STUDY OF MECHANICAL PROPERTIES OF SHORT FIBER GLASS REINFORCED PA 6.6 RECYCLED REPEATEDLY

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Abstract

Some of the parts manufactured with engineering polymers such as short fiberglass reinforced polyamide (PA) are rejected for failing quality controls or are removed due to their breakage in service. The recovery of this technical material would be a major economic and environmental savings. The aim of this work is to determine the degree of recycling or number of times that the material can be re-injected maintaining the mechanical properties suitable for the in-service conditions. Then a physic-mechanical characterization for ten successive recycles was performed. The results show that as the number of recycles increases the PA matrix reduces its viscosity. About the mechanical properties, in static conditions, the ductility, strength and toughness decreases linearly with the number of recycles. This data is repeated in dynamic conditions. Observations of the fracture surfaces by SEM show a great appearance of pores from recycling number 5.

1. Introduction

High Speed Spanish Railway sector uses short-fiber glass reinforced polyamide to inject insulating and mechanically resistant components for fasteners to connect the rails to the sleepers in the superstructure track, see Figure 1(a). One of these constituents is the flanged plate; see Figure 1(b). Some of these pieces are removed, due to either defective production or by breakage in service. The main function of these reinforced polymeric fasteners is to reduce the dynamic efforts that the rail transmits towards the concrete sleeper as a consequence of the impacts generated by the passing of trains and also to minimize the corresponding vibrations. They also prevent longitudinal displacement and rotation of the rails, maintaining the gage constant. Moreover, this system helps to isolate electrically the rails as they drive electrical signals to control the traffic on the line.

The recycling of this material for its later re-use would lead to a great saving, from both an environmental and an economic viewpoint, since the cost of raw materials is relatively high (around 3 Euros per kilogram). The service life of this polymeric material is estimated at ten years, depending on the environmental conditions and on the intensity of the railway traffic. In this respect, it is possible to estimate in 5.000 Euros per kilometer the saving that would be reached through the recovery and total reuse of the constituent material of the fastening plates. In Spain there are more than 3.000 km of high-speed tracks which in raw material terms savings could be interesting. Mechanical recycling is a simple and economic process that only requires the crushing of the material and its subsequent molding, without the need for any chemical treatments. However, any recycling is accompanied by degradation in the quality of the material which could limit the useful life or applicability under certain loading conditions. For this reason, it is necessary to perform a physical and mechanical characterization to verify whether fasteners injected with recycled material are suitable for industrial use.

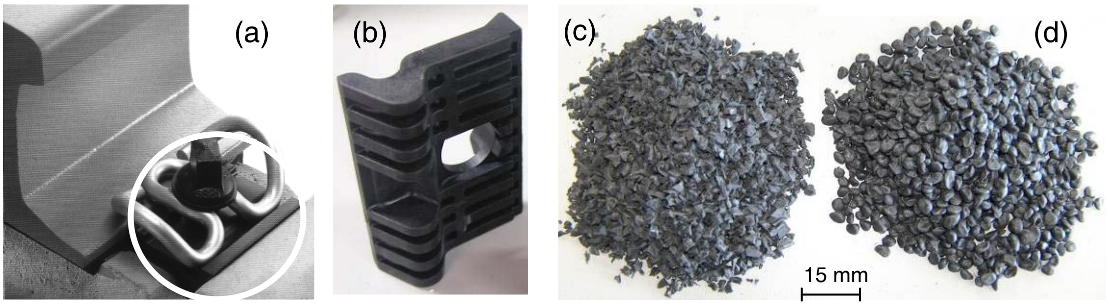

Figure 1. (a) Fastening system, (b) Insulating flanged plate A2. (c) Crushed plate and (d) Raw material

