

RESEARCH PAPER

Nano-dimension pore structure analysis of poly (ethyleneterephthalate) knitted materials: An insight combining SEM images

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ABSTRACT

Scanning electron microscopy (SEM), an easily acquired and widely applied image acquisition and analysis method, has rarely been used to study the pore structure of poly (ethylene terephthalate) knitted fabrics after ultraviolet irradiation and simultaneous ozone gas exposure. In this work, we present an investigation of nano-dimension detection of the pores and fractures using SEM observations. The morphological characteristics of the poly (ethylene terephthalate) fabric surface can be revealed by SEM method. By detecting 9 high resolution SEM images, the pores morphology of different scales were acquired. In addition, the studied poly (ethylene terephthalate) knitted substrate shows different types of pores and holes with multi-resolution at the surface layer after ultraviolet irradiation and ozone gas exposure for 80 minutes. This work demonstrates that the combination of two dimensional (2D) SEM results is both effective in the detection of surface morphology and significant in revealing the pore structure of materials at nano-dimension scale. Sub-micron porosity with pore radii as small as 2.5–10 nm was observed in SEM cross-sections. The formation of nano-dimensional pores on the surface of the fabric is because of the physical etching (due to the ion bombardment in the radiation chamber).

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INTRODUCTION

Scanning electron microscopy (SEM) is utilized to examine the pore morphology and topography of the surface of materials [1]. This investigation began in a work to explore the changes in the surface morphology and topography of poly (ethylene terephthalate) knitted materials by means of the sample's SEM images after ultraviolet irradiation and simultaneous ozone gas exposure for 80 minutes. The main innovation of this paper is twofold:

- 1) Combining SEM 2D results to comprehensively study the nano pores morphology of poly (ethylene terephthalate) knitted materials, and
- 2) Obtaining high-resolution SEM images.

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Moreover, in this regard, to the best knowledge of the authors, papers on the SEM observation of the pore structures on the poly (ethylene terephthalate) knitted materials could not be found in the literature.

METHODOLOGY

Sample preparation and experimental procedure

The following fabrics are applied throughout this exploration: Pure poly (ethylene terephthalate) fabrics (100 %) with the weight, thickness and Yarn linear density of 18.45 g/m², 0.781 mm and 150/144 dtex/filament, respectively. These fabrics were kindly supplied by Nature Works LLC Company, USA.

The poly (ethylene terephthalate) fabrics were scoured with 2 g/L anionic/non+ionic detergent,



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Fig. 1. SEM unit



Fig. 2: Sputter coating unit.

1 g/L Kieralon Jet B conc. (non-ionic surfactant, BASF) and 1 g/L sodium carbonate ('soda ash') at 60 °C for 15 min at a liquor ratio of 10: 1, and washed thoroughly. After scouring, the fabrics were rinsed with cold water for 10 min and dried in ambient conditions to remove mill dirt and lubricants.

The poly (ethylene terephthalate) fabrics were irradiated in a Ultraviolet/Ozone gas irradiation cabinet for 80 min (40 min face-up and 40 min face-down) (Ultraviolet/O3 radiation cabinet: 11 mW/cm² intensity UV lamps without outer envelope (6 Lamps, made in Poland) is placed in a cubic box with the side length of 60 cm. Strips of samples are placed around the source at a suitable distance (~2 cm). Atomic oxygen is generated both when molecular oxygen is subjected to the 184.9 nm radiation and when ozone is irradiated at 253.7 nm. The radiation at 253.7 nm is absorbed by most

hydrocarbons and also by ozone).

When the irradiation was completed, the sample was rinsed adequately with cold water (30+40 °C) at room temperature for 10 min in a liquor to goods ratio of 20:1, and then oven dried at 60 °C for 30 min.

Acquisition of high-resolution images

In this study, the acquisition of high resolution images was conducted on a XL30MODEL/PHYLIPS Company/Netherland SEM (Fig. 1).

In order to avoid problems due to charge build-up, the fabrics were previously sputter-coated with gold palladium for two minutes in a SCDOOS MODEL/Bal+Tech Company/Switzerland sputter coating unit (Fig. 2).

