Some effects of multiple injection moulding on selected properties of ABS

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Materials

ABSTRACT

Purpose: The purpose of this work was to investigate the influence of multiple (up to 10 times) injection moulding of acrylonitrile butadiene styrene (ABS) on some properties of the obtained moulded pieces.

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 injection mouldings, minor decreases (ca. 2.0 %) in the tensile strength 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 transition temperatures (from DSC) of butadiene and acrylonitrile styrene fractions don't vary with the number of injection mouldings and are ca. 61 and +104ºC, respectively.

Research limitations/implications: In order to confirm that degradation process occurs in ABS during injection mouldings, further investigation is necessary, mostly that on variations in the mean molecular weight of ABS.

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

Originality/value: It has been observed that the largest changes in the tensile strength and melt flow rate of ABS occur during its first injection moulding and a melt flow rate increases slightly with the number of injection mouldings. The temperatures of phase transitions and thermal as well as the storage modulus and damping coefficient of ABS do not essentially change after repeated injection mouldings.

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

1. Introduction

In recent years, much attention has been paid to material recycling of plastics, which exhibits many advantages as compared to other kinds of such processes. These are lower costs as well as possibilities to apply conventional techniques and commonly used processing equipment like injection moulding presses, extruders and thermoforming machines. 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 [1].

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 quality of a polymer to be worse. Thus, the moulded products of high quality are difficult to be obtained [2].

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. Increasing interest in polymeric composites, including materials made of recycled polymers, is the result of their very good specific strength and stiffness [3-8]. Very interesting application of these materials is piping industry [9,10].

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, using to determine the composition of a plastic used to produce an object subjected to n technological procedures, with any x ($0 < x < 1$) which a relative contribution of the recycled polymer being added. It follows from this formula that when 30 wt.% of a recycled polymer (x = 0.3) has been added, then a fraction in a given product of the polymer processed more than three times is 2.7 wt.%. [11].

The acrylonitrile-butadiene-styrene (ABS) terpolymer is an important constructional plastic. Copolymerisation of styrene and acrylonitrile carried out in the polybutadiene latex medium (Fig. 1) is one of the basic methods for manufacturing ABS.
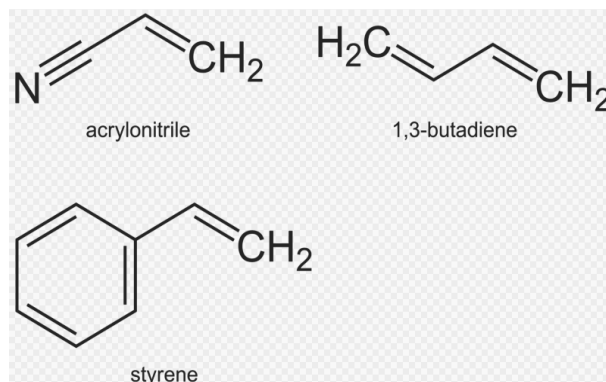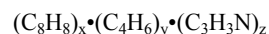

Fig. 1. Chemical formulas of acrylonitryle, styrene and polybutadiene used for ABS polymerization

The chemical formula of ABS is as follow:

$$(C_8H_8)_x \bullet (C_4H_6)_y \bullet (C_3H_3N)_z$$

A typical ABS contains from 15 to 35% acrylonitrile, 5 to 30% butadiene and 40 to 60% styrene but these proportions may vary in a relatively wide range. The result is a long chains of polybutadiene crosslinked with shorter chains of poly(styrene-co-acrylonitrile). The nitrile groups, being polar, attract each other and join the chains together, making ABS stronger than each if its component.

The ABS properties essentially depend on the properties of each component: acrylonitrile enhances hardness and chemical resistance, butadiene acts as plasticiser while increasing impact strength, and styrene improves thermal and processing properties. Thus, high hardness and impact strength, high chemical and thermal resistance, negligible creep as well as easy processing are the most significant advantages of ABS while a relatively low fatigue strength is a disadvantage [12-14].

