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Influence of Shelf Life on Mechanical Properties of Glass Fibre Reinforced Composites

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Abstract: *The present paper was focused on studying the influence of shelf life of an epoxy matrix on the mechanical properties of glass fibre reinforced composites. For the study, two types of the same epoxy system were used, one during its shelf life and one out of its shelf life. The reinforcement used consisted in E-glass fibre fabric. Mechanical investigations were realized in order to compare the materials in terms of loss of mechanical strength and elastic properties. Therefore, three mechanical tests were performed: tensile tests, in-plane shear and open-hole tensile tests. The results showed that the shelf life affects the mechanical properties of the polymeric composite. A decrease of 24% in tensile strength was recorded along with a 28% decrease of the in-plane shear strength and 55% of open-hole tensile strength for the composite manufactured with the out of shelf life epoxy system compared with the other composite. An overall reduction of mechanical strength and elastic properties of the composite material was observed, primarily due to polymeric matrix degradation, which after long periods it could be prone to brittleness and susceptible to delamination and fracture. The thermogravimetric analysis showed that thermal induced changes are happening at a higher speed in the out of shelf life composite, a lower mass loss being registered for new epoxy composite.*

Keywords: *epoxy, GFRP, shelf life, open-hole, in-plane shear*

1.Introduction

Since their discovery in 1940s, polymeric composites were continuously studied and integrated in multiple applications, including aerospace [1, 2], automotive industry [3-5] and wind turbines [6, 7]. Carbon fibre reinforced polymeric composites (CFRP) are the most used composite materials for advanced applications where high loads are implied, while in applications where lower loads or low signal attenuating properties are implied, the glass fibre reinforced polymeric composites (GFRP) are preferred [8]. Due to their advantages and cost related reasons, GFRP are suitable materials for low range unmanned aerial vehicles (UAVs) components, especially research or civil grade UAVs. Many glass fibres types and resins are available, but the most used GFRP precursors are the E-glass and epoxy resins due to their low cost and high performances compared with other GFRPs precursors [8 - 10].

Over the years, many studies were conducted regarding the properties of glass fibre reinforced polymeric composites and their usage [11, 12], for example Spanu et.al. [13] conducted a study regarding the compressive properties of GFRP, they obtained many results for the same material as it was characterized by a high degree of inhomogeneity. Bunea et.al. [14] tested various polymeric composites reinforced with different fibres, including glass fibres, Iliescu et al. [15] studied the mechanical characteristics of GFRP both by theoretical and experimental methods. Stefanescu et.al. [16] studied the low velocity impact properties of different hybrid fibre reinforced composite laminates and it was concluded that the best performances were obtained for laminates that have glass fibre external layers. Beside these studies, many researches were conducted in the last decade regarding hybrid fibre

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reinforced polymeric composites [7, 17, 18], 3D printed fibre reinforced composites [19-21], and even self-healing fibre reinforced polymeric composites [22, 23].

Although various studies were conducted on fibre reinforced polymeric composites, there are still some aspects that have to be addressed, like what happens with the uncured polymeric systems which exceed their shelf life (or their expiration date) or recycling methods that can be applied for such materials. Usually, product suppliers sell polymeric systems like epoxy (composed of epoxy resin and hardener) in large quantities which sometimes cannot be used if you're not a mass producer. In order to reduce waste, the influence of shelf life on resin systems should be addressed to determine if they can be used after the shelf-life date was surpassed.

Currently, a limited number of research papers are available regarding the influence of shelf life of polymers on its properties, moreover the existing papers are focused primary on dentistry polymeric composites. For example, Cees de Lange et. al. [24] studied the shelf life and storage conditions influence on resin-based composites' properties. The materials were kept for various periods of time (up to 15 months) in two different environments, in the dental office to simulate storage under normal working conditions and in a refrigerator. After conducting the tests, they observed that the rate of strengthening and ultimate strength of composite resins depend on the material's storage conditions and a decrease in the ultimate strength could be a sign of less effective polymerization. Other study [25] showed that the mechanical properties of resin composites designed for dental restauration weren't affected after the expiration date was exceeded by 1 year.

