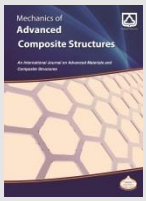
Mechanics of Advanced Composite Structures 6 (2019) 239 - 247

Semnan University

Mechanics of Advanced Composite Structures

journal homepage: http://MACS.journals.semnan.ac.ir



Study on Compression and Flexural Behavior of ABS-SiO_2 Polymer Matrix Composite Fabricated by Hot Extrusion

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PAPER INFO

Paper history:
Received 2018-03-11
Received in revised form 2019-02-21
Accepted 2019-04-26

Keywords:
Acrylonitril-Butadiene- Styrene
Nano-Silica
Extrusion
Compression
Flexural Behavior

ABSTRACT

In the present work, an experimental study was done to prepare Acrylonitrile-Butadiene-Styrene matrix composite reinforced by Nano-silica particles. The hot extrusion method was utilized here to fabricate the composite specimens used for flexural and compression tests. In order to identify the effect of SiO_2 content and extrusion temperature, 12 experiments have been carried out and the obtained results were discussed according to scanning electron microscopy (SEM) images of the sample cross section. In addition, crack propagation and barreling phenomenon were discussed by variation process factors. Obtained results revealed that addition of nano-SiO_2 up to 3% would cause the improvement in both flexural and compression strengths while a further increase in reinforcement content causes reduction of composite strength. Furthermore, samples prepared at extrusion temperature of 210°C, have lower strength compared to those fabricated by 180°C temperature. Finally, it was found that increase in SiO_2 content and decrease in extrusion temperature would increase the brittleness of composite.

1. Introduction

Acrylonitrile-Butadiene-Styrene (ABS) is usually used as a commercial material due to its desirable physical properties as well as low fabrication cost. The main applications of ABS polymer are in automobile parts, household items and electrical housing. The main advantages of ABS polymer are its good stain resistance, impact resistance even at low temperatures, good abrasion resistance, being tough and stiff. However, this material suffers from some problems such as low mechanical properties (e.g. ultimate strength and hardness) [1]. In this regard, researchers focused on enhancing mechanical properties by reinforcing with Nano-ceramic materials to produce polymer matrix Nano-composite (Nano-PMC) [2, 3]. There are several nano-particles to prepare a polymernano-composite. Limited work has been done on different nano-particles such as clay, MMT, mica, talc and alumina. in ABS polymeric systems. There are still only a few reports about the preparation of ABS nano-composites and limited work has been done on alumina nano-particles in ABS polymeric systems. Lee et al. [4] reported a study on fabrication of ABS based nano-composite reinforced with clay through emulsion technique. Also, Wang and Hu [5] studied on characterization of delaminated nano-composite through direct melt intercalation. Stretz et al. [6] discussed the dispersion of clay particles in an ABS matrix comparing this system with a Styrene-Acrylonitrile (SAN) polymer based nano-composite where it was observed that the ABS/MMT composite clay resides in the Styrene acrylonitrile phase of ABS and accumulates preferential at the rubber particles surface. Pourabas and Raeesi [7] prepared an ABS/clay nano-composite by use of solvent/non-solvent method that works by different kinds of mixture. In another attempt Jang and

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DOI: 10.22075/macs.2019.14241.1142

Wilkie [8] studied on the effect of the clay on the thermal degradation behavior of ABS and found that, the addition of nan ceramic material significantly improves the aforementioned performance. Modesti et al [9] investigate the effect of the clay surfactant on the morphology of the developed composite. Tenget al. [10] fabricated alumina based nano-composite and studied the effect of particle size on microstructure and mechanical properties of the prepared material. They found the fracture mode of the fabricated composite changes to intergranular and strength increases, impressively. Kuaet al. [11] investigated effect of Nano sized alumina on mechanical and thermal stability of PEEK nano-composite and found improvement in both the properties in the case of increase in Nano-alumina content.

