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EFFECT OF THERMAL CYCLING ON THE TENSILE BEHAVIOR OF POLYMER COMPOSITES REINFORCED BY BASALT AND CARBON FIBERS

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Abstract

The aim of the present work was to investigate the effect of thermal cycling on the tensile behavior of three types of polymer-matrix composites — a phenolic resin reinforced with woven basalt fibers, woven carbon fibers, and hybrid basalt and carbon fibers — in an ambient environment. For this purpose, tensile tests were performed on specimens previously subjected to a certain number of thermal cycles. The ultimate tensile strength of the specimen reinforced with woven basalt fibers had by 5% after thermal cycling, but the strength of the specimen with woven carbon fibers had reduced to a value by 11% higher than that before thermal cycling.

Keywords: Polymer-matrix composite, Thermal cycling, Tensile strength, Basalt fibers, Carbon fibers

1. Introduction

Structural polymer matrix composites are rarely subjected to hundreds of thermal cycles

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from extremely low temperatures to elevated maximum service temperatures. However, 20 years ago, composites were first considered for satellite applications in which dimensional stability was critical during exposure to thermal fluctuations, when orbiting the earth [1, 2]. In aeronautical applications, structural parts can be subjected to cyclic temperature variations (approximately between -50ºC and 130ºC) depending on fly stages, or the high temperature variations being sustained during the supersonic flight [3]. The study of the exposure of such materials to environmental conditions such as varying temperature, ultraviolet radiation, etc. is of outmost importance, in order to assess the impact of these important fatigue factors on their mechanical behavior.

Because of the structural heterogeneity of composite materials, the thermal fatigue damage process is rather complex, and consists of a variety of damage modes such as matrix cracking, debonding between fiber and matrix, fiber fracture or delamination between plies [4].

During thermal cycling, both matrix and fibers expand or contract, according to their coefficients of thermal expansion. As they are internally constrained, the temperature fluctuations cause the stress to build-up at the interface and consequently the adjacent plies which stacked together with different fiber orientations in the laminate. It appears that one of the most severe problems concerning the survivability of most composites is the bond breakage between their constituents. Such a breakage is caused by either extension of pre-existing cracks or by the creation of new cracks at the material interfaces. For example, upon thermal or mechanical loading of the composites, micro-stresses are developed at the interface between various constituents. If these stresses exceed the corresponding bonding strength of the composite, they result in crack formation. Such interface cracking is the earliest form of damage, which is frequently observed in the composite structures [5].

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The first damage observed in cyclic thermal loading is matrix micro-cracking. Micro-cracks do not only change the thermal and mechanical properties of the composites, but they also constitute pathways through which the corrosive agents may penetrate into the material. Moreover, micro-cracks act as nuclei for further damage types such as delamination and longitudinal splitting [4]. Engineering designs based on static properties without considering degradation of composite strength and modulus caused by temperature variations may lead to and eventually cause catastrophic disaster [4].

Ray [6] studied the influence of thermal cycling on interfacial damage in thermosetting matrix reinforced with aramid fibers. He showed that the post-curing effects may be because of further cross-linking in the matrix and may also introduce more adhesion at the interface. The thermal conditioning may change the chemistry of the fiber/matrix interface in forming an interpenetrating network [7] and also improve the adhesion at the interface either by mechanical interlocking or by a surface chemistry mechanism. The cryogenic conditioning at -80ºC for repeated times may impart different degrees of shrinkage compressive strength at the interface. This could help in strengthening the adhesion at the fiber/matrix interface.

Polymer-matrix composites containing continuous carbon fibers are widely used for lightweight structures, such as aircraft components and rotating machinery. These structures may encounter temperature excursions during the use, whether because of exhaust gas, de-icing, ambient temperature variations or engine operation. Because of large difference between the coefficient of thermal expansion (CTE) of carbon fibers (typically around -6×10^{-6} $^{o}C^{-1}$) in the longitudinal direction and that of the polymer matrix (typically ranging from 20 to 120×10^{-6} $^{o}C^{-1}$), temperature variations result in changes in the amount of thermal stress in the composite. The thermal stress is particularly significant, when the laminate have fibers in different directions in each lamina, as the CTE of each lamina is highly anisotropic. Such

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thermal stress variations may cause thermal fatigue. Damage degrades the modulus of composites, thus enhancing vibrations and then aggravating further damage [8].