A case study on the influence of epoxy shelf life was published online by Watson [26]. The experiments were made on a 15 years old epoxy resin. In this case study was mentioned that the problems with older epoxy aren't related to the material's age but to the presence of contamination like moisture, hardener-resin cross contamination, dirt or contact with other chemicals. Other studies are available regarding the durability of epoxy resin and fibre composites after immersion in different fluids [27] or regarding the feasibility of using recyclable epoxy resin and recovered glass fibres for manufacturing recycled thermoset composites [28, 29].

The lack of many data regarding the out of shelf life polymeric composites' properties and the manufacturing waste reduction were the main reasons that led to the present study. A GFRP material intended for manufacturing of small range UAVs components was used, and the influence of the polymeric matrix shelf life on the GFRP mechanical properties was studied.

2.Materials and methods

For the present study two types of epoxy glass fibre reinforced composites (GFRP) were manufactured using the same twill 2x2 E-glass fibre (280 g/m^2) and two epoxy systems composed of Resoltech 1050 epoxy resin and Resoltech 1058 hardener, one system was used during its shelf-life (further named New) and one was used out of its shelf-life (according to the manufacturer's indications, it was expired for 2 years and it was further named Old). Even if the epoxy system's shelf life was surpassed, it was kept all over the period in good environmental conditions (all elements were kept in sealed original containers at room temperature of 24°C ± 4°C and shielded from light). The epoxy system components were mixed according to the material data sheet, by weight percent (100:35).

The mechanical testing matrix was selected based on the application for which the composites were intended for, manufacturing of components for a small UAV that is intended to be used as a research tool. The composite material is subjected to tensile and flexural forces and the components are assembled by different means (for example some components can be joined by rivets/ fasteners) and can cause stress concentrators. Therefore, three mechanical tests were selected for this study: tensile test, in-plane shear test and open-hole test.

Two type of composite laminates were manufactured by hand lay-up with both epoxy systems: laminates with a $[0°, 90°]_4$ stacking sequence and laminates with $[45°, -45°]_4$ stacking sequence. The curing process was realized in a vacuum assisted oven (POL EKO SLN 240) at 60°C for 5 h using an 850 mbar vacuum pressure, and afterwards were kept at room temperature for one week.

Three type of specimens were cut using a water jet cutting machine: specimens for tensile tests (dimensions 250x25x2 mm established based on ASTM D 3039/D 3039M standard [30], characterized by a [0°, 90°]$_4$ stacking sequence), specimens for in-plane shear tests (dimensions 250x25x2 mm established based on ASTM D3518/D3518M [31], characterized by a [+45°, -45°]$_4$ stacking sequence) and specimens for open-hole tensile tests (dimensions 300x36x2 mm with a 6 mm centred hole established based on ASTM D5766/D 5766M [32], characterized by a stacking sequence of [0°, 90°]$_4$). No tabs were used for all specimens. Each specimen batch was composed of 6 specimens per test, as if one specimen was damaged during the cutting process or during handling it could be excluded from the batch and a minimum value of 5 specimens per batch could be tested.

Mechanical tests were performed at room temperature using Instron 3369 testing machine with a load cell of 50 kN and using a displacement of 2 mm/min. Damage modes of tensile and open-hole specimens were established by visual analysis according to standard failure modes presented in Table 1.

Table 1. Tensile test failure codes/typical modes according to [30]

Failure type		**Failure area**		**Failure location**	
Angled	A	Inside grip/tab	I	Bottom	B
Edge Delamination	D	At grip/tab	A	Top	T
Grip/Tab	G	<1W from grip/tab	W	Left	L
Lateral	L	Gage	G	Right	R
Multi-mode	M (xyz)	Multiple areas	M	Middle	M
Long. Splitting	S	Various	V	Various	V
eXplosive	X	Unknown	U	Unknown	U
Other	O	-	-	-	-

Electron microscopy analysis was performed in the fractured area of the in-plane shear tested specimens and open-hole tensile tested specimens. For this analysis a FEI F50 Inspect SEM was used.

Samples for thermogravimetric analysis (TGA) were taken from the composites laminates in order to study the decomposition process of the composite material, mainly of the polymeric matrix. This analysis can provide important information regarding the thermal stability of the polymeric composite. The thermal analysis was conducted on a Shimadzu DTG-TA-50H equipment at a heating rate of 10°C, samples were scanned in air atmosphere, at a ramp rate of 10°C/min from ambient temperature to 500°C, while mass loss trace was simultaneously recorded with the equipment software. This analysis was performed in order to observe the mass loss with increasing temperature and phase changes for both composite samples.

