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SLOW CRACK GROWTH RESISTANCE OF NON-VIRGIN POLYMERS

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ABSTRACT

In order to improve the circular economy of polymers in terms of sustainability and reduced carbon footprint, the increased use of recyclates has become a topic of major importance. Due to their long design lifetimes, pipe applications represent an excellent opportunity to create added value to non-virgin polymers. In this context, special attention must be paid to the resistance of such materials against slow crack growth (SCG) which is the essential material property for structural long-term performance of 50 years or even more. The current paper demonstrated the applicability of the Cyclic Cracked Round Bar (CRB) Test according ISO 18489 for a quick and sensitive SCG characterization and material ranking of different non-virgin PE, PP and PVC. Moreover, by systematical mixing of PE and PP it is shown that already a small amount of polymeric impurity has a big impact on the long-term relevant resistance against SCG.

INTRODUCTION

Recycling of polymers has become a major economic, environmental, and social challenge for our current and future society [1]. Polymer products have conquered many areas of industrial and social life and they provide an outstanding contribution to the high living standard and technical development of our modern society. Since mid of the past century the global production of polymers has grown exponentially, reaching 311 million tons in 2014 after a twentyfold increase since 1960, and it is expected to reach up to 1.2 billion tons annually by 2050 [2]. Worldwide 69 % of all non-fiber plastic products ever made are based on polyethylene (PE, 36 %), polypropylene (PP, 21 %) and polyvinyl-chloride (PVC, 12 %) [3]. In 2016, the global production of polymeric materials counts 335 million tons, with a share of 50 % from Asia, 19 % from Europe and 18 % from the NAFTA region [4]. Within the 28 countries of the European Union (EU) plus Norway and Switzerland, from a total demand for polymer resins of 49.9 million tons, a share of 49.1 % was related to polyolefins (PE, PP) followed by 10 % of PVC [4]. The three main application segments have been identified with packaging (39.9 %), building & construction (19.7 %) and automotive (10 %) [4].

In order to improve the circular economy and the sustainable use, to reduce carbon footprint and to increase resource efficiency, polymers have become one of the five priority areas addressed in the "EU action plan for the Circular Economy" which sets a clear commitment to prepare strategies that focus on the challenges posed by polymers throughout the value chain and taking into account their entire life-cycle, such as reuse, recyclability, biodegradability, the presence of hazardous substances and concerns in marine litter [5]. The numbers mentioned above emphasize that, if any sector of industry wants to increase added value of polymer products, special attention must be addressed to the materials PE, PP and PVC. In fact, these materials are also globally dominating the products in building & construction and pipes, respectively. To support the current goal of the EU for the reduction of greenhouse gas emissions by 20 % until the year 2020, the European polymer pipe industry has voluntarily committed the target to use 250,000 tons of recycled materials per year by 2020 [8]. In this context, international platforms currently discuss possibilities of considering the use or extension of recycled materials for the manufacturing of polymer pipes. According to existing standards, the use of recycled materials is strictly forbidden for pressure piping systems. However, high potential has been identified in the field of non-pressure pipe applications. In compliance with relevant international and national product standards, in 2014 already more than 105'000 tons of polyolefin recyclate (PE and PP) and more than 87'000 tons of PVC recyclate was manufactured into non-pressure pipe systems such as sewage and stormwater applications [6].

During their designed service life of several decades, the structural reliability of piping systems is of essential importance. Premature failures in supply and sewage systems must be avoided as leaking or broken pipes may cause severe damage of infrastructural constructions or may result in environmental pollution. In contrast to the positive ecological and environmental considerations, it must be considered that the use of recycled polymers can be accompanied by a negative influence on mechanical properties. Especially for the slow crack growth (SCG) resistance, which is the prime property for the long-term failure resistance of stress loaded components, a significant reduction has been verified for recyclates within numerous studies [7–14]. For PE pressure pipe grades, the investigation of SCG properties is a fundamental basis for material classification and lifetime prediction. Based on comprehensive investigations during the past decades, the polymer physical mechanisms of SCG in PE are well understood today. Specific modifications in the molecular structure such as bimodal molecular mass distribution or the controlled implementation of short-chain branches resulted in a significant increase of the strength and SCG resistance [10,11,15–19].

