WORKBOOK

RESEARCH METHODS
OF POLYMER MATERIALS

LUBLIN 2014

KAPITAŁ LUDZKI
NARODOWA STRATEGIA SPÓJNOŚCI

UNIA EUROPEJSKA
EUROPEJSKI
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Projekt współfinansowany ze środków Unii Europejskiej w ramach Europejskiego Funduszu Społecznego

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1. ABSORPTION

1.1. Introduction

As a result of water and oil absorption of certain polymers, an increase in material's volume and decrease in intermolecular binding strength occurs. The consequences of this include a decrease in elasticity, hardness, strength, reduction of temperature, vitrification, deterioration of dielectric properties and material anomalies.

Measurement method of water and oil absorptiveness is based on determination of change in mass, in linear dimensions and in chosen mechanical properties that occur by the action of water or oil, without external loads affecting the sample. It is used for solid materials and porous materials, but the measurement of boiling water absorption is not used in the case of materials that change shape in the temperature of 100^{o}C.

Water absorptiveness according to PN-EN ISO 62 [9] is defined as a ratio of water mass absorbed by the sample to the mass of this sample in a dry state, and it's expressed in percentages by weight. The rapidity of water absorption by the polymers materials is very low. Highly absorbable are the materials containing numerous polar groups e.g. cellulose, whereas practically non-absorbable are the ones with non-polar molecules e.g. polyethylene. Determination of water absorptiveness is used for solid materials, as well as porous materials. The designation may also include the materials containing water-soluble substances. We can divide the research methods of water absorptiveness on the ones conducted in cold water and the ones conducted in boiling water. The method can also be used when the studied material contains substances soluble in water. The choice of oil, time and temperature of the test should be appropriate in regard to the application conditions of the product made out of the given material.

The measurement of oil absorptiveness is a process of weighed and measured samples held in oil for 7 days. Type of oil and its temperature are selected depending on the application conditions. If the above-mentioned period of time is too short to assess the resilience, then the test is extended for a period of time that is a multiple of 7 days. Subsequently, the samples are washed with detergent, rinsed three times in running water and distilled water, dried with the use of blotting paper, measured and weighed. Afterwards, the samples are dried in the temperature of 50±2^{o}C to a constant mass and placed in the desiccator achieving the

temperature of 23±2°C, and then the mass, as well as dimensions of the sample are determined again. Subsequently, the following changes are calculated: change of sample mass after the immersion, change of dimension after the immersion, loss of mass after the drying and conditioning, change of dimension after the drying and conditioning.

1.2. Purpose of the exercise

The purpose of this exercise is to learn the research methods of polymer materials absorption. Also, this exercise aims to determine the water and oil absorption for at least two different thermoplastic materials or materials that contain organic fillers.

1.3. Test stand

Test stand for conducting this exercise includes the following elements:
- analytical balance with weighing precision of 1 mg, type PRL TA14
- calliper, micrometer,
- glass or enamelled dish with a capacity of 0,5 - 1,0 dm^3,
- thermal chamber, thermostat maintaining the temperature with accuracy of ±2°C,

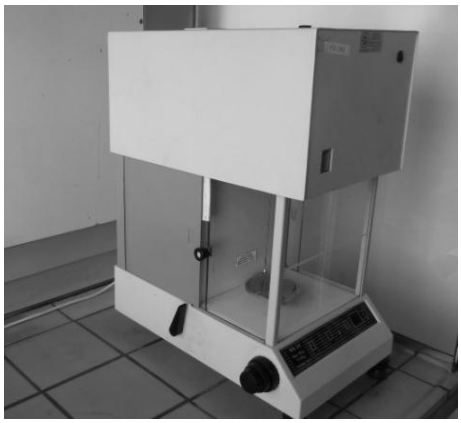


Fig.1.1 Appearance of analytical balance PRL TA14 and test samples during the water absorption test

1.4. Program of the exercise

In this research program the following factors were assumed as factors examined:
a) directly:
- mass of the sample before immersion in water, m_1, mg,
- mass of the sample after removal from water, m_2, mg
- surface of the sample, A, mm^2
- mass of the sample before immersion in oil, m_3, mg
- mass of the sample after removal from the substance and drying with blotting paper, m_4,

- mass of the sample after removal from the substance, drying and conditioning, m_5, mg
- linear dimension of the sample (length or width) measured before the immersion, l, mm
- linear dimension of the sample measured after the immersion and drying, l_1, mm

b) indirectly (consequential factors):

- amount of water absorbed by the sample, x_1, mg,
- amount of water absorbed by surface unit of the sample, x_2, mg/mm^2,
- amount of water absorbed by the sample, x_3, %,
- change in mass of the sample y_1,%,
- change in the specified dimension of the sample after immersion in oil, y_2, %,
- change in mass of the sample after drying and conditioning y_3, %.

As a variable factor, types of the tested samples from polymer materials were assumed.

Determination of resistance to oil must be carried out in accordance with the requirements of PN-EN ISO 175 [10] Plastics. Methods for determining the effects of immersion in liquid chemicals: Before the designation, the samples are dried in temperature of 50±2^{o}C in vacuum dryer for 24±1 h, and then they are cooled in a desiccator in temperature of 23±2^{o}C. After cooling, the samples are weighed with precision of 1 mg and measured with the accuracy of 0,01 mm. After weighing, the samples are placed in oil (e.g. in engine mineral oil Lotos 15W/40) for a period of 7 days. Then, they are removed, washed with a detergent, rinsed three times in running and distilled water, dried with blotting paper, weighed and measured. Afterwards, the samples are dried in temperature of 50±2^{o}C to a constant mass, cooled in a desiccator to the temperature of 23±2^{o}C, and then measured and weighed.

Fig.1.2 Appearance of the test samples during oil absorption test

1.5. Results, report

The results of measurements and tests must be summarized in tables that can be found in the report template. Graphic interpretation of the results of conducted tests, surface roughness for various types of samples must be presented in the form of block diagrams. Correctly prepared report should contain: purpose of the exercise, proceedings of the exercise, results of

measurements and calculations of water and oil absorption summarized in tables, conclusions arising from the conducted exercise.

2. HARDNESS

2.1 Introduction

The hardness in reference to polymer materials is supposed to be understood as resistance of the material during insertion of vertically loaded indenter (in the shape of ball, cone or pyramid). The hardness may also be specified as a resistance of the material to local permanent deformation, caused by the action of the load focused on a small surface area. Hardness measuring methods are reference methods and they are of indirect nature. The variety of hardness measurement methods makes the results that are obtained in various manners, in most cases incomparable between different methods. Similar situation occurs when one method allows different types of load and shapes of indenters. Therefore, comparison of the hardness is only possible in range of one method or in case of methods that have the same measurement principle.

Hardness measurement methods are divided into two basic groups:

- used to measure hardness of the materials in highly elastic state, elastomers and very soft materials, for example: Shore method, IRH method, Schiltknecht method and Richardson method,
- used to measure hardness of the materials in glassy state, for example: ball indentation method, Brinell method and Rockwell method.

When measuring hardness of the materials in highly elastic state and elastomers, the concept of hardness is quite arbitrary, therefore such concepts as rubber elasticity (designated by Shore method) or rubber softness (designated by Schopper method) have been introduced.

