# Effect of multiple injection moulding on some properties of polycarbonate

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## ABSTRACT

**Purpose:** The aim of this work was to investigate the influence of multiple (up to 6 times) injection moulding of polycarbonate (PC) on some properties of the obtained moulded pieces. The investigation results are to be used to evaluate the usefulness of the multiple processing of PC and management of the PC technological waste by material recycling.

**Design/methodology/approach:** The investigated samples were obtained during the industrial injection moulding. There were determined the sample mechanical properties (by a tensile test), melt flow rate, temperatures of phase transitions (by differential scanning calorimetry, DSC), temperatures of thermal degradation (by thermogravimetric analysis, TGA), as well as storage modulus and damping coefficient (both by dynamic mechanical analysis, DMA).

**Findings:** After the first and second injection mouldings, minor increases (ca. 2.2 wt.% in sum) in the tensile strength of polycarbonate were observed. After the next injection mouldings, this quantity did not change much. The melt flow rate increased along with the number of injection mouldings. The glass temperature was approximately constant while the other phase transition temperatures decreased by ca. 7º C after the first injection moulding and they did not essentially change after each of the next five processing procedures. The storage modulus and damping coefficient of polycarbonate were not noticeably affected by the number of injection mouldings.

**Research limitations/implications:** In order to confirm the claim that degradation processes, occurring in polycarbonate during the first two injection mouldings, cause an increase in the van der Waals interactions between the polymer macromolecules, further investigation is necessary, mostly that on variations in the mean molecular weight of polycarbonate.

**Practical implications:** The studies carried out by now indicate that there are no arguments not to subject the polycarbonate technological waste to the management by material recycling.

**Originality/value:** The method for calculating the fraction of multi-processed plastic in a final product is presented. It has been observed that the largest changes in the examined properties of polycarbonate occur during its first injection moulding. It is stated that a minor degradation of polycarbonate macromolecules increases slightly its tensile strength, caused probably by enhancement of the van der Waals forces.

**Keywords:** Engineering polymers; Multiple injection moulding; Recycling; Mechanical and thermal properties

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## MATERIALS

# 1. Introduction

The material recycling of plastics exhibits many advantages as compared to other kinds of such process. These are lower costs as well as possibilities to apply conventional techniques and commonly used processing equipment like injection moulding presses and extruders. Thus, the material recycling is widely utilised in the reuse of waste occurring during the industrial processing of plastics. Mostly, it consists in shredding the waste and adding it in a proper proportion to the original polymer, applied in a given industrial process. The material recycling is also used in the management of post-consumer plastic waste. In this case, however, the procedure is more expensive and more complex because the waste has additionally to be segregated and washed. To improve the functional qualities, including thermomechanical properties, of the plastic produced by the material recycling of blends of various polymers, appropriate compatibilisers can be applied [1-3].

During reprocessing of a polymer, its degradation caused by a shear stress, heat, oxygen, and catalyst residues occurs. This process takes place not only in the polymers susceptible to these factors, as, e.g., in poly(vinyl chloride), but also in the polymers more resistant to the mentioned agents, like, e.g., in polyethylene. The degradation may cause physical properties and functional qualities of a polymer to be worse. Thus, the moulded products of high quality are difficult to be obtained [4].

The plastics produced by the material recycling contain multi-processed polymers. More and more intensive management of post-consumer plastic waste increases the number of processing cycles of the same polymer. Therefore, recognition of an effect of the multiple injection moulding of various polymers on physical properties of the obtained materials is very important.

In basic processing procedures, including extruding, injection moulding, and thermoforming, the technological plastic wastes are subjected to the management by the material recycling, i.e., by shredding the plastic and adding it to the original polymer in a definite proportion. In this case, the advantages of the method consist in lack of necessity of the waste segregation and washing and in possibility to reuse it in the place where it is produced (no need for transportation and storage). Because, during the processing, the individual plastics degrade to various degrees, it is necessary to determine experimentally the acceptable percentage of each recycled polymer being added to the original one, in order to preserve functional qualities of the final products.