However, for the characterization of SCG properties an increased resistance is connected to inconvenient long and expensive testing times. To address this issue, several accelerated fracture mechanics test methods were developed such as the Notched Pipe Test (NPT) [20,21], the Pennsylvania Edge-Notch Tensile Test (PENT) [22–24], the Full Notch Creep Tensile Test (FNCT) [25,26] and most recently the Strain Hardening Test [27–29] as well as the Cyclic Cracked Round Bar (CRB) Test [30–33]. All these tests allow a relative ranking based on SCG resistance.

Although the long-term stress loading situation of non-pressure pipes is different to pressure pipes, SCG has also been identified as one main criterion for the lifetime of non-pressure pipes. While for PE comprehensive experience is available with the above mentioned methods, for other relevant non-pressure pipe materials like PP or PVC a comparable knowledge does not exist. Therefore, in a first step the current paper is dedicated to investigate the general applicability and sensitivity of the Cyclic CRB Test for a SCG characterization of recycled PE, PP and PVC. In a second step, the Cyclic CRB Test will be used to analyze the effect of PP mixed in PE and vice versa in order to evaluate the impact of polyolefin impurities.

BACKGROUND

The use of non-virgin (reprocessed or recycled) polymers is of high interest for the polymer pipe industry. In general, the quantitative effects of non-virgin polymers on lifetime relevant long-term properties with focus of 50 to 100 years is not well known today. In one study severe premature damages of several pressure pipe systems have been investigated where the use of PE recyclate led to a dramatic decrease of the SCG resistance and pipes failed by brittle cracks within only few months after installation [34]. Further studies have shown that for PE and PP in general mechanical properties like tensile strength and strain at break reduce significantly with increasing content of recyclate [12]. With special focus on high density PE pipe grades, it was demonstrated that the use of recyclate most probably has negative effects on the thermo-oxidative aging resistance of the material [13]. A further study demonstrated that blending virgin high density PE pipe grade with only 10 % of virgin high density PE blow molding grade reduces the SCG failure time in PENT test dramatically by more than 95 % [14]. On the other hand, it was proven with PVC for cables that after two repeated remolding and processing steps neither the material composition nor the mechanical properties or the thermal stability changed significantly [35]. In a different study, rigid PVC was investigated after up to five re-extrusion steps and the results showed a slight increase in mechanical properties like elongation at break and tensile strength which was attributed to an increase in the degree of gelation. Moreover, after oven aging also a decrease in the activation energy was observed, indicating shorter aging resistance with increasing number of extrusion steps [36]. For pipe PVC it was demonstrated that even after eight regrinding steps, mechanical properties and thermal resistance have not changed [37]. The same study also concluded that with the quality criteria of K=67 recycled PVC may be used in core layers of multi-layer pipes up to 100 % [37]. While these studies show promising results for the use of recycled PVC, it has also been reported that PVC sewer pipes have failed by SCG after 34 years of service. While for this pipe again no material aging was detected, inherent defects based on foreign inclusions were identified as crack initiating impurities [38].

Basically the characterization of SCG properties in solid materials is possible with methods based on Linear Elastic Fracture Mechanics (LEFM). Initially developed for metals, a large body of scientific work has demonstrated that LEFM also provides reliable concepts for SCG characterization in polymers [8,17,32,39–44]. Especially for the purpose of material ranking of PE pipe grades these studies have also shown that accelerated results on specimen scale are in good agreement with internal pipe pressure tests. To meet the demand for quicker testing of SCG in modern PE pipe grades the Cyclic CRB Test was developed and finally standardized in ISO 18489 [30–33,45,46]. Moreover, it has already been demonstrated that LEFM concepts in general are also suitable to characterize PP and PVC [47–51]. First studies also confirm that the Cyclic CRB Test can be used to reliably characterize the SCG resistance of these materials in relatively short testing times and at ambient temperatures [52,53].

