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EXPERIMENTAL DEVICE WITH DATA ACQUISITION FOR MEASUREMENT OF TEMPERATURE OF DEFLECTION UNDER LOAD

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Abstract. *The paper presents an experimental device designed to measure the bending temperature of deflection under load, one of the most important thermomechanical characteristics of polymeric materials. The device allows the application of a prescribed load which has the effect of inducing a standard stress in a rectangular specimen through three-point bending. Subjected to this bending stress, the test specimen is heated, at a constant speed, until a prescribed deformation (deflection) is registered. The temperature and deflection are recorded continuously by a data acquisition system designed for monitoring the process and subsequent analysis of the experimental results. In order to verify the functionality of the experimental device, preliminary tests were carried out on some polymer materials that have polypropylene in their composition.*

Keywords: *temperature of deflection under load, data acquision, polymers, thermomechanical analysis*

1. INTRODUCTION

One of the most important problems associated with the use of industrial components made of polymer materials is related to the temperature limit up to which they can be used safely. After polymerization proces, at room temperature, thermoplastic polymers have a completely amorphous structure (material keep the disorder associated with melting structure) or a specific degree of crystalinity (some molecular chains are partial aligned). Elastomers are all amorphous at room temperature and exhibit a large and reversible extensibility. Isotactic polypropylene PP is a typical exemple of semi-crystaline polymer. [1]
If materials are heated, many of their properties change due to the fact that thermal energy supplied to the specimen will change the potential energy of constituent molecular chains. If the glass transition temperature is not reached, other amorphous polymer excepted elastomers are usually hard and brittle due to a low mobility of molecular chains. Because there are many factors which are involved in material behaviour (length of molecular chain, monomer nature, croslinking and crystalization degree) is dificult to make prediction on values of these characteristics. In order to measure, when polymer are heated, mechanical characteristics, shrinkage and thermal expansion thermomechanical analysis (TMA) is widely used.

There are two main classes of test types: based on evaluation of changes in the volume (expansion test) or based on evaluation of mechanical characteristics changes (penetration, tension or flexure tests). The basic difference is that in case of expansion test it is not a external force applied to the specimen. Usually, because is necesary a force control, instruments for termomechanical analysis are operated in vertical orientation and some heavies are used as force generator.

The expansion test is commonly used for measurement of CTE (coefficient of thermal expansion) and T_g (glass transition temperature) as an alternative to DSC (differential scanning calorimetry). Penetration test, in which a small tip pressed with a calibrated force penetrates the specimen, reveal the temperature (softening point). [2] This temperature is related to glass transition temperature and in case of amorphous polymers is close to T_g but in case of semi-crystalline polymers is higher than T_g [3].
Similar with softening point is temperature of deflection under load witch is measured in a tree point bending configuration.
Methods for temperature of deflection under load mesurement are specified in ISO 75-1 Determination of temperature of deflection under load. General test method [4] and ISO 75-2 gives also requirement for testing polymeric materials [5].

The result obtained using the test method described is not the limit temperature in which a polymeric material can be used in safe condition or a design parameter for prediction of endurance of material at high temperature.

1.1 Test condition

According to ISO 75-2 (1993) all test specimens shall have a rectangular shape with dimensions l x b x h (80mm x 10mm x 4mm). The specimen is subjected to three point bending under a constant load in order to produce a nominal flexural stress specified in standard (0.45, 1.8, or 8 MPa).

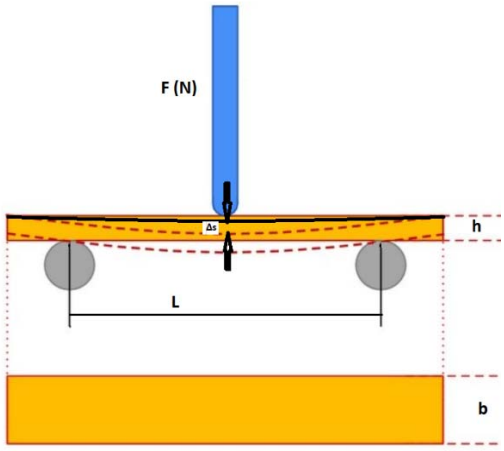


Figure 1. Three point bending loading configoration

The bending force applied is corelated with produced flexural stress by ecuation (1).

