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**CHANGES IN THE STRUCTURE AND MECHANICAL
PROPERTIES OF POLYETHYLENE TEREPHTHALATE AS A
RESULT OF RECYCLING**

THESIS BOOK OF PHD DISSERTATION

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The review and the defense report of the doctoral dissertation can be viewed at the Dean's Office of the Faculty of Mechanical Engineering of the Budapest University of Technology and Economics

1. Introduction

The recycling of plastic waste attracts more and more attention, due to the increasing use of plastic, and social and legislative pressure. Polyethylene terephthalate (PET) receives special emphasis, as a disposable packaging material of drinks with a short life cycle. A great deal of PET waste is generated each year. Recycled, secondary PET can be a good alternative of the original PET and also other, engineering plastics.

PET is typically used in fiber and bottle production. It started to be widely used as a packaging material for soft drinks and mineral water in the 1970s, thanks to the good mechanical and physical properties and low density of the material. Its use is still increasing. The use of PET grew from 57 kt in 2009 to 108 kt in 2017 in Hungary.

Mineral water and soft drink bottles have a short life cycle and their use generates a large amount of waste. Sustainable development requires this waste to be handled properly—this is laid down in various directives of the EU. Typical uses of recycled PET are fibers (textile industry), plastic sheets and strips, technical parts, and nowadays more and more bottles are also produced from recycled PET.

A serious problem with products extruded or injection molded from recycled PET is the deterioration of mechanical properties, especially a high degree of brittleness. This is connected to the morphological properties of the material (e.g. molecular chain length, orientation, crystal structure), which can be affected by several parameters, such as the polyvinyl chloride (PVC) content of the material, drying, or the processing technology.

During the various recycling technologies, the molecular length of the polymer typically decreases, which is even more prominent after repeated recycling. Contaminants in the PET (e.g. PVC or adhesives) make it even worse. A decrease in molecular length not only makes processing more difficult but also impairs mechanical properties.

According to the latest research, PET is not considered a two-phase (crystalline and amorphous) system, but a three-phase (crystalline, rigid amorphous and mobile amorphous) system. This theory states that the amorphous phase consists of two parts of different behavior: the rigid amorphous part is molecules immediately next to the crystalline part, therefore they are less mobile than the molecules of the mobile amorphous part (the traditional amorphous part). The properties of the two amorphous parts and their effect on the mechanical properties of the product are different.

In my PhD, I analyze recycled (once and several times) PET and products made from recycled PET with special recycling technologies, with special attention to mechanical and morphological properties. Finding the connection between the properties of the products and

its morphological structure can facilitate the production of better quality products from recycled plastic, and the application of suitable technologies and the right technological parameters.

2. Literature review, goals

In the literature review, I reviewed the processes during the recycling of PET, and their effect on the crystallization characteristics of PET. The crystalline structure forming during processing largely determines the mechanical properties of the product therefore the processes need to be studied in detail when mechanical properties are examined and interpreted. Much research focuses on the crystallization processes of PET, such as, crystallization during cooling from melt, cold crystallization or crystallization during various forms of heat treatment. These publications detail the processes and compare morphological results with mechanical properties, but only a few explain certain mechanical characteristics (e.g. tensile strength) with the changes in the crystalline structure presented in detail. Research does not usually focus on the changing of mechanical properties over time. Among testing methods, modulated differential scanning calorimetry and fracture tests deserve special attention. The former can provide detailed information about the process of crystallization, while the latter can characterize toughness, one of the most critical properties of recycled PET.

In the second part, I review solid-phase polycondensation, which is a popular way of increasing molecular weight. The articles detail the process and the effects of numerous parameters (temperature, reaction time, contaminations, initial molecular weight) on the efficiency of the reaction. Most research focus on the effects some other characteristics, in addition to the parameters of the reaction, on the achievable IV value, or use a material with an already increased IV value, and examine the product. A large part of the research present morphological characteristics—mechanical tests are rarer. I have not found literature that focuses on mechanical tests on a sample immediately after an SSP reaction—the publications examine samples that underwent an SSP reaction but also another processing step after it.