EXPERIMENTAL

For the basic evaluation of the applicability and sensitivity of the Cyclic CRB Test, 24 different non-virgin materials, 7 x PE (labeled as nvPE), 12 x PP (nvPP) and 5 x PVC-U (nvPVC), reprocessed or recycled, are summarized in the first part of this study. All materials are commercially available grades which are recommended for extrusion. For confidentiality reasons, no further material data will be provided within this paper. For each polymer, at least one representative virgin pipe grade was analyzed in order to create reference data. The second part of this study evaluates the effect of polymeric impurities which is often observed in polyolefin recyclates. Therefore, commercially available virgin PE80 and PP-B for pipe extrusion were systematically blended with different mixing ratios. The manufacturing of all CRB specimens was realized according ISO 18489 [33].

All materials were characterized in terms of density, differential scanning calorimetry (DSC), melt flow rate (MFR) and shear viscosity. As the main intention of the current paper is about the applicability and sensitivity of the Cyclic CRB Tests, results of the morphological characterization will not be discussed in detail here. The cyclic CRB Tests were conducted on a servo-hydraulic closed-loop testing system of the type MTS Table Top (MTS Systems GmbH, Berlin, GER) according ISO 18489 [33]. The force controlled cyclic load was applied with a frequency of f=10 Hz for PE and PVC and f=5 Hz in case of PP [51,54]. All tests were conducted at ambient temperatures of T=23 °C.

DISCUSSION

The following figures are adopted from [53] and summarize the results of the Cyclic CRB Tests for the investigated materials in which the failure cycle numbers N_f is shown as a function of the applied stress load $\Delta\sigma_0$ in log-log scale. The results for PE in Fig. 1,a are additionally compared to typical failure regions of commercially available PE80, PE100 and PE100-RC pipe grades [31]. Apart from the virgin grades PE100 and PE100-RC, all nvPE were tested at somewhat lower testing loads than recommended in ISO 18489. The reference pipe grade materials PE100 and PE100-RC virgin are in compliance with the expected SCG resistance, but the failure curves of all nvPE are clearly below the known performance of PE pipe grades. While nvPE-1, nvPE-3 and nvPE-7 almost match the region of PE80, the SCG resistance of the other materials is shorter by almost two magnitudes in time compared to the virgin PE100. Although the non-virgin grades show significantly shorter SCG resistance, the slope of all failure curves remains parallel indicating that the basic polymer physics of SCG is not changing. The testing time for a single CRB specimen of the non-virgin grades was between some hours and up to three days.

Representative for all non-virgin PE grades, Fig. 1,b shows a scanning electron microscopy (SEM) image of the brittle CRB fracture surface of material nvPE-2. Beside the typical fibrils, which are a result of the discontinuous crack growth characteristics of PE and which confirm the polymer physical characteristics of SCG, also spherical inclusions can be recognized. It was confirmed by DSC that a significant amount of PP exists in the recycling grades. Due to the immiscible characteristics of PE and PP, a phase separation can be noticed in Fig. 1,b. Each of these spheres creates a material inhomogeneity and defect, respectively, which finally reduces the SCG resistance of the material. A relatively high MFR also indicates that PE of lower molecular mass for other applications such as blow molding or injection molding is embedded, which also affects the SCG resistance negatively.

