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ANALYSIS OF POLYCARBONATE RHEOLOGICAL PROPERTIES– MELT VOLUME FLOW RATE (MVR)

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Abstract: *Paper deals with verification of polycarbonate rheological properties. It was measuring and comparing rheological properties of polycarbonate plastic material produced by producer which are used for plastic parts production in automobile industry. This rheological property is very important for plastic parts production by injection moulding. Samples were removed in two different times from materials used for production and after that value of melt volume - flow rate were verified by HAAKE Meltflixer equipment.*

Key words: *polycarbonate (PC), injection moulding, regranulate.*

1. INTRODUCTION TO RHEOMETRY

Rheology describes the deformation of a body under the influence of stresses. "Bodies" in this context can be solids, liquids, or gases. Ideal solids deform elastically. The energy required for the deformation is fully recovered when the stresses are removed.

Ideal fluids such as liquids and gases deform irreversibly – they flow.

The energy required for the deformation is dissipated within the fluid in the form of heat and cannot be recovered simply by removing the stresses. The real bodies we encounter are neither ideal solids nor ideal fluids.

Real solids can also deform irreversibly under the influence of forces of sufficient magnitude – they creep, they flow.

Example. Steel – a typical solid - can be forced to flow as in the case of sheet steel when it is pressed into a form, for example for automobile body parts. Only a few liquids of technical or practical importance come close to ideal liquids in their behaviour. The vast majority of liquids show a rheological behaviour that classifies them to a region somewhere between the liquids and the solids: they are in varying extents both elastic and viscous and may therefore be named "visco-elastic".

Solids can be subjected to both tensile and shear stresses while such as water can only be sheared. This classification of the rheological behaviour of materials related to their response to applied stresses must be further extended by the introduction of the time-scale of any deformation process:

It is written in the Bible that "everything flows, if you wait long enough, even mountains" For all materials a characteristic time factor "λ" can be determined which is infinite in size for ideal elastic solids and almost zero for liquids such as water ($\lambda_w = 10^{-12}$ s). On the other hand deformation processes relate to characteristic time values "*t*". A high "Deborah number" (λ / t) defines a solid-like behaviour and a low "Deborah number" defines a liquid like behaviour.

Two examples may help to improve the understanding of the above:

a) If water is ejected from a nozzle at a very high speed and the droplets hit a hard wall, they will flatten. The droplets then spring back and recover their sphere shape in "no time" as the result of elasticity and surface tension. At these extremely fast deformation processes – "*t*" being very small results in the Deborah – number being high – even water with it's low λ – value reacts elastically.

b) The famous glass windows of the Cathedral of Chartres in France have "flown" since they were produced some 600 years ago. The glass panes had a uniform thickness from top to bottom in mediaeval times, but today the glass molecules have flown under the influence of gravity so that the thickness at the top is now "paper-thin" while the pane thickness has more than doubled at the bottom. The very long time t of this flow process results in a small Deborah-number. Thus one can state that solid glass, in spite of its high lambda-value at room temperature under conditions as stated above, belongs to the group of fluids if you wait long enough! An important conclusion of the concept of Deborah numbers is: substances such as water or glass cannot be classed as liquids or solids as such, but rather they exhibit a liquid or solid behaviour under certain conditions of stress, shear rates or time. Figure 1 shows ideal solids subjected to shear stresses react with strain [1].

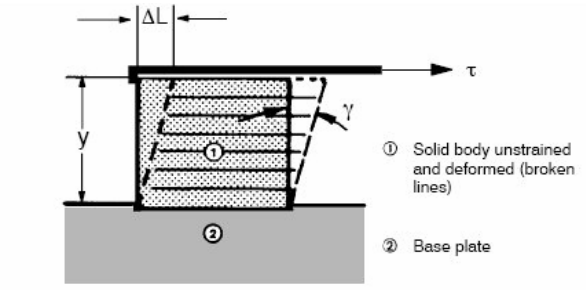

Fig. 1. Deformation of a solid body [5].

Fig. 2. Flow between two parallel plates [5].

Instruments which measure the visco-elastic properties of solids, semi-solids and fluids are named "rheometers". Instruments which are limited in their use for the measurement of the viscous flow behaviour of fluids are described as "viscometers".