$$F = \frac{2\,\sigma_f\, b h^2}{3L} \qquad (1)$$

where
F – bending force (N)
σ_f – nominal stress (MPa)
b - specimen width (mm)
h – specimen thickness (mm)
L – span between supports

The loading assembly (shown in Figure 1) is pased on a heating bath and is controlled heated at a constant rate of (2^0C/min) until the initial deflection of the specimen (due to load applied) has increased by the standard deflection Δs.
The standard deflection are related to the flexural strain increase by ecuation:

$$\Delta s = \frac{L^2 \cdot \Delta\varepsilon_f}{600 \cdot h} \qquad (2)$$

where
$\Delta_{\varepsilon f}$ – increase in flexural strain (relative change in length of the outer surface at mid-span) during heating process (%); $\Delta_{\varepsilon f}$=0.2% according to ISO 75-2
Δs – Standard deflection - increase in deflection (displacement of a point measured at mid-span during flexure) corresponding to the flexural strain increase $\Delta_{\varepsilon f}$ (mm)
T_f – temperature of deflection under load is the temperature at which the specimen deflection reaches the standard deflection Δs. (^{0}C)

2. DESCRIPTION OF THE THERMOMECHANICAL ANALYSIS DEVICE

2.1. Device construction

The experimental device whose schematic representation is shown in Figure 2, has as basic structure a rigid frame that link the lower support points of the test piece, the mounting disk of the linear bearing and the superior platform on which the displacement measuring devices are mounted.
The bending force is applied by means of a centrally located metal rod that slides through the linear bearing.

The bending force applied to the push rod, in three point bending test, it can be changed by adition of known loads (metal discs). One of these metal discs (mandatory made of steel) is required as a sensing element for the inductive proximity transducer, AM1 / D2-5A with a resolution of 1 µm witch has been used already by the autors with resonable results for displacements measurement.[2]
For fast reading of displacement is also used an Mitutoyo dial indicator with 0.01mm resolution, usefull as fast indicator if the test is finished.

As suitable calibrated temperature-mesuring device were chosed two K-type thermocouples located in the proximity of specimen holder. It is also used succesfully temperature transducers based on integrated circuit such as LM335Z which seems to be more efficient than classical thermocouple [6] because the output signal range (larger than specific for thermocouple one) is easier to measure and recorded. More than that in such case is not necessary aditional circuits for signal conditioning as in case of thermocouple.