In fabrication of polymer matrix nano-composite, Silica (SiO_2) nano-particle is one of the most widely used reinforcement. It was used by different researchers to enhance the mechanical properties, thermal stability and surface properties. Lin et al. [12] reported that nano-SiO_2 is beneficial in improving the bonding strength of UF adhesive and subside its free formaldehyde emission. Devi and Maji [13] studied the fabrication of wood polymer nano-composites (WPNC) based on nano-SiO_2. They reported that mechanical properties, water uptake, and thermal stability would be improved by addition of SiO_2 particles in Wood/Polymer matrix. Hsu and Lin [14] reinforced the ABS matrix by adding SiO_2 nano-particles through Sol-Gel method. They used different type of catalyst for preparation of composite material. It was reported that the ABS/SiO_2 fabricated by use of NH_4F as catalyst material has higher strength, finer structure and smoother surface compared to those prepared by HCl catalyzer. Zheng et al. [15] studied the synthesis of silica-graft acrylonitrile-butadiene-styrene (ABS) nano-composites. The silica-graft ABS composites were prepared by an open ring reaction and radical grafting copolymerization of modified silica, styrene, and maleic anhydride (MAH) in ABS/THF solution. Differential scanning calorimetry (DSC) results show that the glass transition temperature (T_g) of ABS-graft-SiO_2 obviously shifts to high temperatures with increasing the silica content.

According to what reviewed above, it is seen that fabrication of ABS-Silica nano-composite through an extrusion process has not been reported so far. Hence, the aim of the present work was to prepare the ABS matrix nano-composite reinforced by nano-silica particles. Here, effect of extrusion temperature and SiO_2 content is fully discussed on flexural and compression strength of nano-composite through SEM images obtained from the cross section. In addition, crack initiation and barreling phenomenon are characterized to show variation of brittleness by adding nano-silica particles and temperature rising.

2. Materials and Method

2.1. Materials

The matrix material chosen for this study was ABS polymer (Absolac-920 Bayer), high flow medium impact grade. Table 1 shows the physical and mechanical properties of ABS. The nano-sized silica particles obtained from NOTRONO CO. (IRAN) was used as reinforcement material with an average particle diameter of 50 nm, density of 3.97 g/cm^3, and 2050 °C melting point. Note that the silica particles are added in lower amounts in volume as compared with conventional polymeric composites. This means that the current nano-composites would not alter much the process ability or density of the ABS matrix.

2.2. Compounding and Sampling

Composite samples for compression and flexural tests were prepared by first mixing the pre-weighed quantities of ABS graduals and Nano-Silica powder at different volumes (0-5 %). Manually, followed by melt mixing in double screw Extruder (BRABENDER 6300) 18 mm with L/D ration of 24/1. The melt temperature during the process varied between 180°C and 210°C and screw speed was kept constant at 60 RPM for all the compositions. In order to improve the mixing of materials, the raw ABS was dried in 60°C vacuum oven for at least 12h to remove the moisture and impurities. The Nano-silica was also dried at 90 °C for 10 hours to prevent from water uptake. The continuous mixing was performed during extrusion and the extruders were cooled by water at the exit of the die, and then air-cooled, after cooling the composite rods cut into graduals form in uniform size through the Pelletizer machine. The nano-composite samples for mechanical characterization were prepared by an injection molding process. The compression test samples (ASTM-D695) and flexural test samples (ASTM-D790) were formed by use of specified dies according to required standard sizes. Fig. 1 illustrates the extrusion machine and the extruded samples for compression and flexural tests.

Table 1 Physical and mechanical properties of ABS

Properties	Value
Tensile strength (MPa)	6.8
Notched impact strength (kJ/m^2)	3-30
Thermal coefficient of expansion	100-150×10^{-6}
Melting point (centigrade)	160
Density (g/cm^3)	0.905

In this work, artefacts have been generated using CNC milling machine, typically used to determine the effect of artefact defect on the mechanical strength of composites. For this purpose, a cylinder radius end drilling tool with 4 mm in diameter was used. The depth of drilling for defected samples was 1.5 in the middle of samples. Damage process was applied in terms of CNC milling machine with 1000 RPM of spindle and feed rate of 30 mm per minute. Fig. 2 shows the drilling process.

2.3. Morphology Analysis

Scanning electron microscopy was used here to determine the dispersion morphology of silica particles in the ABS polymer matrix. Through the SEM micrographs analyze the morphology of developed nano-composite. Scanning Electron Microscope (SEM) at 20 KV. Here, the scanning electron microscope Cam Scan MV2300 was used to study the morphology of ABS/Silica nano-composite. Fig. 3a demonstrates SEM machine used for the experiments.

