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Assessment of the Development of Phase Co-continuity in Immiscible Polymer Blends by Image Analysis of Planar Surfaces

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Abstract

A range of compositions of selected immiscible polymer pairs, PS/HDPE, PS/PP, and PMMA/HDPE, were melt extruded to form uncompatibilized immiscible blends. The morphology and interphase linking of such blends is critical to their performance since no formal chemical bond occurs across the domain interfaces. The prepared blends demonstrated the expected behavior: low volume fraction compositions consisted of one phase dispersed in the matrix of the second phase, and more balanced compositions yielded co-continuous compositions at the phase inversion point. Morphologies were studied by electron microscopy and a simple semi-empirical image analysis model is proposed to enable the quantitative assessment of developing phase continuity in such systems. This model, based on domain section perimeter, root area, and number, correlates well with traditional visual assessments and provides the basis for more quantitative comparisons of immiscible polymer blend structures.

Keywords:

Blends, immiscible, continuity, image analysis, model, HDPE, PMMA, PP, PS

Introduction

Polymer blends have become widely used as an effective approach to combining various functionalities of individual polymers into a composite blend that, ideally, possesses the best properties of the constituents. A specialized subset of these blends are blends prepared from immiscible polymers without the use of compatibilizers so that the interfaces between domains are not chemically bonded. In all blends, but particularly in this latter classification of blends, the development of co-continuity is critically important since the load transfer between domains is completely dependent on intimate contact between the phases. This close contact, sometimes termed "mechanical grafting," as well as other aspects of blend morphology on mechanical properties have been frequently studied [1-4] and significant improvements in the structural properties of yield strength and Young's modulus have been noted in selected systems. [5-8]

The measurement of co-continuity has been a subject of previous and numerous techniques exist in the literature for characterizing such morphologies. [6, 9, 10] One of the most widely used techniques for detecting and quantifying co-continuity is that of solvent extraction. In this approach, a suitable solvent that selectively removes the targeted phase is used to extract a specimen and the degree to which the targeted phase is extracted is a measure of the continuity of that phase in the blend.[11-13]. However, often a suitable solvent cannot be found, particularly when the phases have similar physical and chemical properties, and the extraction methods also suffers from a lack of sensitivity to subtle changes in continuity and its destructive nature.

Image analysis is an attractive alternative to laboratory methods due to the ease and ubiquity of good optical and electron microscopy images in most research laboratories. Morphology and domain identity are quantified in etched or unetched images and a variety of image analysis software programs may be used to generate quantitative data from the structure. Although the determination of three dimensional characteristics, such as co-continuity, from a two-dimensional image has distinct limitations, this technique has become widely used in at least approximating the degree of co-continuity in blend structures. .

Quantification of co-continuity is an important milestone in morphology assessment if comparisons and correlations with processing methods and between compositional parameters is to be achieved. Galloway[14] reported a technique based on measurement of the perimeter per unit area between phases to predict the co-continuous region. He developed an algorithm using MATLAB software to detect and measure polymer-polymer interfaces in a PEO-PS system. The amount of interfaces present in the system showed a local maximum at the boundaries of the co-continuous region. Steinmann[15] used a dimensionless form factor based on the area and perimeter of the domains observed in the blend images to define and detect co-continuity. Heeschen [16] defined morphological parameters such as "co-continuity" and "co-continuity balance" based on the extent to which phases of a blend mutually surround each other, and the relative contribution of each phase to co-continuity respectively.

Studies in our laboratories have focused on the morphologies that develop in uncompatibilized immiscible polymer blends over a wide range of compositions. Naturally, without compatibilizers, the co-continuous range is narrow and more often than not the

compositions we study indicate stages in the development of co-continuity. In this paper we present a novel way of examining developing blend continuity in terms of a scale-independent domain perimeter-area factor applied to assess the degree of coalescence/dispersion and the effects of channel formation and domain linking in blend morphologies as observed from two-dimensional image analysis surfaces.

Experimental

Blends

Polymer blends spanning six combinations of polymer components and singe screw extrusion processing methods were generated to provide a moderately wide variety of materials and morphologies for assessment of co-continuity. PS, PMMA, HDPE, and PP were paired in selected combinations and each blend was extruded in both a simple single screw extruder and a specially designed single screw extruded possessing exceptional mixing and elongational shear elements.

Raw materials used

The four raw materials [PS, PMMA, HDPE, & PP] employed in this study are tabulated in Table 1 along with their melt flow index and physical properties. The polymers were received from the supplier in the form of pellets and blend batches were pre-blended to a homogeneous mix prior to melt extrusion. PMMA was dried for 16 hours[§] at 82^{o}C to remove moisture.

Rheology

[§] *Novadrier™ N7, NovaTec Inc.*

The viscosities of all polymers were measured in a TA Instruments AR 2000 rheometer at the processing temperature of 200^{o}C. This rheometer uses parallel plate geometry and test samples were prepared by compression molding a circular disc of suitable dimension for the apparatus. Viscosity data were collected versus shear rate in the linear visco-elasticity region and these data was used for continuity predictions at shear rates present during extrusion. Such estimates of the compositional location of the co-continuous region in extruded blends are commonly made from viscosity data using a variety of relationships. The relationship that we find most useful is the Jordhamo equation[17]:

$$\frac{\eta_A}{\eta_B} \cong \frac{\Phi_A}{\Phi_B} \qquad [1]$$

From this relationship the co-continuous region is estimated as that composition where the volume fraction (Φ) ratio of the polymers in the blend is equal to the viscosity (η) ratio of the polymers at the shear rate of the extrusion process. For the three systems studied in this work, PS/HDPE, PS/PP, and PMMA/HDPE, the co-continuity point, in volume percent of PS or PMMA, the three systems is 37.7%, 36.4%, and 60% respectively. As a practical consideration, the Jordhamo relationship is an approximation and furthermore co-continuity develops over a range of compositions, not just a single point. Thus, in our studies we construct a window of potential co-continuity around the calculated value of 15% relative, or ±7.5%.