Shin and Kim [9] showed that the strength and stiffness of the graphite/epoxy composites after exposure to thermal cycles (80 cycles, +100ºC to -70ºC) decreased in exponential proportion, as the thermal cycles increased. Paillous and Pailler [10] examined the degradation induced by exposure to a space environment on multi-ply polymer matrix composites. They exposed various graphite/epoxy laminate specimens to thermal cycling. Their study showed that the thermal cycling degraded the matrix by chain scission, cross-linking and micro-crack damage, in which altered the composite properties. Lafarie-Frenot and Rouquie [3] compared the damage development in the carbon/epoxy laminates during isothermal ageing or thermal cycling. They showed that the oxidation phenomenon induced matrix shrinkage which is visible on the surfaces of the samples. This shrinkage, which is non-existent on specimens tested in neutral atmospheres, seems to be favourable to fiber–matrix debonding and matrix crack onset. In that case, a coupling between oxidation and cyclic thermo-mechanical stresses because of temperature variations accelerates all the phases of the damage process.

In composite materials, the oxidation mechanism strongly depends on temperature and oxygen pressure [11], specimen geometry, anisotropy [12–14] and matrix-fiber bonding [15]. In oxidative atmospheres, the coupling between oxidation, occurring at high temperatures, and thermo-mechanical cyclic stresses because of temperature variations accelerate the damage process [16].

Basalt is a natural material that is found in volcanic rocks. When used as (continuous) fibers, basalt can reinforce a new range of composites. It can also be used in combination with other reinforcements (e.g. basalt/carbon). Because of its thermal properties, it is already

used as fire protection in the form of fabrics or tapes [17, 18].

Hybrid composites contain two or more different types of reinforcement fibers which have different mechanical and/or other properties, allowing researchers to design a composite with tailored properties in specific applications [19]. In general, the purpose of hybridization is to achieve a composite architecture which synergises the properties of both materials and/or lowers the cost, since one type of the fibers could be too expensive. Brittle inorganic fibers and ductile organic fibers are often combined to make hybrid composites such as palm/glass, glass/mineral fiber, aramid/glass, etc. [20].

Carbon fibers have excellent tensile strength and modulus, though they are much more expensive than other fibers. As inorganic fibers, basalt fibers have good strength and modulus as well as superior impact resistance, but much better thermal properties than carbon fibers. Therefore, a woven hybrid composite of these two fibers is reasonably priced with adequate tensile, thermal and impact properties. However, it is not clear that how the tensile properties of hybrid composites are changed as temperature fluctuations occur.

Few investigations are done to evaluate the thermal cycling influence on the tensile behavior of reinforced polymer composites, especially for textile composites. In the present study, the effect of thermal cycling on the tensile behavior of carbon, basalt and basalt/carbon hybrid fiber reinforced composites are investigated experimentally. The composite specimens are prepared according to ASTM standards and then subjected to different number of thermal cycles between -30 to +220ºC. The thermal cycled specimens are tested in tensile loading and stress- strain diagrams and the mechanical properties in tensile loading are investigated.

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2. Experimental Procedure

2.1. Materials and Fabrication

The first material was a composite laminate consisted of eight plies of carbon fibers (AC220, Colan Products, Australia) and a phenolic resin (Phenlam® CL2000T, Huntsman Chemical Company, Australia). The second material consisted of four plies of basalt fibers (BAS 630, BasaltexTM, Belgium) and the same phenolic resin. The third material consisted of alternatively three plies of basalt- and three plies of carbon-fiber-reinforced phenolic composite. The materials were fabricated in the form of plates with dimensions 400 × 400 × 2.5 mm. The fiber volume fraction in all the composite specimens was approximately 35%. The physical and mechanical properties of the resin and the fibers used in this work are presented in Table 1. The materials were combined and the plates were fabricated by hand lay-up technique.

