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Investigation of the Accelerated Ageing of Carbon-Epoxy Composites on their Mechanical Properties

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Abstract
In this work, carbon-epoxy composites obtained by an autoclave process were subjected to accelerated ageing in controlled conditions of temperature, water and UV radiation. Each composite was exposed to salt water at 60°C or UV radiation at 60°C over a period of 6 months. Changes within the composites were evaluated by mass and density variation, mechanical testing and also in terms of camouflage. Results did not show significant changes in mass loss or density, and it was found that mechanical properties had decreased slightly. The biggest change is in camouflage. It was observed that during accelerated ageing, the composites' reemission coefficient becomes too high and composites lose camouflage properties.

Keywords
accelerated ageing; carbon-epoxy composites; mechanical test; camouflage.

1. Introduction

Due to their high strength and low weight, composite materials have recently become increasingly popular in the field of civil engineering and military applications [1–4]. Composites can be constructed from many different types of fibers and matrix, with the choice depending on the usage and cost [5]. Considering the unique properties of composites like high strength, high modulus, excellent corrosion resistance, and low density, they can be used as alternative materials to conventional structures, especially in harsh environments [5, 6]. Among the materials which are applied in the industry, carbon-epoxy composites have a place of privilege because of their exceptional stiffness-to-mass ratio and excellent mechanical performance [7–9]. Nonetheless, existing composites have shown some degradation at the material level after being in utility for a number of years [10, 11]. Work performed by Kobayashi et al. [12] presented the degradation of carbon composite materials induced by a thermo-oxidative environment under two pressure conditions – about 0.3 and 0.5 MPa, at 180°C temperature and for an exposure time up to 2 000 hours. The study's results showed that accelerated ageing changes the local shrinkage, deformation, strength and weight. However, no change in the thermo-oxidative degradation mechanism was observed.

The effect of sulphuric acid (H_2SO_4) on the physical and mechanical properties of carbon fibre-reinforced polymer (CFRP) were investigated in study [13]. Accelerated ageing was performed with decreasing pH levels of sulphuric acid solution. Specimens after ageing were examined by means of a mechanical and non-destructive test. Scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS) showed material degradation and the impact of acid on the fibre-epoxy interface. Tensile testing of CFRP and epoxy resin sheets indicated up to 25 % reduction in strength [13]. Research in the field of accelerated ageing processes and how they affect the properties of carbon-epoxy composites was also conducted by Fasana et al. [14], Guzman et al. [15] and Barbosa et al. [16] presented results for carbon fiber/epoxy composites exposed for 3 months to different environmental conditions like: temperature, humidity and UV radiation. The results showed no significant changes in the composite material at the matrix-fiber interface. Fidan and co-workers [17] investigated the scratch resistance of carbon/epoxy composites used in aviation structures. Composites were exposed to the accelerated ageing process over four disparate thermal cycles (250, 500, 750, and 1,000 cycles, over which the temperature changed within the range +50°C to -50°C in one 40-min cycle of the thermal ageing process. Results showed a correlation between scratch properties and the number of accelerated ageing cycles. The worst scratch resistance was reported for the carbon/epoxy composite sample which was aged at 1,000. Accelerated ageing caused an increase in the coefficient of friction and penetration depth values.

As the contact of carbon composites with metallic materials in the presence of moisture promotes galvanic corrosion, which compromises mechanical performance, researchers investigated the effect of galvanic interaction on damage to carbon composites [18-24]. Whitman et al [24] studied the effect of galvanic current on the physicochemical, electrochemical and mechanical properties of an aerospace carbon fiber reinforced epoxy composite. Results showed that the composite was damaged under the cathodic conditions, which appeared linked to H_2O_2. The consequence was epoxy and or fiber sizing degradation,