Extrusion

Blends were extruded in two different machines : a C.W. Brabender Intellitorque Plasti-corder with a screw L/D of 32:1 and fitted with a 3 mm die, and a vertical Randcastle

extruder with custom screw designed to produce a high level of mixing and elongational shear possessing an L/D of 50:1 and 1.5 mm fiber dies at the outlet. Three polymer pairs that combine an amorphous polymer with a semicrystalline polymer were selected from PS, PMMA, HDPE, and PP and a series of compositions bracketing the expected co-continuous region were extruded in both Brabender and Randcastle extruders to give six blend series. The Brabender extruder was operated at 200^{o} C and 60 rpm for all compositions. The Randcastle extruder was run between 200^{o} and 220^{o} C and at 180 rpm. A total of 40 extruder runs were made, as shown in Table II, and samples were collected from each for microscopic analysis.

Microscopy

Specimens from the 40 extrusion samples were cryo-fracturing in liquid nitrogen to produce representative fracture surfaces. In examining samples from various parts of the extrusion stream, very little variability was observed from the beginning to the end of the run, i.e. the extruded rods were homogeneous throughout the run. The fibers were fractured along a plane perpendicular to the extrusion direction and then etched in solvents to enhance imaging. Toluene was used to etch polystyrene and di-methyl-formamide (DMF) was used to etch PMMA. The specimens were then mounted, sputter-coated with and kept overnight under vacuum to remove volatile components from the mounting process. The specimens were observed under a Leo-Zeiss Gemini 982 Field Emission Scanning Electron Microscope at an operating voltage of 5 keV. Images were recorded at various magnifications, but for the present study, only those at 2000 X were used since this magnification produced micrographs that best represented the microstructures of the blends. As a preliminary exercise, a variety of specimens were examined from various parts of the extrusion stream to assess sources of variability in the analysis. The extrusion stream exiting the extruder exhibited very little variability from the beginning to the end of the run, i.e. the extruded rods were homogeneous throughout the run. Some level of variability was observed in individual specimens as a function of radial position, but this variable was effectively averaged by selecting observation points approximately at the radial midpoint. Interestingly, the greatest degree of morphological variability occurred between repeated FESEM images at constant radial position on a single specimen. In reflection, this effect is not unexpected since the statistical variability of the morphology will appear greatest at high magnification as the sampling area

decreases. Given these observations, experimental replication was directed at repeated FESEM imaging and the error bars in the analysis refer to standard deviations based on this source of variability.

Image Analysis

Image analyses on the photo-micrographs obtained from the FESEM was performed using Adobe Photoshop's Image Processing Toolkit. All images were first calibrated using the micron markers on the pictures and then converted into binary images using a threshold filter available in the toolkit. The threshold filter levels were selected by visual observations and judgment of the image before and after setting the threshold with the objective of producing a binarized image that faithfully replicated the optical image with regard phase distribution and domain sizes and shapes. Once the binarized images were generated, the image analysis routines were run with various filters in place to generate values of domain perimeter, domain area, and number of domains in each image. The selection of 2000X magnification mentioned above was a compromise between the enhanced resolution and detail of high magnification with the image analysis error associated with edge effects. Any domains touching the edges of an image are appropriately excluded from the analysis since only partial knowledge exists of that domain's perimeter and area. Thus, magnifications that produce images with a low number of domains will yield poor results due to edge effect errors.

Results and Discussion

Morphology from FESEM Images

Morphology development in polymer blends occurs during the mixing of the molten polymer in various stages of the extruder. Although compositional effects are a strong factor in determining the final structure as discussed previously, the elongational shear and mixing characteristics of the extruder also contribute. Overall, the final morphology is determined by processes of shear-induced dispersion, coalescence and elongation taking place during various stages of mixing. Such processes have been extensively analyzed in previous studies[18-21], and morphology development appears closely linked to a combination of these processes occurring dynamically in the extruder. The FESEM photomicrographs of compositions in the PS/HDPE system extruded by Brabender and Randcastle equipment (figures 1a and 1b) show a progression of morphologies from circular sections of small, apparently cylindrical or spherical domains of PS at low concentrations to increasingly elliptical and ultimately co-continuous morphologies at higher concentrations. For this system, the Jordhamo co-continuity point is 40 weight percent PS. At 20% PS a highly dispersed morphology is observed for both types of extrusion, although the domain sections in the Randcastle specimens appear slightly more elliptical, a precursor of developing co-continuity. At 30% PS the Brabender domain sections are exhibiting a degree of elliptical shape and the Randcastle domains have progressed nearly to co-continuity. This progression of domain section morphologies from circular to elliptical to dumbbell-shaped to co-continuous continues as the PS concentration increases. In comparing figures 1A and B the Randcastle PS domain sections appear to progress more rapidly to co-continuity, reaching an apparent co-continuous state at 40% PS whereas the Brabender PS domain sections reach this point at 45%.

The above paragraph contains subjective assessments of the degree to which continuity is developing in the PS/HDPE system under two thermal processing routes. The subjective nature of this process, and the fact that skilled microscopists and polymer scientists can disagree on the interpretation of the same photomicrograph, suggests that a more quantitative, less subjective methodology is needed to determine not only the presence of single phase continuity or the co-continuity of multiple phases, but the development of continuity precursors in dispersed morphologies.

