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INFLUENȚA MONTMORILLONITULUI ASUPRA PROPRIETĂȚILOR MECANICE ALE COMPOZITELOR LAMINATE POLIAMIDĂ 6/FIBRĂ DE CABON INFLUENCE OF MONTMORILLONITE ADDITION ON POLYAMIDE 6/CARBON FIBER LAMINATED COMPOSITES MECHANICAL PROPERTIES

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In aeronautics applications, there is an increasing interest to replace the use of thermoset matrix in fiber reinforced composites with thermoplastic matrix. This paper presents the obtaining of hybrid carbon fabric reinforced laminated composites based on montmorillonite nanofilled polyamide matrix, using an innovative and cost efficient method involving polymer solvent dissolution, montmorillonite dispersion in different weight contents (1, 2, 4%) relative to the polymer, fabric impregnation with the obtained solution and high temperature pressing. The new laminated nanocomposites were characterized by FTIR spectroscopy to establish nanofiller/polymer chemical interaction, and they were tested in terms of mechanical and thermo-mechanical properties, the fracture cross section being analyzed by SEM and optical microscopy. The hybrid nanocomposites exhibited high mechanical performance, comparable to extensively used carbon fiber reinforced epoxy composites, 4% montmorillonite content generating significant enhancements of both strength and stiffness, as well as heat deflection temperature, results due to both the mechanically improved matrix due to uniformly dispersed montmorillonite, as well as a strong fiber/matrix interface due to solvent impregnation processing.

În aplicațiile din industria aeronautică, există o tendință tot mai accentuată de a înlocui matricile termoreactive utilizate în compozitele ranforsate cu fibre, cu matricile termoplastice. Această lucrare prezintă obținerea unor compozite hibride ranforsate cu țesătură din fibră de carbon, prin intermediul unui procedeu inovator, care presupune costuri medii, care implică dizolvarea polimerului într-un solvent și dispersarea montmorillonitului în diverse concentrații masice (1, 2, 4%) față de polimer, impregnarea țesăturii cu soluția obținută și presarea la temperaturi ridicate. Noile laminate cu matrice nanocompozită au fost caracterizate prin spectroscopie FTIR pentru a stabili interacțiunile polimer/nanofiller și au fost testate din punct de vedere mecanic și termo-mecanic, secțiunea de rupere fiind analizată cu ajutorul microscopiei electronice de baleiaj și microscopiei optice. Nanocompozitele hibride au prezentat performanțe mecanice ridicate, comparabile cu cele prezentate de intens utilizatele compozite cu matrice epoxidică, procente de 4% montmorillonit generând creșteri semnificative ale rezistenței și rigidității, dar și ale temperaturii de deflecție, rezultate datorate atât matricii îmbunătățită din punct de vedere mecanic datorită montmorillonitului dispersat uniform, dar și interfeței puternice între fibre și matrice, formată prin procesul impregnării cu solvent.

Keywords: carbon fiber fabric, montmorillonite, laminated composites, tensile strength, young's modulus

1. Introduction

In the last decades, the use of composite structures in aeronautics and automotive applications has increased tremendously, leading to important mass reduction and fuel consumption reduction [1, 2]. Today, the most used composites in aeronautics industry are CFRP (carbon fiber reinforced plastics), based on unidirectional or bidirectional carbon fiber and epoxy resins, as they exhibit high specific strength and stiffness and low density. The integrity of the composite as a whole

depends upon the effectiveness of load transfer within the composite, that is directly influenced by the interface between the matrix and the fibers [3, 4]. One of the major issues of fiber reinforced composites is represented by the weak interface between the phases that can lead to degradation of the final composite properties and premature failure. This is the reason why studies focus on property enhancement through interface improvements, simultaneously with matrix performance improvements. One promising method is represented by inorganic nanoparticles

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embedding into the organic matrix of the fiber based composites. Nanometric fillers such as layered silicates, carbon nanotubes, nanofibers or metallic nanoparticles [5] exhibit high specific areas, ensuring extensive interface with the matrix, that leads to large contact areas, the nanofiller/matrix interaction supporting a better reinforcing of the fibers. Layered silicates of montmorillonite type are one of the most used inorganic nanofillers for polymeric composites, due to their major advantages such as high cationic exchange capacity, excellent swelling ability and the easy routes that they can be subjected to chemical modification for enhanced compatibility [6, 7].