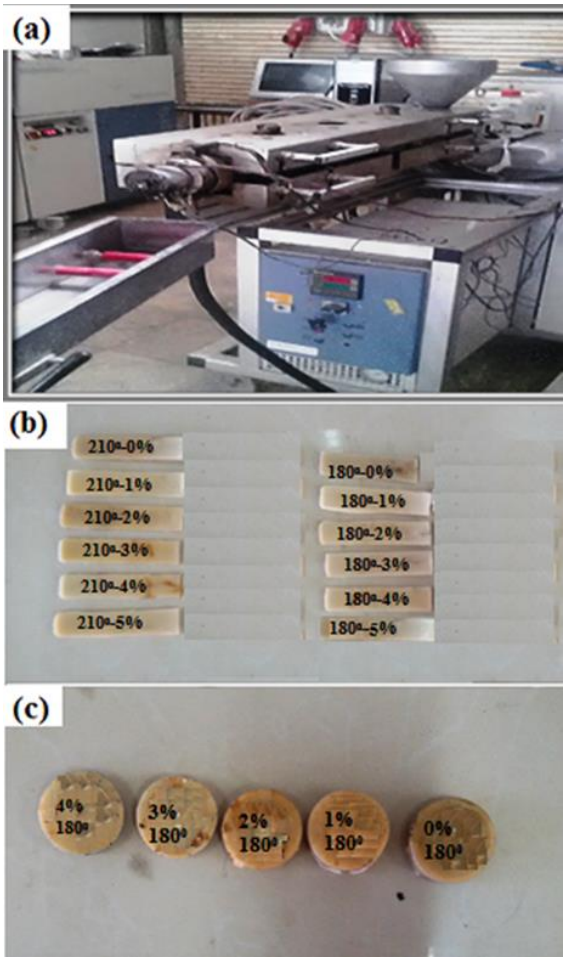


Fig. 1. (a) Extrusion machine (b) Extruded samples for flexural tests (c) Extruded samples for compression tests

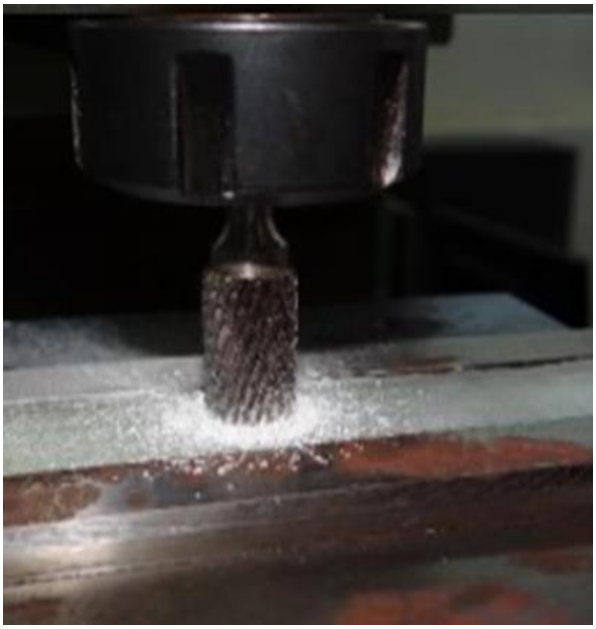


Fig. 2. Driling process in order to make a damaged sample.

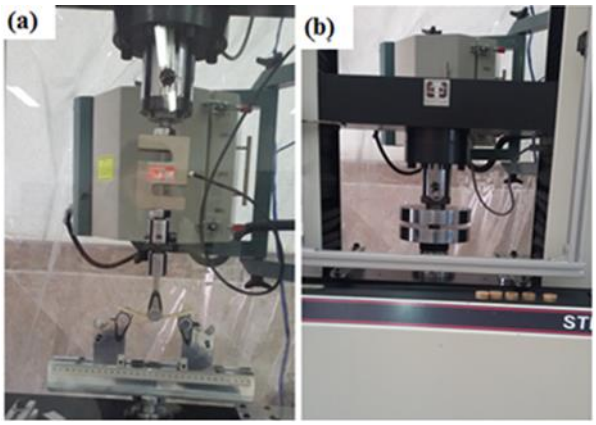


Fig. 3. Devices for characterization and testing (a)Universal test machine for flexural (b) Universal test machine for compression

2.4. Mechanical Testing

The compression and flexural tests were carried out using Universal Testing Machine (SANTAM 30T). The specimens were prepared according to (ASTM-D695) and (ASTM-D790) standards for compression and flexural tests, respectively. During flexural tests, the span length was 100 mm and the crosshead speed was 2.5 mm/min. In addition, for compression test, the tablets were subjected to 5mm/min compression and dimension of the tablets were measured to analyze the barreling phenomenon. Figures 3a and 3b illustrate the test machine for flexural and compression test, respectively.