Continuity Model

In developing a quantitative model for continuity the image factors associated with continuity must first be identified and then quantified in a way compatible with computerized image analysis techniques. All of this is complicated by the fact that most three dimensional continuity is not continuous in two dimensions and that the axis upon which the specimen is sectioned will have a strong effect on the observed structure, particularly for the highly anisotropic, albeit axisymmetric, extrudates generated by Brabender and Randcastle machines. Having recognized the difficulties and complexities of the challenge, and that no model will address all, or even most, structures, we propose a simple model based on several readily observable parameters: the perimeter, the area, and the number of domains observed in a planar section and combined in the following relationship to yield a continuity factor, CF.

$$CF = \frac{\left(\frac{\sum \frac{P_i}{\sqrt{A_i}}}{N} \right)^2}{N^{0.1} \frac{A_T}{A_I}} \qquad [2]$$

Where P is the perimeter and A is the area of the ith domain, the sum is over all domains in the image, A_T is the true area fraction of the phase and A_I is the area fraction measured by the image analysis process. The ratio is a correction term for inhomogeneous or non-representative images and is usually near unity. The continuity factor is zero for a highly dispersed structure with a nearly infinite number of circular domain sections. Values of 10 or 20 are typical for moderately elongated composite structures such as those in figure 1 in the co-continuous region, and values >1000 can be achieved for extremely elongated systems.

The perimeter/area term appears in numerator of the continuity relationship since circular domain sections are not associated with three dimensional network continuity, although they can be characteristic of one dimensional "box of spaghetti" continuity when such a composite is sectioned perpendicular to the longitudinal axis. An increase in elliptical, or other elongated shapes, is a sign of developing continuity and the scale independent and dimensionless ratio P/√A is a measure of this effect. The number of domains is in the denominator of the continuity expression since a truly continuous phase will only have one domain in three-dimensional space. A planar image of such a continuous structure will likely have more than one domain section, but the inverse relationship between the number of domain sections and the continuity of the structure is clear.

The relationship between these variables and the developing continuity of a structure is depicted by a simple graphical model (figures 2 and 3) in which a 20% volume fraction of one phase is blended with 80% of another. Accurate scale images of the schematic morphology are presented on a coordinate axis system where the abscissa is the number of domain sections and the ordinate is ratio of the major to minor axis an ellipse used to model the elongation of the domains. At the origin is a single circular domain section and the CF for this structure has been normalized to 1.0. Moving vertically corresponds to increasing the elliptical nature of the domain(s) which is a sign of developing continuity and a corresponding increase in CF from 1.0 to 4.0 reflects this change. However, if circular domain section shape divides into four small circles, as shown along the abscissa, this change is not associated with developing continuity (CF=0.87) unless this dispersive effect is combined with elongation as illustrated in the upper right of figure 2 where many highly elongated ellipses appear well on the way to developing continuity and the CF = 7.94. Of course the image in the upper right of figure 2 is not continuous. The missing feature is coalescence of these domains to form a continuous structure. Such linking or network formation is schematically illustrated in figure 3 for spherical and elliptical domain sections. Network formation in these images is defined as the number of linkages divided by the number of domain sections, expressed as percent. For circular domain sections the CF is 0.87 for 0% network formation and increases to 4.04 for a 100% networked structure. The elliptical domain sections show a much greater increase in CF (CF=123) as the network degree reaches 100%.

This model seems consistent with our experience working with immiscible polymer blends in that highly elongated structures are rewarded with high CF values and simple dispersion is

penalized. However, dispersion followed by elongation followed by coalescence, or network formation, is highly rewarded and produces the highest CF values consistent with our experience with highly co-continuous structures. The model also possesses a certain intuitive appeal stemming from the fact that true co-continuous composites have only one domain of each polymer and this necessitates a highly elongated and channeled domain structure. However, the model and its applicability to microscopy images has significant limitations. Branch points, often cited as characteristic of co-continuous structures, are not part of this model, although branching and network formation are part of the perimeter/$(\text{area})^{0.5}$ summation term. Perhaps most significant is the fact that this approach, as well as any approach that analyzes a single planar micrograph, is subject to the errors of representing a three dimensional structure based on a two dimensional section.

Co-continuity measurements on immiscible blends

Values of domain section perimeter, area, and number were collected from image analyses of the photomicrographs in Figures 1A-B, as well as for similar photomicrographs for the PMMA/HDPE and PS/PP blends, and inserted into equation [2] to generate quantitative continuity versus composition curves for these three blends series. For each image analysis sequence, one preliminary check consisted of comparing the domain area by image analysis with the actual volume faction as determined by the batch composition. The intent was to identify images that represented inhomogeneous areas of the blend and to generate the area fraction correction term in the denominator of equation [2]. Interestingly, in addition to the expected random variability, a systematic discrepancy was also observed as illustrated in Figure 4. The observed area fraction was only 75% of the expected area fraction. Although this peculiar anomaly has not been fully resolved, it appears to result

from one or more of the following effects: incomplete etching, a slight image threshold bias, and/or edge effects during the image analysis.

The continuity of PS in the PS/HDPE blends as represented by the calculated continuity factor [CF] versus composition (figure 5) shows two major effects. The Randcastle processed blends have a higher level of continuity throughout the composition range compared with the Brabender blends and the peak continuity is achieved at 40% compared with 45%. The difference in peak continuity most likely results from different shear environments in the two extruder. Generally, this quantitative trend appears consistent with the visual trends observed in the micrographs. The Randcastle blends also a statistically insignificant hump in the continuity curve near 30% that mirrors a visual assessment of the micrograph and is not present in the Brabender blend data. Overall, the continuity factor model appears to be providing an accurate and useful quantification of the morphology of these blends.

