Construction of Equipment for Creep Behavior Study

M. Reznicek, V. Senkerik, V. Pata, M. Stanek, A. Skrobak, K. Kyas and D. Manas

***Abstract* —** This article describes the design of devices for measuring creep, in which commercial measuring instruments are used and which allows the measurement of creep at elevated temperatures. Detailed design of the entire device is described, including examples of possible outcomes. The device is designed to measure up to four samples simultaneously and these samples can be loaded with different weight which increases the variability of the equipment. The whole device is designed to be manufacturable in workshops with minimum machinery and thereby reduces the overall cost. Finally the device tested samples PA6.6 and radiation crosslinked filled PP samples.

***Keywords*—** load, strain, creep, result, temperature.

I. INTRODUCTION

Materials used for the making of products are currently very diverse and have a great range of properties that can be changed. We can make these changes by changing the type of material, mixing of materials of different properties, various thermal, chemical and heat treatments. From as early as making the form for plastic products, it is necessary to take into account the material used for the production. Shape also plays a big role respectively technology of manufacturing, which is based on the complexity of the part. By choosing materials with improved structural properties we manufacture more expensive parts, mostly by making expensive waste flow in the form of mouth and distribution channels. The aim of today's manufacturing technology is often fixed to minimize these losses, this can be achieved by choosing the right type of technology or reduce the costs associated with the purchase of material. Substituting high quality materials for lower quality will have reduce prices,

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but in many cases we can get lower quality products that result in a higher failure rate and are prone to destruction.[2]

Therefore, the aim of today's designers is to get maximum information about the material and its properties. Manufacturers of materials therefore focus on development of new blends with the aim to achieve the best performance for customers and provide these materials to maximum material properties, which may play a role in the design and manufacture of the product. In addition to standard material properties emphasis is also placed on the characteristics of long-term stress, which include the measurement of creep.

As already mentioned, creep is a long-term stress of the material, which affects long-lasting equipment or installations that are exposed to elevated temperatures during their usage. Elevated temperatures are accelerating the effect; therefore it is necessary to examine the individual properties of these materials and to inform the designer, who can then use the material effectively. Measurement of creep in metallic materials is not new and is not as significant as creep of polymeric materials that have a strong desire to flow even at room temperatures, where creep of metallic materials has almost no effect.[2]

Another important part in the design will be choosing the type of test. As mentioned in the introduction, creep tests can be performed in two modes, the choice of a mode will have a significant impact on the overall design of the mechanism.

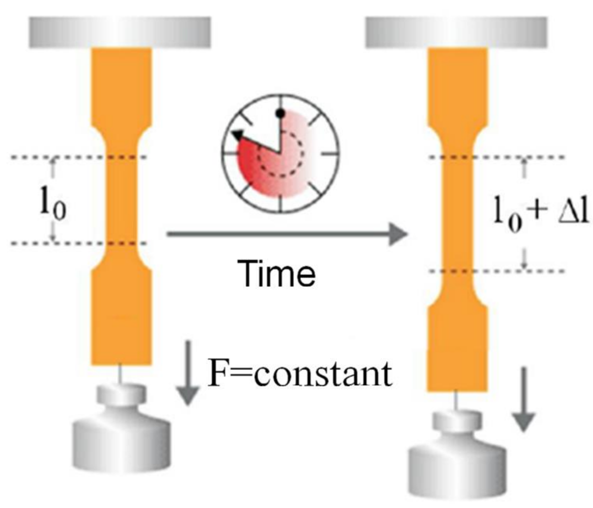

Fig.1 Creep test under a constant load

For these reasons, tests under a constant load was chosen for this design of the equipment. Another important prerequisite for this test is temperature. According to ISO 899-1 norm we differ between test at a room temperature and at higher temperatures. For the mentioned test equipment was required to test at higher temperature. The nominal temperature according to the norm should be within + / - 2 ° C which increases demands on thermal regulation.[3][4]

Each device is limited by the boundary conditions that can be set. Due to the expected use of the device for polymer materials that are radiation modified, the temperature range from 20 ° C to 250 ° C should be sufficient with a sufficient margin. The load is expected to be in the range of 50N - 1000N by weight. Measuring range is 20 mm with 0.01 mm resolution. The equipment should be lightweight with automatic sensing and recording of data.

II. Construction

Each measuring device consists of a number of groups that perform different functions. According to these groups, the device can be divided into parts.

Suitable clamping of the sample is one of the more complex design tasks. Mechanical properties of tested materials are different, and therefore require appropriate selection of the clamping device. In addition to commercially available clamping jaws such as tensile testing clamps from the Zwick company, you can design your own clamps. Due to elevated temperatures, self-locking jaws won’t be used since its clamping force could negatively affect the test results.

