Correlation of the prediction curve of the creep behaviour of polymeric materials

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Abstract: - This study deals with the study corellation of regression curve parameters on the prediction of possibilities of measured data. In this study, the necessary introduction of polymeric materials and their material structure, which influences their physical and mechanical properties is described. Thereafter, the method of testing materials using the DMA device which enables this measurement is presented. In the next section, these measured data are processed by a creep module in which the regression curve methods and their match with the measured data are applied. In the final part of the thesis is evaluated the conformity of the measured data with the data obtained from the regression curve.

Key-Words: - creep, polymer, regression, prediction, long test

1 Introduction

Nowadays, polymeric materials belong to commonly used materials in a number of applications and use. In the practice it is possible to meet a number of methods which enable to study the properties of these materials and to recognize better their behaviour during different conditions of use. These measurements are often unnecessary without the need to choose a certain range of variables which can then be analyzed. It is not possible to always fully influence the choice of the density of this scale, namely the density of the points which are then passed through different lines, curves and splines. However, the density of these points significantly affects the accuracy of the interleaved geometric elements. In interleaving, each used software allows to influence the extent of this interference, and thus the extent to which the interleaved geometry matches the obtained points from the individual measurements. [1-5]

For this purpose, HDPE and LDPE materials from DOW were selected. Polymers are chemicals composed mostly of carbon, hydrogen, oxygen, nitrogen, chlorine and other elements. They are substances which are processed in the liquid state, mostly under elevated temperature and pressure. In this state, it is possible to produce a variety of shapes in the production process. Polymers can be of natural or synthetic origin, from a chemical point of view, they are mainly organic substances composed of large macromolecules. These macromolecules are composed of many repetitive cells in the string called "mer", which is a basic monomeric unit that has the ability to connect under appropriate conditions. From the suffix mer (mer = part) and poly prefix (meaning more from Greek) the word polymer was created.

The length of the macromolecules can be expressed using the molar mass, resp. relative molecular weight. By definition, the macromolecular substance is a compound whose molecular weight exceeds 10^3 g/mol, and the polymer bears a substance having a molecular weight greater than 10^4 g/mol.

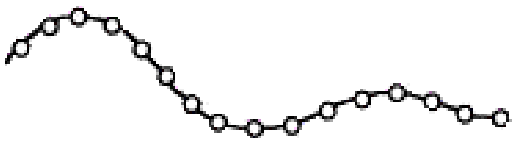

Fig. 1 Linear structure

The manufacture of polymers is linked to them. Nowadays, there is an increasing demand for the resulting plastic product. It should have the lowest weight, high temperature resistance, abrasion resistance, frost resistance, it should be firm and tough. These requirements give a permanent impetus to the emergence of new types of polymers, especially high-tech polymers which are now used for example in the aerospace or aerospace industry [6-9].

Because of different mechanical properties that depend on the internal structure, it is possible to divide the polymers into:

Linear - single monomer molecules join together in a row to form a string. Such polymers have a higher density (e.g. HDPE) due to a spatial arrangement that allows molecules to come closer together.

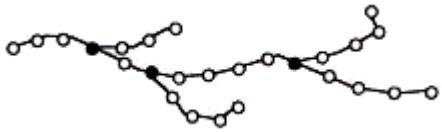

Fig. 2 Branched structure

Branched - will occur if side branches are added to the basic string. These branched macromolecules cannot be as close to each other as they are in the linear branches, and thus have a lower density (e.g. LDPE). Due to branching, the mobility of the macromolecules is impaired (the molten polymer has lower fluidity) and there is also a decrease in the intermolecular forces (the side chains are further apart) which lead to the worse mechanical properties of the polymer. [10-15]

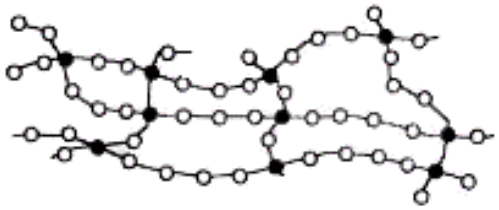

Fig. 3 Crosslinked structure

Crosslinked (block, grafted) - are formed by linking straight or branched chains by means of transverse chemical bonds. Such a c ross-linked macromolecule has a l imited mobility as a w hole. This newly formed network results in a change in polymer properties, increases its hardness, stiffness, increased temperature resistance, but resistance to impact stresses is reduced by crosslinking. The resulting polymer is non-melting and insoluble again.

