Effect of the chemical surrounding on the properties of aged PEEK and Glass Reinforced PA66

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ABSTRACT: A study is presented on the response thermoplastic composite to aging conditions in presence of lubricants. Glass Fiber reinforced Polyamaide 66 (GF PA66) are proposed as commercial material already used in the field while Polyether Ether Ketone (PEEK) is proposed as novel polymeric candidate material due to its chemical stability and high mechanical performance. Accelerated aging tests were carried out to study the response of these materials to external chemical agents under different conditions. Afterwards, a complete set of mechanical and physical tests and characterization procedures were carried out, before and after ageing on suitable samples of the polymers in combination with lubricants, to evaluate the effects of the chemical environment on the mechanical and physical properties of the polymers. GR PA66 presents better global mechanical properties due to the important glass fiber load (30 %). Nevertheless, the values obtained also show that PEEK remains unalterable under different aging conditions. According to the results it can be suggested that PEEK might be considered as a suitable potential alternative, when necessary, in applications with a high resistance to chemical environment requirements.

Keywords: lubricant, Glass Reinforced PA66, PEEK, accelerating aging test, mechanical properties

1. Introduction

In the last decades thermoplastic polymers and polymer-based composites are acquiring high importance substituting metal materials in bearings and engine components.[

[1]] High performance thermoplastic polymers are among preferable options due to good mechanical stability, low weight, and resistance to destructive environments which often, like in the case of cages for bearings of bogie trains, acts as a lubricant. On the other hand, high performance thermoplastic polymers are among preferable options due to good resistance to destructive environments which often like in the case of cages for bearings of bogie trains acts as a lubricant.[[2]]

Polymer cage is a key component achieving reliability and safety of tapered roller bearing units (TBU). Historically, pressed steel cages were applied for TBU. Today, mainly polymer cages are used. They have been in operation since 1990 with excellent results. Pressed steel cages are fitted only on specific customer requests. Usually, pressed steel cage failed after 70 km with a continuously increasing operating temperature, up to complete seizing of the bearing. Under the same conditions, the polymer cage can be operated at least five times longer than a pressed steel cage. Some of the polymer cage benefits consist in reduced friction and roller slip, reduced wear and lower operating temperature, improved safety and performance and safe failure mode without seizing.[[3]] Nowadays, cages are available in three plastic materials: PA46, PA66 and L-

PPS (linear polyphenylene sulphide) with special additives and high temperature resistant design. Also, tapered roller bearings can be fitted with cages made from glass fibre reinforced polyamide and have even longer grease operating life in this case.[4],[5] Glass Fiber reinforced Polyamaide 66 (GF PA66) has been proposed for this study as a commercial material already being investigated in the field while Polyether Ether Ketone (PEEK) has been proposed as a novel polymeric candidate material for these applications due to its chemical stability and high mechanical performance. PEEK polymer has prompted advances across different industrial sectors remaining a source of active innovation. Nevertheless, there are significantly more publications dedicated to study effects of temperature and chemical reagents on GF PA66 rather than on PEEK. This lack of information opens an important window to new publications to shed light on the properties and behaviour of this high-performance polymer whose interest in sectors as railway is being daily increased.

Regarding the presence of the lubricant in the surrounding environment of a bearing cage, the surface of an aged PA66 is susceptible to oxidative aging. This sensitivity can be increased when the material is exposed to certain chemicals like polyglycols without antioxidants presents in oil-based lubricant. Additives can deteriorate PA66 and reduce the mechanical properties significantly. Also, when heated up to 100°C for 1000 h, the thermal influence can increase the crystalline fraction, which also increases the embrittlement.[6] Nevertheless, very small change in flexural properties is detected for GR (glass reinforced) PA66 after aged at 150°C for 487 hr and 1000 hr in presence of two different lubricant mixtures: HFC-134a (refrigerant)/PAG mixture (oil); CFC-12/mineral oil. Only moderate modifications in weight, volumen and flexural modulus.[7] Also, other aging treatments produced in presence of water instead of lubricants have shown a decrease of mechanical.[8]

In summary, PA66 is a material with an important presence in the development of bearing for bogie train and hence there are much more extensive literature already published. On the other hand, the lack of works about PEEK opens a window to propose this high-performance polymer as a well candidate to develop cages. The main tests that have been published to analyse the impact of lubricants and temperature on polymer properties are microscopy, tribology, tensile and flexural tests. In what follows, results corresponding to several selected tests (based in those already published in literature to compare) are shown with the aim of compare mechanical and physical response of PA66 and PEEK to the operation conditions corresponding to bearing cages of a bogie train. During the lab testing, the selected experimental parameters are in the same range of that proposed in most of the papers studied, special attention is dedicated to temperatures and aging time. While oil-based lubricants are widely studied, very few research has been done up to date dedicated to the effect of grease on polymer properties, which also might lead to novel results.