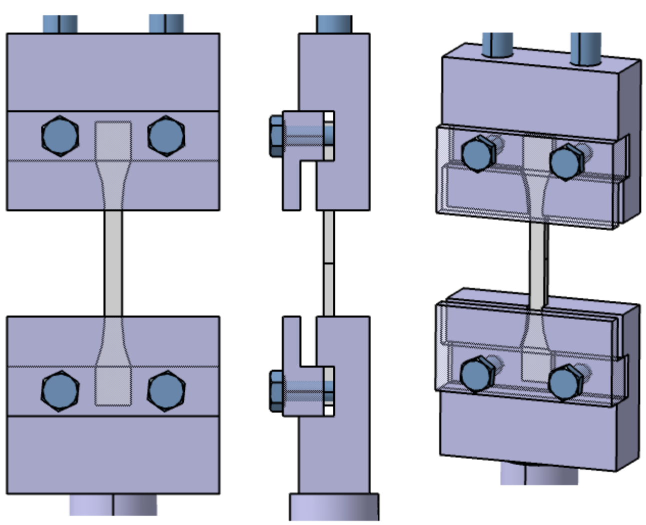

Fig. 2 Clamping of the sample

Used jaws are shaped for small testing samples, which are places in recesses and held by two screws.

Shape of clamping jaws is the same for both upper and lower jaw. Individual jaws are different in places of clamping. The upper jaw is clamped by two screws M10 for fixing the module in place.

The lower jaws for small specimens uses extended cylindrical lock mechanism that allows easy and quick loading of test samples for the measurement.

The advantage of this option is exchangeability of the jaws for jaws with a recess for large samples. The upper jaw is the same except the shape of the clamping of the sample.

The lower jaw is different not only the shape but also the sample clamping mechanism for quick loading, which is now integrated in the body of the jaw, thus compensating the lengths of test samples and gaining the possibility of using the same length rods for both options.

For tests that have costs associated with the operation of the test equipment, designer tries to reduce the cost of a single test. The reduction can be done by reducing operating costs, which are often fixed and cannot be significantly changed.

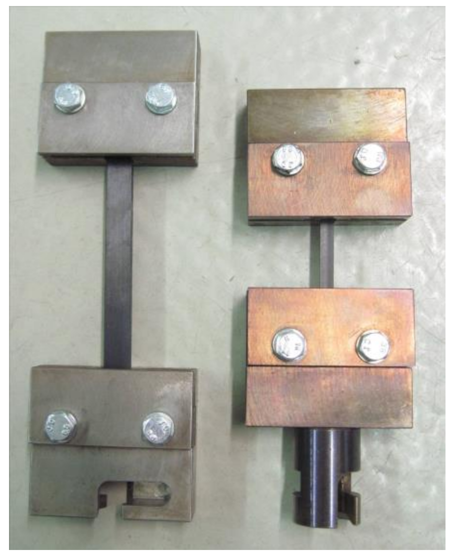

Fig. 3 Clamps for big samples

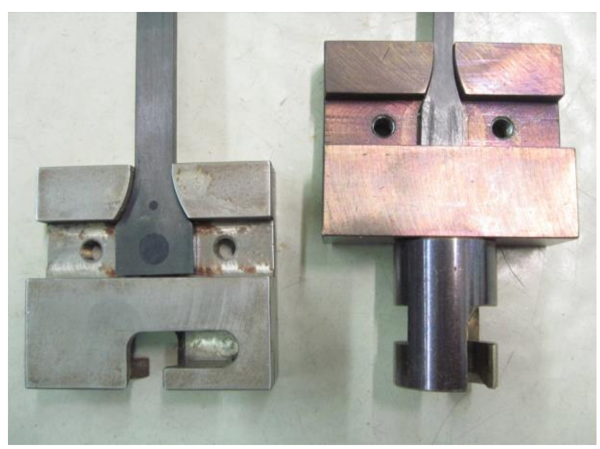

Fig. 4 Comparison of mechanisms for fast loading

Another possibility is to allocate these costs among multiple tests, making price of one sample smaller. In case of tempering chamber it is suitable to test the samples at the same temperature.

Evaluation of the work area temperature chambers with regard to handling and safety areas, four symmetrically placed samples were selected.

The correct choice of location and number of test specimens affects not only convenient clamping of samples but also affects the flow of air inside the chamber. Forced air circulation increases the homogeneity of the temperature field inside the chamber; excessive filling of the chamber can cause inhomogeneous temperatures due to low air permeability and thereby influence the results of elongation due to temperature.

