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Microstructural Orientation of Isotactic Polypropylene Studied by Computerized Scanning Eletron Microscopy Image Analysis

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We evaluate the orientation of microstructural elements of isotatic polypropylene (i-PP) before and after deformation using computerized "Quantikov" software analysis of Scanning Electron Microscopy (SEM) images. We observed that before deformation through uniaxial compression, the polymeric material doesn't exhibit any significant orientation. After deformation at 1349 MPa the material clearly showed preferential orientation that was attested by the orientation axis seen between two petals of the rose of the number of intercepts. This effect was more pronounced after deformation at 2699 MPa.

Keywords: *microscopy, polypropylene, orientation*

1. Introduction

The structural orientation of a polymer is an important factor in the production of polycrystalline and amorphous materials with specific physical properties. However, the parameters and processes that control microstructural evolution have not been yet systematically examined. Traditionally, the engineers have used the empirical knowledge about processing and properties. Exploration of the relation between properties and microstructure serves to establish fundamental knowledge about processing. The characterization of the distribution of microstructural orientation of polymeric materials is important in the prediction of the properties of oriented materials and the better understanding of the influence of different process parameters. Vasconcelos[1] proposed a methodology that help dealing with the complex tasks related to microstructural description. DeHoff[2] explored the tools that are required to understand how microstructures evolve. Garcia and Samios[3] showed that semicrystalline polymeric materials deformed by axial compression presented morphological changes reflecting mainly in the variation of the material crystallinity. Machado and Samios[4] performed a morphology evaluation of semicrystalline polymeric materials deformed through axial forces using SEM. Lima, Villeti and Samios[5] studied morphological and density changes, using deformation dimensional analysis, densimetry, X-ray diffraction and optical polarizing microscopy techniques.

The aim of the present work was to study the orientation of microstructural elements in isotatic polypropylene through computer analysis of SEM images before and after deformation using the software "Quantikov"[6-8] developed for microstructure characterisation[6-11].

The method is widely used in microstructure characterization since it allows to visualize the orientation including the information of the number of orientation axes existent in the system. In order to do that, the center of a co-ordinate system is chosen on the image and the number of intersections by unit length (N_L) for each angle (θ) is determined and values of θ x N_L are plotted in polar co-ordinates. The form of the obtained graph, the so called rose of the number of intercepts, allows the orientation of the selected microstructure to be determined. An isometric system would result in a circle centered in the origin of the polar co-ordinates; systems with a single orientation axis would present roses of intercepts with two petals with the axis passing between them. Orientation in two direction would result in four petals.

The degree of percent orientation (w) can also be determined[6,10]. In order to do that values of N_L for several angles

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are observed and the orientation degree is calculated by the equation (1)

$$\omega = \frac{100(N_L)_{PP} - (N_L)_{PR}}{(N_L)_{PP} + 0{,}571(N_L)_{PP}} \quad (1)$$

Where $(N_L)_{PP}$ is the number of intercepts per unit length observed in a direction perpendicular to the preferred microelements orientation and $(N_L)_{PR}$ represents the number of intercepts per unit length in a parallel direction.

The uniaxial compression ratio, UCR is defined by equation 2.

$$\varepsilon = \frac{l_f - l_i}{l_i} 100 \quad (2)$$

Where the l_f is the final sample compression uniaxial dimension and l_i is the initial one. Measurements for each applied pressure were repeated on at least three different samples and results are expressed as the average values of these measurements. The pressures of 1349 and 2699 MPa were associate with 65% and 90%, UCRs.

2. Materials and Methods

In the present study the plane strain compression in channel die (Fig. 1) was chosen as deformation mode. The advantage of applying this method is that the deformation is homogeneous and avoid spurious cavitation phenomena such as "micronecking" observed by Peterlin during uniaxial drawing[12]. Samples from core region of injection molding isotactic polypropylene (Mw = 438,200 g/mol; Mn = 90,000; Mw/Mn = 5.9) with 3.0 mm thickness 4.7 mm width and 4.7 mm length were plasticaly deformed at ambient temperature using compression pressures of 1349 and 2699 MPa.

