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Novel solar thermal collector systems in polymer design – Part 5: Fatigue characterization of engineering PA grades for pressurized integrated storage collectors

Joerg Fischer^{a*}, Patrick R. Bradler^a, Mathias Schlaeger^a,
Gernot M. Wallner^a, Reinhold W. Lang^a

^aUniversity of Linz - Institute of Polymeric Materials and Testing, Altenberger Strasse 69, 4040 Linz, Austria

Abstract

A novel aging test method considering the superimposed mechanical and environmental (temperature and environmental medium) loads representative for pressurized integrated storage collectors (ICS) is described. Engineering polyamide (PA) grades with short glass fiber (GF) reinforcement, which are of high relevance for endcaps of steel-pipe ICS absorbers or all-polymeric absorber/storage-tanks, are characterized on a specimen level. Therefore, specific test devices and test arrangements for fracture mechanics specimens with or without weld-line are implemented on an electro-dynamic test machine. Fatigue crack growth kinetics data are obtained by conducting cyclic mechanical loads under various environmental testing conditions. The experimental results of two glass-fiber reinforced PA grades, using compact type specimens, performed at two different temperatures (23 °C and 80 °C) and in two environmental media (air and water), are compared in terms of crack growth kinetics. Moreover, the influence of welding on the crack growth kinetics for one PA grade is shown. For all specimens (unwelded and welded) the fatigue crack growth rates are enhanced in water compared to air. In welded specimens the fatigue crack growth resistance is significantly reduced compared to unwelded specimens.

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* Corresponding author. Tel.: +43-732-2468-6617; fax: +43-732-2468-6613.
E-mail address: joerg.fischer@jku.at

1. Introduction

A specific objective of the EC-FP7 project SCOOP (Solar Collectors made of Polymers) was to develop and implement advanced testing methods for the evaluation of the long-term behavior of polymeric materials for solar collectors under service relevant loading conditions. For example, in single-loop integrated storage collectors (e.g., SolCrafte® by KIOTO Clear Energy AG, Austria) the absorber tank is pressurized with maximum internal pressures of 6 bar at ambient temperature and 4 bar at 90 °C [1]. In another associated research work [2,3], conventional (global) aging was used to describe the long-term behavior and to assess the lifetime. It is well known that the mechanical behavior of polymeric materials strongly depends on the mechanical (e.g., loading rate, monotonic vs. static vs. cyclic loading) and environmental (e.g., temperature, gaseous environment, liquid environment) loading conditions [4-12]. Further research work [4,6-8,13,14], revealed that compared to mechanical tests of environmentally preconditioned specimens in air, a different mechanical material response is usually found when superimposing mechanical and environmental loads.

Hence, a main objective of the present research work was to implement a fracture mechanics based in-situ testing approach in which the superimposed mechanical and environmental (temperature and environmental medium) loads are considered properly. For that purpose, a setup developed in a previous research project [15,16] is used. To evaluate the materials crack growth behavior under superimposed environmental loading cyclic fracture mechanics tests are performed. Based on the service relevant loading conditions of integrated storage collectors the superimposed mechanical-environmental tests are conducted at 23 °C and 80 °C in the environmental media air and water.

2. Background

The application of linear-elastic fracture mechanics to characterize the fatigue crack growth behavior in plastics is a well-established procedure [10,17-20]. For a quasi-brittle failure, three different regions are observable and commonly shown in a double logarithmic plot (see Fig. 1). In region I (threshold regime), fatigue crack growth rates da/dN rapidly diminish as the stress intensity factor range ΔK_I is decreased. Conversely, fatigue crack growth rates in region II (stable crack growth regime) frequently exhibit a power law relationship in terms of ΔK of the type

$$\frac{da}{dN} = A \cdot \Delta K_I^m \quad (1)$$

where the parameters A and m are material specific constants depending on the loading conditions. Finally, in region III (unstable crack growth regime), crack growth rates accelerate with increasing ΔK_I as the corresponding ΔK_I value in a cycle approaches the critical stress intensity factor ΔK_{IC} of the material, leading to ultimate failure. When comparing different material grades or various test conditions (e.g., tests performed in air and water, respectively), enhanced crack growth resistance is indicated by a shift of the fatigue crack growth curve towards higher ΔK_I values. For practical considerations also of advantage is a decrease in the slope of the fatigue crack growth curve in region II [10,21,22].