A lot of modifications can be made to improve impact resistance, toughness, and heat resistance. The impact resistance can be improved by increasing the proportions of polybutadiene in relation to styrene. Impact resistance does not fall off rapidly at lower temperatures. Stability under load is excellent with limited range of loads. Thus, changing the proportions of its components, ABS can be prepared in different grades (especially for exstrusion and injection moulding). ABS has useful characteristics within a temperature range from -40 to 100 °C. ABS is widely being applied in the construction of machines, cars, computers, and household equipment. Due to high susceptibility to metallization, it is also used as a base for electronic assemblies and printed circuits. ABS can be processed using the methods applied in processing of thermoplastics, such as mostly injection moulding, extrusion, and vacuum forming [15].

There are different techniques for the ABS waste management. They consist in combustion of this plastic[16] or dissolution in suitable solvents, followed by filtration and precipitation [17]. The management of ABS/polycarbonate blends and degradation of the plastic have also been studied [18-21]. The ABS technological wastes are subjected to the management mostly by the material recycling, although the publications on multi-processing of ABS are scarce [22,23]. In case of the ABS

M. Żenkiewicz, P. Rytlewski, K. Moraczewski, M. Stepczyńska, T. Karasiewicz, R. Malinowski, W. Ostrowicki

recycling by injection moulding, temperature and shear velocity of the plastic being injected significantly influence properties of the moulded pieces [24].

In the past a lot of studies were devoted to polymer multiple recycling. The general trend is some decrease of the mechanical strength and increase of the melt flow ratio observed after several injections or extrusions cycles according to the nature of the polymer being recycled. According to our best knowledge similar results concerning multiple recycling of ABS waste have not been published.

The above mentioned facts inspired the authors to undertake the studies described in this article, namely, to investigate the effect of multiple injection moulding of ABS 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 ABS waste appearing during the injection moulding.

2. Experimental

2.1. Materials

Acrylonitrile-butadiene-styrene (ABS), type Polylac PA-757 (Chi Mei Corporation, Taiwan), of the melt flow rate (MFR) equal 1.62 g/10 min (5 kg, 200°C) and density equal 1.05 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 (σ_M), tensile stress at break (σ_B), tensile strain at tensile strength (ε_M), tensile strain at break (ε_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. 2) 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, 195, 210, and 220°C, the head temperature, 230° C, the mould temperature, 40°C, the injection moulding pressure, 5,5 MPa, the injection moulding cycle time, 40 s, including the cooling time of 27 s.
- The samples for a tensile test were 2-mm thick and dumbbell-shaped, in accordance with an appropriate standard [25]. They were cut out by milling from a fragment of the moulded piece shown in Fig. 2.
- The velocity for testing each sample was 50 mm/min [26]. The parameters σ_M, σ_B, ε_M, ε_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 [27]. For all the samples, the measuring temperature was 200°C and the piston load, 5 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 [28,29]. Samples of 2.3-2.5 mg were used, the measurement temperature range was -70 - +190°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 5° C/min. The aim of these measurements was to examine the effect of multi-injection moulding of ABS 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 [30], the TGA measurements were carried out in the ambient atmosphere, in the temperature range of +20 - +700°C, and with the temperature varying at a rate of 10°C/min. The purpose of these measurements was to investigate the effect of multi-injection moulding of ABS 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 $T_{5\%}$, $T_{50\%}$ and $T_{95\%}$, corresponding to the 5, 50 and 95% mass loses of the samples, respectively, and the sample mass (P_{500}) remaining at 500° C.
- The DMA measurements were performed following a relevant standard [31], in the ambient atmosphere, in the temperature range of 25-160°C, and with the temperature varying at a rate of 3° C/min. The dimensions of the samples were of 60 mm in length, 12.5 mm in with and 2 mm of thickness A dual cantilever setup was used to deform each sample to a strain of 15 μm, at a frequency of 1 Hz. The measurements were aimed at investigation of the effect of multi-injection moulding of ABS 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 ABS. 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 ABS particles less than 8 mm in size. The shredded ABS 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 ten times. The obtained samples were designated with a symbol P_i, where "i" is a number of the applied procedures, fulfilling the inequality $0 \leq i \leq 10$ (P_0 denotes a sample made of original ABS). Thus, some examined quantities (σ_M, MFR) are denoted additionally with an index "i", consistent with the sample symbol index.
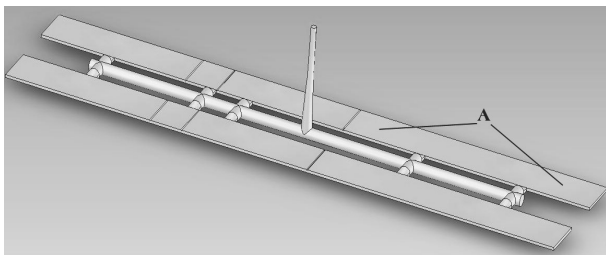

