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THE MICRO-FRACTO-GRAPHIC ANALYZE OF EXTRUDED PRODUCTS FROM POLYAMIDE 6.6 REINFORCED WITH GLASS FIBERS

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Abstract: *In this paper there are presented the results of the micro-fracto-graphic analyse of the samples from the products obtained after extrusion of polyamide 6.6 (PA 6.6) reinforced with glass fibres. Three different degree of reinforcement are used, respectively 10, 20, 30% of glass fibbers. The micro-fracto-graphic analyse of SEM pictures (scanning electron microscope) is performed and the interdependence between the fibres orientation and the processing temperature during extrusion is presented.*
Key words*: extrusion, polyamide 6.6, glass fibber, reinforcement degree, micro-fracto-graphic analyse.*

1. INTRODUCTION

The materials used for the experimental research are from the class of reinforced polymers, respectively polyamides. It was chosen this type of polymer considering the wide range of currently available products on the market and the necessity of increasing their mechanical characteristics.

Polyamides are a class of materials recommended mostly for injection but not for products with infinite length when the extrusion technology is more appropriate.

In the present research, the authors want to determine if the degree of reinforcement of the polymer has any influence upon the material's structure. With this intention, a controlled mixture of polyamide 6.6 with different ratio of reinforcement fibres was done. Glass fibres used for reinforcement are from the short fibres category. Their dimensions are: diameter between 8 and 14 μm and the length between 1-3 mm [1][4][5]. As a result, three types of materials: PA 6.6 reinforced with 10, 20 respectively 30% fibber glass (PA 6.6 – 10, 20, 30% FS) were obtained [3].

The authors want to determine if the reinforcement fibres have the same arrangement during the extrusion process, no matter which the quantity of glass fibber is and to study the mechanical characteristics of extruded material.

For the experiments an extruder Cincinnati Monos +45 mode type was used. This equipment has a single worm gear with a diameter of 45 mm. The geometry of the extruder is a special one, from the last generation, with a bimetallic cylinder realised from special alloys resistant at the corrosion and abrasion. This type of equipment provides a very good homogeneity of the melted material because of the complexity of the worm gear's geometry and the very precise control of the temperatures for all four specific areas of extrusion process (feeding area, compressing area, mixing area and shaping area). With this extruder, tubes with diameters between 20 – 2000 mm and with different wall thickness can be obtained. For this research a tube with exterior diameter of 30 mm and a wall thickness of 4 mm was realised. The processing temperature of the extruder can be set up until reach out the maximum value of 400°C for each area separately [2].

The samples for tensile test and for micro-fracto-graphic analyze were cut, with a special mill, from each tube corresponding to each type of material.

The microscopic analyses were made with an electronic microscope realized by the TESCAN company, VEGA 3SB model. The VEGA 3SB microscope is a compact model from the last generation of scavenging microscopes, being provided with a heat up electronic cannon with tungsten filament. It also has an analytic camera with step-up motorization and also modern optical electrons equipment [2].

2. RESULTS OF THE MICRO-FRACTO-GRAPHIC ANALYSE

In order to perform the experimental research, the working temperature into the feeding zone was set-up and maintained constant at 250 °C and in the compressing zone was maintained at 260 °C [3]. The other values of temperatures, in the homogenous area, respectively shaping area, were set-up according to the experimental planning strategy achieved with the aid of Response Surface Method (RSM), using the rang two compositional central programming [3]. The planning of the experimental strategy was possible by using Design Expert soft version 8.0.5. The worm gear revolution was set-up at a certain value, n = 31 rot/min, and the driving motor of the extruder functioned at a capacity of 70% and the speed of puller is equal with v = 11,19 m/min. All these values were kept constant during the extrusion of all the materials [2].

After the tensile tests were performed, according to the Response Surface Method strategy it was determined for all the materials that the maximum value of the tensile strength is obtained in the case when the value of the temperature in the homogenous area is set-up at 280°C and that from the profiling area is 270°C. Considering the results it can be said that these temperatures' values are the optimal working values. In the study there were considered only the samples that were taken from the breaking area of the products realized with these parameters [3].