Hardness in Shore method

Principle of hardness measurement in Shore method is based on determining the resistance that is exhibited by the tested material during insertion of the needle with a standardized shape and dimensions. The resistance is measured after 3 seconds since the start of insertion, with the help of a spring with known mechanical characteristics. The unit of hardness is arbitrary and is expressed in Shore degrees (^{o}Sh) in the range from 0^o (maximum insertion of the needle) to 100^o (no needled insertion in the material). There are several

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hardness scales determining the rubber hardness. The differences (obviously aside from the results) are due to various shapes of indenter used in measurements and different load values. For A method, a load of 10 N is used, and for the Shore durometer of type C and D used for elastomers, thermoplastics and their composites - a load amounts to 50 N. Such division is an effect of different method application e.g. type A is used for elastic materials, while type C and D are used for harder materials. Reading „0" in Shore scale A means a material with water-like softness, and reading „100" means an incredibly hard material.

Preparation of a sample for measurement requires a disk-shaped material with thickness of at least 6mm. Measurements, like in other methods, should be conducted in certain distance from the sample's edge (in this case 12 mm) in three places that are located at least 5 mm from each other. The measurement is carried out through insertion of needle-shaped indenter into the tested material. After a few seconds, a reading from 100–degree scale should be performed. The result is inversely proportional to indenter's insertion. Such measurement is carried out in accordance with PN-EN ISO 868 [11]. An exemplary device for the hardness measurement with Shore method is measuring apparatus manufactured by Zwick in analogue and digital version. Medium hard and hard elastomers - Shore C, hard elastomers and thermoplastics - Shore D. Type of measurement depends on the shape of indenter.

Fig. 2.1 Devices for measuring the hardness with the use of Shore method, a –Zwick 3130, b - Sauter HTB 100

2.2. Purpose of the exercise

The purpose of this exercise is to learn the determination methods of polymer materials' hardness. Also, this exercise aims to determine the hardness (using Shore method) of three different materials from elastomers group, glassy materials and porous materials.

2.3. Test stand

Determination of hardness using Shore method is carried out with the use of manual durometer manufactured by Omag Affri System s.a.a. model 13. The basic element of the durometer is a needle-shaped indenter with ending in the form of: truncated cone (in the case of scale A) or rounded cone (scale D and OO). The body of this device contains: a spring of known mechanical characteristics, which burdens the needle; and gearing mechanism that transfers the rectilinear motion of the needle to rotary motion of the pointer, which shows the measurement result on the scale. Lower part of the body includes a button that through lever mechanism causes the pushing of the subject table with sample into the needle. Appearance of the durometer for hardness measurement using Shore method is shown in Fig. 2.2.

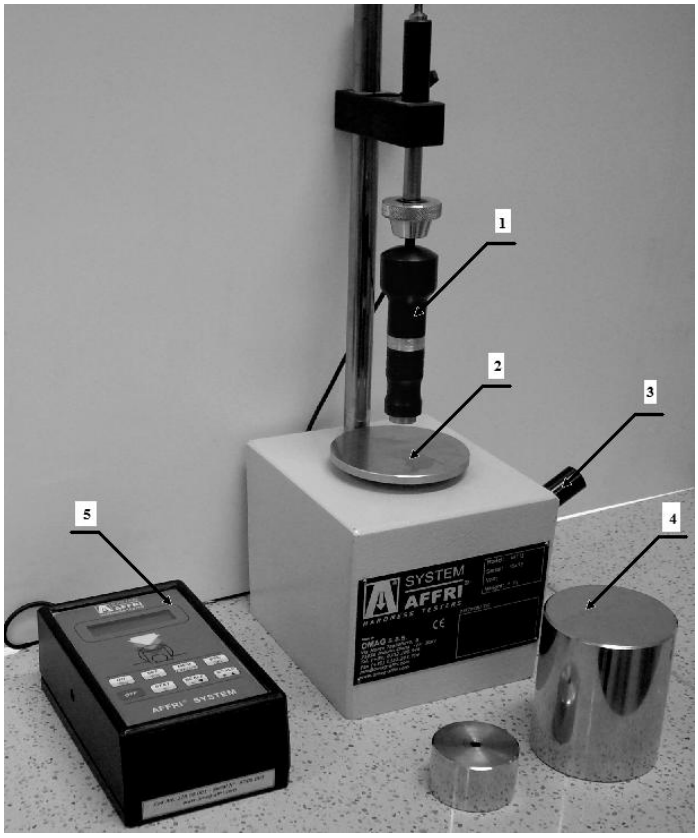

Fig. 2.2. Shore durometer Omag Affri System s. a. a. model 13: 1 – measuring head, 2 – measuring table, 3 – lever, 4 – measuring load, 5 – digital measuring unit.

Shore durometer used in the test has digital system that processes data from measuring device into a digital signal, with backlit reader for all Shore's scales and automatic recognition of the connected measuring head – it enables the selection of hardness scale and has the ability to store up to 100 measurement results. Also, it has three modes for printing the results, static function with a bar graph, it communicates with the computer via interface,

provides instant reading of the results and is equipped with lightweight measuring head with ergonomic handle.

2.4. Program of the exercise

In this research program the following factors were assumed as factors examined:
1) directly: thickness and cross-sectional area of the test sample, mm,
2) indirectly: hardness of the samples, ^{o}Sh
Variable factor is:
- type of the tested material (minimum three types of materials from elastomer group, glassy materials, porous materials).
- type of measuring head, for tests using method A, D, OO,
- type of measuring load, for tests using method A, D, OO.

2.5. Carrying out of the exercise

The essence of the hardness test using Shore method is pushing of the indenter into the tested sample at a specific measuring load. Shape of the indenter and value of measuring load is closely associated with the type of tested material. In the case of Shore OO method for porous materials, the indenter has semicircular shape and the value of the load is very small. In the case of Shore A method, you need to use intender in a shape of truncated cone with standardized dimensions and load that is listed in the standard. In the case of Shore D method, the intender is in the shape of a cone and value of the load is higher than in the previous methods. This is due to application of the mentioned method for hardness measurement in glassy materials of high hardness.
Result of the hardness measurement with the use of Shore method is hardness in Sh^{o} degrees, a value that corresponds to the inverse of indenter's insertion under the affecting constant force. The maximal insertion is a depth of h = 2,5 ± 0,004 mm, which is assigned with a value of 0^{o} Sh.

2.6. Results, report

The results of measurements and tests must be summarized in table 2.1 that can be found in the report template. Graphic interpretation of the results of conducted hardness tests for different types of material must be presented in the form of block diagrams. Correctly prepared report should contain: purpose of the exercise, proceedings of the exercise, results of hardness measurements summarized in tables, interpretation of the results in the form of graphs, conclusions arising from the conducted exercise.

3. TENSILE STRENGTH

3.1 Introduction

Tests regarding tensile strength properties of the materials are one of the main sources of information about the mechanical properties of the materials. Also, they are often used for indirect assessment and research regarding other properties and quality of materials. Obtained results are used for example in the selection of proper material during product design.

Behavioural characteristic of the material during stretching is adequately presented by so called tensile graph (obtained during the tensile test), which shows the course of relations between the two variables: tension and deformation or load and prolongation. Shape of the graph depends mainly on a type of the material, and the basic property of the materials is a small range of rectilinearity of this dependency.

Tensile test is normalized [12] and consists of uniaxial deformation of properly prepared samples and measuring of the resulting forces. Measured values include deformations (prolongations ε) and deforming forces.

Strength testing machines due to their versatility are used for measuring tensile, compression and flexural strength.

Fig. 3.1. Two-column strength testing machines manufactured by: a) Instron, b) Shimadzu [www.instron.com, www.shimadzu.com]

3.2. Purpose of the exercise

The purpose of this exercise is to learn the testing methods of polymer materials tensile strength. Also, this exercise aims to determine the specified tensile strength properties of products made from polymer materials.