Internal removable module is symmetrical, but the vertical columns of the front part are arranged asymmetrically so as to enable a control view of the test sample through the glass in the door of the chamber. The frame is provided with a heat resistant coating up to protecting the structure from corrosion at 250 °C.

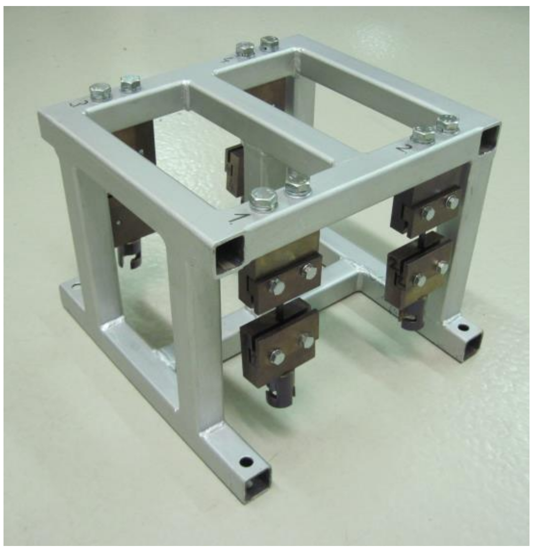

Fig. 5 Fastening mechanism

Small manipulation possibilities for changing of the samples contributed to the design of a small sample supporting frame.

There are four strong jaws attached to the frame with bolts. The testing samples are attached outside of the chamber, then inserted into a fastening mechanism which provide the same position after changing the samples.

Both structures are fabricated from quadrangular cross-section 30x30mm, providing sufficient rigidity and strength of the entire system.

The upper part of the main structure is ergonomically positioned at eye level, allowing the setting of temperature and turning on or off the air circulation.

Below the panel is a thermal chamber for which an older oven was used, fulfilling the conditions of the indoor fan, the maximum temperature of the chamber volume and control indicator. Using the ready-made chamber greatly eased the design and potential problems such as effective insulation, heating, sealing the door, placing of the propeller fan.

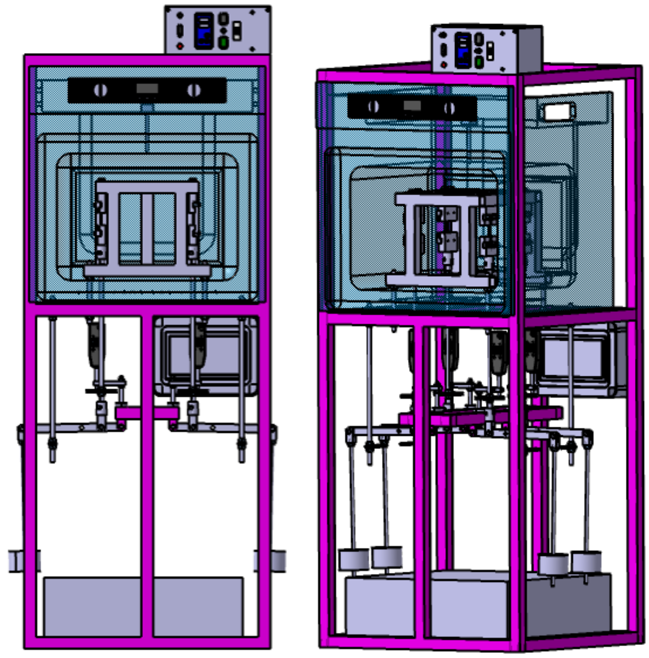

Fig. 6 The overall concept

Under temperature chamber is an auxiliary support structure positioned with measuring devices. This structure is also used for clamping the fixed side of the lever mechanism.

To ensure a higher stiffness and a sufficient resistance to vibration weights were positioned in the lower part of the main structure to increase inertia.

Boxes for dial gauges and temperature sensors placed in the temperature chamber are in the back of the device.

The load of individual samples is performed via a lever mechanism for individual samples, allowing for greater variability in load tests at the same temperature. The lever mechanism is in a ratio of 1:5, with possibility of extension to 1:10.

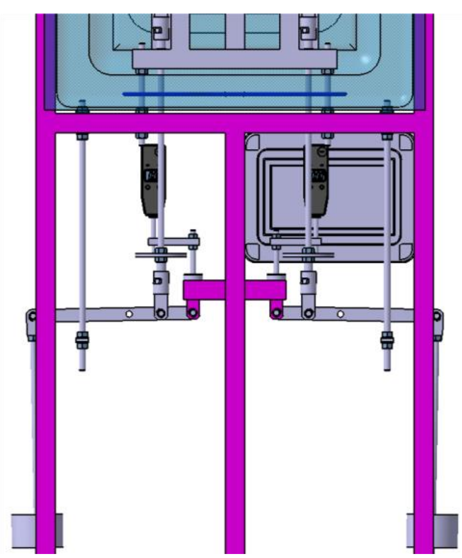

Fig. 7 Lever mechanism

During testing of materials which are thermally very vulnerable to creep the lever mechanism can be completely removed and direct loading of the samples used.