2. Studied material

The material used in the study comes from insulating fasteners for high speed railway tracks (A2) that have not yet been placed on the track. They were selected equally between different manufacturers and, therefore, the raw material also comes from different brands. A2 plates are injected with polyamide 6.6 reinforced with 35% by weight of short fiber glass. Prior to the moulding process, the A2 plates were cleaned and dried at 100ºC for 7 days to eliminate the water inside as the polymeric material is hygroscopic. Then the plates were ground in a mill hardened steel blades until a maximum particle size of 5-6 mm that it is comparable with raw material size as shown in Figure 1(c and d). The ground material was injected into standard tensile (type B) [2], Charpy impact [3] and fracture toughness [4] test specimens setting the following optimal injection parameters: injection molding machine clamping force (35 ton), injection temperature (300ºC), injection pressure (1800 bar) and back pressure (50/400/500 bar). The optimal injection parameters were determined by reference to the guidelines established in the current Standards [5, 6], injection recommendations published by the manufacturer of the raw material [7, 8], the specifications of the injection machine and injection variables published by other researchers [9, 10, and 11].

From ground material, after injection, the obtained specimens were named R1 so that these were pieces that have been obtained after a first recycling. Specimens identified as R2 were obtained from the ground of the R1 pieces, that is, after two recyclings. Pieces coded as R3 have been obtained from the R2 parts, and so on until ten recyclings. All specimens were tested in dry as moulded (DAM) conditions.

3. Experimental methodology

The viscosity of thermoplastics is inversely related to the average molecular weight of the polymer. Therefore, the polymer processing conditions directly affect the viscosity. During the plasticizing process breakage of molecular chains by shearing or overheating takes place, thereby the viscosity measurement before and after the molding determines the amount of the changes. The viscosity of the thermoplastic was characterized by means viscosity number VN [12].

To mechanically characterize the behavior of recycled material of polyamide, the following tests were performed (all of them at room temperature (23 ± 2ºC) and 50±5% of relative humidity):

- *Static efforts*: A servo hydraulic universal testing machine with a load cell capacity of 5kN was used to carry out tensile tests [2]. In order to determine the strain, an extensometer as base of 25 mm was employed.
- *Impact dynamic efforts*: An instrumented pendulum with a load cell capacity of 5J was used to carry out Charpy impact tests [3].
- *Fatigue dynamic efforts:* The accelerated fatigue LOCATI test technique was employed [13, 14] to determine the endurance or fatigue resistance with one single test. The method consists on applying an increasing step load, from a value lower than the fatigue limit, over a constant number of cycles. In this case, blocks of 20,000 sinusoidal wave cycles were applied at a frequency of 5 Hz, between initial tensile loading values of 0,5 and 1,5 kN. The minimum value remained constant in every block and the maximum load was increased by 0,25 kN per block. During the test strain values and surface temperature of the specimen were recorded, for the latter parameter using infrared thermography.
- *Static efforts with defect*: Fracture toughness test [4] by means of three-point bending tests using a servo hydraulic universal testing machine with a load cell capacity of 5kN. The standard specimens used, SENB notched type, with a thickness “B” = 4 mm, width “W” = 10 mm and an initial crack length “a” between 0.45 W and 0.55 W. The tests were carried out at 10 mm / min on two cylindrical supports spaced 4.4 W.

Finally, a study of the porosity detected was performed by means of optical microscopy and fractographic analysis of the fracture surfaces of the specimens subjected to various mechanical tests.

4. Results and discussion

4.1. Viscosity Number

The graph of Figure 2 shows a decrease in the viscosity number as a function of the number of recycling. There is an initial 16% drop between the viscosity number of the raw material (GV) and the first recycling (R1) that is due to the joint effect of the recycling process and the different origin of the raw material (from different suppliers: DSM, DuPont and BASF) that show different original viscosity numbers. After the first recycling with homogenized material, the reduced viscosity number was progressive up to R4 where a tendency change can be seen since the viscosity number begins to stabilize. This fact could indicate that the effect of the molecular chains attrition due to the successive injection couldn´t be detected with this technique of measurement.

