Research Article

A Novel Approach to the Image Analysis of the Phase Morphology in Polymer Blends with Droplet/Matrix Morphology

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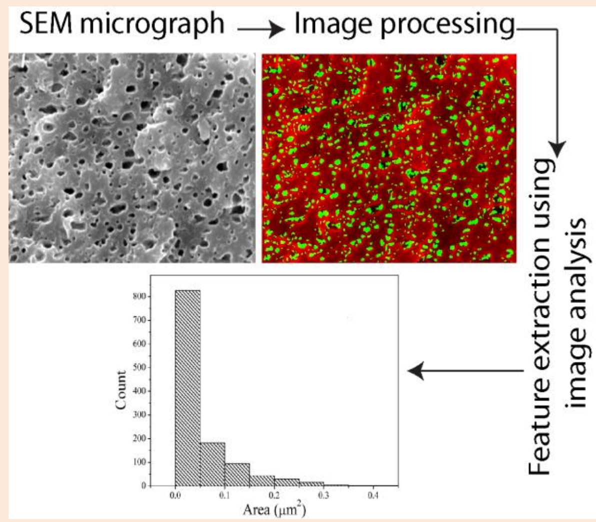
Abstract

Digital image processing is being increasingly implemented in microstructural analysis and is considered to be an ideal tool to quantify the morphology of particles. A novel image processing and analysis method was developed to track and measure the droplets in a polymer blend from its 2D scanning electron micrograph in which the difference in intensity/contrast between the droplets and the matrix is small. The new procedure was able to find the area of each and every droplet domains in the droplet/matrix morphology irrespective to the shape and size of the droplets. This Image processing and subsequent image analysis using image processing toolbox of the MATLAB software has been done to analyze the change in morphology with change in processing conditions in a Thermoplastic Polyurethane- Polydimethylsiloxane rubber blend. An adaptive thresholding algorithm was used to trace the droplets followed by filtering and morphological operations on the image. All polydimethylsiloxane rubber domains were detected using image processing and the areas of each domain were calculated and the comparisons between the blends prepared at different processing conditions were recorded. This procedure can be used in future investigations such as blend dynamics, droplet formation, change in morphology due to compatibilization, blend rheology etc.

Keywords: Phase morphology, polymer blends, image processing, image analysis, scanning electron microscopy, edge detection.



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Introduction

Properties of polymer blends are generally controlled by the properties of the individual components, processing conditions, morphology developed during processing and the interaction between the blend constituents. During processing of the polymer blends there is a large scale change in the texture from pellet, powder or solid block shape to the micrometer sized droplets or particles. This is widely known as ‘morphology development’ in polymer blends. Phase morphology in multi-component polymer based systems constitutes the core physical characteristic to carefully control when designing and studying polymeric materials [1,2]. In immiscible polymer blend systems, the domain range in size from sub-microns to hundreds of microns and spherical, ellipsoidal, cylindrical, ribbon like, co-continuous and sub-inclination types of morphologies can be obtained under various conditions [3]. Scanning electron microscopy is one of the powerful characterization methods which is used to characterize the phase morphology in polymer blends.

Understanding the development of phase morphology, change in morphology during processing and compatibilization are very important areas in polymer technology to achieve the desired end use properties. Imaging techniques combined with image analysis offer the possibility to analyze the size and shape of each individual particle present in a polymer matrix. Image analysis is the process used to extract information from images such as finding shapes, locating edges and counting objects in the image. By combining several image processing techniques such as edge and shape detection, image thresholding and color based segmentation; one can obtain detailed statistics and interpret the image. In a common image processing technique, objects are separated from the background by identifying the abrupt change in the intensity. After their boundaries are detected, the objects are counted and the size distribution can then be analysed [4-6]. Few literatures are available on the image analysis of metal alloys, and polymers [7-9]. Conventional image analysis of the morphology of polymer blends and composites have relied on direct detection of objects in the image by using intensity based edge detection. But the conventional intensity based edge detection is ineffective in the case of the morphology of most of the polymer blends due to the relatively lower difference in contrast/intensity between the different phases in scanning electron micrographs.

