

INVESTIGATION OF THE MECHANICAL- AND MOLECULAR PROPERTIES OF POLYPROPYLENE RECYCLAT

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ABSTRACT

Due to the increasing demand for resource-efficient and sustainable products, recycling and the use of recycled materials are becoming more and more important. The so-called recyclates from the post-consumer- or industrial domestic waste is the resource efficient way of recycling plastics. Recyclates require up to 70 % less secondary energy for their processing than to produce virgin plastics. One of the greatest challenges of recyclates is, to provide them with constant material properties and quantities. For the use of recyclates in heavy loaded applications the materials must be optimized by several additives.

Material properties of recyclates from a domestic waste stream are published for static loading in several studies. Studies who investigate for example full factorial the static, cyclic and creep properties under different temperatures, aging conditions and mean stress ratios are not published or available now. Additionally, it is of great interest to get knowledge of the differences of the molecular structure of recyclat and virgin material.

In this paper a talc filled virgin and recyclat polypropylene (PP) was investigated concerning the mechanical and molecular properties. For the mechanical characterization static tension and fatigue tests on unnotched and notched specimens were carried out. Additionally, the aging effect at high temperature, the stress ratio, and temperatures at 23°C, 50 °C and 80 °C were investigated. The differences in the mechanical properties were described by analytical investigation. By the means of the fatigue investigation on specimen level two notch stress concepts (Highly Stressed Material Volume V_{80} , Stress Gradient χ^*) were used for the lifetime assessment (numerical calculation, part testing) of a door bearing pin of dish washer machine in household application. The lifetime assessment shows that virgin material of the dish washer can be replaced by the recyclat material. Based on an annual material requirement of 6,000 tons of material, a total of 2,500 tons of crude oil or 7,850 tons of CO₂ can be saved.

1 INTRODUCTION

Plastics have become an indispensable part of our daily lives. Plastics fulfil a wide variety of requirements, for example in food packaging, multimedia articles or highly stressed structural components in technical products. The plastic materials used in these applications are available worldwide in consistent quality and grade in reliable supply chains. Plastics can be processed extremely economically and with a high degree of reproducibility by injection moulding. Injection moulding also allows complex geometric shapes to be realised, which increase geometric rigidity and enable the integration of functions. In addition, plastics are ideally suited as lightweight construction materials due to their low density and sufficient strength.

Plastics are refined from crude oil, which as a finite source is only available to us humans for a limited time. The production of plastics requires primary energy, which is bound in the plastic as crude oil, and secondary energy, which is needed as auxiliary energy in the form of heat, steam, or cooling

water for various conversion processes. Thus, depending on the type of plastic, 2 - 6 kilograms of crude oil are required per kilogram of plastic granulate. Therefore, these resources and raw materials must be handled carefully and sustainably. This requires that plastic materials be kept in a recycling for as long as possible to use them as efficiently as possible.

Due to their molecular structure, thermoplastics can be reversibly melted and thus recycled. However, reversible melting cannot be carried out as often as desired, as plastics change their mechanical properties compared to the virgin material through repeated melting, shearing of the molecular chains and mixing of different plastic material streams. In addition, environmental influences change the properties during the service life, as additives and stabilisers are degraded, or oxidation processes occur.

Plastic waste that has already been processed once and has completed an application in use is collected as post-consumer or post-industrial waste. The plastics obtained from this are colloquially referred to as recyclates. In recyclates, the primary energy bound in the plastic is retained and only secondary energy is needed to process it. As a result, only a fraction of the energy is needed for recyclates than for virgin materials. Nevertheless, the use of recycled materials in highly stressed structural components is rather restrained. There are many reasons for this, such as poorer mechanical properties, low market availability, different quality, higher price, or lower user acceptance.

For virgin polypropylene with talc reinforcement, the fatigue properties have been investigated in [1], [2], [3], [4], [5], [6], [7]. In contrast, the studies by [8] and [9] are the only sources that have published structured fatigue tests on PP recyclates from post-consumer waste.

Therefore, this publication aims to present structured investigations on virgin and recycled plastic material. For this purpose, the influence of notches, weld lines, ambient temperature, ageing at temperature and load ratio were investigated by means of static and cyclic tests. Differences in the mechanical properties are described with accompanying analytical investigations such as the GPC analysis and the DSC analysis.

2 EXPERIMENTAL

2.1 MATERIALS

In this study, a recyclat- and a virgin material are investigated. The recyclat material is a black coloured PP homopolymer with 30 weight percent talc reinforcement (rPP MX30) and has the trade name and designation Seculene PPX 1250 TV30 (batch: 249A28090K) from the manufacturer BSB Recycling GmbH.