2. Materials

The following thermoplastic polymers chippings (special for mould injection procedures) were purchased from Biestrefeld:

- PA66 reinforced with Glass Fiber (33%) (Zytel® 80G33HS1L BK104, NYLON RESIN, PA66-I-GF33)
- PEEK. VICTREX® PEEK 90G. High performance thermoplastic material, unreinforced PolyEtherEtherKetone (PEEK)

- Lubricants: oil and grease typically used as lubricants in railway applications were purchased from Shell and Spirax:
    - Solid Lubricant: Shell Gadus Rail S4 High Speed EUFR

- Liquid Lubricant: Spirax S6 AXME 75W-90

3. Experimental

3.1 Samples fabrication

Injection moulding was the technique selected for samples manufacturing according to the standard ASTM D790, which recommends the geometry for subsequent mechanical tests (flexural and tensile). 30 samples per type and material were produced, 120 in total. This important amount allowed an extensive and complete experimental study. Figure 1.e displays an optical picture of the different types of manufactured polymeric coupons: dog-bone type specimens for tensile tests, rectangular for flexural tests, whitish correspond to PEEK and blackish correspond to GR PA66.

3.1. Aging tests

Testing of samples before and after accelerated ageing in lubricant environment to evaluate the effects of oil-based lubricants and grease-based lubricants on structural properties of polymer specimens took place according to standards UNE-EN 12080 and ASTM D543 dedicated to evaluate the resistance of plastics to chemical reagents and which proposed the immersion technique using during this work. Sets of 5 samples (number of samples recommended by standards to ensure reproducibility) for each type of coupon (tensile and flexural) and for each type of material (GR PA66 and PEEK) were immersed in the three different types of surrounding environments (air, oil and grease) during two different times (250h and 500h) and at two different temperatures in an oven (Room Temperature, RT, and 140ºC). Table of figure 1 shows the list of the sample-sets studied and their corresponding experimental conditions for the aging tests: aging time, environment material, and temperature. Row in grey indicates the blank sample used as reference.

Sample marking GR PA66	Sample marking PEEK	Ageing conditions		
		Ageing time	*Environment*	*Temperature*
1.1	2.1	250 h	Grease	140°C
1.2	2.2	500h	Grease	140°C
1.3	2.3	250 h	Grease	Room Temperature
1.4	2.4	500h	Grease	Room Temperature
1.5	2.5	250 h	Oil	140°C
1.6	2.6	500h	Oil	140°C
1.7	2.7	250 h	Oil	Room Temperature
1.8	2.8	500h	Oil	Room Temperature
1.9	2.9	250 h	Air	140°C
1.10	2.10	500h	Air	140°C
1.11	2.11	500h	Air	Room Temperature

Figure 1 Sample- sets studied and their corresponding experimental conditions for the aging tests: aging time, environment material, and temperature. Row in grey indicates the blank sample used as reference.

3.2. Scanning Electron Microscopy

The microscopic examination samples after tensile testing was carried out using a JEOL JSM-7600 (Japan) scanning microscope at different magnifications. SEM has much better resolution and depth of field than optical microscopes, which are key to revealing the topographical features of fracture surfaces. After cutting of specimens to size (height about 20 mm) the specimens with both fracture surfaces were mounted, allowing both faces to be viewed. Afterwards, the specimens were coated using sputter coater with a gold target.

3.3. Tensile tests

Standard UNE-EN ISO 527-2:2012/A/1 was selected to investigate tensile properties of polymeric materials evaluated in this work, GR PA66 and PEEK. Standard test specimens were fabricated following the procedure described in the point 2 of this section. During the testing, samples were subjected to a controlled tension until failure. Some of the most relevant parameters of test are temperature (ºC): 21; test speed (mm/min) from initial section up to 0.3% deformation: 1 mm/min; test speed (mm/min) from 0.3% deformation to final section: 10 mm/min; equipment used: SHIMADZU 5 kN Nº31080.

3.4. Flexural tests

UNE-EN ISO 178:2020 was selected to investigate flexural properties of polymeric materials evaluated in this work: GR PA66 and PEEK under defined conditions. Standard test specimens were fabricated following the procedure described in the point 2 of this section. Some of the most relevant parameters of the test are temperature (ºC): 20; flexural type:3 point bending; support Span (L)(mm): 64; load Rollers diameter (mm): 10; equipment used: SHIMADZU 5 kN Nº31080.

3.5. Tribology

Tribological pin-on-disk testing was performed on samples of the two types of analysed thermoplastic polymers: PEEK and GR PA66 using a disk from 100Cr6 bearing steel and following standard ASTM G99-95a and EN ISO 7148-2:2012. All polymeric samples were preconditioned for period 24 h prior to test at standard atmosphere (temperature 23 ºC, 50 % air humidity). The tribology study was performed on the processed surfaces by the "pin-on-disc" dry and lubricated friction test using a Microtest tribometer (Microtest, S. A., Spain) under the following considerations: Pin parameters: the diameter of polymer samples 3 mm, cross-section about 7 mm2, length 20 mm. Disk parameters: Diameter of 100Cr6 bearing steel disk 45 mm, thickness 5 mm. Steel surface hardness 59HRC. Steel disk surface roughness Ra 0.3 Ra 0,3 (at the contact area). Apparatus: typical pin-on-disk test system with vertical rotation axis. Test conditions: Radius of the trajectory (varied from 5 till 17.5 mm); Temperature of testing (ambient): 23ºC; Linear velocity: 0.1 m/s; Time: 8000 s; Wear distance: 800 m; Specimen test force (load): 7N. The surface micro roughness parameters of the tested samples were measured using a profilometer Surtronic 3+ (Taylor Hobson, UK) with an accuracy (vertical resolution) ± 0.01 µm. Also, a Precisa XR 205SMDR" (Precisa Gravimetrics AG, Dietikon, Switzerland) analytical balance with an accuracy of 0.00001 g was used to measure the mass of the samples before and after tribology test and to evaluate samples mass loss ⍰m.

