

EFFECT OF PROCESS VARIABLES ON THE UNCURED HANDLEABILITY AND
FORMABILITY OF STRETCH BROKEN CARBON FIBER

by

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TABLE OF CONTENTS

1. INTRODUCTION	1
Background	1
Development of Bobcat Machine	6
Stretch Ratio.....	7
Nip Force	8
Line Speed	9
2. OBJECTIVE	10
3. MATERIALS.....	11
Sizing	11
Michelman 181290IX.....	11
GP sizing.....	11
Single tow	11
MSU SBCF	11
Hexcel SBCF	11
4. METHODS	12
Preparation and Verification of Sizing Bath.....	12
Tow Desizing Procedure to Quantify Sizing Application	12
Tow Tenacity	14
Temperature-Controlled Tow Tenacity Test Setup	14
Evaluation of SBCF Reproducibility	16
Evaluation of Reproducibility of Sizing Deposition.....	17
Evaluation of Reproducibility of Tow Tenacity	17
Evaluation of Reproducibility of Tow Tenacity at Intermediate Temperatures.....	18
Evaluation of Reproducibility of Fiber Length Distribution and Mean Fiber Length	18
Preliminary Evaluation of the Effect of Stretch Ratio	19
Preliminary Evaluation of the Effect of Nip Force.....	21
5. RESULTS AND DISCUSSIONS.....	23
Sizing Deposition as a Function of Bath Concentration.....	23
Validation of Elevated Temperature Test Fixture for Tow Tenacity Tensile Test.....	24
Tow Tenacity with Sizing Application	25
Evaluation of SBCF Reproducibility	32
Sizing Deposition.....	32

TABLE OF CONTENTS CONTINUED

Tow Tenacity	33
Evaluation of SBCF Reproducibility: Tow Tenacity at Intermediate Temperatures.....	36
Fiber Length Distribution and Mean Fiber Length.....	38
Effect of Stretch Ratio	43
Sizing Deposition as a Function of Stretch Ratio	43
Fiber Length Distribution as a Function of Stretch Ratio.....	45
Tow Tenacity as a Function of Stretch Ratio.....	51
Effect of Nip Force	53
Sizing Deposition as a Function of Nip Force	53
Fiber Length Distribution as a Function of Nip Force.....	54
Tow Tenacity as a Function of Nip Force	61
6. FUTURE OF SBCF PRODUCTION	64
7. CONCLUSION.....	67
8. RECOMMENDED FUTURE TASKS	69
Tow Tenacity	69
Thermal Behavior	69
Wettability of Sizing	69
REFERENCES CITED.....	70
APPENDICES	74
APPENDIX A: STRETCH RATIO AND NIP FORCE.....	75

LIST OF TABLES

Table	Page
1. Description of SBCF tow batches and associated bath concentrations.....	12
2. Description of materials for the evaluation of SBCF reproducibility	17
3. Description of the materials for the evaluation of the effect of stretch ratio on the MSU SBCF production.	19
4. Description of the materials for the evaluation of the effect of nip force	21
5. Results of analysis of variance (ANOVA) for fiber length as a function of stretch ratio.....	50
6. Comparison of mean using Tukey test for fiber length as a function of stretch ratio.	50
7. Results of analysis of variance (ANOVA) for fiber length as a function of nip force.....	60
8. Comparison of mean using Tukey test for fiber length as a function of nip force.....	60

LIST OF FIGURES

Figure	Page
1. Visualization of sizing application to carbon fiber [11].	2
2. Bobcat machine developed at the stretch broken carbon fiber (SBCF) research group at Montana State University- (a) A spool of single tow unsized continuous carbon fiber tow is introduced as a feed material in the Bobcat machine; (b) Sizing application zone; (c) stretch break zone; (d) Drying zone.....	6
3. A flow diagram of MSU SBCF generation in the Bobcat machine.....	7
4. Schematic diagram of stretch broken tow generation having different stretch ratios.....	8
5. Steps to characterize sizing weight percent on tows: (a) Tows were dipped in 20 ml acetone solution in each beaker for 30 minutes; (b) The beakers were placed in a sonicator for 30 minutes; (c) The tows were transferred to a vacuum assisted filtration setup.	13
6. (a) Schematic of the elevated temperature test fixture. The horizontal Mark 10 tensile test apparatus controls the movement of the grips and recording the load-displacement data; (b) demonstration of the new horizontal test setup with a 152.4 mm (6 inches) tow touching the heated Teflon™ sheet. The gap between the heated Teflon™ and the grip at both sides was 12.7 mm (0.5 inches); (c) the tow was stretched from 152.4 mm (6 inches) to 177.8 mm (7 inches). The temperature reading given by the hot plate does not represent the true temperature of the heated tow. Therefore, a thermocouple was used to manually calibrate the temperature of the heated tow.....	16
7. Resultant sizing deposition on tow vs. measured bath concentration; each point is an average of three samples from each batch of SBCF material.	23
8. Temperature profile for the target temperature of 100°C. The red squares indicate the target temperature. The blue circles indicate the average (ten temperature measurements for each point) recorded temperature along the tow.	25
9. Relationship between the peak load obtained from the load-displacement graphs vs. sizing deposition. The room temperature results (red, square) demonstrate the back-tension ability of the MSU SBCF tows. The elevated temperature results (100°C, blue, circle) indicate tow formability. Each point in the graph is an average of five samples; error bars represent the standard deviation and may lie within the data marker.	26

LIST OF FIGURES CONTINUED

10. Load-displacement graph of the samples tested at room temperature from batches (a) BC2_0033 (control with no sizing applied to it); (b) BC2_0028 (average sizing deposition: 0.31 wt.%); (c) BC2_0029 (average sizing deposition: 1.75 wt.%) and (d) BC2_0027 (average sizing deposition: 6.57 wt.%). Five test coupons were prepared and tested from each batch. The average peak load, standard deviation and coefficient of variation are also included. 28
11. Load-displacement graph of the samples tested at elevated temperature (100°C) from batches (a) BC2_0033 (control with no sizing applied to it); (b) BC2_0028 (average sizing deposition: 0.31 wt.%); (c) BC2_0029 (average sizing deposition: 1.75 wt.%) and (d) BC2_0027 (average sizing deposition: 6.57 wt.%). Five test coupons were prepared and tested from each batch. The average peak load, standard deviation and coefficient of variation are also included..... 29
12. Tensile test coupons prepared from single tow MSU SBCF for the room temperature tow tenacity experiment. (a); pre-tensile test. (b) post-tensile test. The tow failed in a brittle manner under applied tension since sizing acts as a solid binder at room temperature..... 30
13. Tensile test coupons prepared from single tow MSU SBCF for the elevated temperature (100°C) tow tenacity experiment. (a); pre-tensile test. (b) post-tensile test. The tow was stretched from 152.4 (6 inches) mm to 177.8 mm (7 inches) during the elevated temperature tensile test. The decrease in viscosity of the sizing at elevated temperature provided a lubricating effect to the fibers in a tow, which facilitates fiber-fiber sliding during the tow stretching..... 31
14. Comparison of sizing deposition among the samples prepared from BC2_0030, BC2_0031, BC2_0032. BC2_0033 is the control with no sizing applied to it. The results for the Hexcel SBCF are also included. The values in the figure are an average of three samples. 32
15. Room temperature peak load as a function of sizing deposition. Each point is an average value of five samples. BC2_0033 is the control which has no sizing applied to it (DI water was sprayed during the production). A bath concentration of 10 wt.% was used during the production of three replicate batches of MSU SBCF (BC2_0030, BC2_0031, and BC2_0032), which resulted in the average sizing deposition of 1.75 wt.%, 1.86 wt.%, and 1.83 wt.%, respectively. 33
16. Elevated temperature peak load as a function of sizing deposition. The testing temperature was 100°C. Each point is an average value of five readings. 35

LIST OF FIGURES CONTINUED

17. Peak load of single tow MSU SBCF plotted as a function of temperature. The test temperatures were 25°C, 40°C, 50°C, 60°C, 70°C, and 100°C. Each point in the graph is an average value of five readings. Boltzmann fitted curves were also plotted.	36
18. Comparison of fiber length distribution among the three MSU SBCF replicate batches (a) BC2_0030; (b); BC2_0031 (c) BC2_0032; and (d) the Hexcel legacy SBCF.....	38
19. Fiber length distribution plotted as frequency of continuity vs. fiber length for (a) BC2_0030; (b) BC2_0031; (c) BC2_0032; (d) Hexcel SBCF.	40
20. Comparison of mean fiber length between the MSU SBCF tows from three replicate batches (BC2_0030, BC2_0031, and BC2_0032) and Hexcel SBCF. Minimum, maximum, 10 th percentile and 90 th percentile values of fiber length are also shown.....	41
21. Sizing deposition as a function of stretch ratio. Each point is an average value of three readings, error bars show the standard deviation.....	43
22. Fiber length distribution as a function of stretch ratio. The y-axis on the left represents the relative frequency and the one on the right denotes the frequency of continuity.	45
23. Fiber length distribution plotted as frequency of continuity vs. fiber length for (a) 15% stretch ratio; (b) 20% stretch ratio; (c) 25% stretch ratio. The brown box indicates both the upper tail and lower tail region.	48
24. Representation of mean fiber length as a function of stretch ratio. The minimum, maximum, 10 th percentile and 90 th percentile values of the fiber length are also shown.	49
25. Figure: Tow tenacity as a function of stretch ratio. Each point is an average value of 5 measurements. The error bar might lie within the markers.	52
26. Sizing deposition as a function of nip force. Each point in the graph is an average value of three measurements. The error bars might lie within the markers.....	53
27. Fiber length distribution as a function of nip force: (a) 8007 N (1800 lbf); (b) 8896 N (2000 lbf); (c) 9786 N (2200 lbf); (d) 10676 N (2400 lbf); (e) 11565 N (2600 lbf).	55

LIST OF FIGURES CONTINUED

28. Fiber length distribution plotted as frequency of continuity vs. fiber length for the nip forces of (a) 8007 N (1800 lbf); (b) 8896 N (2000 lbf); (c) 9786 N (2200 lbf); (d) 10676 (2400 lbf); (e) 11565 N (2600 lbf). The red box denotes both the upper tail region and the lower tail region.	58
29. Representation of mean fiber length as a function of nip force. The minimum, maximum, 10 th percentile and 90 th percentile values of the fiber length are also shown.	59
30. Tow tenacity as a function of nip force. Each point is an average value of 5 measurements.	61
A.1. Sizing deposition as a function of stretch ratio. Each point is an average value of three readings, error bars show the standard deviation.	76
A.2. Unit mass of desized tows as a function of stretch ratio.	77
A.3. Tow tenacity as a function of stretch ratio. Each point is an average value of 5 measurements. The error bar might lie within the markers.	79
A.4. Sizing deposition as a function of nip force. Each point in the graph is an average value of three measurements. The error bars might lie within the markers.	80
A.5. Unit mass as a function of nip force. The error bars lie within the markers.	81
A.6. Tow tenacity as a function of nip force. Each point is an average value of five measurements.	82

ABSTRACT

Carbon fiber is a high-performance reinforcing material used extensively in aerospace composites. Although carbon fiber is used in both continuous and discontinuous form, the continuous carbon fiber is limited by its inability to stretch due to its low strain to failure during manufacturing structures with complex geometries. Stretch broken carbon fiber (SBCF) is a type of discontinuous and aligned carbon fiber which has the potential to solve this limitation of inextensibility of its continuous counterpart. The discontinuous nature of SBCF enhances its stretchability making this material a prime candidate for manufacturing parts with complex curvatures. SBCF is generated by stretching the fibers using a pair of differentially driven rollers, which breaks the fibers at their intrinsic flaws. Although SBCF can be stretched due to being discontinuous, it compromises the tensile strength due to the lack of fiber continuity. Therefore, a polymeric coating known as sizing is applied to the SBCF to reconstruct its tensile strength. In the context of SBCF production, sizing serves two important functions. Firstly, sizing provides uncured carbon fiber the desired handleability and back-tension ability. Secondly, sizing enhances the formability of SBCF, which is defined as the ease at which a material can be formed into a desired shape without failure. The goal of this work is to investigate the effect of process variables on the generation of stretch broken carbon fiber with consistent and repeatable material properties. The stretch broken carbon fiber research group at Montana State University (MSU) has developed a stretch breaking machine known as 'Bobcat' to generate single tow MSU SBCF. The noteworthy process variables related to MSU SBCF production includes sizing deposition on the tow, stretch ratio, nip force, line speed, fiber length distribution, and tow tenacity. Target amount of sizing deposition on MSU SBCF tow was achieved by choosing an appropriate sizing bath. A temperature-controlled tow tenacity result suggests that MSU SBCF possesses adequate handleability, back-tension ability and formability. MSU SBCF also shows a narrow fiber length distribution and relatively short mean fiber length which indicate improved formability. Reproducibility of these results were observed in the replicate batches of MSU SBCF. Suitable stretch ratio and nip force regimes were identified to optimize MSU SBCF production.

INTRODUCTION

Background

Carbon fiber is a reinforcing material used extensively in the aerospace industry due to its excellent strength and stiffness [1]. It is used in both continuous and discontinuous form. However, the inextensibility of continuous carbon fiber limits its application in forming complex geometries [2]. The precursor for most commercially available carbon fibers (both continuous and discontinuous) is polyacrylonitrile (PAN). These PAN fibers are produced by a wet spinning technique, then drawn, and carbonized. During the carbon fiber production from the PAN based precursor, the unavoidable release of certain decomposition gases e.g., HCN and H₂O leads to the formation of voids and defects on the surface of carbon fiber which potentially compromises its tensile strength [3].

The motivation for using discontinuous carbon fiber is driven by the inextensibility of continuous carbon fiber. One way to produce discontinuous carbon fiber is to chop the continuous carbon fiber at a predetermined length. However, the resultant chopped fibers are still likely to contain these intrinsic flaws. This limitation can be addressed by generating stretch broken carbon fiber (SBCF). SBCF is a type a discontinuous and aligned carbon fiber produced by stretch breaking using a pair of differentially driven rollers. The stretch breaking process facilitates fiber breakage at natural flaws. The resulting fibers are discontinuous, aligned, and reduced in natural flaws while maintaining the strength and stiffness of a continuous carbon fiber [4], [5]. However, the lack of fiber continuity in the SBCF limits its handleability. Which means that tensile strength of the unimpregnated SBCF tow post stretch breaking, is reduced from that

of its continuous counterpart. Surface treatment of carbon fiber, especially of the SBCF, is a potential solution to address this shortcoming. Some noteworthy surface treatments include electrochemical oxidation, plasma treatment, chemical vapor deposition, and chemical grafting [6]. Although these surface treatments enhance fiber-fiber interaction, the issue of high energy consumption involved in these treatments and complications arising during the curing and manufacturing of composites limit the extensive use of these techniques [7]. On the other hand, a significant effort has been invested over the years to improve another promising fiber surface treatment technique, is the application of sizing [8]. Sizing is a thin polymeric coating [9] applied to the fiber surface to enhance fiber-fiber interaction. Sizing application is inexpensive compared to the treatments mentioned above [10]. Sizing can be classified as a solution (including water-soluble) or emulsion [6]. A schematic of a standard sizing application to carbon fiber is shown in Figure 1. Because of discontinuity in the tow, sizing application to SBCF requires alternative application techniques.

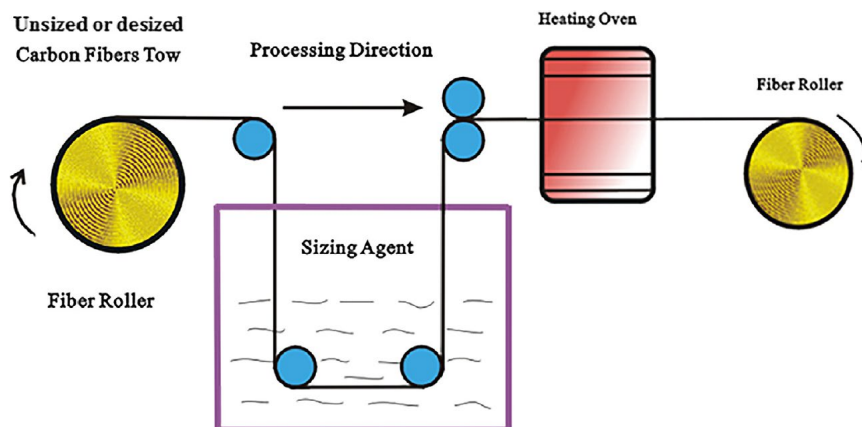


Figure 1: Visualization of sizing application to carbon fiber. Carbon fiber tow is passed through a sizing bath of desired concentration, which forms a thin polymeric coating on the surface of the tow [11].

Sizing in general enhances uncured tow handleability by improving the wettability of fiber surface without any material damage [12]. The increase in wettability of fiber surface by the sizing also improves fiber alignment [13]. Sizing enhances the fiber-fiber interactions by mechanical interlocking or diffusion [14]. Moreover, sizing application to fiber also improves the mechanical performance of the fiber reinforced-cured composites. Yao et al. reported that application of an epoxy based sizing on T700 grade high strength carbon fiber improves the interfacial shear strength of carbon fiber/epoxy composite compared to the composite having unsized carbon fiber [15]. Liu et al. prepared two homemade aqueous epoxy sizing agents and applied them to T300B commercial grade carbon fiber [16]. The contact angle of sized carbon fibers reduced by approximately 23° compared to the unsized carbon fibers, which means sizing improves the wettability of carbon fiber. As a result, the interfacial shear strength of the sized carbon fiber-epoxy composite improved by approximately 70%-76% compared to the unsized carbon fiber composite [16]. Wang et al. reported that when short carbon fiber with no sizing applied to it was used as reinforcement in polyether ether ketone (PEEK) matrix, the tensile strength of the resulting unsized short carbon fiber-PEEK composite was 146.7 MPa [17]. However, when polyimide (PI) sizing was applied to the short carbon fiber (sizing deposition on the short carbon fiber was 1 wt.%), the tensile strength of the PI sized short carbon fiber-PEEK composite was increased to 171.4 MPa. This 16.8% increase in tensile strength of the composite with PI sizing applied to the short carbon fiber compared to the composite with unsized short carbon fiber suggested that the PI sizing improved the interfacial bonding between the short carbon fiber and the PEEK matrix[17].

Sizing application is a potentially viable solution to reconstitute the tensile strength of SBCF. The resulting sized stretch broken carbon fiber attains the strength of a continuous carbon fiber in terms of mechanical performance. Hexcel Corporation has reported stretch broken carbon fiber that is aligned and discontinuous, having a random distribution of fiber lengths with thermosetting sizing applied to it [18]. In the context of stretch broken carbon fiber production, sizing performs two important functions. Firstly, sizing enhances the back-tension ability of SBCF tows. This improvement is because sizing acts as a binder at room temperature. Back-tension of varying magnitude is applied to the carbon fiber tows during the prepreg production. Therefore, it is important for the SBCF tows to maintain strength under variable tension. Secondly, sizing contributes to SBCF formability. Formability of a material is defined as the amount the deformation or strain it can withstand before failure. In the context of SBCF, formability refers to the ease at which it can be formed into a complex geometry. A lower forming force translates to better formability. The forming operations are performed at elevated temperature. At elevated temperatures, sizing reduces in viscosity and starts to flow. This flow provides a lubricating effect to the SBCF tows. The sliding of discontinuous and aligned fibers in the SBCF tow enables the tow to be formed into a desired shape.

One important aspect of sizing deposition on the tow is the amount of sizing deposited on the tow. If the sizing deposition is too high, then the fiber-fiber interaction will be compromised due to the excessive sizing at the fiber-epoxy interface. On the other hand, too little sizing can lead to a poor fiber-epoxy interaction in the cured composite and leads to undesirable fiber-fiber interactions in the tow. If there is not sufficient interaction between fibers in the tow, the SBCF tows can have insufficient tensile strength for handling and forming. Therefore, an optimum

amount of sizing deposition is desirable on the SBCF tows to give adequate back tension ability and formability. Zhang et al. investigated the effect of the amount of sizing deposition on the tensile strength of carbon fiber showed in their work that an increase in the sizing deposition on the single carbon fiber from 0.71 wt.% to 1.20 wt.% resulted in an increase in the single fiber tensile strength from 3.17 GPa to 3.46 GPa whereas, a further increase in the sizing deposition of 1.67 wt.% decreases the tensile strength of the single carbon fiber to 3.34 GPa [19]. He et al. reported that an increase in tensile strength (multifilament tensile strength) from approximately 3.3 GPa to 3.6 GPa was observed when the amount of novolac resin sizing deposited on the carbon fiber was increased from 1.6 wt.% to 4.3 wt.%. However, when the sizing deposition on the carbon fiber tow reached 8.2 wt.% and higher, a decrease in tensile strength was observed [7].

Optimization of sizing application is crucial in producing high quality single tow stretch broken carbon fiber. This optimization can be achieved by evaluating the process variables involved during the SBCF production. The process variables can be categorized into (a) independent process variables and (b) dependent process variables. The independent process variables include sizing bath concentration, sizing flow rate, stretch ratio, nip force, and line speed. These independent process variables can be tuned to optimize the dependent variables, which include sizing deposition on the tow, fiber length distribution, mean fiber length and tow tenacity. Fiber length distribution and mean fiber length are extremely important characteristics of SBCF tows from the perspective of formability of SBCF material. A narrow fiber length distribution and lower mean fiber length is expected to improve forming parts with complex

geometries [18]. Tow tenacity is defined as the tows possessing certain qualities e.g., handleability, back tension ability, and formability and is measured for a single tow in tension.

Development of Bobcat Machine

The stretch broken carbon fiber (SBCF) research group at Montana State University (MSU) has been working on developing and optimizing a stretch breaking machine (Bobcat) for carbon fiber tows. In this machine, a spool of continuous carbon fiber is mechanically stretched to produce stretch broken carbon fiber (SBCF) known as MSU SBCF. The most updated Bobcat machine is shown in Figure 2, where stretch breaking is performed.

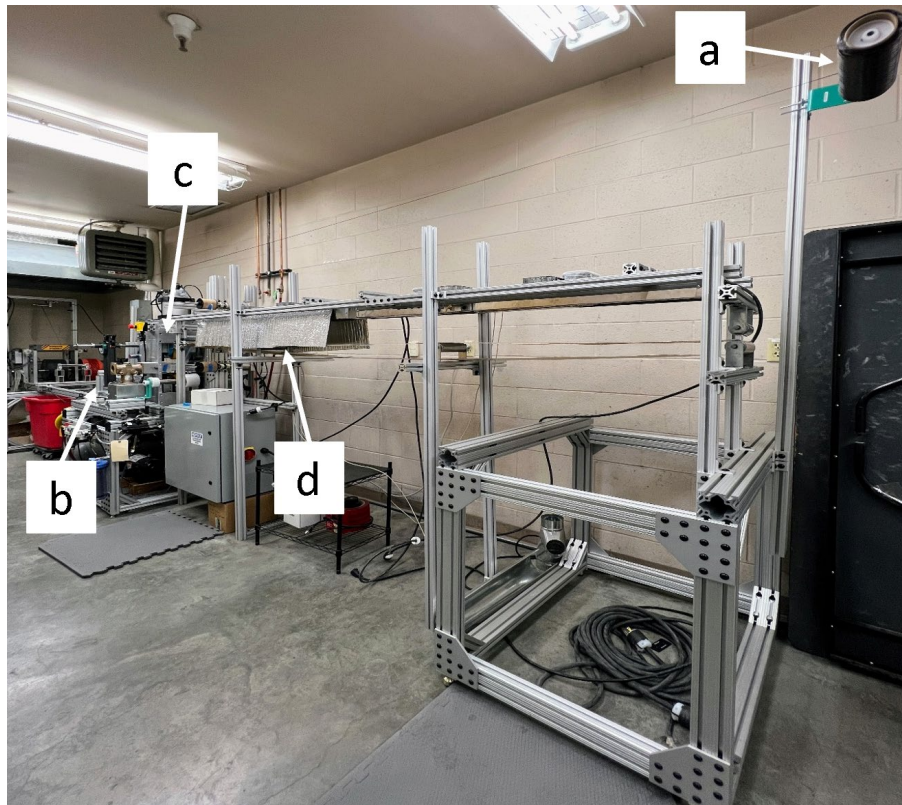


Figure 2: Bobcat machine developed at the stretch broken carbon fiber (SBCF) research group at Montana State University- (a) A spool of single tow unsized continuous carbon fiber tow is introduced as a feed material in the Bobcat machine; (b) Sizing application zone; (c) stretch break zone; (d) Drying zone.

Sizing application is performed by installing a sizing bath of known concentration and a controlled and precise discharge of sizing from the sizing bath to the single tow MSU SBCF. This machine has been equipped with the capability of tuning the relevant process variables to optimize the production of SBCF. These process variables include sizing flow rate, stretch ratio, nip force, and line speed. The aim of this machine is to produce single tow stretch broken carbon fiber known as MSU SBCF. A flow diagram of MSU SBCF production in the Bobcat machine is shown in Figure 3.

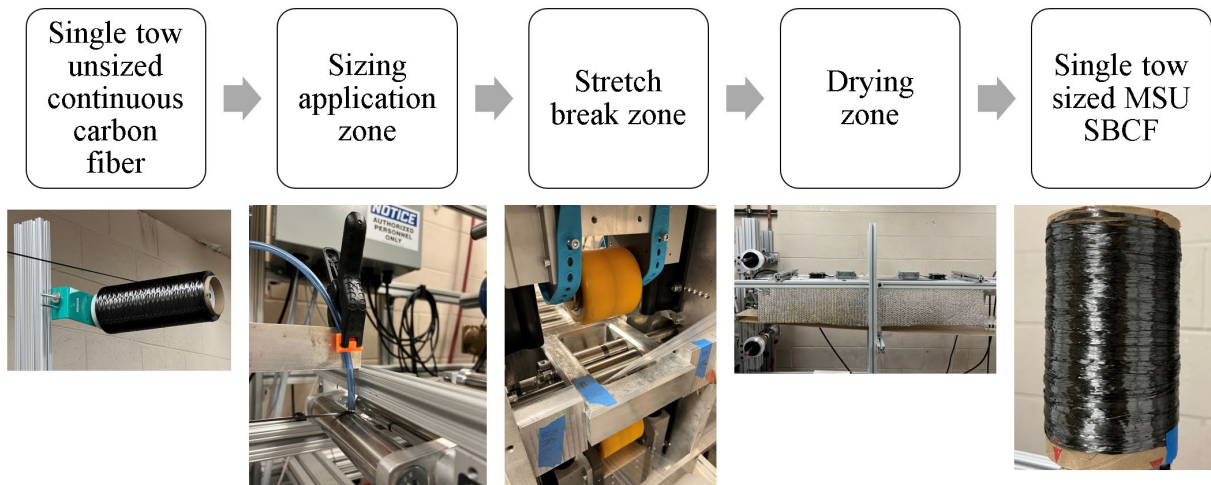


Figure 3: A flow diagram of single tow MSU SBCF production in the Bobcat machine.