This is produced from old battery casings of lead-acid batteries. For this purpose, starter batteries that are no longer in working order are transported to collection points by certified transport companies for recycling. These starter batteries contain lead, zinc, insulators, the electrolyte made of diluted sulphuric acid and the casing made of PP. At the recycling companies, the starter batteries are opened in a crusher so that the diluted sulphuric acid they contain can be separated. The diluted sulphuric acid is processed and sent for further use. Lead, zinc, insulators, and plastic are separated from each other into individual material streams. Lead and zinc are melted down into ingots. The PP casings are crushed and cleaned of adhering dirt and paper labels in special cleaning processes in a caustic bath at high temperature. In some cases, the material stream is supplemented with other PP waste. After drying, the flakes are melted down in a compounding process and the melt is cleaned in special filter systems. Fillers, additives, and colourants are added to the plastic melt, from which pellets are produced again in a pelletising process.

Then virgin material is a PP homopolymer with 30 weight percent talc filling (PP MX30). This serves as the reference material due to its current use.

Table 1 compares the most important physical material properties of the recyclat- and the virgin material from the material data sheets.

	Virgin Material	Recyclat Material
Material type	PP MX30	rPP MX30
Polymer type	Homopolymer	Homopolymer
Flexural modulus	3,800 N/mm ²	3,500 N/mm ² (v = 2mm/min)
Young´s modulus	3,400 N/mm ²	-
Yield stress	33 N/mm ²	33 N/mm ² (v = 50 mm/min)
Yield strain	5 %	5 % (v = 50 mm/min)
MVR	9 cm ³ /10 min (MVR 230/5)	11.5 cm ³ /10 min (MVR 230/5)
Density	1.14 kg/dm ³	1.15 kg/dm ³

Table 1: Properties from the material data sheet of recyclat- and virgin material

2.2 SPECIMENS

For static and cyclic material characterisation, the unnotched and notched specimens shown in Figure 1 were used. The specimens are injected on the upper side by a pin gate. The notches are created by flowing around a core with the respective geometric dimensions. When flowing around the core, a weld line is created which is in the direction of the load but does not influence the mechanical properties. There is a predominant orientation of molecules and fillers of 0 ° to the flow direction of the melt.

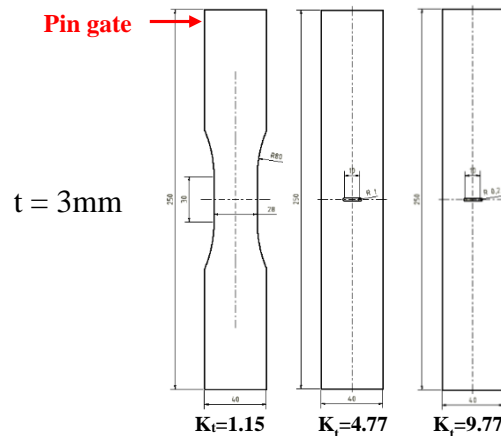


Figure 1: Geometric dimensions of the test specimens

2.3 EXPERIMENTAL PROCEDURE

The static and cyclic tests were carried out on servo-hydraulic testing machines with a force range of up to 63 kN. The test setup used differs in static and cyclic tests only in the application of the measurement sensors, a buckling device for cyclic compression loading and a climatic chamber for $T > 23$ °C (

Figure 2). Calibrated load cells with a nominal load of 25 kN (Interface 1720AJ-25KN) were used to measure the force. The clamping length of the samples in the clamping jaws was 40 mm and the free length of the samples $l_0 = 120$ mm. The tightening torques of the clamping bolts were selected in such a way that the load was introduced into the specimen via frictional locking. The samples were stored in an office climate until they were tested.

During the static tests, the force was recorded using a load cell and the deformation using a strain extensometer (Sandner EXA10-2.5u) to determine the young´s modulus, yield stress and yield strain. In

the cyclic tests, force and deformation is measured by a laser extensometer (Micro Epsilon optoNCDT 1320-10) or inductive displacement sensor (HBM WA T-10). The load was displacement-controlled and was controlled via the servo hydraulic piston. The loading speed was $v = 10 \text{ mm/min}$, and the environment condition was air at a temperature of $23 \text{ }^\circ\text{C}$.

