MECHANICAL, THERMAL, AND MORPHOLOGICAL PROPERTIES OF CARBON FIBER REINFORCED ACRYLONITRILE-BUTADIENE-STYRENE COMPOSITES

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Approval of the Graduate School of Natural and Applied Sciences, Atılım University.

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I certify that thesis satisfies all the requirements as a thesis for the degree of Master of Science.

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ABSTRACT

MECHANICAL, THERMAL AND MORPHOLOGICAL PROPERTIES OF CARBON FIBER REINFORCED ACRYLONITRILE-BUTADIENE-STYRENE COMPOSITES

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In this thesis, short carbon fiber (CF) loaded acrylonitrile-butadiene-styrene (ABS) based composites were prepared. The four different loading ratio of CF as 5%, 10%, 15% and 20% were used and the optimum concentration was tried to obtain. Additionally, sizing layer of CF was removed and effect of the sizing to basic properties of composites was studied. Characterizations were based on mechanical, thermo-mechanical, melt-flow and morphological performances of composites.

According to test results, properties of ABS were enhanced with CF additions. 10% and 15% loading ratios were obtained as optimum level for ABS/CF composites by the help of mechanical test results. Sized CF containing composites display better results than desized ones. MFI test showed that the processability of ABS did not affected by the inclusions of CF. As the SEM images of composites were examined, it was clearly observed that CF surfaces were covered by polymer matrix thanks to sizing layer of CF and debondings were formed for desized CF filled ABS composites.

Keywords: Acrylonitrile-butadiene-styrene, Carbon Fiber, Sizing Layer, Fiber Reinforced Polymer Composites, Melt Mixing.

ÖZ

KARBON ELYAF İLE GÜÇLENDİRİLMİŞ AKRİLONİTRİL-BÜTADİEN-STİREN KOMPOZİTLERİN MEKANİK, ISISAL VE MORFOLOJİK ÖZELLİKLERİ

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Bu tez çalışmasında, kısa karbon elyaf (CF) takviye edilmiş akrilonitril-bütadien-stiren (ABS) bazlı kompozitler hazırlanmıştır. CF için %5, %10, %15 ve %20 şeklinde dört farklı ekleme oranı kullanılmıştır ve en uygun konsantrasyon saptanmaya çalışılmıştır. Buna ek olarak, CF kaplama yüzeyi ve kaplamanın kompozitlerin temel özelliklerine etkisi çalışılmıştır. Karakterizasyonlarda, kompozitlerin mekanik, ısısal-mekanik, eriyik-akış ve morfolojik performansları baz alınmıştır.

Test sonuçlarına göre, ABS’nin özellikleri CF eklemeleriyle ile artmıştır. Mekanik test sonuçları yardımıyla, %10 ve %15 yükleme oranları ABS/CF kompozitleri için en uygun düzey olarak saptanmıştır. Kaplanmış CF içeren kompozitler, kaplanmamışlardan daha iyi sonuçlar sergilemiştir. MFI testi göstermiştir ki; ABS’nin işlenebilirliği CF katılımıyla etkilenmemiştir. Kompozitlerin SEM resimleri incelendiğinde, kolayca anlaşılmaktadır ki; CF kaplama yüzeyi sayesinde CF yüzeyleri polimer matris ile çevrilmiştir ve kaplanmamış CF içeren ABS kompozitlerinde ayrılmalar oluşmuştur.

Anahtar Kelimeler: Akrilonitril-bütadien-stiren, Karbon Elyaf, Kaplama Yüzeyi, Elyaf Takviyeli Polimer Kompozitler, Eriyik Karıştırma.

To My Family

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TABLE OF CONTENTS

LIST OF FIGUERS

LIST OF TABLES

LIST OF ABBREVATIONS

ABS	-	Acrylonitrile-Butadiene-Styrene
CF	-	Carbon Fiber
DMA	-	Dynamic Mechanical Analysis
T_g	-	Glass Transition Temperature
HDPE	-	High Density Polyethylene
MFI	-	Melt Flow Index Test
PAN	-	Poly acrylonitrile
PLA	-	Poly lactic acid
PP	-	Polypropylene
SEM	-	Scanning Electron Microscopy

CHAPTER 1

INTRODUCTION

Carbon fiber reinforced polymer composites have great attention and have found various use in many application areas including transportation, construction, electronics, aerospace and defence industry. The main goal of it’s usage is the reduction of weight by replacing metal parts with carbon fiber containing polymeric structures. Research efforts and application fields based on carbon fiber and its composites are continued to grow steadily and they are considered to advanced materials in many topics.

Acrylonitrile-Butadiene-Styrene (ABS) is a trendy candidate among all of the polymers thanks to their suitable applications in 3D printing field. Research studies have been conducted to optimize and tune of basic properties of ABS copolymer in order to extent its effective use in 3D printing technology. .

In the light of these research highlights, investigations for carbon fiber reinforced ABS composites are based on the effect of sizing layer of fiber and concentration of CF. Basic properties of composites such as mechanical, thermo-mechanical, melt flow and morphological properties can give important clues for material researchers to produce a ABS based composites with desired parameters.

Melt mixing process is widely used for the production of ABS filaments with required colours for their 3D printing applications. This processing technique is also the most preferred one in order to practical optimization and obtain homogeneous dispersion.

Because of these aspects, it was aimed in this thesis study to investigate the fundamental properties of CF reinforced ABS composites by using melt mixing method. Comparisons were done according to influence of concentration and sizing layer of CF samples to their ABS based composites.

CHAPTER 2

BACKGROUND INFORMATION

2.1 Polymeric Composites

Polymer composite materials are defined as composing of two or more different phases including polymer matrix and additives or fillers. Additives play key role for polymer matrix composites that they lead improvements of several performance of polymer [1].

Practical processing and low cost requirements during production of polymer composites make them favourable material in many fields. Lowering weight and cost of the varied parts can be possible by using these promising materials. Replacements of metal components with polymer composite structures become very important due to extension of their application fields primarily in aerospace, defence and transportation industry [2].

2.2 Acrylonitrile-Butadiene-Styrene

The structure of ABS copolymer is composes of polybutadiene groups dispersed in styrene and acrylonitrile parts. Polybutadiene is the elastomeric unit whereas styrene and acrylonitrile show thermoplastic behaviour. These thermoplastic phases are responsible for mainly the toughness, dimensional stability and chemical resistance properties of ABS copolymer. The proportions of these parts exhibit varied ratios as nearly 15% to 35% for acrylonitrile, 5% to 30% for butadiene and 40% to 60% for styrene units in the main structure of ABS [3].

ABS has been found different applications such as toys, sporting goods, telephone panels, helmets, electronic parts and home equipments. In addition to these main application areas, ABS becomes popular recently for their effective usage in 3D printing technology [4,5].

2.3 Carbon Fiber

Carbon fibers are classified as advanced materials because of their extra ordinary performances including high strength and high modulus in addition to low weight and low density. Their higher production cost is the most non-advantageous point that causes some limitations on their usage [6].

The CF filaments compose of 12,000 or 24,000 individual filaments which they provide ultimate strength to deformations. There are different kinds of CFs but poly acrylonitrile (PAN) based CF is the most favourable grade used in many fields. Production of CF consists of several steps such as carbonization of PAN fibers, oxidation, winding and rowing. At the last step of their production, CFs are subjected to polymer emulsion in order to cover a polymeric layer of CF surface which is named as sizing process. This process leads to improve compatibility of CF with polymers in the case of their composite production applications [7].

