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An Etching Technique for Morphological Investigation of TPU/ABS Blends by SEM

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ABSTRACT

Blends of thermoplastic polyurethane elastomer (TPU) and ABS resin (acrylonitrile–butadiene–styrene) were observed by SEM (scanning electron microscopy) before and after their treatment with methyl ethyl ketone (MEK). Samples were treated with MEK for different periods of time and dried at two temperatures in order to optimize the etching conditions. It was found that 3 h of etching is adequate to reveal the phase morphology of these blends. In contrast, a 4 h period has provoked serious artifacts in the samples and no information about phase structure could be obtained. At appropriate conditions, this technique can be used to quantitatively estimate the component dispersion in these blends.

1 INTRODUCTION

Technological development of polymer blends has shown that the mechanical properties of these materials are dependent not only on their chemical nature but also on their morphology.[1]

The morphology of polymer blends can be determined by a number of optical and electron microscopy techniques.[2] The specimen preparation is an important task in polymer microscopy because of the inherent lack of contrast. This may be overcome by methods like staining, etching, replication, shadowing and metal coating.[3]

This paper presents an optimization of the etching technique as an

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aid in the investigation of phase structures of blends composed of thermoplastic polyurethane elastomer (TPU) and ABS resin (acrylonitrile–butadiene–styrene) by SEM (scanning electron microscopy).

2 EXPERIMENTAL

2.1 Sample preparation

A commercial TPU elastomer (COFADE Fabricadora de Elastômero Ltda) based on a polyester, diphenyl methane diisocianate (MDI) and butane diol was used as the matrix in mixtures with ABS resin (commercial product from NITRIFLEX S.A. Indústria e Comércio). Blends containing 10, 30 and 50 phr of ABS were prepared on a roll mill at 80 °C and 15 rpm, for 30 min. The specimens to be observed in the microscope were obtained after being submitted to the Charpy impact test. For this test the samples were cut from sheets (15 cm × 15 cm × 0·6 cm), compression molded at 195 °C for 15 min. The impact test was carried out on frozen samples obtained after immersion for 15 min in a ethanol/dry ice bath.[4] The fracture surfaces were observed by scanning electron microscopy (SEM).

2.2 Etching

Each mixture (10, 30 and 50 phr of ABS in a TPU matrix) was etched by using methyl ethyl ketone (MEK) at room temperature for periods of 2, 3 and 4 h. Since MEK is a good solvent for styrene acrylonitrile copolymer (SAN), a component of the ABS resin,[5] this procedure leads to the ideal period of time for the etching process. After this treatment, all samples were dried under vacuum for about 6 h at two different temperatures: 40 °C and 60 °C.

2.3 Microscopy

Before the analysis, the fractured surfaces were coated with a thin film of gold to produce secondary electrons and to prevent charging. A

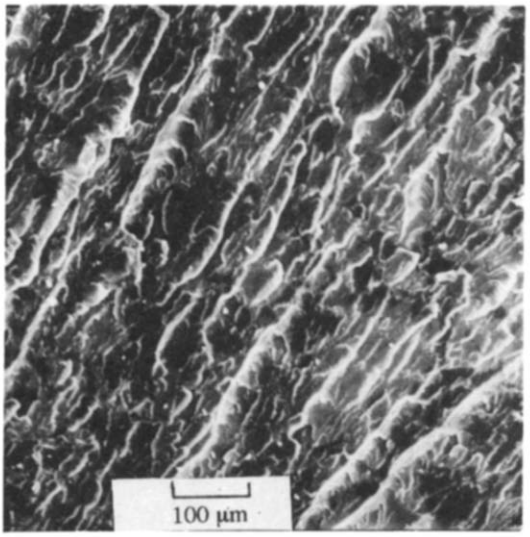

Fig. 1. Fractured surface of TPU/ABS 50/50 blend.

JEOL-JSMU3 model SEM, working at an accelerating voltage of 20 kV, was used.

3 RESULTS AND DISCUSSION

An SEM photomicrograph of a non-etched fractured surface of a TPU/ABS alloy, is presented in Fig. 1. As can be seen, no distinct domains of the dispersed phase were visible even at a high ABS content. Only the stress-whitening characteristic of ABS fracture could be observed.[6] This is still more evident when compared with Figs 2 and 3, which show SEM photomicrographs of pure TPU and ABS, respectively.