For a more accurate interpretation of micro-fracto-graphic images, the polymeric matrix, in our case polyamide 6.6 and the reinforcement fibber, both in a pure state (without impurities), were microscopically analyzed at a scale of 100 microns (μm), (figures 1 and 2). In figure 1 and 2 can be observed the pure polymer and glass fibber, before they were mixed together and the composite was realized.

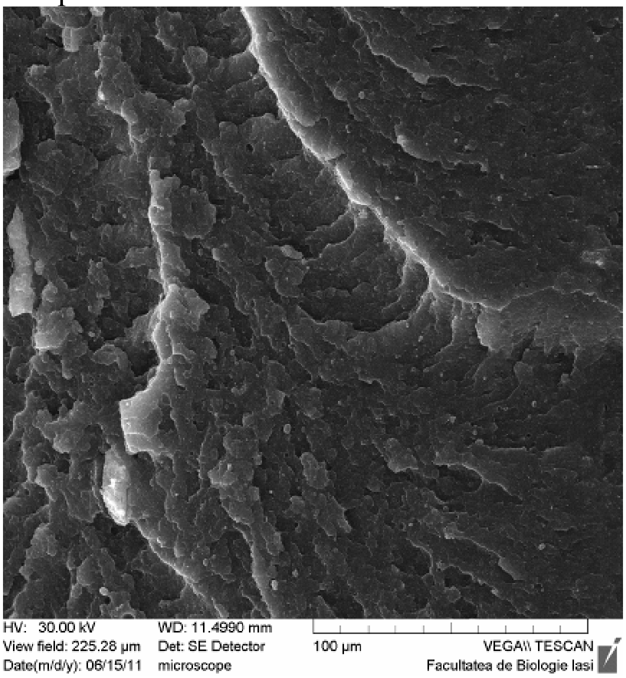


Fig.1. The micro-fracto-graphic of *PA 6.6 mark – nature state* (non-reinforced)

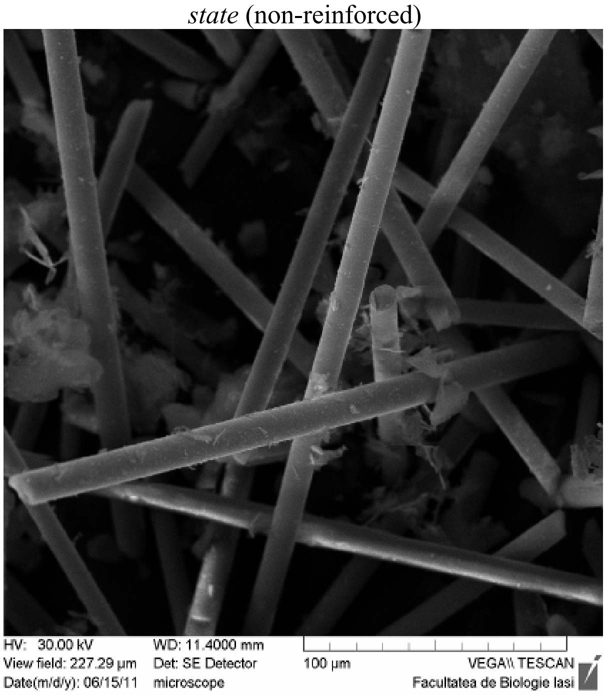
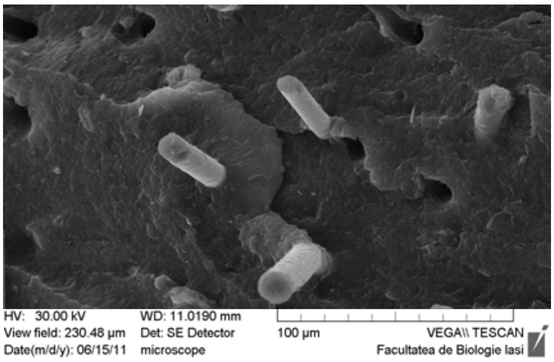