In the case of direct loading of samples the locking mechanism is not fully used in both parts, but only at the top. Calibrated weights of different weights are applied to load the samples.

The load is read out of tensile diagram for the material at the same temperature as the creep test runs. The highest permissible load of samples is the limit of proportionality tension diagram. For easier adjustment, the calculated load value is rounded down which is then calibrated on all stands if required.

Due to the increased possibility of ruptured specimens or excessive stretching, all levers are equipped with catching mechanism. This mechanism ensures the extreme position of extension for viscous materials and as a stop at break in brittle materials.

As a connecting part of the lever mechanism, hardened pins secured by external secure ring are used.

Measurement of stretching is performed using dial gauges from Mitutoyo with extended measurement range of 25.4 mm and Digimatic connector that allows the computer to communicate with the gauge. The thermal chamber does not allow the probe to touch the jaw, thus stretching is measured outside the temperature chamber. By using these commercially available measuring instruments with sufficient resolution and the maximum permissible error allowed significantly reduce the cost of the equipment. The advantage of using these sensors is in their compactness and almost no requirements for their attachment. Clamping is done using the clamping part with diameter of eight millimeters. Another option how to measure the stretching is using various linear gauges using different principles. After attempts to improve the accuracy of measurement by these methods, they were abandoned because of the high demands for the attachment and adjustment of such sensors.

Using the standard sensors from Mitutoyo, which are mounted across the diameter of 8 mm, it is possible, if necessary, quick and easy replacement of these sensors to other with a lower maximum permissible error according to the needs of the experiment or the material properties of the sample.

The digital dial gauges used Digimatic output and were connected to a computer. To ensure high repeatability a dedicated program was made to regularly measure the stretching. The program was written in Python 2.7 and stores the data in a *.txt text file. The recorded data can be easily evaluated in mathematical programs and used to construct a graph.

Note

Rok,mes,den,hod,min,s	cas (s)	COM1	COM2	COM3	COM4	Tep1	akt_tep	poz_tep
2013,3,12,6,57,38	0	+2,99	+0,79	+0,85	+2,87	40,06	40,6	40,0
2013,3,12,6,57,43	5	+3,05	+0,82	+0,86	+2,87	40,06	40,6	40,0
2013,3,12,6,57,48	10	+3,08	+0,83	+0,87	+2,88	40,06	40,6	40,0
2013,3,12,6,57,53	15	+3,09	+0,84	+0,89	+2,89	40,06	40,6	40,0
2013,3,12,6,57,58	20	+3,11	+0,85	+0,89	+2,90	40,06	40,5	40,0
2013,3,12,6,58,3	25	+3,13	+0,86	+0,90	+2,91	40,00	40,5	40,0
2013,3,12,6,58,8	30	+3,14	+0,87	+0,91	+2,92	40,06	40,5	40,0
2013,3,12,6,58,13	35	+3,16	+0,87	+0,92	+2,92	40,06	40,5	40,0
2013,3,12,6,58,18	40	+3,17	+0,88	+0,93	+2,93	40,12	40,5	40,0
2013,3,12,6,58,23	45	+3,18	+0,89	+0,94	+2,94	40,19	40,5	40,0
2013,3,12,6,58,28	50	+3,19	+0,89	+0,94	+2,94	40,19	40,5	40,0
2013,3,12,6,58,33	55	+3,20	+0,90	+0,95	+2,95	40,25	40,6	40,0
2013,3,12,6,58,38	60	+3,21	+0,91	+0,96	+2,96	40,25	40,6	40,0
2013,3,12,6,58,43	65	+3,23	+0,91	+0,96	+2,96	40,25	40,6	40,0
2013,3,12,6,58,48	70	+3,24	+0,92	+0,97	+2,97	40,25	40,6	40,0

Fig. 8 Sample of data recording

Data are saved after each loop to storage on a hard drive, thus ensuring a backup in case of power failure. This feature also allows analysis of data obtained during the initial measurements and in case of wrong data or achieving certain values of the flow and ending the measurement.