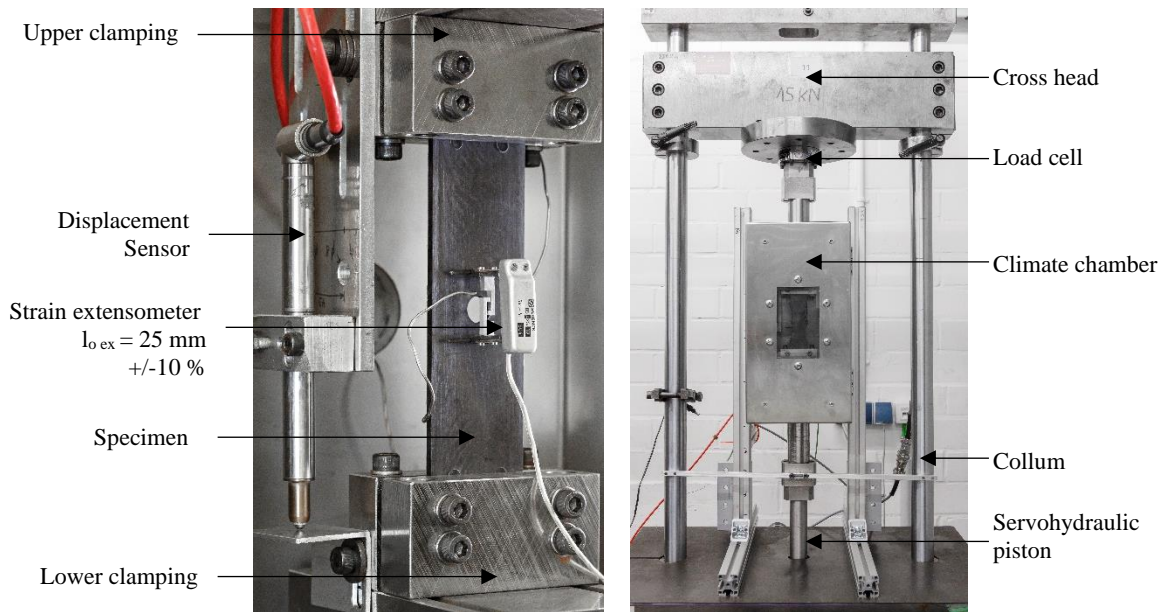


Figure 2: Set-up of the measuring equipment (left), test machine for static and cyclic loading with climate chamber (right)

For the cyclic tests a calibrated laser extensometer (Micro Epsilon optoNCDT 1320-10) or inductive displacement sensor (HBM WA T-10) (

Figure 2, left) was used to measure deformation under cyclic loading. There the deformation was measured in the free clamping length of $l_{0, \text{Sensor}} = 120 \text{ mm}$. The investigated ambient temperatures are $23 \text{ }^\circ\text{C}$ and $80 \text{ }^\circ\text{C}$. The temperature of $80 \text{ }^\circ\text{C}$. was applied using a climatic circulating air chamber. For this purpose, the local temperature on the sample was controlled. A constant test frequency of $f = 1 \text{ Hz}$ was used for the considered cycle interval up to $N = 2 \cdot 10^5$ cycles to minimize time-dependent effects and local warming caused by the influence of the test frequency. The cyclic tests were carried out force controlled. For alternating loading of $R = -1$, a anti buckling support was used to avoid buckling of the specimens under compression load. For tensile loading $R = 0$, no anti buckling support was used.

During the cyclic testing, a photographic image of the current state of the specimen was taken at intervals of $N = 200$ load cycles to investigate the damage progress and crack propagation in the highly loaded region. For this purpose, the cyclic loading was interrupted after $N = 200$ load cycles and the sample was loaded to the top load of the loading amplitude to carry out a photographic image. Additionally, the peak values were measured at maximum and minimum force for each load cycle and the hysteresis of force and deformation was recorded by 100 points per load cycle.

3 EXPERIMENTAL ANALYSES

3.1 ANALYTIC INVESTIGATION

By means of High Temperature Gel Permeation Chromatography (HT-GPC), the PP MX30 and the rPP MX30 were investigated with regards to their molar mass distribution. HT-GPC investigations were carried out on material samples of:

- Granules (granules of PP MX30 and rPP MX30)
- Injection moulded samples (Specimen $K_t = 1.15$ (Figure 1) of PP MX30 and rPP MX30)

Low molecular weight compounds have an exact molar mass, whereas polymers have a molar mass distribution. The molar mass distribution reflects the proportional distribution of the molar mass of the contained molecules. With a narrow molar mass distribution, there is a high uniformity of the molecular chains, resulting in a narrower thermal softening range.

Figure 3 shows the molar mass distribution of PP MX30 and rPP MX30 of the different material samples. In general, for both materials, the material samples of the granulate have a higher molar mass distribution than those of the specimens. This is due to further thermal stress and shear from processing. For the rPP MX30, there is a lower molar mass and a narrower molar mass distribution than for the PP MX30.

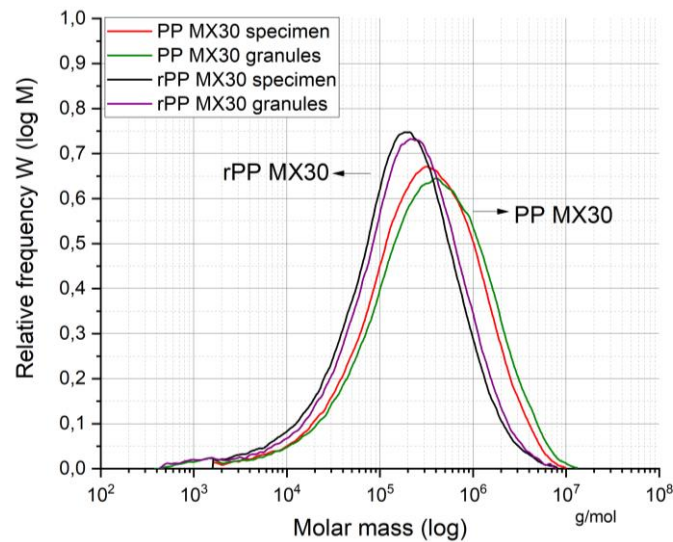


Figure 3: Molar mass distribution from GPC analysis of PP MX30 and rPP MX30 of granules and injection-moulded test specimens