In this case, it has to be taken into account that a fraction of the plastic has been multi-processed. This can be described by a recurrence formula:

$$S_1 = r_1 \qquad (1)$$
$$S_2 = (1 - x) \cdot r_1 + x \cdot r_2 \qquad (2)$$
$$S_n = (1 - x) \cdot r_1 + x \cdot S_{n-1} \qquad (3)$$

where $S_n$ is a composition of a plastic used to produce an object subjected to n technological procedures, $r_n$ – a fraction of a polymer processed n times, n – a natural number, and x – a relative contribution of the recycled polymer being added. From the relations (1), (2), and (3), it follows that

$$S_n = (1 - x) \cdot r_1 + (x - x^2) \cdot r_2 + ... + (x^{n-2} - x^{n-1}) \cdot r_{n-1} + x^{n-1} \cdot r_n \qquad (4)$$

A simple computer programme, using the relation (4) and the Matlab language, has been developed, which enables to determine the composition of a plastic used to produce an object subjected to n technological procedures, with any x of the range $0 < x < 1$.

Exemplary results of calculations for n = 4 (which is described in more details below) and for x= 0.1 and x= 0.3 are listed in Tables 1 and 2. It follows from the tables that when 10 wt.% of a recycled polymer (x= 0.1) has been added, then a fraction in a given product of the polymer processed more than three times is 0.1 wt.% whereas if 30 wt.% of a recycled polymer (x= 0.3) has been added, then this fraction is 2.7 wt.%.

Polycarbonates are linear polyesters of carbonic acid and diols, containing carbonate units. As to practical applications, mostly aromatic polycarbonates are used. They are being produced in the processes of polycondensation of bisphenol A and phosgene and are commonly called polycarbonate (PC). PC is a polymer of superior mechanical (including high impact strength), thermal (including very low thermal expansion in a wide temperature range), electrical, and optical properties.

Table 1.
Percentages of individual fractions of a plastic in an object formed in the *n*th processing procedure (*n* = 1-4), with the 90% contribution of the original polymer added in each procedure (*x* = 0.1; more details in the text)

| Number of a subsequent processing procedure | Contributions of fractions [wt.%] | | | | | |
|---|---|---|---|---|---|---|
| | $r_1$ | $r_2$ | $r_3$ | $r_4$ | $r_5$ | $r_6$ |
| 1 | 100 | - | - | - | - | - |
| 2 | 90 | 10 | - | - | - | - |
| 3 | 90 | 9 | 1 | - | - | - |
| 4 | 90 | 9 | 0.9 | 0.1 | - | - |
| 5 | 90 | 9 | 0.9 | 0.09 | 0.01 | - |
| 6 | 90 | 9 | 0.9 | 0.09 | 0.009 | 0.001 |

Table 2.
Percentages of individual fractions of a plastic in an object formed in the $n$th processing procedure ($n$ = 1-4), with the 70% contribution of the original polymer added in each procedure ($x$ = 0.3; more details in the text)

| Number of a subsequent processing procedure | Contributions of fractions [wt.%] | | | | | |
|---|---|---|---|---|---|---|
| | $r_1$ | $r_2$ | $r_3$ | $r_4$ | $r_5$ | $r_6$ |
| 1 | 100 | - | - | - | - | - |
| 2 | 70 | 30 | - | - | - | - |
| 3 | 70 | 21 | 9 | - | - | - |
| 4 | 70 | 21 | 6.3 | 2.7 | - | - |
| 5 | 70 | 21 | 6.3 | 1.89 | 0.81 | - |
| 6 | 70 | 21 | 6.3 | 1.89 | 0.567 | 0.243 |

Therefore, it is considered as a constructional material, applied in such important products as airplane cockpits, bullet-proof windows, astronaut helmets, medical equipment, and construction parts of various mechanisms, as well as in electrical and electronic engineering. PC can be processed using the methods applied in processing of thermoplastics, such as mostly injection moulding, extrusion, and vacuum forming [5,6].