which lead to debonding and cracking. In addition to carbon composites, glass, basalt and acrylic composites have also been subjected to accelerated ageing processes. Geretschläger and Wallner [25] investigated the behavior of a glass fiber-reinforced polyamide in hot air, elevated temperatures and water. For water exposure, tensile strength and Young's modulus were reduced as well as the glass transition temperature. For hot air exposure mechanical properties were unchanged but the sample mass was reduced. To confirm the degradation process, a tensile test was performed. The molar mass was about 25 % of the initial value at the onset of material cracking. Davies et al. [26] exposed four different composites (acrylic composites, basalt composites, flax fiber composites, carbon-reinforced polyamides) to conditions in deep sea pressure vessels. Water tanks were controlled at temperatures from 4 to 90°C, with continuous water renewal. The results showed that acrylic matrix composites are stable in seawater, basalt fiber has a retentive property as good as that of glass fiber, flax fiber composites show high weight gains, and that the degradation of carbon-reinforced polyamides is quite limited and predictable.

Exposure to UV radiation can rapidly accelerate the degradation of polymeric materials, especially impacting the surface morphology [27]. Ultraviolet radiation leads either a photolytic fission of chemical bonds (photolysis) or photooxidation, which causes the breakage of polymer chains. This phenomenon, known as a chain scission, produces free radicals and consequently reduces the molecular weight of polymer chains [28]. The mechanism of UV degradation involves the absorption of photons by chromophores present in the polymer matrix, usually in the form of double/triple bonds (especially aromatic), hydroperoxides, carbonyls and residues in the form of catalysts, resulting in surface discoloration and loss of gloss [29].

In a real environment polymer degradation is rarely caused by UV radiation exclusively. In fact, various factors influence polymer degradation simultaneously, leading to matrix deterioration. This includes high temperature, mechanical friction, water diffusion causing hydrolytic polymer degradation, evaporation of volatiles and others. Thus, the term synergistic degradation is frequently used to describe this complex behavior [30].

Ideally, composites should be tested in real time and in realistic environmental conditions. Usually, this is not profitable, because the time involved in such studies would delay product development. For this reason accelerated ageing techniques are preferred. A good method for fast identification of the impact of the main degradation mechanisms on polymer composites is accelerated ageing. Although many investigations of composites' accelerated-ageing are available in the literature [31, 32], the effects of ageing on the properties of these materials have not been fully described and understood yet. For this reason, the durability of composites, including carbon-epoxy composites, under environmental effects must be performed to ensure the safe design of composites and further promote the application of these materials in the future. For this purpose, accelerated ageing tests of carbon-epoxy composites were performed and the results presented in this paper. The primary goal of this research was to determine the durability of carbon-epoxy composites obtained under the influence of environmental conditions imitating the ageing process. Based on Kośla's research [33, 34] and literature, accelerated ageing was performed under two sets of conditions and the Arrhenius formula was also applied. Accelerated ageing was achieved by manipulating: time, temperature, UV and water conditions. Despite being a preliminary study, this work successfully revealed that there were no significant changes in the mechanical properties of the carbon epoxy composites, but at the same time, changes in camouflage properties at wavelengths within the range 700-1100 nm of the IR spectrum were observed. The novelty of the works presented in the article is the development of carbon composites based on a newly developed resin structure (patent no. P.443393) and the assessment of the properties of the composites produced, especially in the context of their camouflage ability. The novelty of the work is also the assessment of the impact of accelerated aging processes on physical, mechanical and camouflage properties. The camouflage capabilities of carbon-epoxy composites are important due to their subsequent use in the construction of UAVs used by the army, police and border services. UAVs dedicated to work in the above-mentioned units should be as undetectable as possible for the environment, which affects the success of operations.

2. Materials and Methods

2.1. Materials

Sample 1: Prepreg was made of Style 462-2 (GRM System, Poland) carbon fabric impregnated with epoxy resin prepared by the Adam Mickiewicz University Foundation, Poland (resin content with a weight percent of around 30 %). The composition of the resin is in accordance with the patent application of the Republic of Poland Patent Office no. P.443393 "A multi-component composition that hardens and modifies epoxy resins". Due to the content of the project co-financing agreement, the property rights to the subject of dissolution are vested in the Ministry of National Defense and details of the resin cannot be published at this stage. The polymerization process took place in autoclaves at the Silesian Science and Technology Centre of Aviation Industry Ltd. (temperature 110°C during 4-5 h). Carbon composites were obtained from 10 layers of prepregs. The final composite had an area density of 3150 g/m^2.