Due to the lower molar mass of the rPP MX30, shorter molecular chains are present compared to the PP MX30. The lower molar mass of rPP MX30 is not only due to the previous component life and multiple processing of the recyclate. Rather, the difference is since the recyclate consists of a material mix in which each material contained in its basic formulation has a different molecular weight distribution. Compared to PP MX30, the narrower molecular weight distribution of rPP MX30 results in a narrower thermal softening range, lower shrinkage, lower residual stress, and a lower tendency to warp.

Using the Differential Scanning Calorimetry (DSC) analysis, the crystallinity of PP MX30 and rPP MX30 has been investigated. The change in crystallinity of both materials is to be specifically compared and how the ageing of 1,152 h at 120 °C in air affects the crystallinity. For this purpose, material samples were taken from unaged and aged specimens $K_t = 1.15$ (Figure 1) in the highly loaded region for the DSC analysis. The DSC analysis was carried out in accordance with DIN EN ISO 11357-1.

Figure 4 compares the calculated degrees of crystallisation as a function of the two heating and cooling processes. In general, the PP MX30 has a higher crystallinity than the rPP MX30. For PP MX30 and rPP MX30 a post-crystallisation due to ageing of 1,152 h at 120 °C can be seen. In the DSC investigations a glass transition temperature of $T_g = 6.5$ °C for PP MX30 and $T_g = 5$ °C for the rPP MX30 was determined.

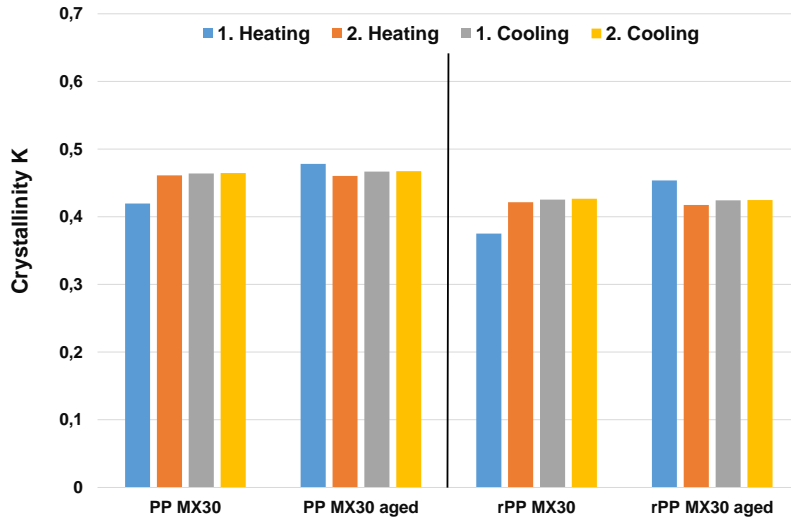


Figure 4: Comparison of the degrees of crystallisation from the DSC analysis of unaged / aged PP MX30 / rPP MX30

3.1 QUASISTATIC TENSION TESTS

The stress-strain curves of the quasi-static tensile tests have a yield point and thus close to a more ductile material behaviour according to DIN EN ISO 527-1. After reaching the yield point, the specimens constrict, whereby the stress decreases until the specimen breaks.

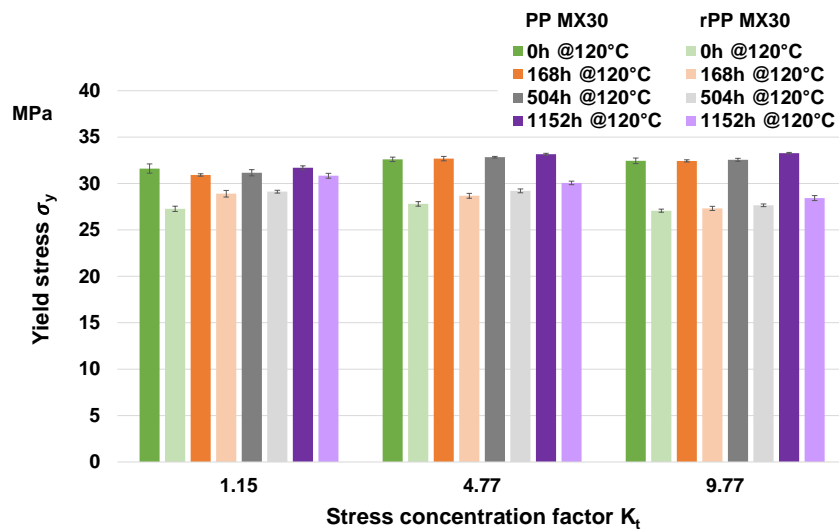


Figure 5: Yield stress under static load of PP MX30 / rPP MX30, different stress concentration factors and storage time