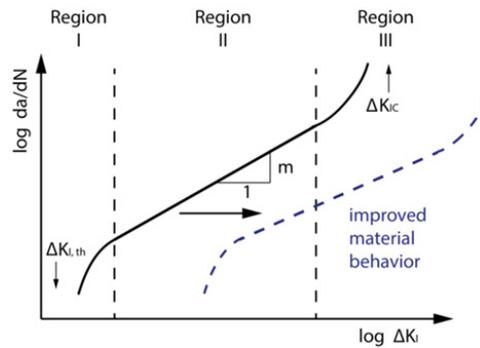


Fig. 1. Dependency of the crack growth rate da/dN on the stress intensity factor ΔK_i .

3. Methodology and experimental

3.1. Materials and specimen preparation

The tests were conducted with two commercial engineering polyamide (PA) grades varying in their glass fiber content. Such composites are of high relevance for integrated storage collectors (e.g., for the endcap of ICS absorbers based on steel pipes or absorber/storage tank components made from engineering plastics). An overview of the investigated polymeric matrix materials along with information to material designation, glass fiber content, commercial grade name and manufacturer is provided in Table 1.

Table 1. Materials

Material designation	Matrix polymer type	Glass fiber content	Commercial grade name	Manufacturer
PA66-GF30	Polyamide 66	30 w%	SCHULAMID® 66 GF 30	A. Schulmann GmbH, Germany
PA66-GF50	Polyamide 66	50 w%	Leona 90W50	Asahi Kasei Corp., Belgium

For the fracture mechanic tests compact-type (CT) specimens (see Fig. 2a) were used. To characterize the influence of vibration welding on the crack growth behavior within welded all-polymeric absorber/storage-tanks tests were performed with unwelded and welded CT specimens. The unwelded CT specimens were manufactured out of injection-molded plates via milling. Additionally, a razor notch was cut into the specimens prior to fatigue test. Figure 2b shows the two arrangements for the sample extraction out of the injection molded plates with the notch direction (and thus the crack growth direction) parallel and transverse to the melt flow direction, respectively.

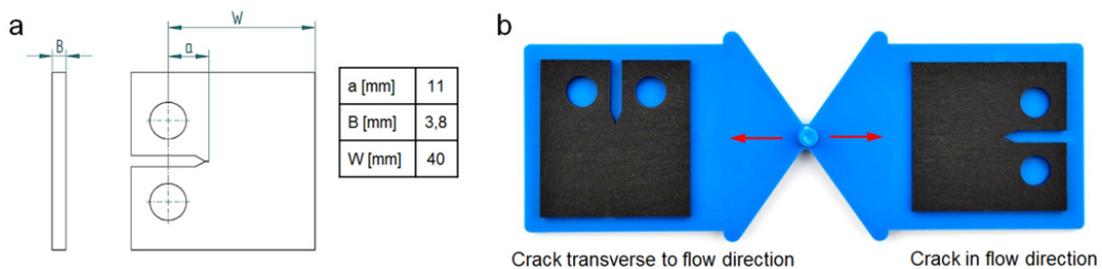


Fig. 2. (a) Compact type (CT) specimen; (b) Illustration of sample extraction for unwelded specimens transverse and in flow direction.

To obtain the welded CT specimens, the injection molded plates were cut and subsequently vibration welded. During milling focus was given to position the notch base on the welding line to ensure crack propagation (fracture) in the welding zone (see Fig. 3).

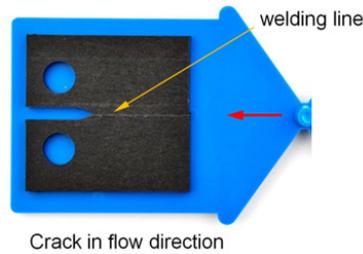


Fig. 3. Illustration of sample extraction for welded specimens in flow direction.

3.2. Fatigue testing, data reduction and fracture surface analysis

The crack growth behavior was characterized in air or deionized water (in the following referred to as H₂O) environment at temperatures of 23 °C and 80 °C, whereby the specimens tested in H₂O were pre-aged in water for 7 days at 80 °C to ensure water-saturation of both polyamide grades. An overview of the test program is shown in Table 2. These tests were performed as in-situ tests using a media containment device developed by Schoeffl et al. [15,16]. To detect the crack length of the specimen the setup was equipped with a specific adapted camera and software system.