3.3. Test stand

Determination of materials strength properties during static stretching is conducted with the use of strength testing machine manufactured by Zwick Z010 (Fig. 3.2).
Strength testing machine consists of the tensile system (including steel frame, two handles, movable bar), device for the measurement of tensile load affecting the sample and the extensometer. Material sample is mounted in the fixed top handle (2) of the machine, attached to the frame, and in the bottom handle (3), fixed to the movable bar (4) that slides along the frame (1).

Fig. 3. 2. Appearance of the strength testing machine, along with the accessories during static stretching of the materials: 1 – steel frame, 2 – top handle, 3 – bottom handle, 4 – movable bar, 5 – control unit, 6 - computer station

Control unit (5) and computer station (6) are used for electronic control of the machine operation. Strength testing machine Zwick enables the operation with maximum tensile force up to 10 kN and tensile speed up to 200 mm/min.

Samples of the material are placed in the machine's handles (Fig. 3.3) and during the measurement, while the load is increasing, you need to continuously record both the load and prolongation of measuring section of the sample. From the curve obtained in strength-prolongation system, you can read the characteristic values and use them to determine strength properties of the material. Appropriate software enables the direct visualization of relation between tension and deformation, as well as all other strength properties on the computer screen.

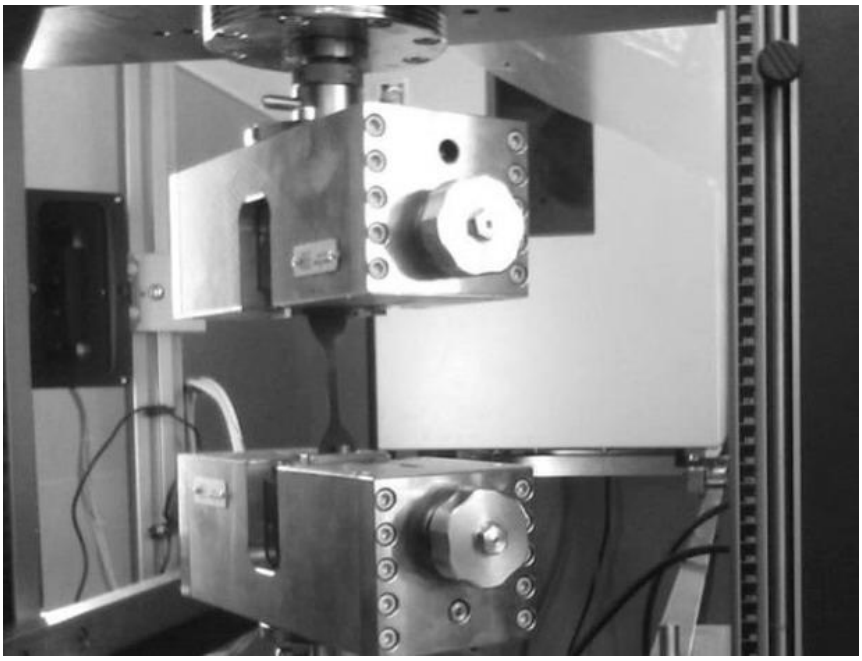

Fig. 3.3. Measuring system Zwick Z010 machine with the test sample of films

3.4. Program of the exercise

In this research program the following factors were assumed as factors examined:

1) directly
   - cross-sectional area of strength test sample, A, mm,
   - maximum tensile force, F_M, N,
   - force at which the sample broke, F_B, N,
   - change in measuring section length in the moment of breaking Δl_B, mm,
   - change in sample section length at maximum tensile force, Δl_M, mm,

2) indirectly

- tensile strength (maximum tensile tension), σ_M, MPa,
- tension in the moment of breaking, σ_B, MPa,
- relative deformation in the moment of breaking, ε_B, %
- relative deformation at maximum stretching tension, ε_M, %.

Variable factor is:

- type of the tested material (minimum two types of material or one type of material with fillers).
- technological conditions of obtaining the products by extrusion of injection process

3.5. Carrying out of the exercise

In order to properly carry out this exercise you must perform the following actions:

1. Prepare the sample in the form of little paddles, obtained through the injection process or cut the sample using little press, at least 3 samples for each type of material, for tensile strength tests.
2. Determination of tensile strength properties must be conducted using strength testing machine along with necessary accessories, in accordance with the following standard: PN-EN ISO 527-1: 2010. Plastics. Determination of mechanical properties in relation to static stretching.
3. Measure the thickness, as well as width of samples' measuring section and calculate initial surface A of samples' cross-section.
4. Fix the first sample in the top and bottom handle of the strength testing machine.
5. Set the desired tensile speed and start the strength testing machine, while examining the recording of the load/prolongation graph, until the breaking moment of measuring sample.
6. Determine the factors examined directly and indirectly, in accordance with the exercise program.
7. Repeat these actions for other samples – minimum of 3 samples for each type of material.

3.6. Results, report

The results of measurements and tests must be summarized in table 1 and 2 that can be found in the report template. Graphic interpretation of the tests results conducted for tensile and stress strength in respect to relative deformation, for different types of materials must be presented in the form of block diagrams.

4. FLEXURAL STRENGTH

4.1 Introduction

Bending occurs when the outer forces affecting the beam that are perpendicular to its axis, can be replaced with resultant pair of forces, lying on the plane passing through its axis. The moment of this pair of forces is the bending moment M_g in the given cross-section.
Bending test consists of placing the material sample on the endings and exerting point-wise load, perpendicular to the longitudinal axis and symmetrically in regard to the supports. The distance between the support points must be closely defined and standardized [13]. Such test is used only in
regard to brittle (but stiff) materials, which break during the test. The tensions that occur during bending (destructive tensions) in the moment when sample breaks, are called flexural strength $б_g$.
In the case of homogeneous samples, the distribution of tensions as a function of their height has linear nature within the range of elastic deformations (Fig. 4.1). Beyond this range, neutral layer of the sample moves up or down, and the relation between tension and deformation becomes curved.
This method is used for unfilled and reinforced with fibrous fillers rigid polymer materials, in addition to porous materials.

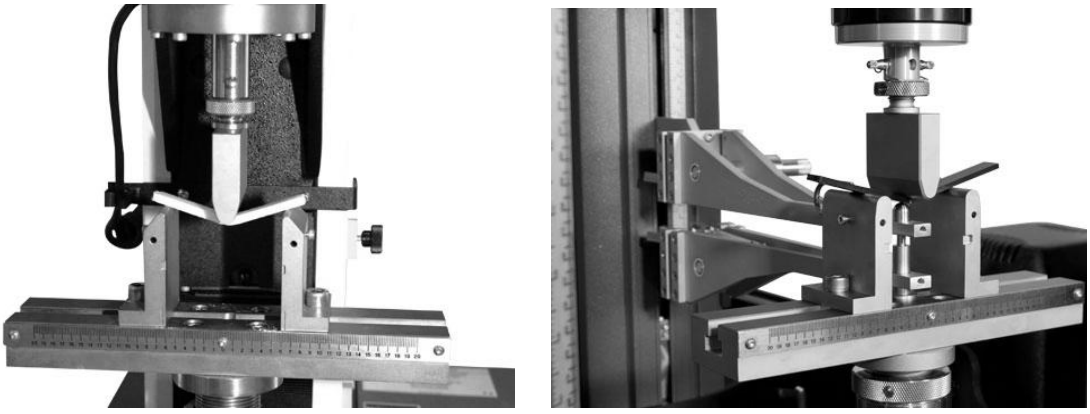

Fig. 4.1. Distribution of tensions in the freely-supported bended beam – example of bending [www.instron.pl]

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During the bending under applied force (F), the top surface of the sample gets smaller, and bottom part gets prolonged. In result of this, in the top part compressive tensions occur and in the bottom part tensile tensions occur. In the middle part, the tensions compensate themselves. Destruction of the sample usually takes place in the middle narrow part, in which due to small cross-section the tensions are the greatest. The middle part is affected by the biggest deformation.