Figure 5 shows the results of the yield stress. In general, the yield stress of the rPP MX30 material is about 15 % lower than that of the PP MX30 for all influencing parameters investigated. For PP MX30 and rPP MX30 is investigated no influence of the stress concentration factor to the yield stress. This is

caused by the plasticization of the notch root, stress redistribution and the test surroundings above the glass transition temperature T_g (PP MX30 $T_g = 6.5$ °C, rPP MX30 $T_g = 5$ °C). Ageing at 120 °C in air causes no influence on the yield stress for all removal times for the PP MX30. For the rPP MX30, the yield stress increases slightly for higher removal times compared to the unaged state. After 1,152 h at 120 °C in air, the natural-coloured samples of PP MX30 have clearly discoloured. In contrast, no change in the colour impression of the black coloured rPP MX30 is visible.

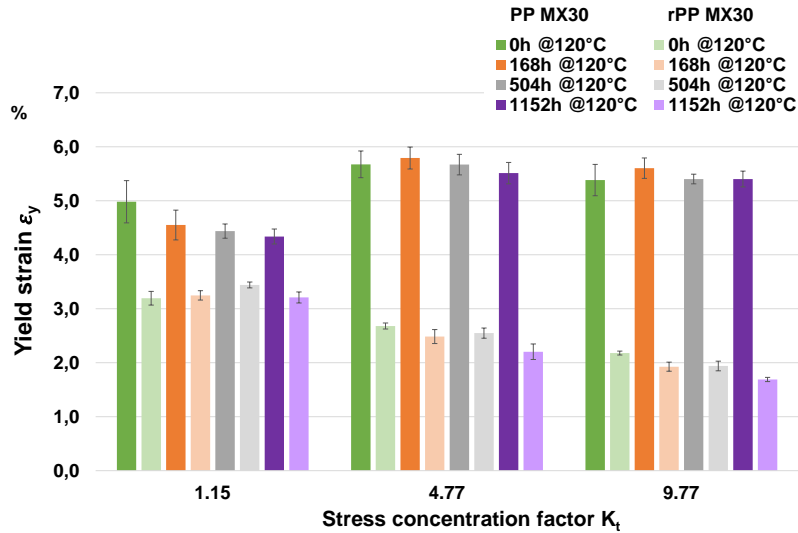


Figure 6: Yield strain under static load of PP MX30 / rPP MX30, different stress concentration factors and storage time

Figure 6 shows the yield strain determined in the static tension tests. The yield strain is significantly lower for the rPP MX30 than for the PP MX30. For the PP MX30, a decrease in the yield strain with increasing ageing time can only be seen for $K_t = 1.15$. For $K_t = 4.77$ and $K_t = 9.77$, only a slight decrease of the yield strain is detected. It is to note, that the yield strain decreases for the rPP MX30 for $K_t = 4.77$ and $K_t = 9.77$, but for PP MX30 the yield strain increases $K_t = 4.77$ and $K_t = 9.77$. This effect is caused for PP MX30 by plasticization in the notch root and the tough and ductile material behaviour. According to [10], thermo-oxidative ageing (polymer damage caused by heat in the presence of oxygen) reduces the elongation behaviour and causes yellowing effect of PP MX30.

The characteristic values of yield stress, yield strain and young's modulus are listed in

Table 2. There the data from the material data sheet and the data investigated in this study is compared. Different specimen geometries, specimen thicknesses and load velocities were used in the data sheet and this study. This different test properties cause a slight deviation, so that the results can not directly compared to each other. In addition, different processing parameters and the use of different testing machines can also lead to slightly different in the results.

Source	PP MX30		rPP MX30	
	Data sheet	Testing	Data sheet	Testing
Young's modulus E [N/mm ²]	3,400	3,870	-	3,396
Yield stress σ_y [N/mm ²]	33	31.6	33	27.3
Yield strain ϵ_y [%]	5	4.98	5	3.19

Table 2 Comparison of the data sheet- and investigated values of PP MX30 / rPP MX30

Based on the test results, there is higher young's modulus for PP MX30 than in the data sheet and the same yield stress and yield strain. For rPP MX30, a lower yield stress and yield strain was determined in the test than specified in the data sheet.