The CF versus composition curves for two other blend systems, PMMA/HDPE and PS/PP (fig 6 -7) show similar trends. The PMMA/HDPE blends demonstrate similar continuity for the two extruder types until the 40% PMMA composition is reached at which point the Randcastle blends show distinctly higher continuity. This trend continues to the point of maximum continuity, 65% PMMA, consistent with expectations from the Jordhamo relationship. The PS/PP system did not develop the expected level of continuity in the Jordhamo region, indeed only a slight increase of continuity is observed by the 40% PS composition and no significant differences exist between the Randcastle and Brabender processing. A moderately sharp increase in continuity is observed for the Brabender blends

at 45% PS, but no reliable data were collected at high compositions due to problems obtaining a clean and accurate etch of high PS blends.

Summary and Conclusions

Three pairs of immiscible polymers were melt-blended in two types of single screw extruders and the morphologies developed over a range of compositions were examined by electron microscopy. Subjective visual assessment of the micrographs revealed the expected blend morphology of one dispersed phase in a continuous matrix for compositions far from the phase inversion composition, with increasingly elliptical-shaped domain sections and coalescence occurring as the blend composition moves towards the phase inversion point. Ultimately, co-continuous morphologies were observed at compositions close to the phase inversion point as calculated by empirical relationships based on rheological relationships. A quantitative semi-empirical model of developing co-continuity was set forth and used to quantify the heretofore subjective process of identifying the developing continuity of a phase in such immiscible polymer blends. Graphs of calculated continuity factors correlate well with expectations based on visual observation and provide a quantitative basis for the comparison of structures.

Acknowledgements

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Tables

Table I: Raw Materials and Selected Physical Properties

Polymer	Melt Flow Data: Index (g/10 min.)	Melt Flow Data: Load [kg]	Melt Flow Data: Temperature [^{o}C]	Density (g/cc)	Tensile Yield (MPa)	Flexural Modulus (MPa)
High density polyethylene (HDPE)	0.35	2.16	190	0.952	27	1309
Virgin Grade polystyrene (PS)	7.0	5.0	200	1.04	53.78	3447.3
Polypropylene (PP)	0.65	2.16	190	0.9	33.5	1275
Poly-methyl Methacrylate (PMMA)	2.30	3.8	230	1.19	70.3	3102.6

Table 2
Blend Formulations for Image Analysis

PS/HDPE		**PS/PP**		**PMMA/HDPE**	
PS weight	*PS volume*	*PS weight*	*PS volume*	*PMMA weight*	*PMMA volume*
20.0%	18.5%	20.0%	17.6%	20.0%	16.8%
30.0%	28.0%	30.0%	26.9%	30.0%	25.8%
35.0%	32.9%	35.0%	31.6%	35.0%	30.4%
40.0%	37.7%	40.0%	36.4%	40.0%	35.0%
45.0%	42.7%	45.0%	41.2%	50.0%	44.7%
50.0%	47.6%			60.0%	54.8%
				65.0%	60.0%
				70.0%	65.4%

All blends were processed both in the Brabender
and the Randcastle single screw extruders

Figure Captions

Figure 1A. Morphologies of PS/HDPE blends processed in a Brabender single screw extruder. Volume fraction of PS is shown in insets. Original magnification, 2000X. See micrometer bar on figure for actual magnification.

Figure 1B. Morphologies of PS/HDPE blends processed in a Randcastle single screw extruder. Volume fraction of PS is shown in insets. Original magnification, 2000X. See micrometer bar on figure for actual magnification.

Figure 2. Schematic continuity model – the effect of dispersion/coalescence and eccentricity on the continuity factor for a 20/80 blend with no network connectivity.

Figure 3. Effect of ellipse linking on continuity factor for two structures in a 20/80 blend.

Figure 4. Phase fraction measured by image analysis compared with actual batch composition, volume percent.

Figure 5. Image analysis continuity factor for PS/HDPE blends.

Figure 6. Image analysis continuity factor for PMMA/HDPE blends

Figure 7. Image analysis continuity factor for PS/PP blends.

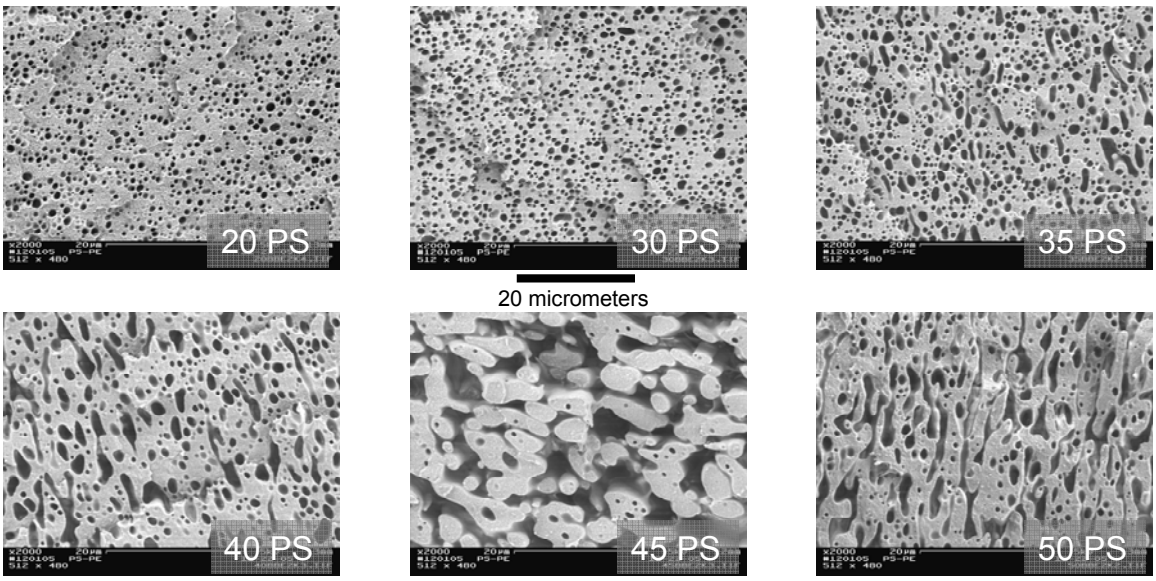

Figure 1A. Morphologies of PS/HDPE blends processed in a Brabender single screw extruder. Volume fraction of PS is shown in insets. Original magnification, 2000X. See micrometer bar on figure for actual magnification.