In the used machine, usually the three-point flexural strength tests are implemented, which are described in the following standards: ISO 178 and ASTM D 790. Typical test results, besides flexural strength σ_g, provide bending module E, tension at 3,5 % deformation and tensions, as well as prolongations at plasticity limit and at breaking of the sample. Results of the bending test particularly show behaviour of the material, near the sample's surface. In comparison to the breaking test, measured inflections are approximately four times greater, than length changes in the breaking test.

Strength testing machines can compensate the deformation of device frame, force sensor and bending tool with the use of specialized software. This enables sufficiently precise measurement using the beam movement sensor of the strength testing machine. The handling of the strength testing machine is therefore simplified, particularly when used in quality control tests.

4.2. Purpose of the exercise

The purpose of this exercise is to learn the test methods of polymer materials flexural strength. Also, this exercise aims to determine the selected flexural strength properties of products made from polymer materials.

4.3. Test stand

Test stand necessary to carry out this exercise includes strength testing machine - Zwick Z010 with accessories and computer station equipped with testExpert II software.

Measurement samples must be prepared in accordance with the requirements provided in the standard applicable to the tested material.

Dimensions of samples used for cuboidal measurements are as follows:

a) length l= 80mm
b) width b= 10±0,5mm
c) height h= 4±0,2mm

Strength testing machine manufactured by Z010 has been described in detail in workbook 3 "Tensile strength". The difference in structure consists of implementation of special system for testing flexural strength, which is shown in Fig. 4.1.

Control unit and computer station are used for electronic control of machine operation. Operation of the bending system is controlled by the computer; and software (as well as machine accessories) enables you to carry out the bending test. Appropriate software enables the direct visualization of relation between tension and deformation, as well as all other strength properties on the computer screen.

4.4. Program of the exercise

In this research program the following factors were assumed as factors examined:

1) directly
   - cross-sectional area of strength test sample, A, mm,
   - distance between the supports of the bending system, l_r, mm,
   - bending force F_g, N,
2) indirectly
   - indicator of cross-section resistance to bending *W*, mm^3,
   - bending moment M_g, Nm and bending tension σ_g, MPa,
   - coefficient of longitudinal elasticity during bending, E_g, MPa

Variable factor is:
- type of the tested material (minimum two types of material).

4.5. Carrying out of the exercise

During determination of flexural strength properties, you must conduct the following actions:

1. Measure the thickness, as well as width of samples' measuring section and based on that calculate surface A of samples' cross-section.
2. Fix the first sample in the bending system of the machine.
3. Set the desired tensile speed and start the strength testing machine, while examining the recording of the load/prolongation graph, until the breaking moment of measuring sample
4. Determine the factors examined directly and indirectly, in accordance with program.
5. Repeat these actions for other samples – a minimum of 3 samples for each type of material.

4.6. Results, report

The results of measurements and tests must be summarized in tables that can be found in the report template. Correctly prepared report should contain: purpose of the exercise, proceedings of the exercise, results of measurements and calculations for flexural strength summarized in a table, conclusions arising from the conducted exercise.

5. IMPACT STRENGTH

5.1. Introduction

Impact strength tests belong to the group of dynamic test methods of polymer materials properties. Impact strength is a measure of materials' brittleness, and its test is based on determination of the work required for dynamic destruction of the sample per unit of surface area of its cross-section. There are several known methods for testing the impact strength, but three of them are of utmost importance: Charpy method, test on Dynstat device and Izod method. These methods use pendulum hammers as measuring instruments.

Charpy method

One of the most popular methods for measuring impact strength – Charpy method is based on breaking the sample that is shaped as cuboidal beam, placed horizontally and supported on both side, with one stroke of pendulum hammer to middle of the beam. During impact strength measurement, you determine the work used for dynamic breaking of the sample in relation to its initial cross-sectional area. The results obtained from these methods are not comparable in regard to each other and can't be converted into each other. Conditions for applying the impact strength test using Charpy method are specified in the following standard: PN-EN ISO 179 [15].

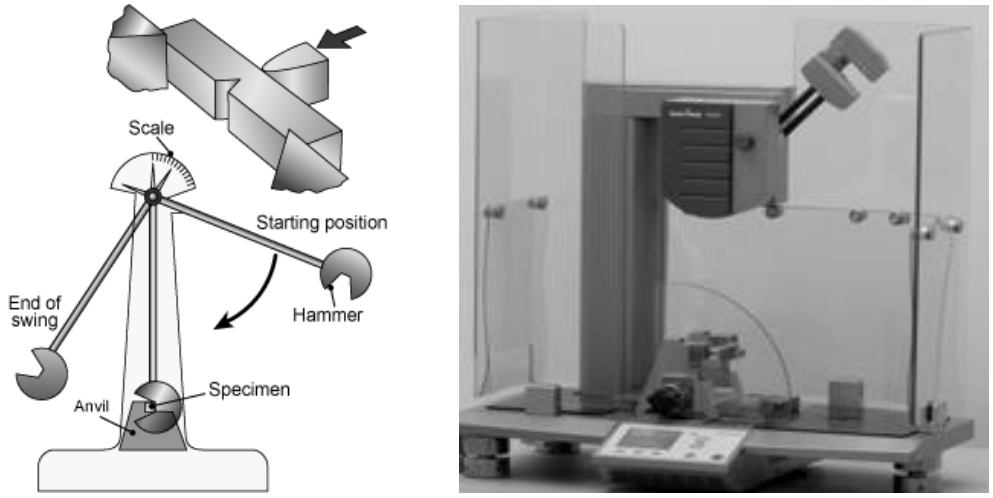

Fig.5.1. The principle of impact strength designation with the use of Charpy method; an example of pendulum hammer manufactured by Zwick; general appearance and during the impact strength test using Charpy method.

This standard provides provisions for applying impact strength tests of stiff polymer materials using two methods: to samples with notch and without notch. In the case of applying the impact strength test to samples with notch, the stroke should be directed at a sample surface located opposite the notch. Schematic of impact strength principle using Charpy method is shown in Fig.5.1.

The hammer is set in vertical position and a check is conducted - whether hammer released without the sample shows energy 0, then it is set in vertical position again. Cross-section of the sample is measured in the place of the stroke with precision of ±0,1mm. The sample is placed on the supports and the indicator of the driver is set to maximum scale value. In order to test the sample with notch, the hammer should strike the surface opposite to the notch. The hammer must be released carefully and without tremors from the side, where splinters do not fall and then you must read used energy indicated by breaking of the samples.

Izod method

Impact strength test using Izod method is usually conducted on the same pendulum hammers, which are used for Charpy method. Izod method differs from Charpy method in regard to the fixing manner of the sample, sample dimensions and impact velocity of the pendulum hammer. This method is applied only to impact strength tests of samples with notch. The notch has a triangular cross-section with the aperture angle of 45º and depth of 2,5 mm, and thus it requires milling with appropriate cutting tool. Impact strength of the samples with notch is determined analogically to Charpy method, using the relations specified in the standard [16].