The basic element of the thermal chamber is an old oven, which has sufficient thermal insulation, oversized sufficiently and appropriately positioned heating system with resistance heating coils. Another advantage is factory-built air fan for high temperature to ensure a homogeneous temperature within the chamber during the entire test. Original bimetal temperature regulator has been replaced by an electronic control unit temperature to maintain the temperature within +/- 2 ° C from the nominal temperature. Temperature monitoring for PID controller Ht60B from the company HTH8 was realized by PT100 temperature sensor placed in the oven.

The advantage of this controller is that it is able to connect to computer via RS232 using Modbus communication protocol to control and record control parameters such as the desired temperature and actual temperature.

Fig. 9 Control panel

With automatic control of the temperature chamber, it doesn’t have to be attended. By using the automatic calibration mode of control parameters, ensure proper operation during the whole measurement is ensured. Another advantage of the control unit is so-called ramp function that ensured the temperature rises linearly to avoid temperature overshoot and the possible degradation or deterioration of the sample.

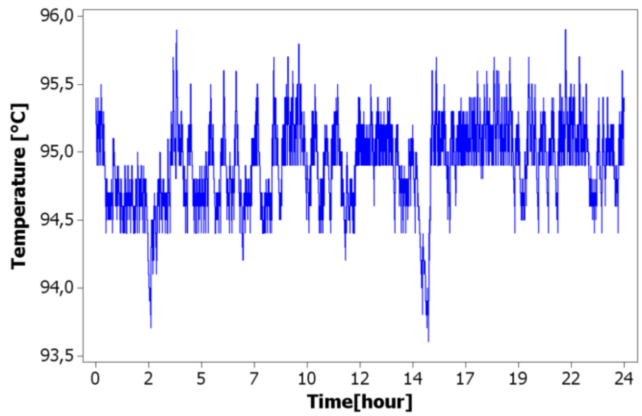

Fig. 10 Temperature during the measurements

III. Method

The shape of small samples is given by norm but some dimensions are determined only by intervals. For our test creep at elevated temperatures the sizes are determined by the Fig. 11 shown below.

A. Prepare of samples

PA6.6 was chosen as the material. It is semi-crystalline to crystalline material, which has one of the highest melting points among all commercially available polyamides. Classes are characterized by strength and stiffness, which is maintained even at elevated temperatures.PA6.6 after molding absorbs moisture, water retention is not as high as for PA6. The moisture absorption depends on the composition of the material, wall thickness, and environmental conditions.

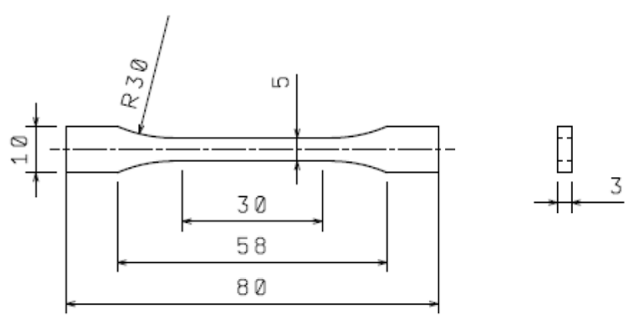

Fig. 11 The sizes of small sample

Test specimens were molded at the injection molding machine ARBUGR Allrouder 170U under the same process conditions throughout the whole injection.

Extent of water absorption affects dimensional stability properties which must be taken into account for the design of the product. Various modifiers are added to improve the mechanical properties. Glass fibers are the most common filler.


Fig. 12 ARBURG Allrouder 170U

Tab I. Technical parameters

Manufacturer	ARBURG
Model	Allrouder 170U
Clamping force	150kN
Maximal size of mold	170x170x200m
Screw diameter	22mm
Maximal injection volume	34cm3
Weight	1740kg

Adding elastomers such as EPDM or SBR improves impact resistance. Viscosity of PA66 is small, therefore it flows well, which enables forming of thin components. Flowing is not as good as in PA6. The viscosity depends on the temperature. It is resistant to most solvents, but not to strong acids or oxidizing agents. It is used in the automotive industry, covers devices, in general where impact resistance and strength is required.

Tab II. Process parameters

Track of dosage	55mm
Mold temperature	90°C
Input range temperature	250°C
Transition zone temp.	280°C
Output range temperature	285°C
Nozzle temperature	290°C
Injection speed	40mm/s
Switching point	15mm
Lockout	800bar
Cooling time	20s

To get the value of the load we first performed a tensile test on a tensile testing machine Zwick Roell 1456. After evaluation of this test, counterweights were choosen. For PA6.6 material a 9 kg counterweight was chosen. For this size of counterweight a series of creep tests at constant load were made. A series of creep tests was carried out at constant temperature for this material.