3.Results and discussions

3.1. Tensile testing

Two batch of specimens were tested. In case of the new epoxy composite batch, one specimen was damaged during the cutting process thereby only 5 specimens were tested. The average mechanical test results are presented in Table 2 and stress – strain curves can be observed in Figure 1a and b.

Table 2. Average results registered in case of the two composite materials

Material/Property	**Tensile strength [MPa]**	**Strain [mm/mm]**	**Elastic modulus [GPa]**
Composite 1 (New resin system)	**233**	**0.04**	**5.68**
St.dev.	9.72	0.005	1.38
CV [%]	4.17	11.80	24.35
Composite 2 (Old resin system)	**177**	**0.028**	**6.63**
St.dev.	9.18	0.001	0.24
CV [%]	5.20	4.16	3.64

St.dev. – standard deviation
CV – coefficient of statistic variation

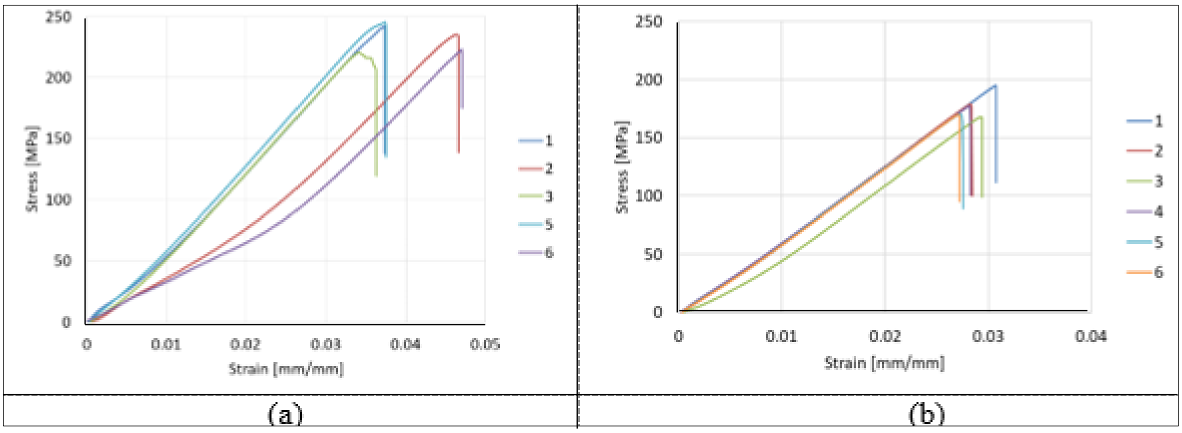


Figure 1. Stress-strain curves: a) new epoxy composite; b) old epoxy composite

As can be observed from both Table 2 and Figure 1, the tests revealed that the new epoxy composite had an average tensile strength higher than the average value determined in case of old epoxy composite (a reduction with approximative 24% was recorded). A difference was observed also in case of the material's elastic modulus, the old composite being characterized by a higher value (calculated in the strain domain between 0.005 and 0.025 mm/mm). Table 3 presents the failure codes for the tensile test specimens and in plane shear test failure modes.

Table 3. Failure codes according to [30]

Tensile test failure modes		**In plane shear test failure modes**	
New epoxy comp.	**Old epoxy comp.**	**New epoxy comp.**	**Old epoxy comp.**
LWT	LWM	AWT	LWB
LWB	LWT	AGT	AWT
LWM	LWL	AWT	MWB
LWT	EWL	AWB	AGM
LWM	GAT	AGT	AWB
-	XAT	AGB	AGM

3.2. In-plane shear testing

The average in-plane shear test results are presented in Table 4 and stress – strain curves can be observed in Figure 2 a,b.