Fig. 2. A photograph of a moulded piece made of ABS (A – a fragment from which samples were cut out to determine their mechanical properties in a static tension test)

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 ($0 \leq i \leq 10$, $0 \leq j \leq 10$), then the compared quantities significantly differ one from another statistically.

3. Results and discussion

3.1. Tensile stress-strain characteristics

A plots obtained from the tensile test performed for the samples P_0 prepared from the moulded pieces shown in Fig.2 are presented in Fig. 3. 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.4, together with standard deviations (SD_M or SD_B) of the results from their mean values (σ_M or σ_B). The value of σ_{M0} is larger as compared to σ_{M1} - σ_{M10} by 0.5 - 1.5 MPa, respectively. The Snedecor's F-test proves that the value of σ_{M0} differ significantly from the others, whereas no significant differences are observed between the

values of σ_{M1} - σ_{M10}. This test indicates also that there are no significant differences between the values of σ_{B0} and σ_{B10} and between the values of σ_{B0} and most of others samples σ_{Bi}. For all the studied samples, the inequality $SD_B > SD_M$ is obeyed ($0.2 < SD_M < 0.4$ MPa and $1.5 < SD_B < 5.2$ MPa).
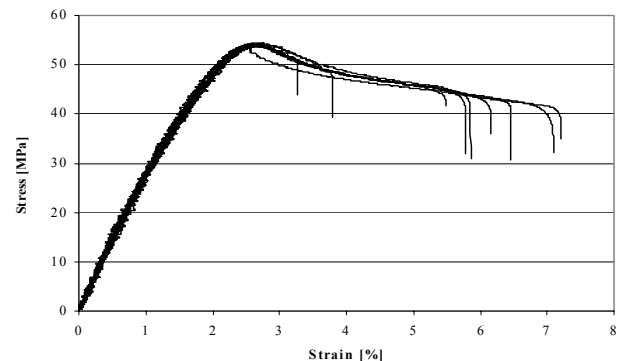

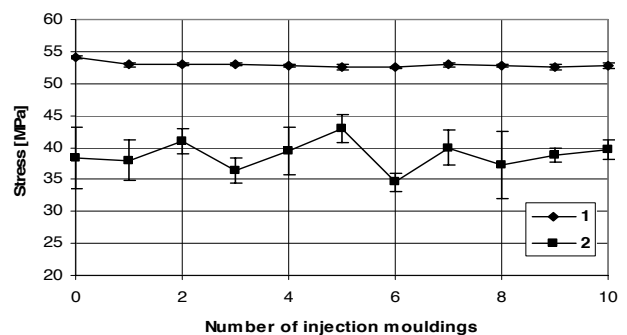Fig. 3. The stress-strain curves of the samples P_0

Fig. 4. The effect of the number of injection mouldings on tensile strength (1) and tensile stress at break (2) of ABS
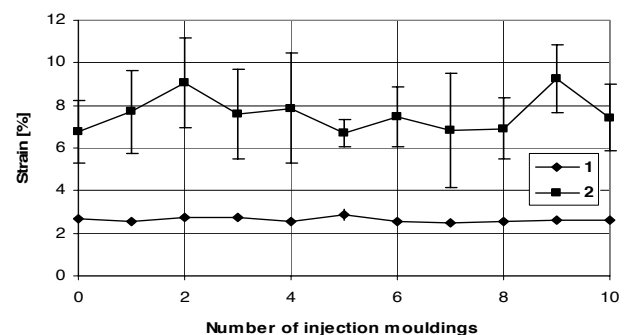