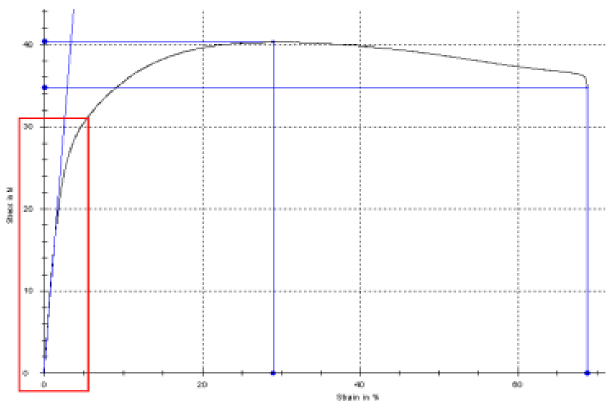

Fig. 13 Tensile curve

B. Non cross-linking material

Creep tests under constant load lasted 24 hours. The values of elongation were recorded at 5-second intervals. Load size of 9 kg was chosen for the PA6.6 material. The temperature at which the test was carried out was 80 ° C. Series of tests were performed, each series with the same load.

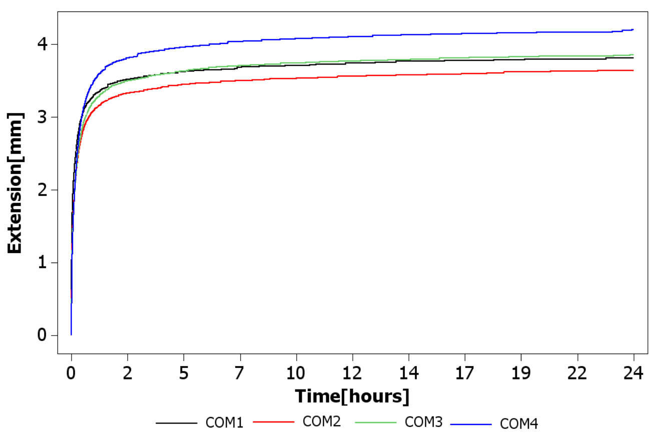

Fig. 14 Elongation of samples PA6.6

It is necessary to check for gross errors, biased and suspicious values. This inspection was carried out using boxplots diagrams. For better clarity and character, the data in the graph below are displays in four hour intervals.

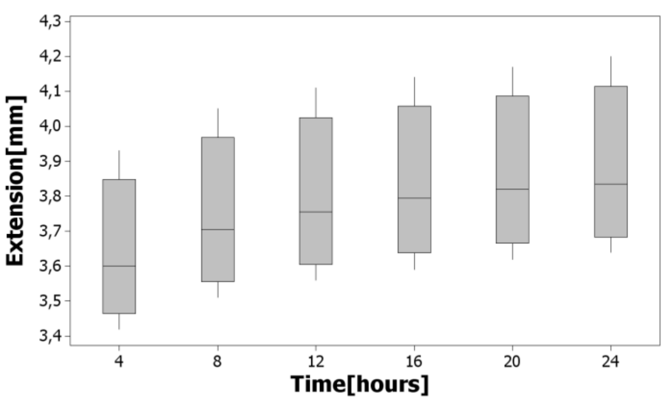

Fig. 15 Check of measured values

As can be seen from the measured values in the data record, there are no biased or suspicious values. Furthermore, we can see a very similar shape of boxes in controlled times that show us the same character data.

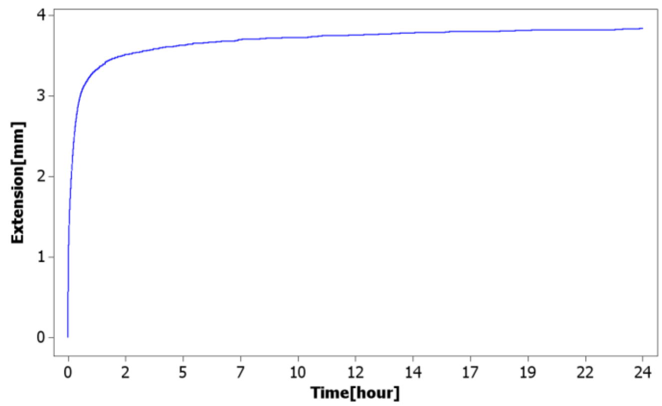

Fig. 16 Graph of median PA6.6

From these data a graph of the median - maxima / minima can be constructed, which we will represent the overall result of the measurement.