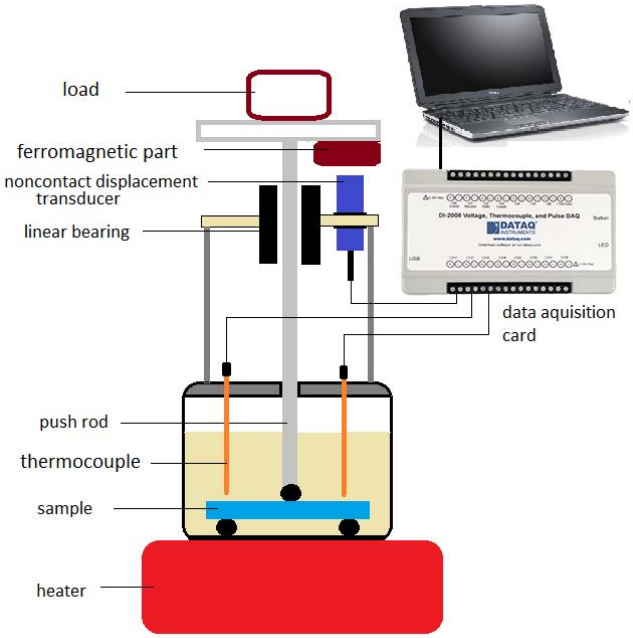
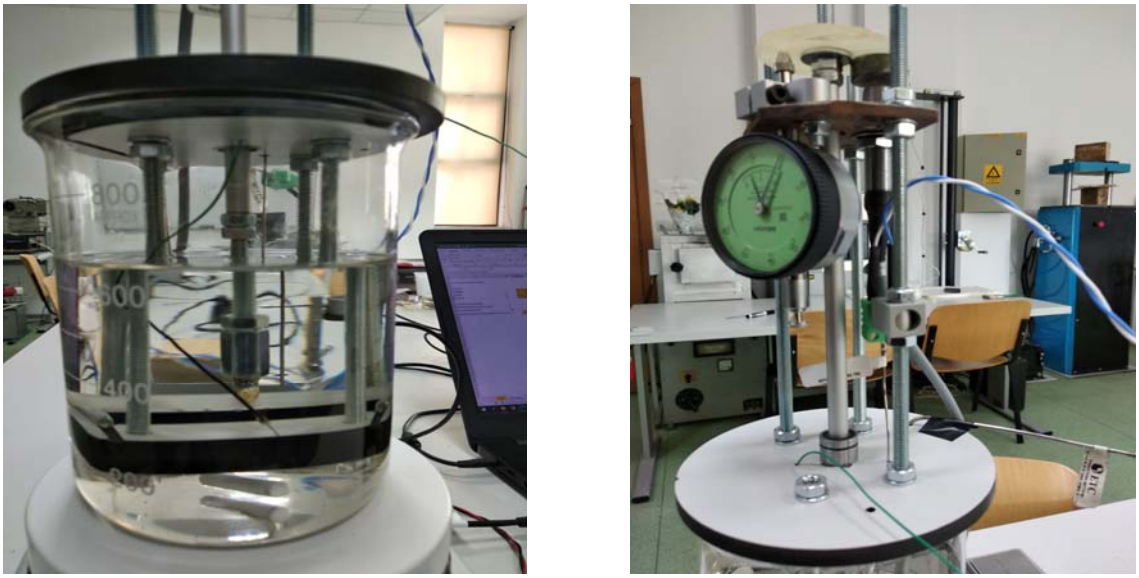


Figure 2. Block diagram of laboratory device for temperature of deflection under load determination

The heating equipment is a bath containing paraffine oil (stable over the heating temperature range) in which the rigid frame is immersed aro und 70 mm (Figure 3). The bath is heated by using a heater with temperature controller and a magnetic stirrer in order tor reach an uniform temperature in entire bath.



(a) (b)

Figure 3. An overwiev of apparatus (a) heated area (b) displacement transducers area

The displacement and temperature signals (generated by the transducers) are recorded by using a data acquisition card. Since thermocouples are used as temperature transducers, it is necessary for the data acquisition system to contain signal conditioning in order to compensate the cold junction temperature. Also because the level of measured signals are so different (voltage output of displacement transducer are in 0÷10V range and in case of thermocouples is just few mV) it is required a very versatite and configurable data acquisition card.

A resonable choise was the DI 245 data acquisition card, produced by DataQ Instruments which is able to communicate with PC through USB port.

The WinDaq Waveform Browser software for ready-to-run measurement, downloadable from the DataQ Instruments website is an appropriate software platform for comunication with PC and configuration of data acquisition sesion.

Mesurement system included four differential and isolated analog input channels with feature for programmable as voltage or thermocouple inputs. There are 8 types of thermocouples supported by DI 245, as it can be seen in Figure 4 and cold junction compensation is automatically enabled.

Each channel can be programmed to aquire data as voltage, due to an internal amplifier, into a programmable range from (-10÷+10mV) to (-50÷+50V) in order to increase measurement accuracy by using entire capacity of incorporated ADC (analog/digital converter).