Table 2. Test program

Sample	Unwelded	Unwelded	Unwelded	Unwelded	Welded	Welded
	Air – 23°C	H ₂ O – 23°C	Air – 80°C	H ₂ O – 80°C	Air – 23°C	H ₂ O – 80°C
PA66-GF30	x	x	x	x	x	x
PA66-GF50	x			x		

The fatigue tests were done on the dynamic testing machine ElectroPuls E3000 (Instron; Norwood, USA) with a fixed frequency of 5 Hz under sinusoidal loading. For all tests the R -ratio (i.e., the ratio between minimum to maximum stress intensity factor) was set to $R=0.1$. Depending on material and environmental conditions, the initial stress intensity factor was chosen appropriately. The calculation of the stress intensity factor ΔK_I with the unit MPa*m^{0.5} was done with Eq. 2 [23], where a is the crack length, B the thickness of the specimen, ΔF the difference between the maximum and minimum applied force of the sinusoidal loading and W the width of the specimen. The equation for the shape factor $f(a/W)$ can be calculated according to Eq. 3 [23] and describes a non-dimensional correction function.

$$\Delta K_I = \frac{\Delta F}{B \cdot \sqrt{W}} \cdot f\left(\frac{a}{W}\right) \quad (2)$$

$$f\left(\frac{a}{W}\right) = \frac{\left(2 + \frac{a}{W}\right)}{\left(1 - \frac{a}{W}\right)^{3/2}} \cdot \left(0.886 + 4.64 \cdot \left(\frac{a}{W}\right) - 13.32 \cdot \left(\frac{a}{W}\right)^2 + 14.72 \cdot \left(\frac{a}{W}\right)^3 - 5.6 \cdot \left(\frac{a}{W}\right)^4\right) \quad (3)$$

To obtain microscopic images of the fracture surfaces, the confocal laser microscope LEXT OLS 4000 (Olympus Austria GmbH, A) was used. The fracture surfaces were analyzed by layered scanning using a laser with a wavelength of 405 nm and an objective with a magnification of 20x.

4. Results and discussion

Injection molding of glass fiber reinforced polymeric materials leads to a multi-layer structure considering the fiber orientation. Due to the shear flow in the mold, on the product surfaces, fibers tend to align in flow direction, whereas in the product central or core layer, fibers are mainly oriented transverse to flow direction [19,24-26]. Fiber orientation dependent fatigue crack growth data of unwelded PA66-GF30 CT specimens are depicted in Fig. 4. In good agreement with previous papers on the fatigue crack growth behavior in short glass fiber reinforced materials [18,24,25,27] it was found that the fatigue crack growth resistance of the transverse-to-flow direction extracted CT specimen was clearly higher than that of the in-flow direction extracted CT specimen. This observation reflects the fiber orientation distribution in these specimens which is dominated by the fibers oriented in-flow direction in surface near regions of the specimen, thus being aligned transverse to the notch and crack direction in the specimen with the improved crack growth behavior. In terms of service performance, it is however the least resistant crack growth path which may control the service life. Hence, all further investigations were performed with specimens extracted with the notch in-flow direction.

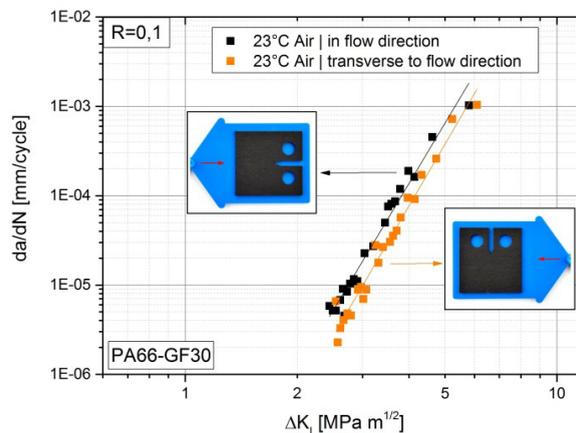


Fig. 4. Fatigue crack growth curves for PA66-GF30 specimens tested in and transverse flow direction.

