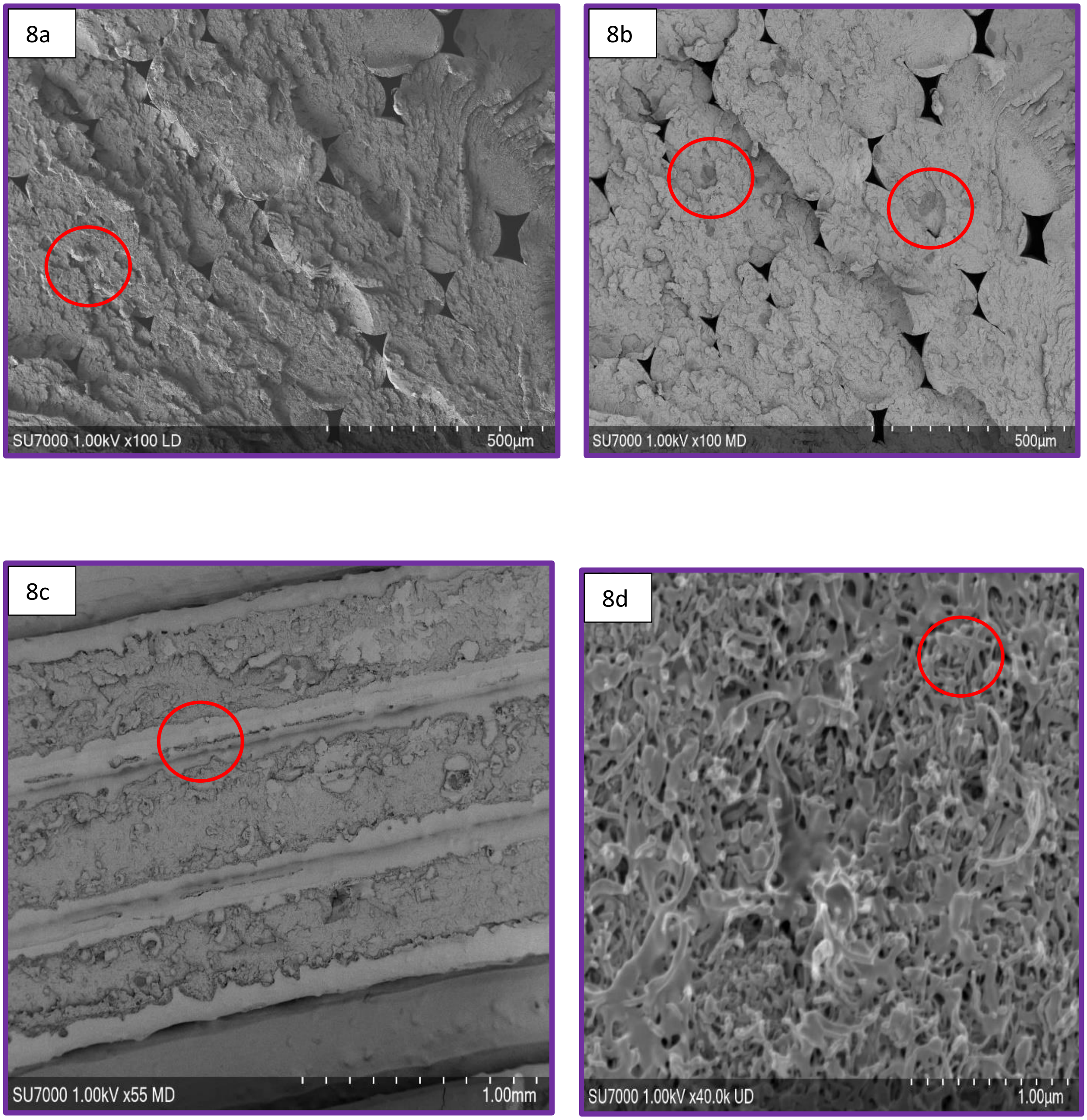


Figure 8: 8a) PETG virgin material micrograph 8b) PETG infused MWCNT micrograph 8c) surface topology of MWCNT concentration at crack 8d) Randomly placed MWCNTs nano filler material