A careful selection of etching time is very important so as to avoid, or at least minimize, artifact formation. It was found from Fig. 4 that a 4 h period[5] is too long and inadequate, with harmful effects on SEM observation. On the other hand, 2 h of etching is insufficient to reveal the phase contrast, as can be seen in Fig. 5, and maybe compared with Fig. 1 for non-etched samples. Figure 6(a) shows that phase dispersed ABS was revealed as relatively uniform particles after 3 h of etching. The average particle size becomes larger for higher ABS content, disclosing an interconnected morphology, as presented in Fig. 6(b). It was verified that drying temperatures higher than 40 °C have harmful

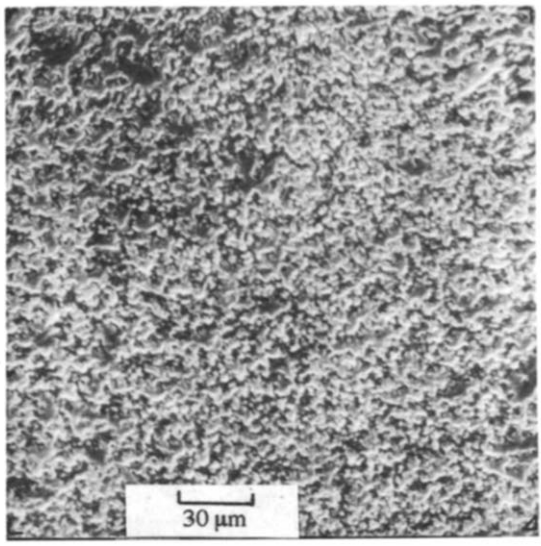

Fig. 2. Fractured surface of TPU.

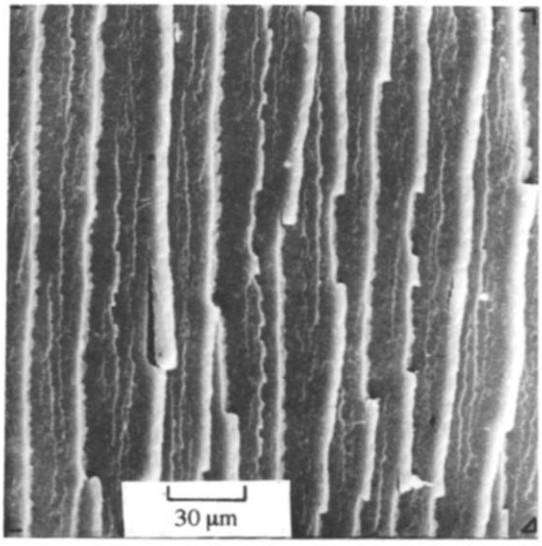

Fig. 3. Fractured surface of ABS.

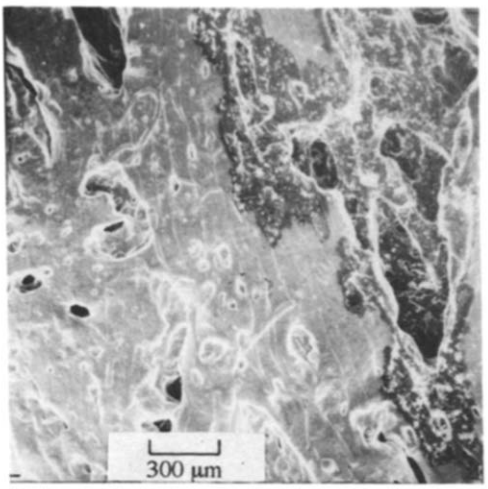

Fig. 4. Fractured surface of TPU/ABS 90/10 blend after 4 h of etching with MEK and dried at 40 °C under vacuum.

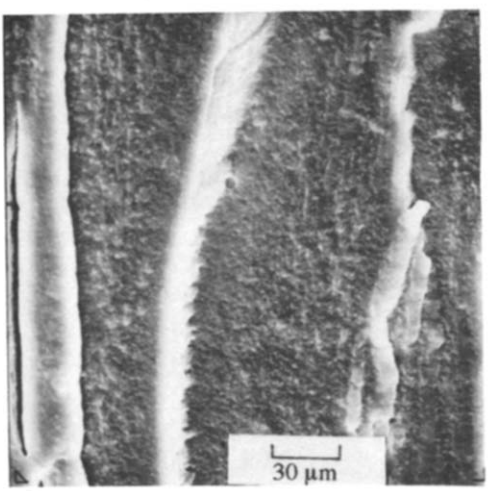

Fig. 5. Fractured surface of TPU/ABS 50/50 blend after 2 h of etching with MEK and dried at 40 °C under vacuum.