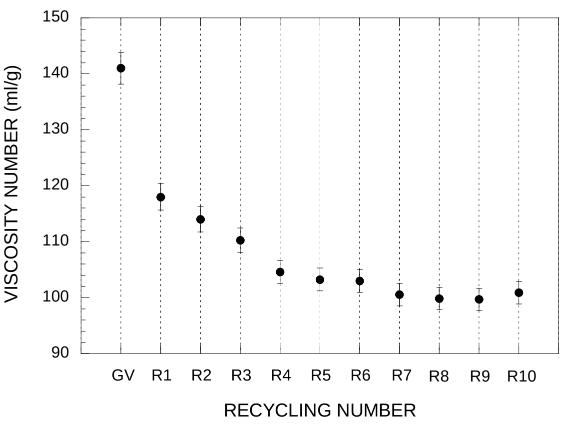

Figure 2. Evolution of viscosity number as recycled material.

4.2. Static Behavior: Tensile test

Figure 3(a) shows the tensile behavior of one of the three specimens tested for each conditions of recycling. It can be seen that the curves show, as the number of recycling increases, a general reduction in strength, flexibility and deformability. The graphs of Figure 3(b and c) show in detail the decrease in the value of the parameters measured in all the specimens tested as the number of recycling increases. It can be appreciated that the stabilization of the viscosity number determined in the preceding paragraph from R4, is not reflected in the tensile mechanical behavior. It should be noted that the mechanical study examines the macroscopic behavior of the material considering matrix, fiber and interface deterioration together [15] and also outer situations, nondependent on the material, such as metal inclusions or porosity, while the viscosity number only analyzes the situation for the polymeric matrix.