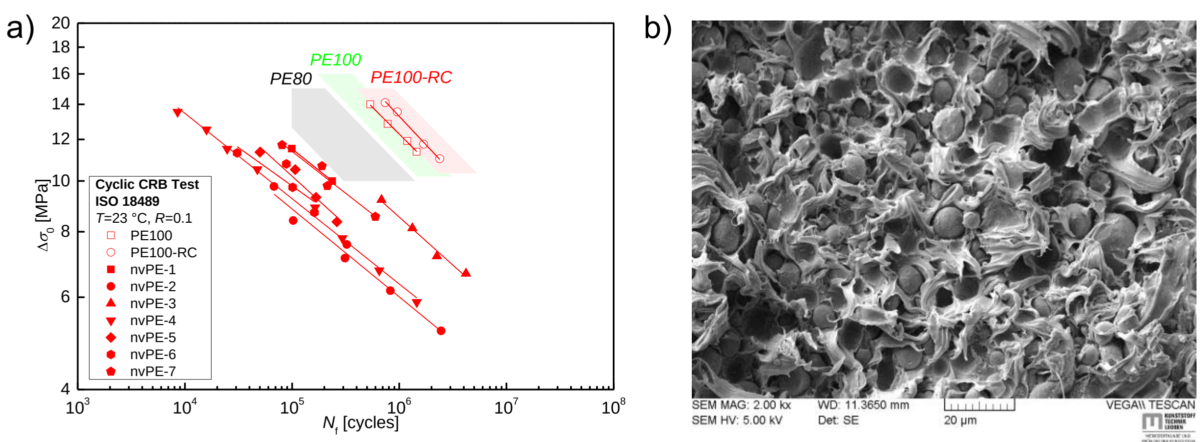

Fig. 1: a) Results of Cyclic CRB Test of virgin and non-virgin PE including reference failure regions [31]; b) Representative SEM fracture surface image of nvPE-2.

The CRB failure curves of the PP grades are shown in Fig. 2,a. Again the reference material PP-B virgin shows the best performance indicated by the longest tests. All non-virgin grades show clearly shorter N_f as a result of lower SCG resistance. Similar to the investigated PE's, the slope of the nvPP failure curves remain almost parallel. The difference in brittle failure curves compared to the reference PP-B virgin is significant between one and two magnitudes in time. Except nvPP-3, all nvPP's were tested within the recommended loading range of ISO 18489. However, due to the reduced test frequency of *f*=5 Hz [51,54] the testing times until failure increased up to approx. one week per specimen for the better performing materials. The SCG performance of PP at loads above the recommended values for PE was not in focus of this study, but it can be expected that an optimization of the test protocol will lead to a further reduction in testing times.

Representative for all nvPP, a SEM image of a CRB fracture surface of nvPP-2 is shown in Fig 2,b. Beside the existence of fibrillation as a confirmation for SCG, an inorganic impurity of about 50 µm in size can be noticed. The DSC curves also confirmed PE fractions inside the material. Moreover a relatively high MFR was measured which indicates that other grades than for extrusion are included into the material. All these effects reduce the SCG resistance in PP significantly.