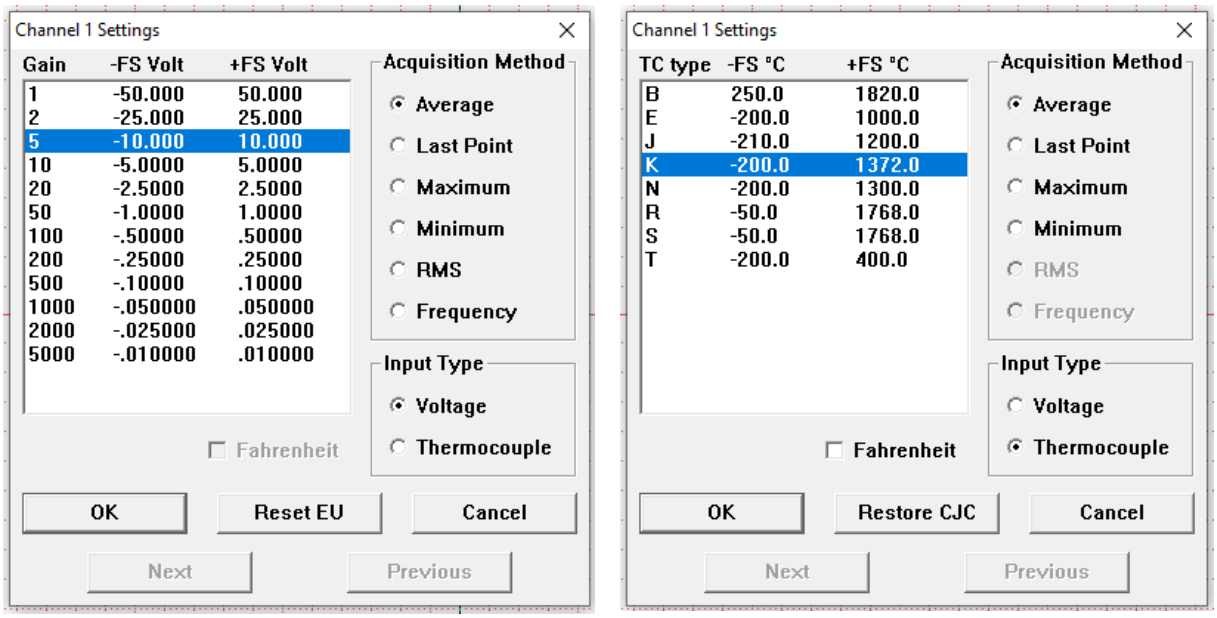


Figure 4. WinDag software (windows for parameters settings)

Maximum sample throughput rate is 2kHz in case of single enabled channel or 200Hz in case of two or more enabled channels and a minimum sample throughput rate 0.709 samples per hour.[7]
WinDaq data acquisition software can be used to record signals waveforms while monitoring a real-time display of the waveform on-screen and also to review and analyze registered waveform.
The software also has the ability to export files recorded during an experiment to Microsoft Excel for furter analysis.

2.2. Calibration

Because measurement of sample displacement Δs is the main objective of the test and contact transducers such as LVDT or resistive types involve large resistent forces (due to their springs) it was mandatory to use an uncontact transducer.
Our choise was an inductive sensor with analog output (signal conditioning included) which provide an output in 0÷10V range as function of the distance between transducer and an ferromagnetic object. In the detection area range 0÷6mm the output is unlinear as can be seen in FIgure 5. Calibration measurement has been performed by using as detected object a steel cylinder with diameter φ=20mm and thickness h=8.5mm which later was included as part of the experimental device.
Nonlinearity of transducer output is also reported by producer, in tehnical datasheet [8] .
The metalic frame and the rod are made by the same material and the proper expansion of the frame is fully compensated so it is not neccesary any furter corection in displacement values.

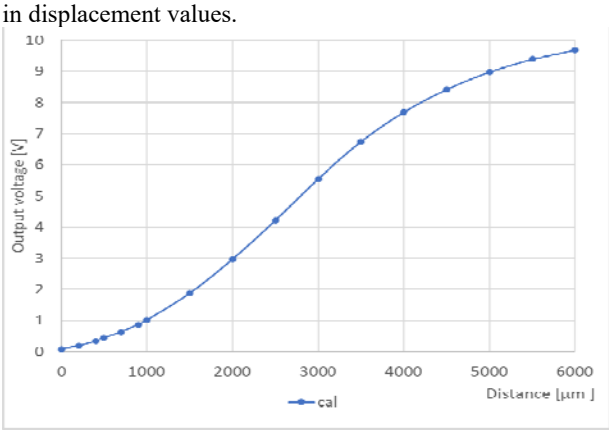


Fig. 5 The static characteristic of displacement transducer

The straith region of static characteristic of the transducer is into 2÷3 mm range and the distance from detected object it is recomended to be in this range durring entire experiment in order to record reliable data. The effect of the mass of the ansamble (rod, support disk, ferromagnetic refference object) were taken into account as contribution to the bending force. Also was taken into account, as negative contribution, the force exerted by the spring of the dial indicator.