Stretch Ratio

During the stretch breaking process, a differential speed is applied between the two drive rollers at the stretch break zone. This differential speed causes the stretching of continuous tow and subsequent breakage into a discontinuous stretch broken tow. If the drive rollers rotate at the same speed, then no stretch breaking takes place. If, for example, the second drive roller rotates at a speed 20% faster than the first drive roller, then stretch broken material will be generated having a stretch ratio of 20%. The concept of stretch ratio is described in Figure 4.

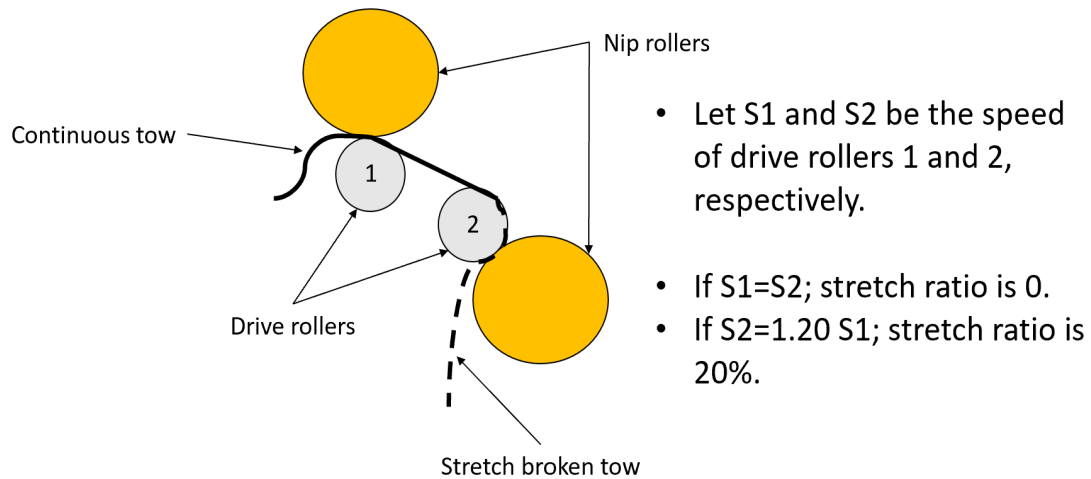


Figure 4: Schematic diagram of stretch broken tow generation having different stretch ratios.

The Bobcat machine can be tuned to accurately control and adjust the stretch ratio. By varying the stretch ratio, process variables such fiber distribution, fiber count, and cross-sectional area can be evaluated. It is expected that lowering the stretch ratio might result in producing SBCF tows having longer fibers and wider fiber distribution due to the lower overall strain on the fibers. Hexcel also investigated the effect of nip force on tow tenacity of SBCF of 76 mm gauge length at elevated temperature (100°C). When the nip force was reduced from the standard value to 36% of its standard value, the tow tenacity (measured in peak load) increased from approximately 20 N to 125 N. The reason for this increase in tow tenacity was attributed to the greater percentage of longer fibers and wider fiber length distribution as the nip force was decreased [18].

Nip Force

The Bobcat machine is equipped with a pair of nip rollers made from polyurethane. When continuous carbon fiber goes through the stretch break rollers, the engagement between the stretch break rollers and the nip rollers takes place, which effectively generates MSU SBCF

from the combined effect of stretching and nip force exerted by the nip roller on the tow. It is expected that increasing the nip force ensures a uniform fiber break with minimal fiber slip in a tow. Hexcel reported the effect of nip force on tow tenacity of SBCF tows of 76 mm gauge length at 100°C. When nip force was lowered from the standard value to 67% of the standard value, the tow tenacity increased from approximately 150N to 390 N. The higher amount of longer fibers and a higher variability in fiber distribution lead to the higher tow tenacity with decreasing nip force [18]. However, using a very high nip force value during the SBCF production might damage the fibers.

Line Speed

Line speed is defined as the rate at which the single tow travels from feeding the continuous carbon fiber in the Bobcat machine to the spooling of the sized single tow MSU SBCF. Not much is known about the effect of line speed on the generation of single tow SBCF. However, it is expected that with increase in line speed, the sizing deposition on the SBCF tows can be lowered. Hence, line speed is also a very important process variable in controlling and optimizing the sizing deposition on the tow. A higher line speed is also desirable to produce spools of single tow sized SBCF at higher rate at a relatively short time period with no compromise in the material properties (such as fiber length) of the SBCF.

OBJECTIVE

The preliminary goal of this research is to develop an understanding of the effect of sizing combined with the relevant process variables on the performance of the uncured single tow MSU SBCF. This goal consists of the following tasks:

1. Target sizing deposition on the MSU SBCF tows ranges from 1.5 wt.%- 2 wt.%.
2. Evaluation of the sized MSU SBCF in terms of sizing deposition on the tow, fiber length, tow handleability, back tension ability, and formability.
3. Evaluation of the reproducibility of MSU SBCF with consistent and reliable material properties.
4. Preliminary evaluation of the effect of process variables (stretch ratio and nip force) on the MSU SBCF production.

Results obtained from the preliminary goal will be assessed to establish a robust and reliable stretch breaking system with a precisely controlled sizing application process capable of generating spools of single tow MSU SBCF at a large scale.

MATERIALS

Sizing

Michelman 181290IX

This epoxy-based sizing was obtained from Michelman Inc. (Cincinnati, OH, USA). This is a water-soluble sizing agent with a solids content of 46 wt.%.

GP sizing

GP is a proprietary sizing prepared by Hexcel Corporation (Stamford, CT, USA). Not much is known about the chemical formula and the preparation of GP sizing.

Single tow

MSU SBCF

Spool of single tow un-sized continuous IM7-12k carbon fiber were obtained from Hexcel Corporation. Tows were stretch broken in MSU's Bobcat machine, a prototype machine for stretch breaking carbon fiber tows. Michelman 181290IX sizing was diluted to the desired concentration and applied to the MSU SBCF.

Hexcel SBCF

Legacy material was available from a previous program by Hexcel for stretch breaking carbon fiber. For this SBCF, Hexcel also started with IM7-12k carbon fiber tows. Stretch breaking was performed by Hexcel and sized with proprietary GP sizing as part of their process.

METHODS

Preparation and Verification of Sizing Bath

To establish a relationship between the sizing deposition on the tow from different sizing bath concentrations as applied during the stretch breaking process, three batches of MSU SBCF tows (BC2_0028, BC2_0029, BC2_0027) were generated in the Bobcat machine. The target bath concentrations chosen were 1.5 wt.%, 10 wt.% and 25 wt.% solids. The solids content of the sizing bath was measured to ensure that these target values were achieved. The comparison between target and measured values of sizing bath concentrations are shown in Table 1, which were in close agreement.

Table 1: Description of SBCF tow batches and associated bath concentrations.

Batch no.	Tow type	Sizing type	Target bath concentration (wt.%)	Measured bath concentration (wt.%)
BC2_0028	IM7 12k SBCF	Michelman 181290IX	1.5	1.48
BC2_0029	IM7 12k SBCF	Michelman 181290IX	10	9.99
BC2_0027	IM7 12k SBCF	Michelman 181290IX	25	24.26

Tow Desizing Procedure to Quantify Sizing Application

A refined procedure to accurately measure the weight percent of sizing deposited on SBCF tows was developed. The tow was weighed before and after being completely desized using a solvent wash with laboratory grade acetone (98% purity). The steps involved in characterizing the sizing weight percent as applied to tows is shown in Figure 5.

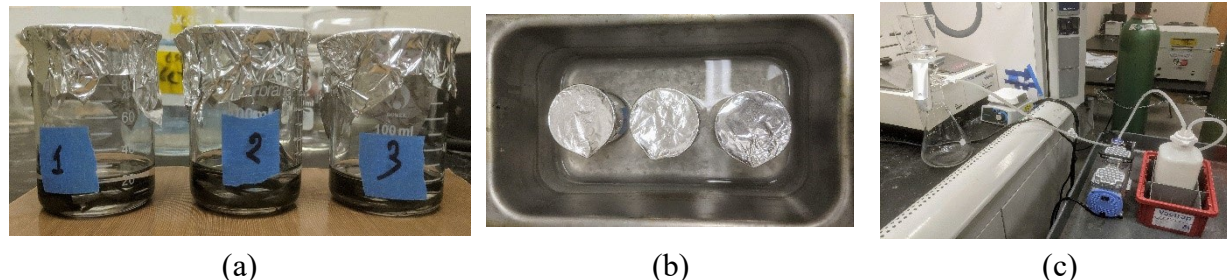


Figure 5: Steps to characterize sizing weight percent on tows: (a) Tows were dipped in 20 ml acetone solution in each beaker for 30 minutes; (b) The beakers were placed in a sonicator for 30 minutes; (c) The tows were transferred to a vacuum assisted filtration setup.

A vacuum assisted filtration setup was adopted to ensure that no fibers were lost during the filtration process. A quantitative glass filter was used having a pore diameter of $1.2\ \mu\text{m}$ (less than the fiber diameter of $5\ \mu\text{m}$). The samples were prepared by chopping 304.8 mm (12 inch) long sections from the spooled tow at random locations. Three samples were prepared for the desizing procedure.

To verify if the sizing was removed completely, three un-sized continuous IM7-12k tows having a length of 254 mm (10 inches) were weighted and sizing was applied to them. In this case, a previous generation of sizing spray booth developed by SBCF research group was used. A sizing bath of 2 wt.% was placed inside the pressure pot which is attached to an air compressor that maintains the desired pressure inside the pressure pot. The outlet pressure of the pressure pot was adjusted to 30 psi. There are two nozzles attached in the carriage. The nozzles are pointed toward each other and are perpendicular to the sample holder. The carriage is attached to translational control. The speed of the carrier with the two nozzles recorded during the spraying was 40 mm/sec. It took 6.35 seconds to size the entire length of the tow. The nozzle used in this sizing booth was a full cone misting spray nozzle. The orifice diameter was 0.01 inches with a spray angle of 80° . After the sizing application, the tows were then desized with acetone. Then

the desized tows were weighted again. The percentage change in weight for the three tows were -0.07% , -0.24% , and -0.06% , respectively. The values suggest that the complete desizing of the tows was achieved with minimal loss of fiber.

Tow Tenacity

The goal of the tow tenacity testing is to evaluate the ability of the SBCF tows to hold back-tension and their potential formability prior to resin infusion. Tensile test coupons were prepared from single tows of each batch to evaluate tow tenacity. For all tensile tests, a Mark 10 mechanical test unit was used. A temperature-controlled tow tenacity setup was used for elevated (forming) temperature evaluation (see below). The gauge length of the test samples was 152.4 mm (6 inches) for all the samples, well above the sizes measured for the fiber length distribution. The crosshead speed during the test was 2.54 mm/min. Five test coupons were prepared from each batch for each temperature condition (room temperature, 100°C : chosen to be consistent with ongoing forming tests). Control materials, SBCF without sizing, were also tested. A total of 40 test coupons were prepared: 20 for the room temperature and 20 for the elevated temperature tow tenacity tests.

Temperature-Controlled Tow Tenacity Test Setup

To ensure a uniform temperature profile along the gauge length of the tow during the elevated tensile tests, the Mark 10 was reconfigured from the standard vertical position to a horizontal position to allow more uniform heating of the tow. Direct heating via conduction was achieved using a hot plate with an aluminum plate in contact with the tow being tested. A Teflon™ sheet was placed on the aluminum plate to prevent sample sticking, particularly

necessary at elevated temperatures. The elevated temperature test setup is shown in Figure 6. The setup applies heat to a 127 mm section of the sample gauge length. A gap of 12.7 mm was maintained at 100°C. A test coupon of Hexcel SBCF tow having a gauge length of 152.4 mm was used. When the hot plate was turned on, the target temperature on the Teflon™ sheet was reached and stabilized after 5 minutes. This stabilization procedure was subsequently used for all elevated temperature tests prior to introducing the tow. The tows reached the target temperature in 1 minute. The temperature was recorded using a K-type thermocouple along the gauge section of the tow at an interval of 12.7 mm. All the readings were recorded once the temperature had come to a steady state.

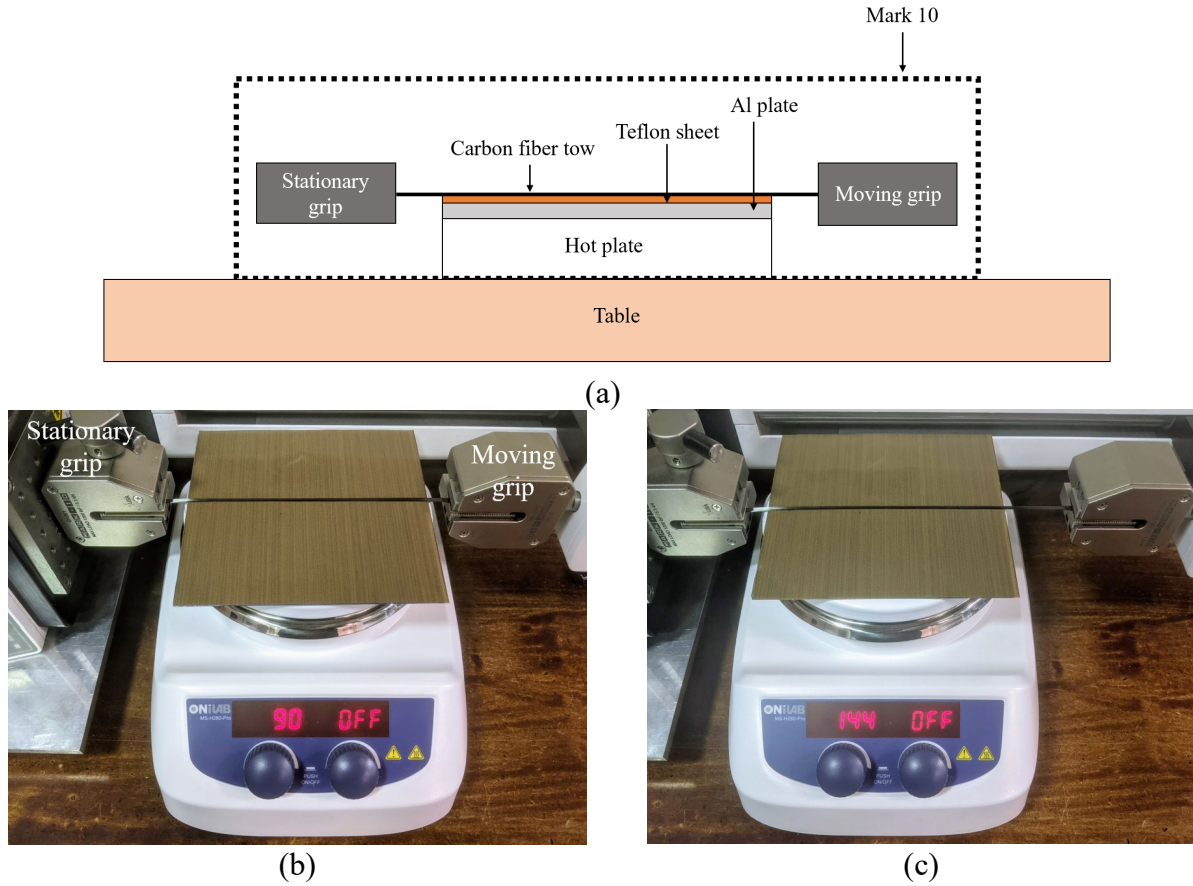


Figure 6: (a) Schematic of the elevated temperature test fixture. The horizontal Mark 10 tensile test apparatus controls the movement of the grips and recording the load-displacement data; (b) demonstration of the new horizontal test setup with a 152.4 mm (6 inches) tow touching the heated Teflon™ sheet. The gap between the heated Teflon™ and the grip at both sides was 12.7 mm (0.5 inches); (c) the tow was stretched from 152.4 mm (6 inches) to 177.8 mm (7 inches). The temperature reading given by the hot plate does not represent the true temperature of the heated tow. Therefore, a thermocouple was used to manually calibrate the temperature of the heated tow.

Evaluation of SBCF Reproducibility

To evaluate the process and sizing application reproducibility, three replicate batches of MSU SBCF were generated keeping production parameters constant. These parameters had been previously established for the Bobcat machine to successfully generate SBCF tows. The

description of these batches is presented in Table 2. In the results section, Hexcel SBCF is also included as a comparison for the MSU SBCF in terms of sizing deposition and tow tenacity.

Table 2: Description of materials for the evaluation of SBCF reproducibility

Batch No.	Tow type	Sizing	Target bath concentration (wt.%)	Measured Bath concentration (wt.%)
BC2_0033	IM7 12k SBCF	Un-sized (control); Sprayed DI water during manufacture	0	0
BC2_0030	IM7 12k SBCF	Michelman 181290IX	10	9.99
BC2_0031	IM7 12k SBCF	Michelman 181290IX	10	9.99
BC2_0032	IM7 12k SBCF	Michelman 181290IX	10	9.99
Hexcel SBCF	IM7 12k SBCF	Hexcel GP	unknown	unknown

Evaluation of Reproducibility of Sizing Deposition

Three batches of MSU SBCF tows were produced using the same sizing bath concentration. These batches are BC2_0030, BC2_0031 and BC2_0032. The control (BC2_0033) and the Hexcel SBCF were also included for comparison. The procedures to determine sizing bath concentration and the sizing deposited on the tows were used to verify both of these parameters for each batch.

Evaluation of Reproducibility of Tow Tenacity

To evaluate the consistency of the tow tenacity measurements, tensile test coupons were prepared for evaluation at both room and elevated temperature (100 °C) using the same procedure as mentioned in the ‘Tow tenacity’ section. A total of 50 samples were tested for this

experiment; 25 samples were tested at room temperature and 25 samples at elevated temperature. The elevated temperature during the test was 100°C.

Evaluation of Reproducibility of Tow Tenacity at Intermediate Temperatures Along with the tow tenacity of single tow MSU SBCF being tested at room temperature and at 100°C, the consistency of tow tenacity was also evaluated at the intermediate temperatures of 40°C, 50°C, 60°C, and 70°C. Besides evaluating the tow tenacity consistency at intermediate temperatures, this experiment also provides a preliminary insight into determining a suitable temperature regime to apply back-tension of various magnitude during the prepreg production. Five tensile test coupons were prepared for each test temperature. A total of 60 samples were tested.

Evaluation of Reproducibility of Fiber Length Distribution and Mean Fiber Length

An analysis of the fiber length distribution and the mean fiber length of the MSU SBCF generated from the Bobcat machine was performed using ASTM D 5103 “Standard Test Method for Length and Length Distribution of Manufactured Staple Fibers (Single-Fiber Test)”. Fifty individual filaments were extracted and measured by hand from each SBCF tow sample, in batches of ten so that the sampling could be randomized by pulling the fibers from different portions of the sample. The measured lengths were then plotted both as a function of relative frequency and as a function of frequency of continuity. Relative frequency visualizes the distribution of a given length of fibers as a percentage with respect to the entire pool of fibers. Relative frequency is plotted in the form of histogram. After trying several combinations of bin width and number of bins, a bin width of 3 mm was selected. A bin width of 3 mm was chosen to resolve adequate resolution of the distribution of fiber length in the histogram. Frequency of continuity is a measure of the percentage of the dataset greater than a given value. For example,

in the context of fiber length, 100% frequency of continuity means that the length of all of the fibers from a pool of fibers is greater than the minimum length of fiber from that pool. 0% frequency of continuity means that none of the fibers are greater than the maximum length of fiber from that pool of fibers. Relative frequency is presented in the form of S-curve.

The fiber length distribution was further evaluated by identifying the tail regions of the S-curve by identifying two tail regions. In the S-curve data of frequency of continuity vs. fiber length, two tail regions were defined; an upper tail region where the frequency of continuity ranges from 90%-100% and a lower tail region having frequency of continuity ranging from 10%-0%.

Preliminary Evaluation of the Effect of Stretch Ratio

To evaluate stretch ratio, three batches of single tow MSU SBCF were generated in the Bobcat machine. The sizing bath was prepared using Michelman 181290IX. Verification of the sizing bath concentration was performed to ensure the target bath concentration of 10 wt.% (and resultant deposition of sizing on the tow of ~2%) was achieved. The measured average bath concentration was 10.03 wt.% (standard deviation: 0.01; coefficient of variation: 0.14%). The nip force of 10676 N and the sizing flow rate of 0.6 ml/min were not changed. The stretch ratios chosen were 15%, 20%, and 25%. A summary of the run conditions is shown in Table 3.

Table 3: Description of the materials for the evaluation of the effect of stretch ratio on the MSU SBCF production.

Batch no.	Stretch ratio (%)	Nip force (N)	Sizing flow rate (ml/min)	Target bath concentration (wt.%)
BC2 0043	15	8007	0.6	10
BC2 0039	20	8007	0.6	10
BC2 0038	25	8007	0.6	10

The MSU SBCF generated by varying the stretch ratio was evaluated in terms of sizing deposition on the tow, tow tenacity, fiber length distribution, and mean fiber length.

During the sizing deposition characterization, both the amount of sizing deposited on the tow and the unit mass of the tow were measured. The unit mass of the desized tow was calculated by dividing the mass of the tow by its length (304.8 mm). The average sizing deposition on the tow was then divided by the unit mass of the desized tow to obtain the average sizing deposition per unit mass of the tow, which was plotted as a function of stretch ratio.

Analysis of Variance (ANOVA) of the mean fiber length as a function of stretch ratio was performed to determine if the mean fiber lengths are statistically significant when the stretch ratio was increased from 15% to 25%. The result obtained was used to track the significant change in mean fiber length in the 15-25% stretch ratio regime. ANOVA of raw data of fiber length was performed in Origin Pro (OriginLab Corporation, Northampton, MA, USA) software. The significance level was set to 0.05. A p-value (probability) of less than 0.05 means the result is statistically significant. If the p-value is greater than 0.05, then the result is not statistically significant. However, p-value only gives information about the statistically significant result in a process. It cannot identify the regimes at which the results are statistically significant. Therefore, the comparison of mean was performed using Tukey method to accurately identify the regimes where statistically significant results are observed. The sig equals 0 obtained from the Tukey method indicates that the differences of means is not significant at 0.05 significance level. The sig equals 1 indicates that the differences of means is significant at 0.05 significance level. The procedure for the ANOVA described for the stretch ratio experiment was also adopted for the nip force experiment.

For the tow tenacity characterization, ten samples were prepared from each stretch ratio materials; five samples were tested at room temperature and five samples were tested at elevated temperature (100°C). Overall, 30 samples were prepared and tested for the evaluation of the effect of stretch ratio on the SBCF production. The average peak load obtained from the tow tenacity experiment was divided by the unit mass of the tow to obtain average peak load per unit mass and plotted as a function of stretch ratio.

Preliminary Evaluation of the Effect of Nip Force

To evaluate the effect of nip force on tow tenacity, five batches of MSU SBCF were generated and sized with a 10 wt.% bath of Michelman 181290IX. The stretch ratio of 25% was maintained and the volumetric flow rate of sizing was 0.6 ml/min. The nip force was changed from 8007 N (1800 lbf) to 11565 N (2600 lbf). The reason for choosing this range is that the polyurethane nip rollers in the Bobcat machine can apply nip force in this range without causing any interruption and damage. The description of the materials for the nip force experiment is shown in Table 4.

Table 4: Description of the materials for the evaluation of the effect of nip force

Batch no.	Stretch ratio (%)	Nip force (N)	Sizing flow rate (ml/min)	Sizing bath concentration (wt.%)
BC2_0055	25	8007 (1800 lbf)	0.6	10
BC2_0054	25	8896 (2000 lbf)	0.6	10
BC2_0049	25	9786 (2200 lbf)	0.6	10
BC2_0046	25	10676 N (2400 lbf)	0.6	10
BC2_0050	25	11565 N (2600 lbf)	0.6	10

The MSU SBCF generated by varying the stretch ratio will be evaluated in terms of sizing deposition on the tow, tow tenacity, fiber length distribution, and mean fiber length.

During the sizing deposition characterization, both the amount of sizing deposited on the tow and the unit mass of the tow were measured. The unit mass of the desized tow was calculated by dividing the mass of the tow by its length (304.8 mm). The average sizing deposition on the tow was then divided by the unit mass of the desized tow to obtain the average sizing deposition per unit mass of the tow, which was plotted as a function of nip force.

Analysis of Variance (ANOVA) of the mean fiber length as a function of nip force was performed to determine if the mean fiber lengths are statistically significant when the nip force was increased from 8007 N to 11565 N.

For the tow tenacity characterization, ten samples were prepared from each batch of single tow MSU SBCF produced by varying the nip force from 8007-11565 N; five samples were tested at room temperature and five samples were tested at elevated temperature (100°C). Overall, 50 samples were prepared and tested for the evaluation of the effect of stretch ratio on the SBCF production. The average peak load was divided by the unit mass of the tow to obtain the average sizing deposition per unit mass.

RESULTS AND DISCUSSIONS

Sizing Deposition as a Function of Bath Concentration

The measured amount of sizing applied to the SBCF tows for each initial sizing bath concentration that was evaluated is shown in Figure 7.