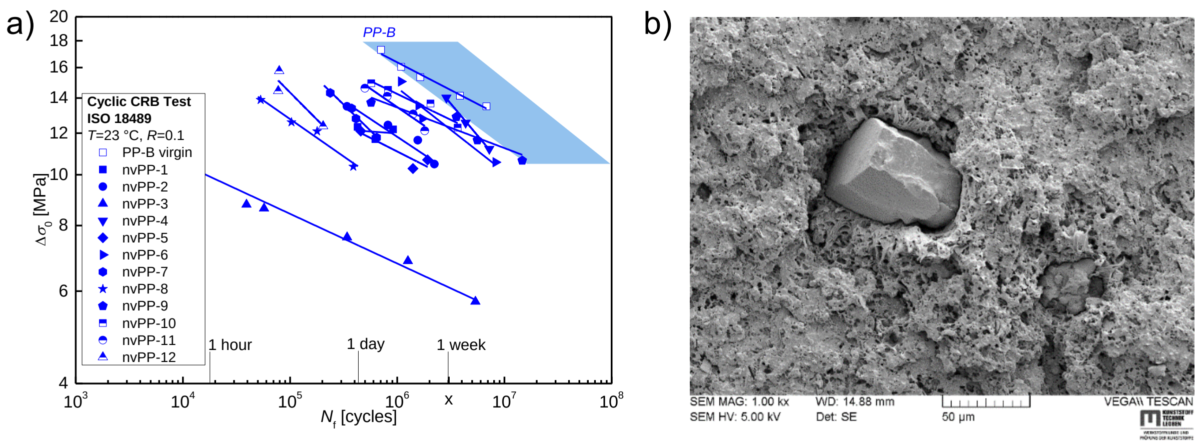

Fig. 2: a) Results of Cyclic CRB Test of virgin and non-virgin PP including reference failure region; b) Representative SEM fracture surface image of nvPP-2.

The results of the CRB Tests on the PVC grades are summarized in Fig 3,a. To get a better overview about the failure performance of PVC, compared to ISO 18489 the loading range has been extended to higher and lower $\Delta\sigma_0$. A potentially increase of the specimen temperature due to hysteretic heating was additionally evaluated with an IR sensor to be insignificant. Like for the polyolefins, the failure curves show a linear correlation in which N_f increases with decreasing $\Delta\sigma_0$. The highest SCG resistance was measured for the reference PVC-U virgin, while all nvPCV grades show lower N_f. The failure curves of all materials were almost parallel to each other and testing times were below one day per specimen.

Figure 3,b shows a SEM image of the brittle failure region on a CRB fracture surface of nvPVC-4. The fibrillary structure is a clear confirmation for the evidence of SCG. No indications for inorganic impurities can be recognized.

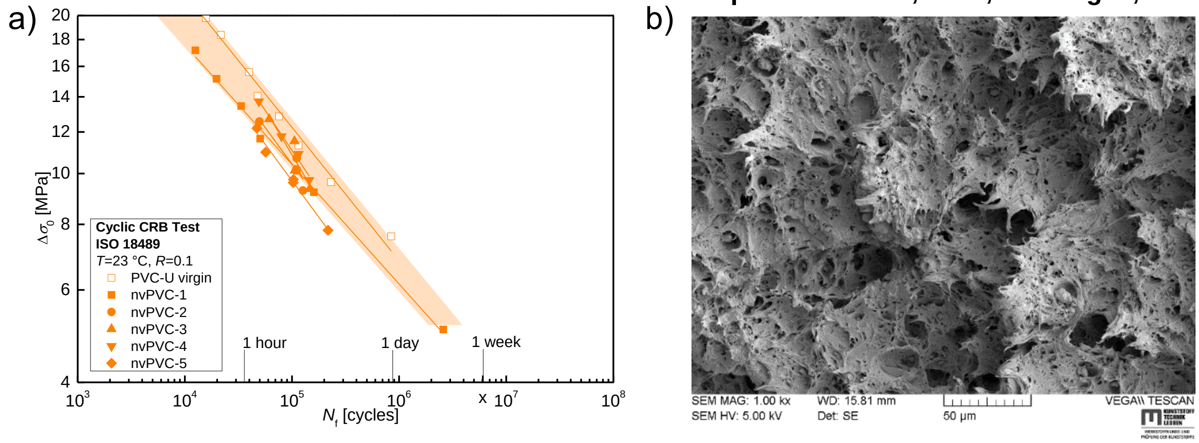

Fig. 3: a) Results of Cyclic CRB Test of virgin and non-virgin PVC including reference failure region; b) Representative SEM fracture surface image of nvPVC-4.