3. TESTING OF DEVICE FUNCTIONALITY

3.1 Tested specimens

In order to test device functionality has been cut samples with rectangular shape from compression- moulded sheet, made by polypropylene(PP) and mixture polypropylene + elastomer. The test specimens dimensions are 80x10x3 mm and 80x10x6mm according to the sheet thickness.

The large application area of PP is due to the fact that has the lowest density among commodity plastics. Despite these popularity there are still problems with PP characteristics at low temperatures. By blending PP with various elastomers results combination with very high impact strength which can be a solution for low temperature behaviour.[9]

Table 1 and Figure 6 show the shape and compositions of tested specimens.

Table 1. Nature of tested materials

Composition	Sample code	Thickness [mm]
Polypropylene	PP	3
Polypropylene+30% elastomer1	PP+EL1	3
Polypropylene+80% elastomer2	PP+EL2 p1	3
Polypropylene+80% elastomer2	PP+EL2 p2	6

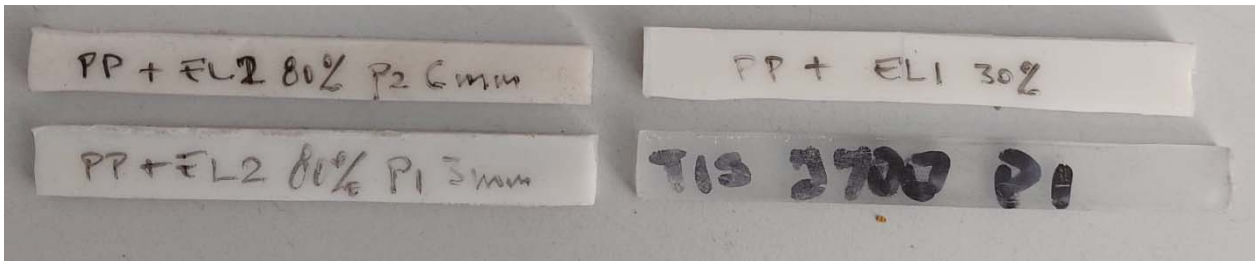


Figure 6. The specimens used in tests

3.2 Experimental results

In order to test the accuracy of the data was tested initially a polypropylene specimen an material with well known termomechanical characteristics.

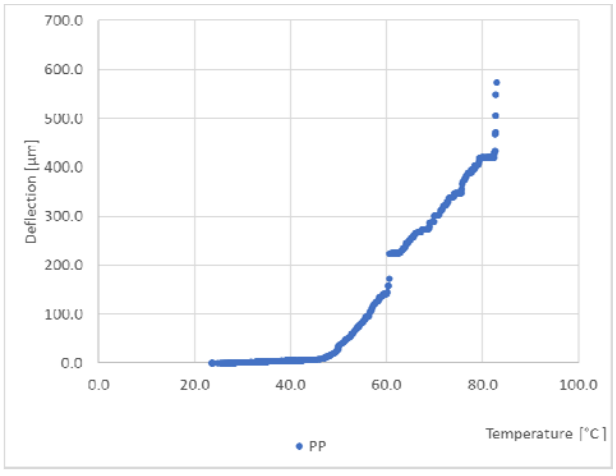


Figure 7.a Deflection vs. Temperature curve for polypropylene - entire curve s=f(T)