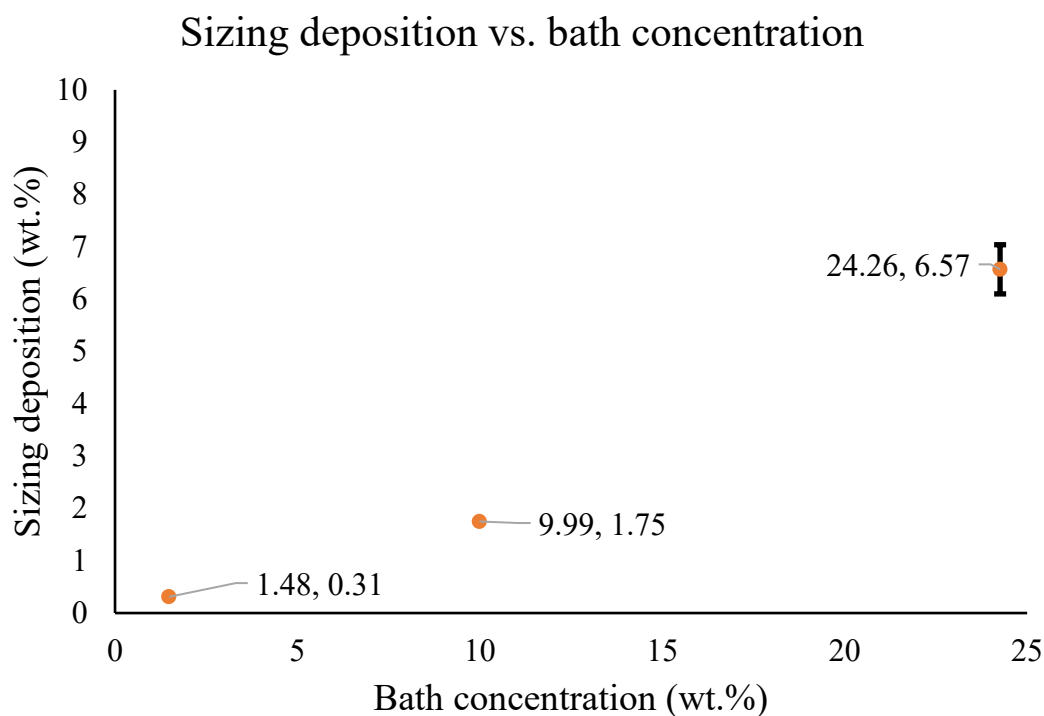


Figure 7: Resultant sizing deposition on tow vs. measured bath concentration; each point is an average of three samples from each batch of SBCF material.

For the bath concentrations of 1.48 wt.%, 9.99 wt.% and 24.26 wt.%, the average (standard deviation) sizing deposition was 0.31 wt.% (0.02%), 1.75 wt.% (0.01%) and 6.57 wt.% (0.47%), respectively. A bath concentration of approximately 10 wt. % solids result in a final sizing application on the SBCF tows in the target range of ~1.5wt. %-2.0 wt.% solids. This target

bath concentration was kept consistent for future tests on process repeatability based on these and the tow tenacity results.

Validation of Elevated Temperature Test Fixture for Tow Tenacity Tensile Test

The temperature profile of the heated tows along the gauge section of the tow at an interval of 12.7 mm is shown in Figure 8. Each point in the graph is an average value of 10 temperature readings with a standard deviation of 0.1 to 0.3. The temperature in the middle section of the tow (38.1 mm to 88.9 mm) is in good agreement with the target temperature. At the edges however, the temperature was 4°C to 5°C lower than middle section. The fiber distribution data combined with a temperature-tow tenacity sensitivity analysis indicate that this temperature drop within the last 30 mm of the tow on each side falls within an acceptable range and should not significantly affect the tow tenacity results.

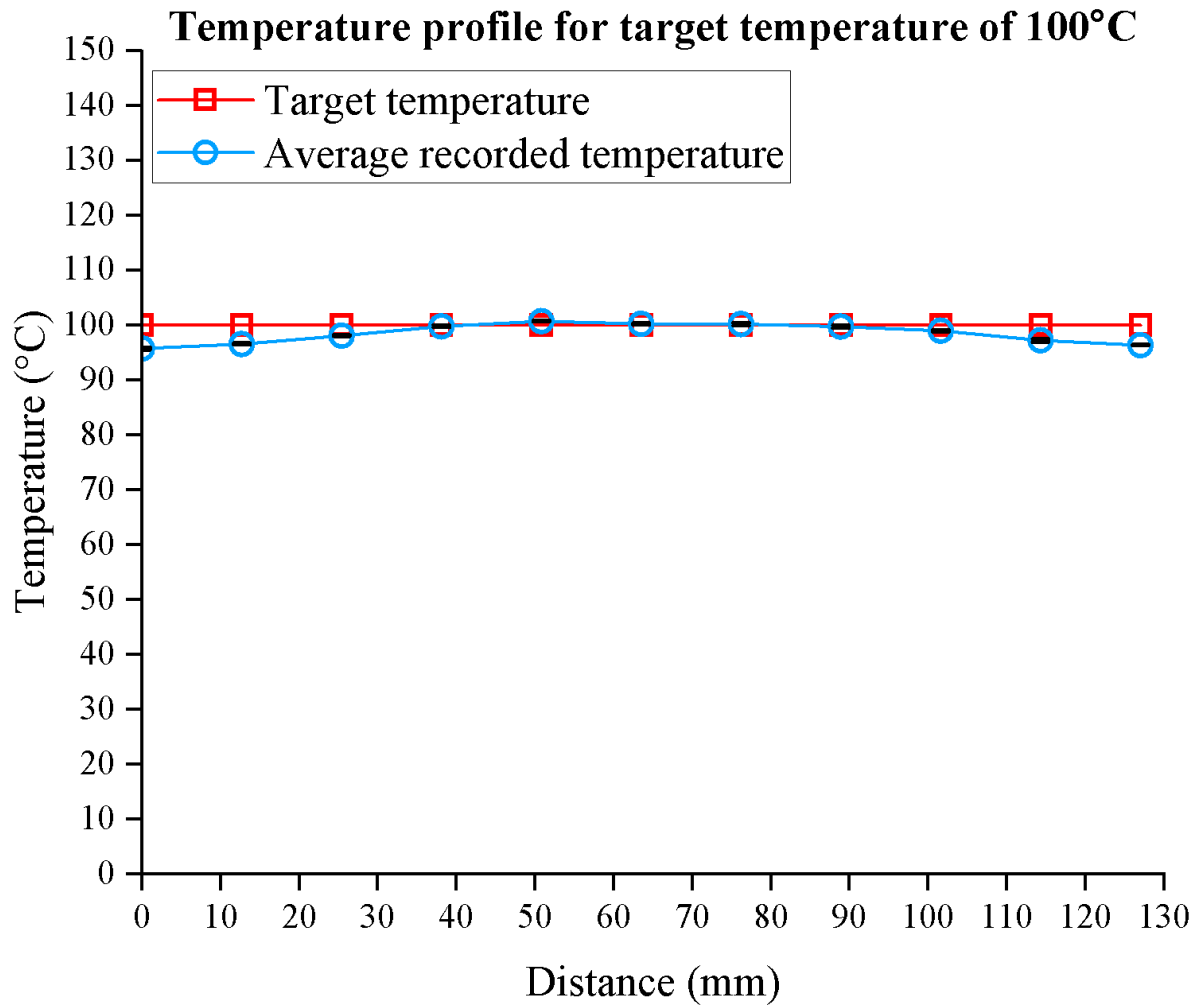


Figure 8: Temperature profile for the target temperature of 100°C. The red squares indicate the target temperature. The blue circles indicate the average (ten temperature measurements for each point) recorded temperature along the tow.

Tow Tenacity with Sizing Application

The peak loads obtained from the tensile tests were plotted as a function of sizing deposition on the tow in Figure 9.

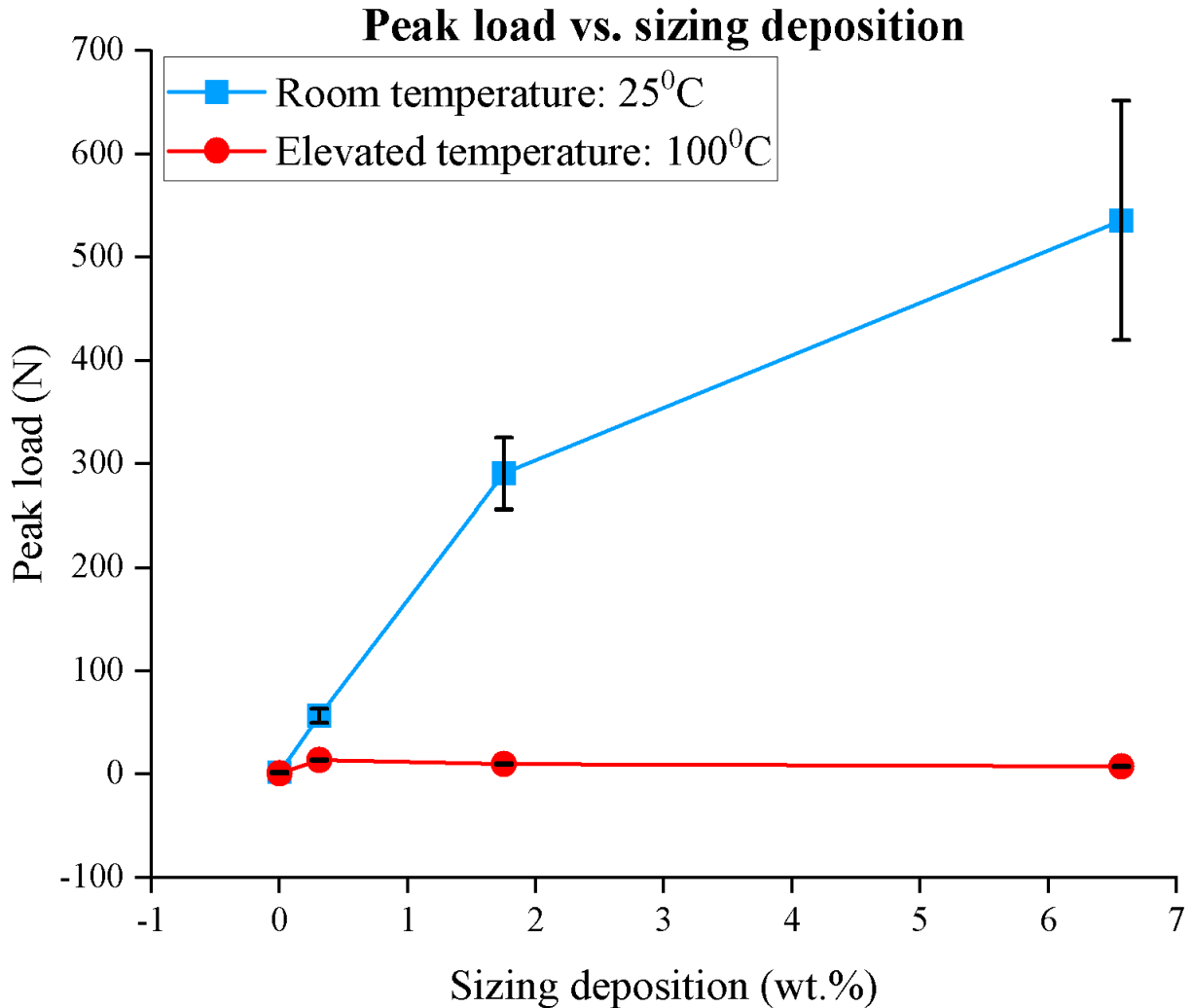


Figure 9: Relationship between the peak load obtained from the load-displacement graphs vs. sizing deposition. The room temperature results (red, square) demonstrate the back-tension ability of the MSU SBCF tows. The elevated temperature results (100°C, blue, circle) indicate tow formability. Each point in the graph is an average of five samples; error bars represent the standard deviation and may lie within the data marker.

When there is no sizing on the tow (control), the peak load measured at room temperature is 1.77 (0.61) N. The lack of fiber continuity and binding between the fibers from the sizing leads to these low values, and resultant challenges in tow handling. Sizing application on SBCF at ~1.75 wt. % can achieve the desired peak loads linked with handleability (at 1.75 wt. %, the average peak load is 290.86 (34.57) N). Sizing application in conjunction with the stretch

breaking process in the Bobcat Machine has been optimized through multiple trials. This result demonstrates a peak load comparable to that of the Hexcel legacy material obtained in the SBCF tows generated in the Bobcat machine while having sizing within the target application window of less than 2 wt.%.

At elevated temperature (100°C), the peak load decreases with an increase in the sizing deposition. This behavior is hypothesized to be due to softening of sizing, reducing the overall viscosity of the sized fibers during extension in the Mark 10. The control, which has no sizing, also shows a lower peak load of 0.92 (0.14) N when compared to the room temperature peak load of 1.77 (0.61) N. A possible explanation for the lower peak load here is changes to the friction at the tow-hot plate interface. The lower peak loads at 100°C support the potential for room-temperature handling with elevated temperature forming of the MSU SBCF. In addition to this indication of formability, various test methods are also being explored at MSU to evaluate the formability of SBCF, which include the plunger test, ply-on-ply and ply-on-tool friction test, dome formability test, and true stress-strain measurements [20]–[23]. Combined with the tow tenacity tests, a full picture of SBCF formability is ongoing. Based on these results, the 10 wt.% bath concentration was chosen to be used for the sizing deposition on the next batches of MSU SBCF tows. The load displacement curves for the room temperature and elevated temperature (100°C) tow tenacity tests are shown in Figure 10 and Figure 11 respectively.

Comparison of tow tenacity at room temperature

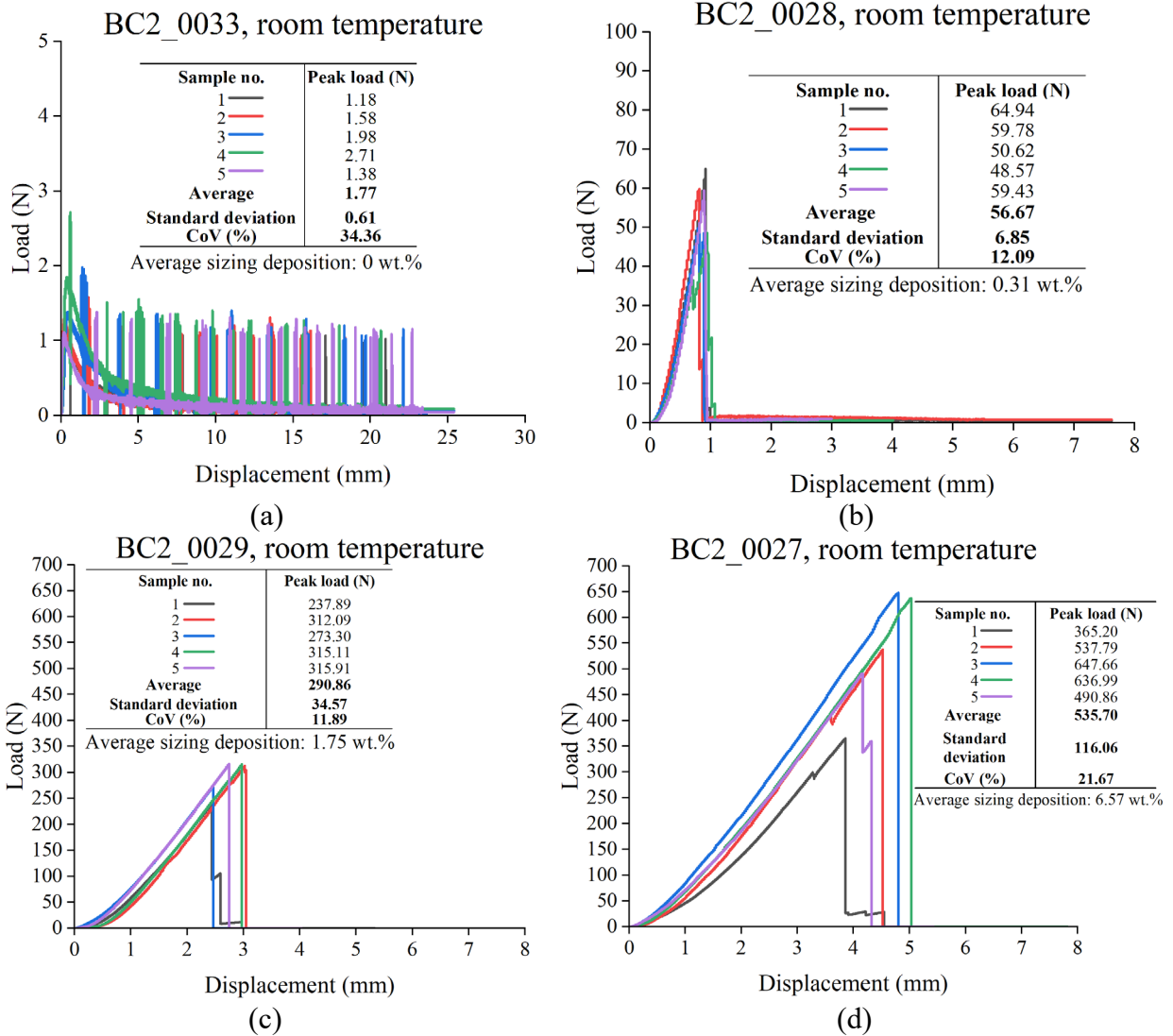


Figure 10: Load-displacement graph of the samples tested at room temperature from batches (a) BC2_0033 (control with no sizing applied to it); (b) BC2_0028 (average sizing deposition: 0.31 wt.%); (c) BC2_0029 (average sizing deposition: 1.75 wt.%) and (d) BC2_0027 (average sizing deposition: 6.57 wt.%). Five test coupons were prepared and tested from each batch. The average peak load, standard deviation and coefficient of variation are also included.

Comparison tow tenacity at 100°C

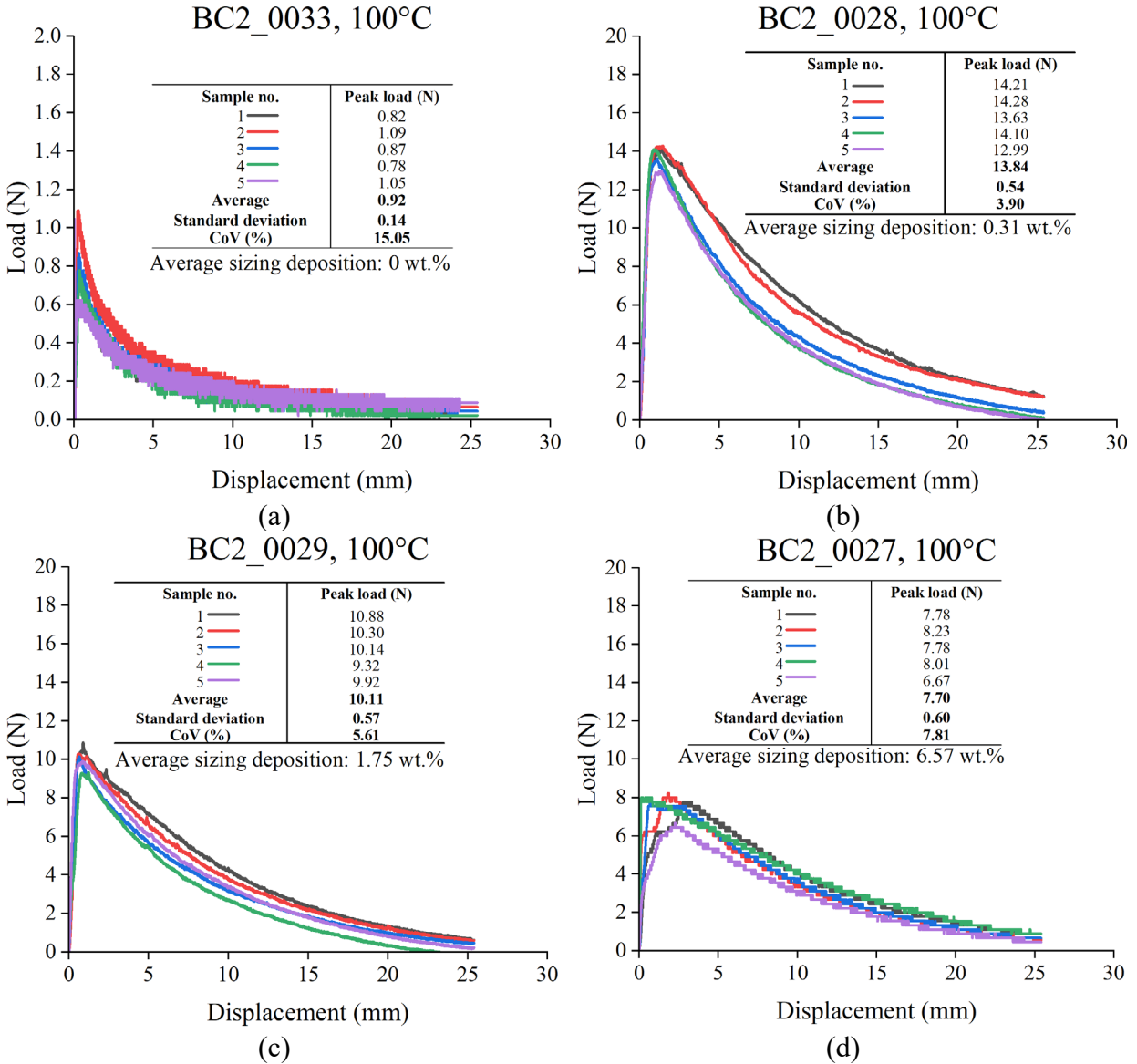


Figure 11: Load-displacement graph of the samples tested at elevated temperature (100°C) from batches (a) BC2_0033 (control with no sizing applied to it); (b) BC2_0028 (average sizing deposition: 0.31 wt.%); (c) BC2_0029 (average sizing deposition: 1.75 wt.%) and (d) BC2_0027 (average sizing deposition: 6.57 wt.%). Five test coupons were prepared and tested from each batch. The average peak load, standard deviation and coefficient of variation are also included.

Tensile test coupons of single tow sized MSU SBCF pre-tensile test and post-tensile test at room temperature are shown in Figure 12.

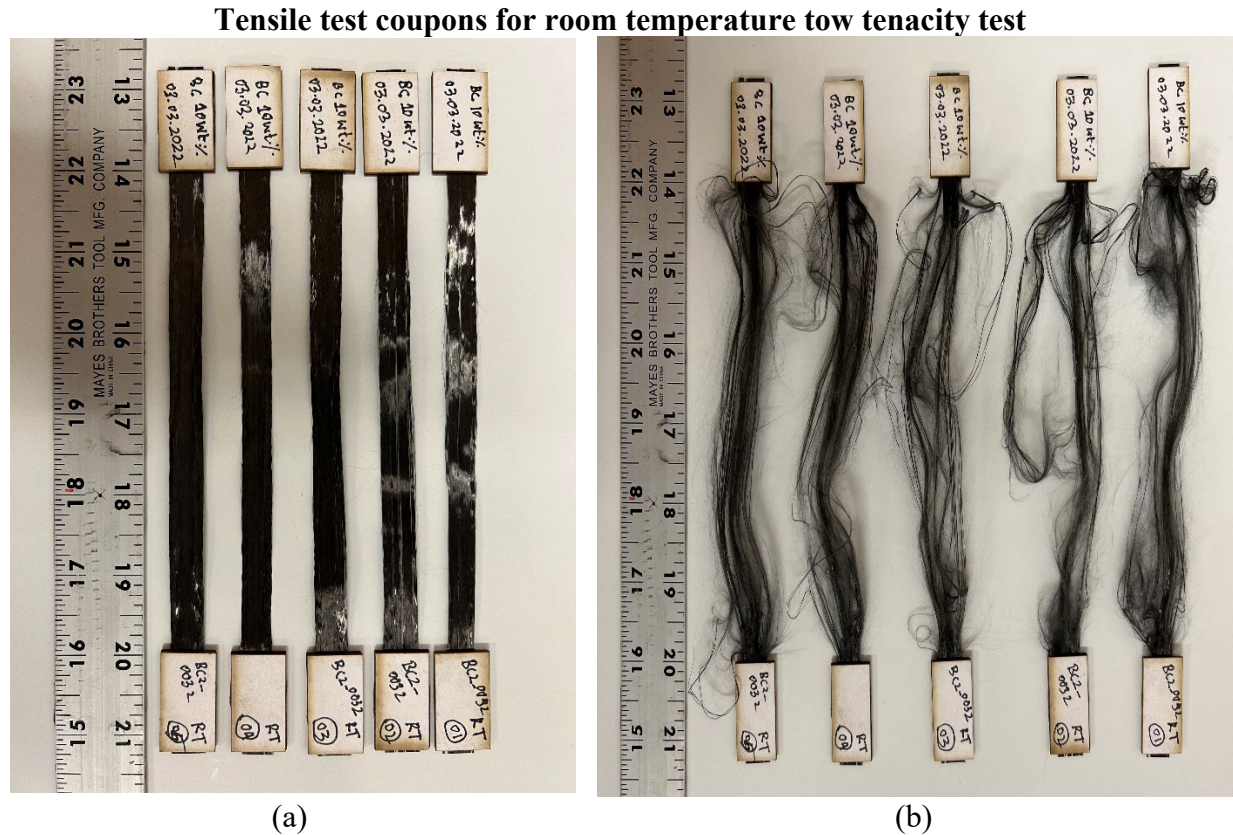


Figure 12: Tensile test coupons prepared from single tow MSU SBCF for the room temperature tow tenacity experiment. (a); pre-tensile test. (b) post-tensile test. The tow failed in a brittle manner under applied tension since sizing acts as a solid binder at room temperature.

Tensile test coupons of single tow sized MSU SBCF pre-tensile test and post-tensile test at elevated temperature (100°C) are shown in Figure 13.