To assess the influence of different environmental conditions, the effect of testing PA66-GF30 at temperatures of 23 °C and 80 °C in air or water environment is illustrated in Fig. 5. Comparing the crack growth data resulting from tests in air environment, for the CT specimen tested at elevated temperature, a lower crack growth resistance was detected. This effect is reflected by a shift of the crack growth curve to lower ΔK_I values at similar crack growth rates. The crack growth curves obtained by superimposed testing in H₂O show steeper slopes than those detected in air but tend to converge at lower crack growth rates. It should be noted, however, that for a given ΔK_I level, larger values for the crack opening displacement were observed for specimens tested in water. This is clearly related to the plasticization effect in polyamides associated with enhanced moisture uptake when submerged in water, which acts to reduce the material modulus and hence the specimen stiffness [28,29]. The results obtained exhibiting water and in particular hot water as the more critical environment compared to hot air are in good agreement with conventional (no superposition of loading conditions) aging data results obtained in previous research work [2,3].

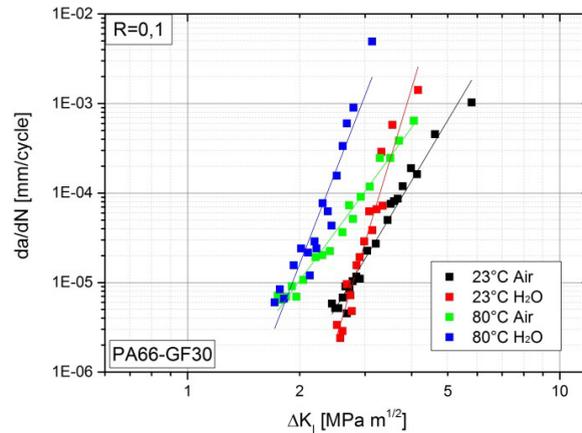


Fig. 5. Fatigue crack growth kinetics for PA66-GF30 specimens tested at various environmental conditions.

The effect of the short glass fiber content on fatigue crack growth behavior in PA66 is compared in Fig. 6 illustrating data for PA66-GF30 and PA66-GF50. The comparison is provided for two environmental conditions, 23 °C in air and 80 °C in water. For both conditions, the fatigue crack growth resistance of PA66-GF50 is higher than the one of PA66-GF30. However, while the crack growth resistance at 23 °C in air is distinctly different, the data at 80 °C in water clearly converge for both materials at lower crack growth rates.

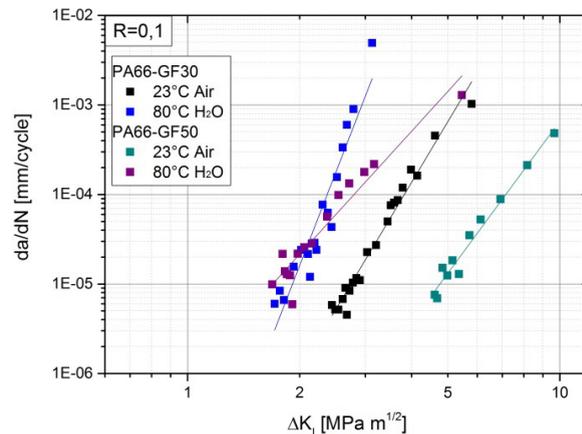


Fig. 6. Fatigue crack growth kinetics for PA66-GF30 and PA66-GF50 specimens tested at various environmental conditions.

As mentioned above, the two parts of absorber/storage-tanks of all-polymeric single-loop integrated storage collectors are commonly welded together. For a better knowledge of the influence of a weld-line on the crack growth rate, investigations were performed with PA66-GF30 at two selected environmental conditions utilizing and comparing specimens without and with weld-lines. The corresponding fatigue crack growth curves are depicted in Fig. 7 and show a significantly lower crack growth resistance for the weld-line specimens. This behavior is related to the unfavorable fiber orientation in the fracture plane of the weld-line which is generated by the welding process, i.e. fibers are predominantly oriented in the weld-line plane. In Fig. 8 the dominating fiber orientation distributions across the specimen thickness are illustrated by the fracture surface images of specimens without (Fig. 8a) and with (Fig. 8b) a weld-line.

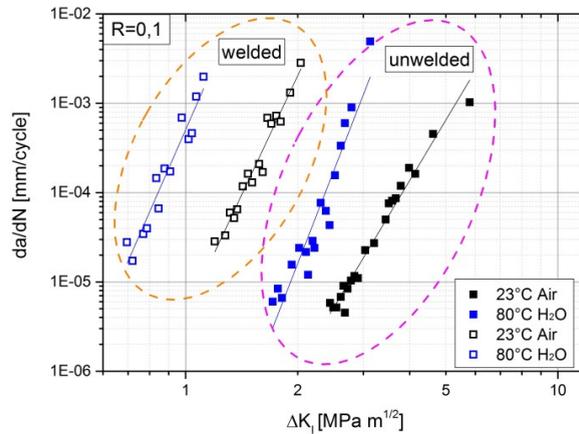


Fig. 7. Fatigue crack growth kinetics for unwelded and welded PA66-GF30 specimens tested at various environmental conditions.