In the third part, I examined PET foaming. Almost all literature emphasize that the foaming of recycled PET in a traditional way is close to impossible due to the low molecular weight, therefore some kind of procedure is necessary to increase molecular weight. This is usually an SSP reaction or the application of a chain extender. In most publications, PET is batch foamed in high pressure reactor, or extrusion physical foaming is applied. Little research focuses on foaming during injection molding.

After the literature review, I set the following goals:

- to examine the crystalline structure and mechanical characteristics of recycled PET and products injection molded from it, as a function of time since manufacturing, and to present in detail how the crystalline structure forms and changes in time,
- to investigate the effect of the SSP reaction on the crystalline structure and mechanical properties – by developing a method with which the SSP reaction can be modeled in the lab and the mechanical properties of products immediately after the reaction can be examined,
- to produce products by chemical foaming in injection molding, during which I examine the effect of injection molding parameters and the various foaming agents on the foam structure and the morphology.

3. Materials and measuring methods

3.1. Materials used

Polymers

In the experiments, I processed polymers of various IV values:

- Polymer of increased IV value (“Increased IV”). It is produced by Polymetrix (formerly Bühler, Switzerland). The company increased the IV value using their own SSP procedure. I measured the IV value: 0.92 ± 0.10 dl/g.
- Original PET (“Orig”): NeopET80 produced by Neogroup (Lithuania). It is widely used in plastic bottle manufacturing. I measured its IV value: 0.78 ± 0.10 dl/g.
- Bottle regrind (“regrind”) – cleaned PET bottle regrind, made in the PET bottle recycling plant of Jász-Plasztik Kft. I measured its IV value: 0.75 ± 0.20 dl/g.
- Low IV polymer (“Low IV”): pellets made from PET bottle regrind by extrusion without drying. The bottles were selectively collected by the public. I measured the IV value: 0.46 ± 0.20 dl/g.

Additives

During the nucleating tests, I used Cloisite 116 (Byk, Germany) montmorillonite (without surface treatment). Its density is 340 g/dm^3 , its characteristic particle size (d_{50}) $<15 \text{ }\mu\text{m}$.

I used two kinds of foaming agents: Tracel IM 3170 MS (Tramaco, Germany) exothermic foaming agents with 120 ml/g gas generation and a decomposition temperature of $170 \text{ }^\circ\text{C}$, and Tracel IM 7200 (Tramaco, Germany) endotherm with 50 ml/g gas generation and a decomposition temperature of $135\text{-}220 \text{ }^\circ\text{C}$. These data are from the technical datasheet.

3.2. Test methods

Determining moisture content

I measured the moisture content of the materials with an FMX HydroTracer (Aboni, Germany) moisture meter at Jász-Plasztik Kft. During measurement, the moisture content of the material evaporates, and then reacts with a reagent (calcium hydride). As a result, hydrogen is generated, which is detected by a gas sensor and the equipment calculates the moisture content using this.

Measuring intrinsic viscosity

I measured the IV value of the samples with a RPV-1 (PSL Rheotek, United Kingdom) computer-controlled optical sensor viscosimeter. The device automatically determines the IV value of the samples, according to the ASTM D4603 standard. The solvent is the 60:40 mixture of phenol and 1,1,2,2- tetrachloroethane (Sigma-Aldrich, USA), test temperature is 30 °C, concentration is 0.5 g/dl. The IV values were calculated with the Billmeyer equation (4):

$$IV = \frac{\eta_{rel} - 3 + 3 \ln(\eta_{rel})}{4c}, \quad (4)$$

where IV [dl/g] is intrinsic viscosity, η_{rel} [dl/g] is relative viscosity, and c [g/dl] is the concentration of the solution.

DSC and MDSC tests

I performed the DSC and MDSC tests using a DSC Q2000 (TA Instruments, USA) device. I determined the proportion of the crystalline, rigid amorphous and mobile amorphous phases based on the three-phase morphological model. I used a heat-cool-heat cycle between 30-300 °C, with a heating and cooling rate of 10 °C/min. I produced the registered curves with Universal Analysis 2000 (TA Instruments, version 4.7A) software. For the peak separation procedure, I used the Calisto 1.47 (Setaram, France) calorimetry software at Jász-Plasztik Kft.