Tensile test coupons for room temperature tow tenacity test

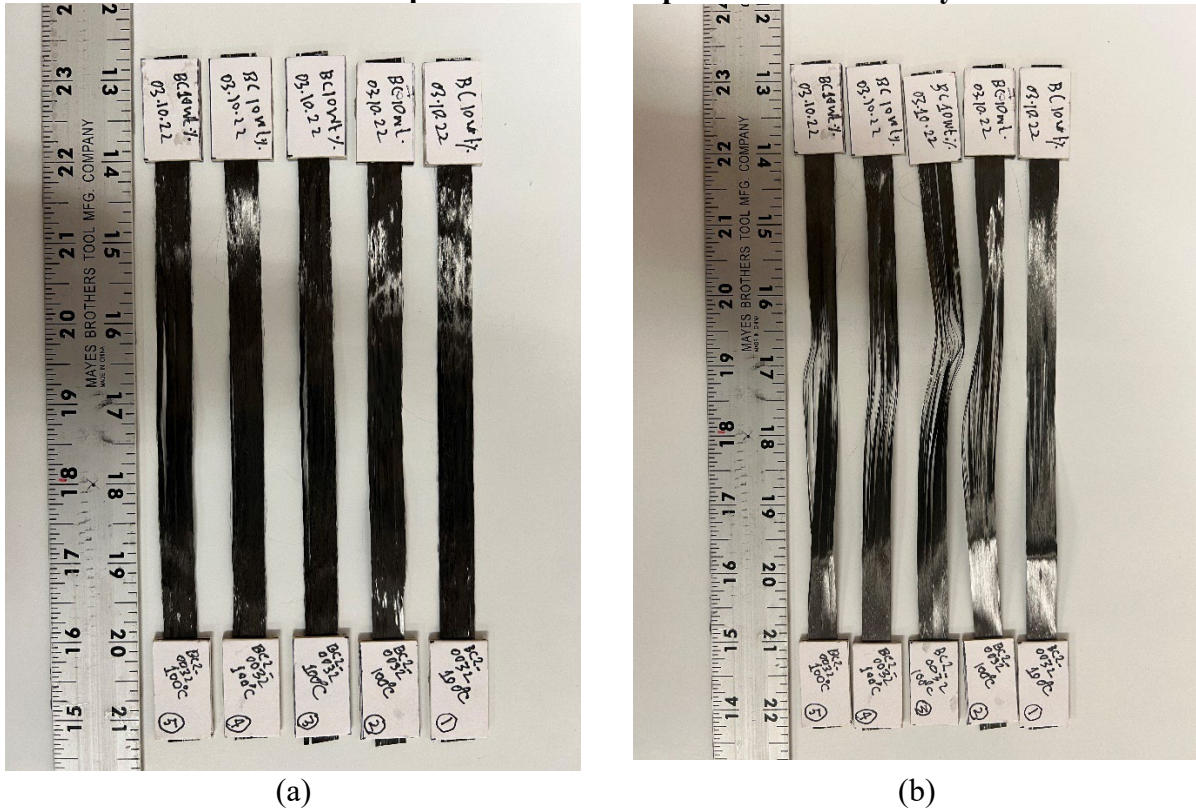


Figure 13: Tensile test coupons prepared from single tow MSU SBCF for the elevated temperature (100°C) tow tenacity experiment. (a); pre-tensile test. (b) post-tensile test. The tow was stretched from 152.4 (6 inches) mm to 177.8 mm (7 inches) during the elevated temperature tensile test. The decrease in viscosity of the sizing at elevated temperature provided a lubricating effect to the fibers in a tow, which facilitates fiber-fiber sliding during the tow stretching.

Evaluation of SBCF Reproducibility

Sizing Deposition

The results showing repeatability in sizing deposition on the tows for three batches from a 10 wt.% sizing bath are presented in Figure 14.

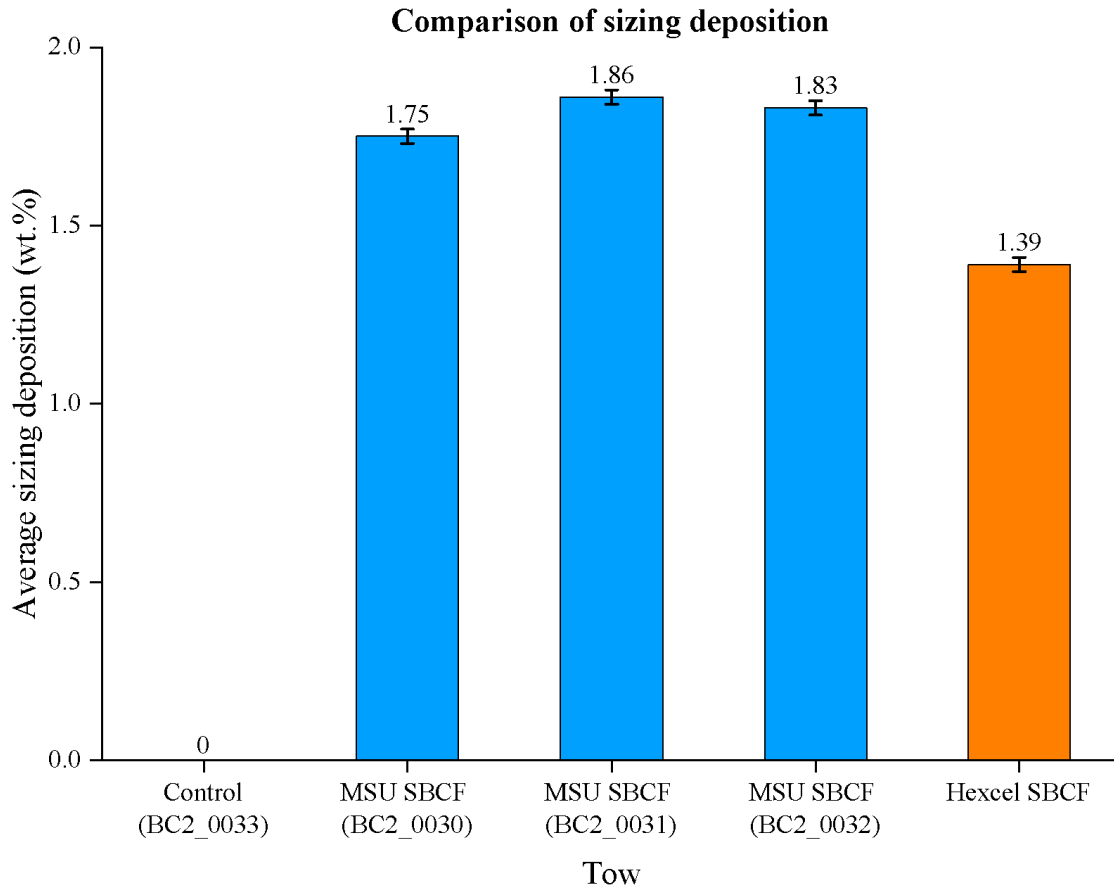


Figure 14: Comparison of sizing deposition among the samples prepared from BC2_0030, BC2_0031, BC2_0032. BC2_0033 is the control with no sizing applied to it. The results for the Hexcel SBCF are also included. The values in the figure are an average of three samples.

The average sizing deposition for process replicates BC2_0030, BC2_0031 and BC2_0032 is 1.75 wt.%, 1.86 wt.% and 1.83 wt.%, respectively. A slight variation in the sizing deposition is observed. Given that the materials from the three batches were generated with

identical parameters, this variation may be statistically significant between the BC2_0030 and other batches. Over the three batches, the sizing application on the tows is repeatable. The sizing deposition on the Hexcel SBCF tow was lower, 1.39 wt.%.

Tow Tenacity

The tow tenacity results for three replicate batches of single tow MSU SBCF at room temperature is shown in Figure 15.

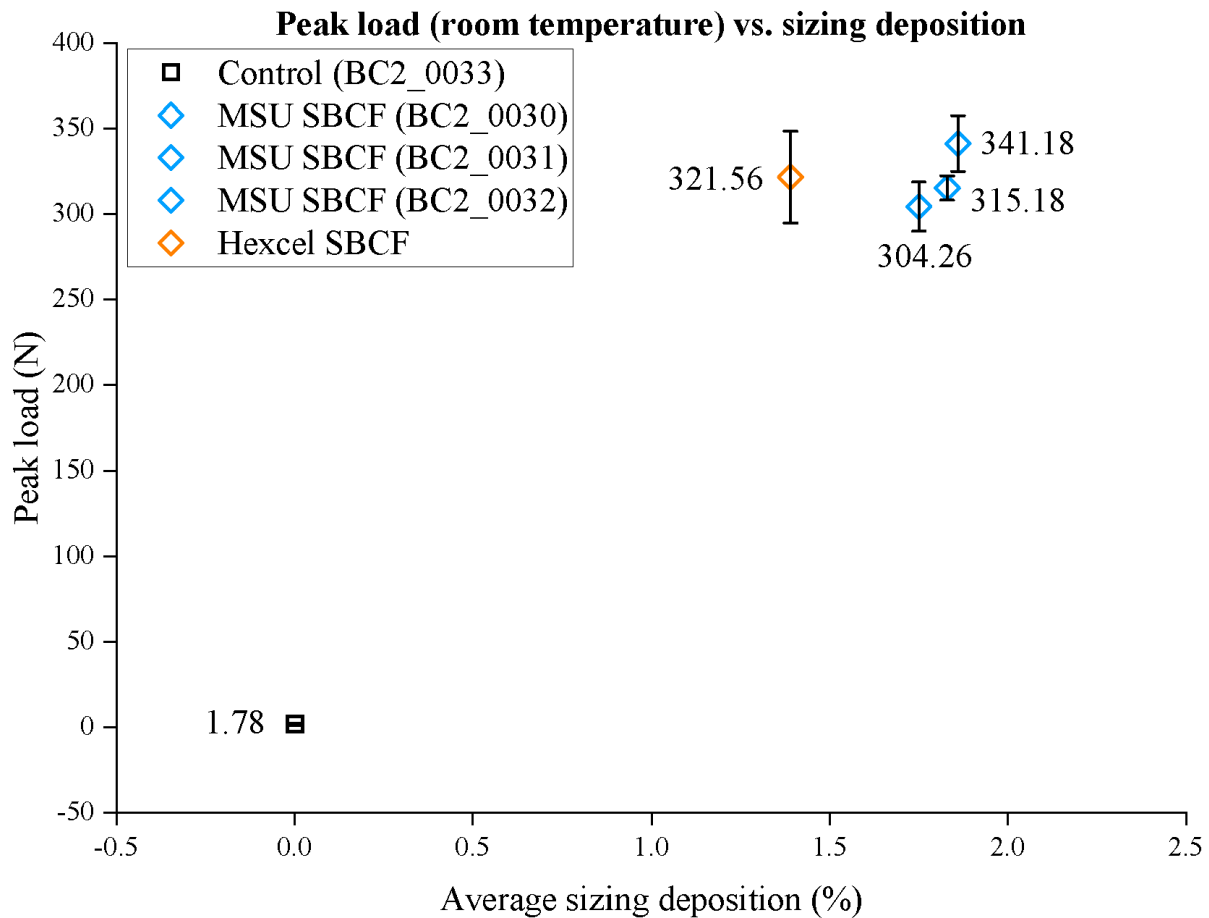


Figure 15: Room temperature peak load as a function of sizing deposition. Each point is an average value of five samples. BC2_0033 is the control which has no sizing applied to it (DI water was sprayed during the production). A bath concentration of 10 wt.% was used during the production of three replicate batches of MSU SBCF (BC2_0030, BC2_0031, and BC2_0032), which resulted in the average sizing deposition of 1.75 wt.%, 1.86 wt.%, and 1.83 wt.%, respectively.

The test coupons prepared from BC2_0033, which is the control showed a very low peak load of 1.77 (0.61) N, as expected due to the absence of sizing. For the test coupons prepared from BC2_0030, BC2_0031, and BC2_0032 the peak loads are 304.26 (14.37) N, 341.18 (16.32) N and 315.18 (7.16) N, respectively (values in the parenthesis denote standard deviation). These values are repeatable and comparable to the 321.56 (26.91) N for the legacy Hexcel SBCF materials. The values of the standard deviation are lower for the MSU SBCF, indicating less variability in sizing application.

The elevated temperature (100°C) tow tenacity results are presented in Figure 16.

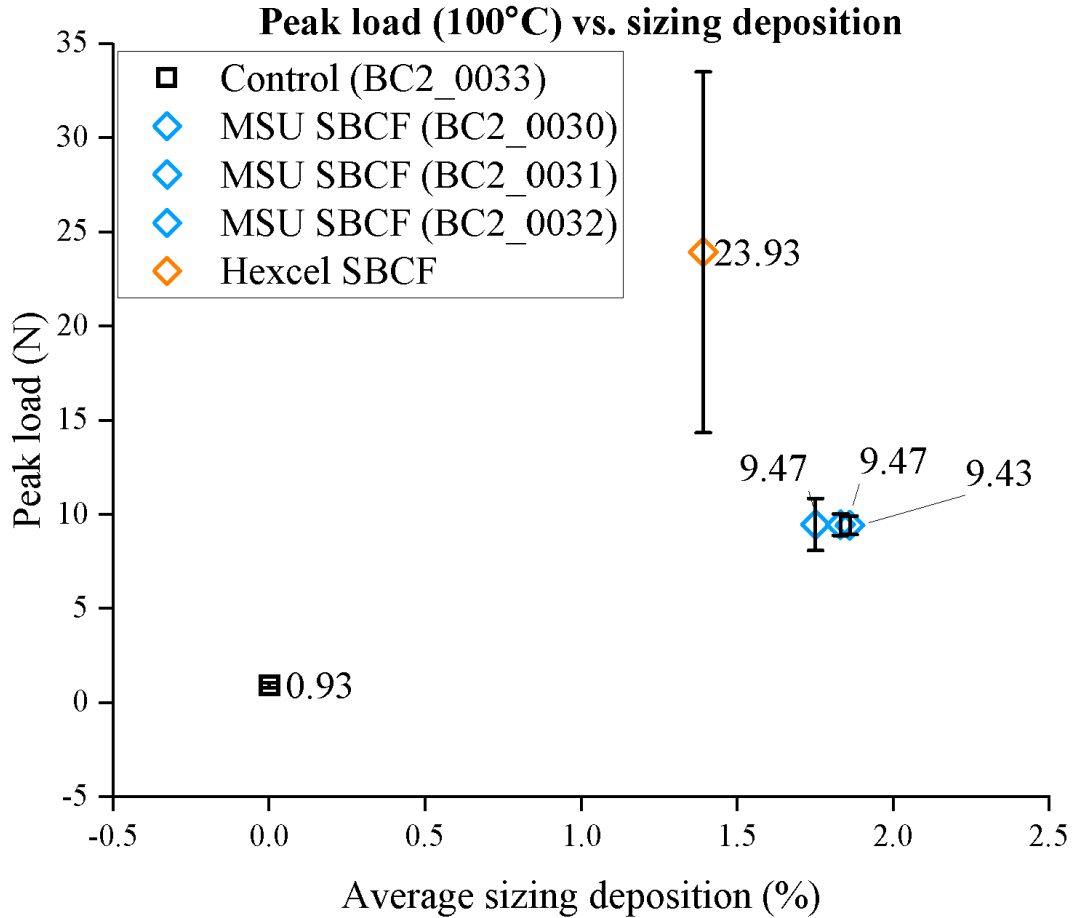


Figure 16: Elevated temperature peak load as a function of sizing deposition. The testing temperature was 100°C. Each point is an average value of five readings.

The control showed a very low peak load of 0.92 (0.14) N. Since the elevated temperature tenacity test was performed by placing the samples on a hot plate, the friction between the surface of the hot plate and the tow surface contributes to the tenacity measured at room and elevated temperatures and is potentially temperature dependent. The test coupons from the three batches (BC2_0030, BC2_0031 and BC2_0032) showed an average peak load of 9.47 (0.38) N, 9.43 (0.49) N and 9.47 (0.58) N, respectively. These results show very repeatable

elevated temperature tenacity in the MSU SBCF, a promising result for predicting formability.

Hexcel SBCF shows the relatively higher peak load of 23.93 (9.58) N.

Evaluation of SBCF Reproducibility: Tow Tenacity at Intermediate Temperatures Along with room temperature and 100°C, the peak load of two replicate single tow MSU SBCF batches (BC2_0032, BC2_0034) obtained at 40°C, 50°C, 60°C, and 70°C are also presented in Figure 17.

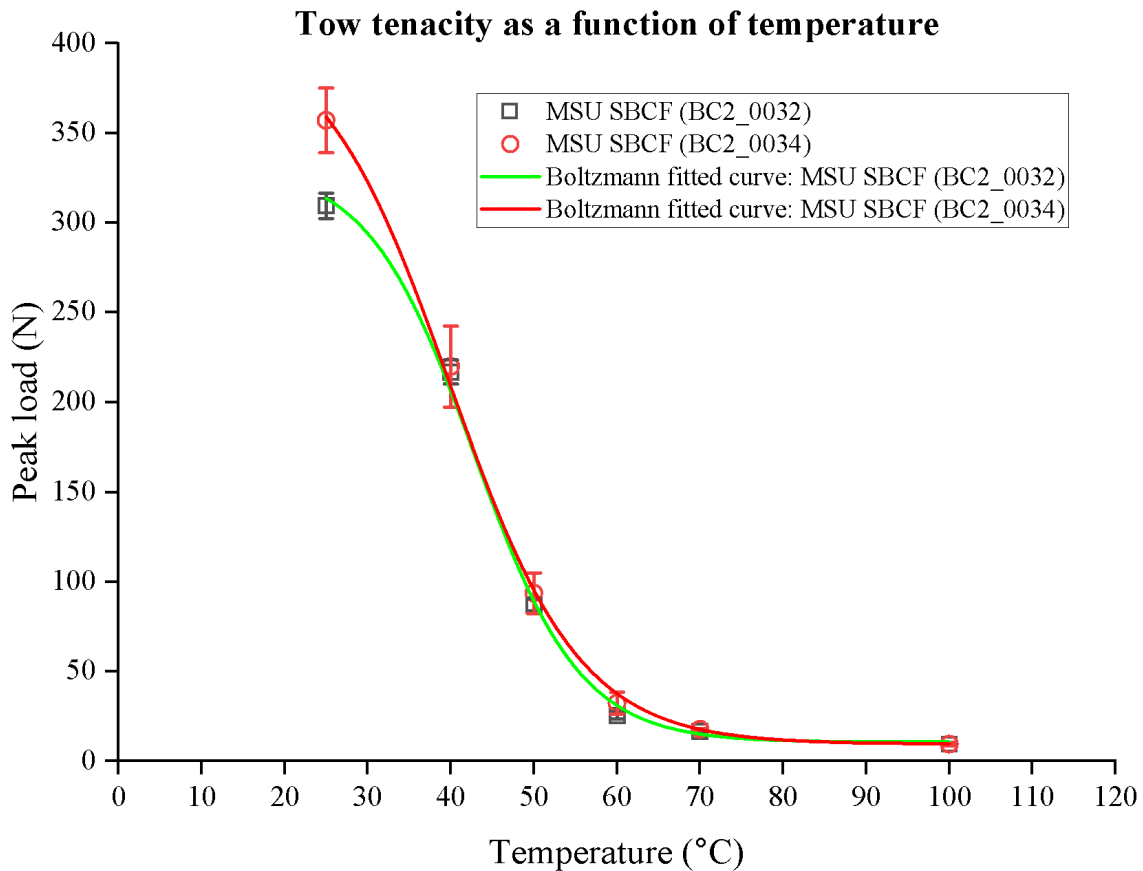


Figure 17: Peak load of single tow MSU SBCF plotted as a function of temperature. The test temperatures were 25°C, 40°C, 50°C, 60°C, 70°C, and 100°C. Each point in the graph is an average value of five readings. Boltzmann fitted curves were also plotted.

A declining trend of peak load from approximately 350N to 30N was observed from 25°C to 60°C. The flow of sizing possibly initiated in this temperature regime leading to the

declining trend. This peak load regime suggest that the back-tension can potentially be applied in this temperature regime without negatively affecting the mechanical performance of the MSU SBCF. From 60°C- 100°C, the peak loads were very low and did not change significantly. Both replicate batches of single tow MSU SBCF showed comparable values of peak loads, which suggests the reproducibility of this result.

Fiber Length Distribution and Mean Fiber Length

The result of the fiber length distribution for each of the replicate batches of single tow MSU SBCF is shown in Figure 18.

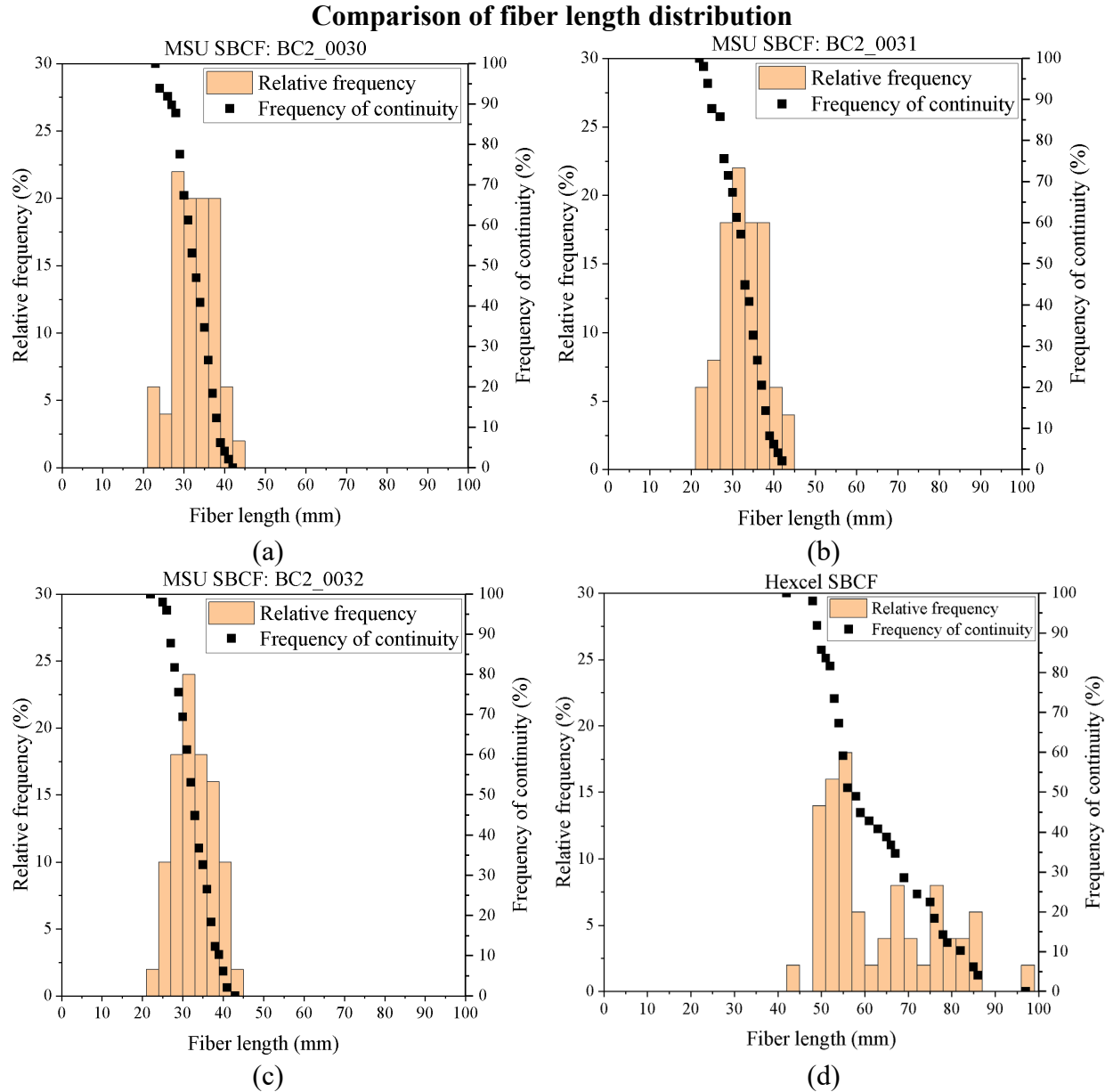


Figure 18: Comparison of fiber length distribution among the three MSU SBCF replicate batches (a) BC2_0030; (b); BC2_0031 (c) BC2_0032; and (d) the Hexcel legacy SBCF.

The fiber length distribution from BC2_0030, BC2_0031, and BC2_0032 are similar in the observed range of fiber lengths. From the plot of relative frequency vs length, BC2_0030 shows the range from 23 mm to 42 mm. For BC2_0031, the length ranges from 22 mm to 42 mm. For BC2_0032, the length ranges from 22 mm to 43 mm. As for the Hexcel SBCF, the fiber length distribution shows a greater spread compared to the MSU SBCF tows (range is 42 mm to 97 mm). The plots of frequency of continuity vs length (scattered black squares) show that the curve for all three MSU SBCF batches is shifted to the left, indicating a narrower fiber length distribution compared to the Hexcel SBCF tows. MSU SBCF shows consistent fiber length distributions for all three batches with a narrower distribution as compared to the Hexcel SBCF.

The plot of fiber length distribution in which both the upper tail and lower tail regions are identified are presented in Figure 19.

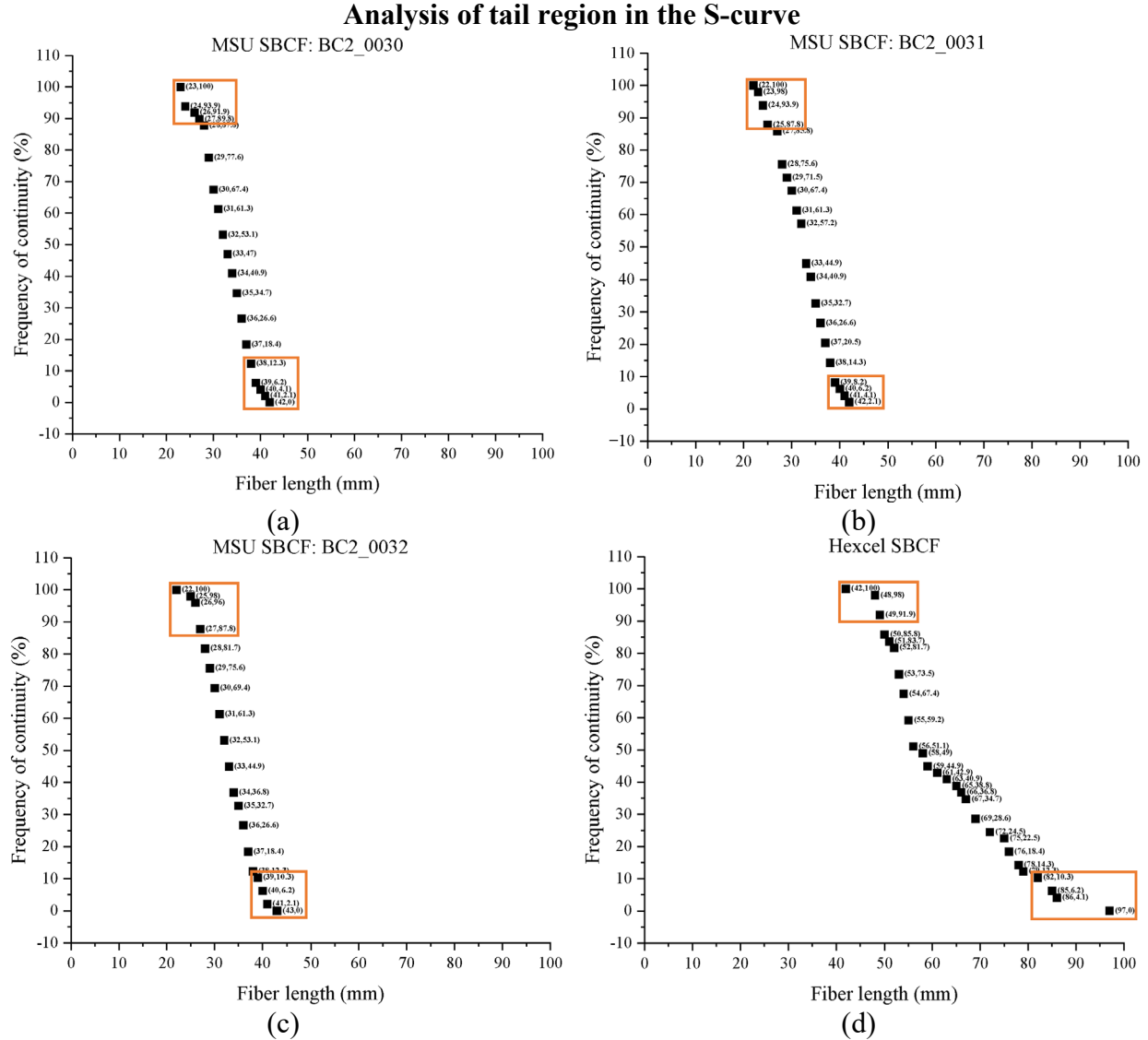


Figure 19: Fiber length distribution plotted as frequency of continuity vs. fiber length for (a) BC2_0030; (b) BC2_0031; (c) BC2_0032; (d) Hexcel SBCF.