The samples were placed in a deep channel die, and then, they were deformed. The upper and bottom surfaces of the chamber were polished with a very fine alumina powder (1.0 μm), avoiding replicas during deformation. By convention, the coordinate reference system was adopted

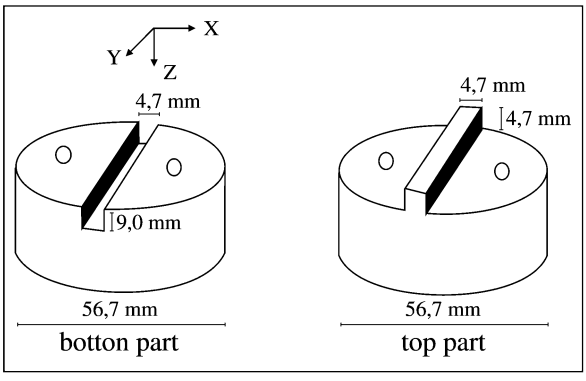

Figure 1. Schematic of the compression channel die showing the flow direction (Y), the loading direction (Z) and the unaffected direction (X).

in relations to the sample, where Z-axis was defined as the loading direction, Y as the flow direction and X as the unaffected axis, that is, the one which is limited by the chamber edges, as shown in Fig. 1.

For the microscopy analysis the following procedures were executed: the samples were placed in liquid nitrogen for 10 min and then rapidly fractured. Afterwards, the specimen were fixed on a stub and metalized with a thin gold layer, using a Sputter Coater (SCD 005/BALTEC) with approximately 20 nm of thickness, in order to eliminate any undesirable charge effects during SEM observations. After these procedures it was possible to do the microscopy analysis. The specimen was observed before and after the deformation process. The instrument used in this study was standard Scanning Electron Microscope Philips model XL30. The images used in this work were obtained by secondary electron mode, with a primary scanning beam of voltage of approximately 15 kV.

Selected portions of the images were processed using the software "Quantikov". Three images of each sample were studied. Firstly the microstructure characteristics related to orientation were enhanced. The digitalized SEM images (or digitalized through photo scanning), with a spectral resolution of 256 shades of gray, were segmented through the selection of the range of gray shades which best enhanced the microstructure orientation. In this procedure all shades of gray laying above the selected threshold are changed to 255 (white) and those below the selected threshold are changed to zero (black). Binary images, formed only by black and white are then obtained. These images were used to determine orientation through the rose of the number of intercepts and the percent of orientation.

3. Results and Discussion

Figure 2(a) is a SEM image from an i-PP sample before compression; Fig. 2(b) shows the processed binary image (black and white) enhancing the microstructure characteristics together with the drawing of the rose of intercepts. It can be seen in this case that the rose of intercepts has a circular shape without sharp and distinct petals leading to the conclusion that this i-PP sample has no significant preferential orientation.

The orientation obtained results, from the rose of the number of intercepts can present an error of approximately 10%. Figure 2(c) demonstrates that spherulitics structures of the sample without deformation have a degree of orientation of 24% and the error involved in this determination is approximately 15%.

Figure 3(a) is a SEM image from the i-PP sample after deformation at 1349 MPa and Fig. 3(b) is the binary image with the rose of intercepts from the corresponding structure orientation. In this case a distinct orientation with a preferential axis passing between the two petals can be observed. In Fig. 3(c) a fibrillar structure observed with a degree of

