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DETERMINATION OF THE HEATSETTING TEMPERATURE OF POLYESTER BY TMA

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Abstract

The determination of the effective temperature of the thermal treatment applied to polyester substrates in the textile process has been broadly studied by differential scanning calorimetry (DSC). In this investigation, the authors have studied the possibilities of the thermomechanical analysis (TMA) as a method for the determination of this temperature. For this purpose, fabrics of polyester heatset in an industrial plant between 160 and 210°C, have been analyzed by DSC and TMA. The good results obtained show the possibilities of this technique for the determination of the effective temperature of a thermal treatment.

Keywords: differential scanning calorimetry, effective temperature, polyester, thermomechanical analysis

Introduction

During the textile process, polyester fibres are subjected to diverse treatments at high temperatures in wet or dry media. The correct and uniform application of these treatments confers to polyester all the qualities that make it appropriate for its textile use. The most intense of these thermal treatments is the heatsetting, where the substrate is subjected during 30–90 s to a temperature of 160–210°C. An insufficient, excessive or not uniform treatment temperature can produce irregularities in the dyed product. For this reason, the objective knowledge of the heatsetting temperature is very important.

Various authors [1–3] have broadly studied the determination of the effective temperature of the thermal treatment applied to polyester substrates by differential scanning calorimetry (DSC). This temperature is determined from the temperature of the peak of a small endotherm previous to the melting peak (premelting endothermic peak, PEP) that appears in the DSC curves. This PEP corresponds [1, 2] to the melting of the crystallites formed in the secondary crystallization which occurs during heatsetting operation.

Given that Themomechanical Analysis (TMA) is an analytical technique normally used to measure dimensional changes in materials with temperature, the crystallinity increase due to heatsetting, which may cause dimensional changes in

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substrates, could be detected. The authors have considered applying this technique to substrates heatset in a wide range of temperatures. For this purpose, fabrics of polyester heatset in an industrial plant between 160 and 210°C for 90 s have been analyzed by DSC and TMA.

Experimental

Material

Polyester fabric, 160 g m^{-2} area density.
Weft: textured semidull polyester, nominal count 167 dtex /30 f.
Warp: bright filament yarn polyester (multilobal), nominal count 120 dtex /46 f.

Treatments

Scouring

The fabric was first washed in water to eliminate sizing. Then it was scoured at 80°C for 30 min with a dissolution containing 0.2% (o.w.f) of Detergent PS (mixture of anionic and non-ionic surfactants) and 0.2% (o.w.f) of Detergent 124 (mixture of surfactants and solvents). Finally rinsing with warm water first, and cold water afterwards.

Heatsetting

Heatsetting was horizontally applied in an industrial stenter (Bruckner) of six sections, in the plant of Tints i Aprestos Valls. Heatsetting was performed at nominal temperatures of 160, 170, 180, 190, 200 and 210°C. The time of residence along the six sections was kept constant (90 s). The resulting crystallinities were of 50.3, 52.8, 53.8, 55.9, 57.0 and 58.7, respectively [4].

Characterization

DSC

Trials have been performed in a Perkin Elmer DSC-7 unit. The following analytical conditions were used for the polyester fibres (3):

Initial temperature: 50°C; Final temperature: 300°C; Heating rate: 20°C min^{-1}; Purging gas: nitrogen, 30 mL min^{-1}

TMA

For thermomechanical analysis a Mettler Toledo TMA/SDTA 840 was used. The substrates were prepared in a special support for fibres to obtain samples 12.8 mm of gage length clamped with a copper staple. The set staple-yarn is introduced in the analyzer and the analysis performed under the following conditions:

Initial temperature: 35°C; Final temperature: 230°C; Heating rate: 10°C min^{-1}; Dynamic strength: 0.17-0.33 N; Frequency: 1/12 Hz; Purging gas: nitrogen, 35 mL min^{-1}

Results

DSC

The temperature of the maximum of the PEP, corresponding to the effective temperature of heatsetting, was measured from the DSC curve. Results are shown in Table 1 and represented in Fig. 1.