Figure 1 depicts the common problems in the image analysis of the phase morphology of the polymer blends. The typical flow mark occurs during the time of sample preparation like cryo-fracturing may interfere the conventional intensity based edge detection technique. Scanning electron micrograph gives normally a 2D photograph but it always contains 3D content. This means a particle in the polymer matrix may show two edges in the SEM photomicrographs i.e. one edge from the upper side of the particle and another from the lower side. Thus conventional edge detection image processing will consider these as two different particles. This might destroy the accuracy of the data. In this study attempts have been made to develop a novel procedure to detect and measure the droplets of all sizes and shapes in a polymer blend with droplet/matrix morphology using image processing toolbox of MATLAB from MathWorks USA. Also attempts have been made to measure the size and area of each droplets which won't detect in normal edge detection because of the relatively small difference in the intensity between the phases of polymer blends.

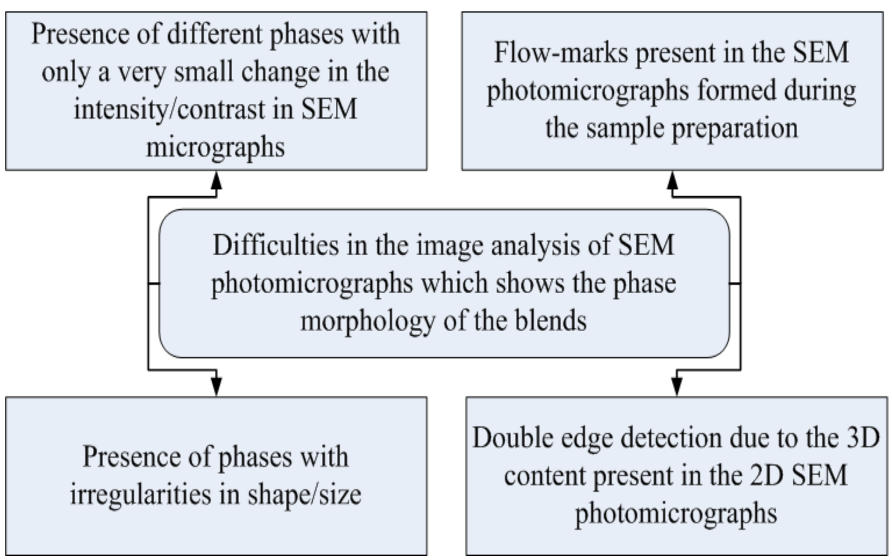

Figure 1 Difficulties in the image analysis of the scanning electron micrographs which shows the phase morphology of the polymer blends.

Experimental

Materials

TEXIN® RxT85A, an aromatic polyether based thermoplastic polyurethane (TPU) was generously supplied by Bayer Material Science, India for the present work. It has a specific gravity of 1.12 and the melt flow index is 4g/10 min at

190°C/8.7kg. PDMS grade, Silastic WC-50TM with a specific gravity of 1.15 was supplied by Dow Corning Inc. (Midland, MI, USA)

Preparation of blends

Blends of TPU and PDMS rubber at a blend ratio of 70:30 were prepared in a Brabender® Plastograph® EC (digital 3.8-kW motor, a torque measuring range of 200 Nm). Two different processing conditions were selected to study the change in morphology by using image analysis. The processing conditions are listed in **Table 1**.

Table 1 Processing conditions for the preparation of T70P30 blend

Specimen ID	Temperature (°C)	Rotor speed (rpm)	Time of mixing(min)
T70P30-1	190	80	2
T70P30-2	210	60	6

The procedure for mixing the blends in the Brabender Plastograph is as follows. First TPU was added into the mixing chamber and allowed it to melt for 2 minutes. Then PDMS was added and mixed for desired time. The time taken for the processing after the addition of PDMS has been taken as the time of mixing. At the end of the blending the melt was taken out and passed through a cold laboratory two roll mill. Then tensile sheets of approximately 2 mm thick were compression molded using thermoplastic compression molding press (Moore Press, GE Moore and Son, Birmingham, UK) at 190°C for 3 min at a pressure of 7 MPa. The designation of the blends is given as TxPy, in which 'T' and 'P' denotes TPU and PDMS and 'x' and 'y' as the weight percentage of TPU and PDMS respectively.

Scanning electron microscopy

A JEOL-JSM-5800 scanning electron microscope was used to study the phase morphology of the binary blend of TPU and PDMS rubber. The blends were subjected to cryo-fracture in liquid nitrogen and the PDMS phase was extracted preferentially from the specimens using toluene as the solvent. Before examination, the fracture surfaces were dried in a heating oven at 70 °C, brought down to room temperature in a desiccator and then sputter-coated with a thin layer of gold in a vacuum chamber. The specimens were subjected scanning electron microscopy at 0° tilt angle.