Sample 2: To compare our materials, prepregs based on commercial resin (Araldite® LY 1556*/Aradur® 1571*/ Hardener XB 3403*/Accelerator 1573*, HUNTSMAN International LLC.) and Style 462-2 (GRM System, Poland) carbon fabric were obtained. The polymerization process took place in autoclaves at the Silesian Science and

Technology Centre of Aviation Industry Ltd. (temperature 110°C during 4-5 h). Carbon composites were obtained from 10 layers of prepregs. The final composite had an area density of 3110 g/m^2.

2.2. Accelerated ageing

Due to the fact that carbon epoxy composites can be used in applications such as aerial or naval autonomous unmanned platforms, accelerated ageing was performed under two sets of conditions:

– accelerated ageing under UV radiation and moisture. The samples were put in a Xenotest climatic chamber, XENOCHROME 320 filter (300-400 nm wavelength). Action cycles: 5 h of UV radiation at 60 ± 3°C, light intensity equal to 45 W/m^2, humidity 10 ± 5 %, 1 h sprinkler irrigation at 25 ± 5°C, and light intensity equal to 0 W/m^2; UV was applied daily for 6 months
– accelerated ageing under water. The samples were put in a closed container enabling complete submersion of samples in 30 % NaCl simulating sea water and the entirety was put in a chamber at 60°C. UV was applied daily for 6 months. To protect the composite against environmental influences, the edges were sealed before submerging in water. The edge sealing involved taking a synthetic resin coating (epoxy resin - EY3804 A/B (H.B. Fuller, USA)) and applying it to the cut edges of the composite component with a brush or from a cartridge.

2.3. Density and mass

The density of solids determined by the hydrostatic weighing method was considered. By classical definition, hydrostatic density measurement involves weighing an object of known mass while it is suspended from a balance in a liquid having some assumed value of density. The indicated loss of mass is equal to the mass of the displaced liquid, making it possible to calculate the true volume of the weighed object, of unknown density. To measure the mass of a weight precisely in air, buoyancy correction of air for the volume of the weight is required. The densities of the objects examined were determined according to the hydrostatic method and were calculated using the formula (1):

$$\rho = \frac{A}{A-B} \times \rho_0 \qquad (1)$$

where: ρ – density of the sample [g/cm^3], A – weight of the sample in air [g], B – weight of the sample in fluid [g], ρ_0 – fluid density = 0.99 [g/cm^3] (temperature of density measurement was 20°C).

Mass was determined by the estimation of three measurements of the samples' masses using an analytical balance [g].

2.4. Thermogravimetric analysis (TGA)

To determine the thermal decomposition of the samples, thermogravimetric analysis was performed. TGA was carried out on a TGA/DSC 3+ Mettler Toledo [TGA/DSC 3+ Mettler Toledo, Switzerland]. The air flow was set to 60 ml/min and a heating profile from 50°C to 850°C with a heating rate of 15°C/min. $T_{5\%}$ - temperature of 5 % weight loss, which was taken as the temperature at the beginning of degradation and char residues were carried out. $T_{50\%}$ - temperature of 50% weight loss.

2.5. Camouflage

Camouflage was determined based on Defense Standard: NO-84-A203:2020 and developed based on the standard procedure PBCH-01/2014 of the Institute of Security Technology "MORATEX". Spectral color characteristics of materials are regulated by the defense standard: NO-84-A-203. This standard outlines the acceptable range of reemision coefficients for individual colors. It includes, for example, requirements for colors of fabrics with camouflage print used in Central European conditions. These are presented in the standard in the form of tables and graphs, percentage limits in which the spectral spectrum should be present, the reflection coefficient in a given wavelength for a given color on the camouflage. The tests were carried out using a UV-VIS-NIR double-beam spectrophotometer [V-670, Jasco, Japan] with a wavelength range of 400-1100 nm and a UV-VIS-NIR integrating ball with a diameter of 150 mm and wavelength range of 200-2200 nm.