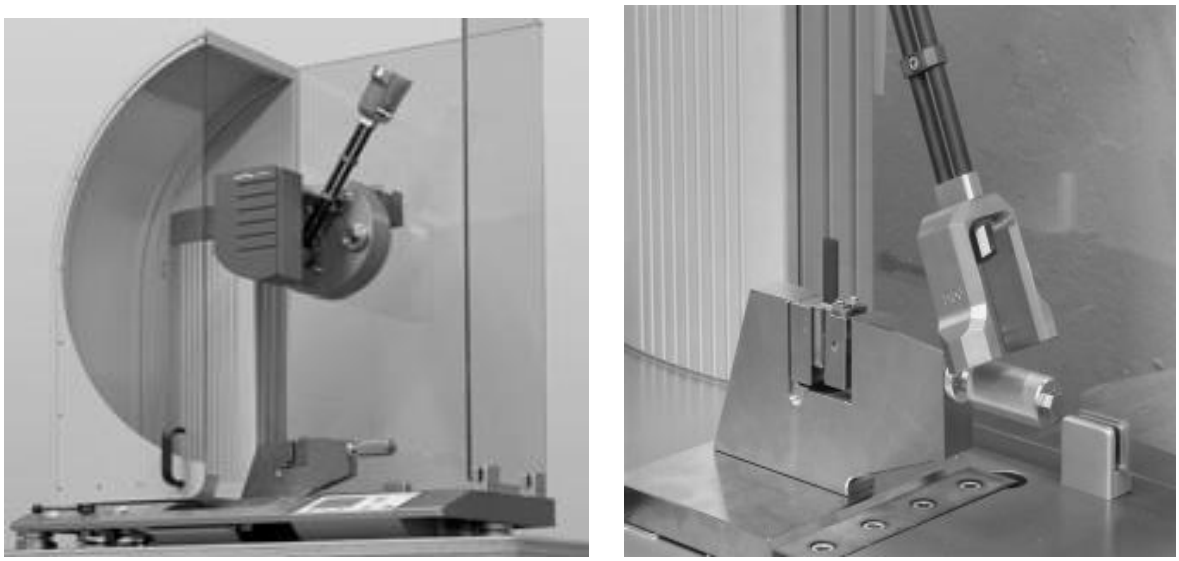

Fig. 5.2. Example of the pendulum hammer manufactured by Zwick; general appearance and during impact strength method using Izod method

In the Izod method, the hammer suspended on the axis moves as free pendulum, and its scale is equipped with driver fulfilling the role of indicator. The hammer falling from position set at the top makes a full rotation and the indicator should show the value 0, because the hammer did not perform any work, besides overcoming air resistance. In order to make the hammer perform work, we must fix a beam on the supports that have 70 mm distance between them. Then, the falling hammer will break it, which absorbs adequate energy, and the indicator will show work used to break the sample. The ratio of the pendulum mass to the frame is very high and allows measuring 80% of the used energy with high accuracy. The frame is made of cast iron to suppress vibrations. Measuring equipment can be place on any table that has appropriate lateral stability. The pendulums can be easily replaced using the release mechanism and this does not require the use of any tools. Automatic recognition of the pendulum using codes eliminates occurring of the errors.

Impact stretching

This test can be defined as a stretching test with relatively high speed of deformation and its conditions are specified in the following standard: PN-EN ISO 8256 [17]. The method of impact stretching can be used to test stiff materials in accordance with ISO 472 standard, but also in regard to the materials that are too flexible or too thin to test their impact strength with the use of Charpy method or Izod method.

Breaking of the sample is done by a single stroke in the lowest movement position of the pendulum. The sample in the moment of breaking is arranged horizontally (Fig.5.3). During the stroke, one end of the sample is held either by the arm or by the pendulum, whereas the second end is held by crosshead handle. Breaking energy E_z is determined based on kinetic energy released by the pendulum during breaking of the sample. You also need to make the corrections that take into account recoil energy (method A) or rebound energy (method B) of the crosshead handle.

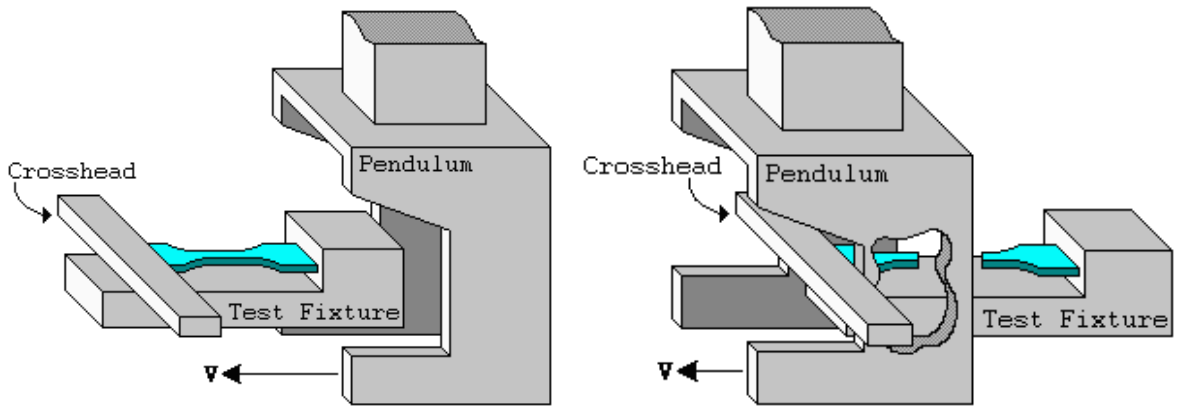

Fig. 5.3. Principle of impact stretching [www.ides.com]

According to the standard [17], five types of the samples can be used for impact stretching tests. The samples are prepared in accordance with guidelines of appropriate standards concerning methods of their preparation and using the recommendations given in the detailed subject standards concerning tested type of the material. For example, the samples made from thermosetting materials are prepared using the press method, and samples made from thermoplastic materials are prepared using injection of extrusion.

5.2. Purpose of the exercise

The purpose of this exercise is to learn the principles of strength impact tests using Izod method and impact stretching. Also, this exercise aims to determine the impact strength properties using Izod method and impact stretching, while applying at least two different thermoplastic materials or materials that contain organic fillers.

5.3. Test stand

Determination of impact strength properties using Izod method and impact stretching properties is carried out with the use of impact hammer manufactured by Comatech Testing Machines,
type 639F. Figure 5.4 shows general appearance of the equipment (handles, pendulums) used for carrying out the impact strength tests with the use of discussed methods.
In order to conduct measurement of the impact strength, you need to select a pendulum hammer (indicated by the lecturer) that has appropriate reserves of energy and desired speed, so that for the breaking of sample it will use not less than 10% and not more than 80% of its energy supplies. For example, in the case of Izod method you need to use pendulum with energy of 2,75 or 5,5 J [16].

Prior to starting the measurements, you need to run control and measurement software of pendulum hammer type 639F. In addition, it's necessary to conduct trial tests without the measuring samples, in order to check whether the total friction losses are not greater than acceptable limits specified in the standard.

According to the standards, the tests must use samples of appropriate types, as recommended by the lecturer. The samples must be prepared in accordance with guidelines of appropriate standards concerning the method of their preparation, with the use of injection, press or extrusion process and pursuant to the requirements of ISO 3167 or ISO 294 standard. Samples from resins are obtained by casting or cutting from cast plates, and in the case of the finished products, samples are obtained by cutting and machining.