3.6. Hardness

Hardness testing was performed on the surface of aged samples of 2 different types of polymeric materials (PEEK and GR PA66) following the standard EN ISO 868:2003. Measurements were taken in five different points for each sample for a better evaluation. Some of the most relevant experimental parameters are: dimensions of samples: thickness 4 mm, width 24 mm, length 48 mm; the temperature of testing:19°C (room temperature); equipment: Shore D digital durometer HDD 100-1 (Saulter AG, Switzerland) with special stand Saulter TI-D and 5 kg weight centered on the axis of indenter; range of hardness measurements (D scale): 1-100; accuracy of durometer ≤± 1 %; reading of scale after 15 s.

Following subsections describe the results obtained for a complete series of tests and characterization procedures to analyse the effect of lubricants during aging of thermoplastic polymeric materials proposed for cages.

4. **Results and discussion**

4.1 Weight Loss

The effect of the presence of different lubricants during aging tests on surrounding environment of proposed polymers was primarily evaluated by evaluating the weight loss of prepared coupons before and after the tests. Results, consisting in lost or won weight percentage are represented in the bars diagram of figure 2, are very revealing. Coupons injected for the tensile tests were used to make these measurements. Results corresponding to GR PA66 are represented at the left side while results corresponding to PEEK are represented at the right side. Solid and grated colors represent results for shorter and longer test time: 250h and 500h respectively. Different colors correspond with different surrounding environment (air, oil and grease) and temperatures (RT and 140ºC).

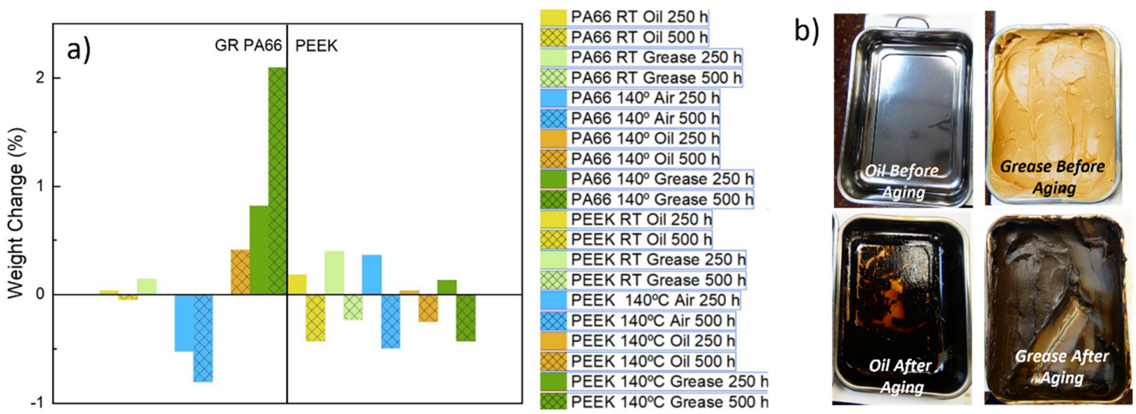

Figure 2 a) Weight loss corresponding to tensile samples for GR PA66 and PEEK after aging under different parameters.b)Pictures showing lubricant before and after the ageing process.

In the case of GR PA66, it is observed that samples aged at RT do not significantly modifies their weight, in this case, the presence of lubricants does not affect independently of their nature. GR PA66 must be heated up to, at least, 140ºC to appreciate the different effects of lubricants presence in the surrounding environment. If this is the case, two different behaviours have been observed depending on if the aging of the polymer was done in presence or not of lubricant. On the one hand if no lubricant was present, weight of samples was decreased, on the other hand, if polymeric coupons were aged immersed in oil or grease an increment of weight was systematically measured, being higher for grease than for oil. This is due to the existence of certain degree of porosity in the polymer. Pores of the polymer host solvents like water or other products of the melting during the injection process that are easily removed at 140ºC giving rise to the small weight reduction observed. But when the samples are soaked in a lubricant during the aging, not only remaining solvents cannot be evacuated from pores, but these will be filled with the material of surrounding medium. Mobility of the molecules of both grease and oil is promoted by the temperature and the long-time of the process. Since grease is denser that oil, the weight increment of samples aged in presence of the solid lubricant is substantially higher than the one corresponding to the samples aged in the liquid one. Finally, time is an important factor which enhance all the effects explained before. On the other hand, the effect caused in the weight loss of PEEK by the different experimental parameters is not as relevant as in the case of the GR PA66. This is due to the remarkable difference in the porosity present in the microstructure of the two polymers. PEEK is a high-performance polymer with very good chemical stability characterized by its optimum mechanical properties due, in part, to its high density. That means that the microstructure of this polymer is close to crystalline whose molecules are very well packed, giving rise to a very low porous degree which makes very difficult to host molecules of any surrounding medium, air, water, or lubricants. The weigh, hence, remains unalterable under any aging treatments or any chemical environment. As seen in figure 2, modifications are systematically low (under 0.5%) and random, which suggest that no experimental condition is determinant in this case.