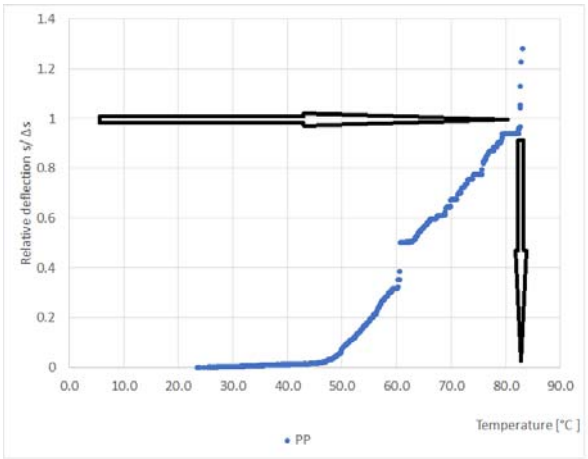


Figure 7.b Relative deflection vs. Temperature curve s/Δs=f(T)

The test result with raw data colected by data acquisition card are shown in Figure 7.a for entire temperature range in which the specimen was heated. In order to emphasize temperature of deflection under load is suitable the representation of relative displacement vs. temperature (Figure 7.b). If Microsoft Excel is used as software for processing experimental data it is posible the direct determination of temperature of deflection under load by using Vlookup function.

Temperature of deflection under load measured (82.6^{0}C) is very close to 81.9ºC which is reported in ISO-75-2 as average result of 7 laboratory work, in case of tests performed with same kind of material and test condition (0.45MPa stress loading and specimen in the flatwise position). Difference (0.7ºC) is less than the within-laboratory standard devation of the average (0.9ºC) and obvious than between-laboratory standard deviation of the average (2.4ºC).[5]

The influence of specimens thickness on the shape of displacement vs. temperature curve were tested by using samples with h= 3mm and h=6mm made by same material (a mixture PP+elastomer) under the same loading conditions σ_f =0.45 (MPa) and an heating rate around (2^{0}C / min).

It is observed that in Figure 8, the curve shapes are different and values of temperature of deflection under load 30.5^{0}C in case of thicker specimen and 32.5^{0}C in case of thinner one.

More than that seems to be a lack of data for specimen noted PP+EL1 p1, despite the fact that the sampling frecquency was the same. These kind of sample with high elastomer ratio (80%) have a mechanical behaviour close ones specific for elastomers (low stiffness). The loading force was just F=0.43N and probably the loading sistem inertia (the friction in the linear bearing) is at origin of the shape, in case of specimen with 3 mm thickness.