Commercial CF grades are mostly purchased in the form of rollers. These types of CFs are used in thermoset polymeric composite productions. Additionally, CF can be chopped into different length and provide reinforcing in thermoplastic composite materials applications [8].

Short or chopped CF reinforced polymer composites have gain importance in recent years thanks to their practical processing and producing light weight and strong composite structures. They found effective usage in especially automotive industry for the weight reduction purposes by replacing them with heavier metal parts [9,10].

2.4 Processing Methods

Melt mixing is the process that is applied widely for production of thermoplastic composites. This process is used with extruder machine which composes feeder, screw, barrel and die zones. The screws provide several functions including transportation of polymer pellets, melting these pellets and dispersing added fillers homogeneously in the molten polymer matrix. The final form of the material after extrusion can be shaped as pellet, chips, sheet, rod, film, tube or pipe which is tuned by the die design [10].

Injection molding is the conventional method to give desired shape for the polymers. Plastics are molten in barrel and they are injected repeatedly by high pressure to a steel mold in this process. after cooling period polymer gains shape of the mold and it released from the mold [11].

2.5 Literature Review

There have been several research studies that conducted on short CF containing composites in the literature.

Cumeiro and Muia investigated the effect of CF type and concentration to rheological behaviour polypropylene (PP) based composites [11]. They observed that reinforcing capability of CF was mainly depend on adhesion between the fibers and polymer matrix.

Karsli and Aytac produced short CF filled PP composites by melt mixing method and they investigated that melting temperature of PP based composites displayed no significant change with increasing CF concentration [12].

Rezaei et al. investigated that short CF addition caused improvement on strength and stiffness of PP/CF composites [13,14].

Wan et al. fabricated the CF reinforced poly lactic acid (PLA) composites. They investigated that increase in interfacial adhesion between CF and polymer matrix yields improvement on mechanical properties of composites [15].

Savas et al. studied the effect of CF addition to high density polyethylene (HDPE) composites [16]. They observed that impact strength of PP increases with CF content and using compatibilizers.

Tayfun et al. performed the production of CF filled polyurethane elastomer composites and they found that mechanical properties of composites enhances gradually with increase in CF ratio [17].

Jiang et al. performed CF containing polyurethane composites. They stated that surface coating of CF caused remarkable increase on tensile strength of composites [18].

CF containing ABS composites were also studied in the literature. Investigations of these composites mostly dealt with the fire performance.

Love et al. fabricated short CF loaded ABS composites and they displayed that tensile strength and modulus of ABS increases with the addition of CF [19,20].

Yang et al. stated that 10% filling ratio of CF caused remarkable increases for flexural and tensile strength values of ABS [21].

Huang et al. investigated that CF addition to ABS results in improvement for strength and modulus values of neat ABS [22].

Hull et al. showed that processing conditions of ABS filaments are significantly affected by CF content in the case of 3D printing applications [23].

Quan et al. demonstrated that short CF reinforced ABS filaments can be optimized for the manufacturing of composite parts by using 3D printing [24].

2.6 Aim of the Study

In this thesis work, the effects of concentration and sizing layer of short CF to mechanical, thermo-mechanical, melt flow and morphological properties of ABS based composites were investigated. The optimum amount of CF for ABS copolymer was tried to obtain by the help of experimental data including mechanical tests. Results of sized and desized CF containing composites were compared in order to evaluate the effect of sizing layer of CF samples.

CHAPTER 3

EXPERIMENTAL

3.1 Materials

In this study, ABS was used as polymer matrix and short CF samples were used as reinforcing agent.

3.1.1 Acrylonitrile-Butadiene-Styrene

ABS copolymer was purchased as chip form by Lanxess, Cologne, Germany. It has the trade name of Lustran ABS M203FC.

3.1.2 Carbon Fiber

CF samples were supplied from Dowaksa, Yalova, Turkey under the trade name of Aksaca – AC 0101. It has chopped form with 3 cm length. This CF grade has surface covering of polyurethane with the sizing ratio of 1.5-2.0 %.

Commercially sized CF was annealed at 450 ˚C for 4 hours for the removal of sizing layer and this desized CF sample was named as DS-CF. Figure 1 represents the Infrared spectra of CF and DS-CF samples. It can be clearly seen from these spectra that mainly hydroxyl gruop peaks of CF around 1000 and 3700 cm^{-1} were dissapeared as compared with DS-CF sample. This finding proves that polymer layer of CF was removed after annealing process.

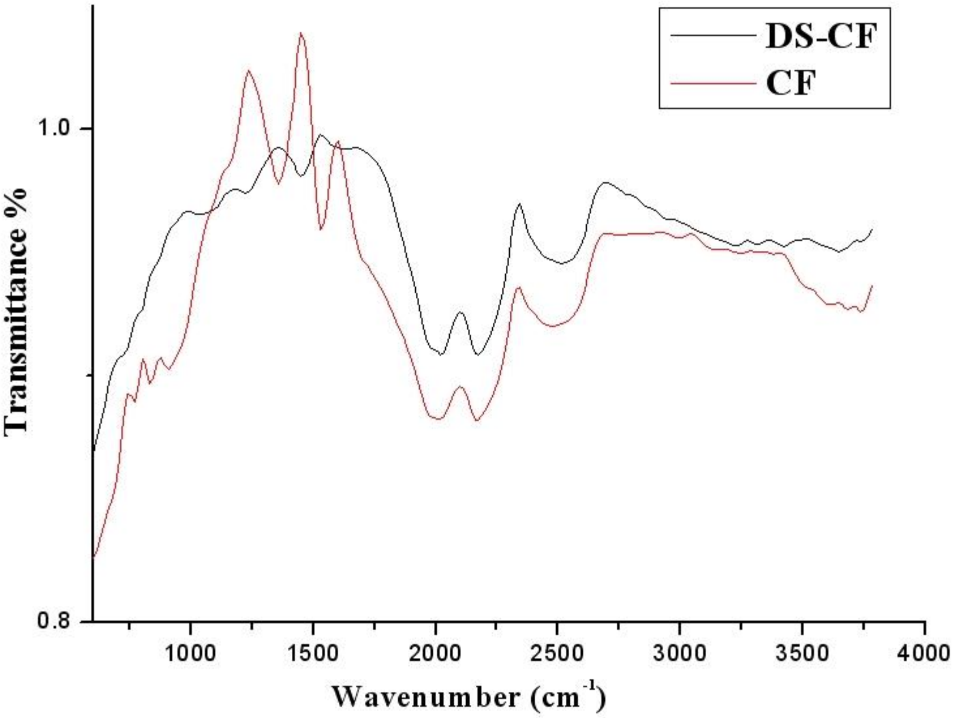

Figure 1. Infrared Spectra of CF and DS-CF

3.2 Production Techniques

3.2.1 Melt Mixing

ABS chips were dried at 100˚C for12 hours to avoid possible moisture before processing. Composites were fabricated by laboratory scale twin screw extruder (15 ml micro-compounder, DSM Xplore, Netherlands). Processing parameters applied during production of composites are listed in Table 1 and image of extruder is represented in Figure 2.