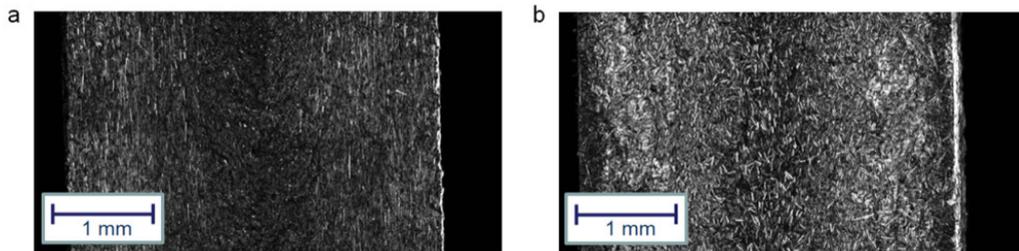


Fig. 8. (a) Fracture surface of an unwelded PA66-GF30 specimen; (b) fracture surface of a welded PA66-GF30 specimen.

5. Summary and outlook

To characterize the fatigue crack growth resistance of engineering polyamide (PA) grades for pressurized integrated storage collectors, two short glass fiber reinforced PA grades differing in glass fiber content (PA66-GF30 and PA66-GF50) were investigated. Fatigue experiments were performed at two temperatures (23 °C and 80 °C) in two environmental media (air and water). Besides injection molded and machined compact-type (CT) specimens also CT specimens with a weld-line in the crack propagation (fracture) plane were tested. As to the glass fiber content, PA66-GF50 revealed improved fatigue crack growth resistance over PA66-GF30. Due to the fact that vibration welding led to a different fiber orientation in the fracture plane, with fibers oriented predominantly in the fracture plane, welded specimens were found to be inferior in their crack growth resistance, as expected. Furthermore, in terms of environmental effects elevated temperatures (80 °C) and a water environment were found to adversely affect the crack growth resistance of the PA grades investigated.

It is believed that superimposed mechanical-environmental testing is appropriate to achieve accelerated information on the long-term behavior of polymers under service-relevant loading conditions. Furthermore, using this method allows for a quick ranking of various potential material candidates for pressurized integrated storage collectors.

While the present paper had its focus on the characterization of two different polyamide grades with superimposed mechanical-environmental test techniques, future research work will focus on the characterization of the environmental dependent crack growth kinetics of further polyamide grades with varying glass fiber contents. Moreover, it is our aim to develop a fracture mechanics based methodology that allows for a quantitative lifetime assessment of single-loop integrated storage collectors under true service conditions utilizing a proper set of fatigue crack growth data.