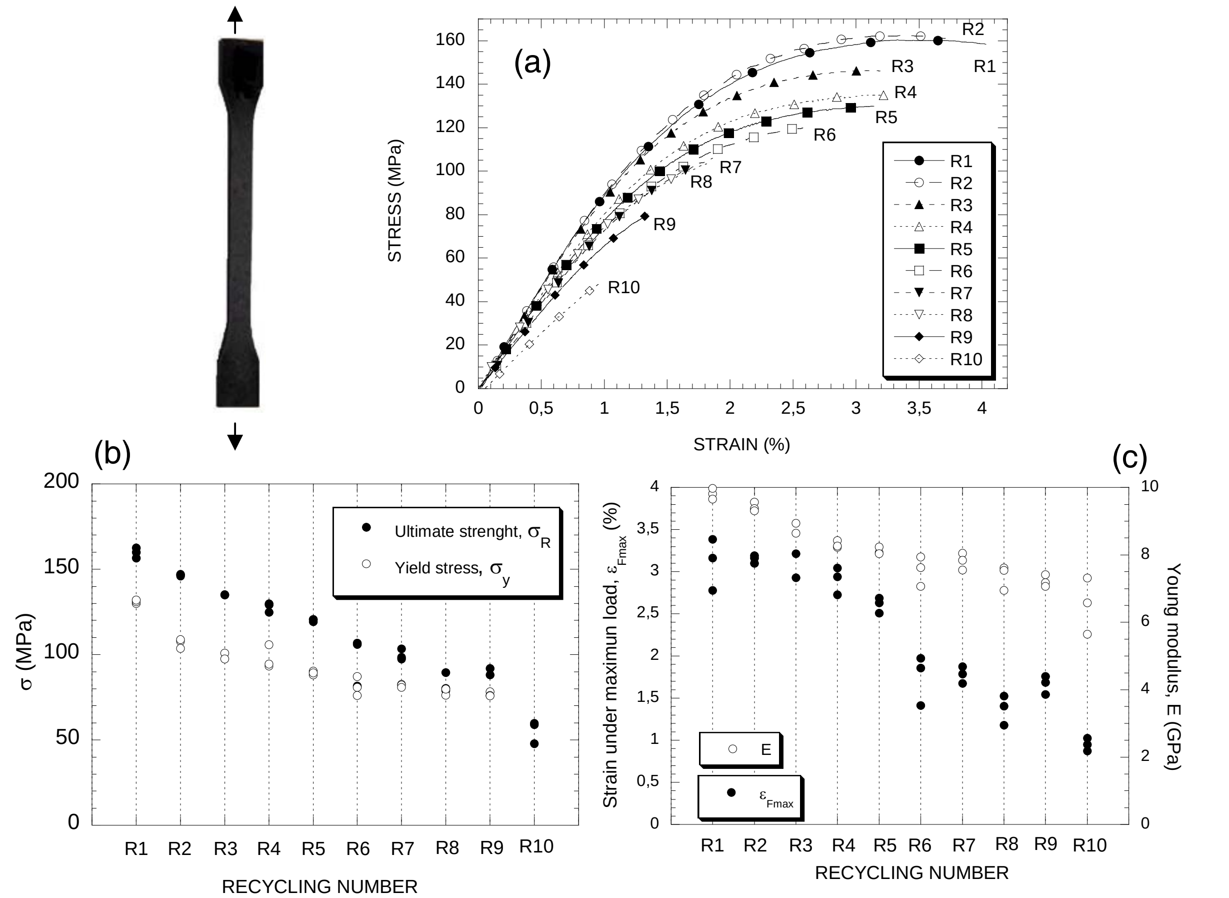

Figure 3. (a) Tensile test curves. (b) Strength parameters evolution (c) Strain and Young's modulus evolution

4.3. Dynamic behavior: Charpy impact test

Regarding to the behavior under dynamic impact loads, the results show the same trend as for those obtained in the tensile test. There is a continued decreasing of resilience as the number of recyclings increased, as can be seen in Figure 4.

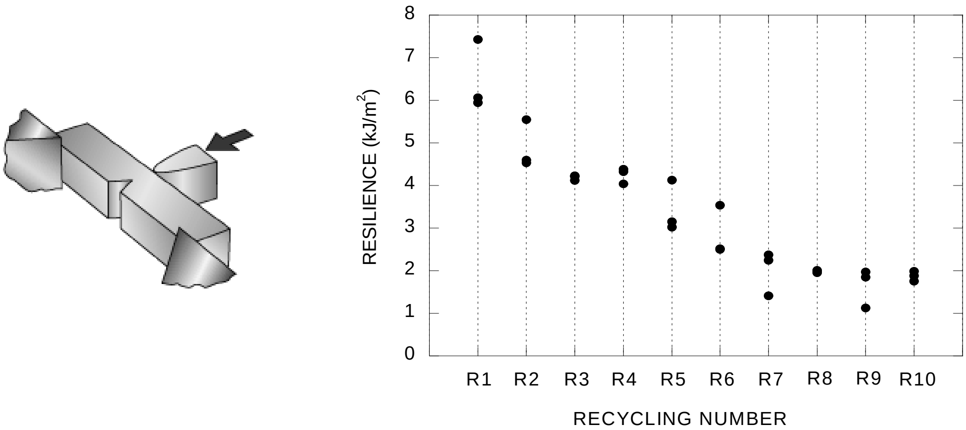

Figure 4. Evolution of resilience with recycling number

4.4. Dynamic behavior: Fatigue accelerate test LOCATI

The graph of Figure 5 shows the evolution of the maximum strain of the specimens as a function of cycle number for the ten recycling conditions.