Based on usable properties and possibilities of use, depending on t he mechanical properties, the polymeric materials can be assembled into the following pyramid.

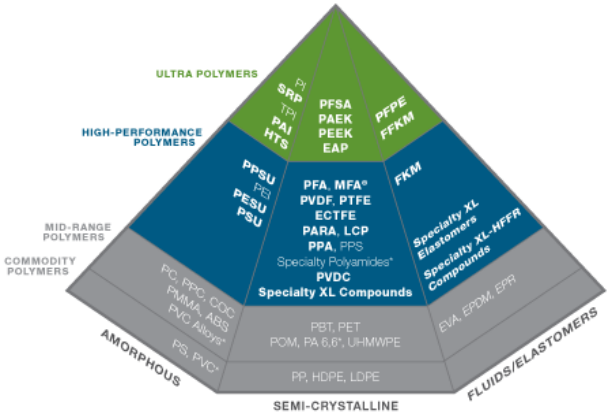

Fig. 4 Separation of polymer

2 Methods

For creep tests it is necessary to prepare test specimens. These are designed to provide repeatability in predefined shapes and dimensions.

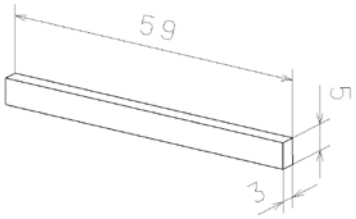

Fig. 5 Sample dimensions

For measuring mechanical tests, standard double-sided blades specimens as standard are used, for the measurement of creep properties on the DMA device samples in the shape of a b lock are used, which can be seen in Fig. 5. This shape and size of samples is adapted for measurement on DMA devices.

Fig. 6 Arburg Allrouder 170 U

The crosslinked material was chosen for the measurement of creep properties for comparison of various options. High-density polyethylene from the company DOW was chosen as a material.
Testing samples were made from this material according to a standard ISO 527-2 1BA for the tensile test.

The processes of injection moulding and radiation cross-linking were performed with the minimum time gap to avoid the influence on the measurement surroundings.

These samples were moulded by the injection moulding machine ARBURG Allrouder 170U according to the same process conditions.

Tab 1. Process conditions

Parameter	**Value**
Injection temperature	200 °C
Barrel temperatures	195 °C, 190 °C, 180 °C
Mold temperature	40 °C
Injection velocity	40 mm/s
Injection pressure	60 MPa
Cooling time	45 s

The samples were irradiated by doses 33 kGy, 66 kGy, 99 kGy, 132 kGy, 165 kGy, 198 kGy. The concurrent measurement followed after irradiation on the device DMA1 from the Company Mettler Toledo and on the device for measurement of creep properties of own produce.

Dynamic mechanical analysis (DMA) is one of the possible methods for measurement of dynamic properties. The samples for this kind of test can be exposed to tension, compression, bending and shear. The device disposes with the mode TMA, which enables to measure the expansion, creep and relaxation time. The device DMA1 in Fig. 7 was used for measurement.

The device DMA1 enables the measurement of brittle and though materials, which are suitable for these type of loading, and it enables evaluation in the program STAR. The load module enables loading from 0.001 to 10 N . The frequency generator enables the variable adjustment of oscillation from 0.001 to 300 Hz.

Fig. 7 Mettler Toledo DMA1

3 Data of results

During evaluating the results, it was necessary to proceed with a slight cut of the measured ones. As can be seen in formula 1, the resulting module is directly proportional to the loading force and initial length L_0 that are constant during measurement and calculation.

$$E_t = \frac{FL_0}{A(\Delta L)_t} \tag{1}$$

However, this creep modulus is inversely proportional to the initial cross section of the test piece and, above all, to the elongation value.
From the measured data one measurement was selected as can be seen in Figure 8. This graph shows the dependence of the creep modulus on time for HDPE material irradiated with a dose of 66 kGy.