Table 1. Compounding Parameters of Composites

Parameters	**Specification**	**Unit**
Processing temperature	230	^{o}C
Mixing time	5	min
Screw speed	100	rpm

Figure 2. Lab-scale Extruder

3.2.2 Injection Molding

Composite samples obtained from extrusion process were shaped by using laboratory scale injection molding instrument (Microinjector, Daca Instruments, UK). Parameters applied during this process are listed in Table 2 and the image of micro injector is displayed in Figure 3.

Table 2. Injection Molding Parameters of Composites

Parameters	**Values**	**Unit**
Barrel temperature	210	^{o}C
Mold temperature	50	^{o}C
Injection pressure	8	Bar
Holding time	5	min

Figure 3. Injection Molding Instrument

Test specimens were shaped as standard dog-bone sample during injection molding process. This test samples have dimensions of 7.6×2.0×80 mm^3 as given in Figure 4. Gauge length of dog-bone shaped test samples is 50 mm.

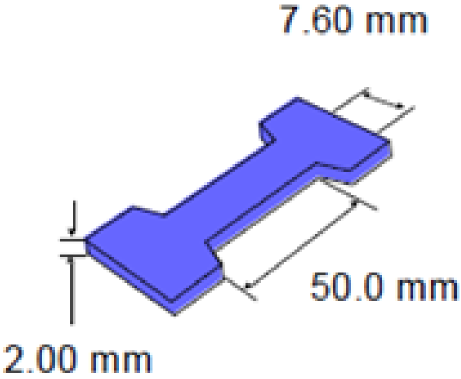

Figure 4. Dimensions of Tensile Test Sample

3.3 Characterization Methods

Characterizations of samples were done according to related standards for each methods. All of the experimantal data were recorded as an avarage of minimum required number of specimens.

3.3.1 Tensile Test

Tensile test measurements of ABS and composites conducted using Instron 5565A tensile testing machine with the accordance of ASTM D-638 standard. Crosshead speed of 5 mm/min and 5 kN load cell were applied. Tensile strength, elongation at break and tensile modulus parameters were recorded as an average of minimum five samples. Representative image of tensile testing machine is givenn in Figure 8.

Figure 5. Tensile Testing Machine

3.3.3 Impact Test

Unnotched Izot impact test was carried out with Coesfeld-Material impact tester. Impact energy measurements were done according to ASTM D256 standard by using a 4 J pendulum. Recorded values represent an average value of at least five samples with standard deviations. Figure 6 represents the impact tester used in this study.

Figure 6. Impact Tester

3.3.4 Dynamic Mechanical Analysis (DMA)

DMA measurements was performed with DMA 8000, Perkin Elmer dynamic mechanical thermal analyser Test specimens obtained from injection molding with dimensions of 7.6x2.0x50 mm^3 were subjected to DMA test in dual cantilever bending mode at the temperature range of -120°C to 140°C with 1 Hz constant frequency and heating rate of 5 °C/min. Figure 7 shows the DMA analyzer used in this work.

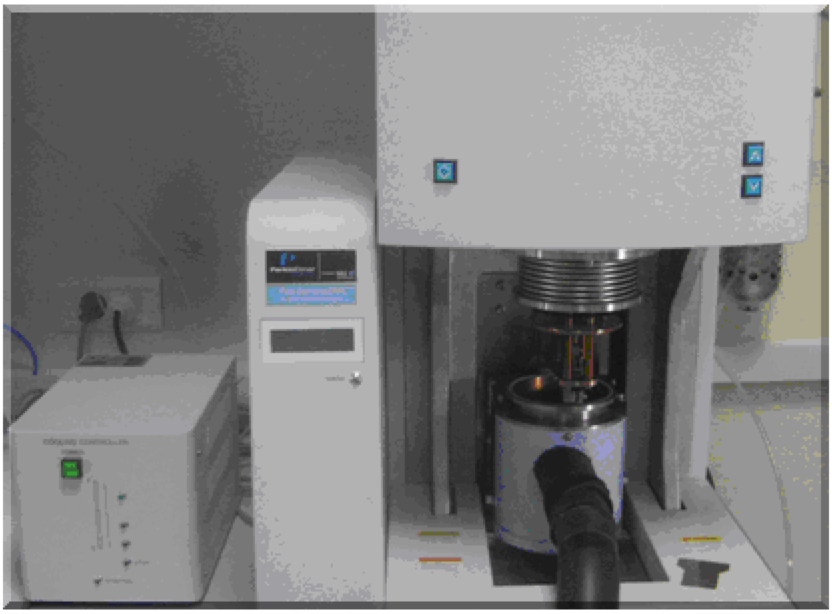

Figure 7. DMA Analyzer

3.3.5 Melt Flow Index Test (MFI)

MFI measurements were performed by using melt flow indexer (Coesfield Meltfixer LT) equipment which is represented in Figure 8. Tests were done under a standard load of 2.16 kg at the processing temperature of ABS and composites (230 ^{o}C). MFI values of each sample were calculated as an average value of at least eight samples with standard deviations.

Figure 8. Melt Flow Indexer

3.3.6 Scanning Electron Microscopy (SEM)

Morphological characterizations of composites were examined by JSM-6400 Electron Microscope. Surfaces of fractured samples were obtained from the impact test and these surfaces coated with a thin layer of gold for the aim of creating conductive surfaces. SEM micro-images were taken at x500, x1000 and x2000 magnifications.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Tensile Properties

Tensile test data of test samples including tensile strength, percent elongation at break and tensile modulus are listed in Table 4. Additionally, strength versus extension curves of composites were given on Appendix as Figure A.1, Figure A.2, Figure A.3 and Figure A.4.

Table 3. Tensile Test Data of ABS and Composites

Samples	**Tensile Strength** (MPa)	**Elongation at Break** (%)	**Tensile Modulus** (GPa)
ABS	35.4±1.7	7.1±0.8	0.92±0.3
ABS/5 DS-CF	39.7±0.7	2.8±0.3	2.20±0.2
ABS/10 DS-CF	52.6±1.1	2.1±0.2	3.32±0.2
ABS/15 DS-CF	50.7±0.9	2.2±0.2	3.34±0.3
ABS/20 DS-CF	44.9±1.0	1.3±0.3	3.80±0.2
ABS/5 CF	57.9±0.9	3.8±0.2	2.53±0.3
ABS/10 CF	74.9±1.3	5.2±0.4	2.65±0.2
ABS/15 CF	88.2±0.7	4.8±0.3	4.03±0.2
ABS/20 CF	98.9±1.1	4.4±0.2	4.55±0.3

According to Table 4, it can be concluded that tensile strength and tensile modulus of unfilled ABS were improved with increase in CF concentration. As a comparison, far higher strength, elongation and modulus values were observed for sized CF additions than desized CF. Nearly two fold and three fold increments were achieved with 10% and 20% loading levels of sized CF, respectively. 10% and 15% adding ratios of desized CF filled gave remarkably higher strength values among other DS-CF additions. These results are in accordance with similar studies [21,22] in which 10% CF content was found as optimum ratio for ABS based composites.

The highest tensile modulus values were obtained for the highest loading ratios for both CF and DS-CF. Percent elongation of neat ABS exhibited reduction after CF additions. DS-CF additions caused sharper decrements as compared with CF.

4.2. Impact Test

Impact strength values are represented as the function of CF loading ratios in the Figure 9. This figure implies that impact strength of neat ABS increased proportionally with increase in CF content.