In order to reduce the influence of human factors on the experimental results, the following procedures were considered:

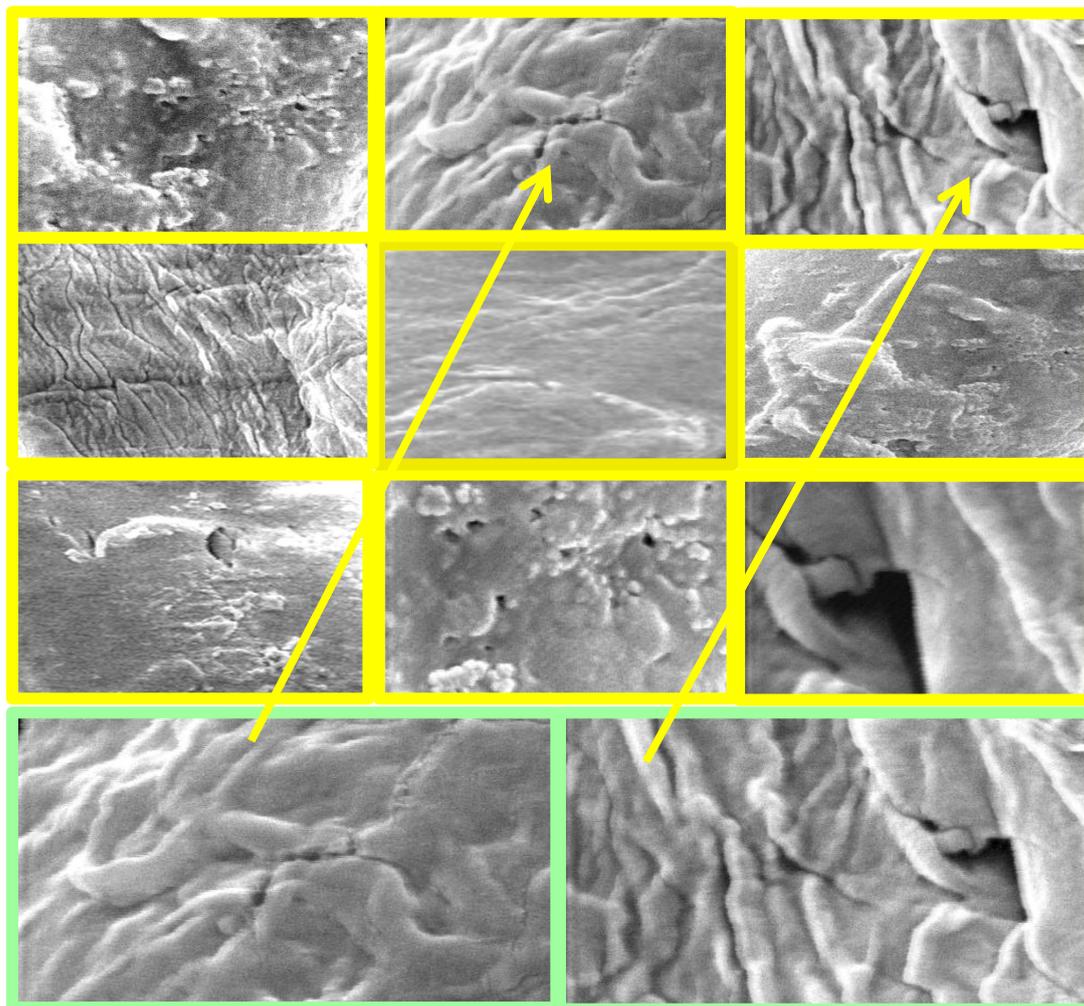


Fig. 3. Flow chart of the experiment

- 1) Reducing the waiting time of the intermediate process;
- 2) Scanning more areas for sufficient data;
- 3) Using an optical microscope with the instrument to observe the samples firstly and select appropriate areas.

The basic testing principles of the experiment is to scan some of the high resolution images (magnifications of 30000×) (Fig. 3).

RESULTS AND DISCUSSION

Morphology analysis

We studied the surface topography of poly (ethylene terephthalate) knitted materials by means of SEM observations. The virgin sample exhibited a markedly smooth surface (Figs. 4).