Table 1. Specification of the fibers and the resin

Specification	Fibers		Phenolic resin
	Basalt	Carbon	
Weave type	Twill 1/3	Plain	_
Thickness (mm)	0.56	0.25	_
Specific surface weight (g/m^2)	630	198	_
Density (g/m^3)	2.70	1.75	1.2
Tensile strength (MPa)	3000	4210	24-45
Tensile modulus (GPa)	89	230	4
Elongation at break (%)	3.15	1.5	1-2
Coef. of thermal expansion (10^{-6} ºC^{-1})	8	-6	20-31
Gel time (min)	_	_	25

2.2. Preparation of Specimens

Specimens were cut from the plates in the desired dimensions of 250×25×2.5 mm, using

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a water jet cutting machine. Each specimen was polished using emery polish paper. In this way, local surface and edge cracks and local surface material heterogeneities were avoided. After cutting to predetermined dimensions according to ASTM D3039 for tensile test, the specimens were labeled and their dimensions and mass were measured. The specimen is pictured in Fig. 1. All Specimens were divided into three groups; basalt/phenolic composites (BFP), carbon/phenolic composites (CFP), and basalt/carbon/phenolic composites (BCFP).

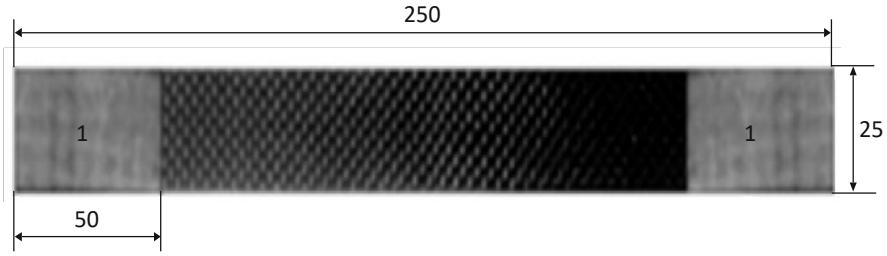

Fig. 1. Composite specimen prepared for tensile testing (dimension in mm). 1 — end tabs.

2.3. Thermal Cycling Process

To investigate thermal cycling effect, the specimens were put into a temperature shock test chamber (Model TS130, Weiss Umwelttechnik GmbH, Germany). The general temperature profile applied to the specimens is shown in Fig. 2. According to this profile, one thermal cycle was designated as the sequence from -30ºC to +220ºC, and a stop of 3 min in each temperature, so that, the total duration of each cycle was 6 min. The complete thermal cycling procedure for all the specimens was as follows:

1. All specimens were placed in freezing environment (-30ºC) for 3 min.

2. All specimens were placed in hot environment (220ºC) for 3 min.

3. After certain successive number of thermal cycles, three specimens were selected and removed from the chamber for tensile testing.

4. The above-mentioned steps were repeated until all the specimens were tested.

5. The thermal cycling was repeated for 5, 10, 15, 20, 25 and 30 cycles.

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An aluminium container was designed and fabricated to avoid direct contacting of the specimens and was placed in the temperature test chamber.

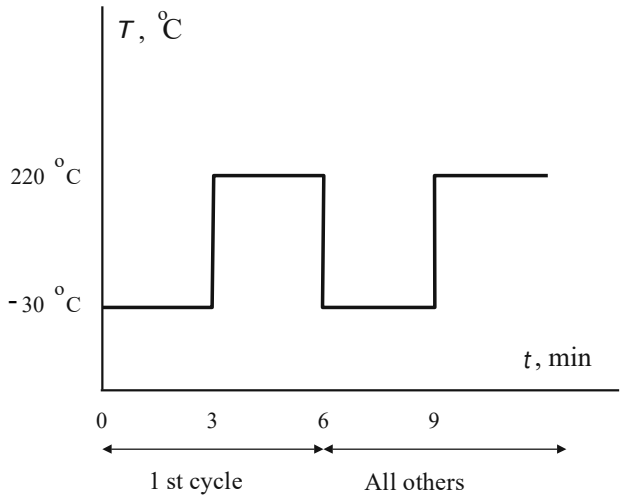

Fig. 2. Profile of thermal cycles.

2.4. Tensile Testing

The tensile tests were performed on an STM-150 20-kN universal testing machine (Santam Co., Iran) according to ASTM D3039. The crosshead speed was 2 mm/min, and the tensile strain was measured by strain gages attached along the longitudinal axis of specimen.

The tests were conducted at an ambient temperature (25°C) and a relative humidity of 85%. Because of the limitation on materials and testing facilities, the tensile properties were determined using only three specimens

3. Results and Discussion

The typical stress–strain curves for the three types of fiber-reinforced phenolic composites after various thermal cyclings are presented in Fig. 3.