KRF was calculated with equation (5):

$$KRF = \frac{\Delta h_m - \sum \Delta h_{cc}}{\Delta h_m^0} 100, \quad (5)$$

where KRF [%] is the crystalline fraction of the sample, Δh_m [J/g] is the specific heat of fusion of the sample, $\sum \Delta h_{cc}$ [J/g] is the heat the sample released during cold crystallization, and Δh_m^0 is the specific heat of fusion of 100% crystalline PET (140,1 J/g).

With the specific heat changes at the glass transition temperature, the amount of MAF can be determined in the samples with equation (6):

$$MAF = \frac{\Delta c_p}{\Delta c_p^0} 100, \quad (6)$$

where MAF [%] is the proportion of the mobile amorphous phase, Δc_p [J/(gK)] is the measured specific heat change, Δc_p^0 [J/(gK)] is the specific heat change of fully amorphous PET at the glass transition temperature (0,405 J/(gK)).

RAF was calculated with equation (7):

$$RAF = 100 - MAF - KRF, \quad (7)$$

where RAF [%] is the proportion of the rigid amorphous phase in the sample, MAF [%] is the proportion of the mobile amorphous phase in the sample, and KRF [%] is the crystalline fraction in the sample.

I performed the MDSC tests between 30-300 °C, at a heating rate of 2 °C/min, with one heating. The temperature modulation was sinusoidal, with an amplitude of $\pm 0,318$ °C and a period of 60 s. The mass of the specimens was 5-8 mg.

I examined the following peaks:

- the cold crystallization peak between 100-130 °C (HK) and the crystalline melting peak between 210-270 °C (KO),
- the melting peak between 130-270 °C (RKO) on the reversible heat flow curve,
- the melting peak between 210-270 °C (NRKO) on the non-reversible heat flow curve, and the crystallization peak between 130-270 °C (NRK).

To determine the area of the peaks, I used a tangential sigmoid baseline. On the non-reversible heat flow curve, the crystallization and crystalline melting peaks overlapped – I decomposed them with the Calisto 1.47 (Setaram, France) calorimetry software at Jász-Plasztik Kft.

Isothermal crystallization

I measured isothermal crystallization with a DSC Q2000 (TA Instruments, USA) device. During the test, I heated the samples to 300 °C at a heating rate of 10 °C/min, then I cooled them at the highest available cooling rate to the isothermal crystallization temperatures: 200, 205, 210, 215 and 220 °C. I used the (8) Avrami formula to evaluate the crystallization peaks:

$$X_t = 1 - e^{-K_t t^n}, \quad (8)$$

where X_t [-] is relative crystallinity as a function of crystallization time, K_t [1/min] is the crystallization speed constant, t [min] is the time of crystallization, n [-] is the Avrami exponent.

I determined the crystallization half-time with equation (9):

$$t_{1/2} = \left(\frac{\ln 2}{K_t} \right)^{1/n}, \quad (9)$$

where $t_{1/2}$ [min] is the crystallization half-time, K_t [1/min] is the crystallization speed constant, and n [-] is the Avrami exponent.

Non-isothermal crystallization

Non-isothermal crystallization tests were performed with a DSC Q2000 (TA Instruments, USA) device. I heated the samples to 300 °C at a heating rate of 10 °C/min, then cooled them at a cooling rate of 4, 8, 16 and 30 °C/min to 20 °C. I evaluated the results with the (10) Ozawa formula:

$$X_t = 1 - e^{-\frac{K_t}{\Phi^{m_o}}}, \quad (10)$$

where X_t [-] is relative crystallinity as a function of crystallization, K_t [1/min] is the crystallization speed constant, Φ [°C/min] is the cooling rate and, m_o [-] is the Ozawa exponent.

Small- and wide angle X-ray diffraction tests

Small angle X-ray scattering (SAXS) tests were performed with CREDO equipment developed and built by Biological Nanochemistry Research Group, at the Hungarian Academy of Sciences, Research Centre for Natural Sciences. Two sample-detector distances were applied: 462 mm and 1516 mm. Scattering curves from two measurements were merged. Scattering vector (q) range was 0,07-6 1/nm. Thickness of investigated samples were 1 mm. Long period was determined from SAXS curves with equation (11):

$$L_{C+A} = \frac{2\pi}{q}, \quad (11)$$

where L_{C+A} [nm] is long period, q [1/nm] scattering vector.