Table 4. Average results registered in case of the two composite materials

Material/Property	**In-plane shear strength [MPa]**	**Strain [mm/mm]**	**Elastic in-plane shear modulus [GPa]**
Composite 1 (New resin system)	**144**	**0.06**	**4.25**
St.dev.	5.17	0.004	0.65
CV [%]	3.59	5.94	15.29
Composite 2 (Old resin system)	**103**	**0.09**	**3.42**
St.dev.	5.80	0.01	0.35
CV [%]	5.66	13.23	10.20

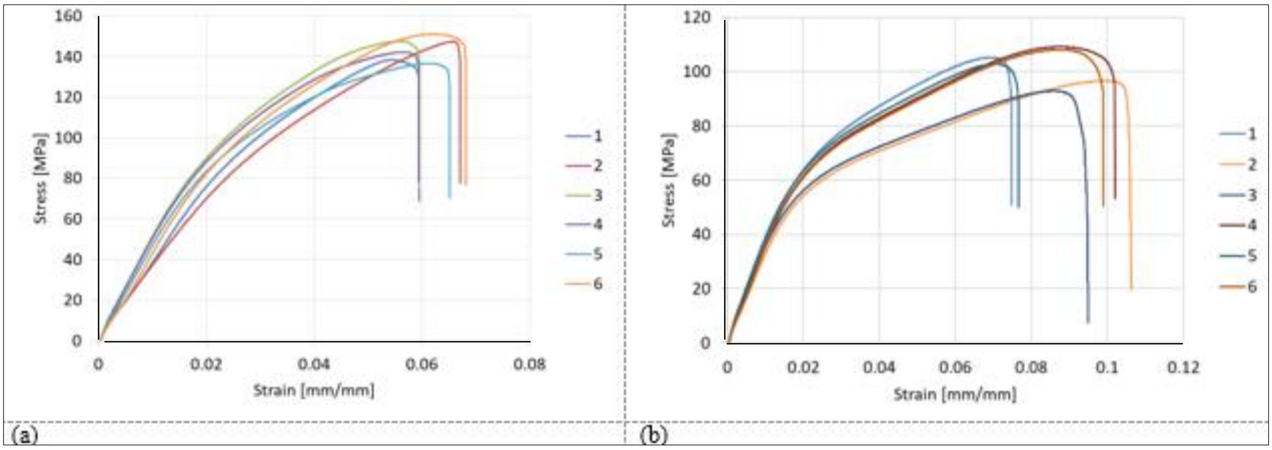


Figure 2. Stress-strain curves resulted in case of in-plane shear tests:
a) new epoxy composite; b) old epoxy composite

Compared with material behaviour registered in case of tensile tests, in-plane shear tests of new epoxy composite presented less scattered results compared with the old epoxy composite. In-plane shear test results showed that the new epoxy composite had a higher mechanical strength compared with the average results of old epoxy composite (an approximative reduction of 28% of the in-plane shear strength was registered). In this case though, the old epoxy composite shows a lower elastic modulus (calculated in the strain domain between 0.005 and 0.015 mm/mm), which was not the case of the results obtained in case of tensile tests.

SEM images realized in the specimens' fractured area (Figures 3 and 4) show broken fibres and damaged polymeric matrix for both composites.