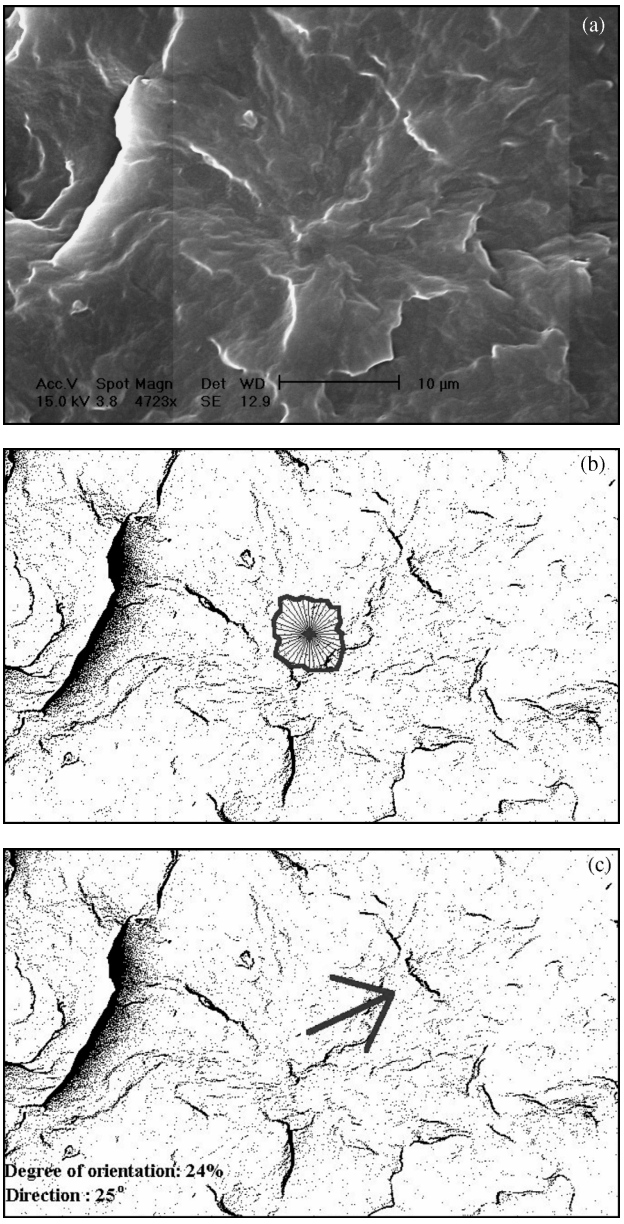

Figure 2. (a) SEM image of i-PP sample without deformation. (b) The same image processed to show the rose of the number of intercepts. (c) The same image processed to show the orientation axis and degree of orientation about 39%. The effect is still more remarkable after compression at 2699 MPa, where the formation of two petals are clearly and well defined. The evaluation of figs. 4(a) (SEM image), 4(b) (processed image) and 4(c) show a very significant degree of orientation of 57%.

4. Conclusions

The use of Scanning Electron Microscopy in combination with "Quantikov" software image analysis, allows the polymer morphological analysis in bulk. The structural morphological changes of i-PP caused by compression

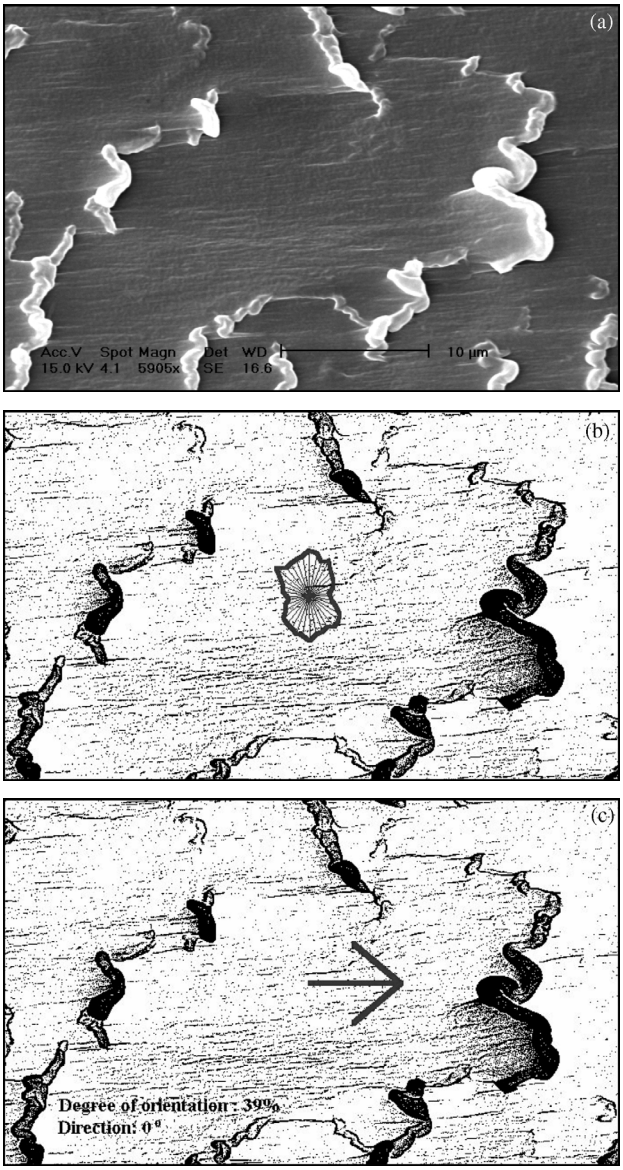

Figure 3. (a) SEM image of i-PP deformed at 1349MPa. (b) The same image processed to show the rose of the number of intercepts. (c) The same image processed to show the orientation axis and degree of orientation.

through axial deformation were evaluated by the analysis of the orientation of microstructural elements through computerized SEM image analysis, before and after deformation.

This methodology permitted initially a qualitative analysis of the SEM images and quantitative orientation results after the use of the "Quantikov" procedure.

As a result of the image processing in the i-PP it was easy to verify that increasing of the applied deformation pressure, causes a significant increase in the microstructure degree of the orientation.

