



High resolution two-dimensional dose mapping of electron beam processed polymers

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ABSTRACT

The recent advances in electron beam processing of polymer samples require increasingly dose mapping techniques with depth and volume resolution capability. In this paper, the use of micro-indentation and thermo-mechanical analysis is proposed to fulfill the necessary requirements in terms of resolution and accuracy. Experimental procedures are used to acquire mechanical data that are successively converted into dose levels based on dedicated calibrated samples. Physical principles, experimental artifacts, and limitations are discussed with special focus on calibration and validation of a simulator for dosimetry in electron beam cross-linking of electrical cables.

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1. Introduction

Dosimetry techniques with high spatial and volume resolution are increasingly needed in radiation processing of materials. This is for instance the case in medical devices, where tools are required for dose mapping in electron beam processed bioresorbable polymers with complex three-dimensional geometries (Dumba, 2011). An additional application field, where both spatial and volume resolution are required, is the calibration and validation of the Monte Carlo simulation tools, which are more and more used for optimum design of radiation processes.

This paper proposes the use of micro-indentation (Oliver and Pharr, 1992) and thermo-mechanical analysis (TMA) (Hatakeyama and Quinn, 1994) to acquire two-dimensional (2D) maps of the local dose-dependent visco-elastic properties in cross-linked polymer samples. Special attention is paid to two-dimensional dose mapping in electron beam processing of cables and to the use of both techniques to calibrate and validate the recently developed Monte Carlo simulation tool EBXLINK3D (Ciappa et al., in press).

The experimental conditions, which have been assumed to measure 1D and 2D dose distributions in bulk and cable-like structures, are described in Section 2. The related experimental results are presented and discussed in Section 3.

2. Experimental

2.1. Irradiation

The samples for TMA and micro-indentation have been irradiated with electrons by a Dynamitron at 870 ± 10 keV energy and at beam current of 10 ± 0.5 mA. The samples have been placed for irradiation on the top of a motorized wood table moving at constant speed perpendicularly to the beam scanning direction. In order to avoid detrimental thermal effects, the total dose has been cumulated by successive partial exposures. The total dose absorbed by the samples has been monitored by radiochromic thin film dosimeters located on bulk polyethylene blocks in the close proximity of the targets. The beam energy has been measured both by the depth of penetration in dedicated, precisely machined polyethylene tablets, calibrated aluminum foils, and by a total dose calorimeter (Ciappa et al., 2010). The distribution of the fluence along the scanning direction of the beam has been characterized at different energies by radiochromic thin film dosimeters mounted on the top of an aluminum bar, whose major axis has been inserted into the irradiation field perpendicularly to the beam scanning direction (Ciappa et al., 2010). The size and the shape of the pencil beam have been measured by the knife-edge technique (Ciappa et al., 2010).

2.1.1. Samples for TMA

For this experiment a 160 ± 5 μ m thick polyethylene foil has been rolled around a copper rod with 5 mm diameter. As shown in Fig. 1, the length of the film has been calculated to avoid wrinkles in the beam direction and has been wrapped very tightly to exclude

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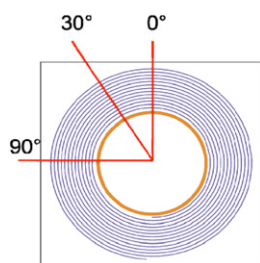


Fig. 1. Schematic cross-section of the roll used for TMA measurements. A 160 μm thick polyethylene film has been rolled around a copper core (13 turns). After irradiation along the 0° direction, samples have been stamped out from the layers 1, 4, 7, 10, and 13 (referred to the surface of the roll) along the indicated angles.

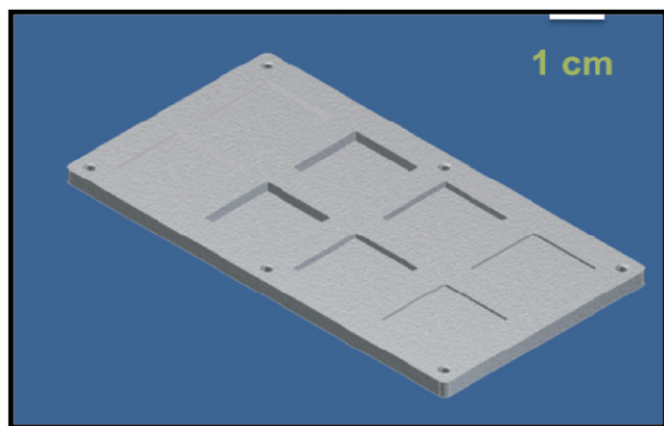


Fig. 2. Monolithic polyethylene tablet used for dose–depth measurements with TMA. The windows have different thicknesses covering the range of interest (Ciappa et al., 2010).