If in the case of epoxy composites, literature presents various studies involving montmorillonite addition in the matrix for improvement of mechanical, thermal or tribological properties of laminates based on carbon, glass or Kevlar fibers [8-12], in the case of polyamide composites reinforced with both fabrics and nanofillers, the experimental information is very limited, due to the difference in processing methods. The few existent studies presenting ternary composites, use the fibers in form of short fiber, Wu et al. [13] developed composites using montmorillonite (3%)/PA6 commercial pellets and short carbon and glass fibers using extrusion processing method, materials that presented up to 40% bending strength and 120% modulus improvements when using montmorillonite/PA6 pellets and 10% by weight short carbon fibers. There are few literature studies presenting carbon fiber fabric composites with polyamide 6 matrix [14], most of them using long [15] and continuous carbon fibers [16-20].

The paper presents the development of novel hybrid fabric reinforced composites based on montmorillonite nanoclay filled polyamide 6 matrix. The processing technology consists of a combined method involving nanocomposite matrix obtaining via solvent homogenization, fabric solvent impregnation and high temperature pressing following a trial established thermal program. The obtained materials were evaluated in terms of density, fiber/matrix ratio, characterized using FTIR spectroscopy and microscopy and SEM analysis and tested in terms of thermal stability under load and tensile and flexural performance, and the fracture region was analyzed using optical microscopy. The results highlight the positive effect of montmorillonite addition on the mechanical and thermo-mechanical properties of polyamide 6/carbon fiber fabric laminated composites.

2. Experimental procedure

The matrix of the laminates consisted of polyamide 6 (PA6) pellets supplied by SC ICEFS Săvinești. The nanofiller used was montmorillonite layered silicate modified with intercalating agent,

Nanomerl.34TCN, purchased from Sigma Aldrich, with the following characteristics: methyl, bis hydroxyethyl octadecylammonium modifier, added in 30-32% by weight content, 1.9 g/cm^3 density and 18-22 Å interlayer spacing. The reinforcing agent was carbon fiber fabric twill weave (FC), produced by ChemieCraft, France, 3K warp, with 193 g/m^2 areal weight and 1.7 g/cm^3 fiber density. The solvent used to dissolve polyamide was formic acid 85% (FA) analytical grade purchased from Precisa SA.

The laminated composites achievement was a multiple stage process that involved nanocomposite processing, fabric solvent impregnation followed by high temperature pressing. The dried polymer pellets were dissolved in formic acid 85% at 10% PA6 in FA weight ratio, under mechanical stirring for 4 hours. The dried montmorillonite (TCN) nanopowder was added in 3 weight contents (1, 2, 4 wt.%) relative to PA6, the mechanical stirring continued for 2 more hours and then the solution was sonicated using Bandelin Sonopuls probe for 15 minutes. Each ply of carbon fiber fabric was impregnated with the obtained solution and stacked up in groups of 11 layers. The solvent was removed at room temperature for 48 h and at 80- 100°C for 8 h. Each laminated composite was pressed using CARVER hot platens press, following an established temperature program, starting with a linear temperature increase from 25-230°C followed by 3-5 min dwell periods between 230-250°C and cooling that took place under pressure down to room temperature. Figure 1 illustrates the thermal pressing program.

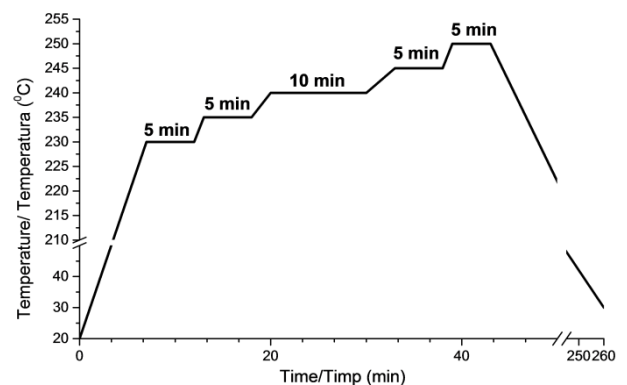


Fig. 1 – Thermal pressing program illustrating the temperature variation in time/ Programul de presare ilustrând variația temperaturii în timp.