Shear induced flow in liquids can occur in 4 laminar flow model cases:

a) Flow between two parallel flat plates, when one plate moves and the other are stationary is displayed on Fig. 2. This creates a laminar flow of layers which resembles the displacement of individual cards in a deck of cards. Conditions remain similar when the upper plate is acting as a stationary doctor blade used in a coating process on flat boards. The coating material, paint or glue is subjected to a laminar flow in the small gap that is formed between the board and the blade.

b) Flow in the annular gap between two concentric cylinders is displayed on Fig. 3. One of the two is assumed to be stationary while the other can rotate. This flow can be understood as the displacement of concentric layers situated inside of each other. A flow of this type is realized for example in sleeve bearings and in rotational rheometers with coaxial cylinder sensor systems.

c) Flow through pipes, tubes, or capillaries is displayed on Fig. 4. A pressure difference between the inlet and the outlet of a capillary forces a Newtonian liquid - the term will be explained in the following – to flow with a parabolic speed distribution across the diameter. This resembles a telescopic displacement of nesting, tube like liquid layers sliding over each other.

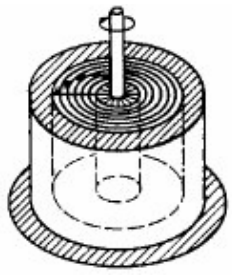

Fig. 3. Circular flow in the annular gap between two coaxial cylinders [5].

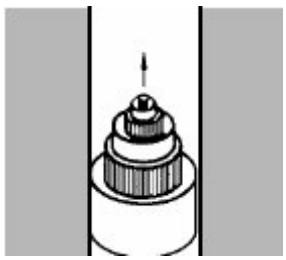

Fig. 4. Flow through capillaries, hoses, tubes or channels [5].

Fig. 5. Flow between a cone and plate or Flow between two parallel plates [5].

A variation of capillary flow is the flow in channels with a rectangular cross-section such as slit capillaries. If those are used for capillary rheometry the channel width should be wide in comparison to the channel depth to minimize the side wall effects.

d) Flow between two parallel plates or between cone and plate sensor systems of rotational rheometers when one of the two is stationary and the other rotates is displayed on Fig. 5. This model resembles twisting a roll of coins causing coins to be displaced by a small angle with respect to adjacent coins. This type of flow is caused in rotational rheometers with the samples placed within the gap of parallel-plate or cone-and-plate sensor systems [5].

2. TYPES OF RHEOMETERS/VISCOMETERS

2.1. Rotational Rheometers/Viscometers

The principle of rotational rheometers with coaxial cylinder, cone and plate and parallel-plate sensor systems allow the design of excellent and versatile absolute rheometers. The range of rotational rheometers and viscometers on the world market varies widely in sophistication and price. The rheological criteria and boundary conditions mentioned before are used to grade types of instruments and explain design features and resulting areas of application.

One might imagine that the coaxial cylinder sensor systems for rotational rheometers/viscometers result from bending both flat plates of the Newton's parallel plate model into an inner and outer cylinder. A liquid sample filling the annular gap between the two cylinders can be exposed to shear for any length of time.

Conditions as in Fig. 3 will lead to laminar flow, and allow a mathematical treatment of measured data delivering test results of shear stresses, shear rates and viscosity in the appropriate physical units. The same can be said for cone and plate and for parallel plate sensor systems, which have special application areas.

Two basic alternatives are open to turn the above geometries into an absolute rheometer/viscometer, which can provide:

a) controlled stress input and determine the resulting shear rate;

b) controlled shear rate input and determine the resulting shear stress.

2.2. Capillary viscometers

Quite a number of simple and sophisticated instruments fall in this group of viscometers which are designed to measure viscosity but not elastic properties even if the samples are visco-elastic.

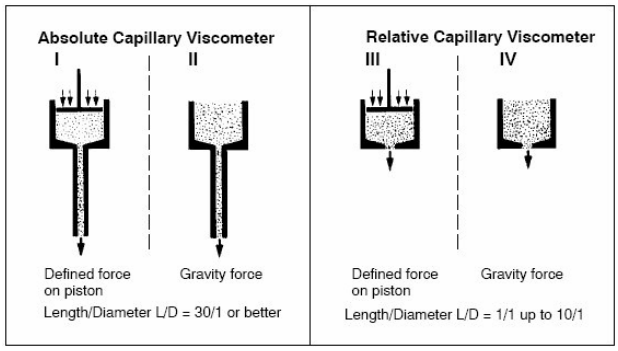

Fig. 6. Schematic comparison of capillary viscometers [5].