2.5. Research Methodology

In order to study the effect of silica content (in six levels) and extrusion temperature (in two levels) on

composite strength, 12 samples for compression and bending tests were prepared according to a full factorial design. In addition, 12 defected samples were also prepared to analyze the effect of defect on strength behavior of fabricated composite. In order to prevent the stochastically observations, each experiment was prepared for three times and average of compression strengths and also the flexural strengths were reported. Table 2 presents the design matrix and observed values of composite strength.

3. Results and Discussion

3.1. Parametric Study

3.1.1. Effect of SiO_2 Content

In order to find the effect of SiO_2 content on strength behavior of fabricated composite, the parameter varied over 0 to 5% weight content while the temperature was kept constant at 180ºC. Fig. 4 illustrates the influence of silica content on bending and compression strength for defect free and defected samples. It is seen from the figure that both the flexural and compression strengths vary in the same behavior by variation of silica content. However, it is seen that for defect free samples, both the strengths increase by the increase in silica content up to 3% in weight. On the other hand, by further increase in reinforcement content, the strength of the composite slightly decreases. However, for defected samples, it is seen that the both strengths continuously increased by increasing the silica content. The interfacial interaction between the SiO_2 and the ABS phases plays a major role in controlling the microstructures and the properties of the composite materials.

So, the morphology and properties of nano-composites were characterized by scanning the electron microscopy (SEM), compress and bending tests. According to the SEM pictures (Fig. 5) used in this research, it can be seen the strong bonding of nano-silica particles and ABS matrices that made a homogenous mixture. Also, the results of flexural and compression tests confirm the existence of a homogenous and strong mixture of nano-silica and ABS. In addition, the well dispersed ABS/SiO_2 hybrids can be obtained by adsorption of silica on the surface of matrice through a strong interaction of silica surface and ABS, which can prevent the macroscopic phase separation in the ABS/SiO_2 hybrid system [16]. According to the Fig. 6(a), the increase in the flexural strength of the specimens by addition of nano-particles can be attributed to the large specific surface area of nano-silica that results in enhancement of the mechanical properties of the ABS composite. In addition, elimination of porosities and cracked surfaces is another reason for improving the mechanical strength of the composite. When the nano-silica particles are added to the ABS matrices, it produces a layered structure and micro-fibers that boosts the endurance limit of the composite against compression forces. This behavior is shown in Fig. 6(b).

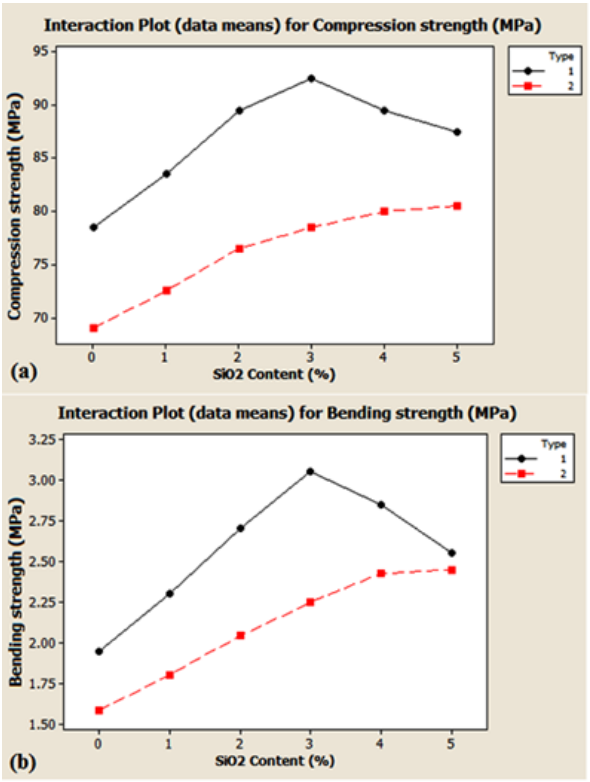


Fig. 4. Effect of SiO_2 content on (a) Compression strength (b) Flexural strength (1: Defect free samples, 2: Defected samples).