It is well known that the degradation of PC in conventional using may be regarded as the sum of all physical and chemical effects occurring during its degradation in manufacturing process, ageing during product life and degradation during recycling. The changes in physical properties of PC during series of subsequent recycling cycles are still not very well understood. The methods of chemical recycling of PC, like depolymerisation occurring during methanolysis, are known already for many years. However, their application is limited to the management of the PC waste produced during synthesis of this polymer [7]. The results of investigation of thermal degradation of PC, occurring during processing of the latter and also during the material recycling, indicate that the degradation is caused mostly by cracking of bonds in main chains and by accompanying processes of hydrolysis and alcoholysis [8]. The examination of the effect of mineral fillers on the stress-strain curves for the recycled PC shows a close resemblance between the results derived from theoretical models of these phenomena and from relevant experiments [9]. The acrylonitrile-butadiene-styrene (ABS) terpolymer grafted with maleic acid anhydride, when introduced to the recycled PC, can significantly increase its mechanical properties, including mostly impact strength [10]. In recent years, the researchers become more and more interested in the recycling of the PC and ABS blends, including electric and electronic wastes[11-13]. Due to specific characteristics of polymeric materials, including the materials made of recycled polymers, the choice of adequate methods for investigation of the polymer properties has to be very careful [14-20].

The PC technological wastes are subjected to the management mostly by the material recycling, although the publications on multi-processing of PC are scarce [21]. This fact inspired the authors to undertake the studies described in this article, namely, to investigate the effect of multiple injection moulding of PC on (i) mechanical strength, (ii) melt flow rate, (iii) temperatures of phase transitions and thermal degradation, as well as (iv) storage modulus and damping coefficient. The investigation results may serve as a basis for evaluation of the possibility of multi-processing and management by the material recycling of the technological PC waste appearing during the injection moulding.

## 2. Experimental

### 2.1. Materials

Polycarbonate (PC), type Xantar 19 UR (DSM Engineering Plastics, Netherlands), of the melt flow rate (MFR) equal 19.4 g/10 min (1.2 kg, 300°C) and density equal 1.2 g/cm$^3$ was used as a studied material.

### 2.2. Instruments

The following instruments were used:
- an industrial injection moulding press, type Demag Extra 150/600 (Demag Plastics Machinery GmbH, Germany), with a screw of the 45-mm diameter;
- a shredding mill, type GRS-300-A221 (Gatecha GmbH, Germany), equipped with sieves of the 7.7-mm opening size;
- a tensile testing machine, type Instron 3367 (Instron, USA), for determination of tensile strength ($\sigma_M$), tensile stress at break ($\sigma_B$), tensile strain at tensile strength ($\varepsilon_M$), tensile strain at break ($\varepsilon_B$), and Young modulus (E);
- an MP 600 plastometer (Tinius Olsen, USA), for the MFR measurements;
- a Q 200 differential scanning calorimeter with mass flow control (TA Instruments, USA), for thermal analysis (DSC);
- a Q 500 thermogravimetric analyser (TA Instruments, USA), for thermal stability analysis (TGA);
- a Q 800 dynamic mechanical analyser (TA Instruments, USA), for thermomechanical analysis (DMA).

### 2.3. Methods

The samples were prepared and examined as described below:
- The moulded pieces (Fig. 1) were obtained as construction elements of storage containers, produced in a large-scale

industrial process. The process parameters were as follows: the temperature distributions along a cylinder of the injection moulding press, 250, 265, and 275ºC, the head temperature, 285º C, the mould temperature, 95ºC, the injection moulding pressure, 7 MPa, the injection moulding cycle time, 38 s, including the cooling time of 24 s.

- The samples for a tensile test were 2-mm thick and dumbbell-shaped, in accordance with an appropriate standard [22]. They were cut out by milling from a fragment of the moulded piece shown in Fig. 1.

- The velocity for testing each sample was 50 mm/min [23]. The parameters $\sigma_M$, $\sigma_B$, $\varepsilon_M$, $\varepsilon_B$, and E were determined using 12 individual samples. The final values of these quantities were derived as arithmetic means of 10 results, two extreme ones being neglected.

- The measurements of MFR were performed according to the procedure specified in an appropriate standard [24]. For all the samples, the measuring temperature was 300ºC and the piston load, 1.2 kg. The MFR was determined using 12 individual samples. The final value of MFR was derived as an arithmetic mean of 10 results, two extreme ones being neglected.

- The DSC measurements were carried out in the ambient atmosphere, according to the procedure specified in relevant standards [25,26]. Samples of 2.3-2.5 mg were used, the measurement temperature range was 110-180°C, and the DSC curves were recorded in three stages, corresponding to the first heating, cooling, and the second heating, while the temperature was varied at a rate of 5º C/min. The aim of these measurements was to examine the effect of multi-injection moulding of PC on its glass temperature ($T_g$). In order to eliminate "thermal history" of the samples, the results were analysed using the data collected during the second heating.

