TENSILE HEAT DISTORTION TEMPERATURE OF POLYMERS FILMS USING THERMOMECHANICAL ANALYSIS

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ABSTRACT

The polymer films analysed in this work are materials used in radiation dosimetry. Tensile Heat Distortion Temperature, THDT, considered as the temperature limit at which the material can support a load for any appreciable time, is an important factor for the development and characterization of polymer films and sheeting. To characterize the dimensional thermal stability of Cellulose Triacetate (CTA), Nylon-6 and MD-polyethylene, commercial quality films, a Shimatzu TMA-50 thermomechanical analyzer was used. Specimens of each material have been analysed at different heating rates and tensile loads to determine their THDT-temperatures. Some of the experimental conditions were equal to those stated in the method described in ASTM-THDT D1637-83 in order to make comparisons of our THDT-values with those of the literature, for similar materials.

The THDT-temperatures decrease, for the analysed materials, with the increase of the tensile load, at each heating rate. Different behaviour is observed when the tensile load is fixed and the heating rate is rised. In this case the THDT-temperatures remain almost the same. Cellulose triacetate and Nylon shrink when different heating rates are applied at a tensile load of 3.5 kg/mm^2 or less. Most of the thermo-mechanical behaviour of these materials can be clarified by the fact that the elastic modulus, at a given load, decreases with the rise of temperature, producing greater deformation at higher temperature values.

I. INTRODUCTION

Most of plastics become soft at some temperature, and in this condition they are easily deformable loosing their shape under a load. In irradiation processes high irradiation doses induce heat and degradation in the irradiated materials, so that the development of a technique to evaluate their thermo-mechanical performance is very important.

Tensile Heat Distortion Temperature, THDT, defines the soften temperature as an arbitrary point in a Temperature-Deflection curve[1]. According to the norm ASTM-THDT D1637-83, the THDT-temperature corresponds to 2% deformation when the sample is heated at a rate of 2°C /min [2].

In the present work, the dimensional thermal stability of commercial quality films of MD-Polyethylene, Nylon-6 and Cellulose Triacetate have been analysed at different heating rates and tensile loads to determine their THDT-temperatures. In order to compare the results obtained in this work with those of the literature [2] for similar materials, some of the experimental conditions were equal to those of the ASTM-THDT D1637-83 standard. For these measurements a Shimatzu TMA-50 thermomechanical analyzer was used.

II. EXPERIMENTAL

Specimens of PE, Nylon and CTA, for the thermomechanical test, with 20 mm gauge length and 5 mm width were prepared by using a home made cutter. The average thickness, of each material, was 92, 90, and 100 μm respectively.

The experimental determinations were carried out using a Shimatzu TMA-50 thermomechanical analyzer. This instrument has a great operational flexibility, temperature stability, displacement resolution of 0.01 μm and other features that make of it, an adequated system for THDT determinations[3]. The temperature calibration was made with the two-point method using Indium (mp: 156.6 °C) and Lead (mp: 327.5 °C) as standards.

To study the THDT behaviour each material was tested at heating rates of 2, 5, and 10°C. At each heating rate, different tensile loads were applied such as 3.5, 11.0, 22.0, and 44.0 kg/mm^2. All measurements for each sample were made between the room temperature and close to its melting point.

To study the heating rate effect on the THDT-temperature , samples were heated at rates of 2, 5 and 10°C/min, and the tensile load was kept constant at 3.5, 11.0, 22.0 or 44.0 kg/mm^2, through each experiment. The tensile load effect on the THDT-temperature was determined keeping constant the heating rate at 2°C/min under tensile loads of 3.5, 11.0, 22.0 or 44.0 kg/mm^2.

III. RESULTS AND DISCUSSION

The thermomechanical materials behaviour is analyzed through elongation-temperature curves obtained under special conditions of tensile loads and heating rates, as stated above.

Figures 1, 2, and 3 show the materials deformation under the action of tensile loads of 3.5, 11.0, 22.0, and 44.0 kg/mm^2, and a constant heating rate of 2°C/min. Under these conditions the effect of the tensile load was determined.