In this study the following nomenclature for the hybrid laminated nanocomposites reinforced with 11 layers of carbon fiber fabric will be used: PA6/11FC for the control sample with no nanofiller and PA6+1%TCN/11FC, PA6+2%TCN/11FC, PA6+4%TCN/11FC where 1, 2 and 4 represent the montmorillonite weight content in the matrix.

The laminated composites nanofilled matrix

was subjected to FTIR spectroscopy using iN10 MX Mid Infrared FT-IR Microscope. The hybrid nanocomposite laminates were subjected to mechanical tests at room temperature (INSTRON 5982). Tensile tests were performed according to SR EN ISO 527 [21], using 5 mm/min tensile rate on dumbbell specimens. 3-point bending (flexural) tests were performed according to SR EN ISO 178/SR EN ISO 14125 [22], using a 2 mm/min speed of test on rectangular specimens, at conventional deflection (1.5 x specimen thickness) and nominal span length (16 x specimen thickness).

The components distribution in the laminated composites cross section was monitored by FTIR microscopy, using iN10 MX Mid Infrared FT-IR Microscope. The fracture cross section and the laminates surface was analyzed using scanning electron microscopy- SEM using QUANTA INSPECT F microscope with field emission gun and 1.2 nm resolution and the fracture mode was evaluated by optical microscopy using Meiji 8520 microscope at 40x magnification.

The materials thermal stability under load was also evaluated by heat deflection temperature testing, using Qualitest HDT1 Tester, according to SR EN ISO 75 [23]. Rectangular specimens were subjected to 3-point bending test at 1.8 MPa flexural stress, in a siliconic oil medium, at 2 °C/min heating rate, until standard deflection depending on sample thickness.

The component content and density of the hybrid laminates were calculated using mathematical formulas taking into consideration the laminate thickness.

3. Results and discussion

3.1. Density and fiber/matrix ratio calculation

The fiber volumetric and weight contents were calculated taking into consideration the average thickness of the sample [24]:

$$V_f = \frac{n \cdot m_{0f}}{e \cdot \rho_f} \quad (1)$$

$$W_f = \frac{V_f \cdot \rho_f}{V_f \cdot \rho_f + V_m \cdot \rho_m} \quad (2)$$

m_{0f} - fabric areal weight (kg/m²), n- number of carbon fabric plies in the laminate, e- thickness of

the final laminate, V_f - fiber volumetric content, V_m – matrix volumetric content, $V_m = 1 - V_f$, W_f - fiber weight content.

The hybrid laminated nanocomposites density values were calculated using the equation:

$$\rho = \rho_f V_f + \rho_m V_m \quad (3)$$

ρ_f - fiber density, ρ_m - matrix density (taking into consideration the montmorillonite content)

Matrix- fiber optimum load transfer is achieved when fiber/matrix ratio is the correct range, range that differs from one combination of fiber/matrix to another. The calculated average fiber/matrix volumetric ratio is 70/30, while weight ratio is 80/20 at an average thickness of 1.7 mm. Regarding the calculated density, the value is insignificantly changed when adding higher nanofiller contents, and the obtained laminates density is approximately 1.57 g /cm³, value that designates them as lightweight materials.

3.2. FTIR spectroscopy

FTIR spectroscopy analyses were performed on the nanofilled matrix of the laminates in order to evaluate the chemical interactions of the montmorillonite with polyamide matrix. The analyzed area was polished before the analysis was performed. Figure 2 presents the spectra of Nanomer134TCN montmorillonite/PA6 and PA6 control sample. No supplementary peak appears proving that formic acid did not alter the chemical structure of the polymer. All spectra show the characteristic peaks of polyamide 6 (stretching of C=O bond at 1640 cm⁻¹ and deformation vibration of N-H group- amide II at 1550 cm⁻¹) [25]. In the case of the nanocomposite samples, with montmorillonite content increase, the intensity of these bands decreases, simultaneously with intensity increase of the peaks in the region 900-1100 cm⁻¹. The peak at approx. 1050 cm⁻¹ is due to Si-O from montmorillonite out-of-plane stretching vibrations, while, the new peak at 920 cm⁻¹ is owned to the bending vibration of Al-OH-Al from montmorillonite structure [26, 27]. The peak intensity increase between 2850- 2960 cm⁻¹ is owned to the bending vibration of CH₃ from quaternary ammonium salt intercalating agent. This observations could suggest a strong interaction between the polymer groups and hydroxyl groups of montmorillonite intercalating agent [28].