- According to the procedure specified in an appropriate standard [27], the TGA measurements were carried out in the ambient atmosphere, in the temperature range of 20-700ºC, and with the temperature varying at a rate of 10ºC/min. The purpose of these measurements was to investigate the effect of multi-injection moulding of PC on the following quantities: the extrapolated temperature of the onset of thermal degradation ($T_{od}$), thermal degradation temperature ($T_d$), determined as a position of the maximum of a dm/dT curve obtained from the relevant TG curve, temperatures $T_{5\%}$ and $T_{50\%}$, corresponding to the 5 and 50% mass loses of the samples, respectively, and the sample mass (P600) remaining at 600º C.

- The DMA measurements were performed following a relevant standard [28], in the ambient atmosphere, in the temperature range of 25-180ºC, and with the temperature varying at a rate of 3º C/min. The measurements were aimed at investigation of the effect of multi-injection moulding of PC on the storage modulus (M) and damping coefficient (K) in a wide temperature range.

In the first stage of the studies, the MFR, DSC, and TGA measurements were carried out, using samples of granulated original PC. Then, a first set of moulded pieces of that material was prepared from which some were used to determine mechanical properties by a tensile properties test and to perform the DMA measurements. The remaining moulded pieces of this

set were shredded by a sieve mill, which enabled to obtain the PC particles less than 8 mm in size. The shredded PC was partially used to perform the MFR, DSC, and TGA measurements while the remaining part was utilised to form the next set of moulded pieces. Such a procedure was repeated six times. The obtained samples were designated with a symbol $P_i$, where "i" is a number of the applied procedures, fulfilling the inequality $0 \leq i \leq 6$ ($P_0$ denotes a sample made of original PC). Thus, some examined quantities ($\sigma_M$, MFR) are denoted additionally with an index "i", consistent with the sample symbol index.
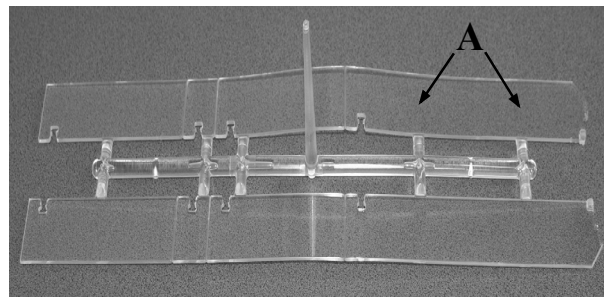


Fig. 1. A photograph of a moulded piece made of polycarbonate (A – a fragment from which samples were cut out to determine their mechanical properties in a static tension test)

When comparing the mean values of the quantities, determined for the injection moulded samples, the test of significance for the respective two means was performed, assuming the significance level $\alpha = 0.05$ and applying the Snedecor's F-test and Student's t-test. If the hypothesis about equality of two means is rejected in favour of an alternative hypothesis that the arithmetic mean of a measured quantity for the $P_i$ sample is greater (or smaller) than that for the $P_j$ sample ($0 \leq i \leq 6$, $0 \leq j \leq 6$), then the compared quantities significantly differ from one another statistically.

## 3. Results and discussion

### 3.1. Tensile stress-strain characteristics

A plot obtained from the tensile test performed for a sample ($P_0$) prepared from the moulded piece shown in Fig. 1 is presented in Fig. 2. The plots for the remaining samples are very similar; therefore, they are not shown.