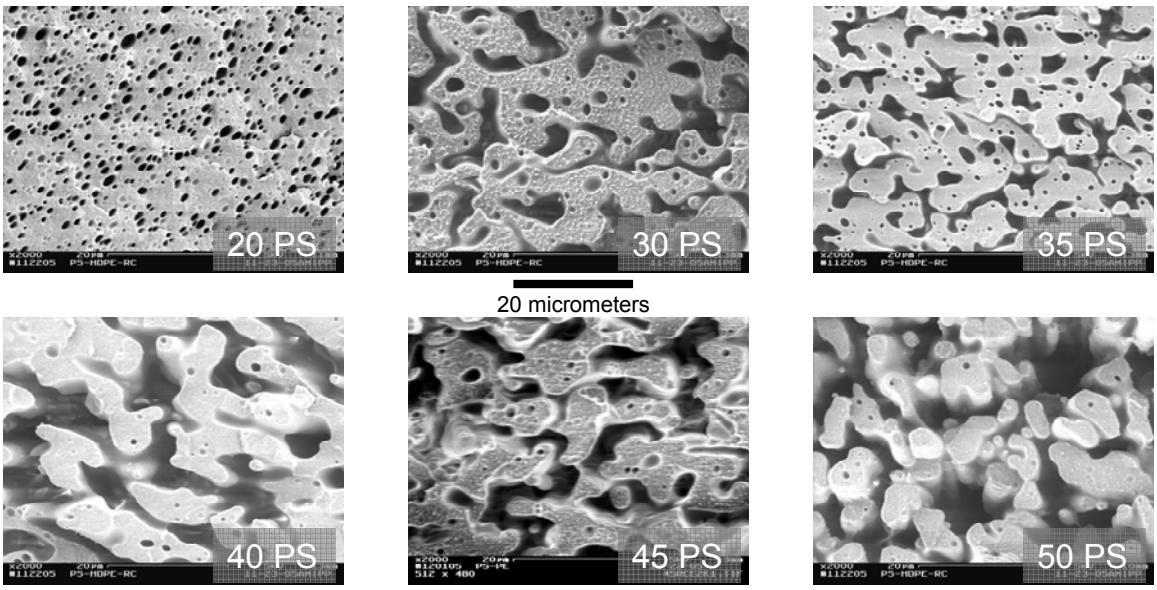

Figure 1B. Morphologies of PS/HDPE blends processed in a Randcastle single screw extruder. Volume fraction of PS is shown in insets. Original magnification, 2000X. See micrometer bar on figure for actual magnification.