any air inclusions with consequent local plasma formation. The angle and depth reference has been defined by a perfectly vertical ($\pm 0.5^\circ$ resolution) cut at both edges of the roll traversing all layers. The irradiation angle of the roll referred to the vertical direction is defined by rotating this mark by means of a goniometric system with approximately $\pm 2^\circ$ resolution. Rolls have been irradiated at 0°, 30° and 90°, according to the description in Fig. 1. Once irradiated, the rolls are unwrapped, and disks (4 mm diameter) are stamped out from the polyethylene film at the different depths defined by the marks, i.e. at 80, 560, 1040, 1520, and 2000 μm measured in radial direction from the surface of the roll. The TMA measurement is performed at the center of the disk with a lateral resolution of ± 0.5 mm. Due to the finite size of the imprint by the TMA (300–700 μm), the angular resolution ranges from 2° to 5° in the outer layers and 3° to 8° in the layer closer to the copper core. Therefore, the dose value delivered by TMA is the value averaged over this angle. The depth resolution is better than ± 5 μm since the thickness of each disk is measured before starting the TMA analysis. All rolls have been exposed three times at the conditions defined in Section 2.1. The total surface dose as measured by radiochromic thin film dosimeters FWT-60 (Far West Technology, 2011) on the top of the polyethylene blocks is 161 ± 5 kGy.

An additional experiment has been performed at the same irradiation conditions, where the dose measured by TMA has been compared quantitatively to the dose measured by radiochromic thin film dosimeters FWT-60. Disks with 10 mm diameter have been stamped out from a 160 μm thick polyethylene foil. Electron range measurements have been conducted using the calibrated polyethylene tablets (Fig. 2), which are used to measure the absorbed dose at depths from 0 to 5000 μm in polyethylene (Ciappa et al., 2010). The measurement of the dose–depth curve has been carried out firstly

by radiochromic thin film dosimeters, then using the polyethylene disks, which have been subsequently characterized by TMA.

The dose calibration of the TMA measurements is performed by TMA characterization of the same polyethylene foil, as used before, which has been mounted on the top of a polyethylene block and irradiated at different dose values measured at the same location by radiochromic thin film dosimeters. The calibration curve provided by the manufacturer has been used for dose calibration of the radiochromic films. Corrections factors for production batch variability, temperature and humidity have been also considered.

2.1.2. TMA system

The thermo-mechanical analysis tool used in this work is the TMA/SDATA840 analyzer by Mettler Toledo. The system is operated in the Thermal Analysis mode, i.e. the indentation depth into the material of a quartz probe driven at constant normal force is measured, while the sample temperature is increased at a constant rate. For the film polyethylene samples, a temperature rate of $10^\circ\text{C}/\text{min}$, a constant force of 0.2 N, and a quartz probe with 0.55 mm bending radius have been chosen to resolve with the best achievable accuracy dose levels in the range from 0 kGy up to 200 kGy. The measurable dose range can be extended (at cost of the resolution in the lower dose range) by increasing the applied force. Since the melting point T_m of the material is close to 120°C , the TMA curves have been acquired in the temperature range from 20°C up to 200°C . The indentation radius at full indentation of the probe (160 μm) is 335 μm , corresponding to a volume of 39×10^{-6} cm³.

2.1.3. Samples for micro-indentation

Ten polyethylene slabs (width 40 mm, length 40 mm, thickness 4 mm) with mirror-like surface quality are stacked and firmly clamped together to simulate a single block without air inclusions. The block is irradiated along a single side (at the conditions described in Section 2.1), in order to obtain an almost ideal one-dimensional dose distribution in the central slabs. Once irradiated, the slabs are unclamped and the central plates are selected for micro-indentation analysis. Slabs are produced by multiple irradiations with surface doses of 50, 100, and 150 kGy, as measured by radiochromic thin film dosimeters FWT-60 mounted on the top of the clamped blocks. Micro-indentation measurements are performed along the depth of the slabs, starting from the irradiated surface. Measurements are also performed on the opposite (non-irradiated) side, as reference.

2.1.4. Micro-indentation tester

An MHT indentation system by CSM Instruments (CSM Instruments, 2011) is used, which is operated in the Micro Hardness mode at room temperature. In the micro-indentation tester, an indenter probe with a known geometry is driven into the material to be tested, by applying an increasing normal load. When reaching a pre-set maximum value, the normal load is reduced until partial or complete relaxation of the material occurs. Then the probe is retracted. This procedure is performed at different known locations. At each stage, the position of the indenter relative to the sample surface is precisely monitored with a differential capacitive sensor. For each loading–unloading cycle, the applied load value is plotted as a function of the corresponding position of the indenter. The resulting load–displacement curves are analyzed to extract the parameters of interest. In particular, the elastic modulus and hardness are calculated according to the power law method developed by Oliver and Pharr (1992). The Vickers hardness is extracted from the ratio of the applied force to the projected contact area of the

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