Desized CF loaded composites displayed slightly lower impact strength values as compared to CF. This may due to beter adhesion of CF with ABS matrix relative to DS-CF.

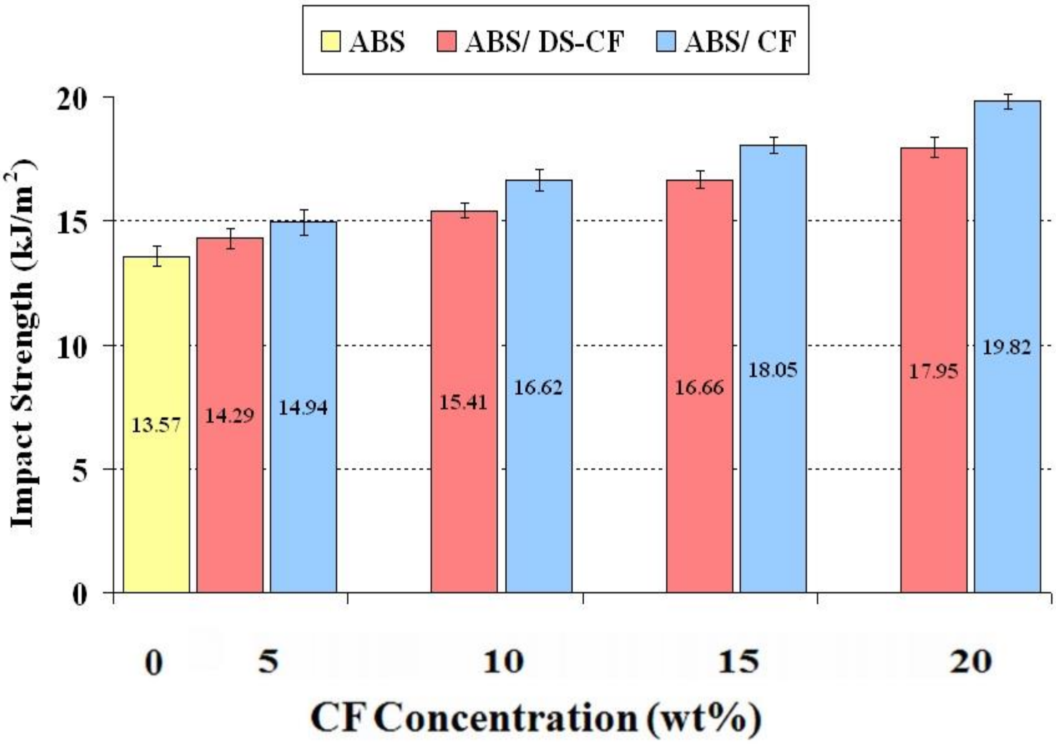

Figure 9. Impact Test Results of ABS and Composites

4.3. Thermo-mechanical Properties

Storage modulus versus temperature curves of ABS and composites were given in Figure 10.

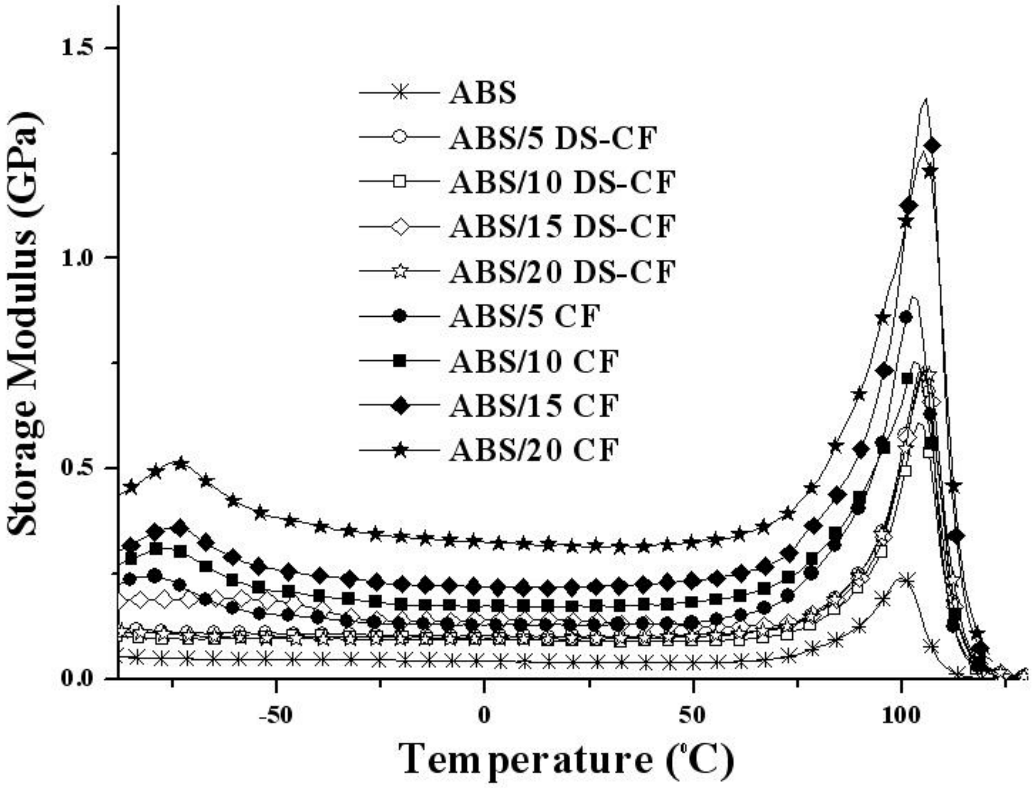

Figure 10. Storage Modulus vs. Temperature Curves

As the Figure 10 represents, the highest storage modulus is observed for 15% CF containing composite. ABS/20 CF composite yields nearly identical storage modulus with ABS/15 CF composite. It is clealy seen from the curves that sized CF samples display remarkably higher modulus values than DS-CF samples.

Tan δ curves versus temperature curves of ABS and composites were shown in Figure 11.