Image processing and analysis of SEM photomicrographs

Image processing and analysis of the scanning electron photo-micrographs of TPU-PDMS blends was performed using MATLAB (matrix laboratory) which is a multi-paradigm numerical computing environment developed by MathWorks, USA. Image Processing Toolbox of MATLAB was used to study the type of morphology, the shape of the PDMS domains and area of each PDMS domains and its distribution in the TPU matrix.

Results and discussions

Scanning electron microscopy studies

Figure 2 shows the SEM photomicrographs of TPU-PDMS rubber blends at a blend ratio of 70:30 processed at two different processing conditions.

The cavities present in the SEM images are the etched portions of dispersed PDMS phase and the land area is TPU, the major matrix. It clearly depicts that variation in the processing conditions alters the phase morphology of the blends. Figure 2(a) of T70P30-1 shows a very well dispersed droplet/matrix morphology where PDMS is the dispersed phase and TPU is the continuous matrix. Most of the PDMS domains are spherical or ellipsoidal in nature. Few bigger PDMS domains also present in the scanning electron micrographs. Scanning electron micrographs of T70P30-2 [Figure 2(b)] also shows droplet/matrix morphology. But the type of droplet is quite different from that in Figure 2(a). Figure 2(b) shows highly elongated droplets of PDMS throughout in the TPU major matrix. Few spherical droplets of PDMS also present in Figure 2(b). This clearly indicates that in polymer blends, the variation in the processing conditions alters the morphology significantly.

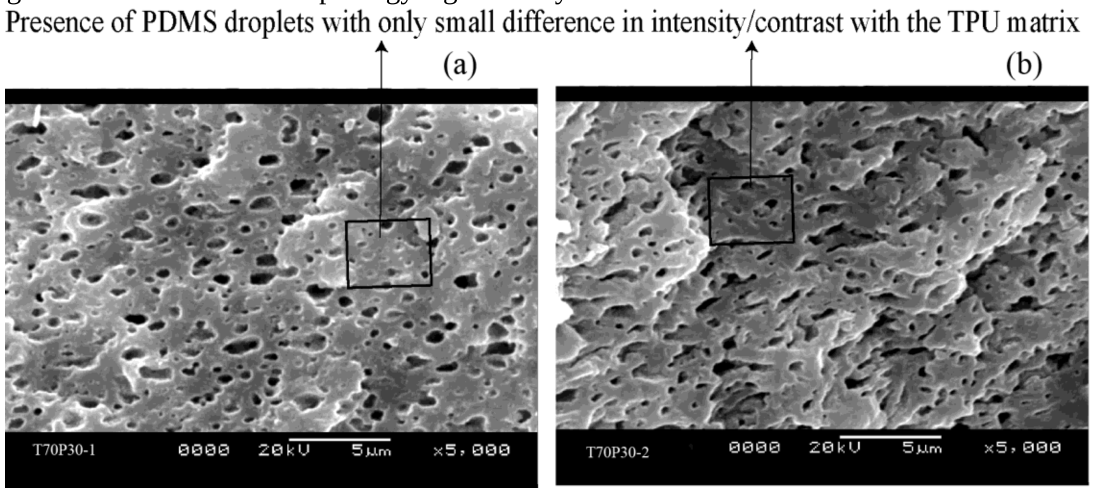

Figure 2 SEM micrographs of the blends (a) T70P30-1 (b) T70P30-2.

Image processing and analysis of scanning electron micrographs

Image processing needs several steps which must be performed one after another to extract the data of interest from the image. In the scanning electron micrographs the etched domains of PDMS is seen as dark cavities and TPU is seen as the land area. But there is not much change in contrast between some of the PDMS domains and the TPU matrix [Figure 2(a) and 2(b)]. This makes very difficult to do conventional intensity based edge detection technique to trace PDMS domains. Attempts have been made to solve this problem to trace each PDMS domains and measure their characteristics like area of each PDMS domains and their distribution. **Figure 3** shows the schematic presentation of the processes done for the image capturing, image processing and image analysis of the phase morphology of polymer blends.