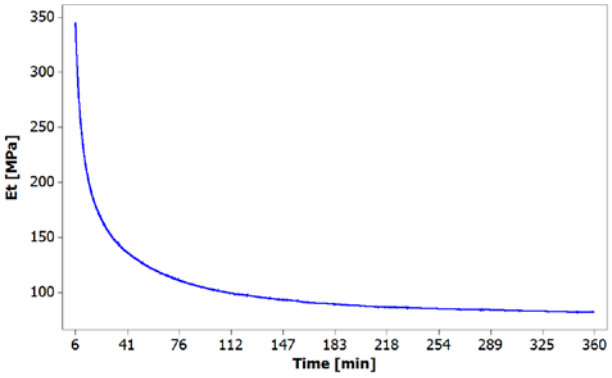

Fig. 8 Creep modulus of HDPE irradiated with a dose of 66 kGy

In Figure 8 the curve of the graph is visible, it is possible to divide the curve into two basic parts as the long-term strain is described. In the first part (primary) there is a high creep modulus that quickly

loses its value. This very steep area ends in approximately 100 minutes of measurement, where the second part of the creep is no l onger so steep and the steady flow (secondary) of the last phase of the creep curve is again the increase in the deformation rate after which the test sample breaks. This part is called tertiary, this area should be avoided by the designer and use only the secondary part with enough reserve than the transition from the linear steady area to the last phase.

Critical trials are very time-consuming, and the most time-consuming behaviour is found. That is why it is possible to neglect the initial (primary) area of the creep and concentrate only on t he secondary area where the steady flow has occurred. This part of the graph can then be seen in Figure 9.

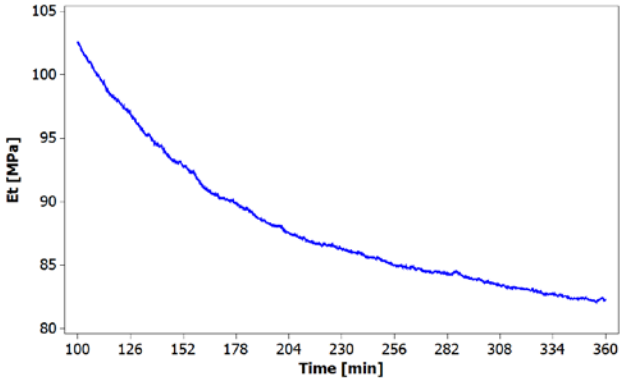

Fig. 9 Secondary area of steady flow

When trying to predict measurement over time, it is the best to use a real data to compare the measurement, and then determine the similarity and predominance of the prediction. From the total measuring time of 360 minutes that are available, the last 60 minutes will be taken, which we will then try to predict.

As was already mentioned, the attempt is to override the course of the crush module after the theoretical end of the measurement at 300 minutes at 360 m inutes. Given the complexities of the mechanism that describes the entire curve, we focus only on the area of the prediction and its close proximity. The goal is to find out how large can be a region for a simple prediction using regression analysis.

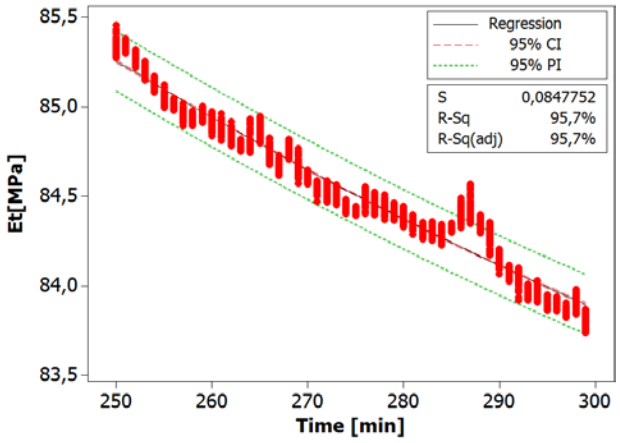

Fig. 10 Regression analysis for 250-300 minutes

In Figure 10, the interleaved data can be seen by the regression curve. The regression curve used is quadratic and its mathematical description is following:

$$E_t = 96.75 - 0.06103t + 0.000060t^2 \qquad (2)$$

The same type of regression function was also interpolated with basic data in 200-300 minutes. Here the probability of stairs with data from the original 95.7% for the range of 250-300 minutes has been stacked to 99.3% as can be seen in Figure 11.

