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The examination of the structure of PP composites with the glass fibre

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ABSTRACT

Purpose: of the examination is determination of selected physical properties: melting and crystallization temperature for polypropylene PP and its composites with glass fiber during observation the melting and congealing process by means of optical microscope in white light.

Design/methodology/approach: Examination of melting and crystallization temperature of polypropylene composites with glass fibre became carried out by means of the set for the microscopic observation of phase transformations. The set consist of polarized microscope and computer system recording. By means of the set the crystal objects were tested during melting and crystallization process of polymer plastic going through the sample in normal light. The melting and crystallization temperatures were determined visuallily. Examination were carried out registering by means of the microphotography at the definite temperature occurring transformations during heating polymer samples and composites with speed of 200ºC /min, and also during cooling with speed of 50ºC/min in white light.

Findings: Fibrous filler in the form of the glass fibre nucleation and modifies the process of the crystallization of the polypropylene composites.

Research limitations/implications: The influence of other fillers on crystallization process of polypropylene composites will be defined in future.

Practical implications: The carried out tests of structure are useful especially for determination how the used filler nucleates and modifies the crystallization process of polypropylene and its composites.

Originality/value: The computer system of recording of the picture enables the carrying out of proceeding processes on-screen of the computer monitor and the recording of single frames and sequences the video, what enables more precise analysis of processes occurring at the high speed.

Keywords: Engineering polymers; Properties; Structure; Melting temperature; Crystallization temperature

MATERIALS

1. Introduction

The main factors influencing on the ability of the polymers to crystallization are [1-10]:

- Chemical structure of the polymer
- Crystalline memory of the polymer
- Presence of strange inclusions and contaminations

Influence of chemical structure of polymer on his ability to crystallization we can consider on a few surfaces. The first surface concerns of the average particle mass and degree of polydispersion. Higher facility to crystallization demonstrates the shorter chains, what is the result of bigger mobility of macromolecule segments and better geometrical conditions to assume straight shape. The high degree of polydispersion is also the factor decreasing crystallization ability of polymer. Imperfection of the chains chemical structure counteracts of forming the regular crystalline structures. Similar influence has the polydispersion of polymer. The other surface concerns the

geometrical structure of macromolecule, thus overall it is possible to state, that the polymer demonstrates the higher ability to crystallization the more simple and regular geometric are its macromolecules. The factors of increasing ability to crystallization are [1-10]:

- the regularity of the three-dimensional structure of the polymer (the configuration of macromolecules),
- the lack of side branches of the main chain,
- little sizes and slight differences of the size of side groups,
- the uniform distribution of side groups.

Together with increasing of flexibility of the macromolecules the melting and crystallization temperature decreases. The last surface concerns the presence of chemical groups able to make intermolecular fixations in monomeric cells, deciding about fixing the chains into the skeleton of space net. The factor, which is called the crystalline memory or polymer's thermal history, is connected with crystalline state of polymer in period before melting and crystallization. In the time of melting the polymer, part of the crystalline aggregates can be completely destroyed. Such residual, very stable crystals can be present in melted polymer through longer time. During the process of the crystallization increases the number of arising nucleus of the crystallization. Essential influence for crystalline ability of polymer has also cooling velocity of polymer. Eventually inclusions and contaminations present in melted polymer can change the number and characteristic of the crystalline aggregates. In some cases inclusions can act similar as additional nucleus of crystallization and can stimulate the crystallization, in others – they can inhibite the process, and in extreme cases – even they can make it impossible. Presence of such additives, like powder fillers, plasticizers and so on, has a significant influence for speed of crystallization and temperature of melting the crystallites. The necessary condition, but not quite enough, to proceed the crystallization process, is lower value of free enthalpy of the solid in comparison with liquid. Besides of this, it must proceed also nucleation and increasing of crystallites. Velocity of the both processes decides about whole velocity of crystallization. It is the higher the bigger is the different of the free enthalpy. The melting temperature (crystallization) of polymer determines the force of intermolecular interaction determining the value of heat (enthalpy) of crystallization $\boldsymbol{AH}$ and rigidity of chains, what has influence on changing the entropy of the process $\boldsymbol{AS}$. In melting temperature: $\boldsymbol{AG = AH - T_mAS = 0}$ [1,2,3,11÷15].

2. Materials, apparatus and methods of tests

The purpose of the present examination is the visual determination of the melting and crystallization temperature of PP composites during observation of the melting and congealing process on optical microscope in white light.

Also examination of structure of matrix and the composites were carried out. For the examination of structure during melting and congealing process the polypropylene and its two composites with glass fibre were used:

- polypropylene with 15 % content of glass fibre,
- polypropylene with 25 % content of glass fibre,

The examination of melting and crystallization temperature of selected polymer materials were carried out with the help of the set for microscope observation of phase transformations, which consist of the following apparatus:

- The table METTLER TOLEDO FP82HT with processor controlling system METTLER TOLEDO FP90
- Polarization microscope „POLAM P.-113"
- The computer system recording of picture – the video camera „Panasonic KR-222", the computer video card „MIRO" and computer with Pentium II 233MHz processor.