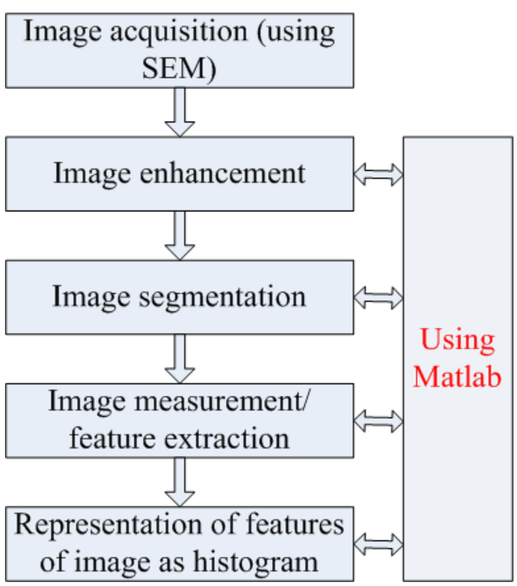

Figure 3 The different stages in the image capturing, processing and analysis.

The procedure of image processing and analysis is represented as a flowchart and it is shown in **Figure 4** and the Matlab code is given in Appendix 1.

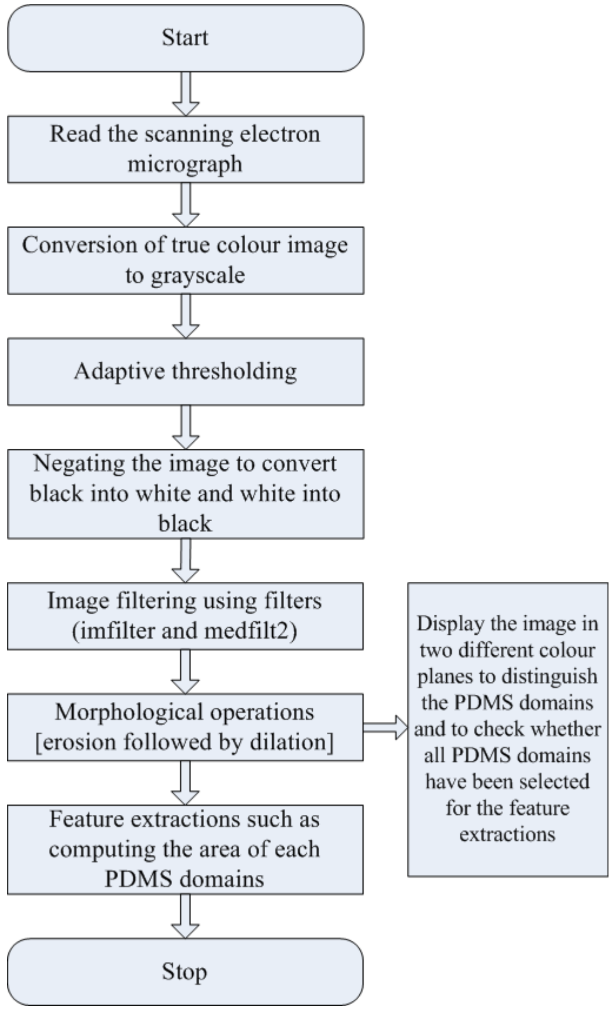

Figure 4 Proposed Flowchart of the image processing procedure.

In order to get a better extraction of data from an image, the image quality should be in an understandable level. For this, image enhancement procedures should be followed and one of such procedure adopted here is the conversion of scanning electron micrograph which is a true color image into a grayscale image. This has been done by eliminating the hue and saturation information while retaining the luminance of the image. The image data in a grayscale image consist of a single channel that represents the intensity, brightness, or density of the image. The gray scale images of the polymer blends are shown in **Figure 5**.