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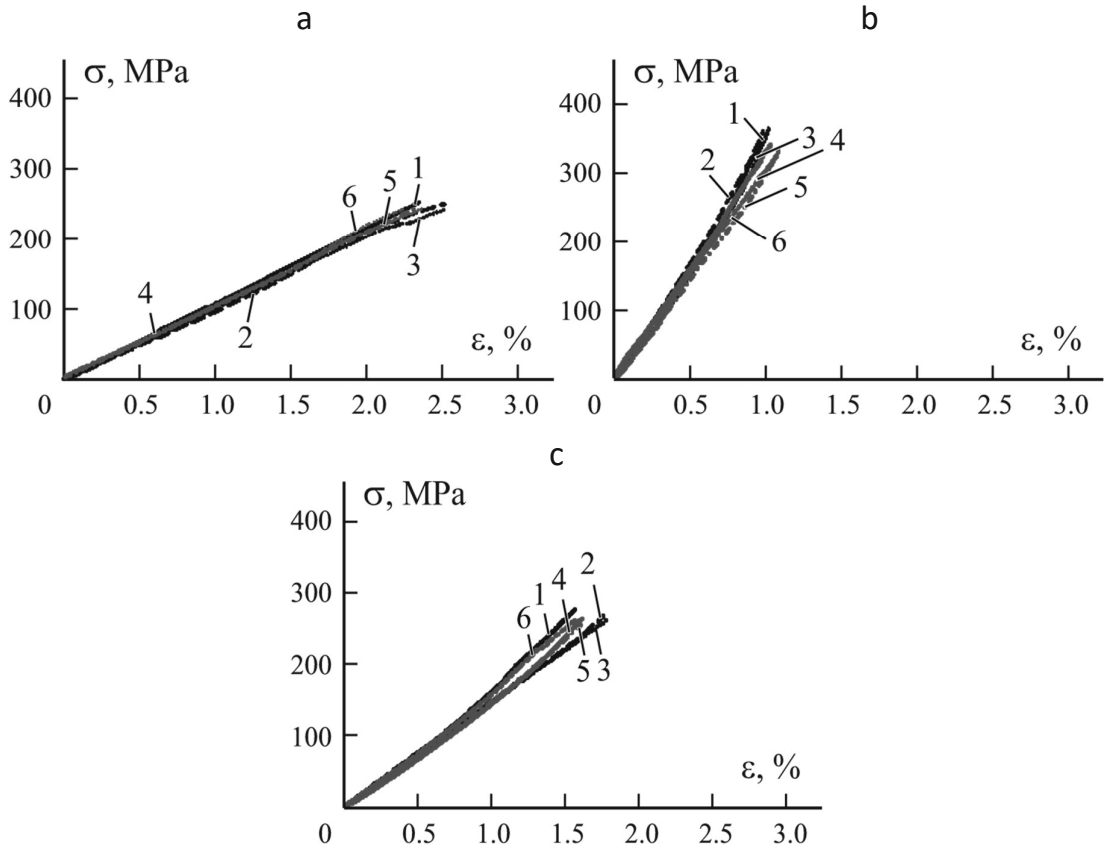

Fig. 3. Tensile stress–strain diagrams σ−ε of BFP (a), BCFP (b), and CFP (c) composites after 5(1), 10 (2), 15 (3), 20 (4), 25 (5), and 30 (6) thermal cycles.

.A thermal cycling definitely causes debonding and/or weakening of the interface between the fibers and the matrix and also between plies because of different coefficients of thermal expansion and/or contraction of the polymer matrix and fiber reinforcement [7].

A higher tensile modulus corresponds to an increased crystallinity and orientation of the crystallographic basal planes parallel to the fiber axis. This phenomenon increases the negative longitudinal coefficients of thermal expansion of fibers. When temperature is decreased, the fibers contract in the radial direction and expand along the longitudinal axis, while the matrix contracts in all directions. Greater differences in the coefficients of thermal expansion between the fibers and matrix increase the thermal stresses at low temperatures

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and cause a corresponding increase in microcracking [21].

The polymeric matrix in this study was a material with a high thermal expansion coefficient of 20-30 · 10^{-6} ºC^{-1}. The differences between the thermal expansion coefficients of basalt fibers, carbon fibers, and polymeric matrix lead to high interfacial strains during the thermal cycling process between –30°C and +220°C. The stresses caused by the strains can lead to the failure of interfacial adhesion between the fibers and matrix, thereby decreasing the tensile properties of composites. When composite specimens are subjected to a thermal cycling under oxidizing atmospheres, the high temperatures probably cause thermo-oxidation of the matrix. This phenomenon primarily affects the external surfaces of specimens [3]. In such a case, the coupling between the oxidation and cyclic thermomechanical stresses because of temperature variations can accelerate all phases of the damage process, particularly in CFP composites.