From Figures 1, 2, and 3 it can be seen that, for different tensile loads, the elongation increases as the temperature is rised. Polyethylene elongation clearly becomes higher with the increase of the applied tensile loads. Nylon, on the other hand, shows a temperature range, from 40 to about 120°C, in which this material shrink slightly with a deformations of about -0.3 %, when the tensile load is 3.5 or 11.0 kg/mm^2, otherwise, the deformation increases with an increase of the tensile loads, and this behaviour is also followed in the shrinking temperature range. Cellulose Triacetate shows a temperature range, from 175 to about 225°C, in which, under a tensile load of 3.5 kg/mm^2 , the material shrinks with a deformation of about -0.25%. Outside this temperature range and load, the deformation of the material becomes higher with the increase of the tensile load.

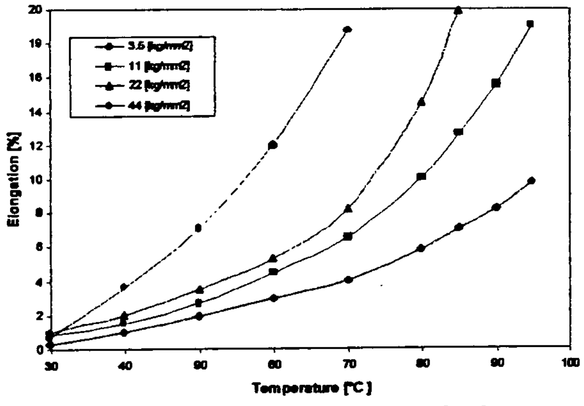

Fig. 1 -Commercial MD-PE film: tensile load effect at a heating rate of 2°C/min.

The decrease of the THDT-temperatures with increasing tensile load can be clarified by the fact that the elastic modulus, at a given load, decreases with temperature. Greater deformations are produced at higher temperatures[4].

The heating rate effect on the thermo-mechanical behaviour were determined by heating the samples at rates of 2, 5, and 10°C/mim, keeping the tensile load constant at 11.0 kg/mm^2 through the measurements. The experimental curves are given in Figures 4, 5, and 6.

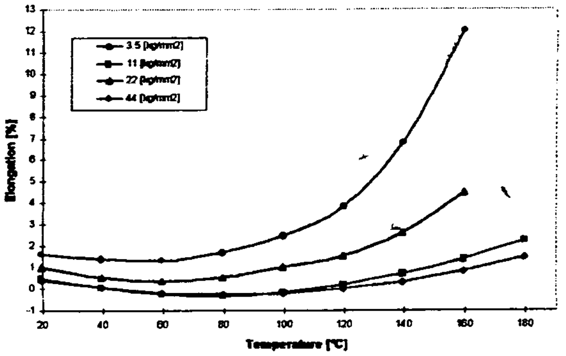

Fig.2- Nylon commercial quality film: tensile load effect at a heating rate of 2°C/min.

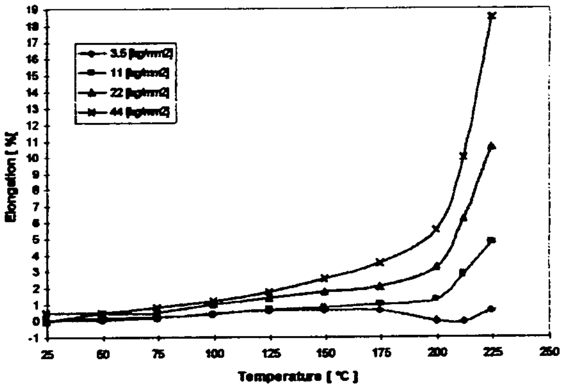

Fig.3- CTA commercial film: tensile load effect at a heating rate of 2°C/min.

Table 1 shows the results of the tensile load effect on the THDT-temperatures of the analysed materials .

TABLE 1.- Tensile Load Effect on the THDT. Heating rate 2°C/min.

Tensile Load [kg/mm2]	2 % Elongation		
	MD-PE	Nylon	CTA
3.5	51.0°C	–	–
11.0	44.2 °C	206.3°C	175.6°C
22.0	40.0°C	163.5°C	130.0°C
44.0	34.5°C	132.2°C	87.5°C

Considering the experimental conditions, since the involved errors are about ± 10 %, no drastic heating rate effect was observed. It can be pointed out that the curves run almost together in the considered temperature ranges, leaving the THDT-temperature quite constant for each material. So it was about 42°C for PE, 151°C for Nylon, and 204°C for CTA. The temperature values for 2% deformation are given in Table 2.