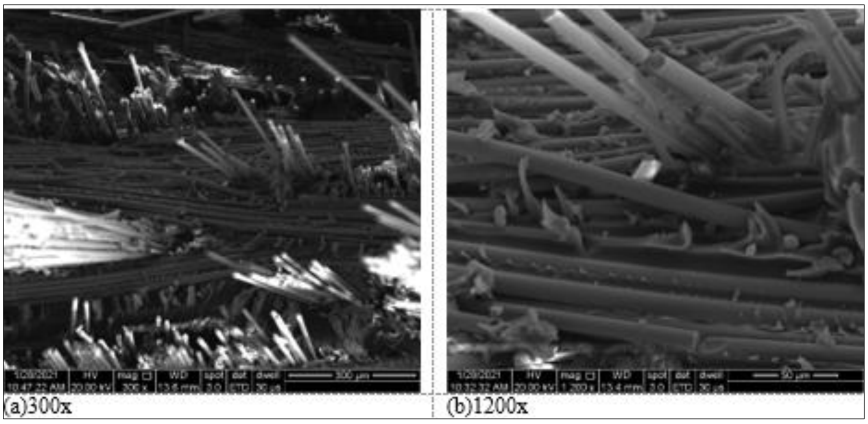


Figure 3. SEM images with the fractured area of the new epoxy composite

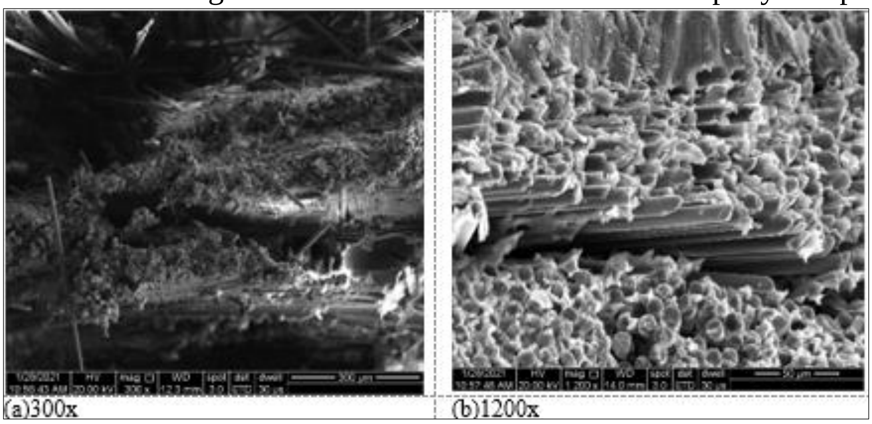


Figure 4. SEM images with the fractured area of the old epoxy composite

3.3.Open-hole testing

The average results determined in case of the two composite materials are presented in Table 5 and stress – strain curves can be observed in Figure 5 a) and b).

Table 5. Average results registered in case of the two composite materials

Material/Property	Tensile strength in case of open-hole specimens [MPa]	Strain [mm/mm]	Elastic modulus [GPa]
Composite 1 (New resin system)	**173**	**0.03**	**7.01**
St.dev.	4.73	0.001	0.35
CV [%]	2.74	2.01	4.98
Composite 2 (Old resin system)	**77.83**	**0.02**	**5.61**
St.dev.	3.29	0.002	0.78
CV [%]	4.22	9.70	13.88

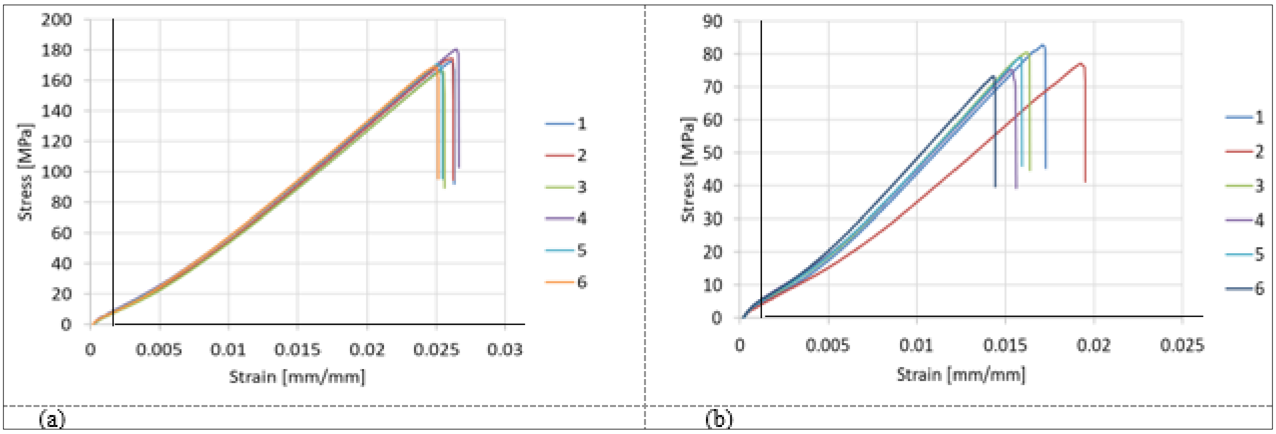


Figure 5. Stress-strain curves: a) new epoxy composite; b) old epoxy composite

As it was observed in case of in-plane shear tests results, open-hole tensile tests results showed that the old epoxy composite show more scattered values and higher coefficient of statistic variations compared with the new epoxy composites. The test results showed that the new epoxy composite had a higher mechanical strength compared with the average results of old epoxy composite (an approximative reduction of 55% of the open-hole tensile strength was registered).

In terms of elastic modulus (calculated in the strain domain between 0.005 and 0.014 mm/mm), the new composite showed a higher elastic modulus compared with the old composite.

Regarding the fracture mode, only one specimen was damaged by AGM mode, while all the other specimens presented an LGM damage code. Figure 6 presents representative images with the open hole testing specimens after they were tested.