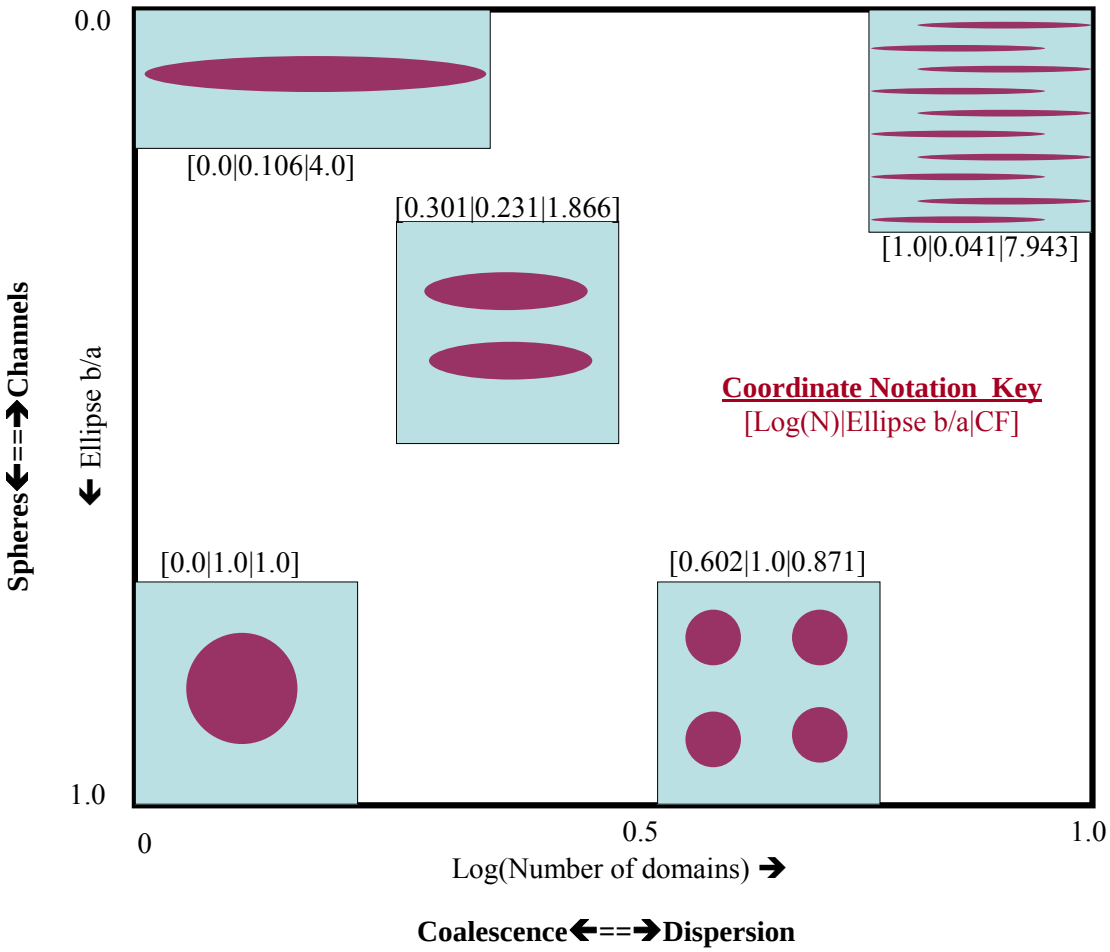

Figure 2. Schematic continuity model – the effect of dispersion/coalescence and eccentricity on the continuity factor for a 20/80 blend with no network connectivity.

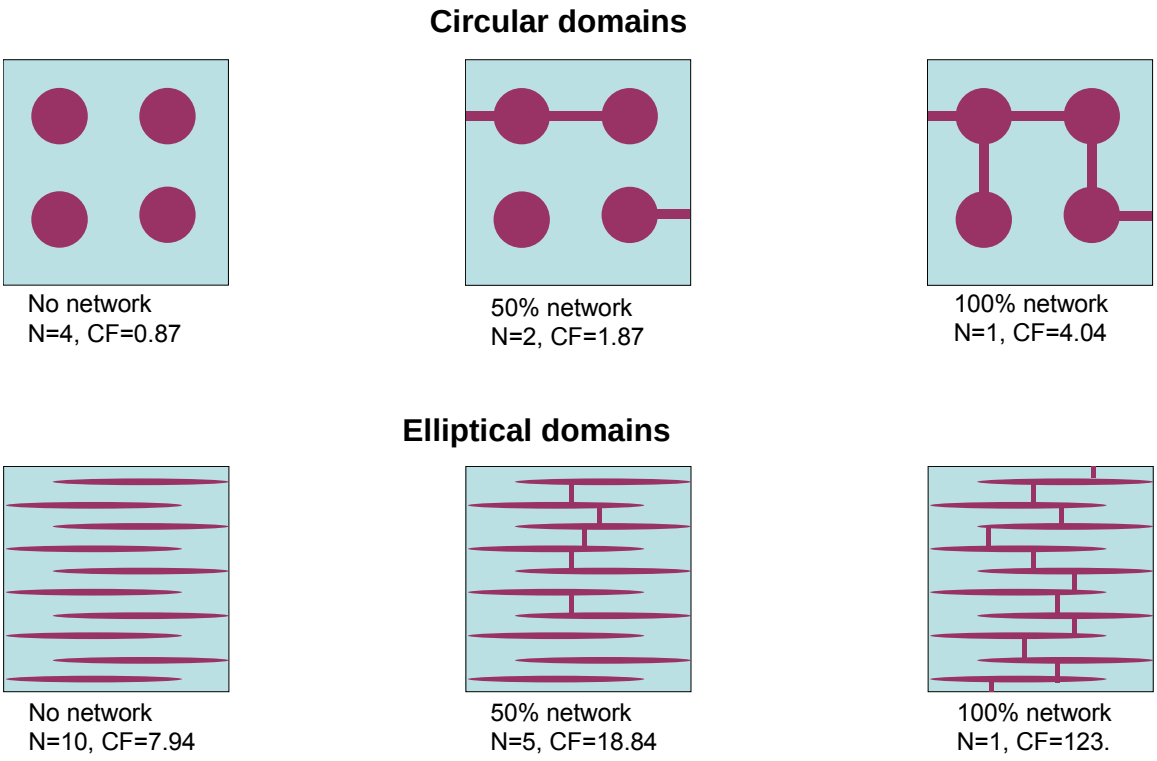

Figure 3. Effect of ellipse linking on continuity factor for two structures in a 20/80 blend.