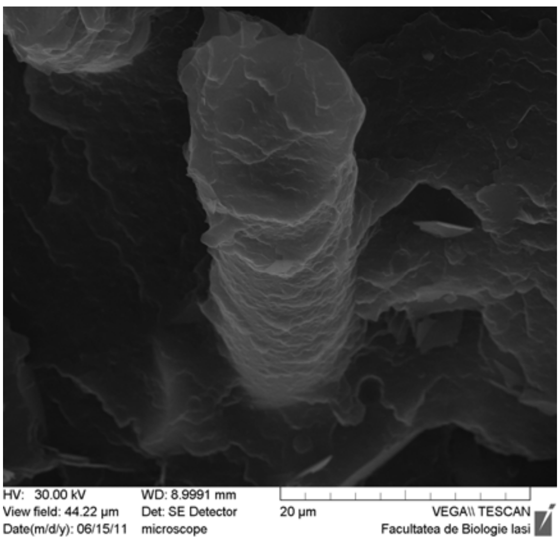
Fig.2. Micro-fracto-graph mark of pure glass fibber

In figure 3 a) and b), there are presented macro-fracto-graph images recorded in the case of extruded product from PA 6.6 – 10% FS.

The macro-fracto-graph analyses of the images was made on a scale of 100 µm (fig. 3a), respectively 20 µm (fig. 3b). The glass fibres from the breaking out area of the samples reveal a uniform arrangement on the polymeric matrix. Since the cutting of the samples was realized on the flow direction, and the breakage resulted on a perpendicular direction, it can be observed the fact that the glass fibbers are uniformly longitudinal disposed, on the flow direction of the polymeric melt. The cavities that appear in the figure are a result of the fibres wresting in the moment of the breakage of the sample during the tensile test. In figure 3b it can be seen a perfect incorporation of the glass fibber into the polymeric matrix.



a) 100 µm scale

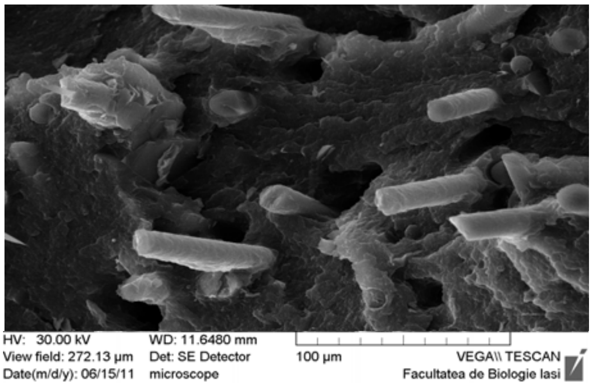


b) 20 µm scale

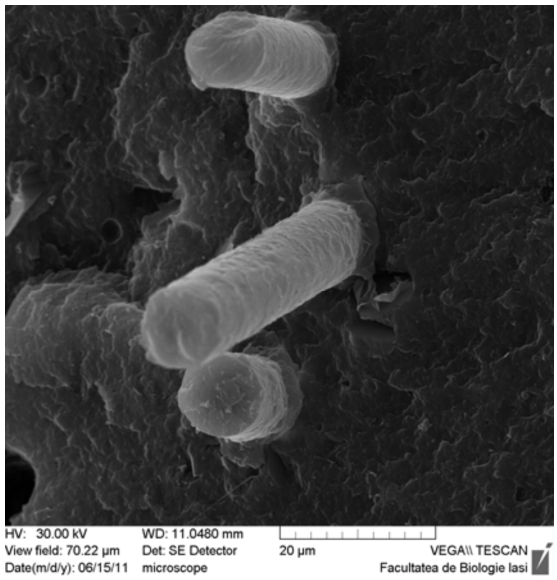
Fig.3. Micro-fracto-graph of extruded product
PA 6.6 – 10 % FS

Analyzing the images from figures 4 a) and b), it can be observed a uniform distribution of the glass fibbers on the breakage section. The micro-fracto-graph interpretations are made at the same scale as in the case presented before (100 respectively 20 µm). It can be also relieved in this case that the appearing of the gaps occurred after pulling out of the fibres after stretching.