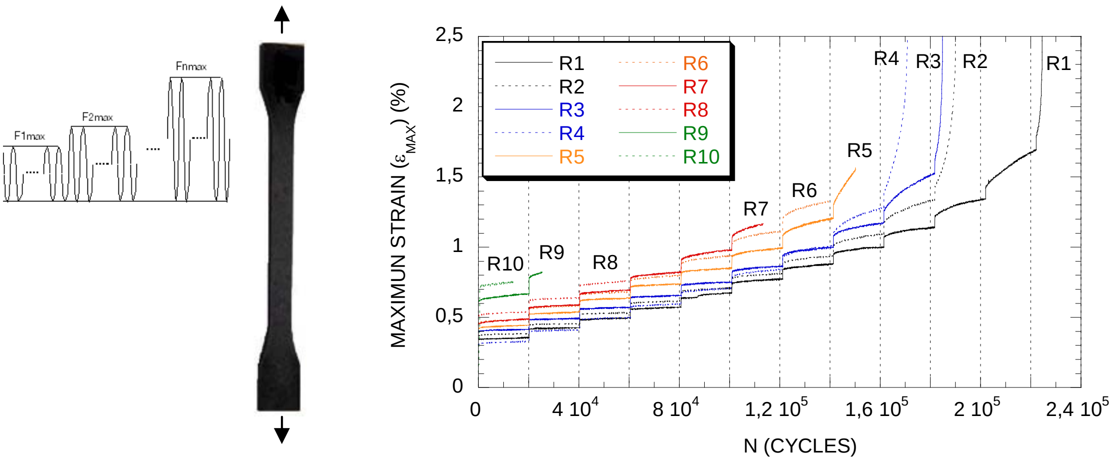

Figure 5. Comparison of maximum strain between the different recyclings in the LOCATI test

Figure 6 shows the two different types of fracture behavior that were observed on the recycled material: thermal failure and mechanical failure. In the graphs the evolution of the maximum and minimum strain, and the surface temperature of the specimen measured by means of infrared thermography with respect to the number of test cycles are shown. Figure 6(a) represents the type of failure due to thermal fatigue, in which the fracture occurs after a large number of cycles during which the levels of strain and temperature fracture increase progressively. At this point, the surface temperature reach critical conditions that indicate their accelerated growth acquiring values near 40°C and 3% before failure. Figure 6(b), by contrast, represents the mechanical fatigue failure, where temperature stabilizes in every loading block. In this case, the temperature level falls below 25°C and the strain does not exceed 1%, without any evidence of accelerated increase of the recorded controlling values at the end of test.

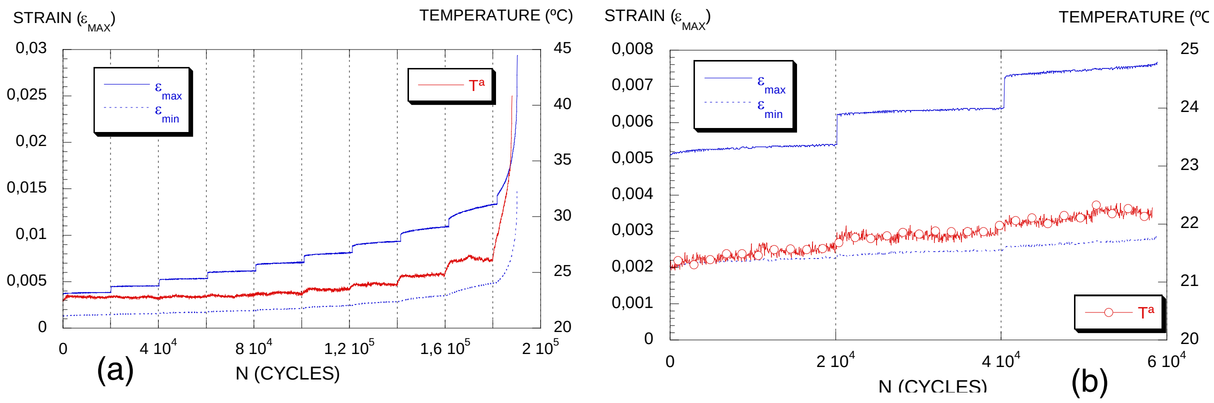

Figure 6. LOCATI test. (a) Thermal fatigue failure (b) Mechanical fatigue failure.

Analyzing the variation of the strain and temperature it can be seen that from recycling R1 to R4, fatigue failure is set as a thermal type, while from R7 to R10 failure is established mechanical type. Conditions R5 and R6 are the border between the two types of failure but in response to the maximum rate strain, R5 corresponds to thermal failure, while R6 would be classified as mechanical failure.