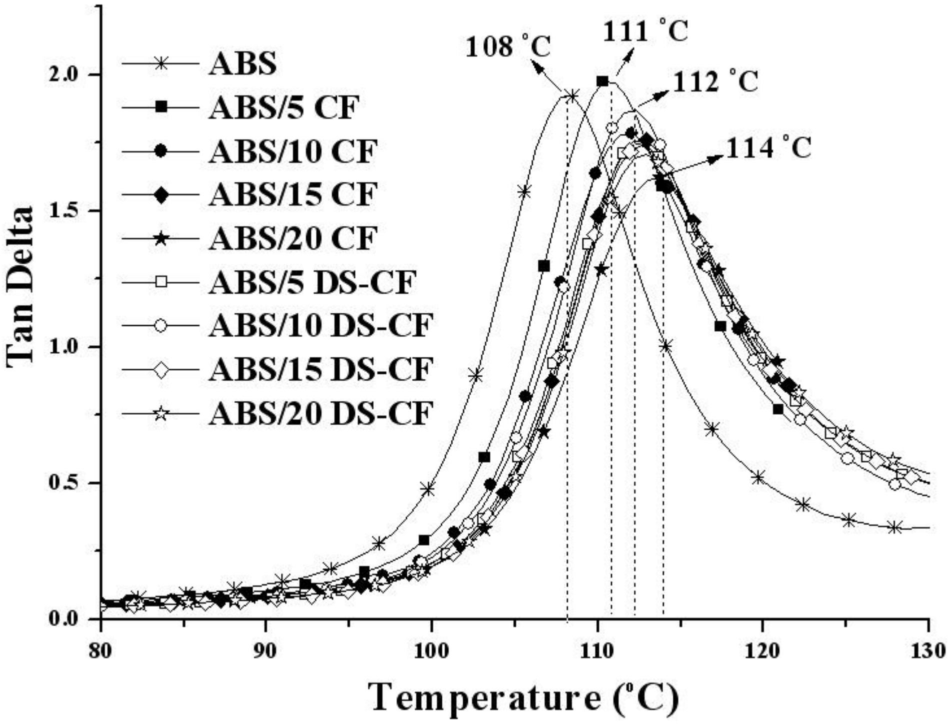

Figure 11. Tan δ vs. Temperature Curves

According to Figure 11, all of the CF additions caused shifting of glass transition temperature (T_g) of neat ABS to the higher temperatures. Improvement of nearly 6 units on T_g of ABS was observed for the highest amount of CF (20%). DS-CF containing composites exhibited almost identical T_g values each other.

The shifting of T_g values are mainly related with the vibration-damping property of the composites as they subjected to constant deformation during the DMA measurements [25].

4.4. Melt Flow Behaviour

MFI test gives information about viscosity cand processability of plastics and polymer composites. MFI values of unfilled ABS and composites are given in Figure 12.

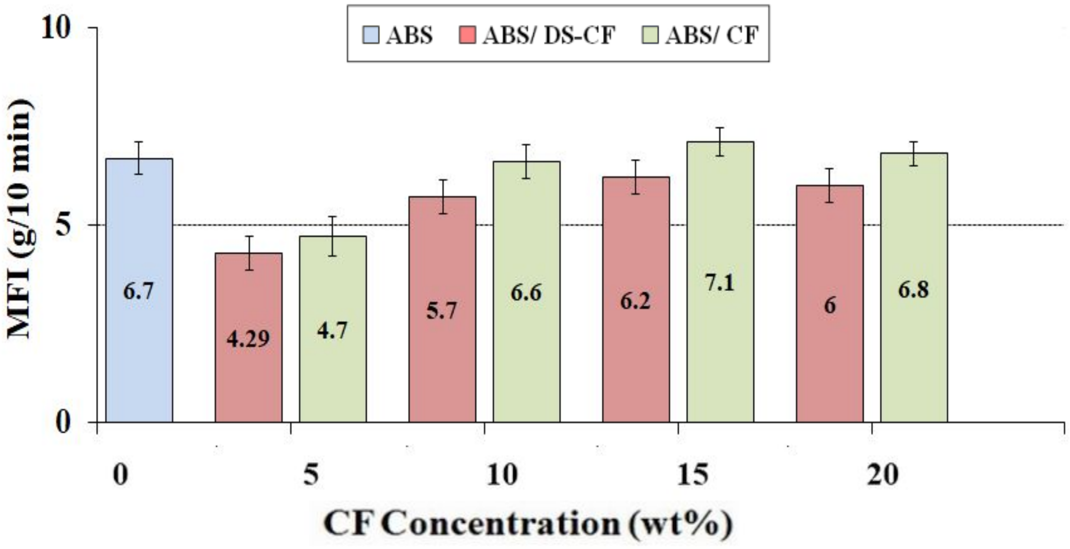

Figure 12. MFI Values of ABS and Composites

Figure 12 reveals that DS-CF containing composites result in slightly lower MFI values with respect to CF containing ones. CF inclusions caused no remarkable changes for blank ABS. This result indicates that production of CF reinforced ABS composites can be operated without acquanted obvious processing problems.

4.5. Morphological Characterization

Fractured surfaces of composites were used in SEM analysis in order to examine their morphological characterization. Micro-images of surfaces were taken at x500, x1000 and x2000 magnifications.

SEM micro-images of 5, 10, 15 and 20 % of both CF and DS-CF containing composites are displayed in Figure 13, Figure 14, Figure 15 and Figure 16, respectively.