For the MSU SBCF materials (BC2_0030, BC2_0031, and BC2_0032), when the frequency of continuity ranges from 87%-100%, the fiber length range is approximately 27mm to 22 mm. When the frequency of continuity ranges from 12%-0%, the range of fiber length is

approximately 38 mm to 43 mm. These tail values suggest that the MSU SBCF generated from the Bobcat machine with the same parameters generates a consistent fiber length distribution. Also as previously noted, compared to MSU SBCF, Hexcel SBCF contains noticeably longer fibers in both the upper and lower tail region.

A comparison of mean fiber length among the replicate batches of MSU SBCF and Hexcel SBCF is shown in Figure 20.

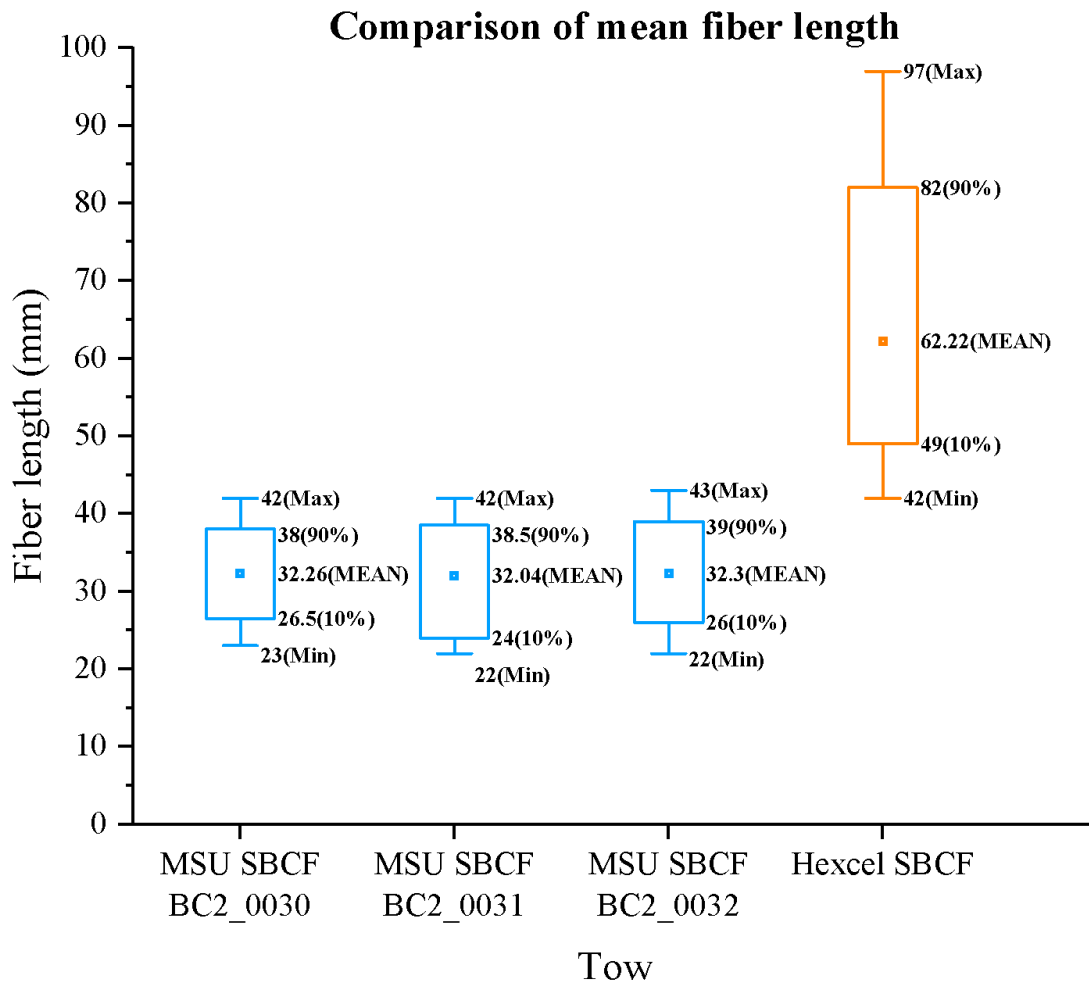


Figure 20: Comparison of mean fiber length between the MSU SBCF tows from three replicate batches (BC2_0030, BC2_0031, and BC2_0032) and Hexcel SBCF. Minimum, maximum, 10th percentile and 90th percentile values of fiber length are also shown.

The mean fiber length of the MSU SBCF tows from BC2_0030, BC2_0031 and BC2_0032 are 32.26 mm, 32.4 mm and 32.3 mm, respectively. The consistency in the values of the mean fiber length for all three batches indicates the consistency and reproducibility of MSU SBCF production. The legacy Hexcel SBCF contains fibers with a higher mean fiber length of 6.22 mm, matching with their prior evaluation of the process.

Effect of Stretch RatioSizing Deposition as a Function of Stretch Ratio

The relationship between the average sizing deposition per unit mass of the tow and the stretch ratio is shown in Figure 21.

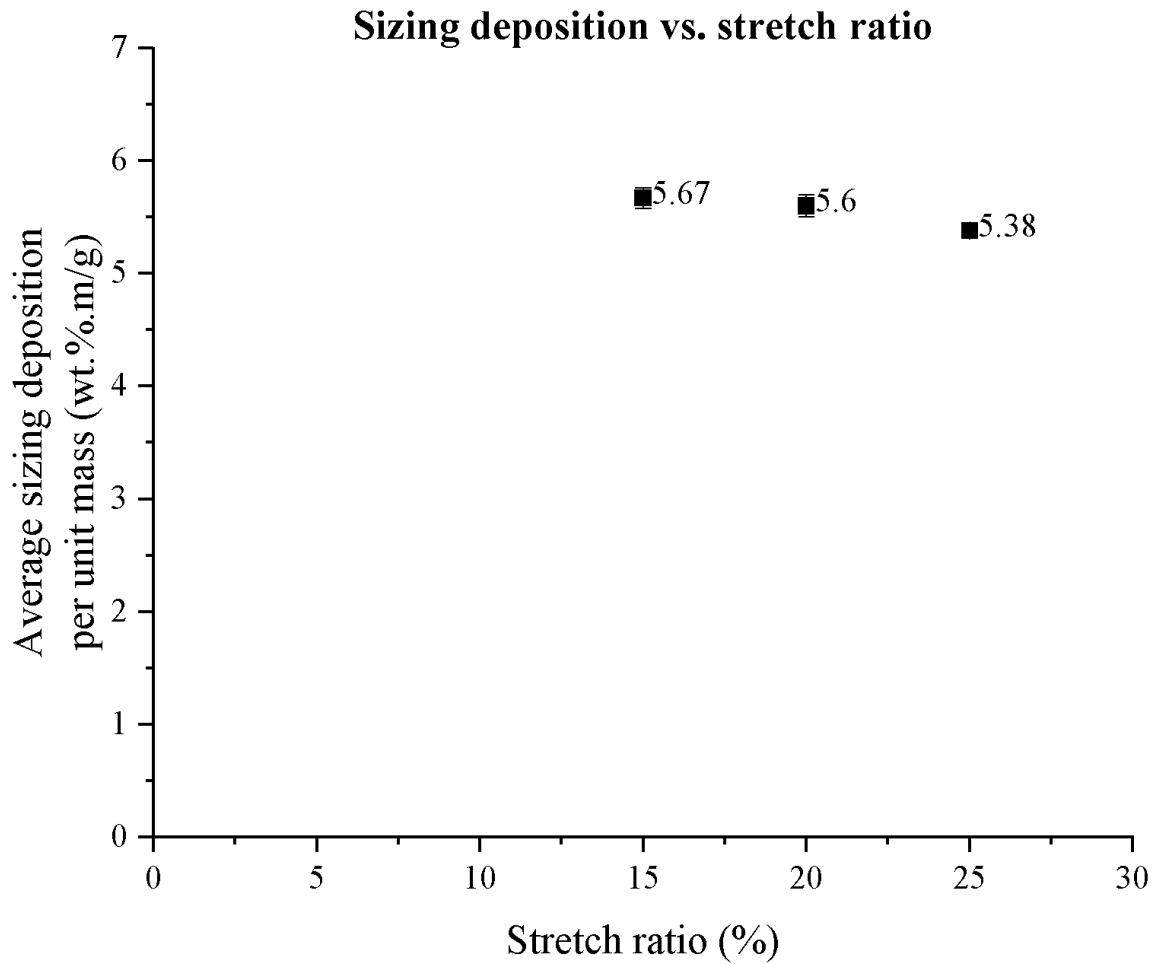


Figure 21: Sizing deposition as a function of stretch ratio. Each point is an average value of three readings, error bars show the standard deviation.

The average sizing deposition per unit mass is 5.67 wt.%.m/g(0.09), 5.60 wt.%.m/g(0.10), and 5.38 wt.%.m/g(0.05) for the stretch ratios 15%, 20%, and 25%, respectively. The values in the parentheses denote the standard deviation. The lower values of

standard deviation indicate uniform sizing deposition per unit mass of the tow at all three stretch ratios. A decreasing trend of average sizing deposition per unit mass is observed with increase in the stretch ratio from 15% to 25%. These results indicate that changes in sizing deposition are linked to changes of tow unit mass and that increasing the tow stretching might potentially reduce the effective cross section area of the tow.

Fiber Length Distribution as a Function of Stretch Ratio

The fiber length distribution of the tows having 15%, 20%, and 25% stretch ratio, respectively is shown in Figure 22.

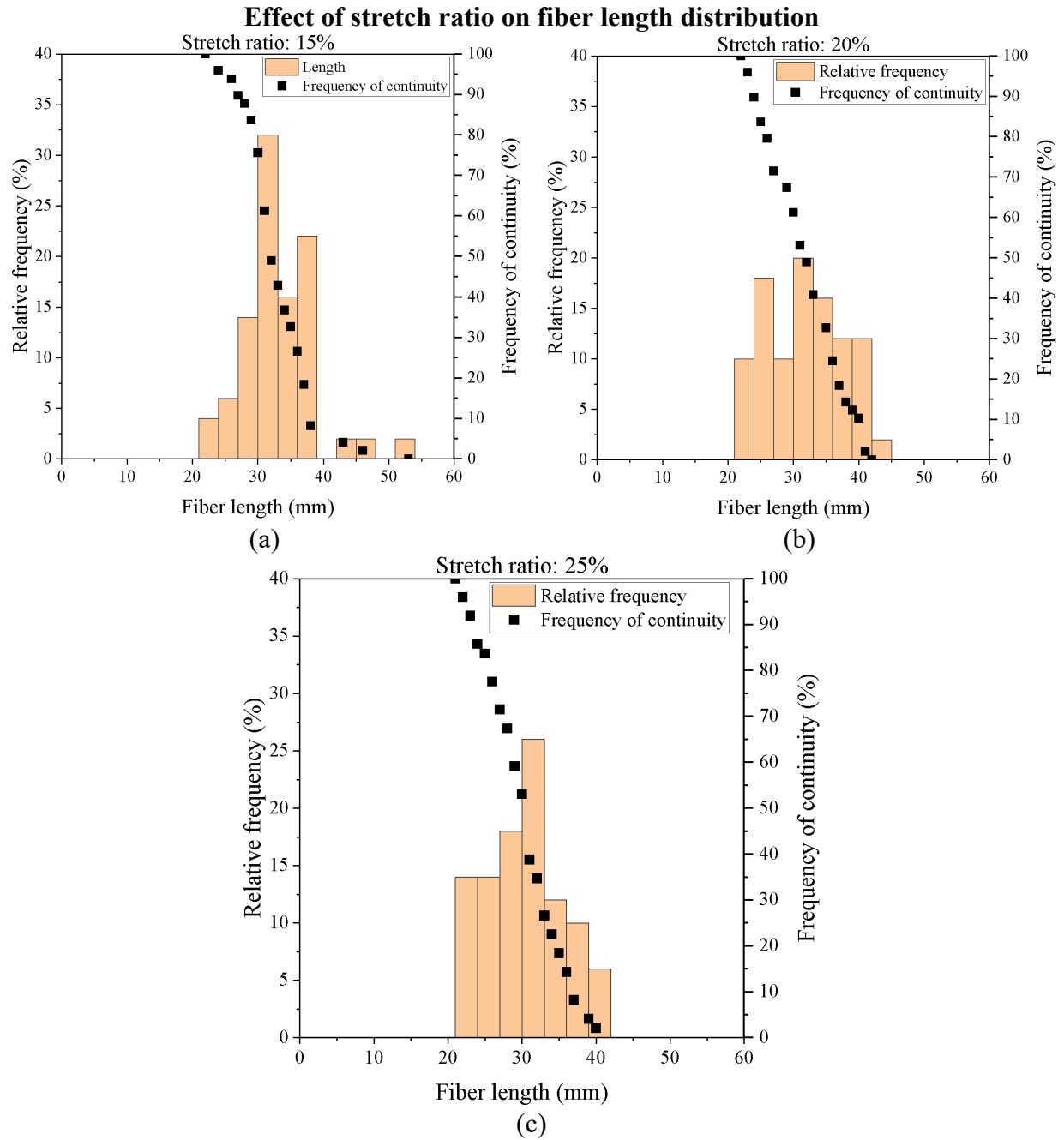
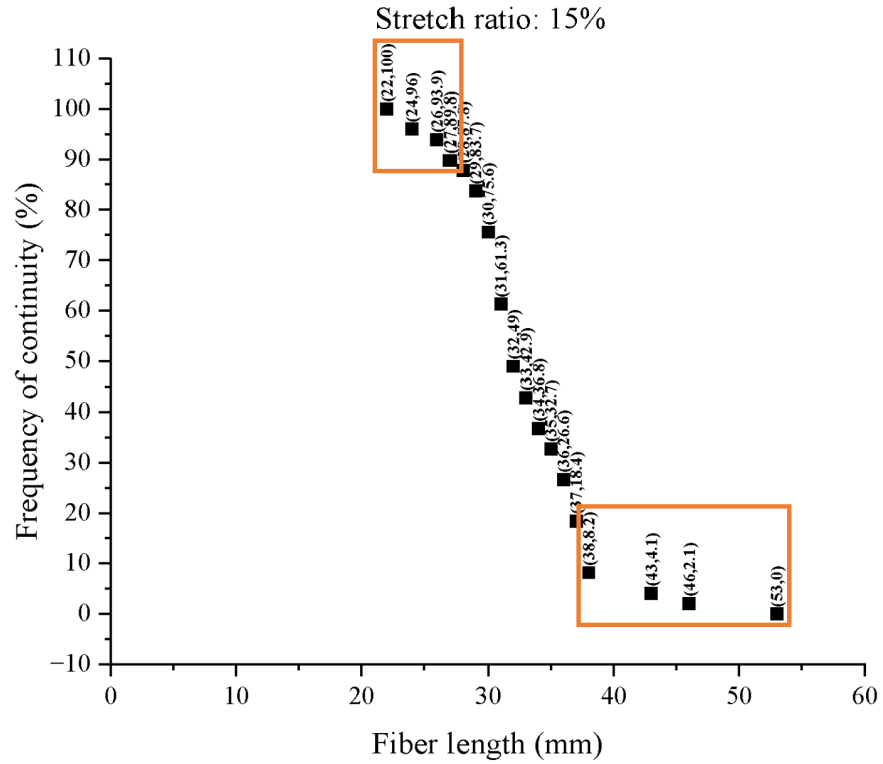


Figure 22: Fiber length distribution as a function of stretch ratio. The y-axis on the left represents the relative frequency and the one on the right denotes the frequency of continuity.

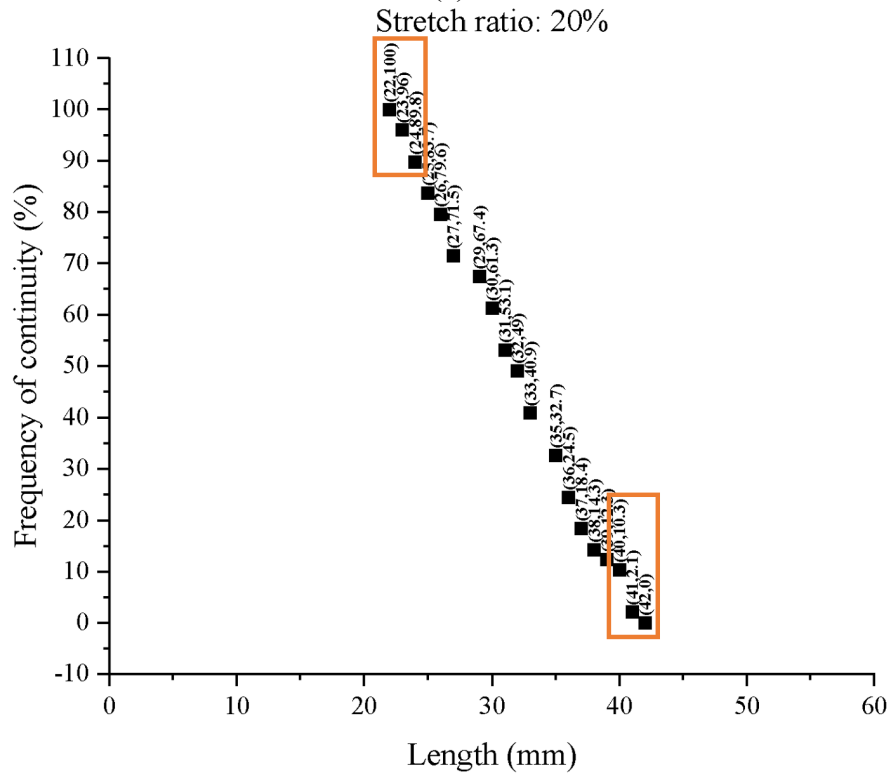
The tows having 15% stretch ratio have a wider fiber length range of 22 mm to 53 mm compared to the tows having 20% (22 mm to 42 mm) and 25% (21 mm to 40 mm) stretch ratio. The tows having 20% and 25% stretch ratio show very similar distributions.

The tail regions in the S-curve of MSU SBCF generated by varying the stretch ratio is shown in Figure 23.

Effect of stretch ratio on the tail regions in the S-curve



(a)



(b)

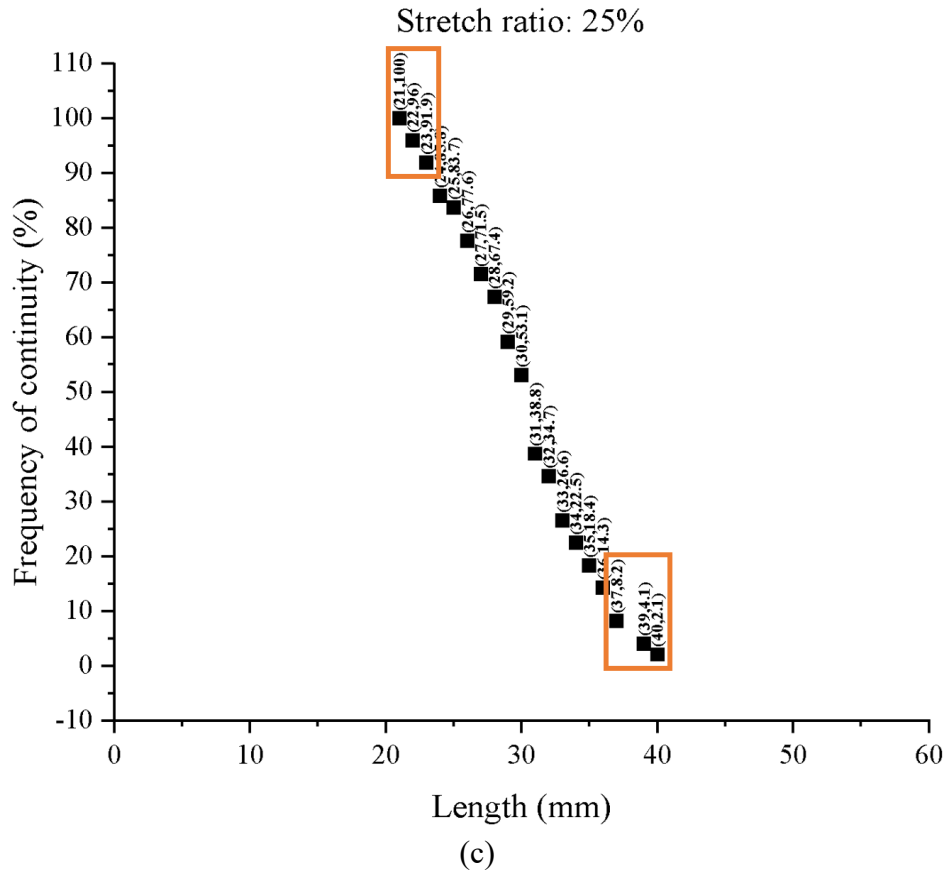


Figure 23: Fiber length distribution plotted as frequency of continuity vs. fiber length for (a) 15% stretch ratio; (b) 20% stretch ratio; (c) 25% stretch ratio. The brown box indicates both the upper tail and lower tail region.

When the stretch ratio is 15%, the fiber length ranges from 27 mm to 22 mm corresponding to the frequency of continuity of 89.8% to 100%. In the lower tail region (frequency of continuity is 8.2% to 0%), the range of fiber length is 38 mm to 53 mm. It is observed that MSU SBCF having 15% stretch ratio contains noticeably longer fibers as seen in the lower tail region. As for the tows made from 20%, and 25% stretch ratio, the tail regions show very similar fiber length distribution. In the upper tail region, the fiber length ranges from 24 mm to 22 mm. The range of fiber length in the lower tail region is approximately 37 mm-42

mm. The lower magnitude of tow stretching at 15% might have resulted in stretch broken fibers having relatively longer length.

The mean fiber length decreases very slightly with an increase in stretch ratio from 15% to 25% as observed in Figure 24. For the tows having 15%, 20% and 25% stretch ratio, the mean fiber lengths are 32.6 mm (5.54), 31.28 (5.73) mm and 29.8 mm (5.02), respectively. A decreasing trend of lower mean fiber length is observed with increase in stretch ratio.

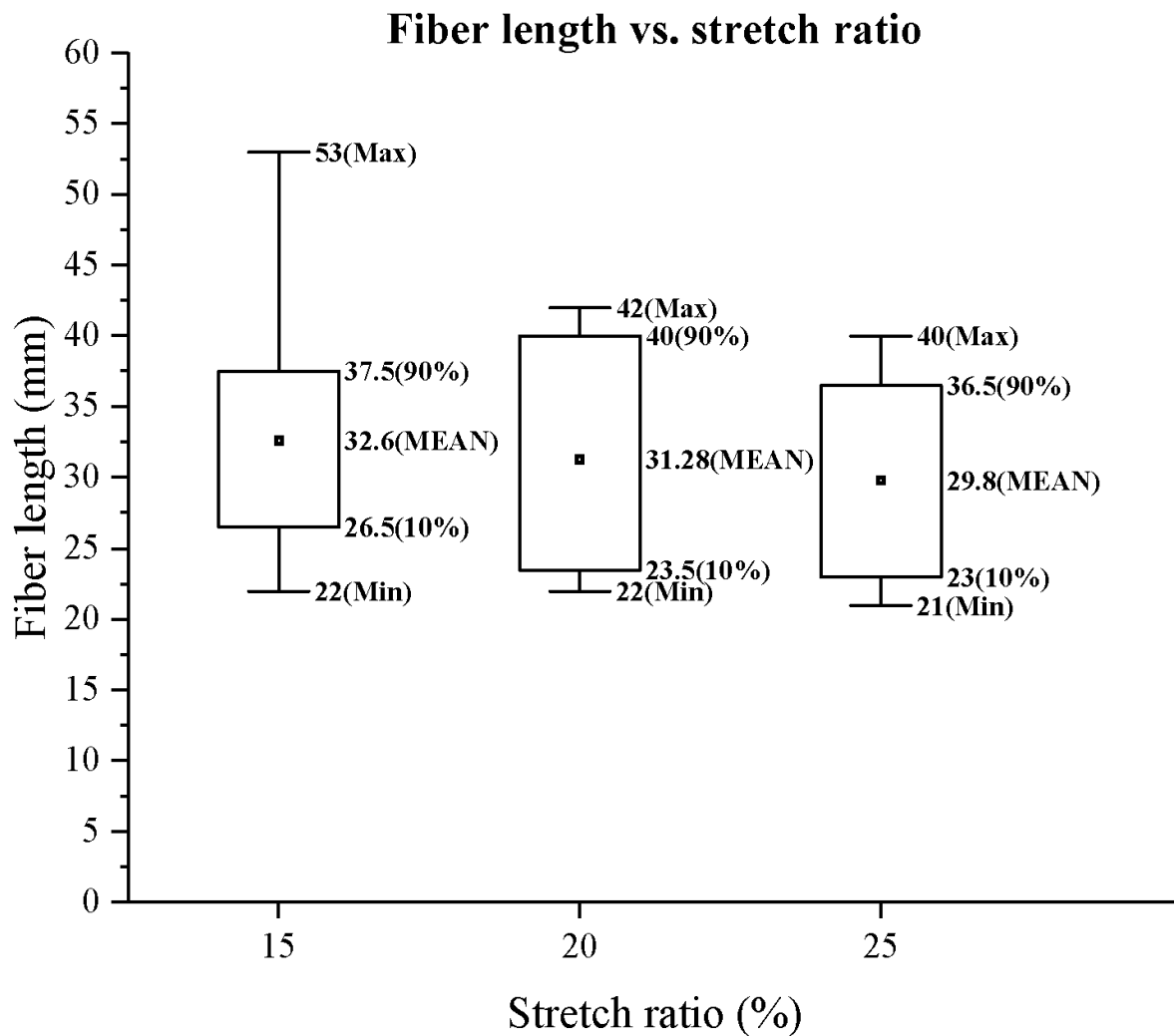


Figure 24: Representation of mean fiber length as a function of stretch ratio. The minimum, maximum, 10th percentile and 90th percentile values of the fiber length are also shown.