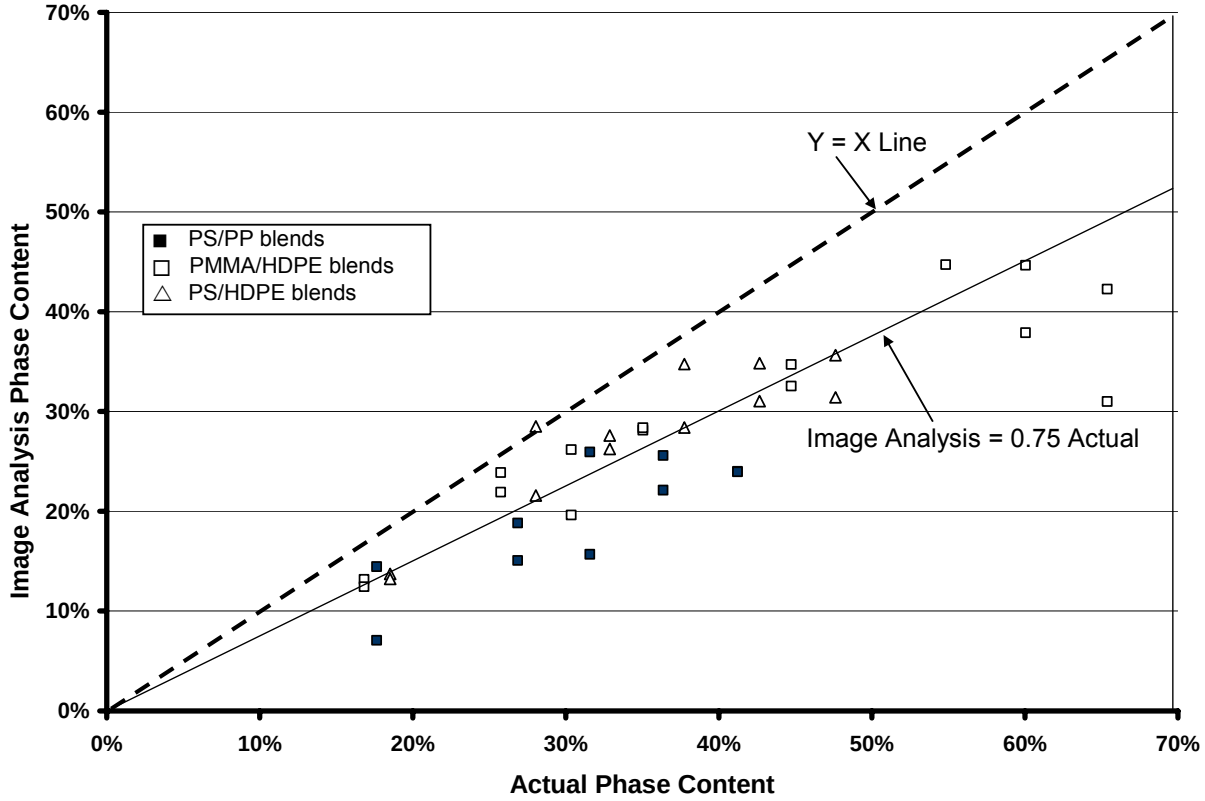

Figure 4. Phase fraction measured by image analysis compared with actual batch composition, volume percent.

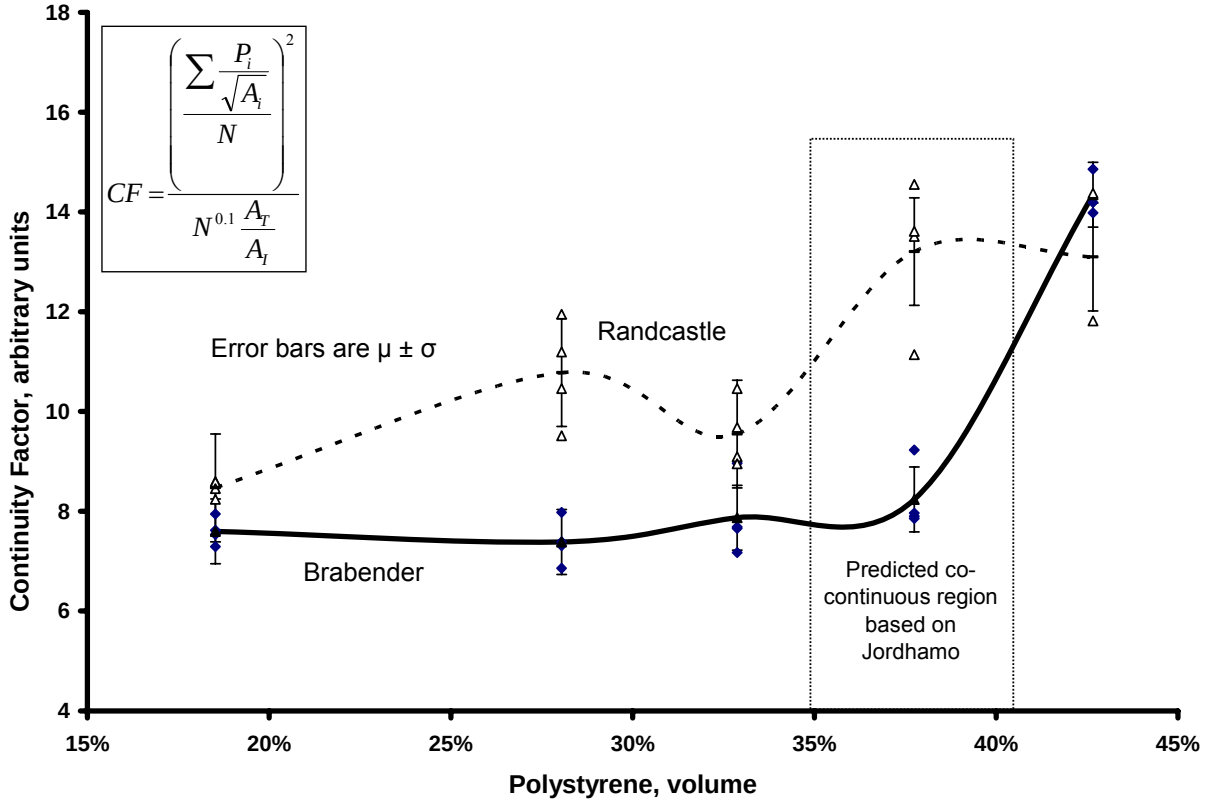

Figure 5. Image analysis continuity factor for PS/HDPE blends.