Fig. 5. The effect of the number of injection mouldings on tensile strain at tensile strength (1) and tensile strain at break (2) of ABS

The decrease in σ_M of sample P_1 as compared to σ_{M0} may be caused by a degradation process. This mechanism is supported by the results of the MFR measurements.
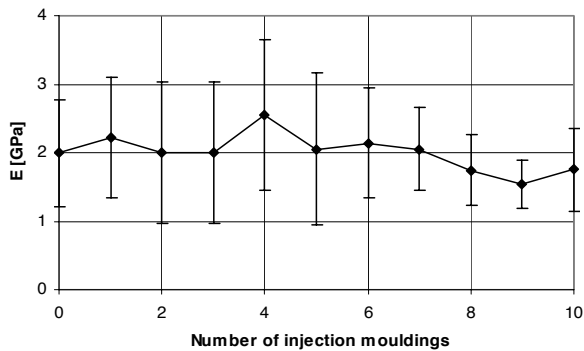
M. Żenkiewicz, P. Rytlewski, K. Moraczewski, M. Stepczyńska, T. Karasiewicz, R. Malinowski, W. Ostrowicki

Fig. 6. The effect of the number of injection mouldings on longitudinal modulus of elasticity (E) of ABS

The values of ε_M for ABS (Fig. 5) are not affected by the subsequent injection mouldings and are equal ca. 2.5 - 2.7% for all the samples. The ε_B values (Fig. 5), also not affected by the subsequent injection mouldings, are much higher (6.7 - 9.3%) and exhibit a significantly higher standard deviation.

The results of measurements of longitudinal modulus of elasticity (the Young's modulus) are presented in Fig.6. The Snedecor's F-test proves there are no significant differences between the values of E_i, because of relatively very high values of standard deviation ($0.4 < SD_E < 1.1$ GPa).

3.2. Melt flow rate

The MFR values varying with the number of injection mouldings are shown in Fig. 7. Contrary to σ_M and σ_B, the MFR values increase monotonically with the subsequent injection mouldings. The largest increase (ca. 14.8%) occurs after the first processing ($MFR_1/MFR_0 = 1.148$), while the total increase in MFR exceeds 27% ($MFR_{10}/MFR_0 = 1.272$). The MFR data indicate that degradation of ABS enhances very slowly with the number of injection mouldings.
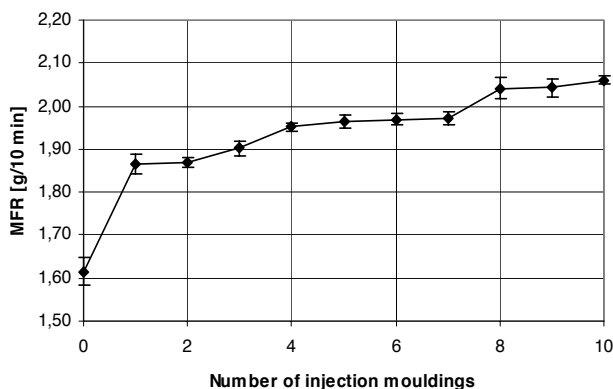

Fig. 7. The effect of the number of injection mouldings on melt flow rate (MFR) of ABS