Fig. 4a shows the effect of thermal cycling on the ultimate tensile strength of thermally conditioned fiber-reinforced composites.

This thermal conditioning resulted in different effects on the BFP, CFP, and BCFP composites. Stability of the tensile strength was noticeable for the BFP composites after 15 cycles, but it reduced for the CFP composites. A gradual stabilization of the tensile modulus for the BFP and BCFP composites seen in Fig. 4b implies that some kinds of permanent stiffening had occurred in the aged material. But the tensile modulus of CFP composites showed a continuous reduction up to 25 cycles.

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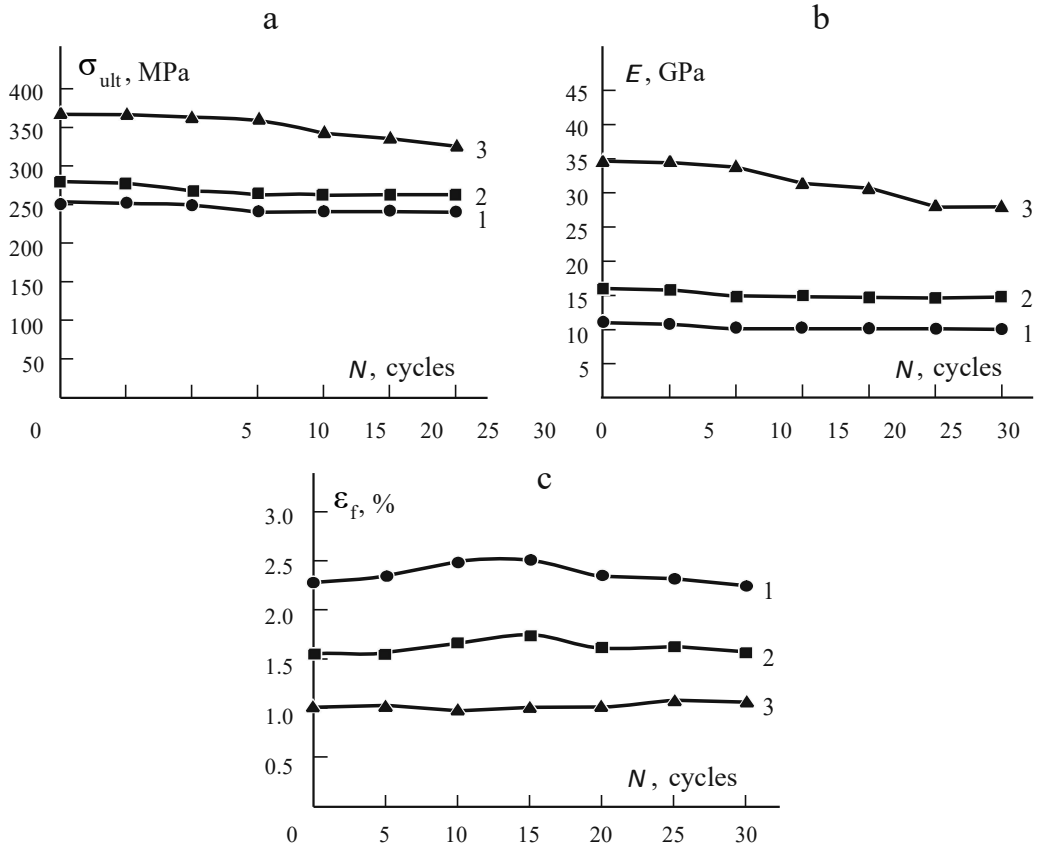

Fig. 4. The ultimate tensile strength σ_{ult} (a), tensile modulus E (b), and strain at failure ε_f (c) of BFP (1), BCFP (2), and CFP (3) composites.

Fig. 4c shows the variation in strain up to failure for the three types of composites investigated. The behavior of the composites was similar, which means that number of cycles did not significantly affected the failure strain. The average measured values of tensile properties for fiber-reinforced phenolic specimens after various thermal cyclings, extracted from Fig. 3, are shown in Table 2. The specific breaking energy was calculated as the area between the stress–strain curves and the X axis.