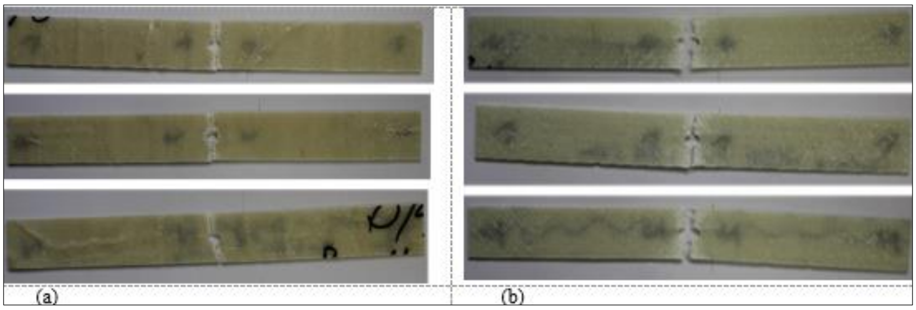


Figure 6. Representative images with specimens after open hole testing: a) old epoxy composite; b) new epoxy composite

SEM images were taken both in the open-hole area and in the specimens' fractured area, they are presented in Figures 7 and 8. As it can be observed from the images presented in Figure 7, the damage in case of the old composite is more extensive, moreover in the images from Figure 8 delamination signs can be observed in the hole area compared with the new epoxy composite where even after testing the open-hole integrity was kept.

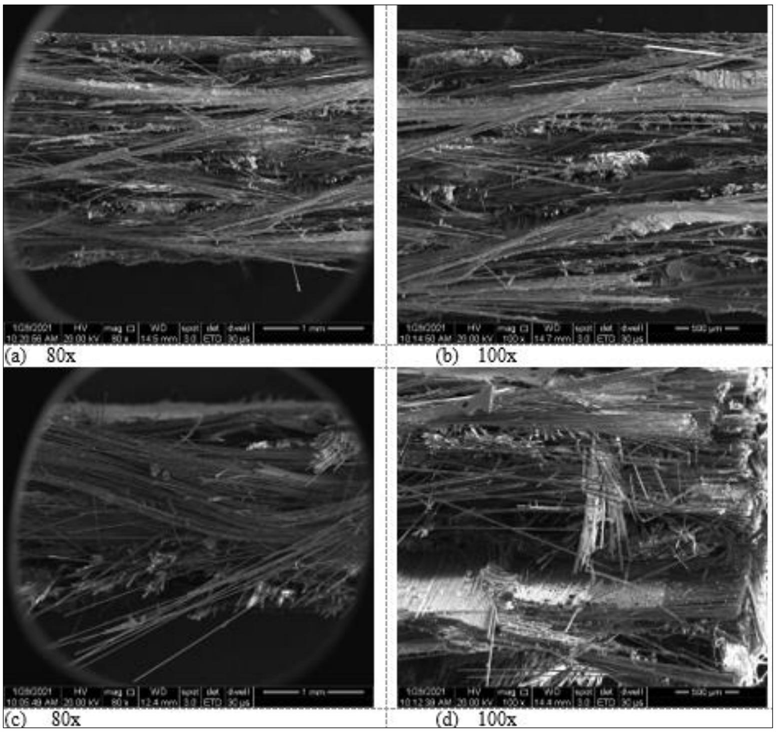


Figure 7. Representative images taken in the fractured area for: a, b) new composite; c, d) old composite