2.6. Mechanical test

Tests of the composites' mechanical parameters like tensile, bending and interlayer shear strength were carried out according to the following:

– PN-EN ISO 14125 Fibre-reinforced plastic composites — Determination of flexural properties
– DIN EN 2850:2017-06, Aerospace series - Carbon fibre thermosetting resin unidirectional laminates - Compression test parallel to fibre direction.

The value of each parameter is the average value of 5 samples. Tests were carried out at ambient temperature using research equipment of the mechanical laboratory at Silesian Science and Technology Centre of Aviation Industry Ltd [MTS Criterion 45, MTS 319.10, MTS, USA]

2.7. SEM microscopic analysis

The samples obtained were examined under a scanning electron microscope (SEM) [Quanta 200 (W), FEI, USA]. The samples were sputtered with a 20 nm gold layer in a Q 150R S vacuum sputtering machine. The studies were carried out in a HiVac (high vacuum) vacuum mode at an electron beam accelerating voltage of 5 kV. An ETD detector was used.

	Before accelerated ageing		After 6 months of accelerated ageing under UV		After 6 months of accelerated ageing under water	
	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2
Mass [g]	2.84 ± 0.02	2.81 ± 0.02	2.87 ± 0.02	2.84 ± 0.02	2.86 ± 0.02	2.81 ± 0.02
Density [g/cm^3]	1.49 ± 0.02	1.49 ± 0.02	1.51 ± 0.02	1.53 ± 0.02	1.52 ± 0.02	1.53 ± 0.02

Table 1. Mass and density of composites during accelerated ageing under UV conditions or water

	Before accelerated ageing		After 6 months of accelerated ageing under UV		After 6 months of accelerated ageing under water	
	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2
$T_{5\%}$	218 ± 27°C	297 ± 19°C	327 ± 21°C	298 ± 15°C	314 ± 22°C	287 ± 17°C
$T_{50\%}$	593 ± 41°C	675 ± 37°C	659 ± 28°C	674 ± 42°C	653 ± 33°C	652 ± 39°C

Table 2. 5% (T5) and 50% (T50) temperature-induced mass loss of samples

3. Results and Discussion

3.1. Density and mass

The change in mass and density of samples exposed to water at 60°C or UV radiation at 60°C was verified. The results of these studies are presented in Table 1.

As can be seen in Table 1, differences in mass change remain within the error limit and do not change significantly. In the case of density testing, for all samples a small increase in the value was observed, however they remained within the assumed tolerance limits.

3.2. Thermogravimetric analysis (TGA)

$T_{5\%}$ and $T_{50\%}$ are presented in Table 2. Figure 1 shows the sample mass reduction of samples during TGA.

Both samples show the same thermal character; no weight loss up to 200°C, which proves their thermal stability in this temperature range. After this temperature degradation of the epoxy resin starts, and after 500°C - the degradation of carbon fibres. The TGA curves of Sample 2 show no effect of ageing, as compared to Sample 1, where an in-crease in the value of T5% and T50% after ageing is observed. The differences in TGA curves are probably due to the chemical structure of the resins, especially, the