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Table 2. Average tensile properties for the fiber reinforced phenolic specimens

Specimen code*	Ultimate tensile strength (MPa)	Tensile modulus (GPa)	Strain at failure (%)	Specific breaking energy (J/cm^3)
BFP-T0	253.4	10.84	2.28	2.95
BFP-T5	251.9	10.74	2.34	3.01
BFP-T10	249.8	10.19	2.49	3.11
BFP-T15	241.2	10.18	2.50	3.10
BFP-T20	241.0	10.11	2.35	2.92
BFP-T25	241.0	10.08	2.31	2.78
BFP-T30	241.0	10.02	2.24	2.67
CFP-T0	368.3	34.57	1.02	1.78
CFP-T5	366.4	34.39	1.01	1.75
CFP-T10	363.6	33.78	0.98	1.68
CFP-T15	359.8	31.31	1.01	1.68
CFP-T20	343.0	30.6	1.02	1.67
CFP-T25	335.9	28.00	1.08	1.69
CFP-T30	326.0	28.02	1.06	1.70
BCFP-T0	279.5	15.83	1.55	2.12
BCFP-T5	277.0	15.70	1.56	2.10
BFCP-T10	267.0	14.80	1.67	2.26
BCFP-T15	263.2	14.75	1.75	2.22
BCFP-T20	262.6	14.61	1.61	2.05
BCFP-T25	262.5	14.56	1.63	2.01
BCFP-T30	262.2	14.59	1.56	1.95

* The number in specimen codes indicate the number of thermal cycles.

The overall conclusion drawn here is that there are a number of mechanisms that can be considered responsible for stiffening of the material during the thermal cycling process. The most reasonable explanation for the stability of tensile properties of the basalt-fiber-reinforced composite was a possible postcuring during the thermal cycling process. This happened because of the high temperature, which was not severe enough to break the chemical bonds of polymer [22-24]; in contrast, it contributed to the creation of free radicals to the molecules of phenolic which had not already reacted, and thus, to a further cross-

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linking [25-28]. Several mechanisms may contribute to the fiber/matrix adhesion, e.g., covalent bonding, physical interactions, and mechanical interactions [7]. These postcuring phenomena could dominate over the de-adhesion effect of thermal cycling. Thus, the stability of tensile properties showed up for the BFP and BCFP composites. The cryogenic condition at –30°C for repeated times may impart different degrees of the compressive shrinkage strength at the interface. This could assist in strengthening adhesion. But the weakening effect of thermal cycling might dominate over the postcuring hardening effect in the CFP composites and consequently, it may result in reduced tensile properties.

Figs. 5 and 6 show the normalized strength $\bar{\sigma}$ and modulus E of specimens at various thermal cycles in comparison with those of unexposed (original) composites specimens. As a general rule, the strength and modulus of the composite specimens after exposure were lower than those of unexposed ones, and they decreased with increasing number of thermal cycles. It is obvious that the CFP composites were more susceptible to damage by thermal cycling than the BFP and BCFP ones. After 30 thermal cycles, the tensile strength of the CFP composites reduced almost by 11%. Also, the tensile modulus showed a rapid decrease with increasing number of thermal cycles and reduced almost by 19%. However, there was observed stabilization after 15 cycles for the BFP and BCFP composites, and the tensile strength and modulus became almost constant.

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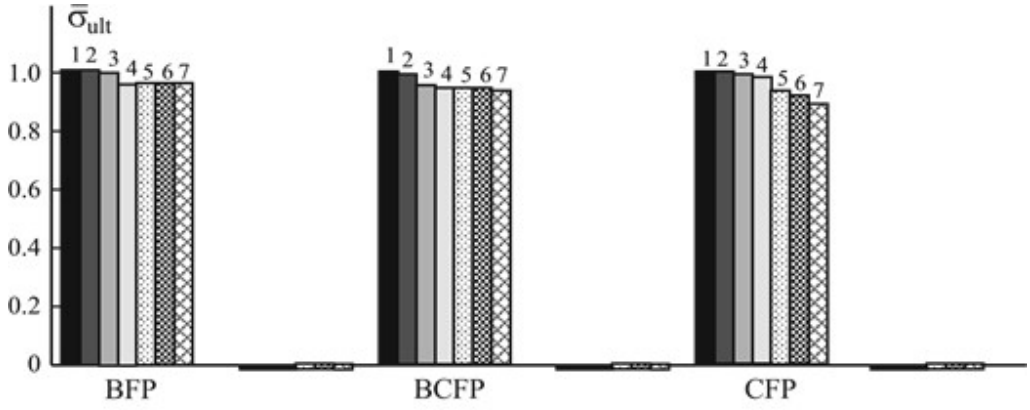

Fig. 5. Degradation of the normalized tensile strength $\bar{\sigma}$ of composites after their exposure to thermal cyclings: $N = 0$ (1), 5 (2), 10 (3), 15 (4), 20 (5), 25 (6), and 30 cycles (7).