Pore structure

Different kinds of pores are developed on the surface of poly (ethylene terephthalate) knitted fabric after ultraviolet irradiation and simultaneous ozone gas exposure (Fig. 5-c). Some nano-dimension raptures and holes were also detected on the poly (ethylene terephthalate) substrate (Fig. 5-a and b). The pores are well developed in the fabric matrix, and different fractures can also be observed by cutting the matrix (Fig. 5-d and e).

It can be said that the formation of nano-dimension pores, fractures and raptures is presumably due to the physical etching effect of ultraviolet irradiation and ozone gas exposure [2, 3]. Physical etching process brings a highly directional flux of energetic, reactive ions to the surface of the

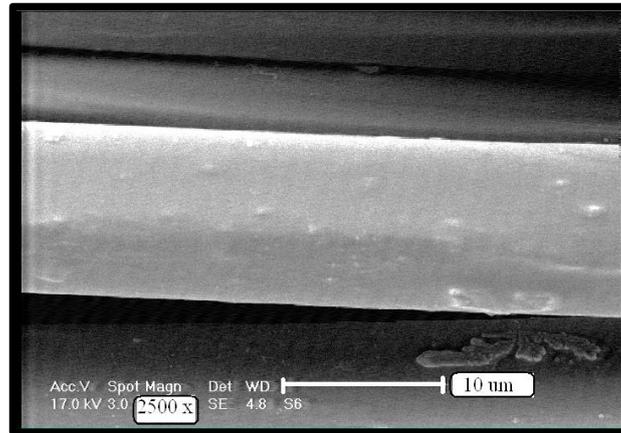


Fig. 4. SEM observation of poly (ethylene terephthalate) knitted material

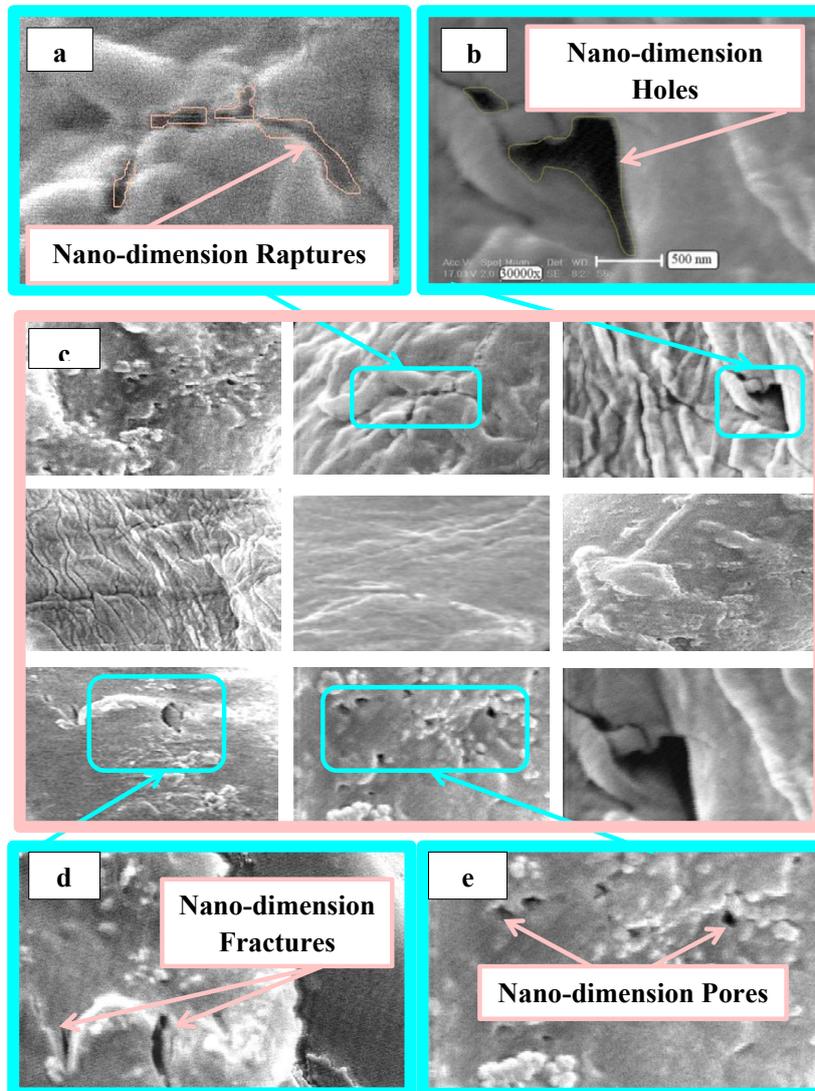


Fig. 5. Pores occurrence by SEM observations on the poly (ethylene terephthalate) surface: (a) nano-dimension raptures, (b) nano-dimension holes, (c) MAPS image, (d) nano-dimension fractures, (e) nano-dimension pores.

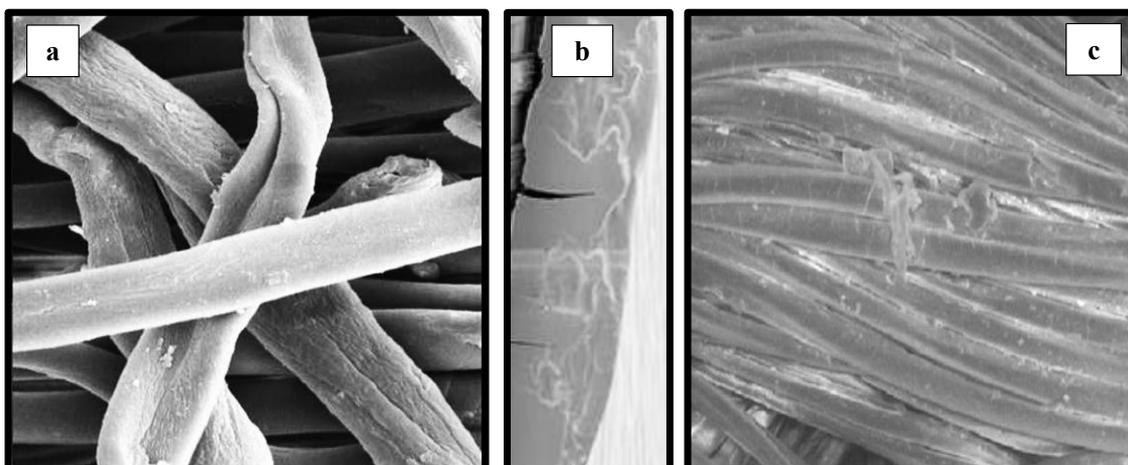


Fig. 6. SEM images of poly (ethylene terephthalate) knitted materials after various irradiation: a) Poly (ethylene terephthalate)/viscose fabric (with a ratio of 65% polyester and 35% viscose) after microwave irradiation[11], b) Poly(ethylene terephthalate) non-textured fabric after gamma irradiation[12], c) Poly(ethylene terephthalate) plain weave fabric after min UVC irradiation[13]