4.2 Tensile tests

Different significant mechanical properties were directly measured via a tensile test before and after the aging procedure with the aim of evaluating the potential effects produce by the different aging conditions described in the table of figure 1.b. A total of 120 specimens were analysed, 5 per material and condition, ensuring the quality of the results and consistency, which might lead to a reliable results report. The respective strain-stress curves characteristic of each case of study are plotted in graphs of figure 3.a for GR PA66 and 3.b for PEEK. In materials engineering, a stress–strain curve is obtained by gradually applying load to a test coupon during the tensile test and measuring the deformation, from which the stress and strain can be determined. These curves, typical of a polymeric material, reveal many important properties of a material represented in bars diagrams of figure 3 for a better comprehension. Each bar corresponds to average values obtained for different tensile properties: c) tensile strength, d) strain at break and e) elastic modulus. Values corresponding to GR PA66 and PEEK are clearly indicated in each graph. For every case, values corresponding to the blank sample used as reference are plot as a black bar. From all the studied magnitudes.

Results are very consistent both for GR PA66 and PEEK, which give an account of the quality of the measurements and the reliability of the analysis.

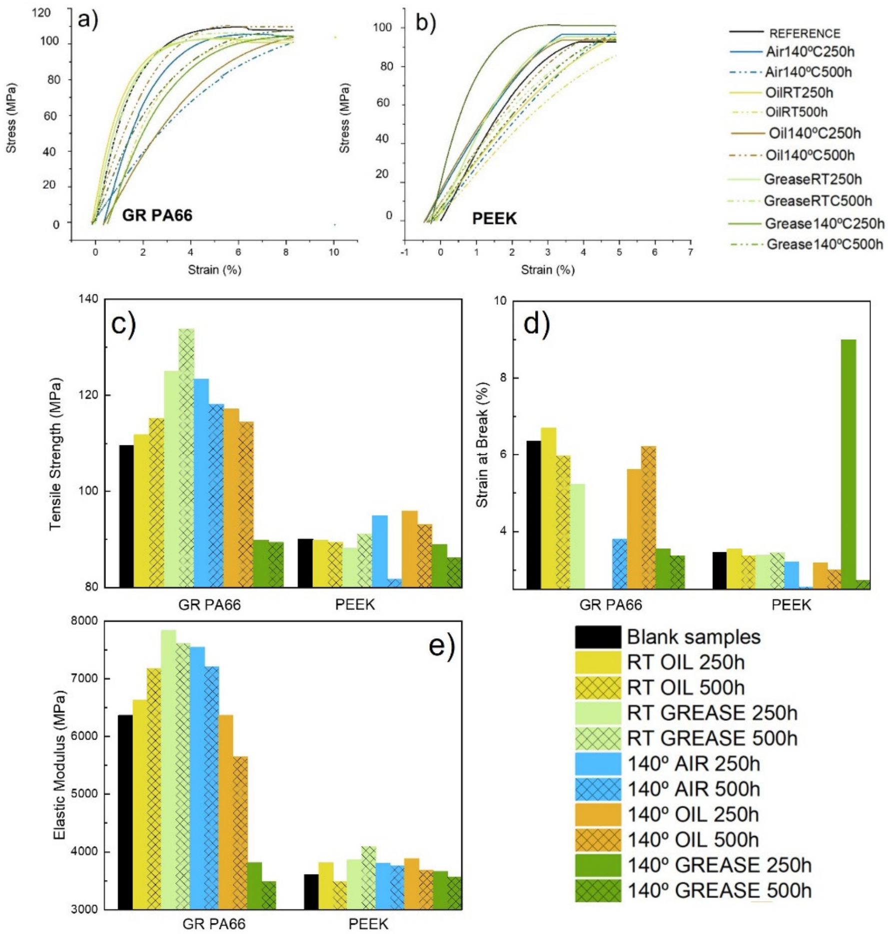

Figure 3 Stress-Strain curves corresponding to the different aging conditions for GF PA66 (a) and PEEK (b). Bars diagrams representing mechanical properties obtained from tensile tests of GR PA66 and PEEK aged under different conditions: c) tensile strength, d) strain at break and e) elastic modulus.