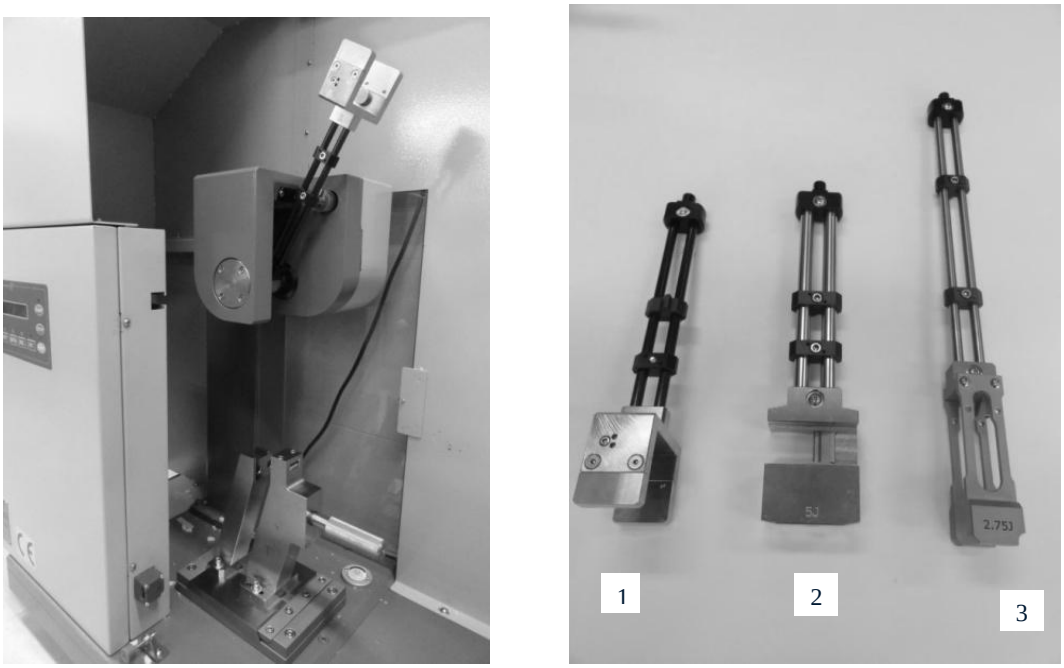

Fig. 5.4. Impact hammer manufactured by Comatech Testing Machines, type 639F: a) appearance of the hammer, along with the accessories for impact stretching tests, b) impact pendulums for impact strength tests using: 1- impact stretching method, 2- Charpy method, 3- Izod method

After carrying out the measurements of the sample, it is placed on the device supports in a manner ensuring that the hammer's blade will hit in the middle of the sample. In the case of sample with notch, it must be placed in a manner ensuring that the middle of the notch is located exactly in the plane of hammer blade movement. If the impact strength measurement is conducted using the method with notch, then the sample is placed on the supports in a way ensuring that the hammer pendulum will hit the surface opposite to the notch. When testing the samples with non-standard dimensions (e.g. samples cut from the plates with a thickness below 10 mm), you must place steel plates behind the supports that have thickness, which will supplement the thickness of the samples to the standard dimensions. After placing the samples on supports in the described manner, you release the hammer lock, but this action must be conducted carefully and without vibrations, which can affect the position of the plate and accuracy of determination of energy absorbed by the sample during hammer stroke.

5.4. Program of the exercise

In this research program the following factors were assumed as factors examined:

1) directly

- width of the sample in Izod method, b_N, mm,
- thickness of the sample in Izod method, h, mm,
- width of the sample in impact stretching method, b_r, mm,

- thickness of the sample without the notch and with the notch in impact stretching method t_r, t_{rk}, mm.
- stroke energy used for breaking of the sample in Izod method, W_B, kJ,
- stroke energy during impact stretching, E_s, kJ,

2) indirectly

- impact strength with notch in Izod method, a_{cN}, kJ/m^2,
- impact stretching strength of the samples without the notch a_{tU}, kJ/m^2,
- impact stretching strength of the samples with the notch a_{tN}, kJ/m^2.

Variable factor is:

- type of the tested material (minimum two types of material or one type of material with fillers for each used method).
- technological conditions for obtaining the samples using extrusion or injection process

5.5. Carrying out of the exercise

In order to properly carry out this exercise you must perform the following actions:

1. Prepare the samples in the shape corresponding to the applied method of impact strength measurement, obtained in the injection process or using little press cut the samples, minimum 3 samples for each type of material – for impact strength tests using Izod method and impact stretching.
2. Measure thickness and width of the measuring section of the samples and based on that calculate initial area *A* of the samples' cross-section.
3. Fix the equipment (handles, pendulum) for designation of impact strength with the use of Izod method.
4. Run control software of the impact hammer and enter measurement data for the first tested sample to the computer program.
5. Fix the first sample in the measuring handles of the machine.
6. Carry out the measurement of the impact strength in accordance with recommendations included in the control program.
7. Determine the factors examined directly and indirectly, in accordance with program of the exercise.
8. Repeat these actions for other samples, minimum 3 samples for each type of the material.
9. Repeat all described actions
10. Repeat all described actions to determine the impact strength with method of impact stretching.

Impact strength of the materials examined with the use of Izod method and impact stretching method should be determined from the dependency in accordance with applicable standard.

6. GEOMETRIC STRUCTURE OF THE SURFACE

6.1 Introduction

Surface of the products made out of the materials is constituted during processing by factors related to processed material, processing tool and conditions of the processing. Factors associated with the processed material mainly include: properties and structure of the material, as well as auxiliaries and relations between them. Factors associated with the processing tool mainly include: type of the material that constitutes the tool and state of its surface layer. Factors associated with condition of the processing mainly include: type and intensity of physical enforcements occurring during processing and cooling of the object after its formation, as well as porous process.

Geometrical structure of the product's surface is formed as a result of geometric and kinematic interaction of the socket surface forming the tool in regard to actual surface of the product and in context of changes in this structure due to relative movement of the material during processing, on the working surface of the tool.

State of the surface geometrical structure is determined with the use of surface roughness parameters, which include e.g. mean arithmetic deviation of surface roughness profile R_a, (μm), surface roughness according to 10 points R_z, (μm), maximum value of surface roughness R_m, (μm), mean spacing of surface roughness S_m, μm. Appearance of the exemplary roughness profile with measured parameters is shown in Fig. 6.1.

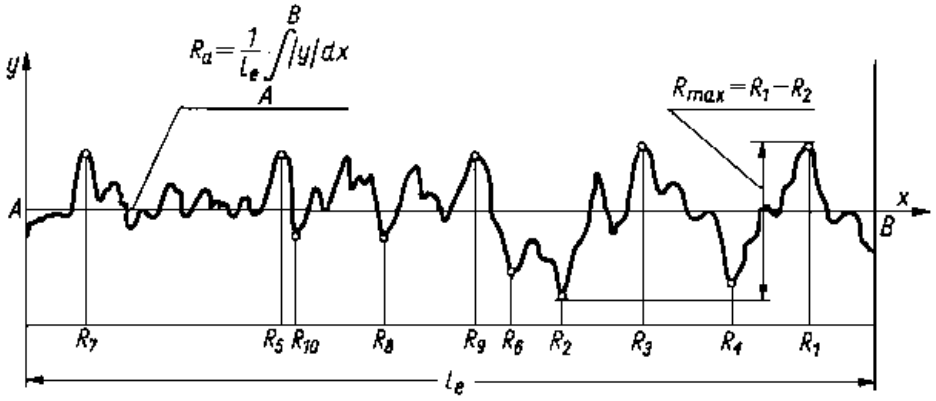

Fig. 6.1. Appearance of exemplary roughness profile with measured values: R_a, R_z, R_m (www.tribologia.org)

Tests for roughness profile are conducted with the use of test stands for roughness tests, which consist of profilographometer with accessories and software. These stands may be stationary or portable (Fig.6.2). Measuring stand enables you to carry out visualization, archiving and processing of the measurements conducted based on the applicable standard [18].