Well designed capillary viscometers allow a liquid to flow as indicated in Fig. 6. Capillaries can have round or slit cross sections. Laminar flow in capillaries can be understood as a sliding of nested tube like layers telescopically over each other.

With respect to the design of capillary viscometers and the quality of their viscosity test results, one differentiates between those which provide variable pressure to force liquid through the capillary or those that use gravity as the driving force. In addition capillaries may have either long or short capillaries or high or low ratios of capillary length L to capillary diameter D. Testing can be done in the CS mode with a pressure provided and the resulting flow rate being measured or in the CR mode, for which a controlled flow rate is preset by means e.g. of a melt pump and the resulting pressure drop along the length of the capillary is determined.

2.3.Melt indexers – model III of Fig. 6

These are relatively simple capillary viscometers mainly used to grade the viscosity of polymer melts. The polymer is melted in a cylindrical reservoir and then extruded out of the exit orifice by means of a plunger carrying one or more dead weights. The exit orifice is really a capillary with a very short length (ratio of L / D = 10 / 1 or smaller). This capillary length is insufficient to guarantee the boundary conditions – constraints of rheometry – of steady state and laminar flow between the entrance of the capillary and the exit orifice. A sizeable amount of the potential energy in the system – here mainly the dead weight on the plunger must be used to accelerate the sample to the flow speed at the exit and to overcome turbulence at the entrance to the capillary. Exit effects must also be accounted for inasmuch as the extrudate leaving the die transports kinetic energy with it which was not present when the sample entered the entrance region of the capillary.

Viscosity is measured by determining the volume of a sample that is extruded through the capillary within a defined time period Δt. Test results gained with melt indexers and polymer melts can vary up to 30% from the corrected, true values obtained using capillary type viscometers with a high L / D ratio such as in Fig. 7.

Melt indexers are customarily used for non-Newtonian polymer melts to determine their workability in production machines. However, shear rates in melt indexers are several decades below those encountered e.g. in dies of extruders or nozzles in injection moulding machines.

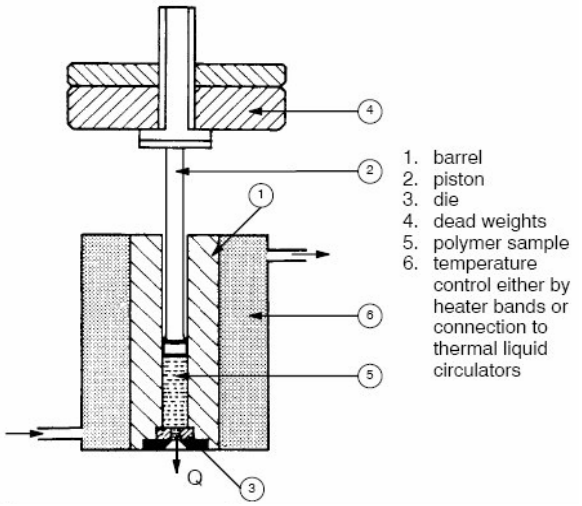

Fig. 7. Schematic drawing of a melt indexer [5].

It is never a good idea to use viscosity values measured at low shear rates and then extrapolate to viscosity values linked to high shear rates if samples such as polymer melts are highly non-Newtonian.

As the result of their relative low price, melt indexers are still quite common in industry for the testing of polymers with a relatively simple processing behaviour such as polyethylene or polystyrene but they are loosing popularity in the laboratories of companies which have to maintain high levels of quality control for raw materials and compounds.

Melt indexers may cause one other problem: if pelletized polymers are molten statically only by heat transfer from the heated barrel during 5 to 10 min the polymer may already start to chemically degrade before the actual start of the test. Bad test results can also be expected if the extrusion takes place before the polymer is not fully molten and when air has been entrapped in the melt.

This will not happen with modern extruder capillary rheometers which use single or even twin-screw extruders to melt and homogenize polymers and their compounds and which will provide a melt of much higher homogeneity in a fraction of the time which melt indexers require [1, 5].