To get an overview about the relative difference in SCG resistance between virgin and non-virgin polymers, the N_f of the failure curves at a testing load of $\Delta\sigma_0$=12.5 MPa were determined and normalized to the respective reference virgin grade. For PE the value of PE100 was selected as reference. The results in Fig 4 clearly show the lower SCG resistance for all non-virgin grades. While the relative lower SCG resistance for the investigated nvPE and nvPP grades is about one to two magnitudes, for the nvPVC grades it is significantly smaller. However, it must be emphasized that the presented results of this study are based on a limited number of PVC and they do not allow a valid conclusion yet that PVC is generally less sensitive to SCG reduction in non-virgin conditions. To answer this question, additional systematically studies with controlled material variations from virgin to non-virgin are necessary. However, it can be concluded that the CRB Test produces real SCG in all materials without the need of increased testing temperature or any additional surfactant, and that the generated data allow a clear and quantitative differentiation of the SCG resistance for all polymers.

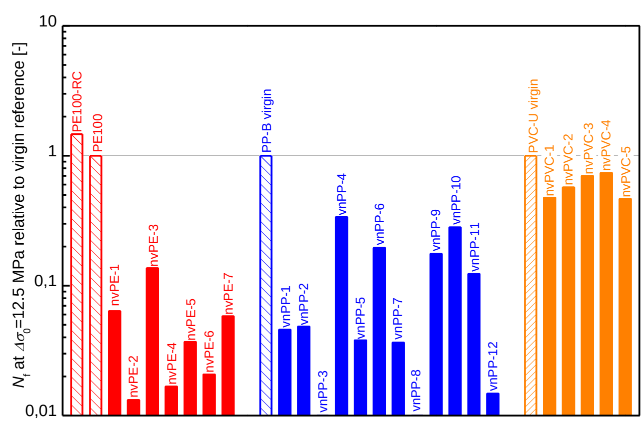

Fig. 4: Relative failure cycle numbers N_f of virgin and non-virgin PE, PP and PVC at a testing load of $\Delta\sigma$0=12.5 MPa normalized to the respective reference virgin grade.

The investigations discussed in Fig. 1 and Fig. 2 showed that polymeric impurities might have a significant effect on the SCG resistance of non-virgin polyolefins. Therefore, the second part of the current paper is dedicated to a systematical study of the effect of PE in PP and vice versa [55]. The CRB failure curves in Fig. 5,a show that already with a PP-B content of only 5 %, the SCG resistance of PE80 is reduced significantly. A continuous further decrease of N_f was measured with increasing PP-B content of 10 % and 20 %. Clearly lowest SCG resistance, accompanied by an increased scatter of the data, was observed for the mixture of 50 % PE80 and 50 % PP-B. A very similar characteristics is shown in Fig. 5,b for mixing PE80 in PP-B. While the pure PP-B has clearly the highest SCG resistance, failure cycle numbers significantly decrease with increasing PE80 content.