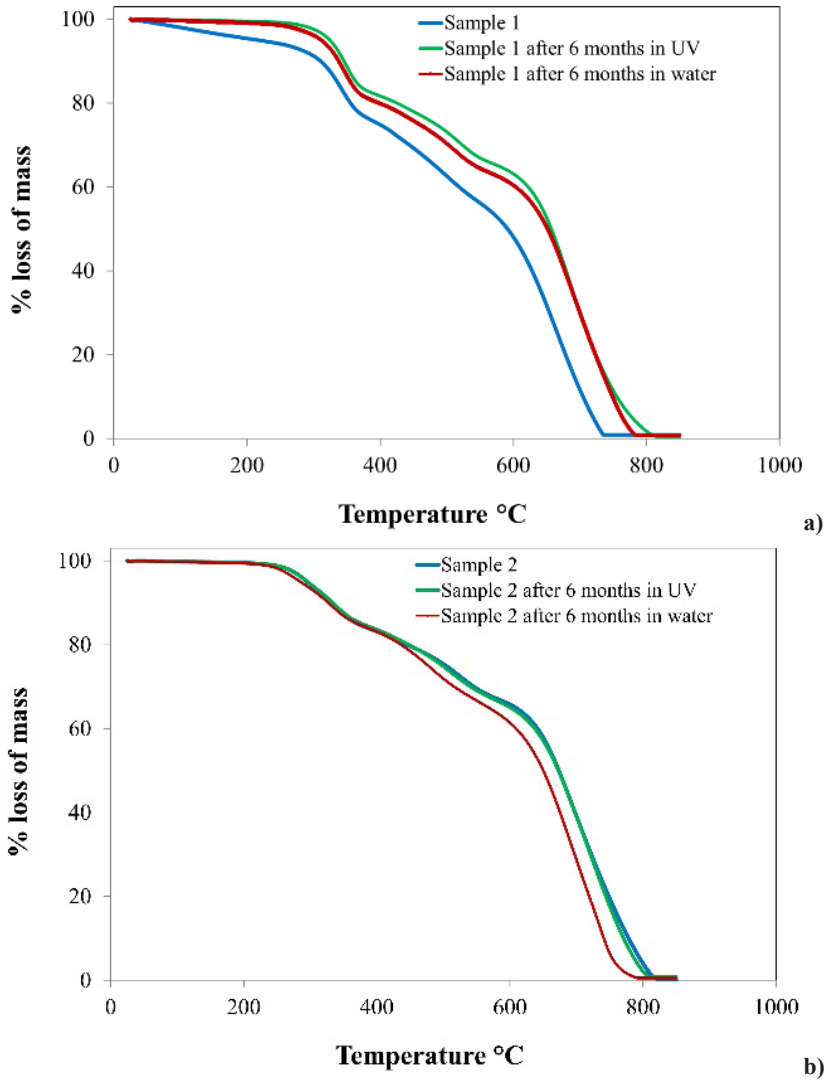


Fig. 1. Thermogravimetric analysis of a) Sample 1 & b) Sample 2

possible photo-radiolysis of the ionic liquid cation, which is one of the resin components, and cation aggregation when under UV-light irradiation. Epoxy resins possess aromatic groups with a strong absorption in the UV range. This makes epoxy structures vulnerable to UV degradation. When oxygen molecules in the air are exposed to UV light radiation, oxygen radicals are produced. Radicals are also formed from broken bonds, which are highly reactive and have a very short life. These formed radicals attack the surface of the epoxy resin and react with them. There are various mechanisms proposed for UV degradation [35]. Additionally, conventional epoxy resin fails in long-term soaking in water as pores can be formed [36]. To avoid or reduce degradation, inorganic and organic UV absorbers, as well as stabilizers have been used. These stabilizers can act as polymer degradation inhibitors. They either stop or at least slow down the degradation reaction rate by scavenging produced radicals. In the case of the developed resin, additives in the form of ionic liquids increased the stability of the resin in degradation processes, therefore the course of the Tg curves for Sample 1, both non-aged and subjected to aging processes, shows much greater differences than in the case of Sample 2. Moreover, the increase in the value of T5% and T50% after ageing is caused probably due to the chemical structure of the resins, especially the possible photo-radiolysis of the ionic liquid cation, which is one of the resin components, and the cation aggregation when under UV-light irradiation. However, the detailed composition of the resin of Sample 1 is in accordance with the patent application of the Republic of Poland Patent Office no. P.443393 "A multi-component composition that hardens and modifies epoxy resins".

3.3. Camouflage

This part of investigations discusses the effectiveness of infrared camouflage at wavelengths in the range 700-1100 nm for the carbon-epoxy composite obtained. The reemission coefficient of the sample before accelerated ageing is shown in Figure 2. The reemission coefficient of accelerated ageing of the composite under UV conditions is shown in Figure 3, and that of the accelerated ageing of the composite under water conditions in Figure 4.