Table 1

Hybrid laminated nanocomposites characteristics/ *Caracteristicile nanocompozitelor laminate*

Sample/ <i>Probă</i>	Thickness/ <i>Grosime</i> , cm	V_f , %	W_f , %	Density/ <i>Densitate</i> , g/cm ³
PA6/11FC	0.169	72	80	1.58
PA6+1%TCN/11FC	0.169	71	80	1.58
PA6+2%TCN/11FC	0.172	70	79	1.57
PA6+4%TCN/11FC	0.174	69	78	1.57

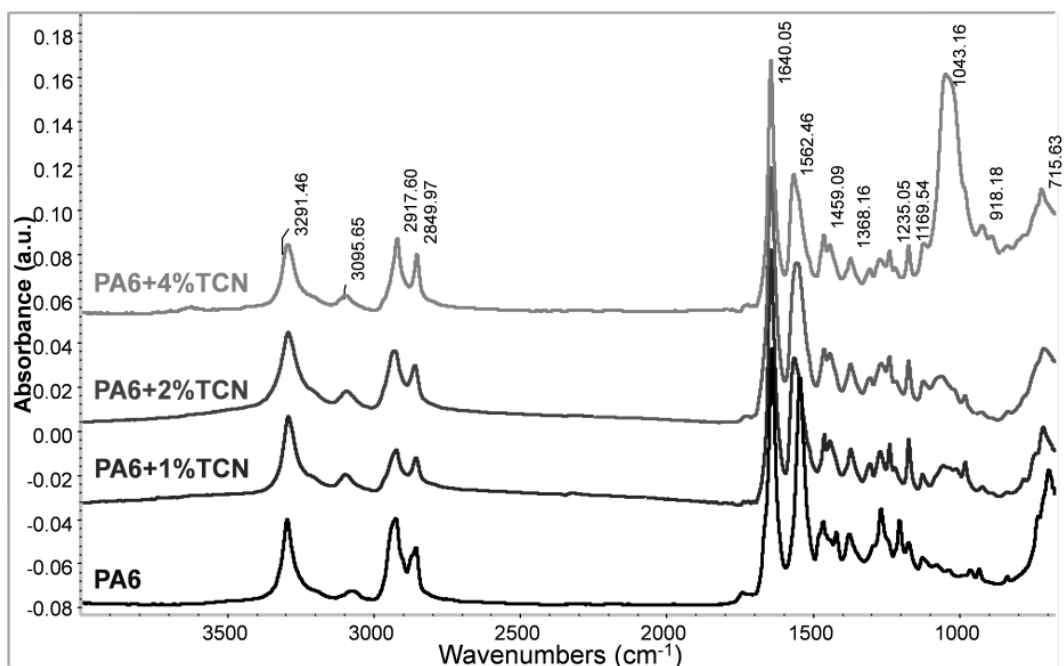


Fig. 2 – FTIR spectra of PA6 control sample and 1, 2 and 4% Nanomerl.34TCN based PA6 samples/ Spectrele FTIR ale probei martor PA6 și ale probelor de PA6 având la bază 1, 2 și 4% Nanomerl.34TCN.