Wide angle X-ray diffraction (WAXD) measurements were performed with X'pert Pro MPD (PANalytical Bv., Netherlands), at the Department of Inorganic and Analytical Chemistry, at the Budapest University of Technology and Economics. Cu K α radiation, Ni filter and X'Celerator detector was used, in a 2θ range of 4-44°. Thickness of investigated samples were 1 mm. Crystalline fraction of investigated samples were determined by dividing the difference of the integrated area of amorphous-crystalline curves by the integrated area of crystalline curves.

Based on results of WAXD and SAXS measurements, sizes of the crystallites were determined by equation (12):

$$L_C = L_{C+A} x_C, \quad (12)$$

where L_C [nm] is the size of the crystallite, L_{C+A} [nm] is long period, x_C [-] is the crystalline fraction determined by WAXD measurement.

DMA test

The DMA tests were carried out with a DMA25 (Metravib, France) dynamic mechanical analyzer. I performed the tests in the temperature range 0-150 °C, at a heating rate of, 3 °C/min, in tensile mode. The frequency was 1 Hz, excitation was 0.1% strain excitation (symmetrically from the clamping point), and clamping length was 6 mm. The cross section of the specimens was irregular due to the type of manufacturing (the material flow became thinner after it exited the extruder), so I determined the cross section with a Stemi 508 (Zeiss, Germany) stereo microscope. The device handles rectangular or circular cross section specimens, therefore I determined an equivalent diameter and set it in the device. A disk with the equivalent diameter had the same area as the actual measured cross section of the specimens. In the evaluation, I used the storage modulus and loss factor curves.

Determining the fragility index

In order to determine the fragility index, I performed DMA tests with a DMA25 (Metravib, France) device, in shear layout. To determine the glass transition temperature, I heated the samples between 0-150 °C, at a heating rate of 2 °C/min. The frequency was 1 Hz, while the amplitude was 15 µm.

I made the measurements for the master curve at isothermal temperatures between 50-101 °C with 3 °C steps. The frequency range was 0.01-10 Hz with 5 measurement points in every decade, in a logarithmic scale. I created the master curve with the software of the device (Dynatest 6.90).

I calculated the fragility index from the C_1 and C_2 constants that belong to the T_g temperature, using equation (13):

$$m = \frac{T_g \cdot C_1}{C_2}, \quad (13)$$

where m is the fragility index [-], T_g is the glass transition temperature determined with a DMA measurement [K], C_1 and C_2 are the constants of the WLF equation at T_g temperature [-].

I analyzed the Angell diagrams with the values of shift factor determined at various temperatures. I determined the eltolási tényező from the master curves of the DMA measurements. I plotted and evaluated the diagrams in the range $T_g/T = 0,95-1$.

Tensile test

I performed the tensile tests with an Instron 3369 (Instron, USA) universal testing system. I determined the initial tensile modulus of elasticity between strains of 0.05-0.25%, at

a crosshead speed of 1 mm/min, using a video extensometer. I measured tensile strength at a crosshead speed of 10 mm/min. I repeated every measurement 5 times.

Flexural test

I performed the three-point bending tests with a Zwick Z020 universal tester (Zwick, Germany). Supporting pin distance was 28 mm, and crosshead speed was 10 mm/min.

Impact test

In the preliminary tests, I determined the impact strength of the samples with a Charpy impact test. It turned out that due to the geometry of the samples (1 mm and 2 mm thick sheets) this method cannot be applied well, therefore in subsequent experiments I performed Izod impact tests.

I performed the Charpy tests with a Resil Impactor (Ceast, Italy) impact tester on notched specimens, with a 2 J hammer, at an impact speed of 2.9 m/s, at room temperature. I repeated each test (with identical samples) ten times.

I performed the Izod tests with a Zwick 5113.100/01 (Zwick, Germany) impact tester on notched specimens with a 2.75 J hammer, and an impact speed of 3.47 m/s, at room temperature. I repeated each test (with identical samples) ten times.

Density test

I determined the density of the foamed samples with an AS 60/220.R2 (Radwag, Poland) analytical balance, by immersion into ethanol. Density was determined with equation (14):

$$\rho_m = \rho_{et} m_l / (m_l - m_{et}), \quad (14)$$

where ρ_m [g/cm³] is the density of the sample, ρ_{et} [g/cm³] is the density of ethanol at the temperature of the test, m_l [g] is the mass of the sample in air, m_{et} [g] is the mass of the sample in ethanol.