Similar with the case of PA 6.6 – 10% material presented above, it can be observed in the image b) from figure 4 the glass fibber's orientation along the longitudinal axis (in the flow direction of the polymeric melt). A perfect incorporation of the glass fibbers into the polymeric matrix can be observed in this case too.



a) 100 µm scale



b) 20 µm scale

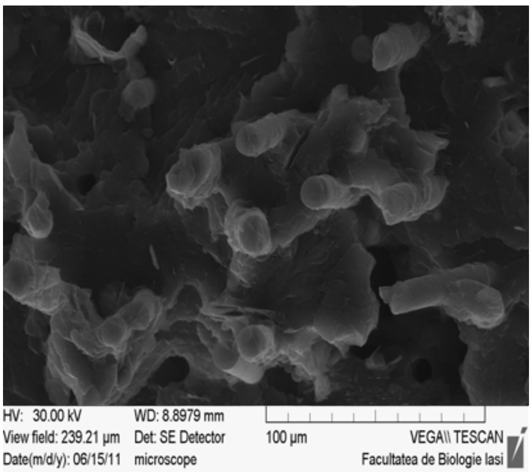
Fig.4. Micro-fracto-graph of extruded product
PA 6.6 – 20 % FS

In figures 5a, respectively 5b, there are presented the micro-fracto-graph images for the polymer's reinforcement with 30% glass fibber.

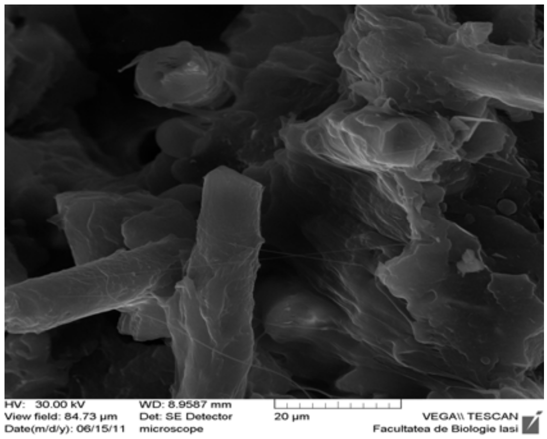
Interpretation of the analyzed images was made at the same scale as in the previous cases. It can be seen in figure 5b an evenly dispersion of the glass fibbers on the surface of the breakage section.

Comparative to the previous cases, after the micro-fracto-graph analyses corresponding with material PA 6.6 – 30% FS, it can be seen that a transversal position of the fibres against the flow direction of the polymer appears (fig. 5b). Considering these observations, one can say that increasing the reinforcement degree (in our case over 20% FS), the orientation of the fibres is no longer only in the direction of polymer's flowing.

From the study of the images from figure 5a and 5b, it can be seen that there are fewer (or even none) gaps, which usually occur after the breakage, and this observation leads to the conclusion that the fibres adherence at the polymeric matrix is increased. This phenomenon is due to the fact that the distance between the glass fibres is smaller, and because of the fibres capacity to storage, they receive heat from the cylinder of the extruder and release it to the polymeric matrix during the process. There is still a small amount of gaps in the breakage area, but the percentage is lower than in the case of materials PA 6.6 – 10 % FS and PA 6.6 – 20 % FS.



a) 100 µm scale



b) 20 µm scale

Fig.5. Micro-fracto-graph of extruded product *PA 6.6 – 30 % FS*

In the image from figure 6 it can be observed the appearance of an extrusion defect (air bubbles) because of the auxiliary times between two successive processes. Air inclusions (bubbles) relieved at a scale of 20 microns are due to the residual humidity from the extruded granular material. The polymer has the capacity to absorb water from the environment, even if before the processing the material is dried in a special oven at a controlled temperature.