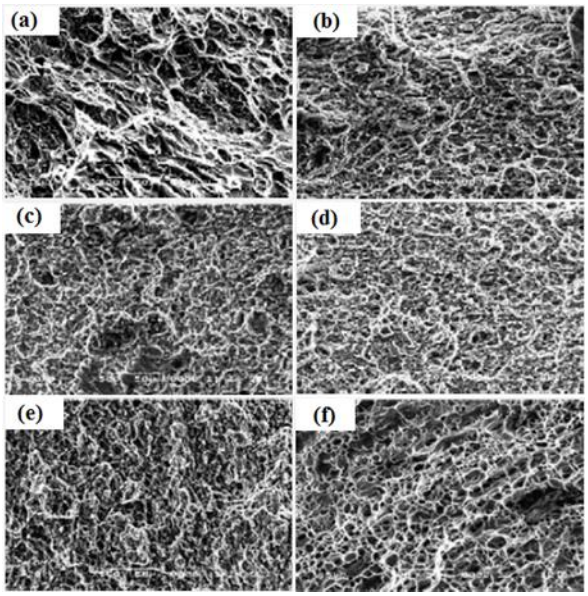
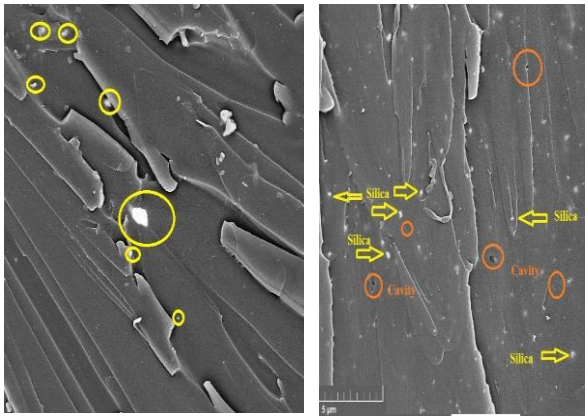


Fig. 5. Morphology of composite cross section under different silica content (a) 0% (b) 1% (c) 2% (d) 3% (e) 4% (f) 5%

Table 2 Design matrix and observed values of composite strength

No	SiO_2 content (%)	Temperature (°C)	Compression strength (MPa)		Flexural strength (MPa)	
			Defect Free	Defected	Defect Free	Defected
1	0	180	81	66	2.1	1.5
2	1	180	88	69	2.5	1.7
3	2	180	96	75	2.8	2
4	3	180	100	77	3	2.3
5	4	180	97	79	2.8	2.5
6	5	180	95	80	2.6	2.5
7	0	210	76	72	1.8	1.68
8	1	210	79	76	2.1	1.91
9	2	210	83	78	2.6	2.09
10	3	210	85	80	3.1	2.2
11	4	210	82	81	2.9	2.35
12	5	210	80	81	2.5	2.4



(a) (b)

Fig. 6. SEM pictures: (a) dispersion of nano silica & (b) porosity decrease

For defect free samples, when the silica contents increase up to 3%, the degree of porosity in cross section of fabricated samples decreases which causes the increase in its density. In such condition, voids in the cross section of the samples decreases and causes increase in the strength. When the SiO_2 content goes beyond a critical value, the composite would be saturated from reinforcement and SiO_2 sediment out in a composite matrix. In such condition, the deposited SiO_2 can disturb the strength of the composite. Fig. 4 illustrates the SEM micrograph from the morphology of the composite under different SiO_2 contents. It could be seen from the figure that by adding SiO_2 up to 3%, the voids are diminished from composite cross section and further increase in the content causes deposition of SiO_2 on matrix surface that roughen the composite structure and has a negative influence on strength [17].

Another point to be interpreted from the Fig. 5 is that for defected specimens, the strength increases continuously by increase in SiO_2 content. Since the defect destroys the strength of the composite, by increase in silica content, the strength increases and deposited silica can protect the samples from fracture. Hence, the composite strength for defected samples would increase by increase in silica content. This can be explained by the characterization of Silica that has high strength and can create a good bonding with epoxy matrix that permits the right stress distribution into both composite phases and the nano-particles and agglomerates act as stress concentrators. Nano-particles have good distribution and good bonding with matrix and also fill the hole and prevent crack growth (Fig 6(a)).