Tensile strength and tensile stress at break as functions of the number of injection mouldings are presented in Fig. 3, together with standard deviations (SDM or SDB) of the results from their mean values ($\sigma_M$ or $\sigma_B$). The values of $\sigma_{M1}$ and $\sigma_{M2}$ are larger as compared to $\sigma_{M0}$ by 0.5 and 1.3 MPa, respectively. The Snedecor's F-test proves that these values differ significantly whereas no significant differences are observed between the values of $\sigma_{M2}$, $\sigma_{M3}$, $\sigma_{M4}$, $\sigma_{M5}$, and $\sigma_{M6}$. This test indicates also that there are no significant differences between the values of $\sigma_B$ for individual samples. For all the studied samples, the inequality SDB > SDM is obeyed.
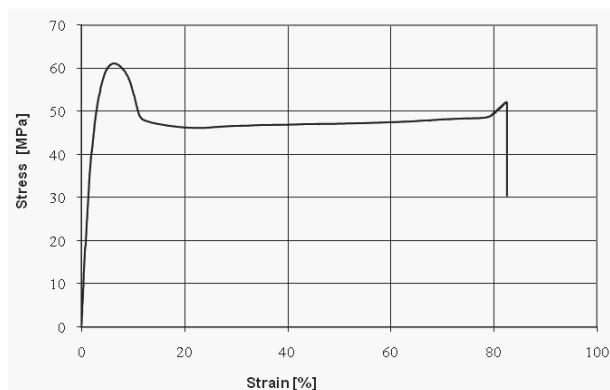
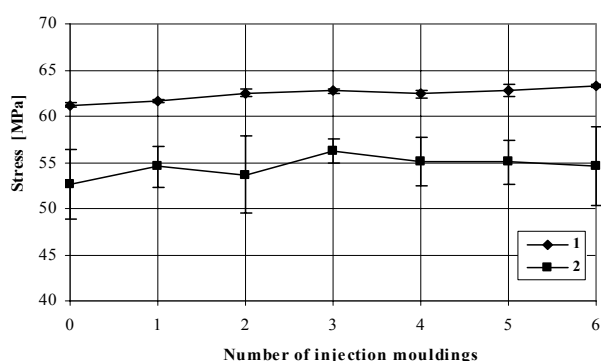Fig. 2. The stress-strain curve of sample $P_0$



Fig. 3. The effect of the number of injection mouldings on tensile strength (1) and tensile stress at break (2) of PC

The increase in $\sigma_M$ of samples $P_1$ and $P_2$ as compared to $\sigma_{M0}$ may be caused by enhancement of the van der Waals forces existing between the PC macromolecules. This, in turn, may be due to a reduction in mean distances between the PC macromolecules, brought about by a decrease in their length due to a degradation process. This mechanism is supported by the results of the MFR measurements and the literature data [21,29].

The values of $\varepsilon_M$ for PC are not affected by the subsequent injection mouldings and are equal ca. 6% for all the samples. The $\varepsilon_B$ values, also not affected by the subsequent injection mouldings, are much higher (75-90%) and exhibit a significantly higher standard deviation. The longitudinal modulus of elasticity (the Young's modulus) does not change considerably with the number of injection mouldings as well and is equal to 2.1-2.6 GPa.

## 3.2. Melt flow rate

The MFR values varying with the number of injection mouldings are shown in Fig. 4.

The plot is relatively well approximated with a second order polynomial: MFR $= -0.0555\,k^2 + 0.7323\,k + 19.63$, with $R^2 = 0.961$ (k is the number of injection mouldings). Contrary to $\sigma_M$ and $\sigma_B$, the MFR values increase monotonically with the subsequent injection mouldings. The largest increase occurs after

the first processing (MFR$_1$/MFR$_0$ $\approx 1.06$), while the total increase in MFR exceeds 13% (MFR$_6$/MFR$_0$ = 1.134). The MFR data indicate that degradation of PC enhances with the number of injection mouldings.

In order to confirm the results of the MFR measurements, the mean molecular weight ($M_V$) of PC being multi-processed purpose, viscosity numbers (VN) of the studied samples were determined according to an appropriate standard [30] by measuring times of outflow of PC solutions from a capillary of a normalised viscometer.
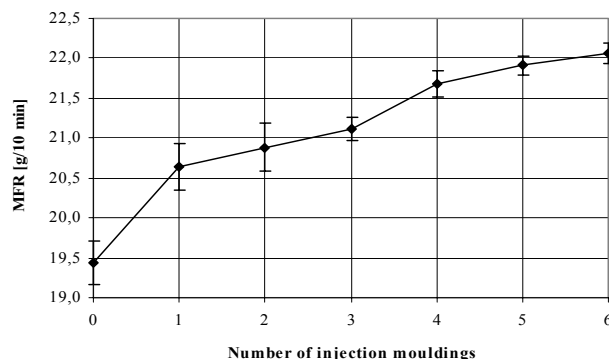


Fig. 4. The effect of the number of injection mouldings on melt flow rate (MFR) of PC