The reemission coefficients of samples before the ageing process meet the assumption of infrared masking, i.e., above 700 nm, thus being within the limits of acceptance for black color. After exposure to UV radiation for a period of 6 months, the composite loses its ability to camouflage in the wavelength range of 400 – 1000 nm due to both samples being above the upper limits for black color (Figure 4). It is known that epoxy resistance to UV is rather poor and visible yellowing is the first indication of photooxidation processes taking place on the surface of the material.

3.4. Mechanical testing

The mechanical properties of non-aged carbon-epoxy composites, composites after 6 month's immersion in salt water and 6 months of accelerated ageing under UV-radiation conditions are shown in Table 3.

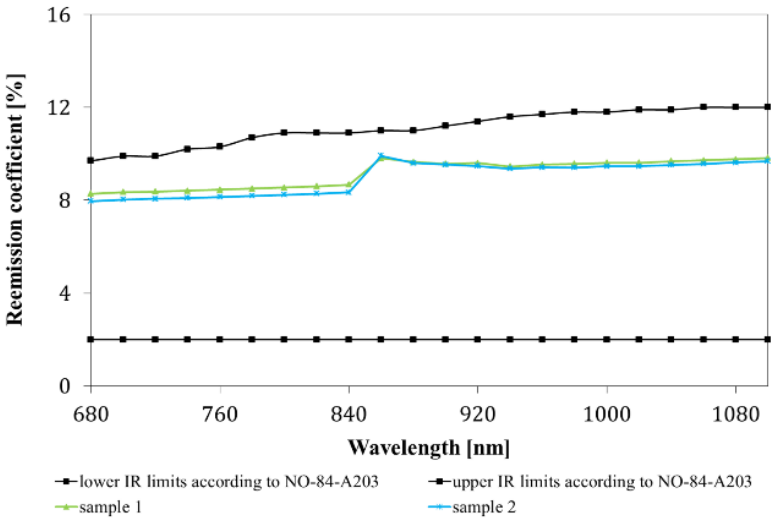


Fig. 2. Reemission coefficient of composite before accelerated ageing

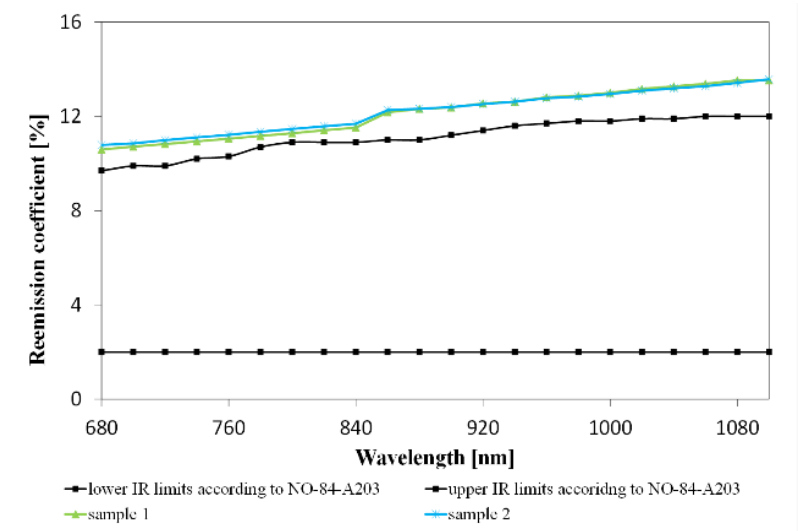


Fig. 3. Reemission coefficient of accelerated ageing of composites under UV conditions after 6 months

	Before accelerated ageing		After 6 months of accelerated ageing under UV		After 6 months of accelerated ageing under water	
	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2
Compressive strength (MPa)	651.2 ± 35.4	591.9 ± 69.2	542.2 ± 42.5	483.5 ± 70.9	468.8 ± 88.5	308.6 ± 121.1
Flexural strength (MPa)	966.1 ± 25.7	943.4 ± 27.4	870.6 ± 27.7	948.3 ± 39.6	902.2 ± 45.7	688.4 ± 36.2
Flexural modulus (GPa)	57.2 ± 1.3	60.3 ± 1.8	55.0 ± 1.7	55.7 ± 4.1	55.0 ± 1.2	57.6 ± 1.0