Table 1 Effective heatsetting temperature of the studied substrates

Nominal temperature of heatsetting/°C	160	170	180	190	200	210
Effective temperature of heatsetting/°C	161.9	175.3	184.8	195.3	206.2	216.2

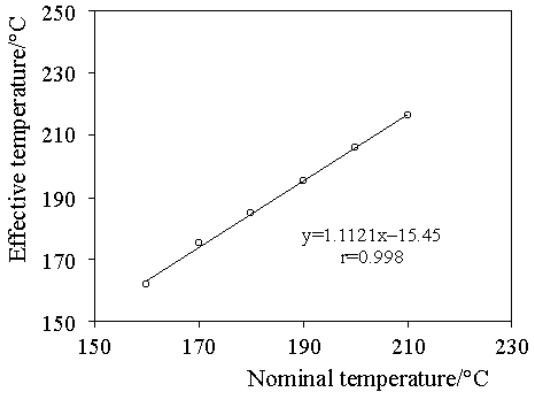

Fig. 1 Relation between effective and nominal temperatures of heatsetting

Table 1 shows that the melting temperature of the small crystallites formed during the heatsetting treatment increases on increasing the nominal temperature where effective temperatures are higher than nominal temperatures.

TMA

An example of the curves obtained is represented in Fig. 2. The amplitude of the periodical deformation induced by the periodical stress applied can be observed. Transition temperatures have been determined using the mean curves that are represented in Fig. 3.

The different inflexions in the thermograms vary on increasing the heatsetting temperature of the studied substrates. In order to obtain a more accurate determination of the transition temperatures, the first derivative as a function of the test temperature has also been determined. Resulting curves (Fig. 4) give information about the deformation rate of the substrates. These curves present two different maxima. The first one appears at

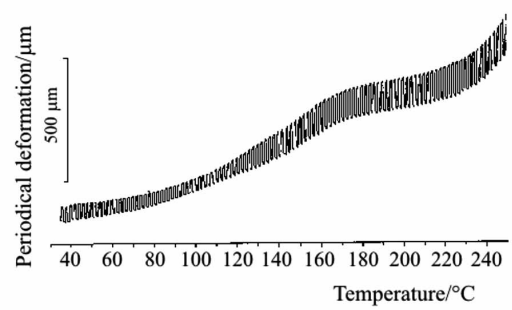

Fig. 2 TMA curve obtained in the indicated conditions for the substrate heatset at 190°C

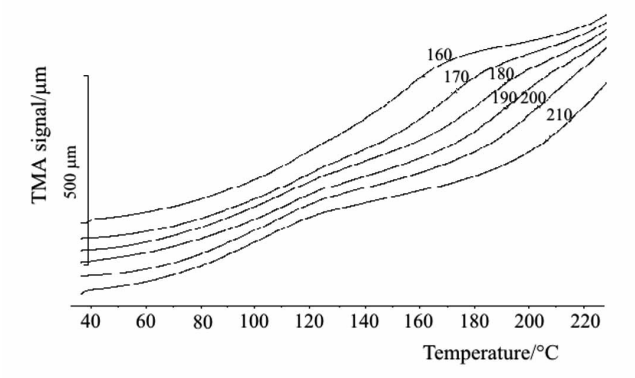

Fig. 3 TMA curves of all heatset substrates

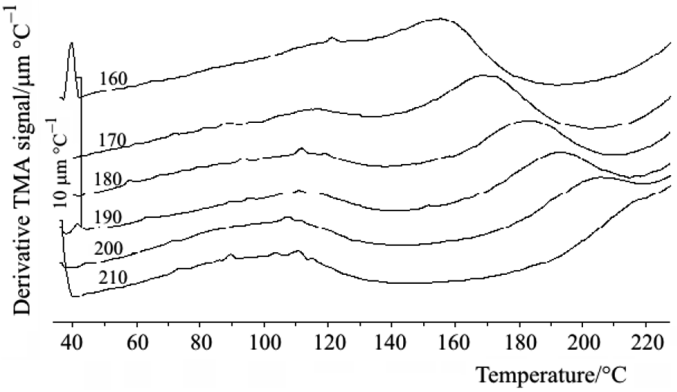

Fig. 4 Derivative TMA curves in function the test temperature for the heatset substrates

lower temperatures and decreases as heatsetting temperature increases, while the second maximum is observed at higher temperatures and increases on increasing the heatsetting temperature. Table 2 includes the pertinent values of both peaks.

Table 2 Characteristic temperatures of the TMA derivative curves for the studied substrates

Nominal temperature of heatsetting/°C	Peak 1/°C	Peak 2/°C
160	118.9	155.6
170	115.1	167.0
180	113.5	182.0
190	111.6	192.7
200	105.4	203.5
210	100.4	217.2

Figure 5 represents the evolution of the first maximum of the curves as a function of the nominal heatsetting temperature. It shows that the first maximum decreases on increasing the heatsetting temperature. As the first maximum of the derivative curve corresponds to an inflexion of the curve, and due to the fact that, when dilatometric measurements are carried out, the inflexion of the curve volume/length *vs.* temperature is associated with the glass transition temperature (T_g) [5], it means that this maximum corresponds to T_g. The decrease of T_g determined by TMA on increasing heatsetting temperature could be produced by a decrease of the orientation of the amorphous phase of polyester fibres [6].