GR PA66 present substantially better mechanical properties than PEEK presenting both better mechanical resistance (higher stress values achieved) and better elasticity (higher strain percentage before breaking). Whereas PEEK is considered one of the thermoplastic polymers with highest mechanical performance, these exceptional properties are well overcome by PA66 due to the generous glass fibre reinforcement (33%). As well, as noticed by measurements of weight loss, the porous nature of GR PA66 comparing with PEEK made it much more receptive to the aging treatment under all conditions. Both, changes respect to the blank sample and effects of aging are barely noticed for PEEK, so the rest of the analysis will be focused on GR PA66, nevertheless, PEEK might be considered as a good candidate material to be used in applications involving thermal aging and/or presence of chemical reagents like lubricants. Considering the environment effect on mechanical properties: both, tensile strength and elastic modulus improve of GR PA66 after aging under almost any surrounding environment condition being the aging in grease at RT the most advantageous condition. There is an exception: grease environment at 140ºC. A significant decrease has been obtained in this case. Regarding the time effect on mechanical properties, it plays different role depending on the temperature: at RT aging time favours mechanical properties in the most part of cases while after aging at 140ºC mechanical properties decreases with the time. On the other hand, temperature also present effect on mechanical properties: important differences have been noticed depending on the chemical environment. When aging is performed in air, temperature enhance mechanical properties of GR PA66 while when the thermal treatment is done in grease, mechanical properties get severely worse at higher temperatures. Temperature on the other hand is not a relevant factor when oil is used as environment reagent.

4.3 Flexural tests

Several aspects of the stress/strain relationship such us flexural strength or flexural modulus were obtained for samples before and after the aging test with the aim of evaluating the potential effects produced by the different aging conditions described in the table of figure 1.b. A total of 120 specimens were analysed, 5 per material and condition, ensuring the quality of the results and consistency. In a similar way than in the case corresponding to tensile test measurements, the respective strain-stress curves characteristic of each case of study are plotted in graphs of figure 4.a for GR PA66 and 4.b for PEEK . Two different bars diagrams, each one corresponding to average values obtained for different flexural properties are as well shown: c) flexural modulus and d) flexural strength. Values corresponding to GR PA66 and PEEK are clearly indicated in each graph. For every case, values corresponding to the blank sample used as reference are plot as a black bar. Again, the porous nature of GR PA66 comparing with PEEK made it much more receptive to the aging treatment under all conditions, so the rest of the analysis will be focused on GR PA66. Nevertheless, PEEK might be considered as a good candidate material to be used in applications involving thermal aging and/or presence of chemical reagents like lubricants.

As observed before, flexural modulus of GR PA66 is significantly higher that flexural modulus of PEEK and the effects of aging effects under different conditions is systematically higher. While PEEK remains almost unalterable, measurements from tensile tests are consisting with those obtained for tensile tests and weight loss experimentations and reinforced the fact that PEEK, even if present worse mechanical properties compared with GR PA66, respond much better to thermal aging in presence of lubricants of different nature. Finally, flexural strength measurements obtained for both material are very similar being almost unalterable when aging treatments under different conditions are applied.

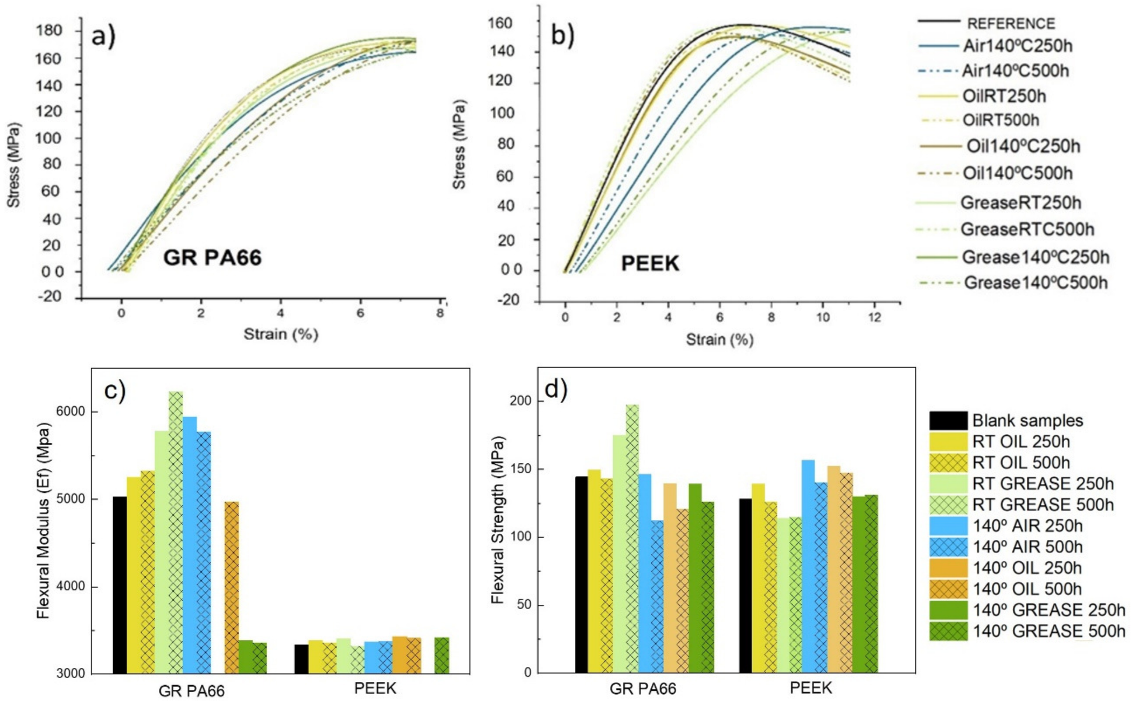

Figure 4 Stress-Strain curves corresponding to the different aging conditions for GF PA66 (a) and PEEK (b). Bars diagrams representing mechanical properties obtained from flexural tests of GR PA66 and PEEK aged under different conditions. Black bars correspond to reference values. c) flexural modulus and d) flexural strength.