To determine if the decreasing trend of mean fiber length with increase in stretch ratio is statistically significant, analysis of variance (ANOVA) was performed. The p-value of 0.03889 presented in Table 5 showed that the mean fiber lengths are statistically significant at 0.05 significance level in the 15-25% stretch ratio regime, which suggest that the stretch ratio has a noticeable effect on the mean fiber length of single tow MSU SBCF.

Table 5: Results of analysis of variance (ANOVA) for fiber length as a function of stretch ratio.

Process variable	DF	Sum of Squares	Mean Square	F Value	p-value
Stretch ratio	2	196.21333	98.10667	3.31985	0.03889
Error	147	4344.08	29.55156		
Total	149	4540.29333			

The mean difference was calculated to identify the stretch ratio regime where the statistically significant change in mean fiber length was observed. A sig value of 0 presented in Table 6 at 15-20% and 20-25% stretch ratio regimes indicate the difference of means is not significant at 0.05 significance level. However, in the 15-25% stretch ratio regime, a sig value of 1 indicates that the difference of means is significant at 0.05 significance level. From the ANOVA analysis, it is evident that the stretch ratio has a dominant effect on the change in mean fiber length when the range of the stretch ratio regime relatively wide (15%-25% stretch ratio). However, the change in mean fiber length might not be statistically significant at a relatively narrow stretch ratio regime (15%-20% and 20%-25% stretch ratio).

Table 6: Comparison of mean using Tukey test for fiber length as a function of stretch ratio.

Stretch ratio	Mean Difference	SEM	q Value	Prob	Alpha	Sig	LCL	UCL
20%-15%	-1.32	1.08723	1.71699	0.44675	0.05	0	-3.89423	1.25423
25%-15%	-2.8	1.08723	3.64211	0.02944	0.05	1	-5.37423	-0.22577
25%-20%	-1.48	1.08723	1.92511	0.36395	0.05	0	-4.05423	1.09423

Tow Tenacity as a Function of Stretch Ratio

The relationship between the average peak load per unit mass of the tow and the stretch ratio is plotted in Figure 25. For the stretch ratio of 15%, 20%, and 25%, the average peak load per unit mass obtained at room temperature were 1038.55 Nm/g (63.53), 1011.74 Nm/g(33.71) and 1026.22 Nm/g (53.94), respectively. Although a decreasing trend was observed for the average sizing deposition per unit mass with increase in stretch ratio, the average peak load per unit mass at all three stretch ratios are comparable. Moreover, the mean fiber length is statistically significant between 15% and 25% stretch ratio but not statistically significant between 15-20% and 20-25% stretch ratio. These results suggest that the change in unit mass of the tow and mean fiber length are possibly not significant enough to observe any noticeable change in the tow tenacity at room temperature in the 15%-25% stretch ratio regime.

The average peak loads per unit mass obtained at elevated temperature are 25.69 Nm/g (0.95), 27.48 Nm/g (2.85) and 29.1 Nm/g(5.46) for the stretch ratio of 15%, 20% and 25%, respectively. These values suggest that change in the average peak load per unit mass of the tow at 100°C is not significant in the 15-25% stretch ratio regime. Therefore, the formability of the single tow MSU SBCF will not possibly change noticeably when the stretch ratio is 15-25%.

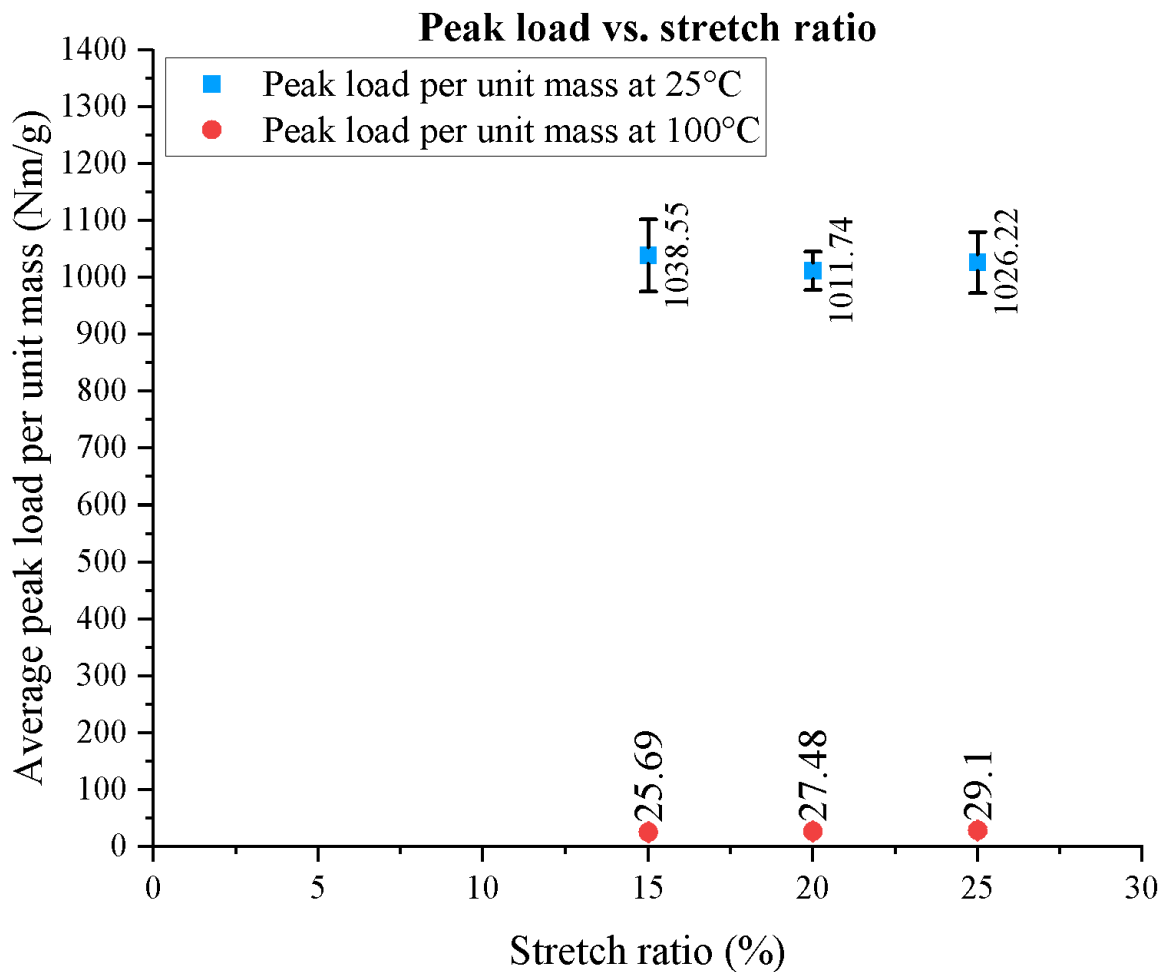


Figure 25: Figure: Tow tenacity as a function of stretch ratio. Each point is an average value of 5 measurements. The error bar might lie within the markers.

Effect of Nip Force

Sizing Deposition as a Function of Nip Force

The relationship between the sizing deposition on the tow and the nip force is presented in Figure 26.

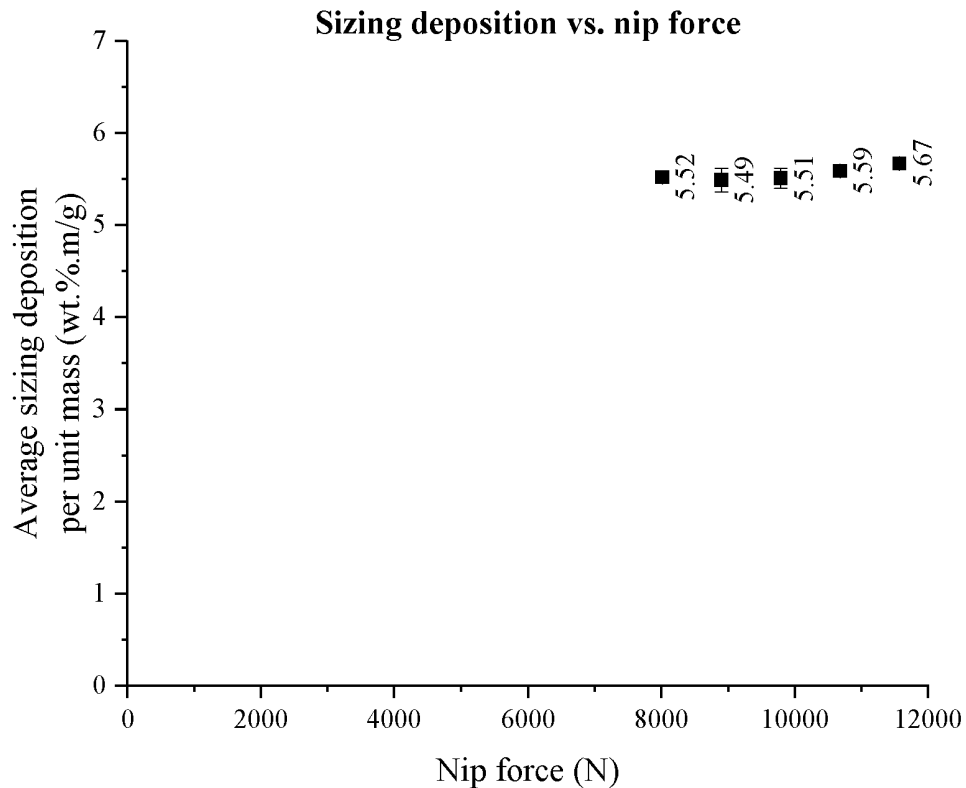
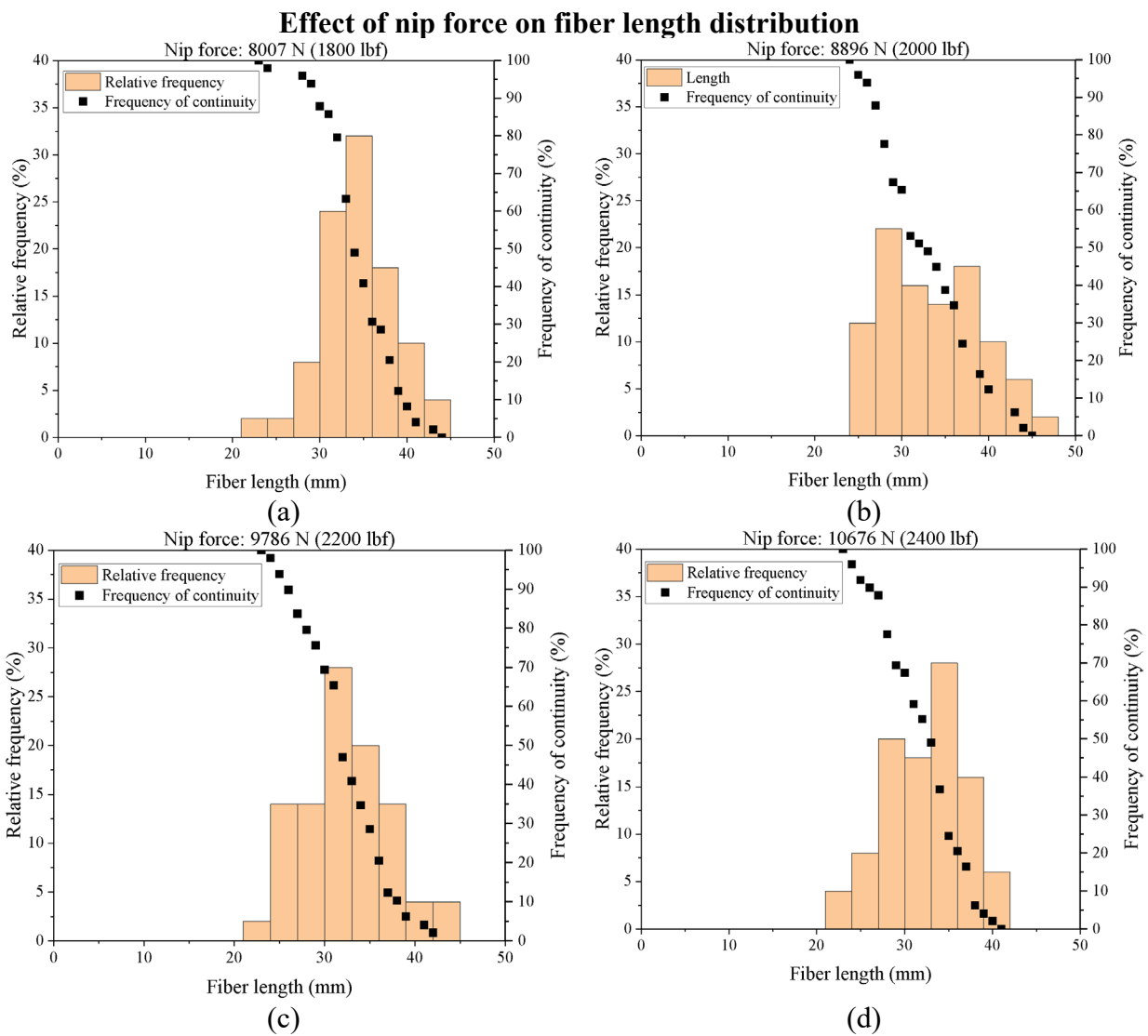


Figure 26: Average sizing deposition per unit mass plotted as a function of nip force. Each point in the graph is an average value of three measurements. The error bars might lie within the markers.

When the nip force was increased from 8007 N to 11565 N, the average sizing deposition per unit mass ranged from 5.49-5.67 wt.%.m/g. The result suggests that increasing the nip force in 8007-11565 N regime during the stretch breaking did not noticeably change the unit mass of the tow. Therefore, nip force probably has no noticeable effect on the sizing deposition on the tow.

Fiber Length Distribution as a Function of Nip Force

The fiber length distribution of the MSU SBCF tows were very similar for the nip force values ranging from 8007 N to 11565 N. The range of the fiber length was approximately 23 mm to 45 mm. Figure 27 shows the fiber length distribution as the nip force was changed from 8007 N to 11565 N.



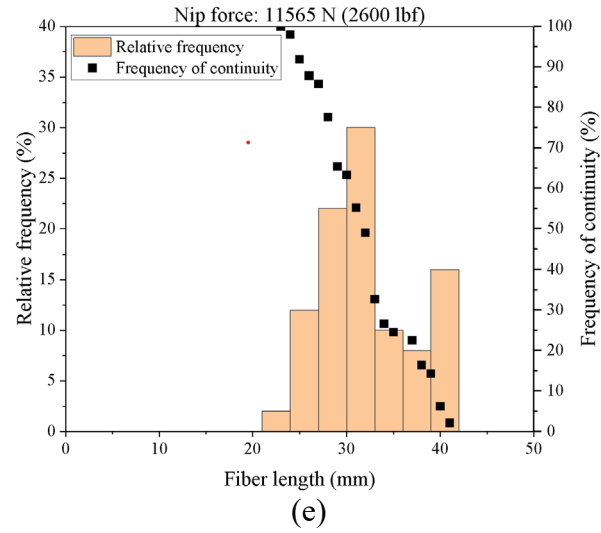
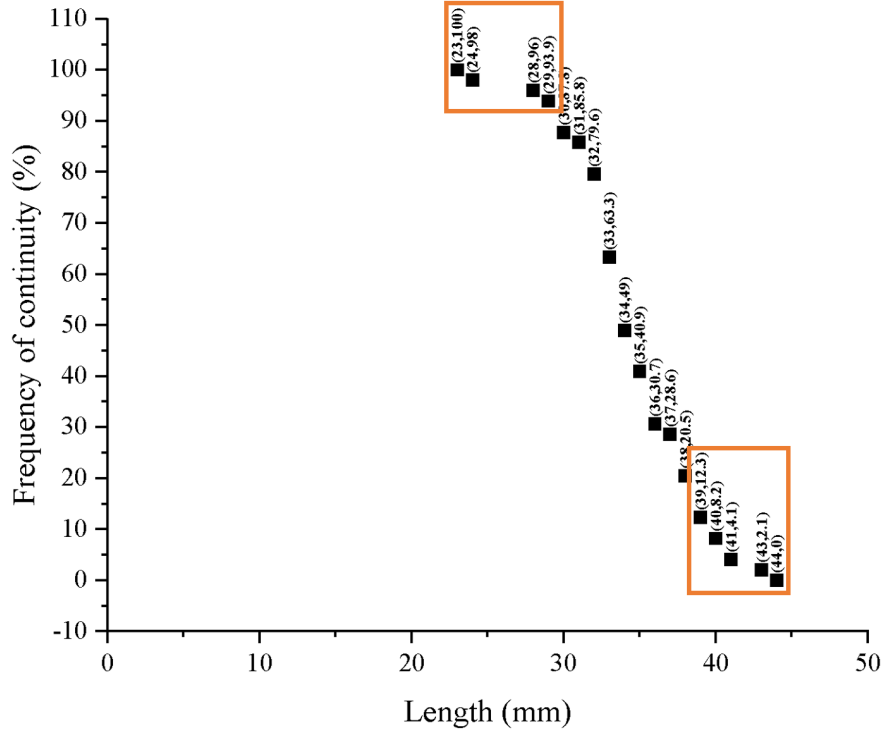


Figure 27: Fiber length distribution as a function of nip force: (a) 8007 N (1800 lbf); (b) 8896 N (2000 lbf); (c) 9786 N (2200 lbf); (d) 10676 (2400 lbf); (e) 11565 N (2600 lbf).

The tail regions in S-curve of single tow MSU SBCF generated by varying the nip force is presented in Figure 28.

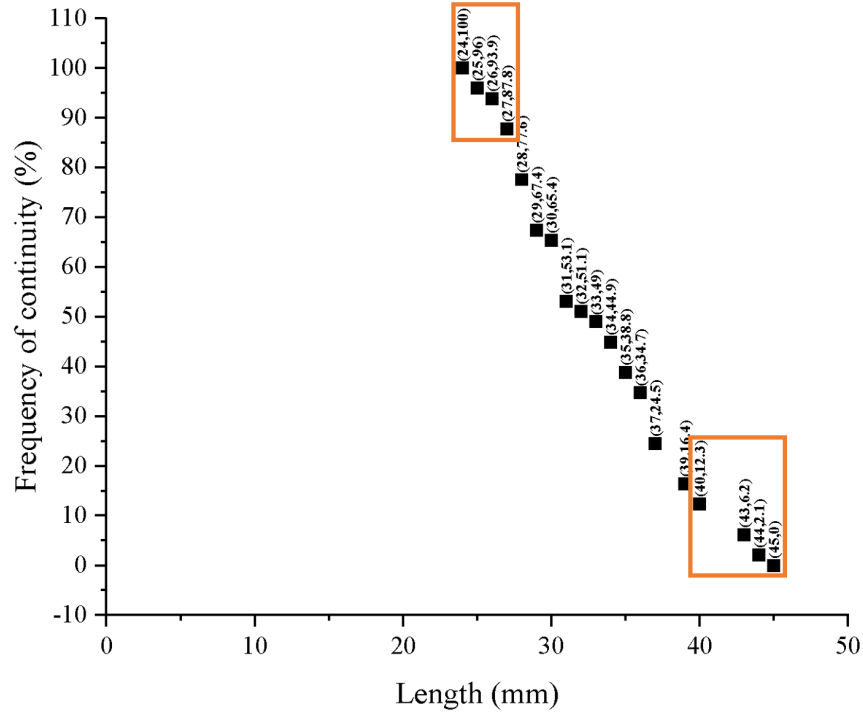
Effect of stretch ratio on the tail regions in the S-curve

Nip force: 8007 N (1800 lbf)



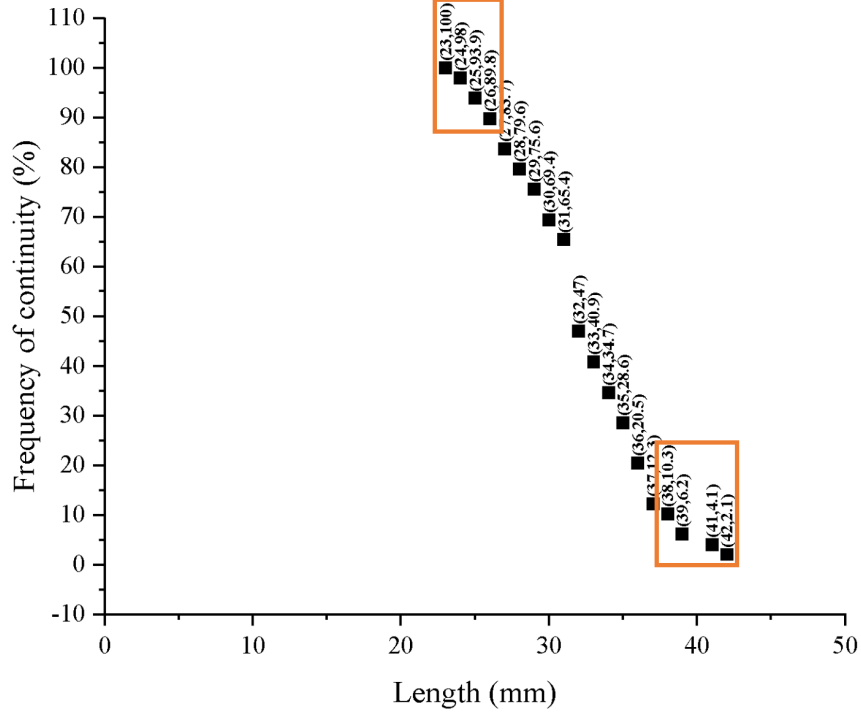
(a)

Nip force: 8896 N (2000 lbf)



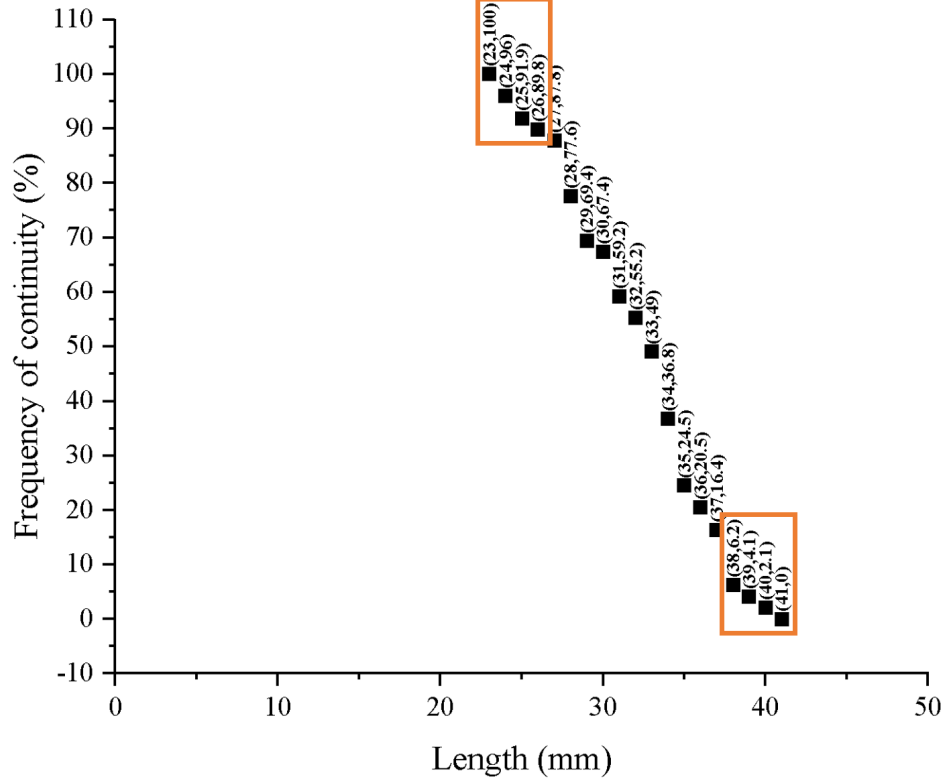
(b)

Nip force: 9786 N (2200 lbf)



(c)

Nip force: 10676 N (2400 lbf)



(d)

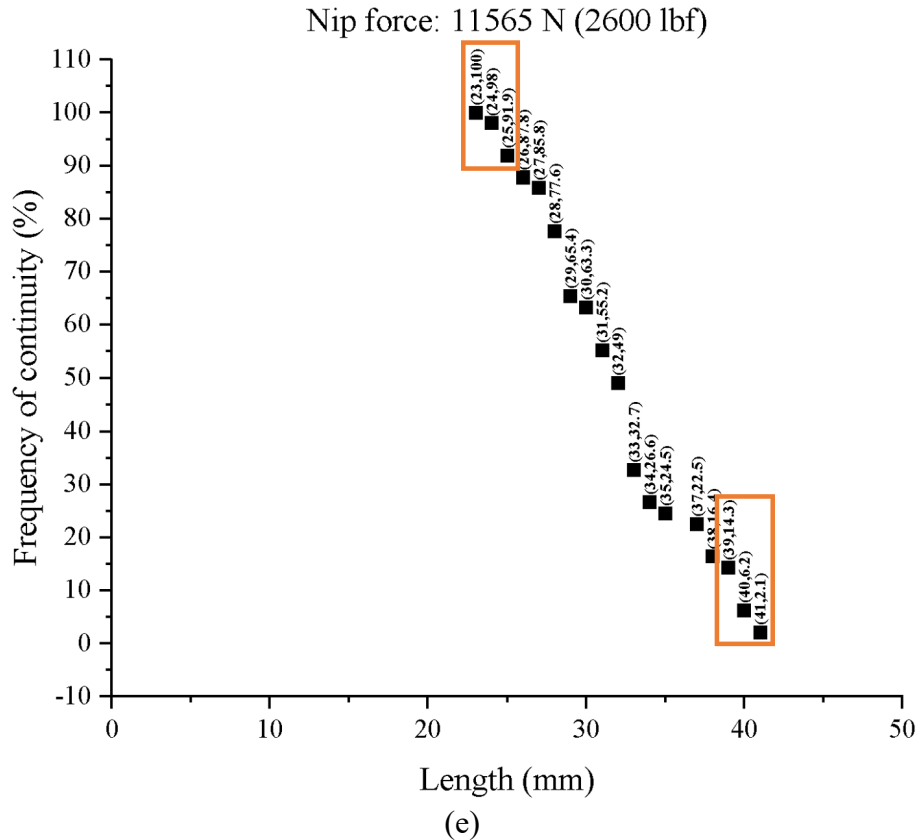


Figure 28: Fiber length distribution plotted as frequency of continuity vs. fiber length for the nip forces of (a) 8007 N (1800 lbf); (b) 8896 N (2000 lbf); (c) 9786 N (2200 lbf); (d) 10676 (2400 lbf); (e) 11565 N (2600 lbf). The red box denotes both the upper tail region and the lower tail region.