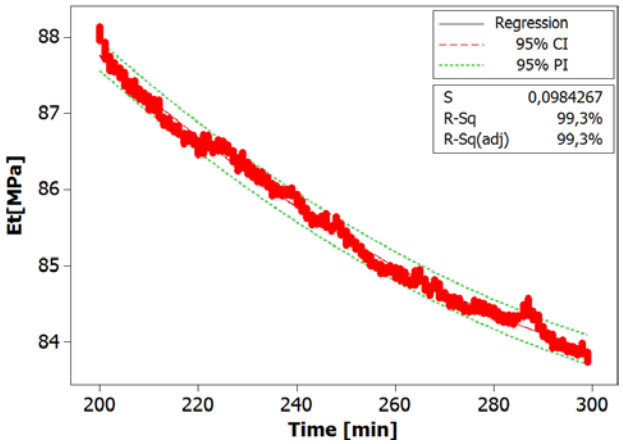

Fig. 11 Regression analysis for 200-300 minutes

The formula for expressing the regression curve shown in Figure 11 can then be written following:

$$E_t = 106.3 - 0.1282t + 0.000179t^2 \qquad (3)$$

The last observed range was a time interval of 150-300 minutes. The depiction of our own regression curve can be seen again in Fig. 12.

Where to write the regression curve is possible following:

$$E_t = 116.6 - 0.2123t + 0.000347t^2 \quad (4)$$

Here is the highest level of match with measured data, which is 99.4%.

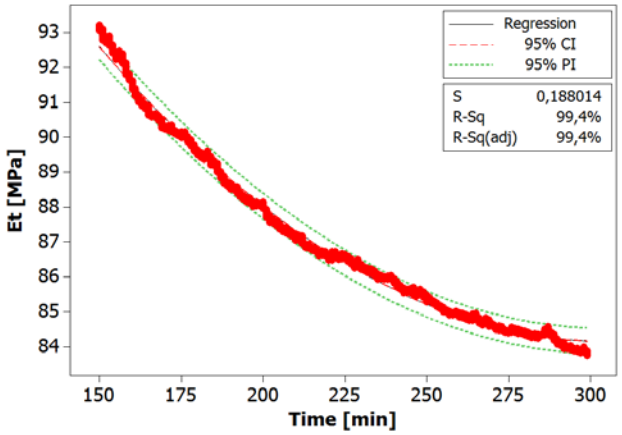

Fig. 12 Regression analysis for 250-300 minutes

From the equations thus obtained, it is possible to obtain the course of the function during the determined time by simple insertion into the equation in time.

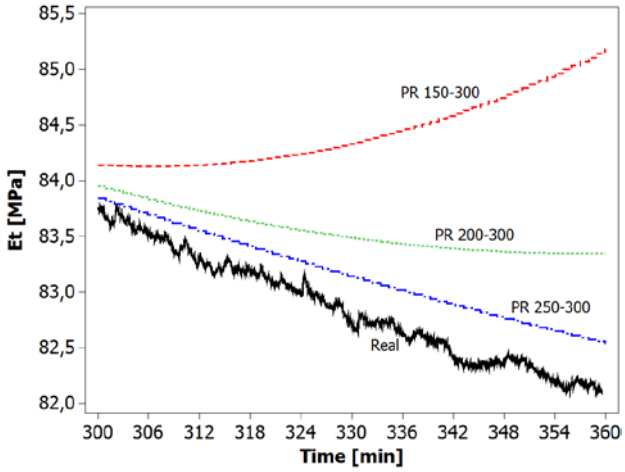

Fig 13 Comparison of prediction and reality 300 - 360 minutes

4 Conclusion

In Figure 14, it is possible to see the prediction error that occurs when applying the application of the regression function equations.

Looking at the prediction calculated from the 150-300 minutes period, we will outweigh the increasing divergence from the actual values. In this case, the longer prediction totally loses its significance, and this longer, theoretically more appropriate interval elicits the biggest mistake. The error curve plotted from the regression curve of 200 -300 minutes is significantly more appropriate with a h alf error compared to the prediction curve obtained from a 150-300 minute stretch.

The last section examined was 250-300 minutes where the formula (4) was obtained from the measured data, which describes the actual data in the shortest section. However, as can be seen in Figure 14, the deviation in the observed area 300 - 360 minutes with the actual measured data is minimal.

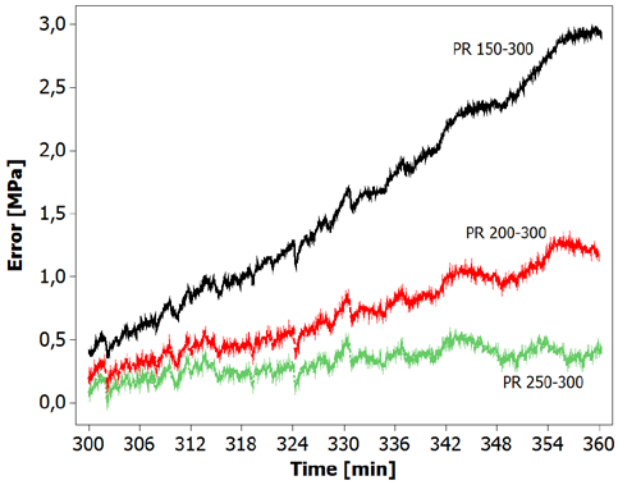

Fig. 14 Predictive error display

In Figure 14 it is also visible that the curvature of the curves, which should be smoothed because they are calculated from regression functions. This vibration is due to confrontation with real-time data. This statement confirms a more detailed view of the one-sided curves, whose trends are repeated only at a y-axis in each time period.