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References

- [1] Solcrafe. Product brochure. Website of Solcrafe, KIOTO Clear Energy AG, St.Veit/Glan (AT); 2014
<http://www.solcrafe.com/sites/all/themes/solcrafe/files/Solcrafe-brochure-EN.pdf> (downloaded 13.12.2014).
- [2] Geretschlaeger KJ. Characterization of pigmented and reinforced polymeric materials for solar energy technologies. Institute of Polymeric Materials and Testing, University of Linz (AT). Dissertation; 2015.
- [3] Geretschlaeger KJ, Wallner GM. Aging characteristics of glass-fibre reinforced polyamide in hot water and air. *Polymer Composites* 2015; submitted.
- [4] DeCoste JB, Malm FS, Wallder VT. Cracking of Stressed Polyethylene: Effect of Chemical Environment. *IndEng Chem* 1951;43:117-21.
- [5] Menges G, Puetz D, Gitschner W. Creep-Properties of Thermoplastics and Duroplastics with regard to mechanical, thermal and chemical (corrosive) influence. *Materialwissenschaft und Werkstofftechnik* 1979;10(6):200-12.
- [6] Lang RW, Stern A, Doerner G. Applicability and limitations of current lifetime prediction models for thermoplastics pipes under internal pressure. *Die angewandte makromolekulare Chemie* 1997;247(1):131-45.
- [7] Stern A, Novotny M, Lang RW. Creep crack growth testing of plastics - I. test configurations and test system design. *Polymer Testing* 1998;17(6):403-22.
- [8] Stern A, Asanger F, Lang RW. Creep crack growth testing of plastics - II. data acquisition, data reduction and experimental results. *Polymer Testing* 1998;17(6):423-41.
- [9] Krishnaswamy RK. Analysis of ductile and brittle failures from creep rupture testing of high-density polyethylen (HDPE) pipes. *Polymer* 2005;56(25):11664-72.
- [10] Lang RW, Pinter G, Balika W. Konzept zur Nachweisführung für Nutzungsdauer und Sicherheit von PE-Druckrohren bei beliebiger Einbausituation, *3R international* 2005;44(1-2):33-41.
- [11] ISO 175: Plastics – Methods of test for the determination of the effects of immersion in liquid chemicals.
- [12] ISO 16770: Plastics – Determination of environmental stress cracking (ESC) of polyethylene – Full notch creep test (FNCT).
- [13] Brown R. (Ed.). *Handbook of Polymer Testing: Physical Methods*. Boca Raton (US): CRC Press; 1999.
- [14] Altstädt V, Kausch HH (Ed.). *Intrinsic Molecular Mobility and Toughness of Polymers II*. Berlin, Heidelberg (DE): Springer; 2005. p. 105-52
- [15] Schoeffl PF. Influence of Liquid Hydrocarbon Environments on High Density Polyethylene or Oil and Gas Applications. Institute of Polymeric Materials and Testing, University of Linz (AT). Dissertation; 2014.
- [16] Schoeffl PF, Bradler PR, Lang RW. Yielding and crack growth testing of polymers under severe liquid media conditions. *Polymer Testing* 2014;40:225-33.
- [17] Hertzberg RW, Manson JA. *Fatigue of engineering plastics*. New York (US): Academic Press; 1980.
- [18] Lang RW, Manson JA, Hertzberg RW. Effect of short glass fibers and particulate fillers on fatigue crack propagation in polyamides. *Polymer Engineering & Science* 1982;22(15):982-7.
- [19] Lang RW. Applicability of linear elastic fracture mechanics to fatigue in Polymers and short-fiber composites. Lehigh University, Bethlehem (US). Dissertation; 1984.
- [20] Hertzberg RW. *Deformation and fracture mechanics of engineering materials*. Toronto (CA): Wiley; 1996.
- [21] Paris P, Erdogan F. A Critical Analysis of Crack Propagation Laws. *Journal of Fluids Engineering* 1963;85(4):528-33.
- [22] Haager M. Bruchmechanische Methoden zur beschleunigten Charakterisierung des langsamen Risswachstums von Polyethylen-Rohrwerkstoffen. Institute of Material Science and Testing of Plastics, University of Leoben (AT). Dissertation; 2006.
- [23] Gross D, Seelig T. *Bruchmechanik: Mit einer Einführung in die Mikromechanik*. Heidelberg (DE): Springer Verlag; 2001.
- [24] Lang RW, Manson JA, Hertzberg RW. Fatigue Crack Propagation in Short-Fiber-Reinforced Plastics. *Advances in Chemistry Series* 206, Washington DC (US): American Chemical Society; 1984. p. 261-77.
- [25] Lang RW, Manson JA, Hertzberg RW. Mechanisms of fatigue fracture in short glass fibre-reinforced polymers. *Journal of Materials Science* 1987;22(11):4015-30.
- [26] Schlaeger M. Einfluss der Umgebungsbedingungen auf die mechanischen Eigenschaften von kurz-glasfaserverstärktem PA 66 unter monotoner und zyklischer Belastung. Institute of Polymeric Materials and Testing, University of Linz (AT). Master Thesis; 2015.
- [27] Lang RW, Manson JA, Hertzberg RW. Effects of Fibrous and Particulate Reinforcements on Fatigue Crack Propagation in Polyamides. *Organic Coatings and Plastics Chemistry* 45, New York (US): American Chemical Society; 1981. p. 778-83.
- [28] Hahn MT, Hertzberg RW, Manson JA, Lang RW, Bretz PE. Effect of test frequency and water content on localized crack-tip heating in nylon-6.6. *Polymer* 1982;23:1675-9.
- [29] Lang RW. *Polymerwerkstoffe 1*. Institute of Polymeric Materials and Testing, University of Linz (AT). Lecture Notes; 2015.