4.4 Tribology

Tribology tests were performed with the aim of evaluating the effect of chemical surrounding media in the wear rate presented by the polymers object of this work. Tests were performed following procedures describe in detail in experimental section. The wear rate mw of the polymers was calculated by dividing the mass loss ▯m by wear distance and expressed in g/m. The average mw values of 5 tests are presented in this report. The friction coefficients were calculated after the test data for the start sliding distance were eliminated. Three different types of lubricants for the tests specimens were evaluated: Dry testing without any lubricants, lubrication with grease and lubrication with oil, for all the aging conditions described in the table of figure 1.b. The surrounding media (air, oil or grease) selected to carried out tribology tests for each samples coincides with the corresponding medium of the aging test. Figure 5 shows bars diagrams corresponding to values obtained for different tribology parameters: (a) kinetic coefficient of friction, (b) volumetric wear rate and losses of volume (c) and mass (d) of polymeric cylindrical pins samples taken using the different media: air, oil and grease. Values corresponding to GR PA66 and PEEK are clearly indicated in each graph. For each material bars corresponding to different temperatures and aging times are well indicated.

On the one hand, for the case of dry testing without any lubricants, results are more relevant for PA66 than for PEEK, which means that accelerating aging in air might affect GR PA66 while PEEK remains almost unalterable. The dynamic friction coefficients are typical for PEEK and PA66 reinforced with glass fiber samples in dry friction conditions. Friction coefficient of PEEK is about 2 times greater than PA66 with glass fiber. However, the friction coefficient of PEEK of the tested samples practically did not change, while friction coefficient values of PA66 with glass fiber depends more strongly on aging conditions. Moreover, the mass and volume losses of PA66 with glass fiber samples are until 2 times largest in comparison with PEEK samples. On the other hand, for the case of lubrication with oil, the comparative characteristics of PA66 GR and PEEK have a similar character and tendency as in the case of dry friction. Results depend on samples aging conditions, which mean that accelerating aging in oil might affect PA66 GR and PEEK. Only for PEEK this effect is significant only at elevated temperatures, and PA66 GR is affected even at room temperature. It should be emphasized that in the case of using an oil lubricant, the friction coefficient of the steel-plastic tribological pair decreased significantly. For PEEK it decreased to 0.17-0.41 compared to the case of dry friction, when it was 0.5-0.53, and for PA66 it decreased to the limits of 0.11-0.17 compared to 0.2-0.25. For PEEK, the use of an oil lubricant has a greater effect, and the friction coefficient characteristics of PA66 GR and PEEK become very similar. Talking about the speed and amount of wear, thanks to the lubricant, it has also been significantly reduced. For PEEK, the volumetric wear rate decreased to 0.055-0.106 mm3/km compared to the case of dry friction, when it was 0.131-0.176 mm3/km, and for PA66 GR it decreased to the limits of 0.043-0.089 mm3/km compared to 0.22-0.265 mm3/km. As can be seen from these data, the use of oil lubricant has a greater effect on the degree and rate of wear of PA66 GR than on PEEK samples. Finally, in the case of lubrication with grease, the comparative characteristics of PA66 GR and PEEK have a similar character and tendency as in the case of using oil as a lubricant. Results depend on samples aging conditions, which mean that accelerating aging in grease might affect PA66 GR and PEEK. Only for PEEK data obtained friction coefficients are quite contradictory and the nature of the influence is difficult to determine, and on PA66 GR the use of grease and the aging process is more pronounced at room temperature. It should be emphasized that in the case of using a grease lubricant, the coefficient of friction of the steel-plastic tribological pair did not change much compared to dry friction. For PEEK it varied between 0.37-0.5 compared to the case of dry friction, when it was 0.5-0.53, and for PA66 it varied within 0.2-0.39 compared to 0.2-0.25. For PEEK and PA66 GR, the use of grease as a lubricant does not give a great effect, as when using oil. The friction coefficient characteristics of PA66 GR and PEEK remain very similar to dry friction. This may be due to the high viscosity of the grease. Talking about the speed and amount of wear, thanks to the lubricant, it has also been significantly reduced. For PEEK, the volumetric wear rate decreased to 0.071-0.106 mm3/km compared to the dry friction case, when it was 0.131-0.176 mm3/km, and for PA66 GR it decreased to the limits of 0.036-0.089 mm3/km compared to 0.22-0.265 mm3/km. As can be seen from these data, the use of lubricant in the form of a grease in this case also has a greater effect on the degree and rate of wear of PA66 GR than on PEEK samples.