The criteria established for define the fatigue limit or endurance is the higher load variation value of the LOCATI test in which the maximum strain is not stabilized and the corresponding strain rate measured in the steady state starts to grow [16]. The graph of Figure 7(a) shows the damaging effect of the material recycling on the fatigue limit, decreasing almost linearly with the number of times that the reprocessing is performed.

4.5. Static behavior with defects: Fracture toughness test

The condition of the thickness of the specimen ($B<2{,}5\cdot(K_{IC}/\sigma_y)^2$) is not fulfilled for low recycling number, while for recycling values above 5 the condition is accomplished. Then the material property, $K_{IC,}$ has been able to determine for high recyclings, whereas the value of toughness for that particular specimen geometry, K_Q, has only been able to determine for low recyclings. Figure 7(b) shows the mean value of $K_{IC/Q}$ parameter for different recyclings. It can be observed that the toughness decreases as the number of recyclings increases.

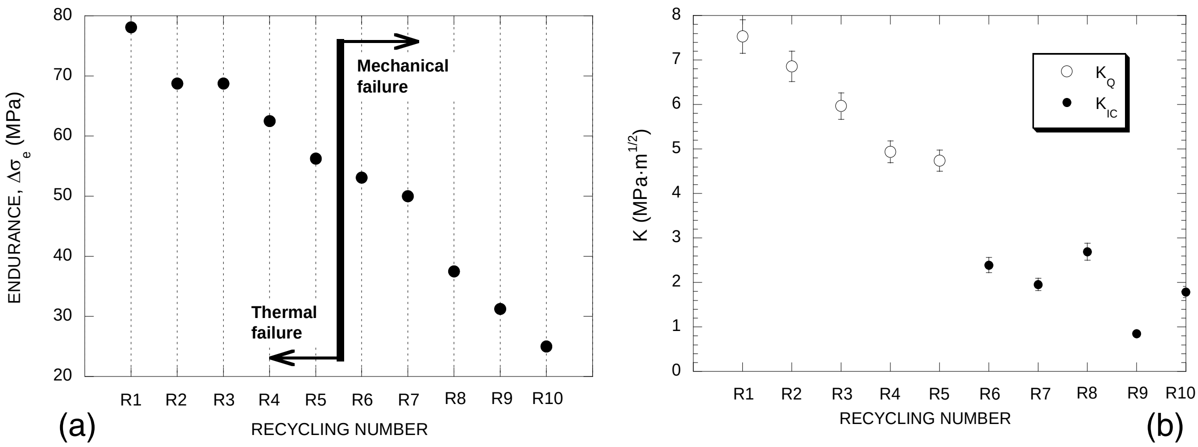

Figure 7. (a) Variation of endurance (b) Evolution of fracture toughness with recycling number.

4.6. Micrographic analysis

Parallel a fractographic analysis of the fracture fatigue surfaces of the tested specimens was performed. The existence of porosity was observed in those with high number of recyclings. Further study of the porosity distribution was carried out by cutting a tested piece of each recycling condition into five different sections along the longitudinal axis and, then, they were analyzed with an optical microscope. It was found that the porosity is only manifested in high recycling conditions, over R5. The porosity is evident in the recycling R10, Figure 8(a), where the porosity increases with the distance from the point of injection, reaches the value up to 15%. The pores are arranged in an annular region of increasing size with the distance from the injection point. The pore size distribution within the annular band is similar in all sections analyzed, being the largest in the inner region and decreasing in size as it approaches the periphery of the specimen.

The micrograph in Figure 8(b) shows the section of fatigue failure of the specimen R10, where the large existing porosity and appearance of the matrix surrounding the pores can be seen. This fact agrees with that observed in the curves of mechanical behavior, the fracture surface is completely brittle showing smooth fracture planes with little strain. Figure 8(c) shows the interconnecting pores through cracks, but with a flat matrix surface, corresponding to whole brittle aspect for fatigue fracture section of specimen R9. By contrast, the figure 8(d) shows the large strain undergone the matrix R1 due to the fatigue, corresponding to a typical thermal fatigue failure. In the fracture surface, regions that present high strain produced during the propagation of the crack, and other regions that remain brittle aspect which is a result of the breakage of the material in the last fatigue cycle coexist.