3.3. Thermal behaviour

The DSC curve corresponding to the second heating of sample P_0 (Fig. 8) reveals three peaks. The first one ($T_{gb} = -60.8°C$) is assigned to the glass transition of butadiene fraction, the second one ($T_{ga} = +104.2°C$) is assigned to the glass transition of acrylonitrile-styrene (SAN) fraction and the third one ($T_{ma} = +129.4°C$) is probably assigned to the melting point of any additive. The DSC curves of the remaining samples are very similar to that in Fig. 8 and are not shown. A peak associated with the melting of the crystalline phase is absent because ABS is an amorphous polymer. The DSC data indicate that the glass temperature is not affected by the number of injection mouldings: $-61.2 < T_{gb} < -60.1°C$ and $+104.0 < T_{gb} < +104,5°C$ for all the samples, while T_{ma} decrease slightly (from 129.4°C to 128.2°C) with increasing of the number of injections moulding. The moulded pieces did not change colour with the subsequent injection mouldings.
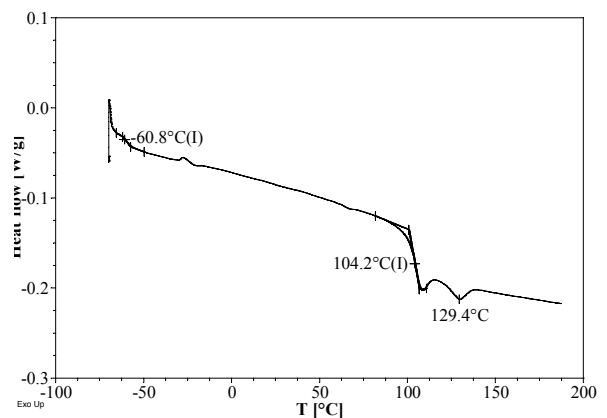

Fig. 8. The DSC curve corresponding to the second heating of sample P_0

These findings are supported by the results of the DSC measurements presented in the literature [32].

3.4. Thermal stability

The TGA (TG and dm/dT) curves (m and T are the sample mass and temperature, respectively) of the P_0 sample are presented in Fig. 9. The TGA curves of the remaining samples are very similar to those in Fig. 9 and are not shown.

These curves were used to determine the effect of multi-injection moulding of ABS on the extrapolated temperature of the onset of thermal degradation (T_{od}), thermal degradation temperature (T_d), temperatures $T_{5\%}$, $T_{50\%}$ and $T_{95\%}$ corresponding to the 5, 50 and 95% mass loses of the samples, respectively, and the sample mass (P_{500}) remaining at 500ºC. The mentioned quantities are plotted in Fig. 10.
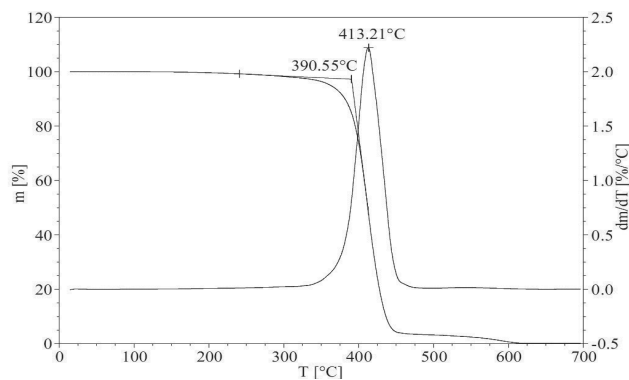
Fig. 9. The TGA curves of sample P_0

As seen, the values of T_{od}, T_d, $T_{5\%}$, $T_{50\%}$ and $T_{95\%}$ does not vary with the number of injection mouldings and are ca. 390, 412, 363, 411 and 445ºC, respectively. The values of P_{500} do not change with the number of injection mouldings and are confined in the range of 1.7-3.2%.
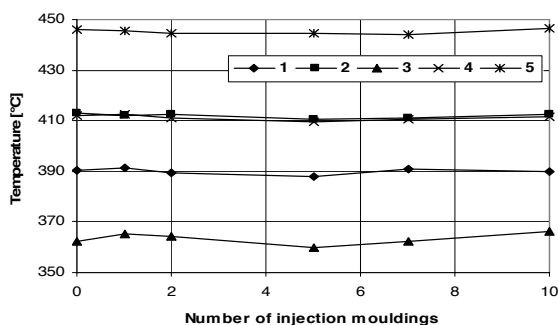

Fig. 10. The effect of repeated injection mouldings of ABS on the extrapolated temperature of the onset of thermal decomposition (1), thermal decomposition temperature (2), and temperatures corresponding to the 5, 50 and 95% mass loses of the samples (3, 4 and 5, respectively). The plots (2) and (4) coincide

3.5. Mechanothermal behaviour

The values of M and K as functions of temperature for sample P_0 are shown in Fig. 11. The plots for the remaining samples are analogous and are not presented.