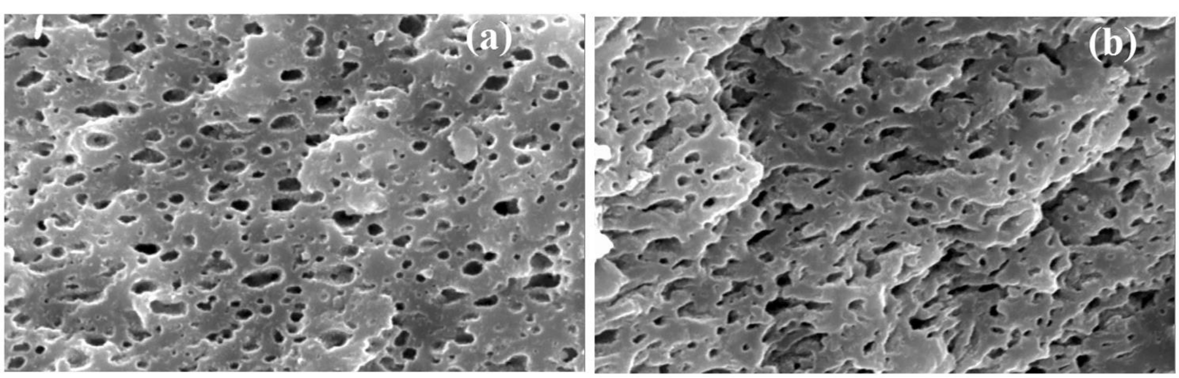

Figure 5 Grayscale images of TPU-PDMS blends (a) T70P30-1 (b) T70P30-2.

In digital image processing, image segmentation is the process of partitioning a digital image into multiple segments of pixel to represent that image in more meaningful to analyze and extract the data from the image. This is typically used to identify objects in the image. So in the present context to identify all PDMS droplets in the TPU matrix, it is necessary to do the image segmentation of the grayscale image. Thresholding is one of the simplest methods of image segmentation. Thresholding is used to create binary image from grayscale image. It is used to segment an image by setting all pixels whose intensity values are above a threshold to a foreground value and all the remaining pixels to a background value. But in this case of phase morphology of TPU-PDMS blends, it is difficult to choose an adequate threshold or number of thresholds to separate each PDMS domains from the background because of the variation in the intensity/contrast between the most of the PDMS domains and the TPU matrix is very small. In order to accommodate change in lighting condition in the image, adaptive thresholding algorithm was used to separate the foreground from the background with non-uniform illumination. Adaptive thresholding changes the threshold dynamically over the image. For each pixel in the image, a threshold has to be calculated. If the pixel value is below the threshold it is set to the background value, otherwise it assumes the foreground value. This solves the issues related to the strong illumination gradient or shadows. This method also solve the problem of low intensity difference between the PDMS phase and TPU in the scanning electron micrographs [Fig. 2(a) and Fig. 2(b)]. After thresholding step, intensity of the resultant image was negated to achieve a complement of the image to get better view of the thresholded image. By this step black and white in the image were reversed. So the black area represents TPU matrix and white portions represent PDMS domains.

In image processing, filters are mainly used to suppress either the high frequencies in the image (smoothing the image) or the low frequencies (enhancing) or detecting the edges in the digital image. Filtering is an operation, in which the value of any given pixel in the output image is determined by applying some algorithm to the values of the pixels in the neighborhood of the corresponding input pixel. The filtering procedures are applied in this case is to distinguish the objects of interest from other objects like noise and the background. Filtering is also used to sharpen the edges and correcting unequal illuminations. Linear spatial filtering using the functions imfilter and medtfilter2 has been used for the filtering procedures. The imfilter function computes the value of each output pixel using double-precision, floating-point arithmetic. If the result exceeds therange of the data type, the imfilter function truncates the result to that data type's allowed range [10]. Median filtering is a nonlinear operation often used in image processing to reduce "salt and pepper" noise. Median filtering is more effective to reduce noise and preserve edges simultaneously. The resulting image after thresholding, negating and filtering is shown in **Figure 6**.

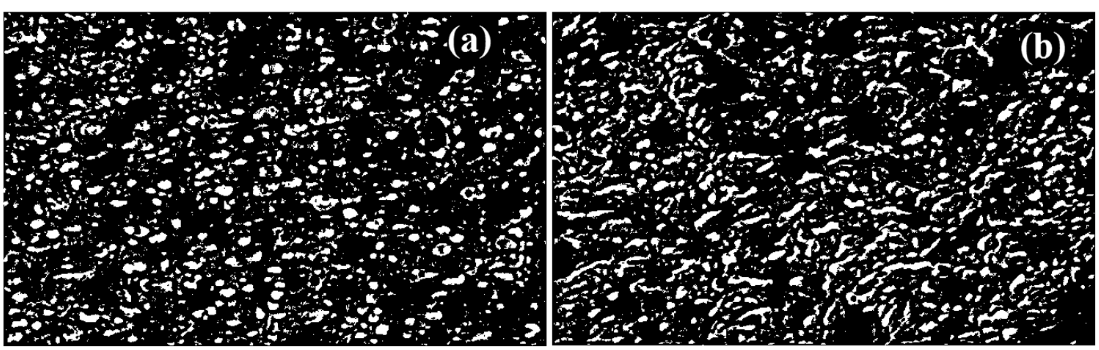

Figure 6 Binary Image after thresholding and negating the image (a) T70P30-1 (b) T70P30-2.