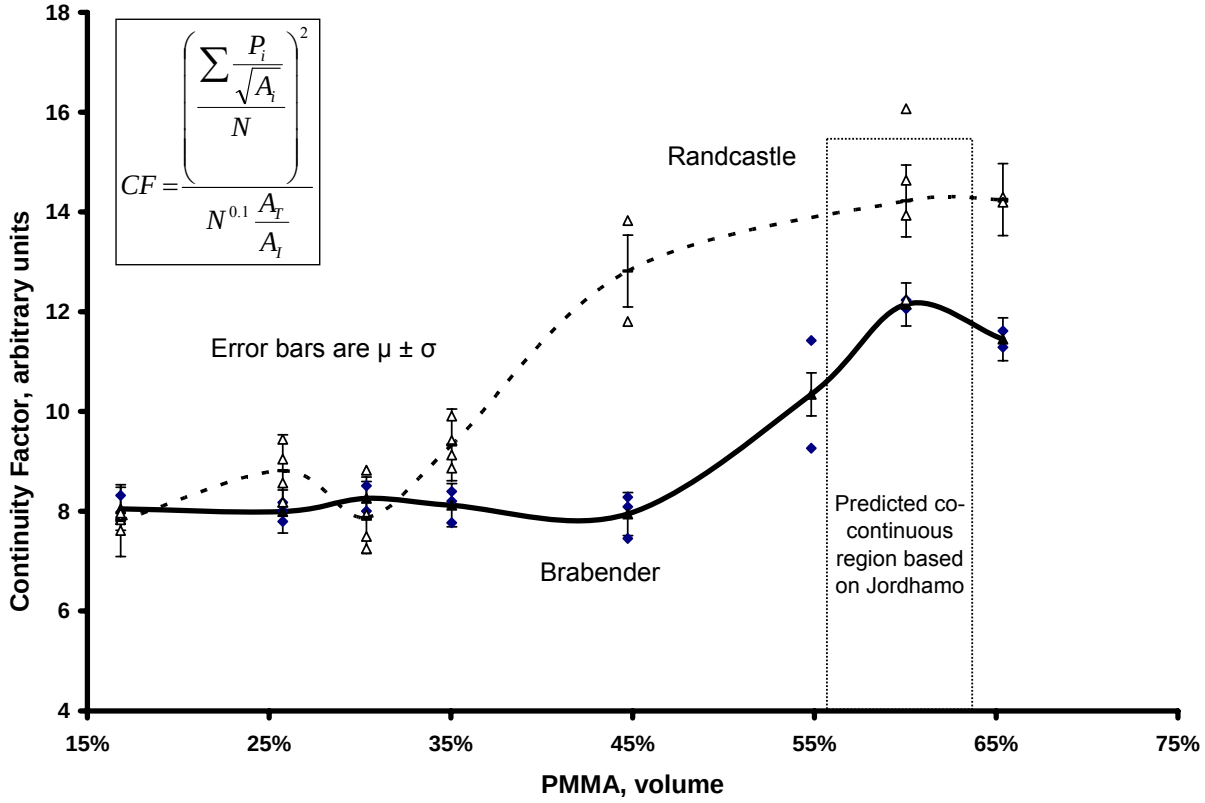

Figure 6. Image analysis continuity factor for PMMA/HDPE blends

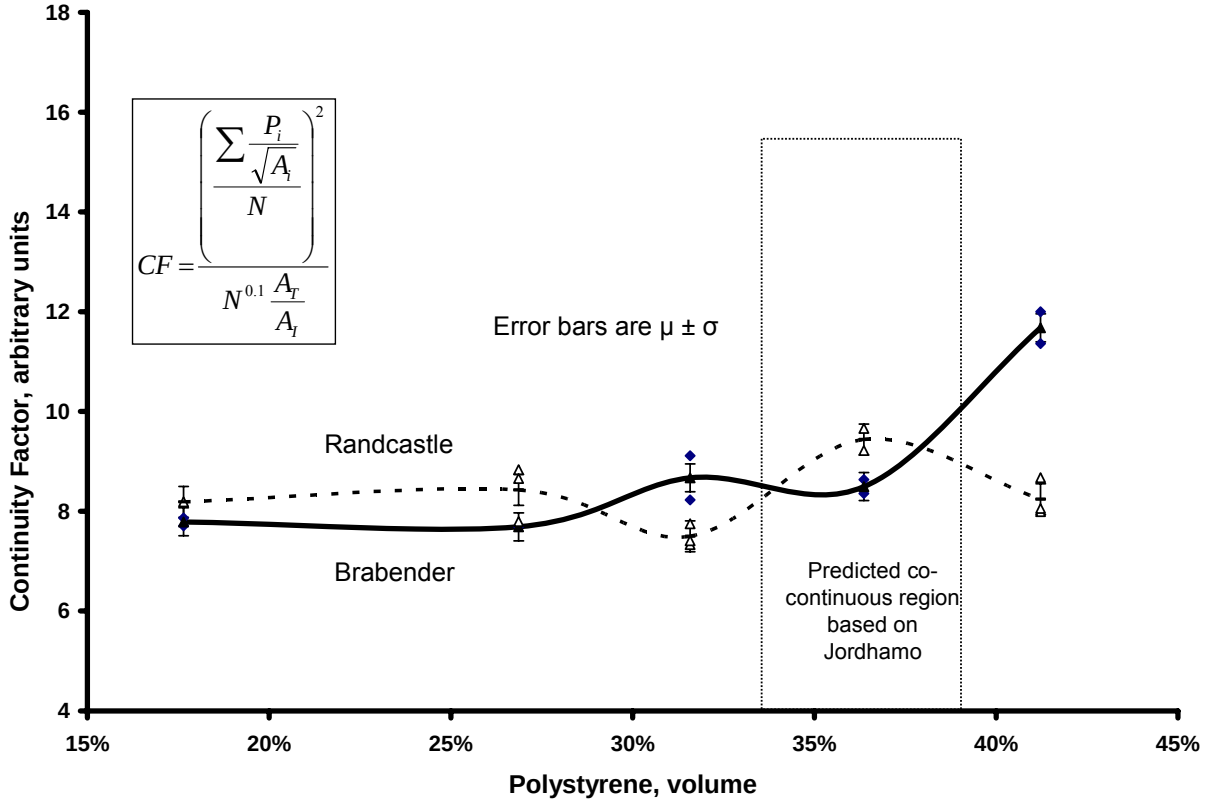

Figure 7. Image analysis continuity factor for PS/PP blends.