During the extrusion, the glass fibbers dimensions change because the tensile phenomenon appears. This phenomenon occurs because of the permanent contact of the fibres with the metallic elements of the extruder (worm gear, cylinder, the worm gear spires etc.)

In the previous researches [3] it was presented that for a reinforcement that exceeds 30% concentration of fibbers in the polymeric matrix, the fabrication through extrusion is very expensive. In the same time the quality of resulted materials is more difficult to control and the equipments are very expensive because they need a proper fabrication to resist to the aggressive wear produced by the fibres.

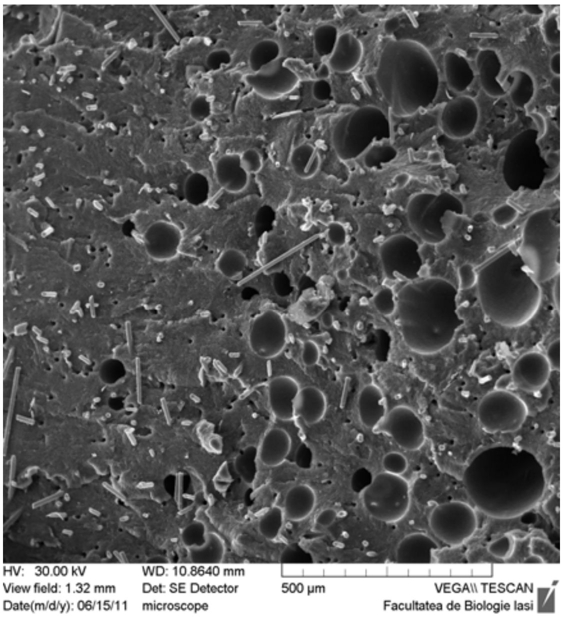


Fig.6. Defect after extrusion

3. CONCLUSIONS

In this paper it was find out that until a concentration of 20 % of glass fibbers in the polymeric matrix, their orientation is preponderant longitudinally, on the flow direction of the polymeric melt. In the case of reinforcement with 30% FS, the orientation of fibres, is not any more preponderant in the same direction with the polymer's flow, but there is a random orientation in most cases. So, it can be affirmed that with increasing of the reinforcement degree (in our case over 20 % FS), the fibres orientation is not only in the same direction with the polymer's flow but also in other directions.

Another remark is that with increasing of the reinforcement degree, a better adherence of the fibres to the polymeric matrix is observed. This is due to the fact that the glass fibres are able to store the heat received from the extruder's cylinder during processing by extrusion and to release it to the polymeric matrix, even after processing. By increasing the reinforcement degree, the distance between the glass fibres is smaller and the quantity of the storage heat released to the polymeric matrix is higher. Due to the increased adherence between fibres and the polymeric matrix, it can be also observed a substantial diminution of the number of gaps that occur after breakage during the tensile test. All these remarks attest the fact that with increasing of the reinforcement degree, but not over the established limit (maximum 30% glass fibbers), there are also increasing the properties and the quality of the extruded products.

4. REFERENCES

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ANALIZA MICROFRACTOGRAFICA A PRODUSELOR EXTRUDATE DIN POLIAMIDA 6.6 ARMATA CU FIBRE DE STICLA

Rezumat: În cadrul acestei lucrări sunt prezentate rezultatele analizei microfractografice a probelor prelevate din produsele obținute prin extrudarea poliamidei 6.6 (PA 6.6) armate cu fibre de sticlă. S-au folosit trei grade de armare diferite, respectiv 10, 20, 30 % fibre de sticlă. Analiza microfractografică a imaginilor SEM obținute, ne permite evidențierea interdependenței modului de aranjare al fibrelor de armare față de temperatura de prelucrare prin extrudare. Odată cu optimizarea temperaturilor de prelucrare prin extrudare, se observă faptul că va crește şi calitatea produselor extrudate.

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