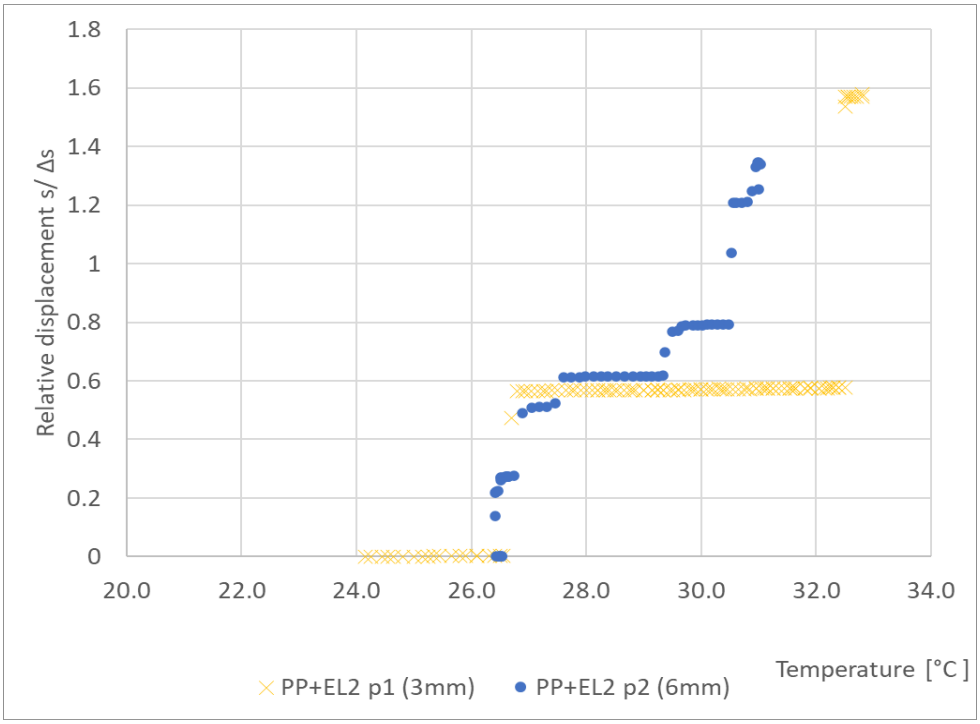


Figure 8. Deflection vs. temperature curve for mixture PP+elastomer

The sample with higher stiffness (thickness=6mm) has a more well defined curve shape and the imprecision in measurement of temperature of deflection under load is lower. As a general conclusion, if material tested have a very low elastic modulus, for better results the thickness of specimens should be biger.

In Figure 9 are shown the curve of PP in comparison with those of PP+elastomer mixtures. The tests were carried out under the same loading conditions σ_f =0.45 (MPa) and an heating rate around (2^{0}C / min).

It is obvious that the temperature of deflection under load is lower for blends and also depends on the composition (elastomer ratio).

The adition of elastomers decreases the stiffness of the material and if the PP/elastomer ratio in the mixture does not exceed a certain value, the displacement-temperature curve is almost a continuum and the temperature of deflection under load is easily emphasised.

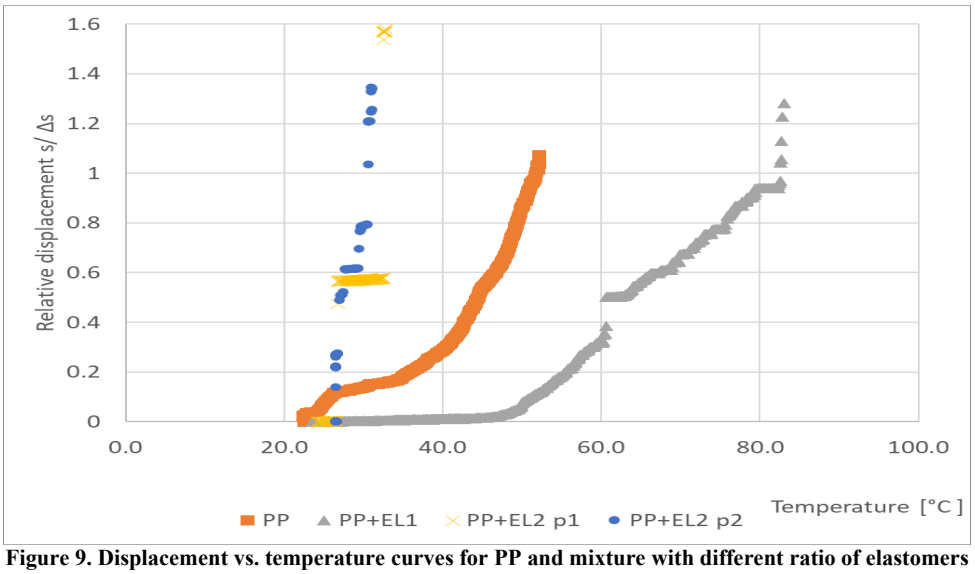


Figure 9. Displacement vs. temperature curves for PP and mixture with different ratio of elastomers

Our future project is focus on using described apparatus to determine the influence of heating rate, load level and sample dimensions on temperature of deflection under load. Also it can be an interesting investigation the correlation between test results in case of temperature of deflection under load and TMA penetration test which reveal softening point.

4. CONCLUSIONS

1. The overall accuracy of experimental device is resonable (the difference between measured temperature and the temperature reported on ISO 75-2 in case of same material is just 0.7^0C).

2. The measuring device ensures the reproducibility of the results if the inductive transducer is placed at the same distance from the detected object. Otherwise there are differencies in displacement estimation and it is mandatory an in situ calibration using the dial indicator.

3. The variation of the sample dimensions has not as effect a significant difference in the values of temperature of deflection under load, but in case of materials with low stiffness are suitable thicker specimens.

4. Data acquisition systems in combination with appropriate software are powerfull tools for process monitorization and data treatment.

5. In case of polypropylene mixed with elastomers the temperature of deflection under load decrease when elastomer ratio increases as was expected.

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