Table 3.
Viscosity numbers (VN), limiting viscosity numbers (LVN), and mean molecular weight ($M_V$) of PC being multi-processed by injection moulding

| Number of injection moulding | VN [mgl/g] | GLL [mgl/g] | $M_V$ [g/mol] |
|---|---|---|---|
| 0 | 50.66 | 47.26 | 21025 |
| 1 | 48.64 | 45.49 | 19969 |
| 2 | 48.31 | 45.21 | 19802 |
| 3 | 47.18 | 44.21 | 19217 |
| 4 | 46.39 | 43.52 | 18810 |
| 5 | 45.81 | 43.01 | 18511 |
| 6 | 46.11 | 43.27 | 18663 |

The results were evaluated statistically. Using the Matthes and Solomon-Goetsman equations [31], limiting viscosity numbers (LVN) of PC for individual samples were obtained. The values of $M_V$ were calculated from the Mark–Houwink–Kuhn–Sakurada relation [32] and are presented in Table 3. They agree quite well with the MFR results.

## 3.3. Thermal behaviour

The DSC curve corresponding to the second heating of sample $P_3$ (Fig. 5) reveals a peak assigned to the glass transition. The DSC curves of the remaining samples are very similar to that in Fig.5 and are not shown. A peak associated with the melting of the crystalline phase is absent because PC is an amorphous polymer. The DSC data indicate that the glass temperature is not affected by the

number of injection mouldings: $T_g = 144.6\text{-}145.5$°C for all the samples. Moreover, the moulded pieces did not change colour with the subsequent injection mouldings.
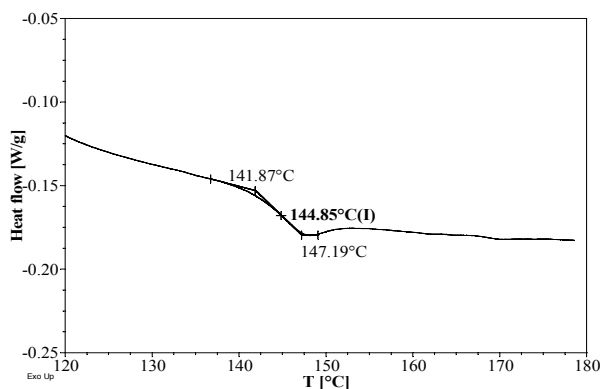


Fig. 5. The DSC curve corresponding to the second heating of sample $P_3$

## 3.4. Thermal stability

The TGA (TG and dm/dT) curves (m and T are the sample mass and temperature, respectively) of the $P_3$ sample are presented in Fig. 6. The TGA curves of the remaining samples are very similar to those in Fig. 6 and are not shown.
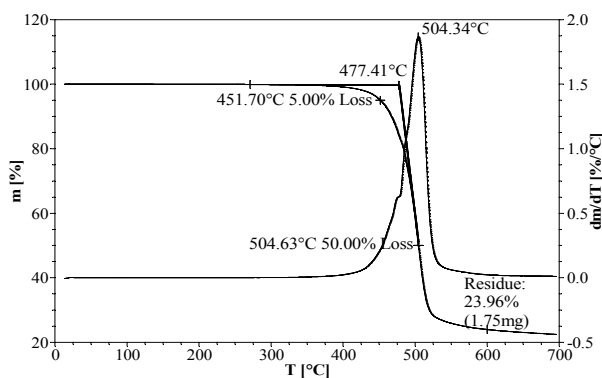


Fig. 6. The TGA curves of sample $P_3$

These curves were used to determine the effect of multi-injection moulding of PC on the extrapolated temperature of the onset of thermal degradation ($T_{od}$), thermal degradation temperature ($T_d$), temperatures $T_{5\%}$ and $T_{50\%}$, corresponding to the 5 and 50% mass loses of the samples, respectively, and the sample mass ($P_{600}$) remaining at 600°C. The mentioned quantities are plotted in Fig. 7.