In summary, the lower yield stress, yield strain and young's modulus of the rPP MX30 is caused by to the lower molar mass and lower crystallinity. Furthermore, the dimensions of the added talc particles can also influence the mechanical properties to higher and lower values. The coating used on the talc particles also plays a decisive role. Post-crystallisation occurs due to ageing, which slightly increases the yield stress of rPP MX30. But contrary to the post-crystallization, a thermo-oxidative process takes place, which does not show any significant influence here (higher temperature or longer aging times would affect a more significant reduction of the properties). Furthermore, the additional manufacturing process and the uses in application cause a slight reduction of the material properties. As conclusion, the material properties of the recyclate material are not that bad as their general reputation.

3.2 FATIGUE TESTS

In the following, the fatigue tests carried out on PP MX30 and rPP MX30 are presented. As a general overview, the S/N-curves are shown in

Figure 7. In general, flat slopes k in the range of $k = 25$ to 45 and low scattering of the S/N-curves were investigated. The low scattering close to constant manufacturing process parameter and material properties. Flat slopes in the were also observed by [1], [2], [5], [6], [7], [11], [12] for talc reinforced PP. Additional creep tests close to the supposition, that failure of the material is more creep dominated. Further analysis to this effect is running.

There the same strength properties are determined as for static loading:

- no influence of the stress concentration factor for both materials at 23 °C and 80 °C
- no strength reducing effect of aging for 1152 h at 120 °C in air
- 10 to 15 % reduced strength for recyclat of the fatigue strength at 23 °C
- 30 to 40 % reduced strength for recyclat of the fatigue strength at 80 °C

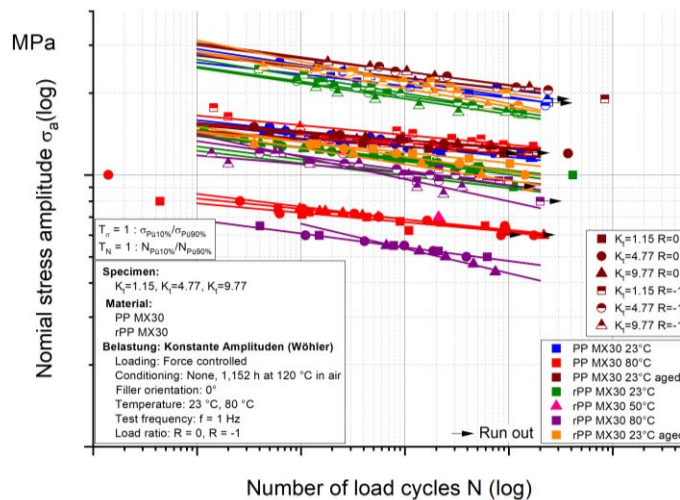


Figure 7: Overview of all determined S/N-curves of PP MX30 and rPP MX30, without weld line (left), with weld line (right)

3.3 LIFETIME ASSESSMENT

The material investigations for static and cyclic loading were used for a lifetime assessment of the door bearing pin (

Figure 8 mid) of a basis carrier of a dish washer machine (

Figure 8 left). The basis carrier has the dimension of 400 x 400 x 200 mm³ and a weight of 2 kg. So, the material substitution of virgin material by a recycled material has a big impact to a more sustainable dish washer. From the material side the recycling material must have nearly the same performance as the virgin material to bear the mechanical loadings. The door bearing pin is the heaviest loaded section of the basis carrier. During the life of 18 years, 15 opening and closing cycles each day cause 10⁵ load cycles. There the loading is considered as constant amplitude loading in tension (R = 0) at a temperature at 50 °C. The temperature of 50 °C can be caused by thermal radiation of an underfloor heating or placing next to an oven.

Based on the mechanical investigation on specimen level a notch stress concept [12] of the Highly Stressed Material Volume V₈₀ and Stress Gradient χ^* was assembled. Therefore, not the total rupture of the specimens was used, rather achieving a level of 10 % relative inelastic strain [13]. The relative inelastic strain will be calculated from the stress strain curve from the static tension test with Eq. 1.

$$\varepsilon_{inel}(\sigma) = e_{tot}(\sigma) - \frac{\sigma}{E} \quad (1)$$

By the means of numerical calculation, static and cyclic tests of the door opening pin, the notch stress concepts were validated.