Surface roughness tests using profilographometers are carried out with contact method, which consists of determining the numerical values of the roughness profile parameters and its mapping in the form of profilograph with known horizontal and vertical magnification. In contrast to optical measuring methods, which only enable examination of small portion of the tested surface, contact method allows you to determine the surface shape of any given length.

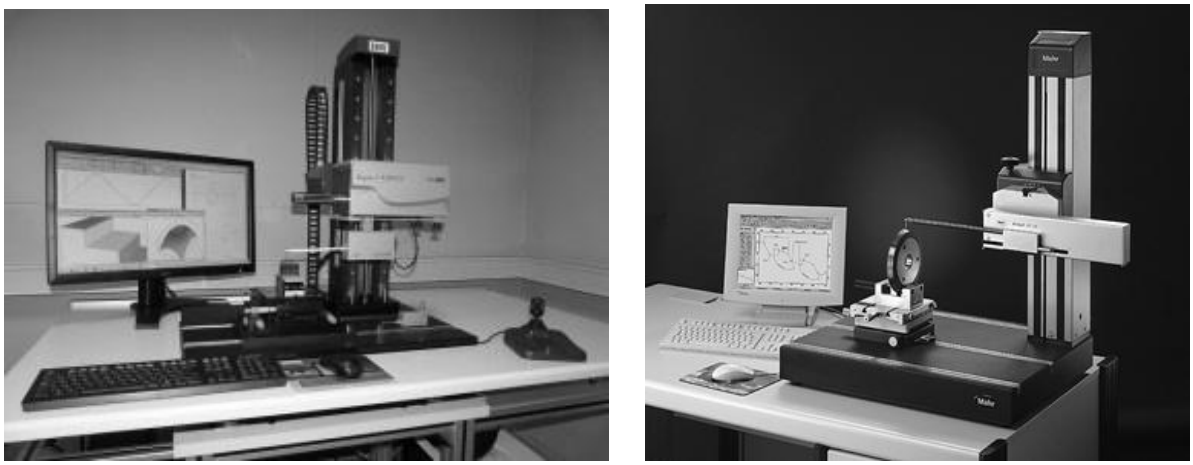

Fig. 6.2. Appearance of the stationary stands for surface roughness tests (www.ios.krakow.pl, www. komag eu. www. izasa.es)

6.2. Purpose of the exercise

The purpose of this exercise is to learn the method of surface roughness test, with the use of at least two different thermoplastic materials or materials that contain organic fillers. Also, this exercise aims to determine specific surface roughness properties of the products made out of thermoplastic materials.

6.3. Test stand

Main element of the stand for surface roughness tests is measuring device profilographmeter

TR 200. Measurements carried out using TR 200 device are based on the contact method of determining surface roughness parameters. This method consists of determining numerical values of the roughness profile parameters and its mapping in the form of profilograph with

known horizontal and vertical magnification. In order to this, a needle with known geometry is used and it moves along the tested surface at a constant speed, while its vertical movements are converted into electric signal. The amplified signal is registered in the form of profilograph, and then, after filtering the waviness, it is converted with the use of microprocessor system into numerical values of the desired roughness parameters.

Test stand for examination of surface roughness consists of the following components: Profilographometer TR-200, which enables quick determination of surface roughness parameters. The variety of the used equipment in combination with the capabilities of software enables the measurement of 13 different roughness parameters on one test stand. Test stand equipment and computer software Data View enables the cooperation with a laptop or desktop PC and suitable printer. This allows for quick management of measuring programs and saving the test results in numerical form and graphical form.

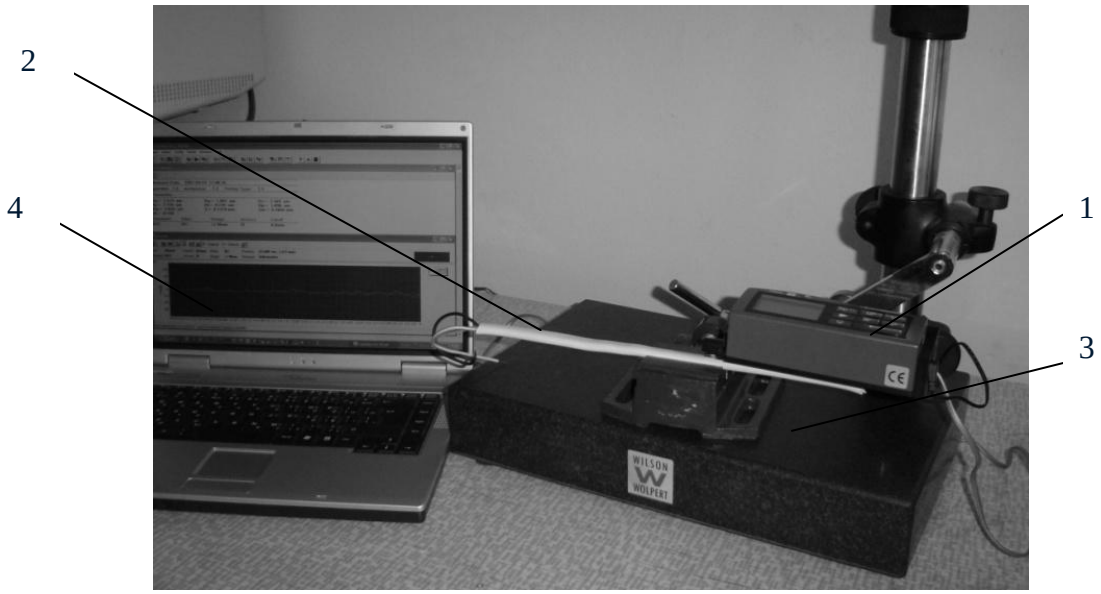

Fig.6.3. General appearance of the test stand for examination of surface roughness: 1- profilographometer, 2- measuring sample (cable), 3- granite base with the fixing arm, 4- PC

6.4. Program of the exercise

In this research program the following factors were assumed as factors examined:

a) directly

- mean arithmetic deviation of the roughness profile R_a, µm,

b) indirectly (consequential factors):

- value of surface roughness according to 10 points R_z, µm,
- maximum value of the surface roughness R_m, µm,
- mean spacing of the surface roughness S_m, µm.

Type of the tested samples surface has been adopted as a variable factor.

Constant factors include:

- structure elements of the Profilographometer TR - 200,
- external conditions – air temperature 24 ± 2°C,
- length of the measuring section $l_n = 0.8$,
- number of measurements - 5.

6.5. Carrying out of the exercise

Surface roughness tests should be carried out with contact method, which consists of determining the numerical values of the roughness profile parameters and its mapping in the form of profilograph with known horizontal and vertical magnification. In contrast to optical measuring methods, which only enable examination of small portion of the tested surface, sensor method provides graph of the surface shape in the plane that is perpendicular to it and in any given length.
During roughness measurement, the sensor is placed on the tested surface and moves along the surface driven by the drive unit that is built into the device. The sensor receives the surface roughness through sharply ended tip (probe). Roughness causes movement of the probe and therefore it causes changing of induction value of the inductor, activating analogue signal corresponding to the roughness of measured surface at the output side of phase-sensitive rectifier.
For each tested sample, you must carry out 5 measurements. All tests should be conducted using sensory gauge of the surface cross-section outline, recorded and presented in the test protocols.

6.6. Results, report

The results of measurements and tests must be summarized in a table that can be found in the report template. Graphical interpretation of the results of conducted tests for surface roughness of different types of samples must be presented in the graph form. Correctly prepared report should contain: purpose of the exercise, proceedings of the exercise, results of measurements and calculations for surface roughness summarized in a table, conclusions arising from the conducted exercise.