Determining the viscosity curve with a capillary rheometer

I determined the flow curves with a Ceast SR20 (Ceast, Italy) capillary rheometer. I performed the measurements at 260, 270, 280 and 290 °C, in a shear rate range of 100-8000 1/s. The capillaries used had a diameter of 2 mm, and their length is 10 and 15 mm. The program calculates the result from the two parallel measurements with Rabinowich correction.

Optical microscopy and CT test

I performed the non-destructive test of the foamed samples using a Modular CT (YXLON, USA) industrial CT device at the Department of Materials Science and Technology of Audi Hungaria Faculty of Automotive Engineering at the Széchenyi István University in

Győr. Resolution was 0.027 mm, the voltage was 200 kV, the current 0,1 mA. The detector was a flat panel, without a filter, with an integration time of 700 ms. The structure of the samples was reconstructed from 1440 images. I used a computer algorithm to evaluate the individual cross-sectional images. The algorithm was developed by the researchers of the Department of Materials Science and Technology of Audi Hungaria Faculty of Automotive Engineering at the Széchenyi István University in Győr. I determined the porosity of the samples from the outer surface of the samples, using CT images. The cell diameters were calculated by the VGStudio MAX 2.2 software.

4. Summary

I investigated the recycling of poly(ethylene terephthalate), with special attention to the effect of recycling on the morphological and mechanical properties. The results presented in my dissertation can be divided into three parts. In the first part, I examined the effect of recycling on the crystalline structure, tensile properties (strength and modulus) and toughness of PET. As extrusion and injection molding are the two most widely used technologies in recycling, I examined their effect on degradation during manufacturing and the morphological and mechanical properties of the product. I also examined how these properties changed over time. In the case of products injection molded from recycled PET, a post-crystallization process was observable within 30 days from manufacturing. This process occurred at room temperature without outside influence. During this process, the crystallinity of the samples increased by 110% compared to crystallinity immediately after manufacturing. The DSC tests proved that the rigid amorphous parts in the samples converted, and this increased the proportion of KRF. In PET, cold crystallization can characteristically occur as a result of heat treatment, and between T_g and the melting point, a crystallization and crystalline melting process occurs as well. These processes can be decomposed with an MDSC test, and analyzed. Irrespective of manufacturing parameters, the initial crystallinity of the sample can be attributed to a distinct melting process and this is also true for post-crystallization, too. In the case of recycled PET, toughness is an especially important mechanical property since the molecular weight of recycled PET is typically lower than the original PET, which can greatly increase rigidity, thus decreasing impact strength. In my experiments, I used the fragility index (determined with a DMA test) in addition to fracture energy, to characterize the toughness of the samples. The results indicate that the two indicators of toughness correlated well. This relationship makes it possible to characterize the toughness of specimens, in whose case traditional impact tests cannot be used (e.g. films).

In the second part of my research, I investigated the effect of the solid phase polycondensation (SSP) reaction on the crystalline structure, and dynamic mechanical properties. I developed a unique method to do this. I performed an SSP reaction in a lab with various parameters, and on various materials with different initial IV values, then I examined the effectiveness of the reaction and the increase in IV. Then I examined the morphological properties of the specimens by DSC, and the mechanical properties by DMA. The morphological tests yielded further data about the crystallization of PET: as a result of high temperature crystallization, a crystallite population appears which can be observed as a separate population, based on its size, up to 220 °C, then above this temperature its size is the same as the size of initial crystallization, and as temperature increases, both populations grow at the same time. Mechanical properties changed as a result of the change in morphological characteristics. The storage modulus of the samples increased as a result of increasing KRF proportion and the conversion of rigid amorphous-mobile amorphous parts.

In the third part of my research, I produced chemically foamed products from recycled PET. I examined the effect of exothermic and endothermic foaming agents on the foam structure. The various foaming agents locally change the temperature of the melt as they decompose, which affects the foam structure across the cross section. As mold temperature increases, porosity changes: an endothermic foaming agent increases it, while an exothermic foaming agent decreases it.