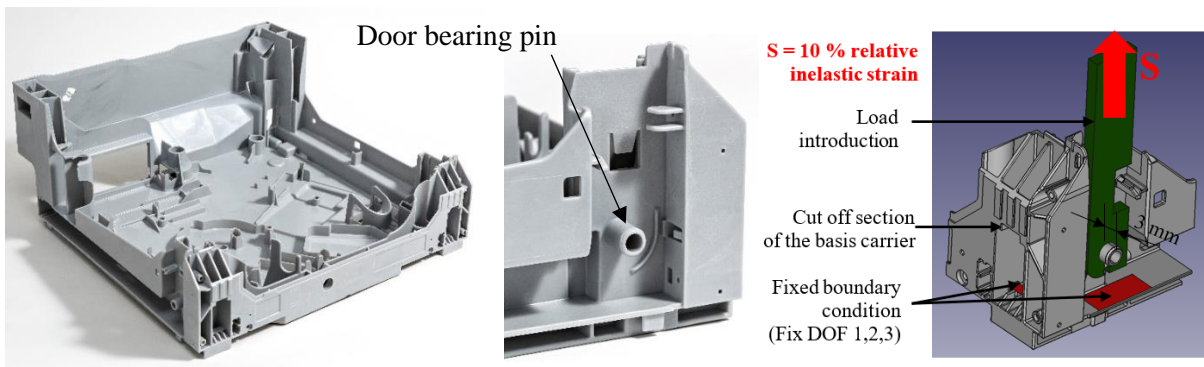


Figure 8: Basis carrier of the dish washer (left), door bearing pin (right)

For the static and cyclic testing of the door bearing pin a section was cut of the basis carrier. The load ratio was R = 0 and the temperature 23 °C and 50 °C. As failure of the door bearing pin is defined a relative inelastic strain of 10 %. This is equivalent to a plastic deformation of 0.4 mm in the notch root of the door bearing pin. Higher deformation causes a failure of the complete dishwasher in that way that the interior of a dishwasher is not sealed, or the door opening does not work comfortable. The plastic deformation at 10 % relative inelastic strain was determined in the static tension test and is displayed in Table 3.

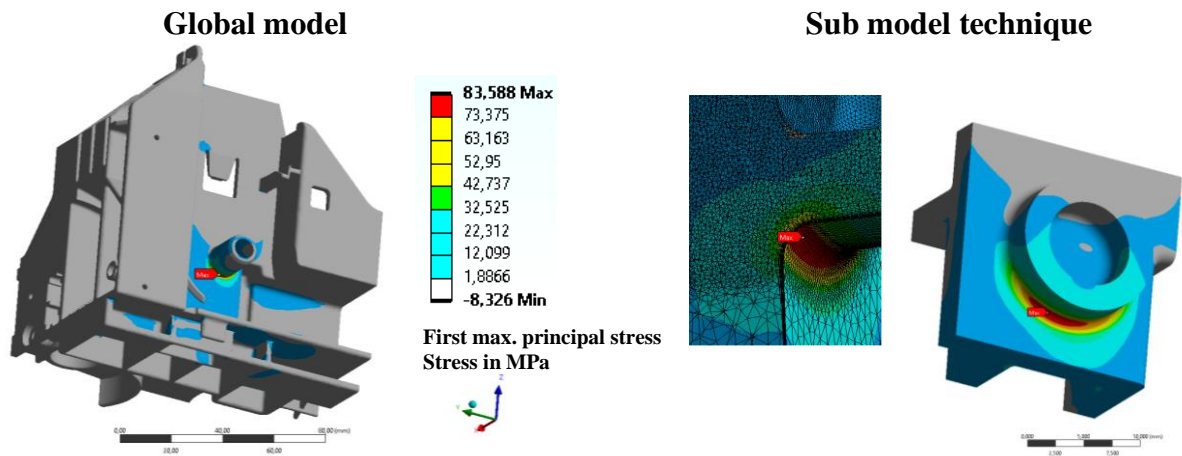


Figure 9: Results of the numerical calculation of the global model and the sub model

The numerical calculation model was meshed with tetramer elements second order. The boundary conditions were applied on the global model in the same way as in the static and cyclic part testing (Figure 8 right). As loading the deflection of $\epsilon_{inel} = 10\%$ was used. The young's modulus was determined by static tension tests and was chosen for $23\text{ }^\circ\text{C}$ ($E = 3,870$, $\nu = 0,32$) and for $50\text{ }^\circ\text{C}$ ($E = 1,600$, $\nu = 0,32$). The operating point with the deflection of $s = 0.178\text{ mm}$ at $50\text{ }^\circ\text{C}$ is caused by a spring with a force of $F = 317\text{ N}$ of the door opening mechanism. A global model- and a sub model was used for the calculation. There the deformation of the global model was mapped on the sub model of the door bearing pin. The sub model technique allows a local finer mesh size where exacter stress, deformation, V_{80} and χ^* can be determined. In

Table 3 are displayed the result of the numerical calculation. There the V_{80} and the χ^* is in the range of the stress concentration factor of $K_t = 4.77$.