Mathematical morphology is a technique of image processing based on geometry. The theory of morphological image processing lies in set theory and mathematical theory of order. By choosing the size and shape of the neighborhood, suitable morphological operation can be performed which is sensitive to specific shapes in the input image [11]. The basic idea in binary morphology is to probe an image with a simple pre-defined shape, drawing conclusions on how this shape fits or misses the shapes in the image. This simple probe is called structuring element, and is itself a binary image [12]. The fundamental morphological operations are dilation and erosion. The morphological dilation operation (Θ) of an image or a point set 'X' with another small point set B (structuring element) is defined as a set of points 'P' belongs to the two dimensional space Z^2 such that P = x+b for every point x belongs to X and the point b belongs to B and this relation is represented as

$$X \ominus B = \{P \in Z^2 \mid P=x+b,\ x \in X,\ b \in B)\} \quad (1)$$

The erosion operation (Θ) of an image or a point set 'X' with another small point set B(structuring element) is defined as a set of points P belongs to the two dimensional space Z^2 such that P+b belongs to X for every b belongs to B and this relation is represented as

$$X \ominus B = \{P \in Z^2 \mid P+b \in X,\ b \in B)\} \quad (2)$$

Dilation adds pixels to the boundaries of objects in an image, while erosion removes pixels on object boundaries. The number of pixels added or removed from the objects in an image depends on the size and shape of the structuring element used to process the image [5,13,14]. This morphological operation has been done by doing an erosion technique followed with dilation on the filtered image [Figure 6]. The resultant image is shown in **Figure 7** and this image has been used to do the image measurements including the area of PDMS domains and its distribution.

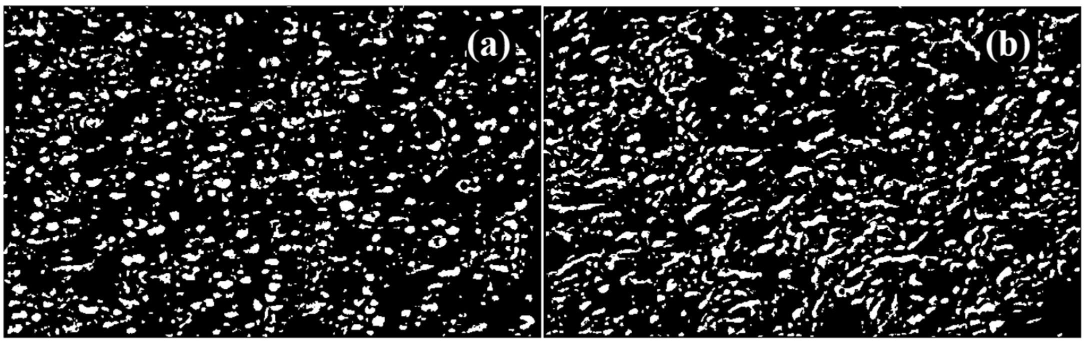

Figure 7 Images after the morphological operations (a) T70P30-1 (b) T70P30-2.

In order to visually distinguish each PDMS domains from the matrix and to check whether all PDMS domains has been detected or not for the image analysis, separate color planes have been given for PDMS domains and TPU matrix and it is shown in **Figure 8**. The PDMS domains are represented by green color and the remaining red land area is TPU major matrix. This has been done by assigning different color planes to the two different intensity areas. Figure 8 confirms that almost every PDMS domains have been detected and selected for image analysis.

