1472
doi:10.1017/S1431927611008233

Microsc. Microanal. 17 (Suppl 2), 2011

Quantifying Dispersion in Polymer Systems by Combining Image Analysis and Statistical Analysis

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It is documented in the literature that nanoparticle dispersion has a critical impact on the properties of nanocomposite materials [1, 2]. Over the years, various methods have been developed to quantify dispersion based on SEM or TEM images. For instance, one can look at dispersion by quantifying the mean inter-particle distance [3-5] (average distance between every combination of particles) but this method is rather insensitive to the dispersion quality. Another approach consists of the so-called Parent / Child model in which a grid is overlaid onto the image and the number of particles in each quadrant is quantified. This method strongly depends on the density of the grid with respect to the image to be analyzed and is not always sensitive to agglomeration. Finally a more recent approach focuses on unreinforced polymer domains and the free-space length between the particles, rather than quantifying the distribution of particles [6]. The premise is that these unreinforced domains will constitute the weak link of the nanocomposite by behaving as the neat polymer.

In the work presented here we focus on developing a method to quantify and analyze the dispersion of an impact modifier in a polymer matrix. Understanding the influence of processing conditions on impact modifier dispersion is critical as it will allow us to build a structure-property matrix linking processing conditions, morphology and mechanical properties.

Representative images of the morphology of the material under various processing conditions were acquired by STEM-in-SEM. The image analysis is performed on images acquired at 10kX – this magnification is low enough to capture a meaningful field of view and high enough to resolve the individual particles. The image analysis is based on the ability to "recognize" the impact modifiers domains due to their contrast with respect to the background (which is achieved via specific staining). Once detected, the particles are allowed to "expand" in all directions at the same time until their expanded area reaches an adjacent expanded domain. The domains keep growing until the entire image is filled and domains can no longer grow (see Figure 1). The method then automatically measures and reports the surface area of each individual expanded domain (domains adjacent to the edges of the images are deleted). The data are then analyzed using the Minitab™ software. The standard deviation of the distribution of the expanded domains is not enough to quantify dispersion as in some instances the smaller expanded domains within one agglomerate negate the larger expanded domains between agglomerates. Therefore we look at the linear fit of the distribution for each sample: a perfectly homogeneous distribution would result in a linear time series with a slope equal to 0 (all expanded domains would have the same size). The "less linear" a distribution is, the less homogeneously dispersed it is. We then quantify the deviation from a perfectly homogeneous distribution with the mean average deviation (MAD), which allows clear differentiation and ranking of the samples (see Figure 2).

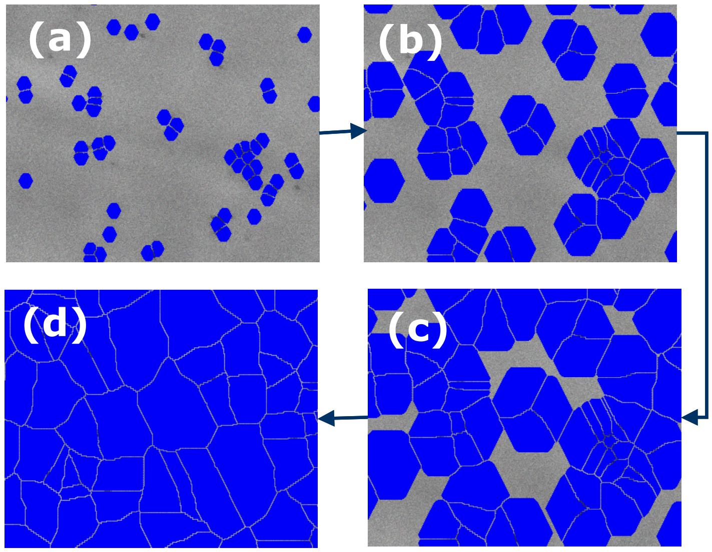

Figure 1: Image analysis approach: a) the particles are automatically detected; b) and c) the domains are dilated and allowed to expand in all directions; d) the process continues until the entire frame is filled.

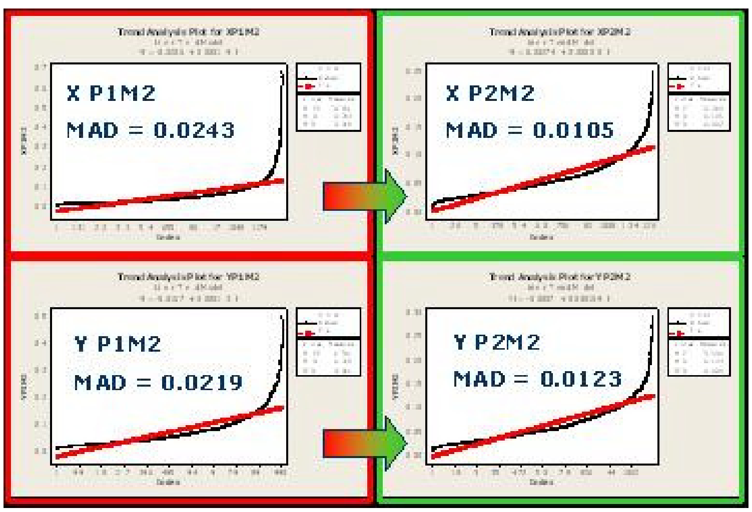

Figure 2: Statistical analysis of the dispersion of the impact modifiers domains using Minitab.

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