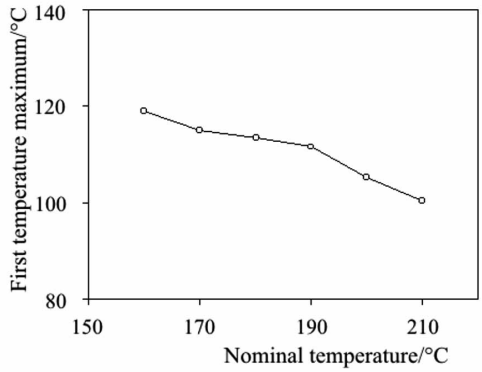

Fig. 5 Relation between the 1st temperature maximum of the derivative TMA curve and nominal temperature of heatsetting

During its manufacturing process polyester fibres are stretched and subjected to some thermal or hydrothermal treatments that increase polyester crystallinity and/or orientation. DSC analysis of polyester fibres can hardly detect the glass transition temperature, however, this temperature can readily be evaluated through the TMA technique.

In order to guarantee that the temperature of this first peak corresponds to T_g, the Young's modulus analysis of the periodical stress applied to the deformation curves has also allowed to calculate the phase angle. As it is well known, the temperature of the maximum shift in the phase angle corresponds to T_g [5]. Results of the temperature of the peak are shown in Table 3.

Table 3 Temperature of the peak of the phase angle of the studied substrates

Nominal temperature of heatsetting/°C	160	170	180	190	200	210
Temperature of the phase angle/°C	118.5	116.0	114.6	113.8	113.4	110.3

The compaction of these values with the temperature of the first peak of the derivative curves (Table 2), could allow concluding that the temperature of the first maximum of the derivative curve corresponds to the glass transition temperature.

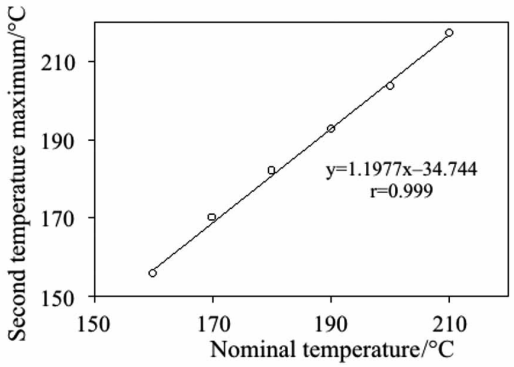

Fig. 6 Relation between the 2nd temperature maximum of the derivative TMA curve and nominal temperature of heatsetting

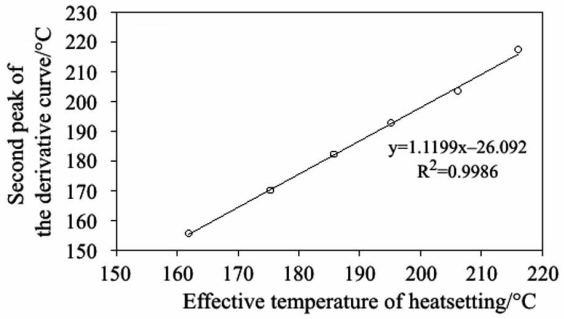

Fig. 7 Relation between the 2nd temperature maximum of the derivative TMA curve and effective temperature of heatsetting obtained by DSC

Figure 6 represents the evolution of the second maximum of the derivative curves as a function of the nominal heatsetting temperature. It shows the existence of an excellent linear correlation between both values (correlation coefficient=0.999).

Values of second peak temperature of the derivative curves obtained by TMA are similar to the effective temperature of heatsetting obtained by DSC. Both temperatures have been compared in Fig 7. that shows an excellent linear correlation (y=1.1199x–26.092; r=0.999) between them. This can be interpreted in the sense that the second peak of the derivative curve obtained by TMA corresponds to the effective heatsetting temperature.

Conclusions

The following conclusions may be drawn from this study:

- The determination of the transition temperatures from TMA curves of heatset polyester fibres is possible through the determination of the first derivative curve, this curve presents two maximums.
- The first maximum of the TMA derivative curve of polyester fibres corresponds to the glass transition temperature. T_g decreases on increasing nominal heatsetting temperature. TMA has allowed determining this transition temperature that is not detectable in the DSC analysis.
- The second maximum of the TMA derivative curve of polyester fibres corresponds to the effective temperature of heatsetting.

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