Cell of the heating table type Mettler Toledo enables observation of the processes proceeding in tested material under microscope. The processor controlling system of heating the cell Melter Toledo FP90 enables programming the velocity of heating process the sample by assignment the temperature of beginning the programmed heating, velocity of increasing the temperature, and final temperature. Besides of this it is possible to change in progress the two last parameters and manually speed up and stop the temperature increasing, and also decrease the temperature for better observe proceeding transformations.

Applying of the polarized microscope for testing of crystalline objects enables observation of the melting process of object in light passing through the sample. Applying of the polarizing microscope for testing the crystal objects enables observation of the melting process of the object in white light passing through the sample, and also enables for optical delimitation the melting temperature of preparation (visible deliquescence of the sample and its mobility on microscopic slide), through using the polarizer - analyzer system in microscope, between which the tested sample is placed, (in polarized light the objects demonstrating crystal properties, like solid crystals, causing the light depolarization, what is visible as luminance of the sample on dark background). The observation can be performed with different magnifications, depending on used objective. The computer system of recording of the picture enables making observation of proceeding processes on screen of the computer monitor and recording single frames and video sequences, what enables to make more precise analysis of processes proceeding with high speed.

The tests were carried out recording proceeding transformations during heating the samples of tested polymers and their composites with speed 20 ^{0}C /min by means of microphotography, and also during cooling with speed 5^{0}C/min in white light and in polarized light, with forty times magnification of objective. Additionally for every sample, recording of video sequences from crystallization process of the composites melted before was carried out.

The video sequences were recorded with different speed and at different start temperatures:

- polypropylene – 1 frame/0,1 ^{0}C start =128 ^{0}C
- polypropylene + 15 % glass fibre – 1 frame/0,1^{0}C start=130 ^{0}C
- polypropylene + 25 % glass fibre – 1 frame/0,1 ^{0}C start=130^{0}C

3. Investigation results and discussion

During examination the visually defined melting and crystallization temperature of the sample were noted:.

- polypropylene,

T_{top} = 168 ^{0}C T_{kryst}= 127-1117 ^{0}C

- polypropylene + 15 % glass fibre,

T_{top} = 169 ^{0}C T_{kryst}= 129,7-122 ^{0}C

- polypropylene + 25 % glass fibre,

T_{top} = 170 ^{0}C T_{kryst}= 131,9-123 ^{0}C

Additionally for some of the samples size of objectives recorded on microphotography were determined:

- polypropylene – spherolites ~ 115-132 μm,
- polypropylene + 15 % glass fibre – spherolites ~ 52 μm, glass fibre ~ 15,6 μm,
- polypropylene + 25 % glass fibre – spherolites ~ 42-51 μm, glass fibre ~ 13-14 μm.

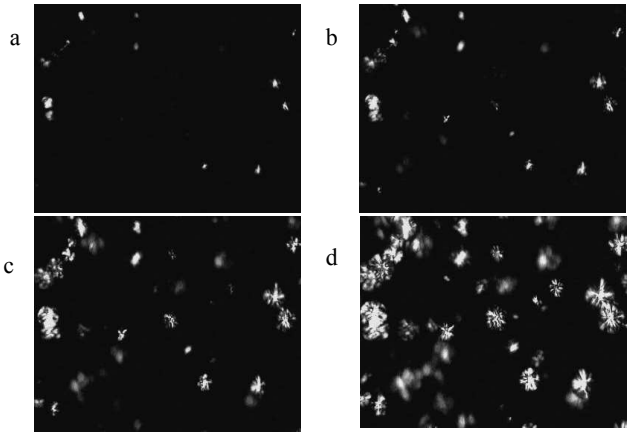

Fig. 1. Structure of PP observed on optical microscope during cooling in polarized light with magnification x 9 and temperature: a) 125 ºC, b) 124 ºC, c) 123 ºC, d) 122 ºC

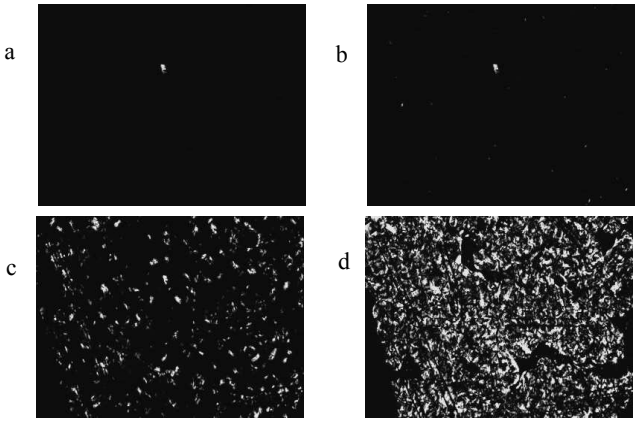

Fig. 2. Structure of PP + 15 % the glass fibre observed on optical microscope during cooling in polarized light and temperature: a) 147 ºC, b) 121 ºC, c) 117 ºC, d) 110 ºC