C. Cross-linking material

Another used material is polypropylene (PP). Polypropylene has good stability, hardness and strength, but low impact toughness. It is not susceptible to internal stress and is well weldable. At temperatures below zero it gets brittle. It has very good electrical and chemical resistance. PP can be used at temperatures from +5 to +100 ° C. Its advantages include low density, high thermal and dimensional stability, high surface hardness and physiological safety. The disadvantages contrary, low abrasion resistance, oxidation resistance and poor bondability. PP cannot be high-frequency welded. Used PP was modified by adding 30% glass fibers and after injection was radiation networked in BGS in Germany.[16][17]

In polymeric materials during the process of networking new links between macromolecular chains are created. This modification makes a significant change in the structure, which will bring new features of the material. The process of creating spatial network has advantages especially in:

• Improved mechanical, chemical and thermal properties.

• Modification of existing product without the unnecessary costs in waste.

• No thermal or pressure influence on the final product.

• Ability to optimize the dose of radiation to improve the properties of a material.

• Possibility of combining multiple materials in a single irradiation process.

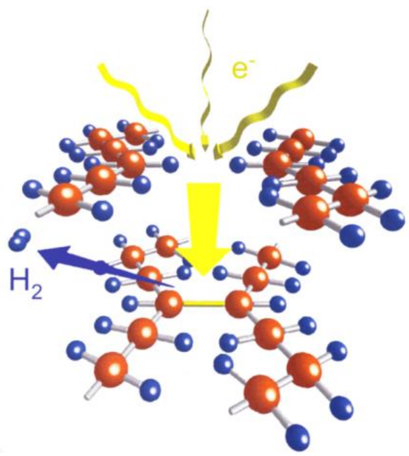

Fig. 17 Principle of radiation crosslinking [1]

The process of cross-linking occurs between two carbon atoms chains lying in close proximity, which prevents the formation of free radicals from CH bonds in polyethylene. When the CH bond breaks, macromolecules break too, which manifests as degradation of the material. With increasing radiation dose the amount of broken macromolecules increases and may have an adverse effect on the final properties.[17]

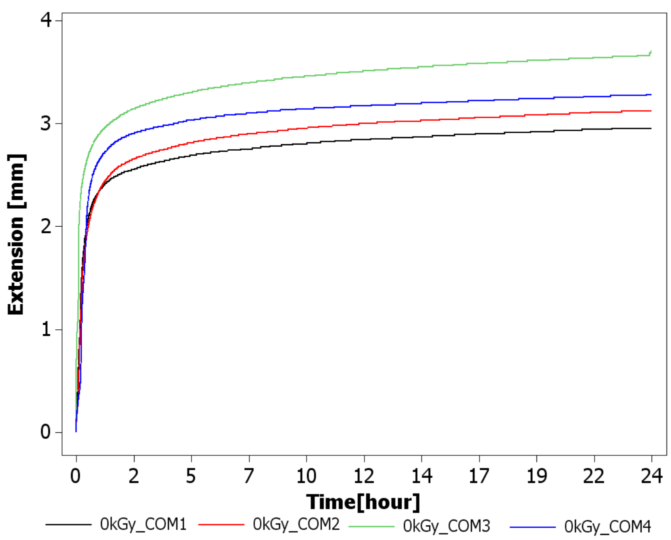

Fig. 18 PP+30%GF 0kGy

The ratio between the cross-linking and breaking of macromolecules is determined by the chemical structure of the polymeric material and the size of the dose. Due to the greater sensitivity of some materials to degradation during processing, crosslinker agents are used. Formation of these compounds not only positively affects the formation of bonds between individual chains, but also reduces the dose of radiation necessary to reach the final degree of crosslinking.[18]

The graph display elongation of small test samples of filled PP 30% GF, the absolute elongation versus time is shown. The shape of the curves shows a similar trend particularly in the second stage of creep. To check the measured data boxplot diagrams were constructed again, which in any case do not display leaning or suspicious values.

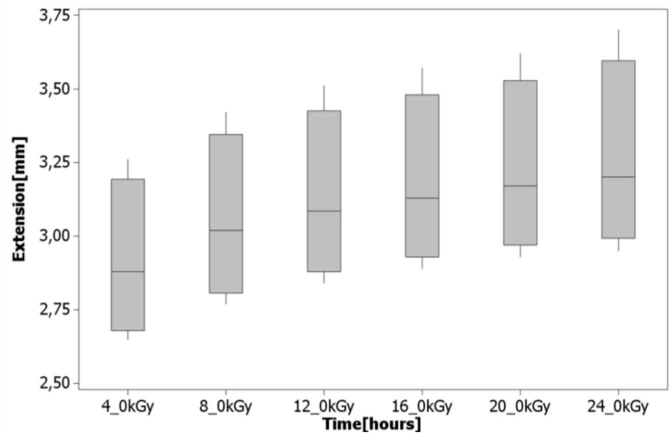

Fig. 19 Boxplot diagram for PP+30%GF 0kGy

The same method was used in the case of radiation doses 33 kGy and 66 kGy.