As seen, they change most after the first injection moulding of PC. The differences between the values of $T_{od}$, $T_d$, $T_{5\%}$, and $T_{50\%}$

for samples $P_0$ and $P_1$ are close to one another and equal ca. 7°C. The next injection mouldings do not considerably influence the mentioned temperatures. The maximum differences for all the samples do not exceed 5°C and directions of the changes vary from sample to sample. The values of $P_{600}$ do not change with the number of injection mouldings and are confined in the range of 23.2-24.4%.
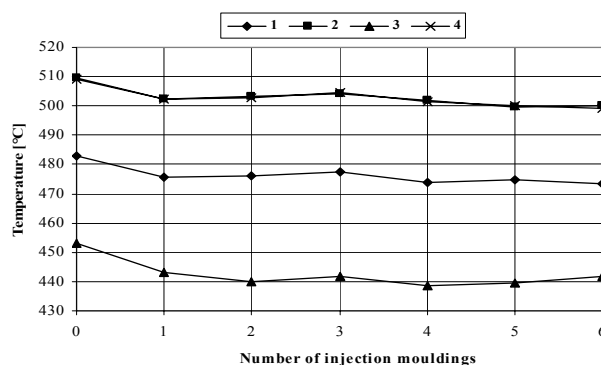


Fig. 7. The effect of repeated injection mouldings of PC on the extrapolated temperature of the onset of thermal decomposition (1), thermal decomposition temperature (2), and temperatures corresponding to the 5 and 50% mass loses of the samples (3 and 4, respectively). The plots (2) and (4) coincide

## 3.5. Mechanothermal behaviour

The values of M and K as functions of temperature for sample $P_3$ are shown in Fig. 8. The plots for the remaining samples are analogous and are not presented.
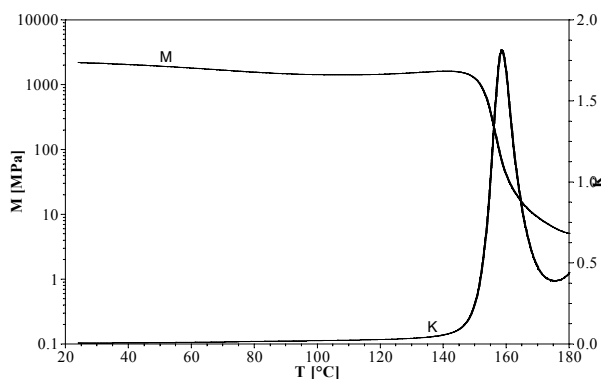


Fig. 8. The DMA curve including variations in storage modulus (M) and damping coefficient (K) of sample $P_3$

As seen, M drops approximately linearly from ca. 2100 to ca. 1370 MPa as the temperature increases from 25 to 100°C. Then, in the temperature range of 100-143°C, M increases to 1580 MPa,

probably due to rearrangement of polymer chains, caused by relaxation of stress generated in this sample during its fabrication [33]. In the temperature range of 143-165°C, M decreases rapidly (down to 14 MPa), which indicates a transition from the glassy state to high-elastic one.

The K value at temperatures up to ca. 140°C is relatively small, but above 140°C, it increases dramatically, which is probably due to the beginning of changes in the PC structure with a final stage being the polymer glass transition. The temperature corresponding to the maximum of K (ca. 158°C) is considered as the glass transition temperature of PC. The glass transition temperatures determined by DMA and TGA differ, which is confirmed in the literature [33].

## 4. Conclusions

- Along with the increase in the number of injection mouldings, the tensile strength of PC initially rises slightly (after the first and second processing procedure) and, then, it remains constant. The other quantities, determined in a tensile test, i.e., tensile stress at break, tensile strain at tensile strength, tensile strain at break, and longitudinal modulus of elasticity do not essentially change. Dispersion of the measured values, expressed as the standard deviation, is much less for the tensile strength than for the tensile stress at break.
- The melt flow rate increases as the number of injection mouldings of PC rises. The largest increase, being above 6% in relation to the original PC, occurs after the first processing procedure. After the sixth injection moulding, the MFR value is larger by more than 13%.
- The glass temperature (from DSC) does not vary with the number of injection mouldings and is ca. 145°C. In all the studied samples, PC does not crystallise from the liquid phase and the process of cold crystallisation does not occur.
- The extrapolated temperature of the onset of thermal degradation, thermal degradation temperature, and temperatures corresponding to the 5 and 50% mass loses of the samples decrease by ca. 7°C after the first injection moulding of PC. These temperatures do not considerably vary after the next five processing procedures.
- Storage modulus and damping coefficient of PC do not essentially change after repeated injection mouldings. However, the glass temperature determined from DMA is by 13°C higher than that from DSC.
- The obtained results indicate that there are no arguments not to apply the material recycling to the management of the polycarbonate technological waste occurring during the injection moulding.

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