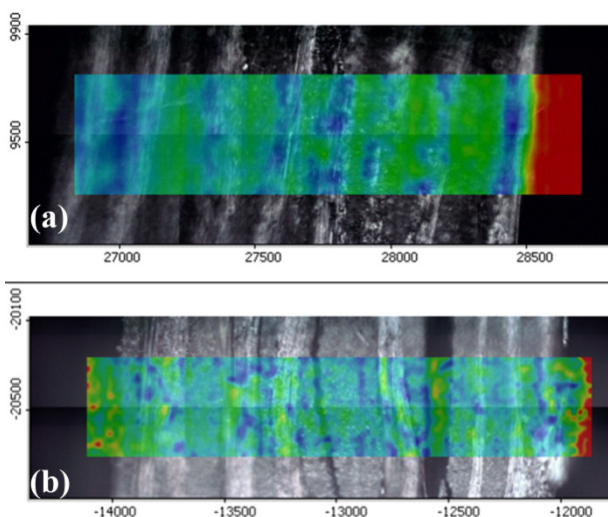


Fig. 3 – 2D image overlapped on video image of (a) PA6+2%TCN/11FC and (b) PA6+4%TCN/11FC/ Imaginea 2D suprapusă peste imaginea video a probei (a) PA6+2%TCN/11FC și (b) PA6+4%TCN/11FC.

3.3. FTIR microscopy

FTIR microscopy highlights the presence of a compound in a sample by monitoring by its characteristic absorbance peak. The monitoring was performed at 1650 cm^{-1} (Figure 3a), one of the highest characteristic peaks in polyamide and 1050 cm^{-1} (Figure 3b) corresponding to the montmorillonite.

It can be seen that polyamide appears in regions with maximum absorbance at 1650 cm^{-1} , while the fabric appears in regions with lower absorbance, but the areas are not strictly delimited, showing that the polyamide penetrated through the fibers of the fabric. Monitoring by

montmorillonite characteristic peak, the image (Figure 3b) highlights a possible uniform distribution of the nanofiller in the matrix, that it is also uniformly distributed in the laminated composite analyzed area. FTIR microscopy offers important information regarding the nanofilled matrix distribution in the overall cross-section of the laminated composites. In order to perform an analysis of the interface between the components, SEM images offer higher magnification level.

3.4. Scanning electron microscopy

SEM analyses of the laminates fracture cross-section and surface offer important information regarding both nanofilled polyamide/fiber interface and montmorillonite distribution.

SEM images of the fracture cross-section (Figure 4a, b) illustrate the good interface between the polyamide matrix and the fibers. The simple and nanofilled polymer embedded the fibers that constitute the fabric, creating a thin polymer layer around the fibers. Figure 4b shows how the montmorillonite nanofilled polyamide layer remains attached on the fiber surface even after tensile fracture, sustaining the idea of a solid matrix and matrix/fiber interface aided by the montmorillonite presence.

This is very important for mechanical performance. Fracture cross-section images prove that the nanofilled polymer covers the fibers all around, while surface images prove that it covers the entire fabric ply, ensuring an extended contact area that allows an extremely efficient mechanical load transfer within the composite.