The test results indicate that in the case of recycled PET, the processing technologies and the preparation and processing parameters greatly affect the properties of the product. It is important that the product has the right morphology because morphology affects mechanical properties. For this, however, the behavior of the material during recycling needs to be known. My research results provide help in a more detailed description and deeper understanding of this behavior. This makes it possible to use recycled PET in areas it is currently less used, such as automotive parts or engineering products.

5. Theses

Thesis 1

I showed that the crystalline fraction of products injection molded from poly(ethylene-terephthalate) increases by $110 \pm 25\%$ during post-crystallization under the glass transition temperature, at room temperature. Due to the conversion of the rigid amorphous phase at the interphase of the crystallites, the size of the crystallites increases, as well as the crystalline fraction. This phenomenon cannot be observed in the case of the regranulates, therefore the

increase of the crystalline fraction can be attributed to the morphology formed during injection molding [1].

Thesis 2

With a modulated differential scanning calorimetry test of recycled poly(ethylene-terephthalate) not only recrystallization can be shown and examined but other melting and crystallization processes can also be identified. When the partly simultaneous crystallization and crystalline melting processes on the non-reversible heat flow curve are decomposed into exothermic and endothermic components, the following relationships can be observed between peaks of the individual curves:

- I showed that the initial crystallinity of the samples determined from their total heat flow curve of the samples is the same as the area of the melting peak on the non-reversible curve.
- I found that the sum of the areas of the cold crystallization and post-crystallization peaks on the non-reversible heat flow curve is the same as the area of the melting peak on the reversible curve.

I proved my claim by investigating 16 different kinds of samples, each injection molded with different technological parameters [2].

Thesis 3

The increase in IV during the SSP reaction of the poly(ethylene-terephthalate) regranulate is given by the formula:

$$IV_{n\ddot{o}v} = A(1-IV_0)$$

where $IV_{n\ddot{o}v}$ [(dl/g)/h] is the hourly increase of IV, $A=0.075$ 1/h constant, and IV_0 [dl/g] is the initial IV of the regranulate. My formula is true with the following conditions:

- initial IV is 0.45 – 0.76 dl/g;
- reaction temperature: 230 °C;
- gas medium: nitrogen;
- reaction time: 0 – 8 hours [3].

Thesis 4

I showed with the method I developed that during the solid phase polycondensation of recycled poly(ethylene-terephthalate) not only the molecular weight of the material but its storage modulus can also be increased considerably. The reason for this is the high-temperature

(above 200 °C) crystallization during the process, and the change in the structure of the mobile amorphous part as a result. During crystallization, the conversion of the rigid amorphous phase increases the crystalline fraction. Another consequence of the process is the orientation of the chains in the mobile amorphous phase, which also increases the storage modulus [3].

Thesis 5

I showed that during the chemical foaming of recycled poly(ethylene-terephthalate), when mold temperature increases, porosity increases when an endothermic foaming agent is used, and decreases when an exothermic foaming agent is used. The decomposition of 4% endothermic foaming agent decreased melt temperature by 9 °C, which also decreased the viscosity of the melt. As a result, in the mold temperature range 25-65 °C, porosity increases by 40%. The decomposition of 4% exothermic foaming agent increases melt temperature by 5 °C, which increases melt viscosity as well. As a result, in the mold temperature range 5-45 °C, porosity decreases by 50% [4].

6. Publications related to the theses

- [1] B. Molnar and F. Ronkay, “Time dependence of morphology and mechanical properties of injection moulded recycled poly(ethylene-terephthalate),” *Int. Polym. Process.*, vol. 32, no. 2, pp. 203–208, 2017.
- [2] B. Molnar and F. Ronkay, “Investigation of Morphology of Recycled PET by Modulated DSC,” *Mater. Sci. Forum*, vol. 885, pp. 263–268, 2017.
- [3] B. Molnar and F. Ronkay, “Effect of solid-state polycondensation on crystalline structure and mechanical properties of recycled polyethylene-terephthalate,” *Polym. Bull.*, 2018. (*in press*)
- [4] F. Ronkay, B. Molnar, and G. Dogossy, “The effect of mold temperature on chemical foaming of injection molded recycled polyethylene-terephthalate,” *Thermochim. Acta*, vol. 651, pp. 65–72, 2017.