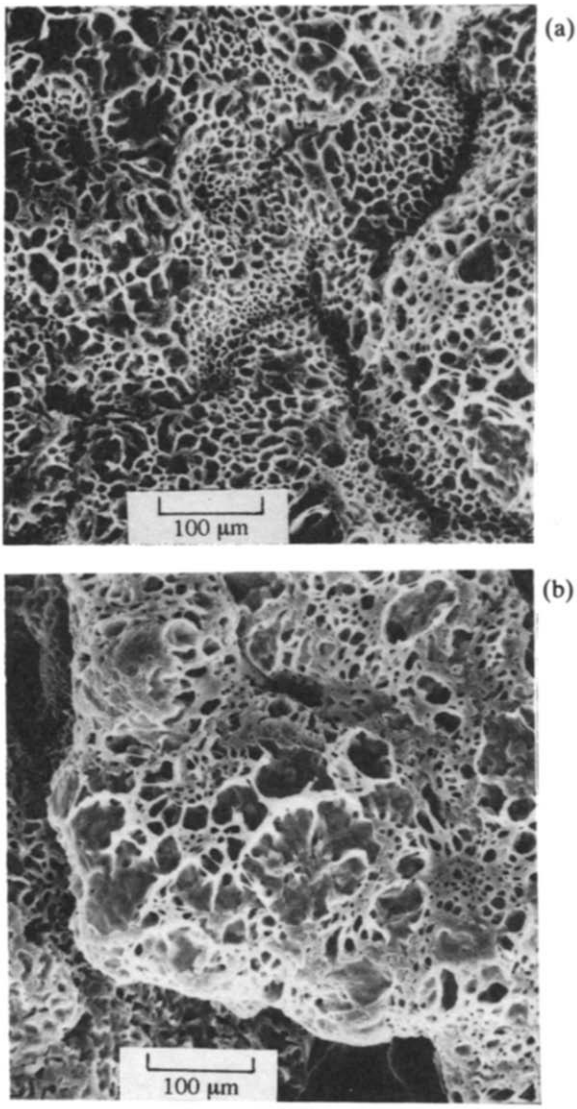

Fig. 6. Fractured surface of TPU/ABS blends after 3 h of etching with MEK and dried at 40 °C under vacuum: (a) TPU/ABS 70/30; (b) TPU/ABS 50/50.

effects on the blends' phase-structure. Figure 7 shows a sample etched for 3 h with MEK and dried at 60 °C under vacuum for about 6 h.

4 CONCLUSIONS

- In TPU/ABS blends, the domains of the ABS dispersed phase at the fractured surface are not visible by the SEM technique unless

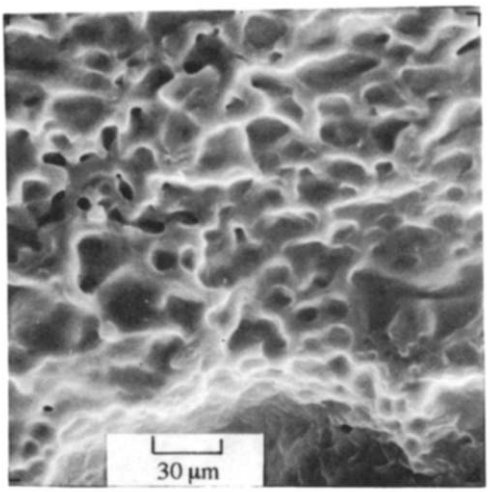

Fig. 7. Fractured surface of TPU/ABS 50/50 blend after 3 h of etching with MEK and dried at 60 °C under vacuum.

etched samples have been used. This is an indication that well dispersed TPU/ABS blends were obtained by the milling procedure.

- The optimization of the etching conditions is very important in order not to damage the TPU matrix. Etching by MEK of the fractured surface of TPU/ABS blends for 3 h has revealed the dispersed phase morphology. The average size of ABS domains was found to be proportional to the ABS content in the blends.
- In order to minimize the danger of artifact evolution, the samples need to be dried at temperatures sufficiently lower than the temperature range in which the polymer undergoes morphological changes.

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