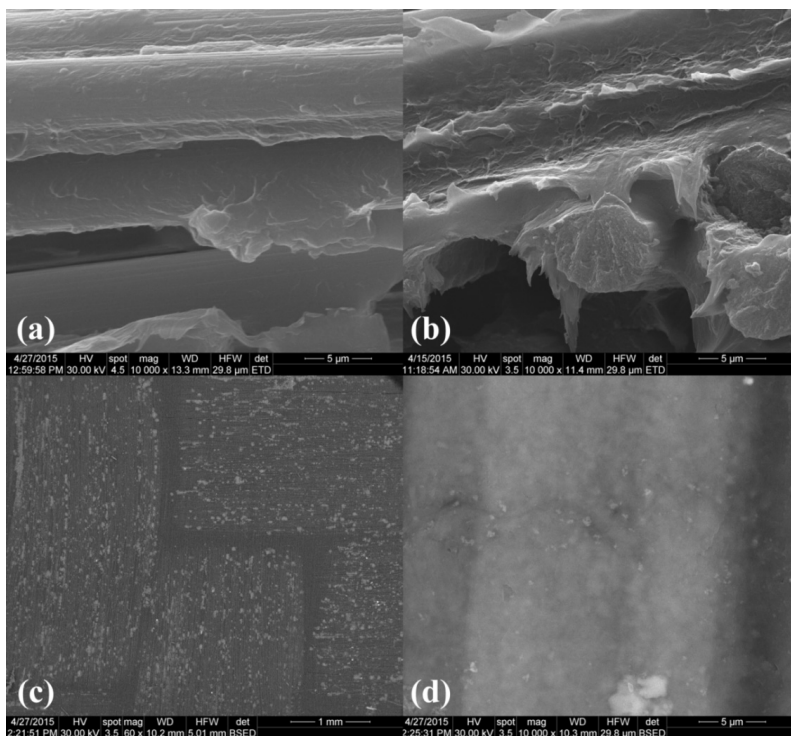


Fig. 4 - SEM images of (a) PA6/11FC-fracture cross-section, (b) PA6+4%TCN/11FC fracture cross-section at low magnification, (c) PA6+4%TCN/11FC surface at high magnification/ Imaginile SEM ale probelor: (a) PA6/11FC-secțiunea de rupere, (b) PA6+4%TCN/11FC- secțiunea de rupere la mărimi mici, (c) PA6+4%TCN/11FC- secțiunea de rupere la mărimi mari.

3.5. Thermal stability under load

Thermal stability under load is illustrated by the heat deflection temperature value (HDT). The average values presented in Table 2 show that montmorillonite addition has positive effects on the materials HDT. 1 and 2% montmorillonite samples present an increase by approximately 10°C, while a significant increase of HDT value is illustrated by the nanofilled PA6/carbon fiber laminates with 4% montmorillonite contents, that is with approximately 30°C higher compared to the control sample.

Table 2

Heat deflection temperature values for the laminated nanocomposites/ Valorile temperaturii de deflecție ale nanocompozitelor laminate

Sample/ Proba	HDT (°C)
PA6/11FC	200.3
PA6+1%TCN/11FC	210.0
PA6+2%TCN/11FC	208.2
PA6+4%TCN/11FC	230.0

3.6. Mechanical Testing

The mechanical testing consisted of tensile and 3-point bending (flexural) tests, that focused

on the modification of strength and Young's modulus of elasticity, in order to compare the nanofilled laminates with the control sample. Table 3 presents the properties average values obtained for each sample, illustrating the overall improved mechanical performance of the nanofilled laminates in both tensile and 3-point bending testing.

It can be noticed that the nanofilled laminated composites mechanical properties in both tensile and 3-point bending increase with montmorillonite content increase in the matrix. Adding 1 and 2% Nanomer I.34TCN in the PA6 matrix generated an increase of tensile strength by 16% and 18% respectively, while flexural strength increased by 11% and 21% respectively compared to the control sample.

The 4% montmorillonite based laminates illustrated the highest increments, showing a 40% increase of tensile and flexural strength compared with the unmodified matrix laminates.

Table 3

Mechanical properties of NanomerI34TCN/PA6/carbon fiber fabric hybrid composites
Proprietățile mecanice ale compozitelor hibride NanomerI34TCN/PA6/țesătura fibră de carbon

Sample Proba	Tensile strength/ Rezistența la tracțiune, MPa	Young's Modulus/ Modulul Young, GPa	Flexural Strength/ Rezistența la încovoiere, MPa	Young's flexural modulus/ Modulul de elasticitate la încovoiere, GPa
PA6/11FC	366.1	39.3	402.4	40.1
PA6+1%TCN/11FC	422.9	60.0	445.5	52.7
PA6+2%TCN/11FC	432.2	62.7	485.2	53.5
PA6+4%TCN/11FC	530.0	66.0	556.2	57.2

In terms of elasticity modulus, the increments were even higher, the samples based on 1, 2 and 4% respectively showed a 50, 60 and 70% increase of Young's modulus. Likewise, Young's flexural modulus increased with montmorillonite content increase, compared with the control sample, the 1, 2 and 4% TCN based samples presented 21, 34 and 43 % higher values.

Therefore, Nanomer134TCN montmorillonite addition into the PA6 matrix via solvent homogenization helps improving both strength and stiffness of the PA6/carbon fiber fabric laminated composites. 4% content improves PA6 mechanical performance, so that the matrix is able to support higher mechanical loads, that consequently allows that lower load amounts are sustained by the fibers. This, together with the strong and extended interface between the nanofilled matrix embedding the fibers of the fabric, illustrated by SEM, allow an optimum load transfer between the phases of the hybrid composite, that contributes to the enhancement of tensile and flexural strength and stiffness. Mechanical tests results were explained also with the aim of optical microscopy analyses.