D.3. Surface Morphology

The surface morphology of all three filaments appeared to be influenced by the presence of MWCNT. MWCNT PLA and MWCNT PETG surfaces exhibited a rougher texture compared to their respective neat counterparts, indicating the presence of MWCNT protruding from the surface. MWCNT ABS surface showed a mixed morphology as shown in figure 9, with regions of roughness attributed to MWCNT protrusions and areas of smoother surface, possibly due to polymer matrix covering MWCNT agglomerates [36].

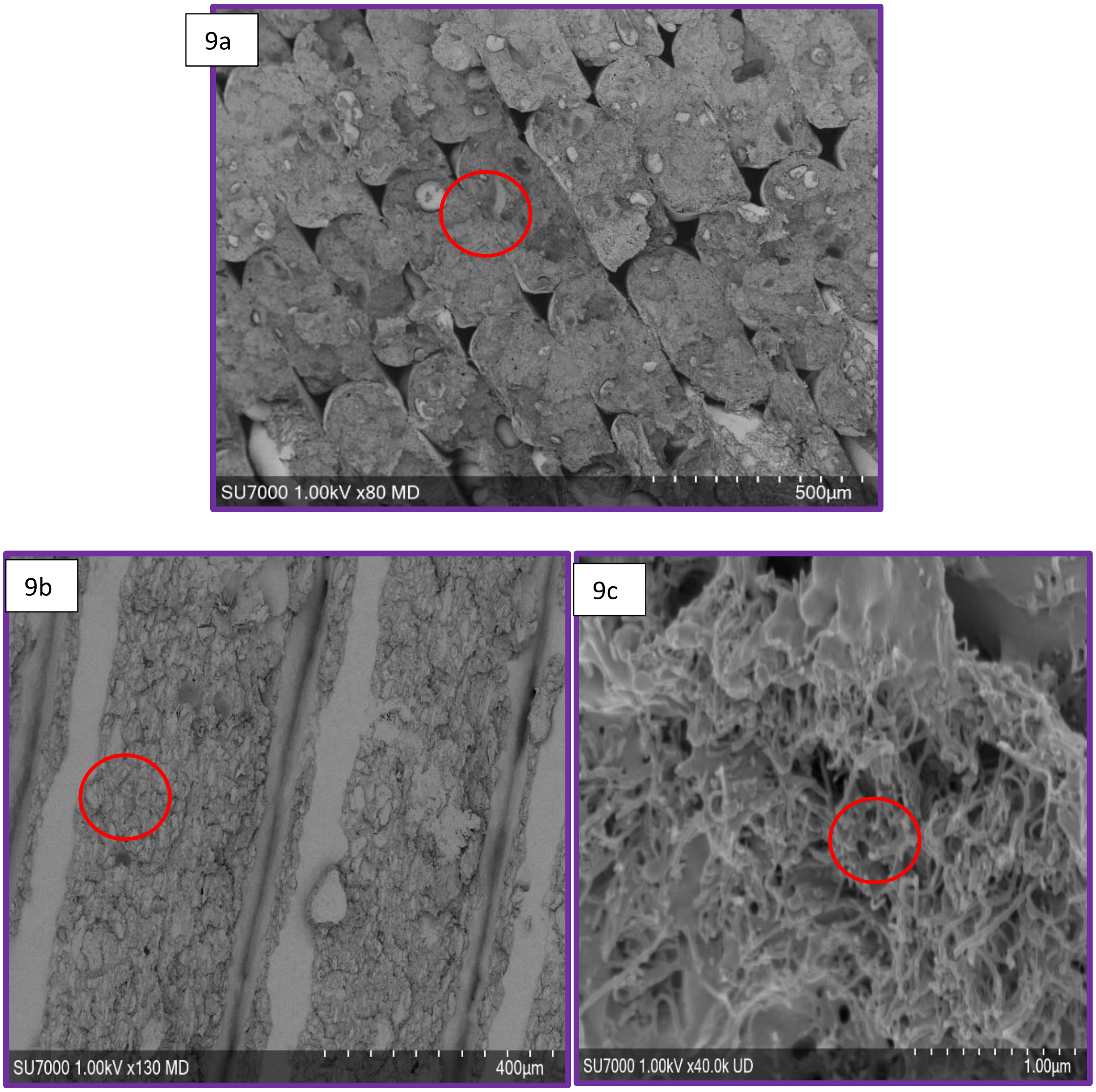


Figure 9: 9a) ABS infused MWCNT micrograph 9b) surface topology of MWCNT concentration at crack 9c) Randomly placed MWCNTs nano filler material

SEM analysis of MWCNT infused filaments revealed varying degrees of dispersion, agglomeration behavior, and surface morphology depending on the polymer matrix. MWCNT PLA and MWCNT PETG exhibited relatively uniform dispersion of MWCNT with minimal agglomeration, whereas MWCNT ABS showed signs of agglomeration and uneven dispersion. These findings provide valuable insights into the microstructural characteristics of MWCNT infused filaments, which are essential for understanding their mechanical, thermal, and electrical properties in AM applications. Further investigation is warranted to optimize the dispersion of MWCNT within the ABS matrix and mitigate agglomeration effects. Additionally, the influence of MWCNT morphology and loading on the properties of the infused filaments should be explored for comprehensive understanding and potential enhancement of their performance.

These outcomes resonate with the intricate interplay between material composition and mechanical characteristics. They provide invaluable insights for engineering design, allowing for the strategic selection of material ratios based on the desired tensile performance and micro level details [37]. As such, this research underscores the importance of tailoring MWCNT based polymer compositions to achieve maximum mechanical behavior, contributing to the advancement of reliable and high-performance materials.

Conclusion