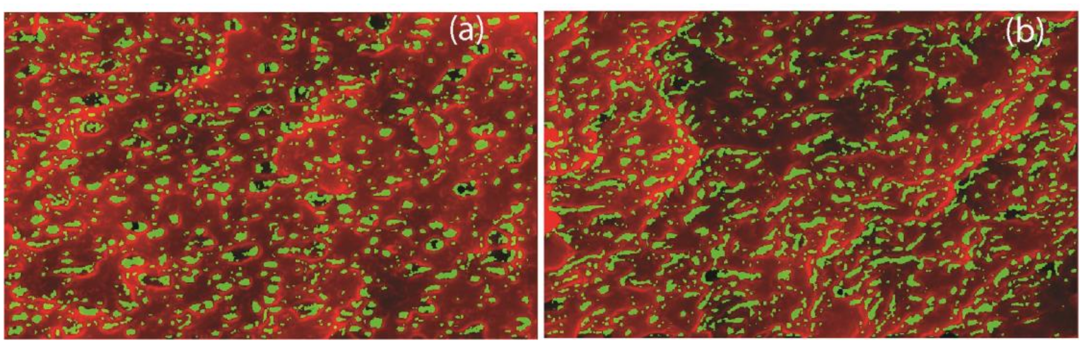

Figure 8 Representation of the droplet/matrix morphology of TPU-PDMS blends in two different colors (a) T70P30-1 (b) T70P30-2.

Final stage is the image measurement process to extract the objects and measurements. Connected-component labeling has been used to get statistics about the image. For example, one application of the connected components labeling is to count the objects in an image. Connected component labeling works by scanning an image by pixel-by-pixel (from top to bottom and left to right) in order to identify connected pixel regions, i.e. regions of adjacent pixels which share the same set of intensity values. This function returns a matrix, the same size as the input image, in which the objects in the input image are distinguished by different integer values in the output matrix [4,6]. The output matrix is of class double and not a binary image. One way to view the output matrix is to display it as a pseudocolor indexed image. The pseudocolour indexed image is shown in **Figure 9**.

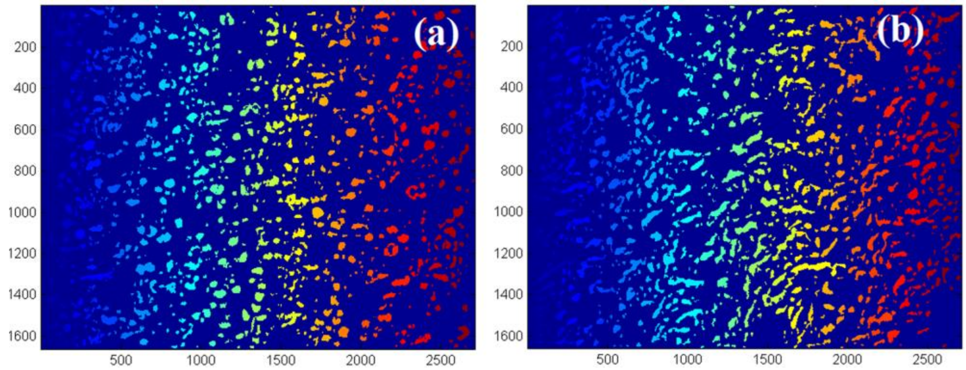

Figure 9 Pseudocolour indexed image of (a) T70P30-1 (b) T70P30-2.

The function bwarea has been used to find the area of each PDMS domains. The bwarea function returns the area of a binary image. The area is a measure of the size of the foreground of the image. Roughly speaking, the area is the number of 'on' pixels in the image. bwarea does not simply count the number of pixels set to on. bwarea actually weights different pixel patterns unequally when computing the area. This weighting compensates for the distortion that is inherent in representing a continuous image with discrete pixels. The result of the calculation of area of each PDMS domains is represented as a histogram in **Figure 10**. Histogram shows that in both blends majority of the particles have area less than 0.3 µm2. The blend T70P30-1 has very few bigger particles whose area is higher than those of 0.3 μm^2. But T70P30-2 shows significant number of bigger particles in the range of 0.4- 0.6 μm^2 due to the presence of highly elongated PDMS domains due to the merging of two or more smaller PDMS domains. This novel way of image processing and analysis is found to be the best way to find the area of each domain in the droplet/matrix morphology.