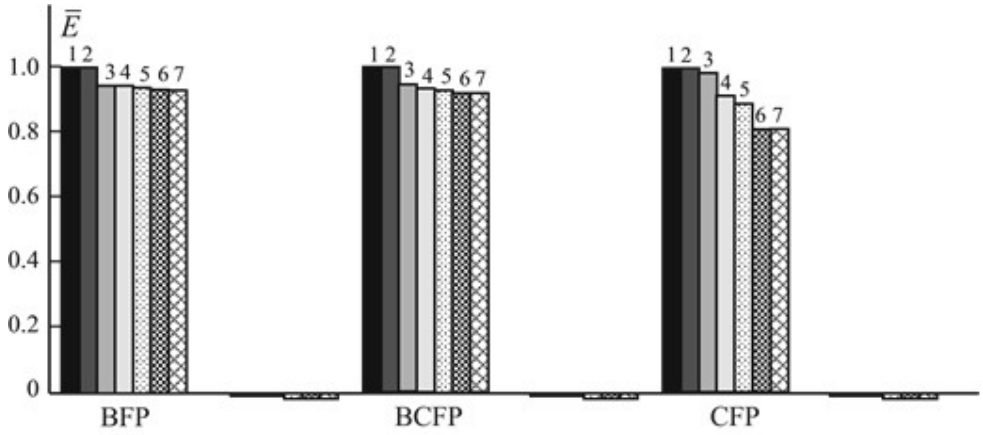

Fig. 6. Degradation of the normalized tensile modulus $\bar{E}$ of composites after their exposure to thermal cyclings. Designations as in Fig. 5.

From the above-mentioned discussion, it is clear that thermal cycling is a complex process requiring continuing research efforts to get a knowledge about the various processes inside the materials.

The fractured specimens revealed that the fracture surfaces of CFP specimens were dominated by a brittle fracture, which is typical of these types of composites. Also, no evidence of interfacial debonding and delamination phenomena was observed between 5 to 30 cycles. Both matrix cracking and fiber breakage could be seen. For the BFP specimens, at

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a low number of thermal cycles, the matrix cracking was the dominating failure mechanism, but at a higher number of thermal cycles, fiber pull-out and delamination were dominant. For the specimens of BCFP composites, the presence of carbon fibers in the BCFP composite led to a higher brittleness as compared with that of BFP composite. The same conclusion could be drawn for the CFP composites. The failure mechanisms were matrix cracking, fiber breakage, and delamination.

4. Conclusions

In this work, the effect of thermal cycling on the tensile behavior of polymer-matrix composites were investigated experimentally. The matrix used for the composites were a phenolic resin reinforced by layers of woven basalt fibers and carbon fibers and also by alternating layers of woven basalt/carbon fibers. The temperature range for thermal cycles was –30 to +220°C. The number of cycles was varied from 5 to 30. The thermal cycling manifested similar kinds of debonding effects for thermally conditioned specimens of carbon/phenolic (CFP) and basalt/phenolic (BFP) composites. In conclusion, the thermal cycling resulted in degradation affects on thermally conditioned carbon/phenolic composites. The same conditioning caused no extensive weakening effect on the basalt/phenolic and basalt/carbon/phenolic composites. It is also reasonable to conclude that carbon/phenolic composites were more susceptible to damage by this type of thermal cycling compared with the basalt/ phenolic and basalt/carbon/phenolic ones. The reduction in the ultimate tensile strength and modulus after thermal cycling was small compared with the results before thermal cycling for the basalt/phenolic and basalt/carbon/phenolic composites, but the effect was high for the carbon/phenolic composites. The effect of thermal cycles on the failure strain was insignificant. The fracture mode of the carbon/phenolic composites was the

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fracture of matrix and fibers, but the dominant failure mechanism of the basalt/phenolic and basalt/carbon/phenolic composites was fiber pull-out and delamination.

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