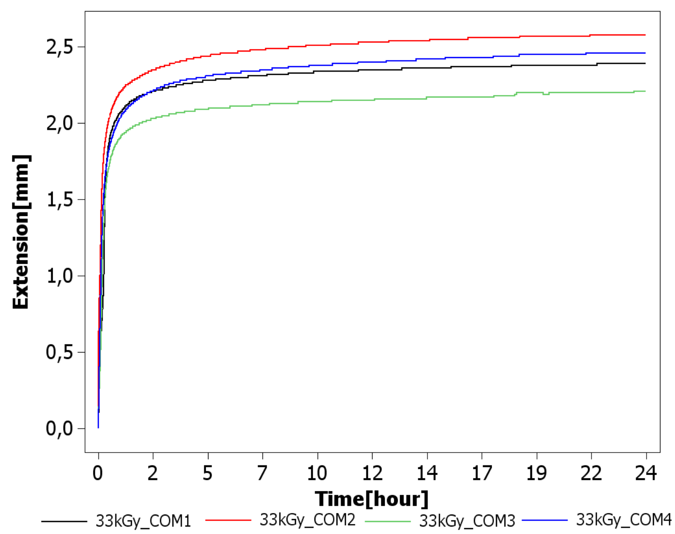

Fig. 20 PP+30%GF 33kGy

When we insert the median values in one of the charts Fig. 22 we can notice a significant difference in the amount of elongation of irradiated and non-irradiated material.

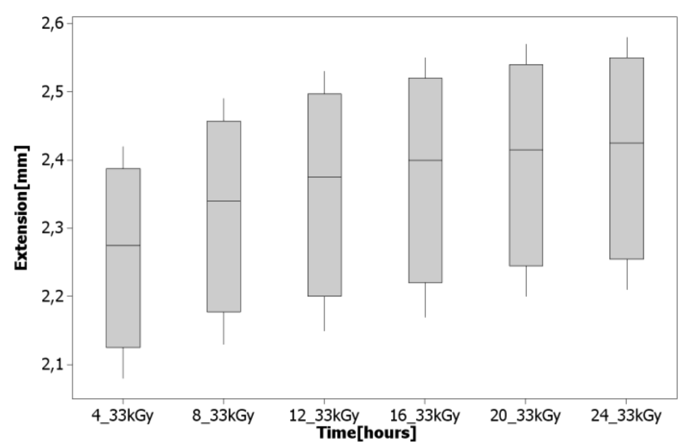

Fig. 21 Boxplot diagram for PP+30%GF 0kGy

The character of the data does not change significantly during the secondary phase of the test, which is also confirmed by balanced slope of the lines intersected with this part.

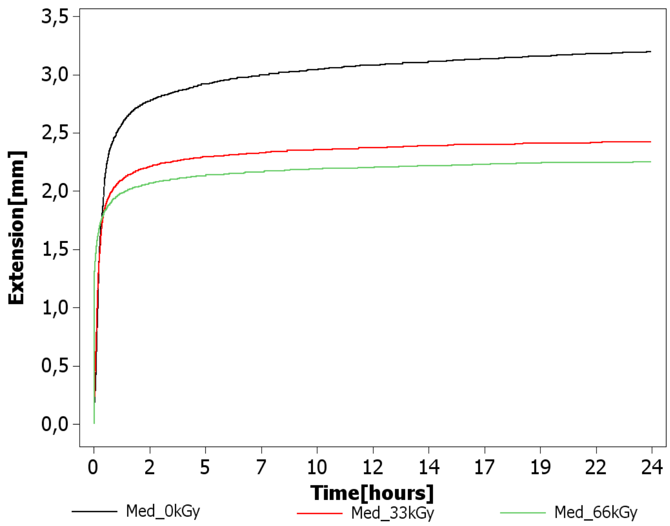

Fig. 22 Medians of PP+30%GF and dose 0,33,66kGy

IV. CONCLUSION

This article deals with the construction of equipment for the measurement of creep at elevated temperatures. The design is described with many technical details and limits of the device. For testing, series of measurements were performed on samples PA6.6 and filled PP with 30% GF. All these measurements have demonstrated the functionality of the device, suggesting new possibilities for the evaluation of creep tests.

The last graph shows an apparent effect of radiation crosslinking to elongation of specimens

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