When nip forced increases from 8007 N to 11565 N, in the upper tail region, range of fiber length is approximately 29 mm- 23 mm. For the lower tail region is range is approximately 39 mm-45 mm. Another observation is that with increase nip force, the range of fiber length decreases gradually in both the upper tail region and lower tail region. An increased pressure applied by the nip roller to the tow might potentially favor the lower spread of the fiber length in the tail regions.

The mean fiber length decreased from 34.1 mm (4.22) to 31.6 mm (5.02) with increase in the nip force from 8007 N to 11565 N, which is presented in Figure 29.

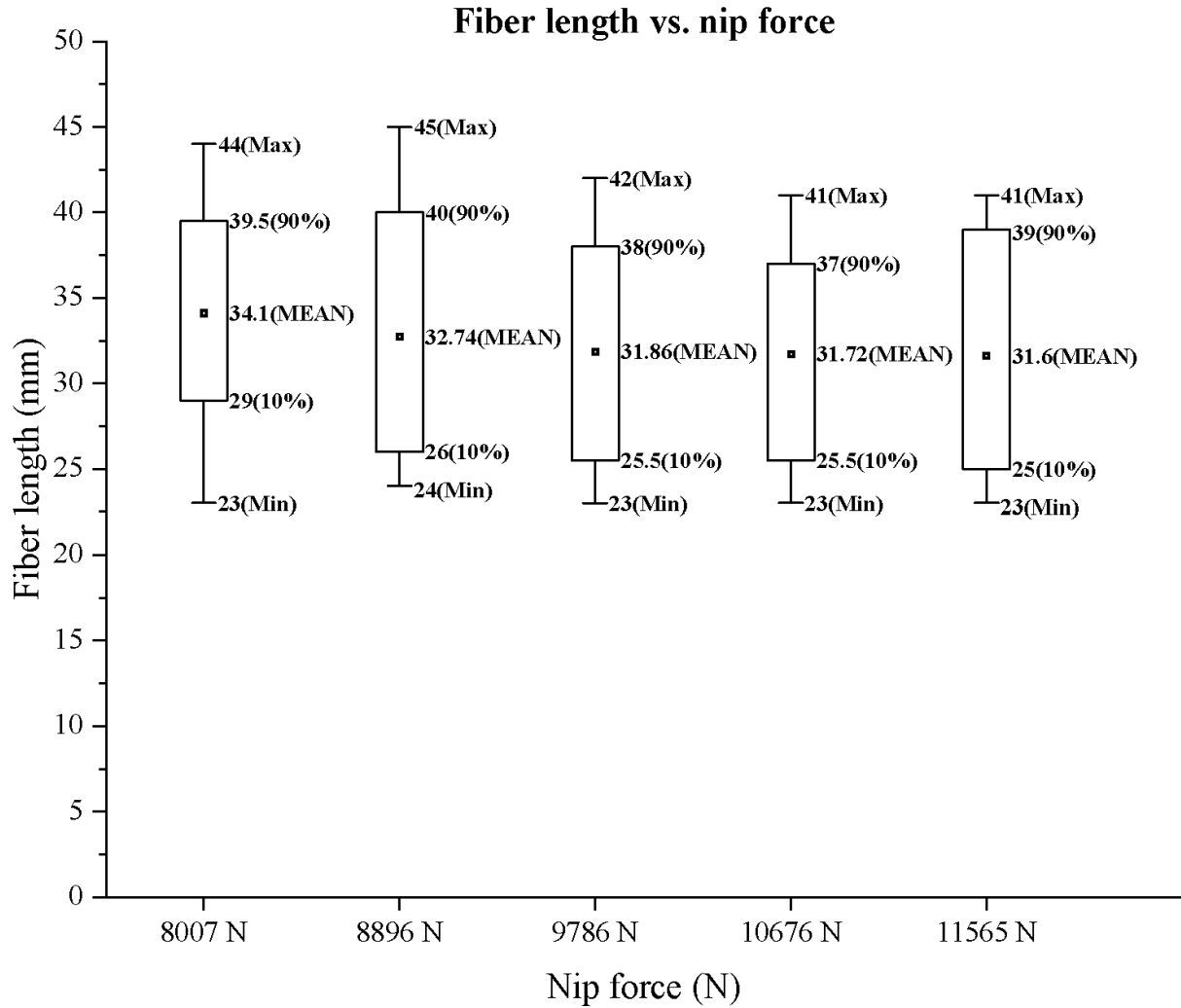


Figure 29: Representation of mean fiber length as a function of nip force. The minimum, maximum, 10th percentile and 90th percentile values of the fiber length are also shown.

Analysis of variance (ANOVA) was performed to determine if the values of the means fiber length are statistically significant when the nip force was increased from 8007 N to 11565 N. The p-value of 0.05613 presented in Table 7 showed that the mean fiber lengths are not statistically significant at 0.05 significance level in the 8007-11565 N nip force regime, which suggest that the nip force has no noticeable effect on the mean fiber length of single tow MSU SBCF.

Table 7: Results of analysis of variance (ANOVA) for fiber length as a function of nip force.

Process variable	DF	Sum of Squares	Mean Square	F Value	p-value
Nip fore	4	219.976	54.994	2.33582	0.05613
Error	245	5768.22	23.54376		
Total	249	5988.196			

The sig value of 0 was observed between all the nip force values which is shown in Table

8. The result obtained from the Tukey method indicates that the difference of the means is not significant at 0.05 significance level.

Table 8: Comparison of mean using Tukey test for fiber length as a function of nip force

Nip force	Mean Difference	SEM	q Value	Prob	Alpha	Sig	LCL	UCL
8896 N-8007 N	-1.36	0.97044	1.98192	0.62733	0.05	0	-4.02696	1.30696
9786 N-8007 N	-2.24	0.97044	3.26434	0.14573	0.05	0	-4.90696	0.42696
9786 N-8896 N	-0.88	0.97044	1.28242	0.89411	0.05	0	-3.54696	1.78696
10676 N-8007 N	-2.38	0.97044	3.46836	0.10516	0.05	0	-5.04696	0.28696
10676 N-8896 N	-1.02	0.97044	1.48644	0.83114	0.05	0	-3.68696	1.64696
10676 N-9786 N	-0.14	0.97044	0.20402	0.9999	0.05	0	-2.80696	2.52696
11565 N-8007 N	-2.5	0.97044	3.64323	0.07799	0.05	0	-5.16696	0.16696
11565 N-8896 N	-1.14	0.97044	1.66132	0.76583	0.05	0	-3.80696	1.52696
11565 N-9786 N	-0.26	0.97044	0.3789	0.99886	0.05	0	-2.92696	2.40696
11565 N-10676 N	-0.12	0.97044	0.17488	0.99995	0.05	0	-2.78696	2.54696

Tow Tenacity as a Function of Nip Force

The effect of nip force on tow tenacity at both room and elevated temperature is presented in Figure 30.

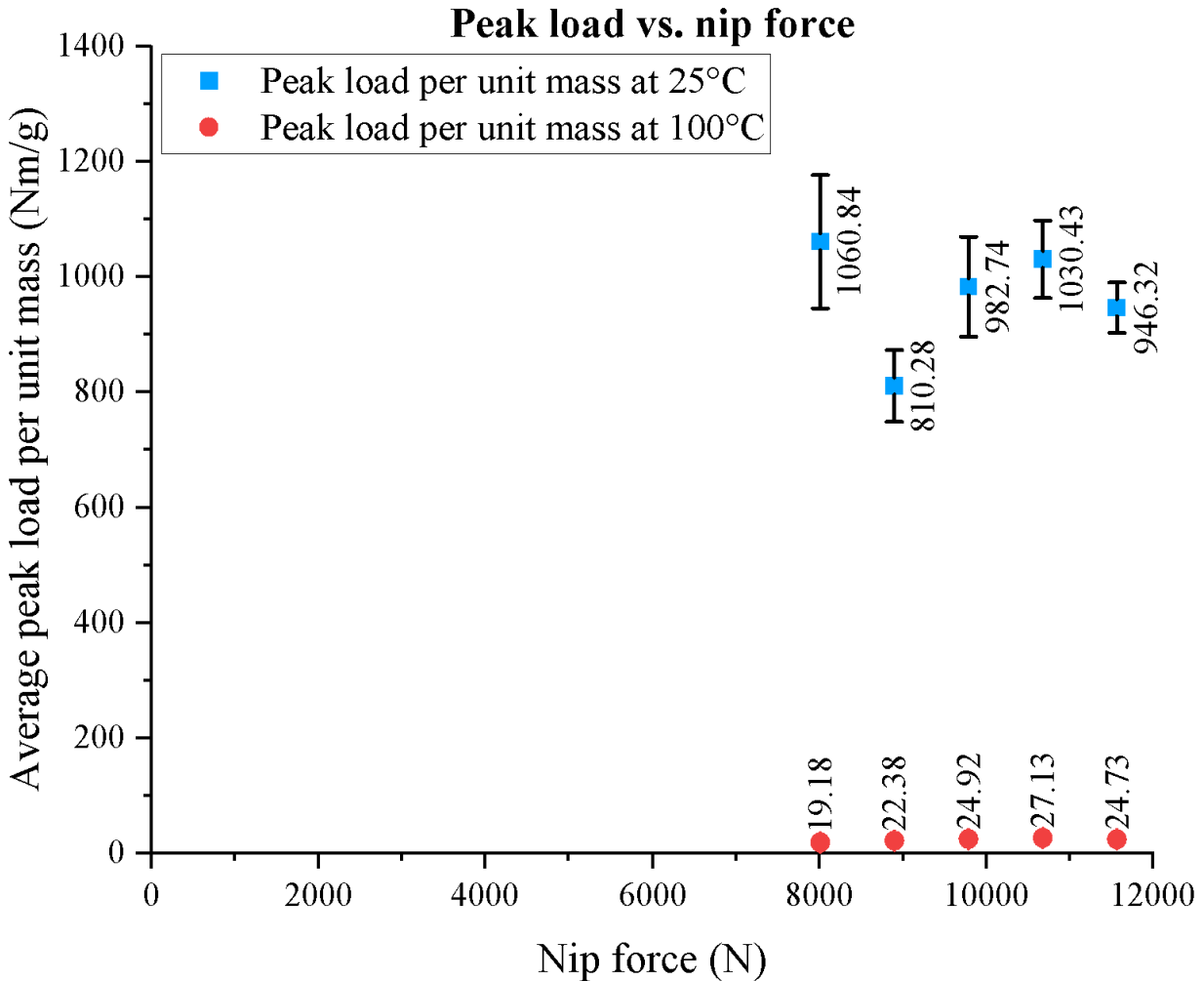


Figure 30: Average peak load per unit mass plotted as a function of the nip force at both 25°C and 100°C. Each point is an average value of five measurements. Error bars may lie within the marker.

At a nip force of 8807 N, the average peak load per unit mass at room temperature was 1060.85 Nm/g (115.85). Although the average peak load per unit mass obtained at this nip force is the highest in the 8007-11565 N regime, the quality of the SBCF material was not consistent.

A noticeable tow splitting, tow folding, and tow waviness were observed along some section of the tow length. The usable length of the tow at nip force of 8007 N was approximately 40%. At lower nip force, the SBCF tow did not possibly experience adequate nip roller pressure during the stretch breaking process. Therefore, there was a greater chance of tow slippage at lower nip force leading to the generation of the approximately 60% of the unusable tows. When the nip force was increased to 8896 N, the average peak load per unit mass decreased to 810.28 Nm/g (62.16). A noticeable tow splitting was observed in the usable tow section produced at 8896 N nip force resulting in significant gaps inside the tows which compromised the quality of the MSU SBCF tows. Approximately 30% of the usable tows were produced at 8896 N, which was the lowest yield of usable tow in 8007-11565 N regime. The peak loads obtained at 9786 N and 10676 N are 982.74 Nm/g (86.88) and 1030.43 Nm/g (67.37), respectively and represented a regime where the usable SBCF tows generated were 80% and 83%, respectively. The higher percentage of usable tows in this regime indicate that the stretch breaking process became more efficient with increase in the nip roller pressure experienced by the tow. The decrease in the standard deviation with increase in nip force also supports the more efficient SBCF production. At 11565 N, the average peak load per unit mass decreased to 946.32 Nm/g (43.46). Again, the issue of tow splitting was observed in this batch of MSU SBCF material with usable material being 71%. A higher nip force of 11565 N possibly damaged some fibers due to excessive force on the tow by the nip roller.

At 100°C the average peak load per unit mass was 19.18 Nm/g (2.65) to 27.13 (1.91) Nm/g when the nip force was increased from 8007-10676 N. The At 11565 N, the average peak load per unit mass decreased to 24.73 Nm/g. The increasing trend of average peak load per unit

mass at 100°C indicate that the percentage of usable tow increased with increasing the nip force. The decrease in the standard deviation was also observed similar to the room temperature tow tenacity result. At 11565 N the average peak load per unit mass decreased to 24.73 Nm/g (0.60). The tow tenacity result at both room and elevated temperature suggest that, the SBCF tows generated using 10676 N nip force appeared to produce higher quantity of usable SBCF without suffering from noticeable tow splitting. The average peak load per unit mass at 9786 N also yielded comparable results, representing an optimal processing regime for the nip force between 9786-10676 N.

FUTURE SCOPE OF SBCF PRODUCTION

The production of stretch broken carbon fiber generation at laboratory-scale by a first generation stretch-break machine (Bobcat) was successful. The continuous development and optimization of the Bobcat machine is ongoing at the hardware level to produce industry grade SBCF material. The improvements at the hardware level are expected to optimize the independent process variables which include sizing application, stretch ratio, nip force, and line speed. During the stretch-brake process, these independent process variables work together to influence the dependent process variables which include sizing deposition on the tow, fiber length distribution, mean fiber length and tow tenacity. The quality of SBCF material produced is governed by the interaction among these process variables. For a particular SBCF production setup, one process variable might be more dominant compared to the other process variables. Identifying the relationship among these process variables along with their relative contribution is crucial in producing SBCF material possessing desired qualities. Therefore, it is extremely important to define the lower limit and upper limit for each of the independent process variables. These limits will in turn dictate the desired upper limit and lower limit of the dependent process variables. The hardware design and the feedback obtained from the laboratory scale experiment will be valuable in designing and building the industry-scale second generation of stretch-break machine.

The experimental results obtained in this work provides insight into the upper limit and lower limit of the process variables which will govern the the feasibility of future SBCF production at industrial capacity. The amount of sizing deposited on the tow is dictated by the concentration of sizing bath. The laboratory grade stretch-break machine (Bobcat) is capable of

generating single tow MSU SBCF having approximately 1.8 wt.% sizing deposition when sizing bath concentration of 10 wt.% is used. The sizing deposition above 2 wt.% is not acceptable since the mechanical performance of the prepreg and the cured composite reinforced with MSU SBCF with sizing deposition more than 2 wt.% will deteriorate. A dry tow tenacity of 300-400 N is desirable for the handleability and back-tension ability. This range of dry tow tenacity is comparable to the single tow continuous carbon fiber (both sized and unsized). The laboratory-scale single tow MSU SBCF with approximately 1.8 wt.% sizing deposition on it is capable of achieving this dry tow tenacity, which is promising in terms of the industry-grade production of SBCF material. A tow tenacity of approximately 10 N at 100°C indicates desirable formability of the single tow MSU SBCF especially in terms forming parts with sharp radii, corners, and curvatures. The elevated temperature tow tenacity of MSU SBCF motivates the development and optimization of a comprehensive set of novel test fixtures to characterize and quantify the formability of MSU SBCF material. During the SBCF production, the drive rollers and nip rollers engage together to cause the stretch breaking. So, the evaluation of these two process variables is also of paramount importance to optimize the SBCF production. A stretch ratio regime of 15-25% was investigated in this present work. The overall trend of sizing deposition on the tow and the tow tenacity was not noticeable in this stretch ratio regime. However, a decreasing trend of statistically significant mean fiber length was observed in the 15-25% stretch ratio regime. The results from the stretch ratio experiment suggest that a wider stretch ratio regime needs to be investigated to obtain a full picture of the lower and upper limit of the sizing deposition on the tow, fiber length distribution and tow tenacity with statistically significant trend. A nip force regime of 9786-10676 N produces greater percentage of usable single tow

MSU SBCF possibly because the stretch breaking process is more efficient in this nip force regime. This result is also crucial in designing the geometry and the arrangement of the nip rollers in the next generation of stretch-break machine.

The first generation of stretch-break machine was equipped with one pair of drive roller arranged in parallel and one pair of nip roller positioned at top and bottom of the drive roller pair. The second generation of stretch-break machine will have three pairs of drive rollers arranged in a row and each pair will be associated with one nip roller placed above them. This newly designed geometry is expected to improve the stretch breaking process since the tow will experience a uniform tension from all three pairs of drive rollers and nip pressure from all three nip rollers simultaneously. The arrangement of nip rollers is expected to improve the tow slippage at lower nip force. The resulting single tow SBCF is expected to exhibit a more uniform fiber length distribution with smaller mean fiber lengths due to an increased number of fiber breaks in the tow. The interaction among the independent process variables might be influenced by the change in geometry. However, since the acceptable upper and lower limit of dependent variables are already predetermined based on the result from the first generation of stretch-break machine, the independent process variables can be adjusted accordingly.

CONCLUSION

From the sizing deposition experiment, target sizing deposition in the range of 1.5-2 wt.% on the single tow MSU SBCF was achieved when a 10 wt.% sizing bath was used. The tow tenacity experiment showed that the single tow MSU SBCF with no sizing applied to it (control) had a very low peak load of 1.77 N at room temperature. A peak load of 290.86 N tested at room temperature corresponding to the 1.75 wt.% sizing deposition was determined to be an ideal balance between the sizing deposition and back-tension ability. A peak load of 10.11 N tested at 100°C suggests that the MSU SBCF is a formable material with potential to be formed into complex shapes during composite production. Three replicate batches of MSU SBCF also showed consistency and repeatability in terms of sizing deposition on the single tow MSU SBCF, tow tenacity at both room and elevated temperature, fiber length distribution, and mean fiber length. When the stretch ratio was increased from 15% to 25%, the average sizing deposition per unit mass decreased from 5.67 wt.%.m/g to 5.38 wt.%.m/g due to the change in mass per unit length of the tow. MSU SBCF generated with 15% Stretch ratio showed a wider fiber length distribution of 22-53 mm compared to 21-42 mm range for 20% and 25% stretch ratio materials. The statistical analysis of the fiber length indicates that the mean fiber lengths are not statistically significant in the 15-20% and 20-25% stretch ratio regime. However, means fiber lengths are statistically significant in the 15-25% stretch ratio regime. When the stretch ratio was increased from 15% to 25%, the average peak load per unit mass at room temperature was 1011.74-1038.55 Nm/g, which suggest the decreasing trend of average sizing deposition per unit mass, fiber length distribution and mean fiber length had no noticeable effect on the tow tenacity at room temperature in the 15-25% stretch ratio regime. Therefore, the single tow MSU SBCF

was expected to possess comparable tow handleability and back tension ability at 15-25% stretch ratio. The average peak load per unit mass obtained at 100°C were 25.69 Nm/g, 27.48 Nm/g, and 29.1 Nm/g corresponding to the stretch ratio of 15%, 20% and 25% respectively, which suggests that single tow MSU SBCF is expected to possess comparable formability in the 15-25% stretch ratio regime. Varying the nip force from 8007 N to 11565 N did not significantly change the average sizing deposition per unit mass of the tow, which indicate the sizing deposition on the single tow MSU SBCF is not nip force dependent in the 8007-11565 N nip force regime. The fiber length distribution of single tow MSU SBCF was approximately 23-45 mm at 8007-11565 N nip force. Statistical analysis of fiber length showed that the differences in mean fiber lengths are not statistically significant in 8007-11565 nip force regime. The tow tenacity characterization suggests that the SBCF tows generated using 10676 N nip force are the most consistent material in terms of the percentage of usable length of tow having desirable handleability, back-tension ability and formability. Comparable results were also observed for the SBCF tows produced using the nip force of 9786 N, which indicates an optimal nip force processing regime of 9786-10676 N.

RECOMMENDED FUTURE TASKS

Tow Tenacity

1. New batches of single tow MSU SBCF will be generated to reevaluate the effect of nip force on the MSU SBCF materials. Nip force will be varied from 8007N to 11565 N.
2. Effect of line speed on the tow tenacity will also be evaluated. MSU SBCF produced so far has been generated using a line speed of 1.22 m/min (4 FPM). Line speed will be increased to 3.05 m/min (10 FPM) to generate MSU SBCF materials.

Thermal Behavior

1. Rheological characterization of Michelman 181290IX sizing will be performed to determine its change in viscosity with temperature and relate this experiment to understand the formability of MSU SBCF.
2. Characterization of sizing by differential scanning calorimetry (DSC) will be performed to determine the temperature at which sizing starts to soften.
3. Thermogravimetric analysis (TGA) of sizing will be performed to determine thermal stability of sizing at the forming operation temperature.

Wettability of Sizing

1. Contact angle and surface energy will be determined to understand the wettability performance of sizing.

REFERENCES CITED

- [1] M. Sharma, S. Gao, E. Mäder, H. Sharma, L. Yew, and J. Bijwe, “Carbon fiber surfaces and composite interphases,” *Compos. Sci. Technol.*, vol. 102, pp. 35–50, 2014, doi: 10.1016/j.compscitech.2014.07.005.
- [2] J. C. Janicki, D. S. Bajwa, D. Cairns, R. Amendola, C. Ryan, and A. Dynkin, “Gauge length and temperature influence on the tensile properties of stretch broken carbon fiber tows,” *Compos. Part A Appl. Sci. Manuf.*, vol. 146, no. February, p. 106426, 2021, doi: 10.1016/j.compositesa.2021.106426.
- [3] S. Wu, Y. Liu, Y. Ge, L. Ran, K. Peng, and M. Yi, “Surface structures of PAN-based carbon fibers and their influences on the interface formation and mechanical properties of carbon-carbon composites,” *Compos. Part A Appl. Sci. Manuf.*, vol. 90, pp. 480–488, 2016, doi: 10.1016/j.compositesa.2016.08.023.
- [4] G. Jacobsen, “Mechanical characterization of stretch broken carbon fiber materials - IM7 fiber in 8552 resin,” *Int. SAMPE Symp. Exhib.*, no. December 2007, 2010.
- [5] E. B. Chris Ridgard, Douglas Cairns, “ADVANCES IN THE DEVELOPMENT OF STRETCH BROKEN CARBON FIBER FOR PRIMARY AIRCRAFT STRUCTURE,” *CAMX Anaheim CA*, 2022.
- [6] H. Zheng *et al.*, “Recent advances of interphases in carbon fiber-reinforced polymer composites: A review,” *Compos. Part B Eng.*, vol. 233, no. January, p. 109639, 2022, doi: 10.1016/j.compositesb.2022.109639.
- [7] H. He, K. Li, and F. Gao, “Improvement of the bonding between carbon fibers and an epoxy matrix using a simple sizing process with a novolac resin,” *Constr. Build. Mater.*, vol. 116, pp. 87–92, 2016, doi: 10.1016/j.conbuildmat.2016.04.125.
- [8] Y. Athulya Wickramasingha *et al.*, “Surface modification of carbon fiber as a protective strategy against thermal degradation,” *Compos. Part A Appl. Sci. Manuf.*, vol. 153, no. November 2021, p. 106740, 2022, doi: 10.1016/j.compositesa.2021.106740.
- [9] J. Moosburger-Will *et al.*, “Interaction between carbon fibers and polymer sizing: Influence of fiber surface chemistry and sizing reactivity,” *Appl. Surf. Sci.*, vol. 439, pp. 305–312, May 2018, doi: 10.1016/j.apsusc.2017.12.251.
- [10] M. Lai, L. Jiang, X. Wang, H. Zhou, Z. Huang, and H. Zhou, “Effects of multi-walled carbon nanotube/graphene oxide-based sizing on interfacial and tribological properties of continuous carbon fiber/poly(ether ether ketone) composites,” *Mater. Chem. Phys.*, vol. 276, no. April 2021, p. 125344, 2022, doi: 10.1016/j.matchemphys.2021.125344.
- [11] W. Jiao *et al.*, “Preparation of carbon fiber unsaturated sizing agent for enhancing interfacial strength of carbon fiber/vinyl ester resin composite,” *Appl. Surf. Sci.*, vol. 439, pp. 88–95, 2018, doi: 10.1016/j.apsusc.2017.12.226.