The microstructure changes from isotropic structure of the undeformed samples, where UCR=0% (Fig 2a) to

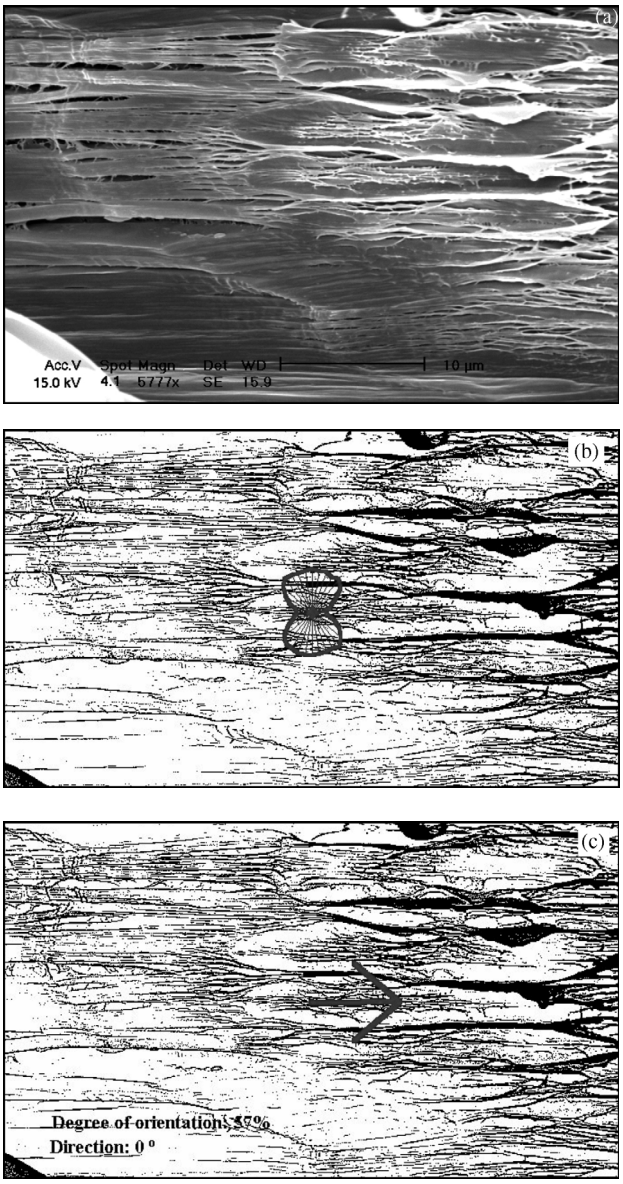

Figure 4. (a) SEM image of i-PP deformed at 2699MPa. (b) The same image processed to show the rose of the number of intercepts. (c) The same image processed to show the orientation axis and degree of orientation.

lamellar (Fig. 3a) for a moderate deformation (1349 MPa UCR = 65%) and to fibrillar (Fig. 4a) for the strong deformations (2699 MPa UCR = 90%) is followed by a degree of orientation from 24% to 39% and 57% respectively.

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References

1. Vasconcelos, V.; Vasconcelos, W.L. *Materials Research*, v. 2, n. 3, p.127, 1999.
2. DeHoff, R.T. *Materials. Research*, v. 2, n. 3, p. 111, 1999.
3. Garcia, I.T.S.; Samios, D. *Polymer*, v. 39, n. 12, p. 2563, 1998.
4. Machado, G.; Samios, D. *Anais do V Interamerican Electron Microscopy Congress,* Isla. Margarita,Venezuela, outubro/1999.
5. Lima, M.F.S.; Villeti, M.; Samios, D. *J. Polym. Eng.*, v. 17, p. 139, 1997.
6. Pinto, L.C.M. Tese de Doutorado: *"Quantikov - Um Analisador Microestrutral para o Ambiente Windows",* São Paulo, 1996.
7. Machado, G.; Luca, M. A.; D. Samios. *Anais do 23ª Reunião Anual da SBQ,* Poços de Caldas, MG, maio/2000.
8. Pinto, L.C.M. Short reference guide of the "Quantikov" software. Available on the Internet (1998).
9. Dehoff, R.T.; Rhines, F.N. *Quantitative Microscopy*, N.York, Mcgraw-Hill Book Company, (1968).
10. The Welding Engineer's Weld Pool, *Quantitative Stereology.* Available on the Internet http://home.istar.ca/~bsant/QS/QS.html.
11. Nemati, K.M; Monteiro, P.J..M; Scrivener, K.L. Analysis of compressive Stress – Induced Cracks in Concrete, *American Concrete Institute Materials Journal*. Available on the Internet .
12. A. Peterlin, *Journal of Materials Science*, v. 6, p. 490, 1971.