Table 3. Mechanical parameters of composites before and after accelerated ageing

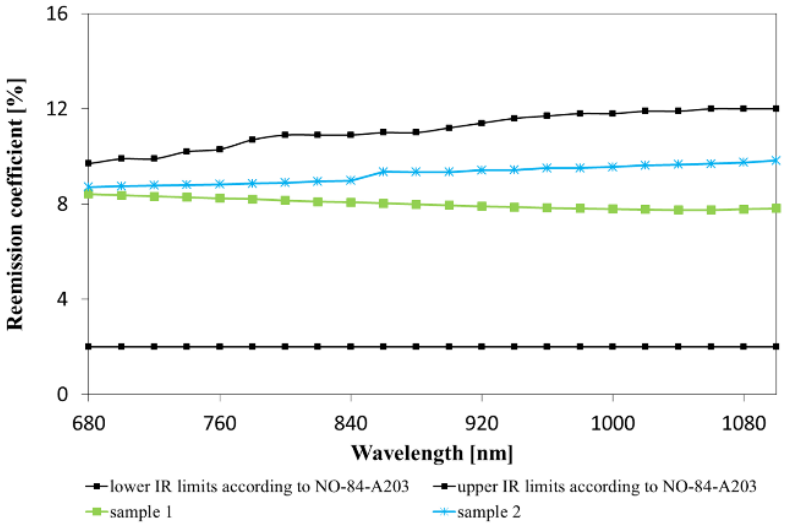


Fig. 4. Reemission coefficient of accelerated ageing of composites under water conditions after 6 months

Analysis of the results of strength tests shows that the compressive strength decreased as a result of the ageing processes. After ageing under UV radiation, a decrease in strength of around 17 % was observed for Sample 1 and around 18 % for Sample 2. After ageing in seawater, it decreased to around 28 % for Sample 1 and around 47 % for Sample 2. Results suggested that Sample 1, with the newly developed resin, is more resistant to accelerated ageing than Sample 2. In the case of flexural strength, a decrease in the value of strength after ageing can be observed. A greater decrease was observed in the ageing process under UV radiation than in sea water. Moduli for the tested composites after the ageing processes did not change significantly, and their differences remain within the error limits. This means that the elasticity of the material remained unchanged or slightly decreased; therefore, the composite did not undergo significant destruction/damage causing a change in the elastic characteristics of the composite. Various parts of an epoxy backbone are susceptible to photodegradation effects in different ways.

3.5. SEM microscopic analysis

Scanning electron microscopy of the non-aged carbon-epoxy composites and composites after 6 month's immersion in salt water and 6 months of accelerated ageing under UV-radiation conditions are shown in the Figures 5 and 6.

After accelerated ageing under UV-radiation conditions as well as in water, many cavities of different size and shape as well as cracks on the surface were observed, which is proof of the degradation of the samples. The effects are much more pronounced for samples aged under UV, where some degradation phenomena are visible, in particular some evidence of de-bonding.