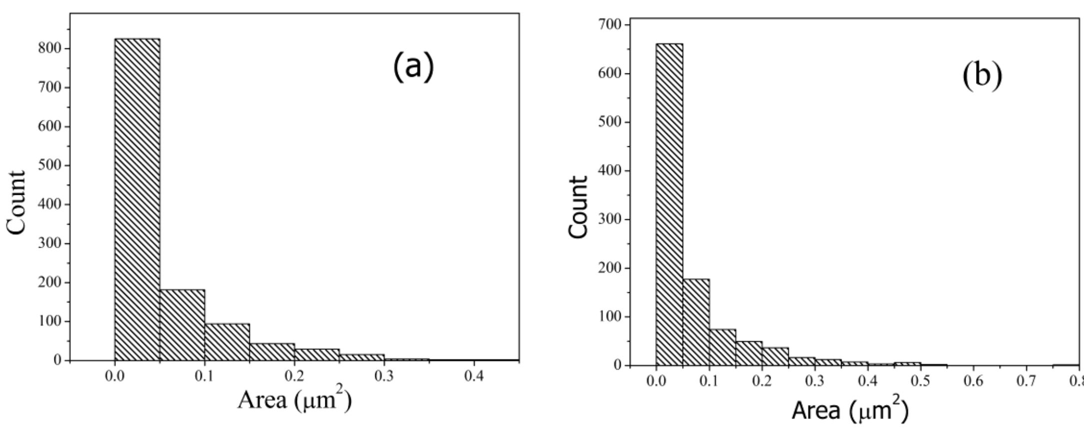

Figure 10 Histogram of area of PDMS domains in the TPU matrix (a) T70P30-1 (b) T70P30-2.

Conclusions

It is demonstrated that scanning electron microscopic images of polymer blends with droplet/matrix morphology can be analyzed using the image processing procedure by the image processing toolbox of the software MATLAB. Droplets with spherical, ellipsoidal or highly elongated shapes can also be processed and interpreted using this procedure. The binary image processing with adaptive thresholding, morphological operations and filtering methods are used to find the edge detection and noise removal in the image processing and to trace the PDMS droplets. Two images of blends with unequal size distributions and shapes caused by the difference in processing conditions in the melt mixing are quantitatively differentiated. This method can also be used to compare the size of the domains and its distribution in blends and the effect of compatibilization on the phase morphology of the polymer blends etc.

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Appendix 1

1. MATLAB code for adaptive thresholding

```
function bw=adaptivethreshold(IM,ws,C,tm)
%ADAPTIVETHRESHOLD An adaptive thresholding algorithm that separates the
%foreground from the background with nonuniform illumination.
% bw=adaptivethreshold(IM,ws,C) outputs a binary image bw with the local
% threshold mean-C or median-C to the image IM. ws is the local window size tm is 0 or 1, a switch between mean and median. tm=0 mean(default); tm=1 median.
% Contributed by GuangleiXiong (xgl99@mails.tsinghua.edu.cn) at Tsinghua University, Beijing, China.
%
if (nargin<3)
error('You must provide the image IM, the window size ws, and C.');
elseif (nargin==3)
tm=0;
elseif (tm~=0 && tm~=1)
error('tm must be 0 or 1.');
end
IM=mat2gray(IM);
if tm==0
mIM=imfilter(IM,fspecial('average',ws),'replicate');
else
```

```
mIM=medfilt2(IM,[wsws]);
end
sIM=mIM-IM-C;
bw=im2bw(sIM,0);
bw=imcomplement(bw);
```

2. MATLAB code for the image processing and analysis

```
img=imread('Sample.JPG');
gray=rgb2gray(img);
minsize=50; %% mimimum and maximum sizes to be detected
maxsize=10000;
bw=adaptivethreshold(gray,60,.051,1);
bw1=1-bw;
 CC = bwconncomp(bw1);
numPixels = cellfun(@numel,CC.PixelIdxList);
[biggest,idx] = max(numPixels);
BW=bw1;
for k=1:size(numPixels,2)
ifnumPixels(k)<minsize || numPixels(k)>maxsize
BW(CC.PixelIdxList{k}) = 0;
end
end
 BW = imerode(BW,ones(5));
BW = imdilate(BW,ones(5));
figure(1),imshow(img);
figure(2),imshow(bw1);
figure(3),imshow(BW);
rgb = img;
rgb(:,:,1)=gray;
rgb(:,:,2) = uint8(BW*255);
rgb(:,:,3)=zeros(size(gray));
figure(5),imshow(rgb);
rgb(:,:,1)=gray;
rgb(:,:,2) = uint8(BW*255);
rgb(:,:,3)=zeros(size(gray));
figure(4),rgb(:,:,2);
[L, num] = bwlabel(BW);
imagesc(L);
 STATS = regionprops(L, 'Area');
```


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