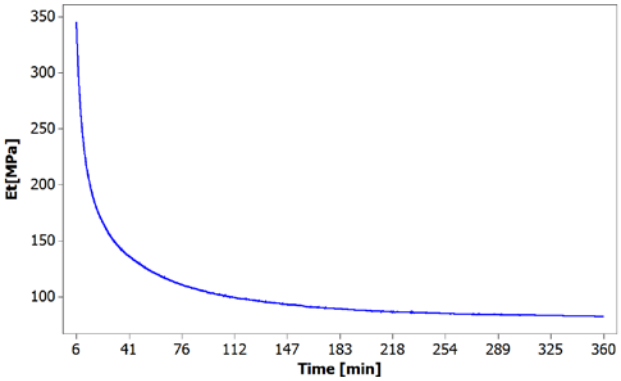

Fig. 15. Total combined creep curve

Figure 15 shows a possible output from a combination of the use of real waveforms and curves computed from the regression equation. As

shown in Figure 14, the prediction from the time range of 250-300 minutes showed the smallest deviation from the real values. The calculated area in the 300 - 360 minute interval is completely homogeneous with the previous measurement and represents the hourly saving time on the measuring device.

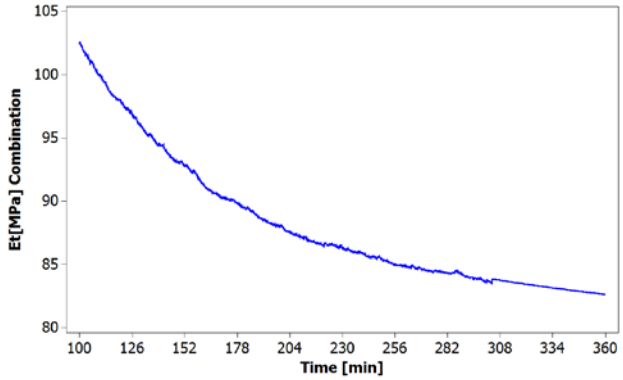

Fig. 16. Secondary combined creep curve

For a more detailed view of the secondary region of the creep, which is shown in Figure 16, a continuous relationship to the preceding sections is visible. From the calculated time of 300 minutes, it is possible to notice the stabilization of the entire curve, which causes the use of regression equations.

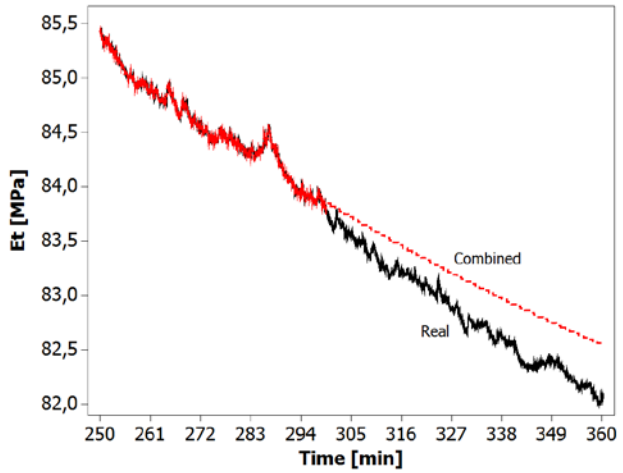

Fig. 17 Detailed view of predicting stairs

In Figure 17, a detailed view of the prediction curve differences from the data from the 250-300 minutes interval and the curves measured in measure is displayed. Is clear that, at a 60-minute prediction time, we have a d ifference of approximately 0.3 MPa, which is only 0.3% error when measured by values. This error can be considered negligible.

Concluding this article, we can state that on the basis of the data obtained from the measurement of the creep it is possible to perform a relatively easy prediction of the measured data in a short period of time. Given the nature of crunching, the use of shorter slots before the start of the prediction is more appropriate because of a minor error in the observed area.

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