In recyclings prior to R5, the mechanism of crack growth in the brittle area cannot longer use the pores as preferred fracture ways because they do not exist, and instead cracks uses the empty accommodation of the fibers, that have lost their adhesion with the matrix, to move along a perpendicular plane to the efforts applied (Figure 8(e)).

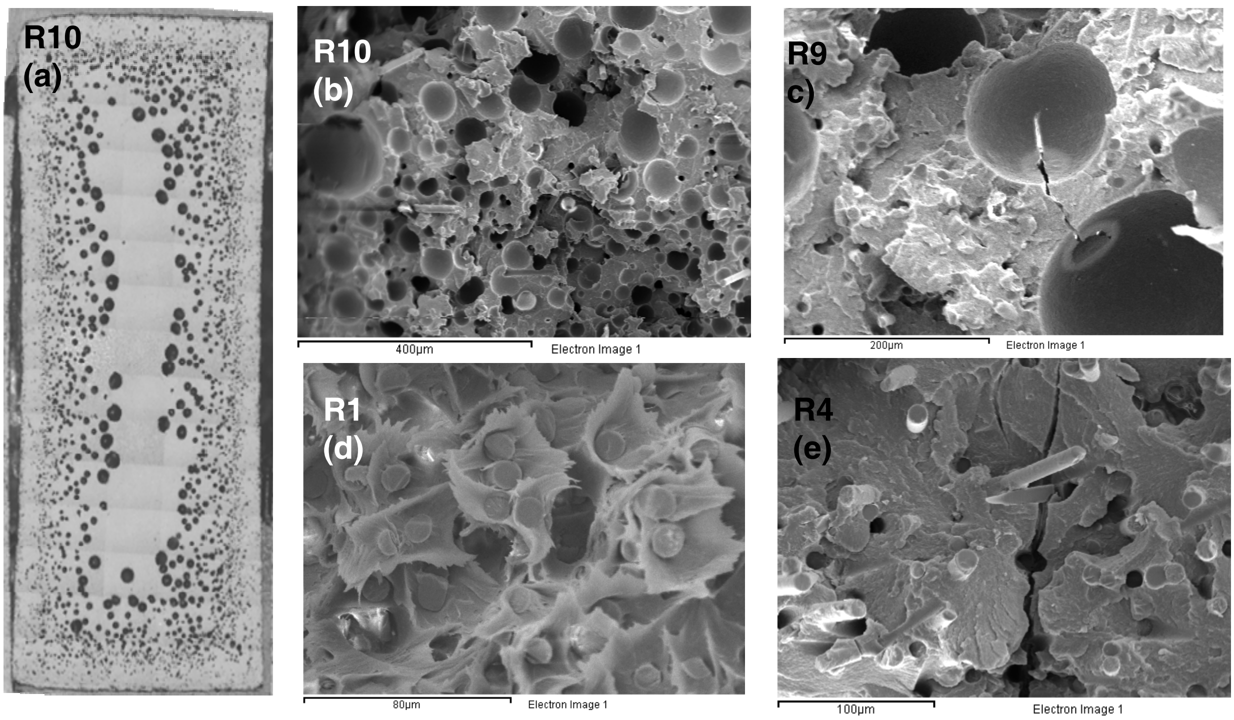

Figure 8. Images micrographic analysis.

5. Conclusions

It can be noted that in the mechanical characterization of the standard specimens recycled repeatedly up to 10 times, an almost linear decline in the analyzed mechanical properties was observed. Moreover also it was verified that from recycled R5 the porosity increases, reaching 15% in the case of R10. This fact accelerates the loss of mechanical properties, causes a loss in remaining net section, and the pores act as preferential paths for the propagation of the cracks. The analyzed results suggest that up to a third mechanical recycling the material could be used with a loss of mechanical properties less than 20%.

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