materials [4-6]. By doing so, a precisely controlled patterning of the substrate takes place as the unmasked sample is etched away by the reactive ions [7, 8]. In *physical etching*, ion bombardment through directional momentum transfer causes the physical sputtering of atoms [9, 10].

Fig. 6 shows the results of other research about the surface analysis of poly (ethylene terephthalate) knitted materials after different irradiations with SEM instrument.

Some other techniques such as Brunauer Emmett Teller method (BET) have extensive application in materials science to compute the solids surface areas through the physical absorption of gas molecules [14,15]. Finally, the author proposes this alternative strategy for the assessment of the surface poly (ethylene terephthalate) knitted materials after ultraviolet irradiation and ozone gas exposure. This strategy is expected to increase the success of surface morphology characterization.

CONCLUSIONS

Observations combining SEM tests were conducted to reveal the pore structure of poly (ethylene terephthalate) knitted material. The following conclusions can be drawn:

(1) Combining SEM observations in a nano-scale is effective to reveal pore structure in 2D dimensions. The SEM provides information about pore structure and surface morphology.

(2) SEM images provide information about poly (ethylene terephthalate) knitted material surfaces in a larger observation size with high resolution

degrees, which is of significance in revealing pores structures at different scales.

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CONFLICTS OF INTEREST

There are no conflicts to declare.

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