- [12] H. Lyu, N. Jiang, J. Hu, Y. Li, N. Zhou, and D. Zhang, "Preparing water-based phosphorylated PEEK sizing agent for CF/PEEK interface enhancement," *Compos. Sci. Technol.*, vol. 217, no. June 2021, p. 109096, 2022, doi: 10.1016/j.compscitech.2021.109096.
- [13] Z. Dai, F. Shi, B. Zhang, M. Li, and Z. Zhang, "Effect of sizing on carbon fiber surface properties and fibers/epoxy interfacial adhesion," *Appl. Surf. Sci.*, vol. 257, no. 15, pp. 6980–6985, 2011, doi: 10.1016/j.apsusc.2011.03.047.
- [14] E. Laukmanis *et al.*, "Effect of the interplay between fiber surface chemistry and sizing reactivity on fiber matrix interaction in carbon fiber reinforced epoxy resin reinforced epoxy resin," *Compos. Interfaces*, vol. 00, no. 00, pp. 1–31, 2022, doi: 10.1080/09276440.2022.2068249.
- [15] L. Yao *et al.*, "Applied Surface Science Comparison of sizing effect of T700 grade carbon fiber on interfacial properties of fiber / BMI and fiber / epoxy," vol. 263, pp. 326–333, 2012.
- [16] F. Liu, Z. Shi, and Y. Dong, "Improved wettability and interfacial adhesion in carbon fibre/epoxy composites via an aqueous epoxy sizing agent," *Compos. Part A Appl. Sci. Manuf.*, vol. 112, pp. 337–345, Sep. 2018, doi: 10.1016/j.compositesa.2018.06.026.
- [17] T. Wang *et al.*, "Improving the Interfacial Adhesion of Carbon Fiber/Polyether Ether Ketone Composites by Polyimide Coating," *ChemistrySelect*, vol. 5, no. 19, pp. 5507–5514, 2020, doi: 10.1002/slct.202001232.
- [18] G. Jacobsen and W. C. Schimpf, "PROCESS DEVELOPMENT AND CHARACTERIZATION OF STRETCH BROKEN CARBON FIBER MATERIALS."
- [19] D. S. R. L. Zhang, Y. D. Huang, N. Li, L. Liu, "Effect of the Concentration of the Sizing Agent on the Carbon Fibers Surface and Interface Properties of Its Composites," *J. Appl. Polym. Sci.*, vol. 116, no. 5, pp. 2658–2667, 2010, doi: 10.1002/app.
- [20] R. A. Yoni Shchemelinin, Jared W. Nelson, Cecily Ryan, Dilpreet Bajwa, Douglas Cairns, "Bulge and dome testing to investigate the formability of continuous and stretch broken carbon fiber prepreg laminates," *CAMX Anaheim CA*, 2022.
- [21] D. N. Matthew C. Egloff, Roberta Amendola, Douglas Cairns, Dilpreet Bajwa, Cecily Ryan, Bridget Powers, "A Predictive Forming Model for Stretch-Broken Carbon Fiber Composites as a Function of State of Stress, Forming Rate, and Resin Viscosity," *CAMX Anaheim CA*, 2022.
- [22] C. R. Dalton B. Nold, Dilpreet Bajwa, Douglas Cairns, Roberta Amendola, Cecily Ryan, "Tensile Properties of Stretch Broken Carbon Fiber Prepreg," *CAMX Anaheim CA*, 2022.
- [23] D. C. Tasnia J. Nur, Zachary T. White, Brendon Bossert, Matthew C. Egloff, Cecily Ryan,

Dilpreet Bajwa, Roberta Amendola, “Novel experimental unit to evaluate inter-ply and tool-ply friction in carbon fiber reinforced polymer composites prepregs,” *CAMX Anaheim CA*, 2022.

APPENDICES

APPENDIX A

STRETCH RATIO AND NIP FORCE

Effect of Stretch RatioSizing Deposition as a Function of Stretch Ratio

The relationship between sizing deposition and stretch ratio is presented in Figure A.1.

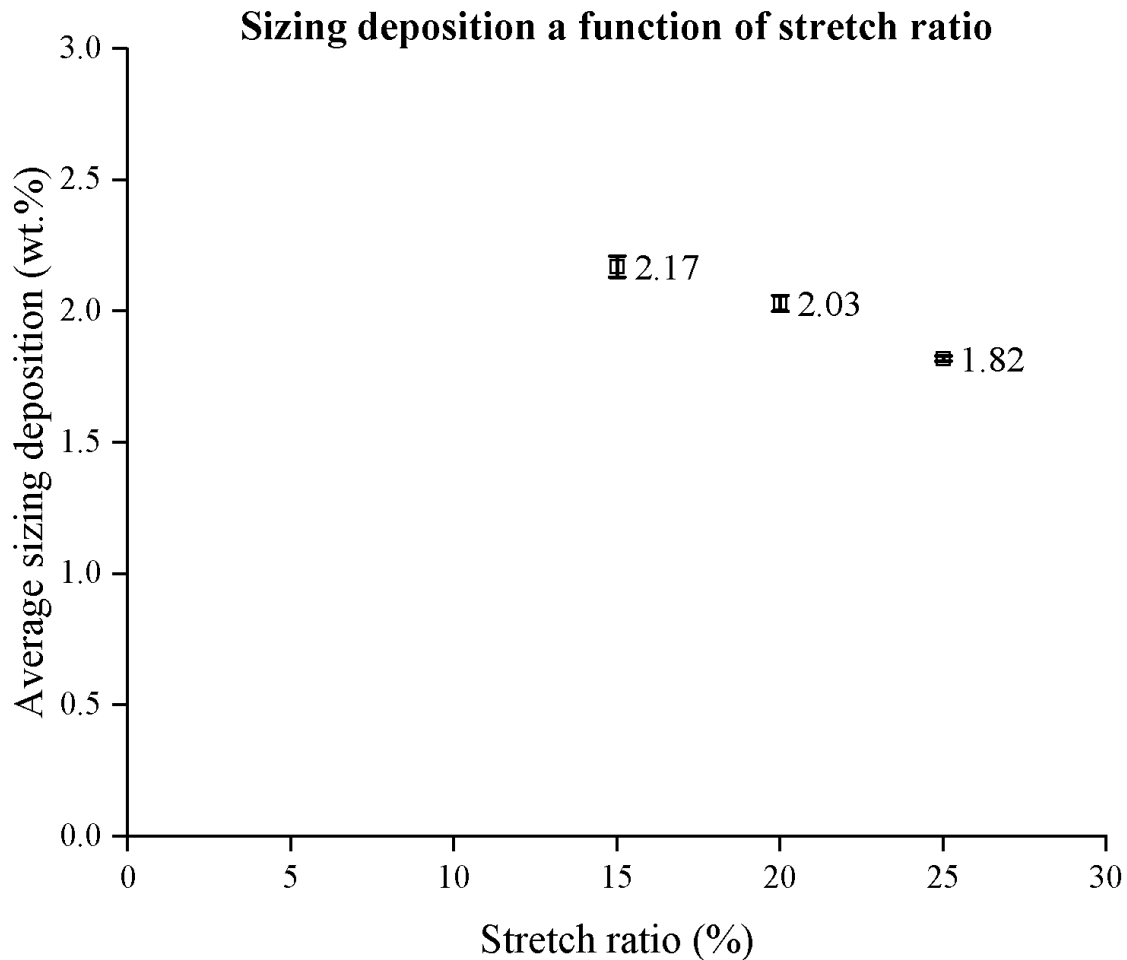


Figure A.1: Sizing deposition as a function of stretch ratio. Each point is an average value of three readings, error bars show the standard deviation.

The average sizing deposition on the tow having 15% stretch ratio is 2.17 wt.% (0.04). When the stretch ratio increases to 20%, the average sizing deposition came out to be 2.03 wt.% (0.03). Finally, for tows having 25% stretch ratio, the sizing deposition is 1.82 wt.% (0.01). It is

evident that the amount of sizing deposited on the tow decreases with an increase in the stretch ratio. The reason for observing this trend is likely due to the change in mass per unit length of the tow. The change in mass per unit length of the tow with change in stretch ratio is shown in Figure A.2.

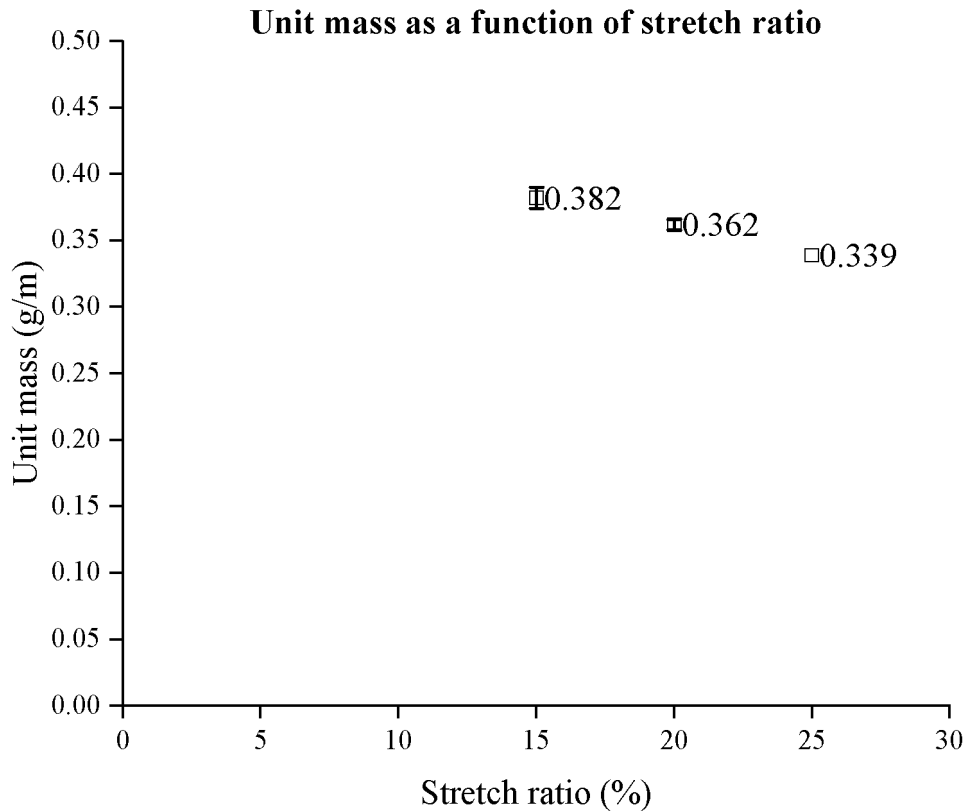


Figure A.2: Unit mass of desized tows as a function of stretch ratio.

For the stretch ratio of 15%, the unit mass of the desized tow is 0.382 g/m (0.008). When the stretch ratio is increased to 20%, the unit mass of the tow decreased to 0.362 g/m (0.004). When the stretch ratio was further increased to 25%, the unit mass of the desized tow was 0.339 g/m (0). Increasing the stretch ratio decreases the unit mass of the tow. These results indicate that

changes in sizing deposition are linked to changes of tow unit mass and that increasing the tow stretching might potentially reduce the effective cross section area of the tow.

Tow Tenacity as a Function of Stretch Ratio

For the evaluation of tow tenacity, a temperature-controlled tow tenacity test setup was used. The goal of this assessment was to determine the effect of stretch ratio on back tension ability and formability of the MSU SBCF tows. Ten tensile coupons were prepared from each batch; 5 coupons to be tested at room temperature and 5 coupons to be tested at 100°C. The gauge length of the test coupons was 6 inches. The crosshead speed during the tensile test was 0.1 in/min. Overall, 30 tensile test coupons were prepared and tested. The room temperature results give an idea about the back tension ability of the tow. The results at 100°C give an idea about the formability of the MSU SNCF tows. The effect of stretch ratio on the tow tenacity at both room and elevated temperature is shown in Figure A.3.

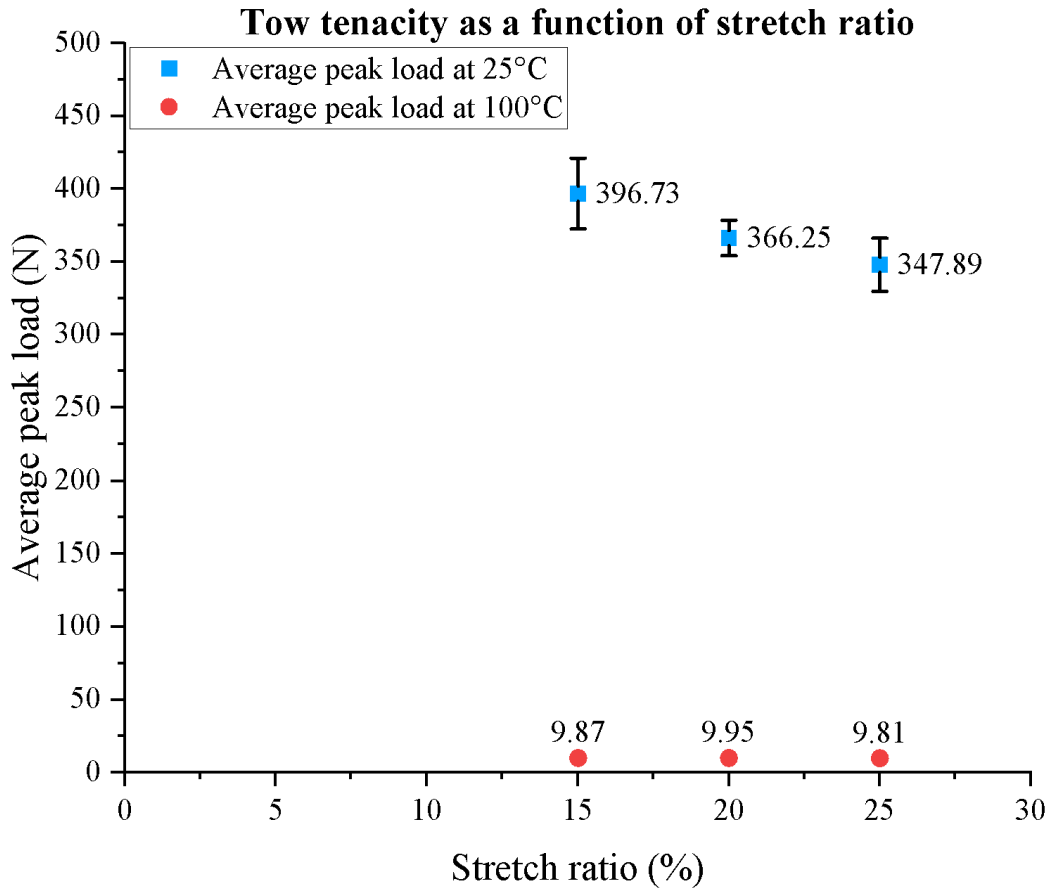


Figure A.3: Tow tenacity as a function of stretch ratio. Each point is an average value of 5 measurements. The error bar might lie within the markers.

For the stretch ratio of 15%, 20%, and 25%, the peak loads obtained at room temperature were 396.73 N (24.27), 366.25 N (12.20) and 347.89 N (18.29), respectively. A combination of slightly higher sizing deposition, higher unit mass and longer mean fiber length might have contributed to higher peak load with a decrease in stretch ratio. It is to be noted here that the 15% stretch ratio materials were the more difficult to generate in the Bobcat machine followed by 20% and 25% stretch ratio materials. A stretch ratio of 15% approaches the lower limit of the operation of Bobcat machine. For the tows having 25% stretch ratio, a peak load of 347.89 N is obtained at room temperature, which is still sufficient for the room temperature back tension

ability required during the prepreg production, although softening with elevated temperatures during production may still present a challenge.

The peak loads obtained at elevated temperature are 9.87 N (1.85), 9.95 N (1.03) and 9.81 N (0.36) for the stretch ratio of 15%, 20% and 25% respectively. All three values suggest that increasing the stretch ratio from 15% to 25% potentially will not affect the formability of MSU SBCF tows.

Effect of Nip Force

Sizing Deposition as a Function of Nip Force

The relationship between the sizing deposition on the tow and the nip force is presented in Figure A.4.

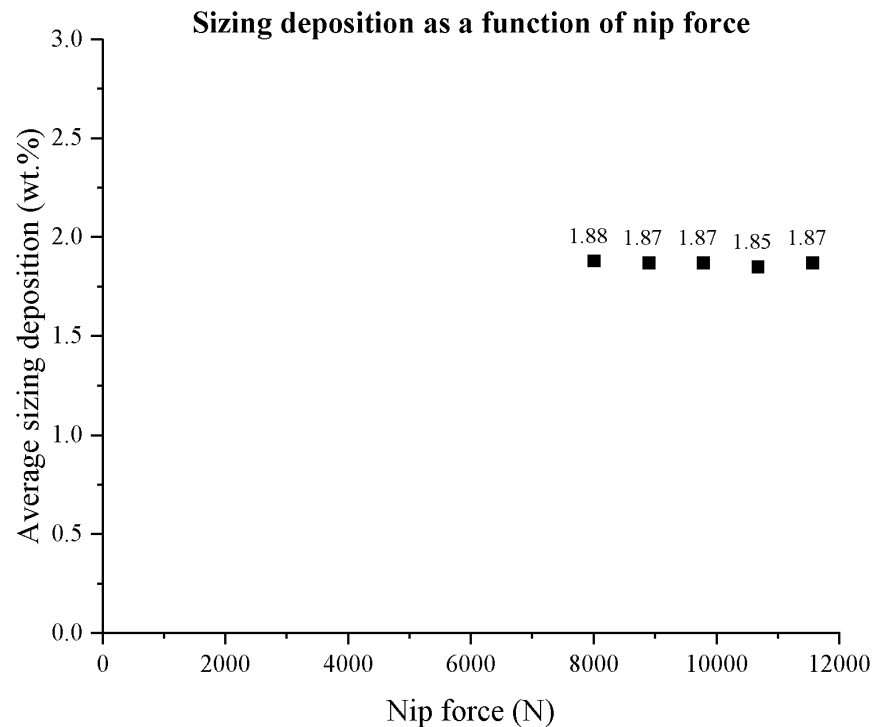


Figure A.4: Sizing deposition as a function of nip force. Each point in the graph is an average value of three measurements. The error bars might lie within the markers.

The sizing deposition was in the range of 1.85 wt.% to 1.88 wt.% for the tows having nip force ranging from 8007 N (1800 lbf) to 11565 N (2600 lbf). The result suggests that changing the nip force does not significantly change the sizing deposition on the tow. The unit mass of the desized tows shown in Figure A.5 also supports the result of sizing deposition. The unit mass decreased from 0.341 g/m to 0.330 g/m with increase in nip force from 8007 N (1800 lbf) to 11565 N (2600 lbf). This change is not significant when compared to the change in unit mass with stretch ratio.

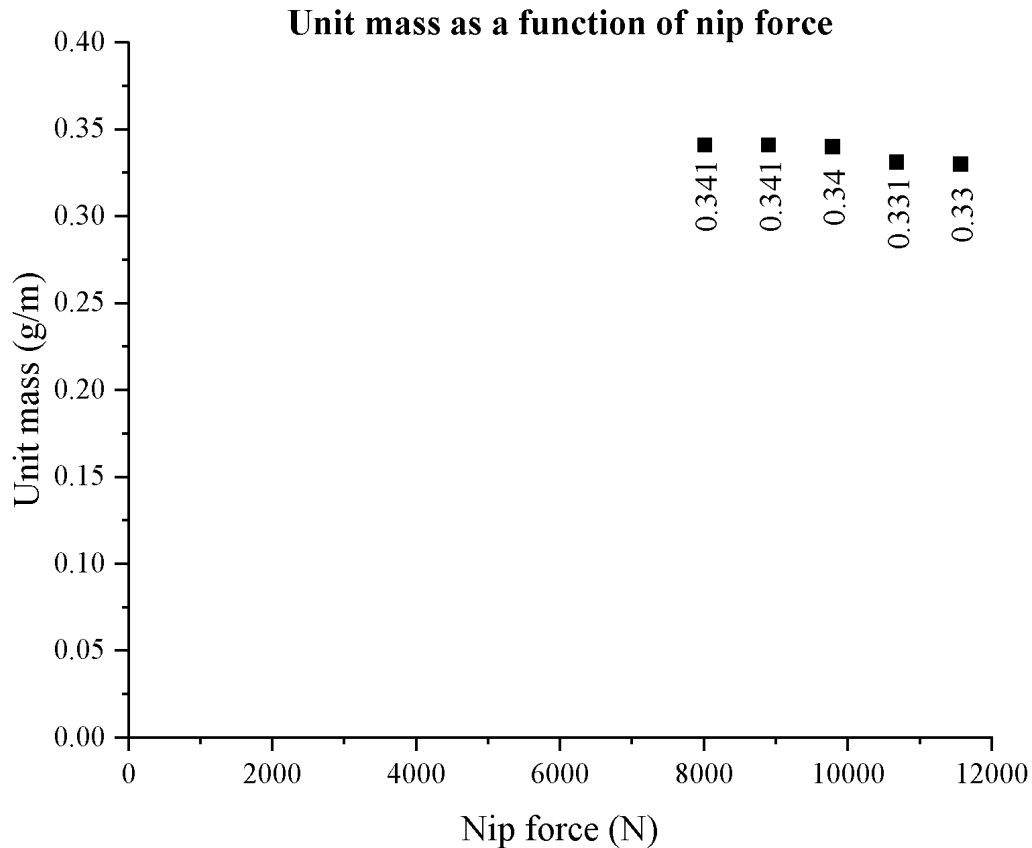


Figure A.5: Unit mass as a function of nip force. The error bars lie within the markers.

Tow Tenacity as a Function of Nip Force

The effect of nip force on tow tenacity at both room and elevated temperature is presented in Figure A.6.

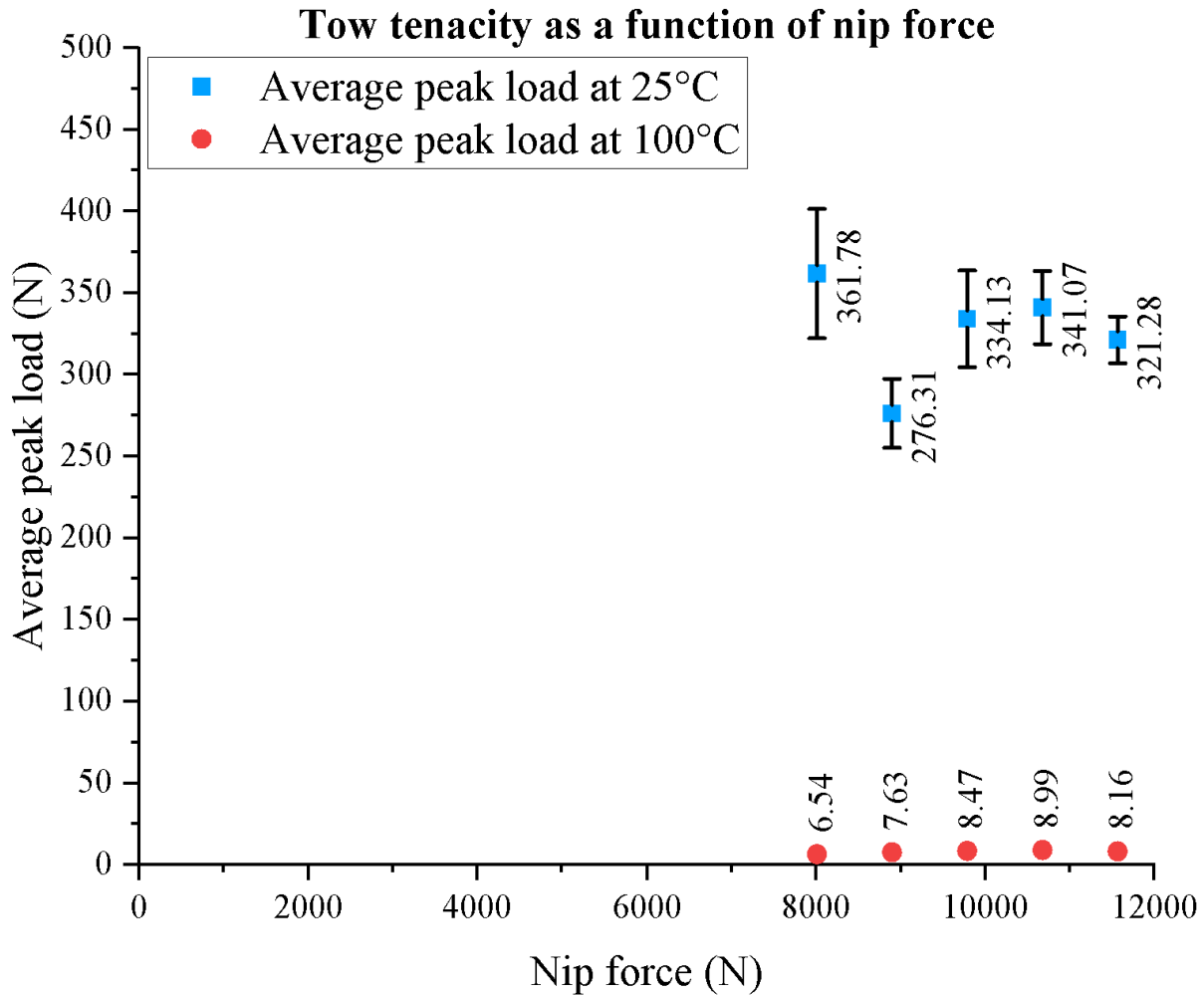


Figure A.6: Tow tenacity as a function of nip force. Each point is an average value of five measurements.

At a nip force of 8807 N, the peak load at room temperature was 361.78 N (39.53). Although the peak load obtained at this nip force is the highest, the quality of the SBCF material was not consistent. A noticeable tow splitting was observed along some section of the tow length. Also, from the fiber length distribution, approximately 43% of the individual fibers were

in the range of 31 mm to 34 mm which might have contributed to the higher overall peak load. When the nip force was increased to 8896 N, the peak load decreased to 276.31 (21.22). The reason for the lower peak load at 8896 nip force is that the tow was split into lots of individual tows. This splitting resulted in significant gaps inside the tows which compromised the quality of the MSU SBCF tows. The peak loads obtained at 9786 N and 10676 N are 334.13 N (29.54) and 341.07 N (22.30), respectively and represent a regime where good quality SBCF tows are generated. At 11565 N, the peak load decreased to 312.28 N (14.34). Again, the issue of tow splitting was observed in this batch of MSU SBCF material. From the visual observations, the nip forces of 9786 N and 10676 N generated consistent tow without any significant tow splitting while also resulting in adequately high, room temperature peak loads.

At elevated temperature, the peak load increases from 6.54 N to 8.99 N when the nip force was increased from 8007 N to 10676 N. At a nip force of 11565 N, the peak load decreased slightly to 8.16 N. Although the mean fiber length decreases with an increase in the nip force, it was expected that the fiber the peak load would also show the same trend. However, the issue of tow splitting was observed to be more severe for the 8007 N, 8896 N, and 11565 N nip force materials which adversely affects the tow tenacity at elevated temperature. Considering both room and elevated temperature peak loads, the SBCF tows generated using 10676 N nip force appears to be the most consistent material since it provides the desired back tension ability and formability without suffering from noticeable tow splitting. The samples generated using 9786 N also yielded comparable results, representing an optimal processing regime for the nip force between 9786 N to 10676 N.