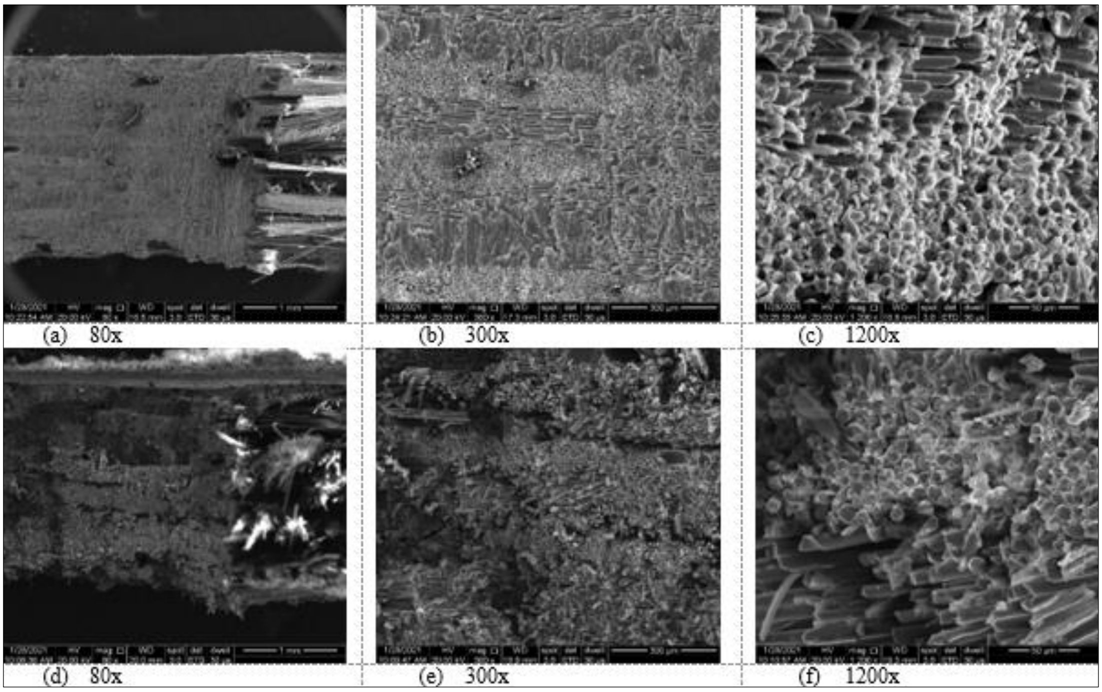
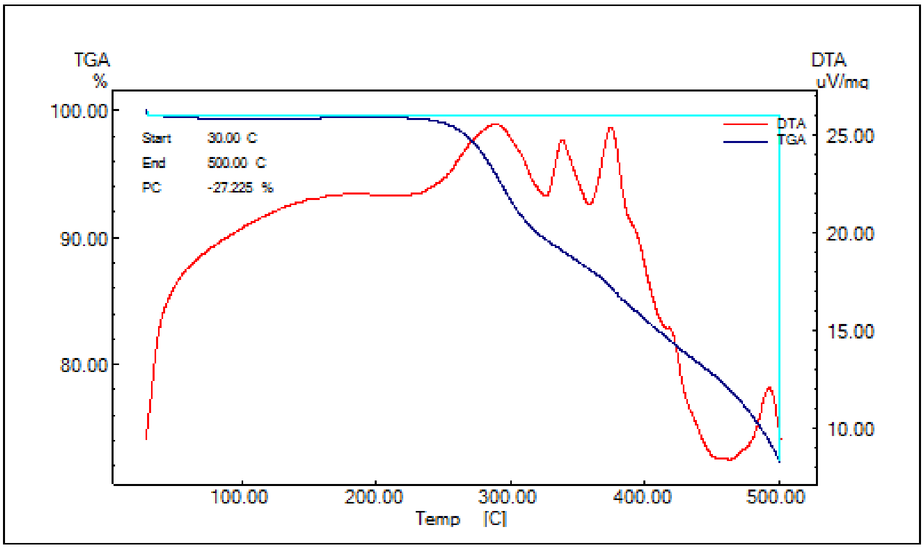


Figure 8. Representative images taken in the open-hole area for: a, b, c) new composite; d, e, f) old composite