Finally, it was observed that the compression and flexural strength of undamaged samples had about 20% and 63% enhancement with adding 3% nano silica in comparison with the neat samples. Furthermore, with increasing the extrusion temperature from 180^{oc} to 210^{oc}, the compression and flexural strength of composite decreased about 36% and 6% respectively. In addition, the compression and flexural strength of defected samples had about 20% and 58% enhancement with increasing of nano-silica to 5%.

3.1.2. Effect of Temperature

In order to find the extrusion temperature on strength behavior of fabricated composite, the parameter varied over 180ºC and 210ºC while the SiO_2 content was kept constant at 3%. Fig. 7 shows the influence of temperature on both compression and flexural strength for defected and defect free samples. It could be seen from the figure that both the flexural and compression strength vary in the same behavior by variation of extrusion temperature. However, it is apparent from the figure that for defect free samples the strength decreases by increase in

extrusion temperature; while, for defected specimens the strength increased by increase in temperature.

For defect free samples, when the melt temperature increases, the sensitivity of material to degradation also increases which results in formation of porosity in morphology of samples that destroys composite strength. Fig. 8 illustrates the cross section of composite fabricated under different extrusion temperatures. It is seen that by increasing the extrusion temperature, the void dimensions in the morphology of composite increases which causes low composite strength.

However, it is evident from the figure that for defected samples, the composite strength increases by increase in the extrusion temperature. When the temperature increases, the viscosity of the melt material decreases and the ductility of the composite increases. In such condition, during mechanical testing, the material subjected to compression or bending can move easily and fill the defect, to some extent. Consequently, the composite samples would be protected from failure and the strength of composite samples would increase.

3.1.3. Interaction of SiO_2 Content and Temperature

Fig. 9 illustrates the simultaneous effect of extrusion temperature and silica content on strength behavior of fabricated nano-composite material. It is seen from the figure that for both the extrusion temperatures, the bending and compression strength increased by increase in SiO_2 content up to 3%. However, irrespective of extrusion temperature, by further increase in SiO_2 content, compression and flexural strength decrease, slightly. Moreover, it is ascertained from the Fig. 9 that at lower SiO_2 content (i.e. 0 and 1%), the difference between strength values under different temperature can be neglected. However, when the SiO_2 content exceeds 1%, the strength values for 180ºC extrusion temperature is impressively higher than those fabricated in 210ºC. This trend could be attributed to expansion of SiO_2 at elevated temperature.

When the silica content is relatively low, the expansion of nano-composite and relative porosity is low, and therefore, there is no highlighted difference between strength values at different temperatures. However, by increase in SiO_2 content, the expansion of composite and relatively porosity at elevated temperature is higher that damages the strength of the fabricated composite when it is processed at 210ºC extrusion temperature.

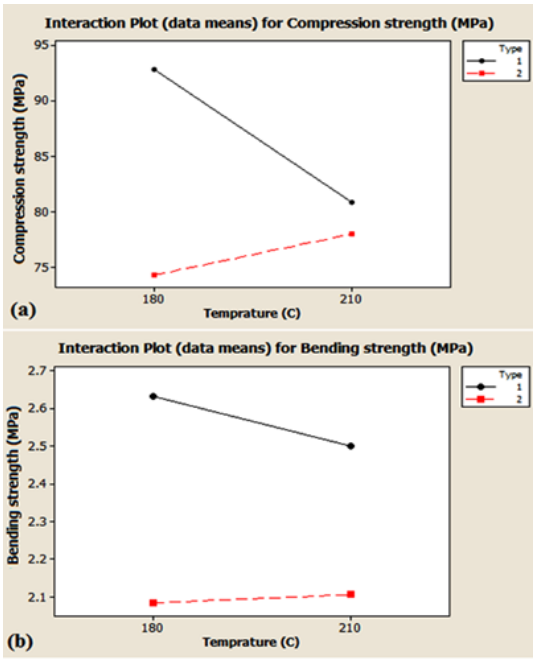


Fig. 7. Effect of temperature on (a) Compression strength (b) Flexural strength (1: Defect free samples, 2: Defected samples)