In summary, the use of any type of lubricant has a positive effect and significantly reduces the wear rate. Only for PEEK the influence of the presence of any lubricant is less significant. Talking about the coefficient of friction, its values are very dependent on the type of lubricant used, and the greatest effect of reducing the coefficient can be achieved by using a more liquid lubricant - oil.

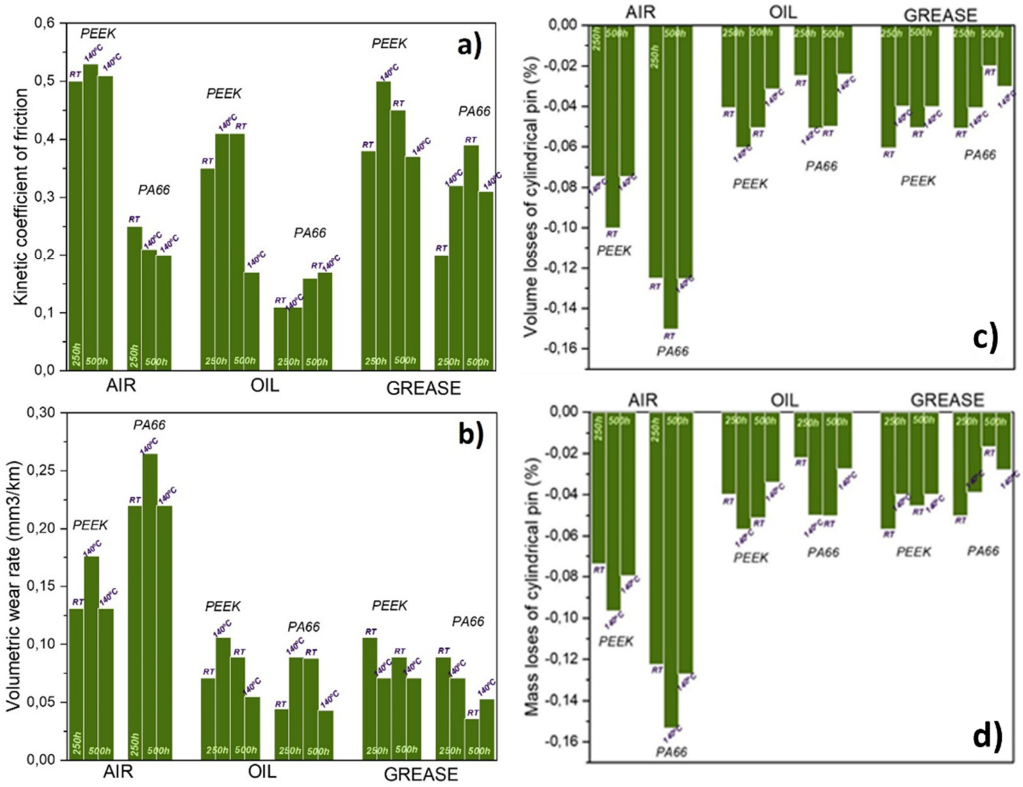

Figure 5 Measured values for kinetic coefficient of friction (a) and volumetric wear rate (b) corresponding to polymeric samples aged under different experimental parameters. Measured values for losses corresponding to volume (a) mass (b) of polymeric cylindrical pins samples aged under different experimental parameters.

4.5 <u>Hardness</u>

Hardness measurements were taken following the procedure explained in the experimental section for 5 different points in each sample of GR PA66 and PEEK respectively, for all the experimental accelerating aging test parameters described in the table of figure 1.b, corresponding to surrounding environment (air, oil and grease), aging temperature (RT and 140ºC) and time (250h and 500h). Results are plotted in bars diagram of the figure 6.

Typical hardness obtained for GR PA66 lays in the range from D68 till D82. Average hardness of aged samples varied from 74.5 till 76.5 while the medium measured value for hardness of all samples is 75.5. The difference of measurements respect to the blank sample is very low (within the range ± 1 %). On the other hand, typical hardness of PEEK can be from D82 till D95. Average hardness of aged samples varied from 81.8 till 82.5. The medium hardness of all samples is 82.1. Again, the difference of measurements respect to the blank sample lays in the range ≤± 1 %.

Very similar conclusions for GR PA66 and PEEK might be obtained. Differences in hardness measurement results are within the measurement error limits and the applied ageing conditions does not influence the hardness of tested GR PA66 and PEEK samples.

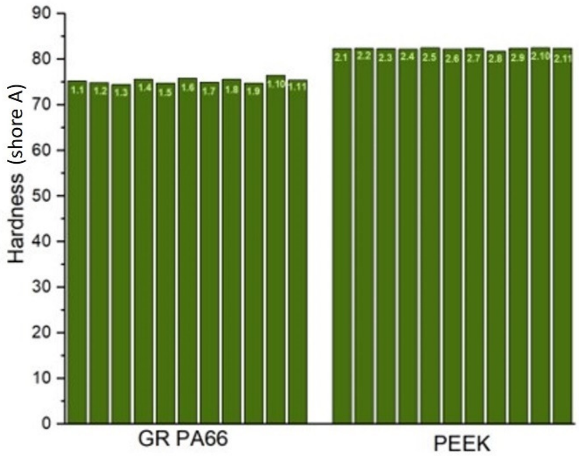

Figure 6 Bars diagrams corresponding to measured values for hardness for each sample aging under conditions of the table of the figure 1 well indicated for GR PA66 and PEEK.