Temperatur T [°C]	Deflection at $\epsilon_{inel} 10\%$ S_{tot} mm	First max. principal stress σ_{max} MPa	Highly stresses material volume V_{80} mm ³	Stress gradient χ^* 1/mm
23 (part testing)	0.38	83.62	0.498	2.016
50 (part testing)	0.385	38.91	0.485	2.084
50 (operating point)	0.173	17.50	0.437	2.016

Table 3: Results of the numerical calculation of the door bearing pin of the sub model

The results of the calculated test data are contrasted to material curves of the virgin material PP MX30 and the recycelat material rPP MX30. There the calculated test data must fit to the material curves of PP MX30 at $23\text{ }^\circ\text{C}$ and $50\text{ }^\circ\text{C}$. These results are displayed in

Figure 10. There is to mention, that the lifetime estimation fits perfect for PP MX30 at $23\text{ }^\circ\text{C}$. But for $50\text{ }^\circ\text{C}$ there is a lifetime overestimation. In the design process the local stresses (reduction of wall thickness) of the basis carrier will be adapted to the material curve. In application the component will fail much earlier than the expected lifetime of $N = 10^5$ load cycles.

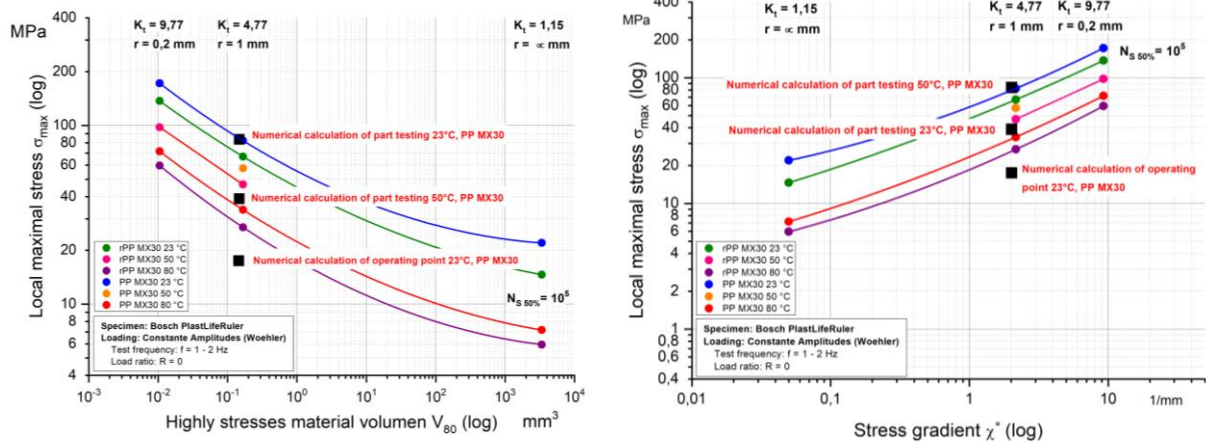


Figure 10: Results of the lifetime assessment by the notch stress concept highly stressed material volume V_{80} (left) and the stress gradient (right)

In the application are no premature failures of the door bearing pin occurring, so that the actual design is reliable for 18 years with 10^5 load cycles. It can be concluded from this, that recyclat material can substitute the virgin material.

The differences of the lifetime assessment to the experience of the application can be caused by several effects:

- The real temperatures in the field application are underneath of 50 °C.
- The real loading in application (time depended loading and relieve) does not correlate to the cyclic loading with constant amplitudes in specimen- and part testing.
- The linear elastic numerical calculation does not depict the complex viscos elastic material behaviour.
- The multi axial stress state in the door bearing pin does not correlate with stress state on specimen level.

All these issues can influence the quality of the lifetime assessment.

4. CONCLUSION

The virgin and the recyclat material are extensively investigated concerning their mechanical properties under static and cyclic loading. Additionally analytic investigations described the differences of the mechanical properties of the virgin- and the recyclat material. The lower mechanical properties of the recyclat material are not only caused by the additional manufacturing process and further use an application. Furthermore, differences in the filler dimension, filler distribution, filler sizing, the crystallinity, and the molar mass distribution cause differences of the material properties. Especially the molar mass distribution is primarily caused by the mixture of different waste stream. Furthermore, nothing is known about differences in the additives used in the virgin- and the recyclat material.

For unreinforced or particle reinforced polymers often occur high deformation, which lead to a failure at the component much earlier. Using the rupture as failure criterion often leads to an unrealistic lifetime assessment. In this study the concept of relative inelastic strain (here 10 %) works quite well as failure criterion for static and cyclic loading.

The notch stress concept of the Highly Stressed Material Volume V_{80} and the Stress Gradient χ^* can be used for the lifetime assessment of structural parts made of talc filled PP. This is demonstrated on the door bearing pin of the basis carrier of the dish washer. At the temperature of 23 °C there is a good

agreement between assets and actual lifetime. For higher temperatures further optimisation is necessary. The virgin material of the basis carrier can be substitute by the recyclat material. In this dish washer application with 6.000 tons material each year, 2.500 tons crude oil or 7.800 tones CO₂ can be saved each year. By using this recyclat material in the dish washer application a more sustainable and more resource efficient complete system can be achieved. This contributes to fulfil the sustainability development goals (SGD) of the United Nation especially to the goal 12 “Ensure sustainable consumption and production patterns”.

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