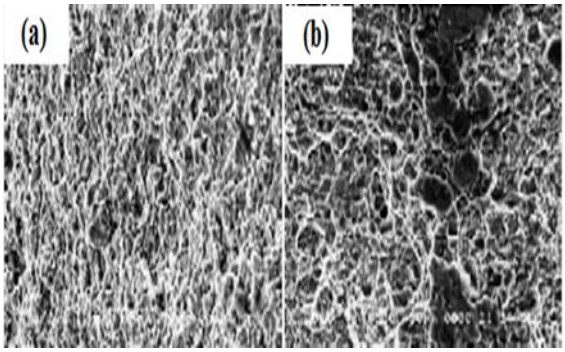


Fig. 8. Morphology of the composite under different tempera-ture

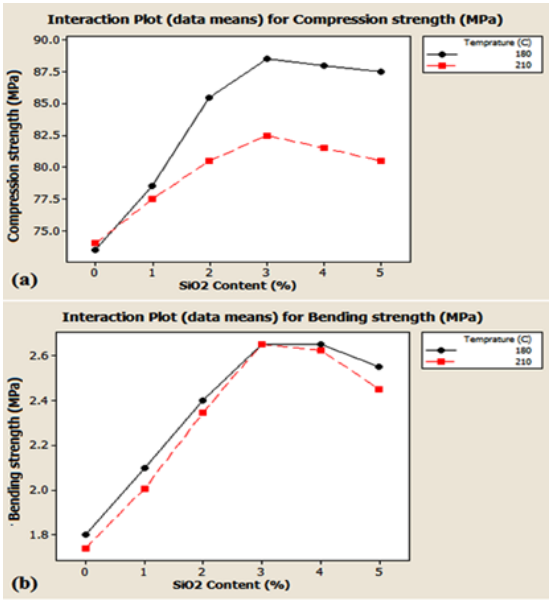


Fig. 9. Simultaneous effect of SiO2 content and extrusion temperature on (a) Compression strength (b) Flexural strength

3.2. Fracture Behavior

In order to analyze the behavior of material during mechanical tests, stress-strain curves of the fabricated composites were obtained and are presented in Fig. 10.

It could be seen from the figure that by adding SiO_2 content up to 3%, the strength increases and by further increase in reinforcement content, the strength would reduce. However, by increase in SiO_2 content, the strain values decrease. Increase in strength and decrease in strain implies on brittle behavior of fabricated nano-composite by addition of SiO_2. The SiO_2 is a brittle ceramic phase with high strength. Hence, by addition of silica as reinforcement, the brittleness of fabricated composite increases.

Fracture observation of the samples showed that by addition of SiO_2 content up to 3%, the barreling phenomenon is restricted and by further increases in silica amount, the crack occurs and propagates in the surface of the samples subjected to compression test. Hence, by addition of silica from 0% to 3%, the diameter of the compressed sample decreases and the height increases. On the other hand, at SiO_2 content of 4% and 5%, the crack is formed and propagates in the specimen. In addition, the length of the crack for specimen containing 5% silica is relatively higher than that of having 4% silica. Furthermore, in the samples with high content of nano-silica, agglomerates act as stress concentrators. The reduction of the mechanical strength of high filler content (4% & 5%) is related to the presence of the higher quantity of clusters due to the agglomeration that tend to reduce the strength of composite and also the saturation phenomenon was occurred [18]. According to the results, it can be concluded that the damage process results in a strain hardening and leads to more fracture stress and stress concentraion, but on the other hand, defection process, could lead to separation of the interface of nano- silica particles and the ABS which ultimately, reduced the mechanical strength of the composite samples. Fig. 11 illustrates that by increase in the extrusion temperature, the strength decreases.

However, the strain values increases. It means that by increase in extrusion temperature, the fabricated material shows the ductile behavior. When the temperature increases, the sensitivity of viscous material for expansion increases and in such condition, the deformation of material during mechanical testing is higher that causes higher strain value. Sample observations after fracture show that at 3% SiO_2 content, the diameter of the sample fabricated in 210ºC is relatively higher than that fabricated in 180ºC. In other words, the degree of barreling for the samples fabricated at elevated temperature is relatively higher than that for samples fabricated in low temperature.