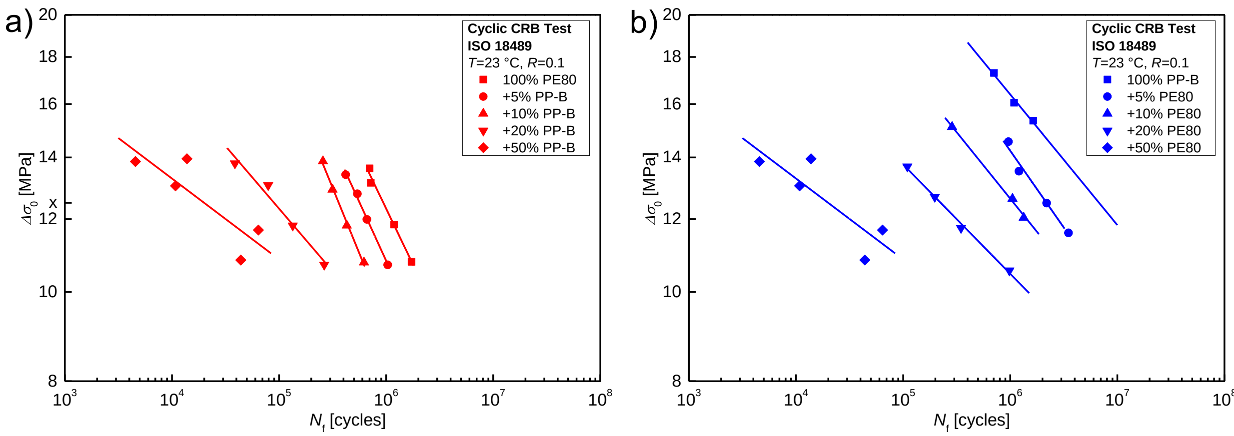

Fig. 5: Results of Cyclic CRB Test of PE/PP mixtures: a) PE80 with increasing content of PP-B; b) PP-B with increasing content of PE80 [55].

The relative change of SCG resistance with increasing mixing content is summarized in Fig. 6. While the reduction of N_f for PE80 mixed with 5 % PP-B is about 38 % of the initial value of the clean material, a mixing content of 5 % PE80 in PP-B already reduces the SCG resistance by more than 68 %. These trends continue with increasing mixing content, where a mixing content of 10 % reduces the SCG resistance by 61 % for PE80 and 85 % for PP-B. Generally the data show that PP-B has a higher relative sensitivity on polyolefin impurity than PE80.

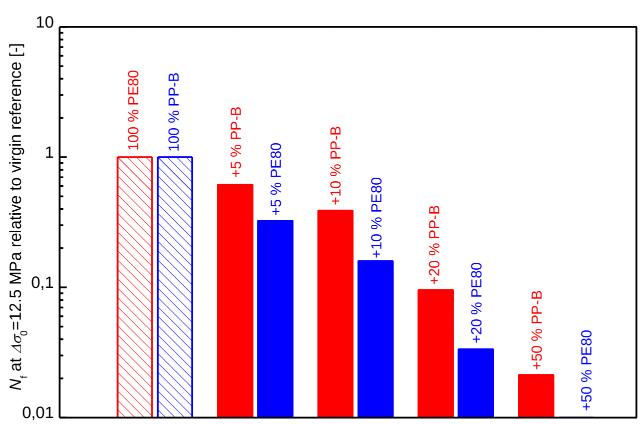

Fig. 6: Relative failure cycle numbers N_f of PE/PP mixtures at a testing load of $\Delta\sigma_0$=12.5 MPa normalized to the respective reference virgin grade.

CONCLUSIONS

The Cyclic CRB Test according ISO 18489 is a direct fracture mechanics test to analyze the SCG resistance of thermoplastic materials. The results of the present paper confirm the general applicability of this test, not only to PE, but also to PP and PVC. Moreover it was demonstrated that this test shows a high sensibility on non-virgin polymers. Especially for non-virgin PE and PP, the SCG resistance is significantly lower by about one to two magnitudes when compared to virgin pipe grades. On the other hand, PVC seem to be less sensible in terms of SCG reduction. However, for PVC it must be emphasized that only a few number of experimental data is available yet and further studies are required to generalize this conclusion.

For PE and PP, impurities with the respective other material has been identified as one main reason for SCG reduction. Therefore a systematical study with mixed PE and PP was conducted. It was demonstrated that already a small amount of 5 % of mixing content results in a considerable reduction in the SCG resistance. The results indicate that, compared to the initial values of the virgin materials, the relative impact of PE in PP is higher than the impact of PP in PE.

For plastic pipes in non-pressure applications, a minimum lifetime of 100 years will be required in future. For economic and ecological reasons, the additional use of non-virgin materials will become more and more important. The results of this paper emphasize the importance of a careful consideration of SCG properties of non-virgin materials when used for non-pressure pipes. Compared to pressure pipes, non-pressure pipes are exposed to clearly lower long-term loading conditions. Therefore, also non-virgin materials may have the capability to reach lifetimes of one century, even with demonstrated lower SCG resistance. However, to reliably confirm the structural long-term integrity, additional future work should focus on fracture mechanical lifetime predictions of non-pressure pipes made of non-virgin materials.

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