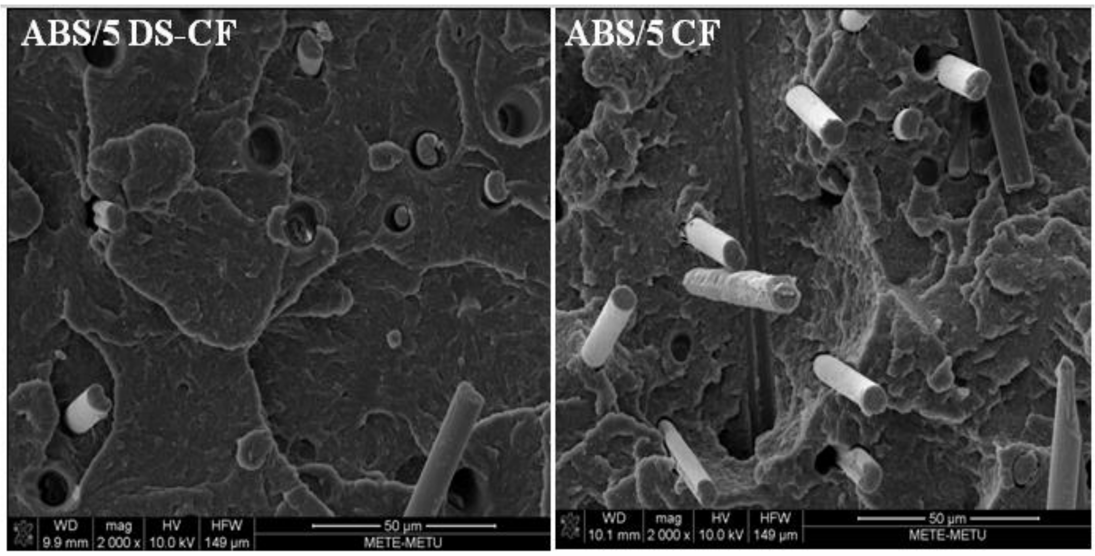

Figure 13. SEM micro images of ABS/5 CF and ABS/5 DS-CF Composites

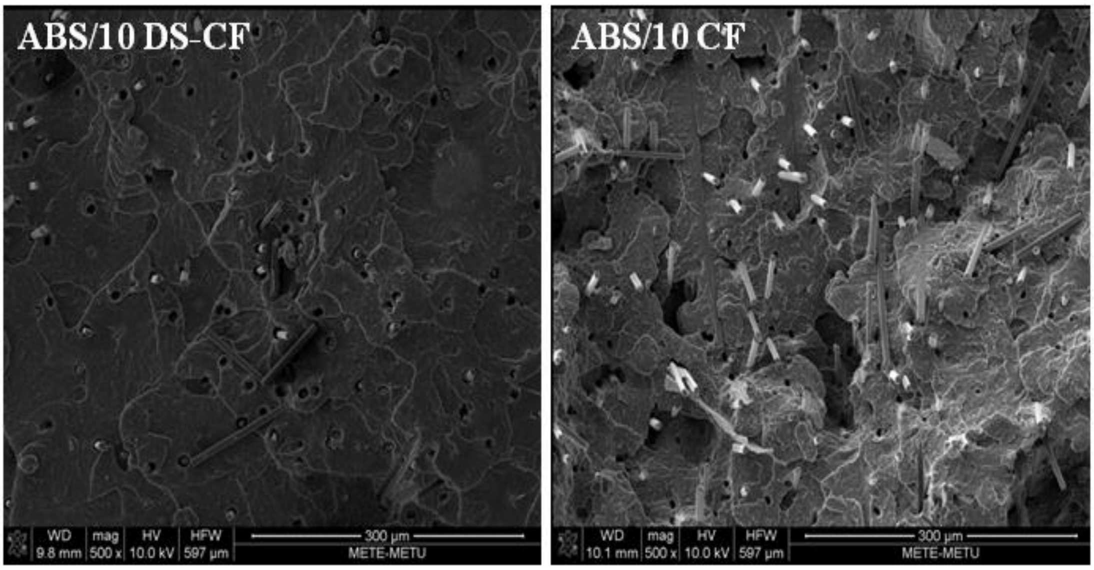

Figure 14. SEM micro images of ABS/10 CF and ABS/10 DS-CF Composites

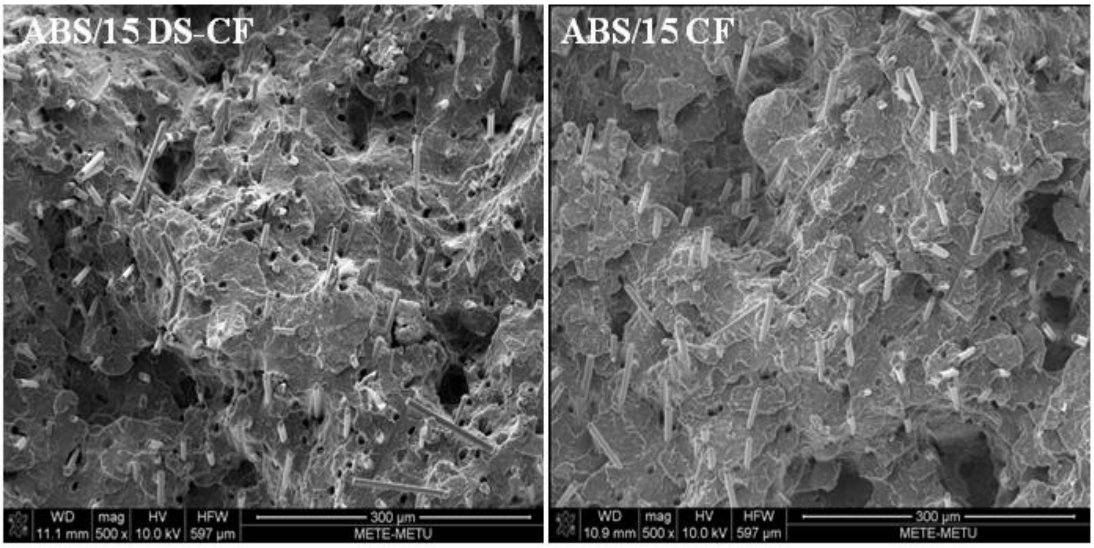

Figure 15. SEM micro images of ABS/15 CF and ABS/15 DS-CF Composites

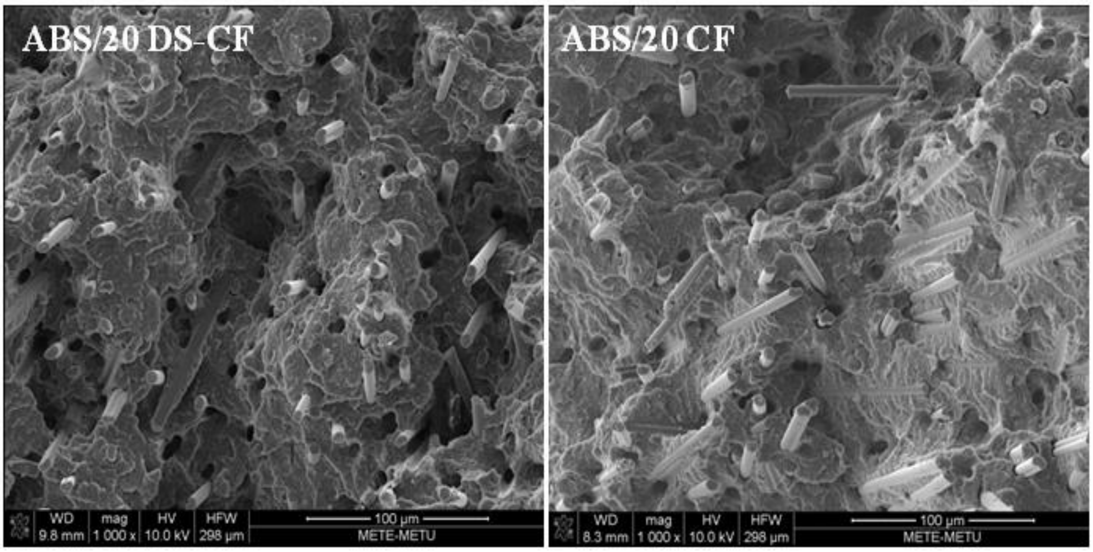

Figure 16. SEM micro images of ABS/20 CF and ABS/20 DS-CF Composites

SEM micro images of selected composites shows that sized CFs disperse uniformly into ABS matrix. This may due to the improvement of compatibility of CF with ABS matrix by the help of commercially sized surface of CF.

It can be seen from the figures that, sized CF surfaces were covered by polymer phase and almost no debonding formation was observed for CF filled ABS composites.

On the other hand, large gaps were formed between DS-CF and ABS matrix which may stem from the lack of adhesion of inert DS-CF surface to polar ABS matrix.

As the CF contents were compared by examination of micro images, it can be observed that fibers were oriented well in one direction for 5% and 10% CF contents. Further addition of CF caused to start disorientations of fibers into polymer matrix. This observation may be the reason of obtaining optimum CF content of 10% on tensile test results.

All of these findings from the SEM analysis proved the previous test results and they can be strong evidence for the effect of sizing layer of CF to ABS matrix.

CHAPTER 5

CONCLUSION

In this thesis study, short CF containing ABS based composites were prepared using extrusion process. Basic properties of composites were examined by tensile and impact tests, DMA, melt flow index measurements and SEM techniques. Effects of surface covering and concentration of CF to ABS matrix were investigated.

According to tensile test results, tensile strength and modulus of neat ABS increased with the incorporation of the CF at higher filling ratios in which 10% and 15% concentrations displayed higher values. On the other hand, percent elongation of ABS decreases drastically with CF loadings.

Impact test revealed that CF additions caused remarkable improvement for impact strength values of ABS. Sized CF filled composites exhibited relatively higher impact strength than desized CF.

DMA analysis showed that storage modulus and glass transition temperature of neat ABS were enhanced with the addition of CF at higher amounts. Loading levels of 15% and 20% CF gave higher modulus values among all of the composites. Sized CF inclusions resulted in higher storage modulus and glass transition temperature values with respect to desized ones.

According to MFI test results, CF additions caused no remarkable changes for MFI value of neat ABS. It is concluded that there will be no obvious problems in the case of processing conditions of composites.

As the SEM micro-images of composites were examined, it was observed that sized CF surfaces were covered by polymer matrix which arises from the compatible interfaces CF between ABS copolymer. Debonding formations were clearly seen between DS-CF and polymer phase due to the their lack of adhesion each other.