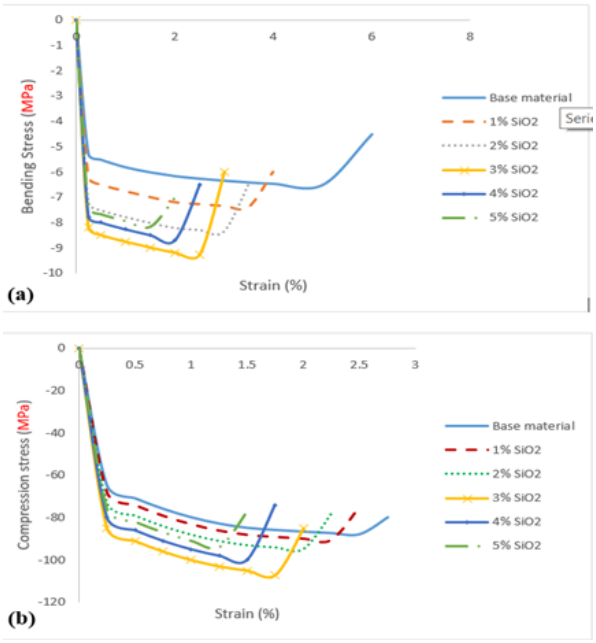


Fig. 10. Stress-strain curve under different silica contenta for (a) Flexural strength (b) Compression strength

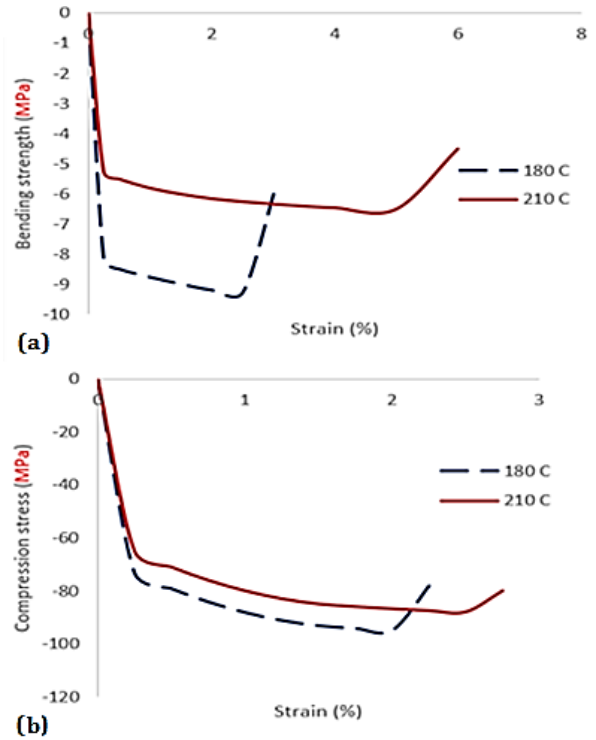


Fig. 11. Stress-strain curve under different extrusion temperature for (a) Flexural strength (b) Compression strength

Fig. 12. Crack growth in composite samples with different content of Silica: a) in 180°C, b)in 210°C

In addition, comparison results between samples containing 5% SiO_2 shows that the length of the crack for the sample fabricated in 210◦C is relatively lower than that for sample fabricated in 180◦C. These observations show that increases in extrusion temperature causes the composite material to have the ductile behavior during mechanical testing. Fig. 12 (a & b) show the crack growth in composite samples with different contents of nano-silica and for two 180◦CS and 210◦C extruding temperatures.

4. Conclusions

In the present work, an experimental study performed to prepare Acrylonitrile-Butadiene-Styrene matrix composite reinforced by Nano-silica particles. Hot extrusion method was utilized here to fabricate the composite specimens used for flexural and compression tests. Experiments were carried out to identify effects of silica content and extrusion temperature on strength behavior. The obtained results could be summarized as follow:

- Results indicated that both the compression and flexural strengths increased by increase in SiO_2 content up to 3%. While by further increase in reinforcement content (i.e. 4% and 5%), the strength decreases. The reason for improvement of strength is restriction of the porosity and for decrease in strength is deposition of silica at high content.
- By increasing the extrusion temperature due to increase in porosity level, the strength of the composite decreases.
- Results indicated that the increase in SiO_2 content causes the composite material showing brittle behavior. It means that the strength increases, but the strain values decrease.
- Due to expansion of composite at elevated temperature, the strain values of composite samples fabricated at 210ºC are relatively higher than those fabricated at 180◦C. It means that increase in extrusion temperature causes the material showing ductile behavior during mechanical testing.
- By increasing the silica up to 3% content, the barreling is restricted and by further increase, crack occurs for fractured composite. In addition, the length of crack at 5% silica was relatively higher than that of 4%.
- By increasing the extrusion temperature, the barreling improves (at lower 3% silica) and the length of the crack (for 4% and 5% content) is restricted.

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