As seen, M drops approximately linearly from ca. 2330 to ca. 11746 MPa as the temperature increases from 25 to 75ºC. Then, in the temperature range of 75-100ºC, M increases slightly to 1760 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 100-118ºC, M decreases rapidly (down to 23 MPa), which is relatively well approximated with a first order polynomial (Fig. 12).

In this range of temperature the drop of 1ºC causes the decrease of M ca. 75 MPa.
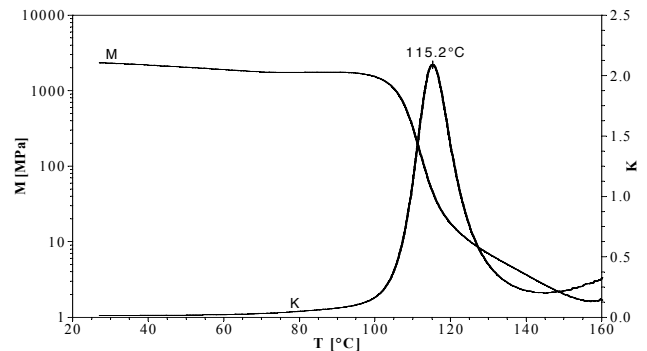
Fig. 11. The DMA curve including variations in storage modulus (M) and damping coefficient (K) of sample P_0
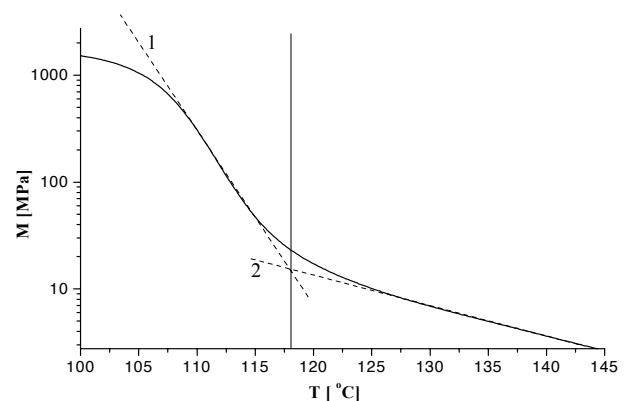
Fig. 12. The linear approximations of the M curve presented in Fig. 11 of two characteristic regions of temperature

Then, the temperature decrease by 1ºC in the temperature range of 118-145ºC is accompanied by the reduction in M by ca. 0.4 MPa, which indicates a change in the nature of the glass transition. A different course of changes in the M values may point out superposition of two glass transitions associated with the acrylonitrile and styrene blocks of the ABS copolymer, the temperatures of which may differ by up to 25°C [15].

The K value at temperatures up to ca. 100ºC is relatively small, but above 100ºC, it increases dramatically, which is probably due to the beginning of changes in the ABS structure with a final stage being the polymer glass transition. The temperature corresponding to the maximum of K (ca. 115ºC) is considered as the glass transition temperature of ABS. The glass transition temperatures determined by DMA and TGA differ, which is confirmed in the literature [33].

4. Conclusions

- After the first injection moulding the tensile strength of ABS decreases slightly (ca. 2% of the virgin ABS) and then

remains approximately 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 ABS rises. The largest increase, being ca. 15% in relation to the original ABS, occurs after the first processing procedure. After the ten injection moulding, the MFR value is larger by more than 27%.
- The glass transition temperatures (from DSC) of butadiene and acrylonitrile-styrene fractions don't vary with the number of injection mouldings and are ca. -61 and +104ºC, respectively. In all the studied samples, ABS 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, 50 and 95% mass loses of the samples do not considerably vary after the subsequent processing procedures.
- Storage modulus and damping coefficient of ABS do not essentially change after repeated injection mouldings. However, the glass temperature determined from DMA is by 11ºC 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 ABS technological waste occurring during the injection moulding.

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