3.7. Fractography

The fracture region of representative specimens of each sample was visualized using optical microscopy, to identify the main fracture mechanisms that led to tensile and flexural failure.

Figure 5 illustrates tensile tested specimens. It can be noticed that the control sample presents as main failure mechanism: crack propagation that led to delamination. Matrix micro-cracking is also noticed, showing that the matrix lower mechanical strength probably lead to inefficient load transfer between matrix and fibers and consequently lower mechanical strength and

stiffness of the composite. In the case of montmorillonite based samples with different contents, the matrix micro-cracking phenomenon is not noticed and fiber break is the main fracture mechanism that led to laminates failure. It can be noticed that the crack propagation phenomenon appears in the lowest extent in the PA6+4%TCN/11FC sample. This confirms the higher mechanical strength and stiffness in tensile ensured by the higher strength nanofilled matrix that was able to withstand higher mechanical loads. This fact along with a proper interface sustained an efficient load transfer from the fibers, so that the mechanical load that the fibers had to support were lower, leading to higher mechanical performance of the composites.

Flexural testing was conducted until conventional deflection meaning that the load was applied until the specimen bended to a value of deflection equal to 16 of the specimen thickness. During flexural test, the top layer suffers compression stresses, while the bottom layer suffers tensile stresses. Some of the specimens of the nanofilled samples did not break until this value, but others presented some cracks after testing on 3-point bending. Figure 6 shows that the flexural load generated interlaminar cracks in the medium region of the specimens of the control sample and PA6+2%TCN/11FC, while external layer only suffered minor cracking due to compression/ tensile stress under bending. One possible reason for this could be the presence of stress concentration sites that influence both material behavior as well as crack propagation. In the case of PA6+4%TCN/11FC samples, the illustrated specimen presented crack propagation between marginal layers, while the inner region was not affected.

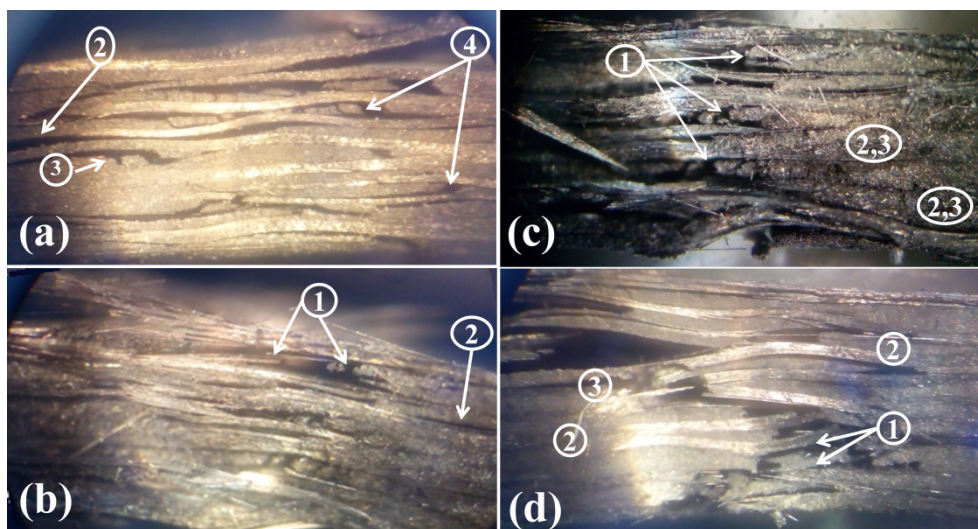


Fig. 5- Optical images of the fracture regions of tensile tested specimens of (a) PA6/11FC, (b) PA6+1%TCN/11FC, (c) PA6+2%TCN/11FC, (d) PA6+4%TCN/11FC and the identified mechanisms: (1) fiber pull-out, (2) crack propagation, (3) delamination, (4) matrix micro-cracking/ *Imaginile de microscopie optică ale regiunilor de rupere ale epruvetelor testate la tracțiune: (a) PA6/11FC, (b) PA6+1%TCN/11FC, (c) PA6+2%TCN/11FC, (d) PA6+4%TCN/11FC și mecanismele de rupere identificate: (1) smulgerea fibrelor, (2) propagarea fisurilor, (3) delaminare, (4) microfisurarea matricii.*