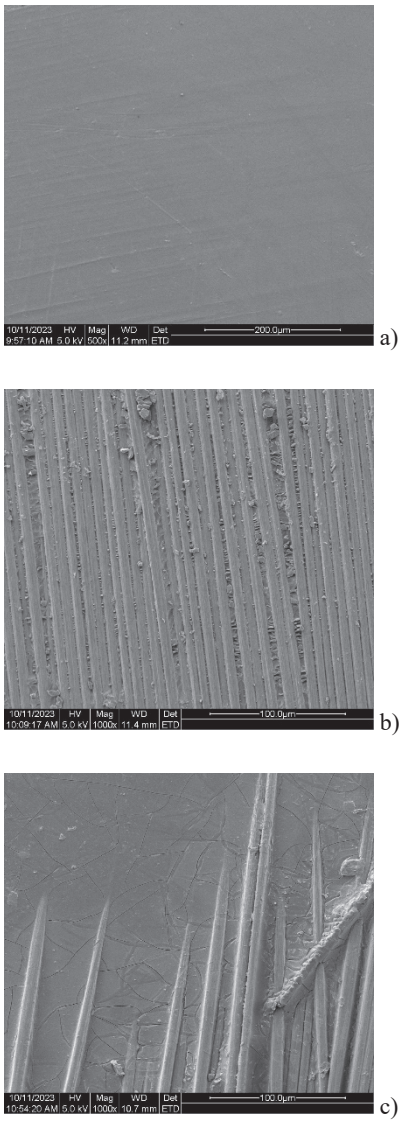
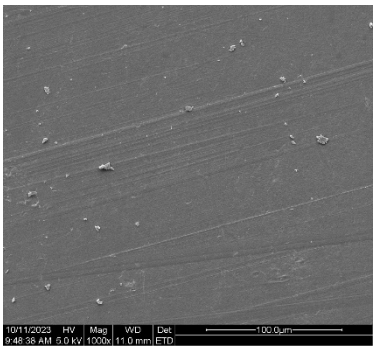
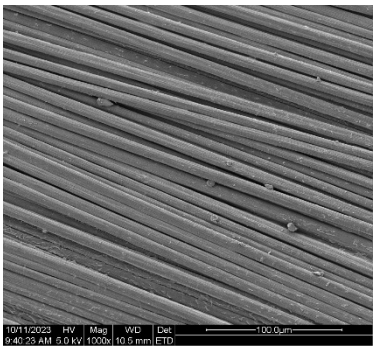


Fig. 5. Scanning electron microscopy of Sample 1 at magnification x1000: a) non-aged b) after 6 months of accelerated ageing under UV-radiation conditions c) after 6 month's immersion in salt water



a)



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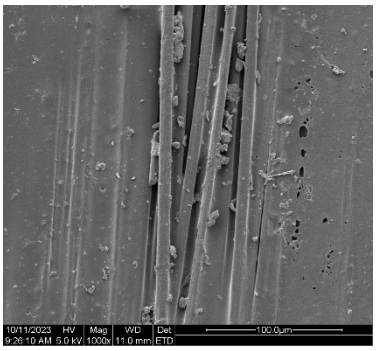

c)

Fig. 6. Scanning electron microscopy of Sample 2 at magnification x1000: a) non-aged b) after 6 months of accelerated ageing under UV-radiation conditions c) after 6 month's immersion in salt water

4. Conclusions

The aim of this study was to investigate the effects of accelerated ageing on composites obtained in autoclave from Style 462-2 carbon fabric impregnated with epoxy resin. Carbon-epoxy composites were exposed to salt water and UV radiation at a temperature of 60°C for a period of 6 months. All samples did not show mass loss or density changes caused by the accelerated ageing procedure. Although significant changes in mass or density were not observed, the carbon-epoxy composites tested in this work were altered by the ageing procedure. Changes within the material are evidenced by a slight decrease in mechanical parameters, such as compressive strength and modulus, shear strength, flexural strength or flexural modulus, and some loss of ability to provide camouflage in the 700-1100 nm wavelength range. Camouflage studies suggest changes in the chemical structure of the polymeric matrix, which were caused by accelerated ageing.

Further studies should be focused on longer accelerated ageing periods, and observation of surface texture and chemical changes is needed in order to evaluate processes occurring within carbon-epoxy composite materials.

To conclude the results it can be seen that:
- significant changes in mass and density were not observed,
- thermal analysis carried out after the accelerated ageing of the tested samples showed no changes in the thermal character of the samples,
- composites obtained are less resistant to UV radiation than to sea water,
- composites after 6 months of accelerated ageing in water still provide camouflage, while composites accelerated under UV do not,
- moduli for tested composites after ageing processes did not change significantly,
- the compressive and flexural strength of the carbon-epoxy composite obtained change in the range 5-28 % after accelerated ageing, which may be the result of chemical changes taking place in the composite matrix as a result of the interaction of UV radiation, temperature and salt water,
- Sample 1, with newly developed resin, is more resistant to accelerated ageing than Sample 2, with commercial resin.

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The Authors declare there is no conflict of interest.

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