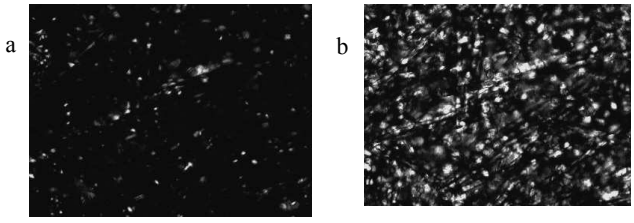

Fig. 3. Structure of PP + 25 % the glass fibre observed on optical microscope during cooling in polarized light with magnification x 9 and temperature: a) 128 ºC, b) 126 ºC

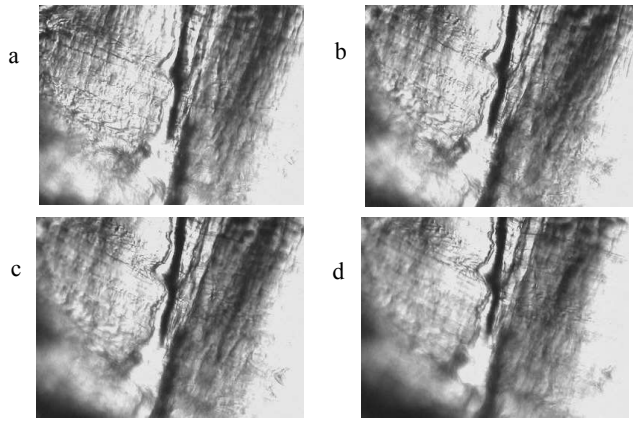

Fig. 4. Structure PP observed on optical microscope during heating in white light with magnification x 9 and temperature: a) 30 ºC, b) 60 ºC, c) 90 ºC, d) 130 ºC

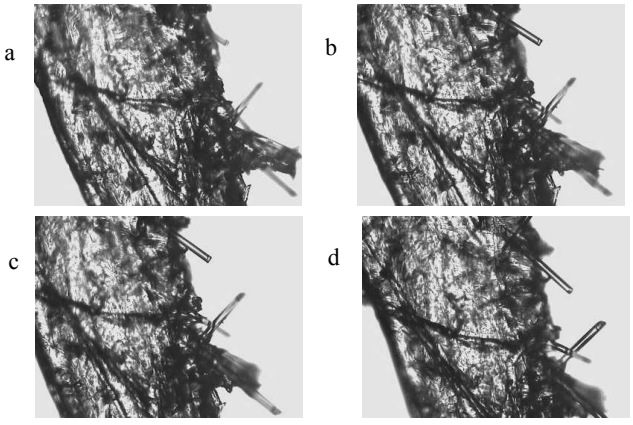

Fig. 5. Structure of PP + 15 % the glass fibre observed on optical microscope during heating in white light with magnification x9 and temperature: a) 30 ºC, b) 60 ºC, c) 90 ºC, d) 130 ºC

a b

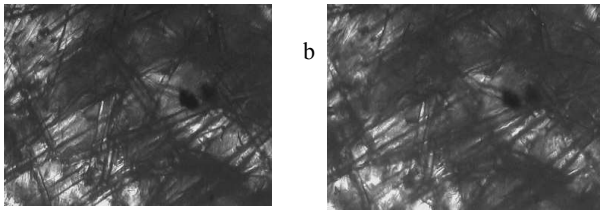

Fig. 6. Structure of PP + 25 % the glass fibre observed on optical microscope during heating in white light with magnification x9 and temperature: a) 30 ºC, b) 60 ºC

4. Conclusions

A result of the carried out tests the selected physical properties were determined, i.e. melting and crystallization temperature and structure of composites obtained in different conditions of the melting and crystallization process. Also the evaluation of proper applying of fibre filler as the factor nucleation the crystallizing process of polypropylene has been done. It was stated also, that the applied fibre filler in form of glass fibre type E, nucleation and modifying the crystallization process of polypropylene, changing simultaneously in significant way the morphologic structure of polymer. On the base of the examination of structure carried out on polarizing microscope together with the computer system recording of picture, the melting and crystallization temperature were defined for the following:

- polypropylene,

T_{top} = 168 ^{0}C T_{kryst}= 127-117 ^{0}C

- polypropylene + 15 % glass fibre,

T_{top} = 167 ^{0}C T_{kryst}= 129,7-122 ^{0}C

- polypropylene + 25 % glass fibre,

T_{top} = 168 ^{0}C T_{kryst}= 129,9-123 ^{0}C

Additionally for some of the samples size of objectives registered on microphotography were determined:

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- polypropylene + 15 % glass fibre – spherolites ~ 52 μm, glass fibre ~ 15,6 μm,
- polypropylene + 25 % glass fibre – spherolites ~ 42-51 μm, glass fibre ~ 13-14 μm,

Polypropylene composites with glass fibre showed slightly higher crystallization temperatures.

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