As an overall conclusion, sized CF samples display better results with respect to desized CF samples.

Generally composites with 10% and 15% CF loading ratios showed remarkably higher mechanical and thermo-mechanical properties among other composites. These concentrations would be more suitable for the applications dealing with ABS based composites.

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APPENDIX

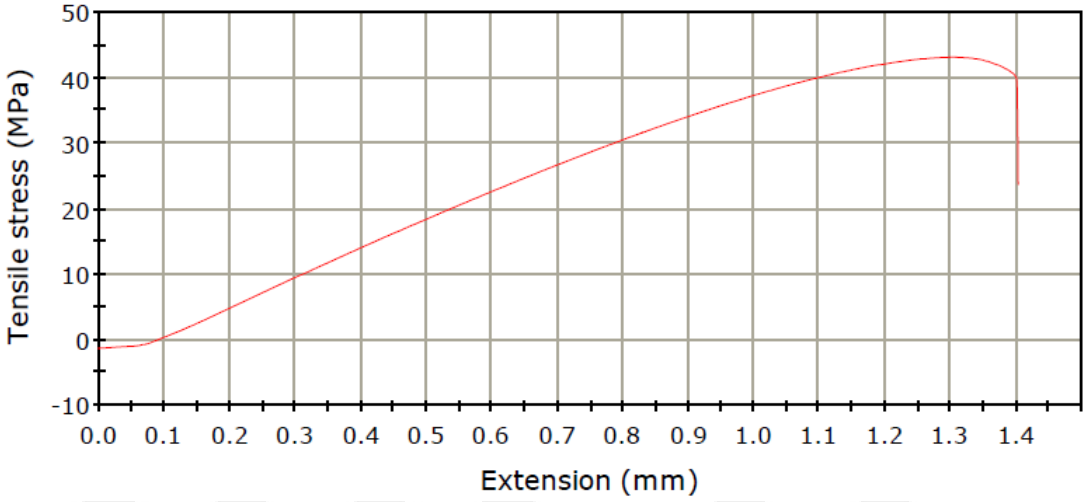

Figure A1. Stress-Strain Curve of ABS/5 DS-CF Composite

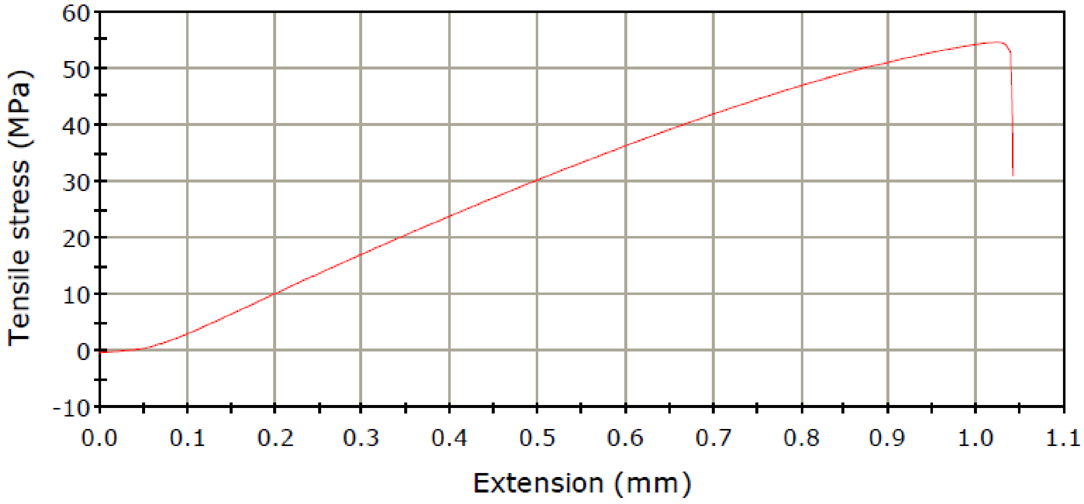

Figure A2. Stress-Strain Curve of ABS/10 DS-CF Composite