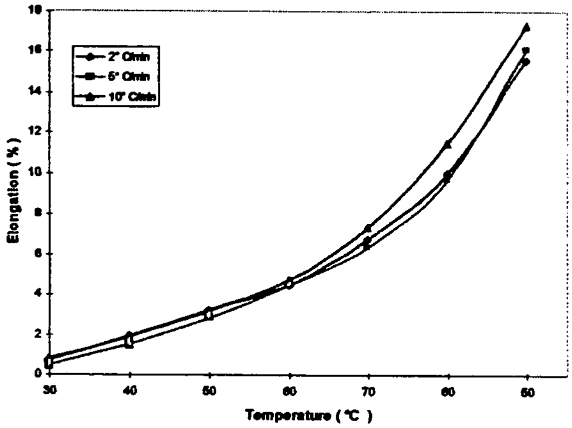

Fig.4- PE commercial film: heating rate effect with tensile load 11.0 kg/mm^2.

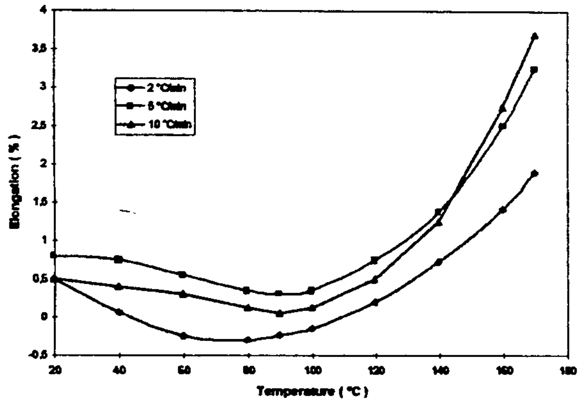

Fig.5- Nylon commercial quality film heating rate effect with tensile load 11.0 kg/mm^2.

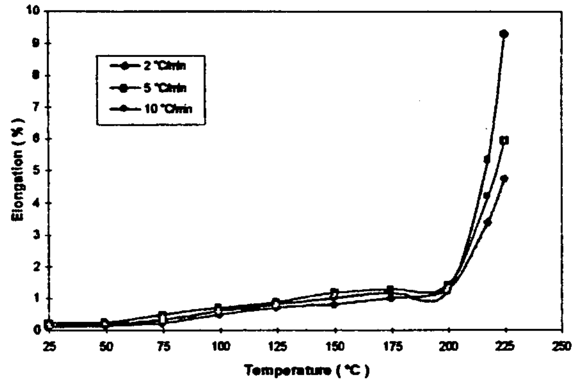

Fig.6 - CTA commercial fim: heating rate effect with tensile load 11.0 kg/mm^2.

TABLE 2.- Heating rate effect on the THDT at 11.0 kg/mm^2

Heating rate [°C/min]	2% Elongation		
	MD-PE	Nylon	CTA
2	40.8°C	--	205.8°C
5	43.8°C	151.7°C	203.5°C
10	41.4°C	150.7°C	204.7°C

IV. CONCLUSIONS

The experimental results show that the THDT-temperature, for each material, decreases with the increase of the tensile load. This behaviour can be clarified by the fact that the material elastic modulus, at a given load, decreases with temperature. Greater deformations are produced at higher temperatures.

The heating rate effect, performed under a constan tensile load and heating the samples at different heating rates, show no drastic differences on the deformation-temperature curves, since their run close together along the temperature range. These results are similar with those founded in the literature for the same material tested under similar conditions [2]. This behaviour can also be clarified by the fact that the elastic modulus of the materials depends on the equilibriun temperature of the whole sample. In films, due to its thickness, this equilibrium temperature is reached with almost no time delay produced by the different heating rates. This situation could be different with bulky samples.

These thermo-mechanical analysis provide valuable information to establish the upper limits of doses rate that these materials can stand during an irradiation process.

REFERENCES

[1] Nielsen, L.E. and Landel R.F., **Mechanical Properties of Polymers and Composites,** Marcel Dekker, Inc.N.Y., 1994.

[2] Moscato, M.J., **The Use of Thermomechanical Analysis as a Viable Alternative for the Determination of the Tensile Heat Distortion Temperature of Polymers Films,** Riga, A.T.and Neag,C.M., Eds. American Society for Testing and Materials, Philadelphia, 1991.

[3] Shimadzu Scientific Instruments, Inc.,**Thermal Analysis System Application into Polymeric Materials - Manual,** Maryland, 1994.

[4] Hawkins,W.L. **Polymer Chemistry,** Materials Thecnology, Volume II, Prentice-Hall, Inc., N.J., 1970.