3. MEASUREMENT OF SAMPLES

The measurements were made in specialized laboratory at our department which consists of Thermo Scientific HAAKE Meltflow MT rheometer, evaluating software, analytical balance and other equipment. Thermo Scientific HAAKE MeltFlow indexers are ideal for injection molding companies to be used for incoming or outgoing quality control of polymers. Thermo Scientific HAAKE MeltFlow indexers comply with the ISO 1133, ASTM D 1238, ASTM D 3364, JIS K 7210 and referring standards. Based on a compact design the HAAKE MeltFlow MT is the ideal table top instrument for small labs and infrequent use in a quality control environment. Equipped with a digital displacement sensor the apparatus measures the melt volume rate (MVR) semi automatically [3].

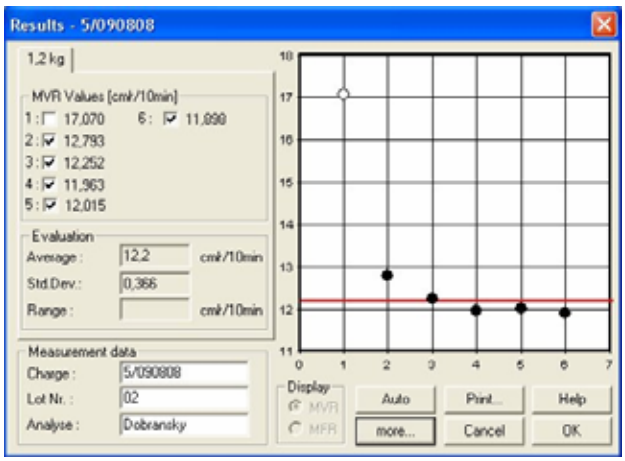

Fig. 8. Demonstration of measured values for first sample in software Meltflixer 2000.

Using the Thermo Scientific HAAKE MeltFlow software the apparatus can automatically measure apparent viscosity data's (shear rate, viscosity, shear stress). Also was used balance Mettler Toledo PL 303 IC with accuracy 0.001g for weighing of sample mass [2, 4].

Table 1 shows measurement conditions which were set up during measurement and which were the same for the both samples.

Figure 8 shows demonstration of measured values for first sample during test from which software Meltflixer 2000 determine final value of melt volume flow rate (MVR). Software also determines next values which are entered into the Table 2.

Test results are declared as arithmetical average of re-determinations *x* with relative standard deviation of arithmetical average *s*.

Table 1

Measurement conditions

	Sample 1	Sample 2
Material:	polycarbonate	polycarbonate
Sample mass:	5.00 g	5.00 g
Temperature:	300 °C	300 °C
Die length:	8.000 mm ±0.025 mm	
Die diameter:	2.095 mm ±0.005 mm	
Weight:	1 200 g	1 200 g

Table 2

Measurement results

Property	Sample 1	Sample 2	Unit
Melt volume rate (MVR)	12.35	11.3	cm^3/10min.
Relative standard deviation of arithmetical average	0.366	0.0639	%
Time of measurement	1.40	1.54	min.
Shear rate	2.2525E1	2.0863E1	1/s
Shear stress	1.0759E4	1.0759E4	Pa
Viscosity	4.7767E2	5.1572E2	Pa.s

4. CONCLUSIONS

Paper deals with verification of polycarbonate rheological properties. It was measuring and comparing rheological properties of polycarbonate plastic materials produced by producer which are used for plastic parts production in automobile industry. During experiment were tested in the same measurement conditions two samples (polycarbonate) which were removed in the different time (1 month) from production process of injection moulding. Samples were tested in specialized laboratory by means of Thermo Scientific HAAKE Meltflow MT rheometer. Samples was evaluated by special software Thermo Haake Meltflixer 2000, which determined melt volume flow rate value, shear rate, shear stress and viscosity what are the basic rheological properties of plastic materials.

The target was determined and verified melt volume flow rate value which is presented in material list and which is important to verify it. Values of melt volume flow rate by both samples were not in allowing tolerance. Required value is from 8 up to 11 cm^3/10min. Value by sample 1 was 12.35 and by sample 2 was 11.3 cm^3/10min. It means that the material has not good flow properties and it is not qualified for production process of injection moulding.

It is necessary to make some changes in material properties, especially in its rheological properties by reason of higher quality of this processing material.

That is necessary to determine material properties again and if the result will be without tolerances material have to be reclaimed by reason of quality process assurance.

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