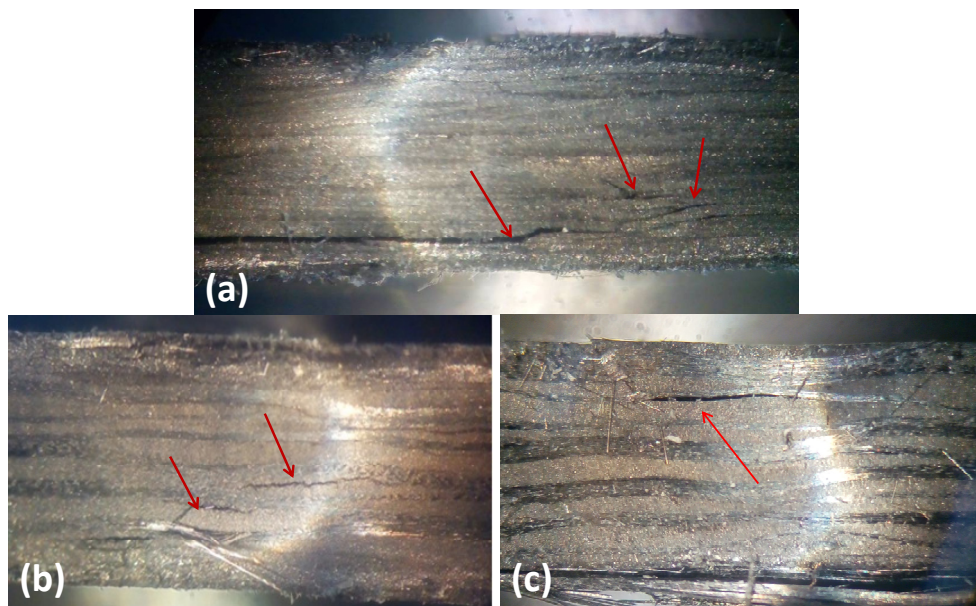


Fig. 6 – Optical microscopy images of the fracture regions of flexural tested specimens of (a) PA6/11FC, (b) PA6+2%TCN/11FC, (c) PA6+4%TCN/11FC / Imaginile de microscopie optică ale zonelor de rupere ale epruvetelor testate la încovoiere: (a) PA6/11FC, (b) PA6+2%TCN/11FC, (c) PA6+4%TCN/11FC.

4. Conclusions

The study presents the development of novel hybrid carbon fabric reinforced composites based on Nanomer134TCN nanoclay filled polyamide 6 matrix. The technology consisting of nanocomposite matrix obtaining via solvent homogenization, fabric solvent impregnation, high temperature pressing following a trial established thermal program has some major advantages. The solvent method enables nanoclay homogenization into the PA6 matrix via ultrasonication, while fabric solvent impregnation leads to extremely high contact areas between matrix and fibers that compose the fabric and thermal pressing stage is a rapid and cost effective process. All these contribute to the obtaining of final hybrid composites with low weight and high mechanical properties that can exhibit 40% higher strength and 40-70% higher rigidity at 4% montmorillonite loadings compared with control sample composites. The mechanical properties of these materials are comparable with standard carbon fiber epoxy composites, used in aeronautic industry, but they present the advantages of lower density, recyclability and stronger mechanical interface ensured by the solvent impregnation and stronger matrix ensured by montmorillonite presence.

Acknowledgements

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MANIFESTĂRI ȘTIINȚIFICE / SCIENTIFIC EVENTS

The seventeenth European Conference for Composite Materials (ECCM17) , Munich (Germany) from June 26th to 30th, 2016.

The conference is organised by the Institute for Carbon Composites of the Technische Universität München (TUM) and the Leading-Edge Cluster MAI Carbon, under the patronage of the European Society for Composite Materials (ESCM). TUM is one of Europe's top universities. It is committed to excellence in research and teaching, interdisciplinary education and the active promotion of promising young scientists. MAI Carbon is funded by the Federal Ministry of Education and Research and consists of companies, training and research facilities, as well as supporting organisations, which are active within the technological field of carbon-fiber reinforced plastics in the region of Munich-Augsburg-Ingolstadt (MAI).

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