This research study provides a concise overview of an experimental investigation conducted on MWCNT specimens with varying concentrations, manufactured using the MEX process. The study focuses on characterizing the tensile properties and SEM behavior. The key findings of this research are summarized as follows:

- It is particularly useful for assessing the quality of synthesized MWCNT samples and monitoring structural modifications induced by functionalization. Numerous studies have demonstrated the versatility of SEM in elucidating the structure-property relationships of MWCNT-based materials, facilitating their design and development for various applications.
- The study demonstrates that the mechanical properties of MWCNT-reinforced PETG and ABS composites are highly dependent on the 3D printing parameters, including nozzle diameter, extrusion temperature, and build orientation. The optimal tensile strength for MWCNT PETG was achieved at 0.8 mm nozzle at 260°C in the XY orientation, whereas

the weakest was found with a 0.4 mm nozzle at 230°C in the XZ orientation. Similarly, for MWCNT ABS, the highest tensile strength occurred with a 0.8 mm nozzle at 220°C in the XY orientation, while the lowest was observed with a 0.4 mm nozzle at 240°C in the XZ orientation. The study concludes that precise control of 3D printing parameters, such as nozzle diameter, extrusion temperature, and build orientation, is crucial for optimizing the mechanical properties of MWCNT-reinforced PETG and ABS composites. The XY orientation typically results in higher tensile strength compared to the XZ orientation, due to the direction of the applied load relative to the layer boundaries, a trend consistently observed across all three materials

Future works

Although this unique study produces a number of knowledge blocks for the nanoscale AM processes, especially for MEX type of operations, further studies could be achieved. The scope of some future studies could be given as follows.

- Improving the software capabilities to design MWCNT with varying concentrations is needed.
- Understanding the fracture behavior of multi material interfaces under bending fatigue.
- Impact testing modelling and characterization.

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