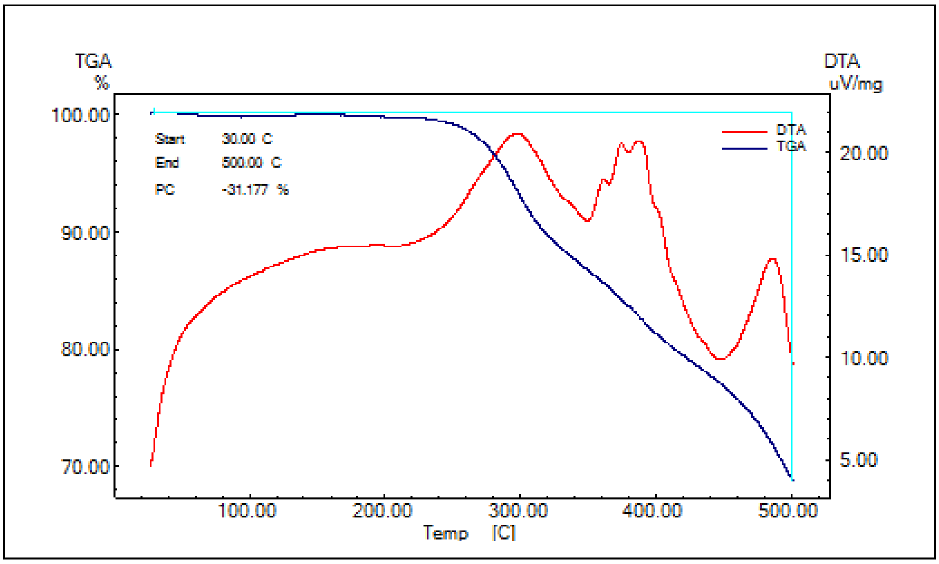
Comparing the results obtained in case of tensile tests and open-hole tensile tests (as the stacking sequence is the same), the stress concentrator reduced the mechanical strength of the composite with approximative 26% in case of new epoxy composite while in case of the old epoxy composites the stress concentrator reduced the mechanical strength by approximative 56%.

If the average results in terms of mechanical strength in case of all kind of tests performed are very clear, the mechanical strength of old epoxy composites is lower compared with the results of new epoxy composites, in case of elastic material properties a univocal deduction cannot be drown. As it was observed in the study made for resin-based composites [24], the decrease in the material's strength could be affected by the efficiency of the curing process.

The TGA was performed in order to determine if there are differences between the two-composite materials from physical phenomena point of view. In Figure 9 are presented two TGA thermograms, for the new epoxy composite, respective old epoxy composite.



(a)



(b)

Figure 9. TGA thermograms for: a) new epoxy composite; b) old epoxy composite

According to the TGA curve of the new polymeric composite the plateau section where no mass changes is approximately until 225°C, afterwards the decomposition of material happens and the mass is reduced. At the end of the analysis, at 500°C, a 27.225% mass change was determined in the sample. According to the TG curve of the old polymeric composite the plateau section where no mass changes is approximately until 180°C, afterwards the decomposition of material happens and the mass is reduced. At the end of the analysis, at 500°C, a 31.177% mass change was determined in the sample. Both DTA curves obtained in case of the two types of composite show endothermic and exothermic transformations showing the dehydration and decomposition of the epoxy matrix.

4.Conclusions

The paper presents a study conducted on GFRP manufactured using the same type of glass fibre fabric and two epoxy systems on different shelf life stages, a new system and an out of shelf life system. The influence of shelf life of the epoxy glass fibre reinforced composites was assessed based on three types of mechanical tests performed and on a thermogravimetric analysis. Based on test results it was concluded that:

- the new epoxy composite had an average tensile strength of 233 MPa, higher than the average value of 177 MPa determined in case of old epoxy composite which experienced a 24% strength reduction;
- the new epoxy composite had an average in-plane shear strength of 144 MPa, higher than 103 MPa registered for the old epoxy composite which experienced a 28% strength reduction;
- the new epoxy composite had an average open-hole strength of 173 MPa, higher than 77.83 MPa registered for the old epoxy composite which experienced a 55% strength reduction.

An overall reduction of mechanical strength and elastic properties of the composite materials was observed, primarily due to polymeric matrix degradation, which after long periods it could be prone to brittleness and susceptible to delamination and fracture. The TGA showed that changes in the epoxy matrix are more rapid in case of the old system. A future study should be conducted in order to determine if the cause of the mechanical properties reduction and the acceleration of reactions within the out of shelf life polymeric matrix are caused by the out of shelf life hardener used.

Even if the mechanical properties of the epoxy resin out of its shelf life were lower than the properties of the new epoxy system, it still can be used to manufacture UAV system components that aren't subjected to high loads (i.e. launch ramp).

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31.***ASTM D3518/D3518M - Standard Test Method for In-Plane Shear Response of Polymer Matrix Composite Materials by Tensile Test of a ± 45° Laminate
32.***ASTM D5766/D 5766M - Standard Test Method for Open-Hole Tensile Strength of Polymer Matrix Composite Laminate

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