4.6 Scanning Electron Microscopy

A detailed SEM analysis of all coupons aged under different conditions described before was carried out with the aim of evaluate a potential effect of aging conditions on microstructure which might explain the different findings exposed in this work. Results are quite consisting with the rest of this study. No relevant modifications in the aspect are observed for samples aged under RT for any of the surrounding media or for samples aged at 140ºC in air or oil. These observations extend both for polymeric matrix as for fiber fillers. But it can be observed that when samples are subjected to aging at 140ºC in presence of grease some differences can appreciated in the polymeric section. Figure 7 shows pictures corresponding to the cross section of coupons made with GR PA66 ((a) and (c)) and to PEEK ((b) and (d)) before ((a) and (b)) and after ((c) and (d)) an aging test in presence of grease at 140ºC and during 500h, all pictures were taken at the same magnification: x1200. From pictures it can be appreciated how granularity in these cases seem to enhance, which can suggest certain degradation which might explain the lack of certain properties as will be seen in previous sections. Also, consistently with the rest of results of this work, from the examination of the pictures corresponding to PEEK, no change in the aspect of materials can be appreciated under any of the experimental parameters used due to the high chemical stability presented by this polymer. This proves again the good response of PEEK to ageing procedures and chemical environment and shows the potential of PEEK as cages materials.

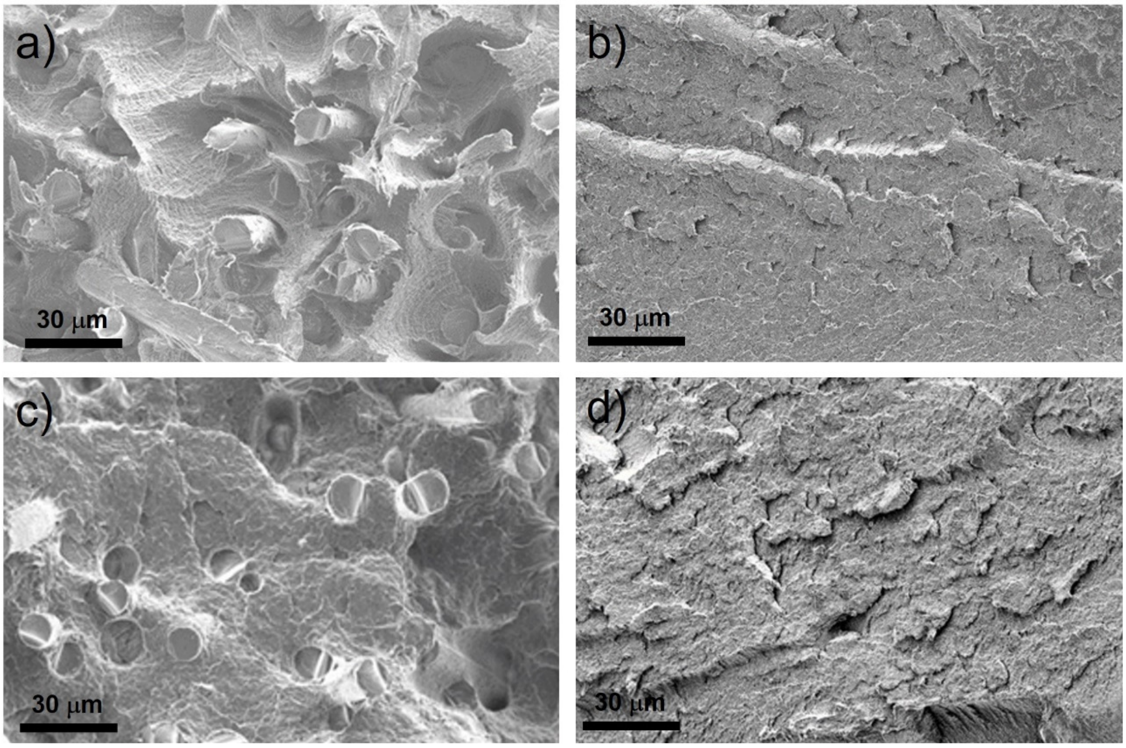

Figure 7 SEM pictures corresponding to GR PA66 (a) and (c) and to PEEK (b) and (d) before (a) and (b) and after (c) and (d) an aging test in presence of grease at 140ºC and during 500h.

5. **Conclusions**

This extensive study has led to many results whose analysis has been carefully detailed along the whole section and whose contents must be compile as follows:

- GR PA66 present better global properties due to the important glass fiber load (30 %). Hence better results have been achieved for mechanical properties: flexural and tensile testing. Values obtained in these tests shows that PEEK remains unalterable under different aging conditions.
- SEM pictures, weight loss measurements as well as hardness and tribology testing show that PEEK responds slightly better than GR PA66 to the aging tests under different parameters studied.
- Temperature of 140ºC and grease lubricant are the only two external parameters that affect (slightly) different physical and mechanical properties evaluated for GR PA66.

6. **Acknowledgements**

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