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7. VICAT SOFTENING TEMPERATURE

7.1 Introduction

Thermal properties of the polymers include a group of phenomena associated in physical sense with changes in functional characteristics (mechanical properties) under the influence of temperature increase. Thermal properties of the polymers are more related to constitutional and structural factors of the substance, than in the case of mechanical properties. Knowledge of the thermal properties of the materials is very important in the context of the use of the products that are made out of them. In contrast to metals and ceramic materials, these products can be used in much lower temperature range. For the most of them, the upper functional temperature is in the range of 80÷ 150 ˚C.

During temperature increase, polymer materials are affected by structural changes and chemical reactions, which intensify along with the temperature increase and can lead to thermal decomposition of the material – degradation, destruction and sometimes depolymerisation. During the use of products made from polymer materials, along with temperature change, their physical properties also significantly change, particularly mechanical properties. Also, sometimes change of physical state of the material occurs. Transition of the material from one physical state to another is characterized by the nominal temperature – vitrification temperature, softening temperature and melting temperature.

There are numerous methods for experimental determination of acceptable temperature for the use of given material. Most of them rely on the measurement of temperature at which, under given load, previously assumed material sample deformations occur, in the standardized test conditions.

The most important method used only to thermoplastic materials is a method of determining softening temperature according to Vicat [19]. This method consists of determination of temperature at which the normalized needle of the measuring device of Vicat's apparatus is inserted into the surface of measuring sample to certain depth, under the influence of the specified load and at a uniform rate of temperature increase.

Indenter of normalized dimensions (length 3 mm, circular section of the tip 1mm ± 0, 015mm^2) is inserted into the sample to depth of 1 mm, under constant temperature increase

and under the influence of the predetermined loads. Temperature at which 1 mm penetration of the material occurs is the Vicat's softening temperature (ºC).

Depending on the applied rate of temperature increase, there are two variants of softening temperature measurement according to Vicat. First (I) – is used at the rate of temperature increase 50 ± 5°C/h, and the second (II) – at rate 120 ± 10°C/h. In Vicat method we can apply load values of 10 N and 50 N, and conduct measurements at different rates of temperature increase, at 50ºC/h and 120ºC/h. Then, the whole device is placed in an oil bath. It is equipped with a heater, which automatically adjusts the temperature increase. The oil must behave neutrally in regard to the tested material and can't change its properties along with the temperature increase. The test can be conducted in glycerine or air instead of oil. Regardless of the medium, in order to ensure uniform temperature distribution, the liquid or gas must be mixed. Samples used for this test method should be in the shape of a cuboid. Width and length should be 10 mm and height should range from 3 mm to 6,4 mm.

7.2. Purpose of the exercise

The purpose of this exercise is to learn the determination method of softening temperature of the materials using Vicat method. Also, this exercise aims to determine softening temperature of the selected thermoplastic materials.

7.3. Test stand

Test stand enabling the measurement of softening temperature using Vicat method is equipped with measuring device CEAST HV3 and computer station with specialized software VisualTHERM. Test stand allows you to carry out the measurement of 3 samples simultaneously. Device CEAST HV3 consists of an instrument providing loads on samples and determining softening temperature, as well as chamber thermostat.

This device enables you to determine softening temperature under load according to Vicat (VST) and deformation temperature under load (HDT). In these tests the following standards are used: ISO 306 and ISO 75-1/-2.This device is equipped with 3 or 6 working stations depending on the type of conducted tests. Deformation test in the specified temperature conditions and load conditions is carried out with appropriate rate of heating in an oil bath. The whole device is controlled by microprocessor. This device has LVDT transducers and independent temperature sensors, which enable you to obtain very precise results with high repeatability. After the finished test, the cooling system is turned on and it restores the temperature to the value from which the test was started, enabling quick carrying out of the further tests.

Fig. 7.1. Device CEAST HV3 with computer station

Vicat method is conducted in accordance with ISO 306 standard. This method consists of determining temperature at which the indenter of normalized dimensions (length 3 mm, circular section of the tip 1mm ± 0, 015mm^2) is inserted into the sample to depth of 1 mm, under constant temperature increase and under the influence of the predetermined loads. The temperature at which 1 mm penetration of the material occurs, is the Vicat's softening temperature (ºC).

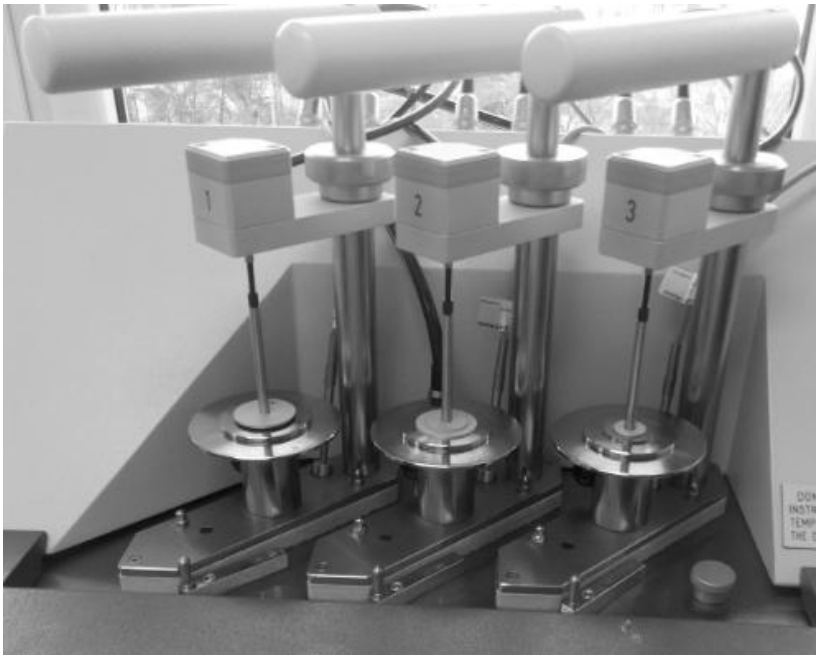

Fig.7.2. Measuring stations immersed in the oil bath during measurement of softening temperature using Vicat method

7.4. Carrying out of the exercise

In order to properly carry out this exercise you must perform the following actions:

- turn on device CEAST HV3, computer and software that controls the stations
- carry out the measurements of tested samples and enter the data into the control program
- place samples horizontally on the base of the instrument providing loads and determining softening temperature, in such way that the indenter's tip of unloaded rod will be in contact with measuring surface of the sample at a distance not smaller than 3 mm from its edge and close the doors of chamber thermostat,
- fix the weights on the device loading plate in such manner that the total load of the sample will be 10 ± 0,2 N for variation A and 50 ± 1 N for variation B,
- set the measuring indicator of the micrometer sensor to 1 ± 0,1 mm,
- set the thermoregulator of temperature increase to 50 ± 5^{o}C during 1 hour or 120 ± 10^{o}C, depending on the adopted variant of designation,
- read with an accuracy of 1^{o}C the Vicat softening temperature, at which the needle of the device will insert itself into the tested sample to depth of 1 ± 0,1 mm,
- the final result of the measurement – Vicat softening temperature – is a mean arithmetic value of the temperature for at least two tested samples, rounded to the nearest whole Celsius degree,
- after the measurement is finished, turn off the thermoregulator and thermostat, and cool it to the surrounding temperature; remove the instrument with fixed samples; remove samples from the handles and examine them.

7.5. Results, report

The results of measurements and tests must be summarized in a table and presented graphically in accordance with the figure that can be found in the report template. Correctly prepared report should contain: purpose of the exercise, proceedings of the exercise, designation conditions for softening temperature VST, results of measurements summarized in the tables, interpretation of results in the form of graphs, conclusions arising from the conducted exercise.

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