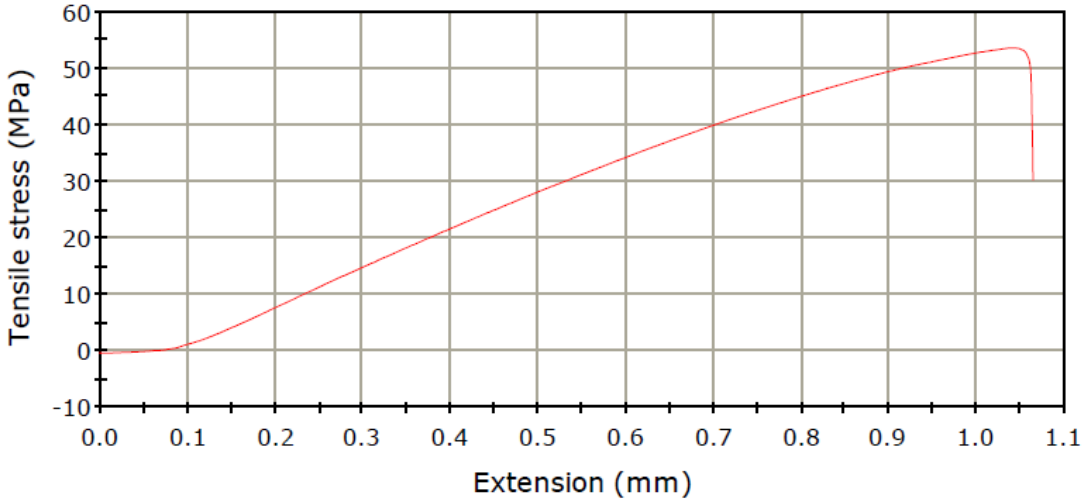

Figure A3. Stress-Strain Curve of ABS/15 DS-CF Composite

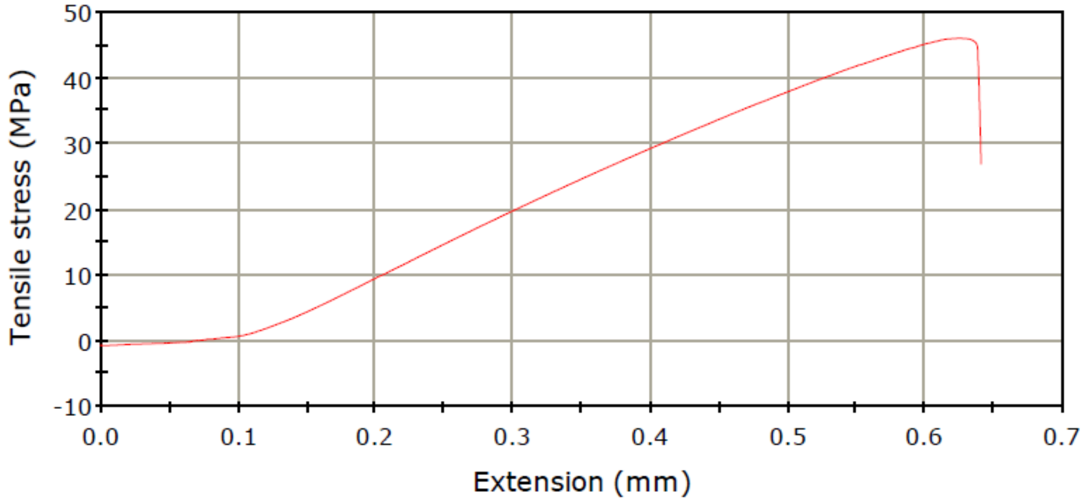

Figure A4. Stress-Strain Curve of ABS/20 DS-CF Composite

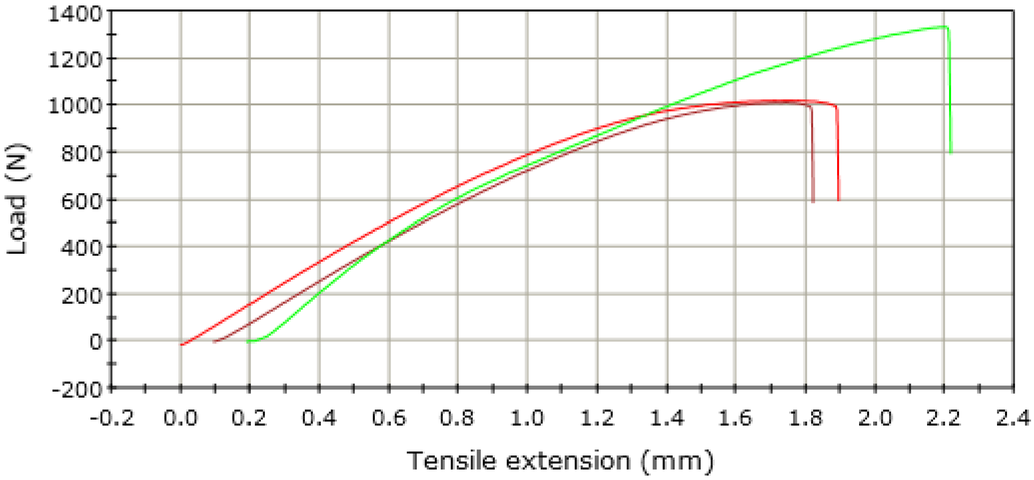

Figure A5. Stress-Strain Curve of ABS/5 CF Composite

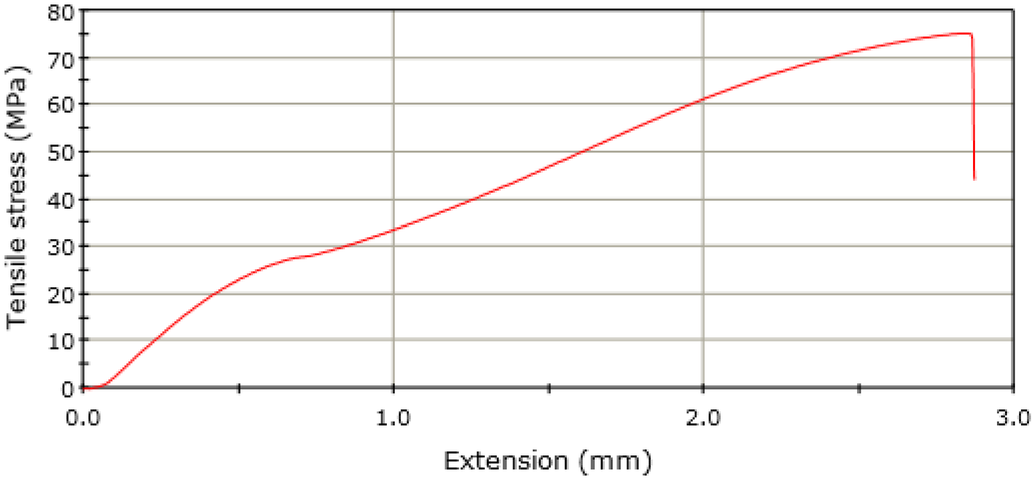

Figure A6. Stress-Strain Curve of ABS/10 CF Composite

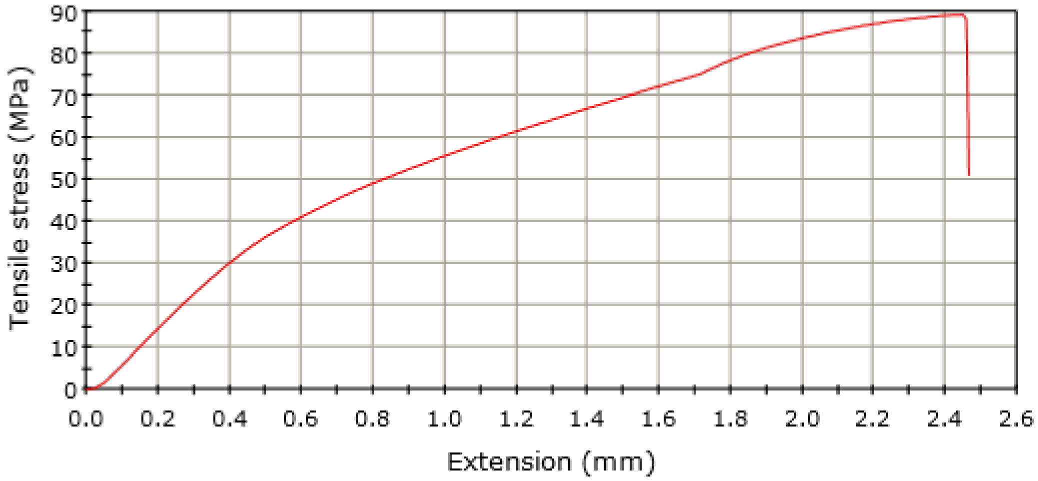

Figure A7. Stress-Strain Curve of ABS/15 CF Composite

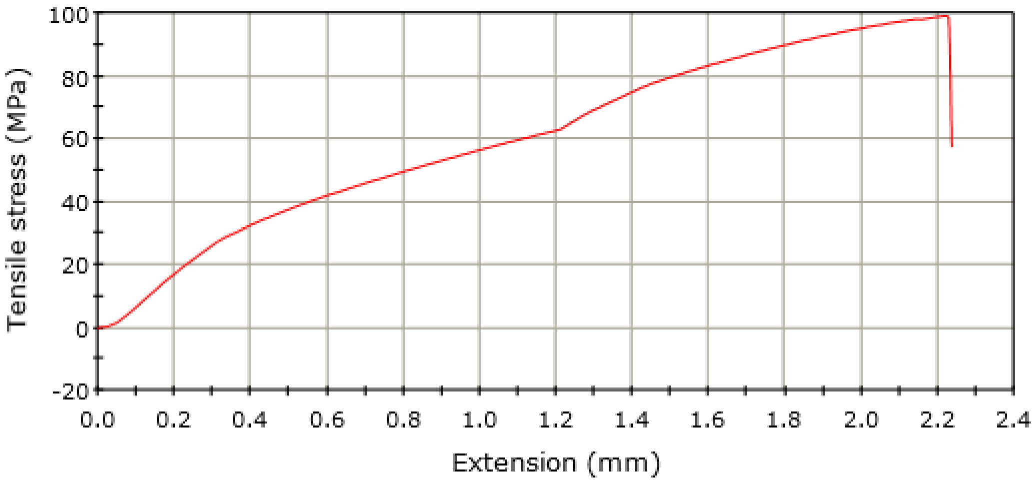

Figure A8. Stress-Strain Curve of ABS/20 CF Composite