

Determination of the phase structure evolution in immiscible polymer blends with electron microscopy

I. Fortelný¹, M. Lapčíková¹, and B. Dimzski^{1,2}

1. Institute of Macromolecular Chemistry AS CR, v.v.i., Heyrovsky Sq. 2, 162 06 Prague 6, Czech Republic
2. Faculty of Technology and Metallurgy, Ss. Cyril and Methodius University in Skopje, 1000 Skopje, Republic of Macedonia

forteln@imc.cas.cz

Keywords: polymer blends, phase structure, coalescence, electron microscopy

Polymer blends represent a very important segment of thermoplastic materials. It is due to the fact that materials with a broad scale of properties can be obtained by the mixing of common polymers in universal mixing device. The phase structure of immiscible polymer blends in solid state is non-equilibrium, frozen by cooling of the blend components below their melt and/or glass temperature. The control of the phase structure in immiscible polymer blends, which is necessary for achievement materials with required properties, is extremely difficult task. Therefore, check of the phase structure in various steps of the blend preparation and processing is very important. Because typical size of phase domains in blends having acceptable mechanical properties changes from tenth to tens micrometers, scanning and transmission electron microscopy seem to be proper tools for study of the blend morphology. Nevertheless, many conditions must be fulfilled for achievement of reliable description of the phase structure in immiscible polymer blends with electron microscopy.

A certain disadvantage of the electron microscopy methods is the fact that the phase structure in polymer blends can change during their cooling. It is commonly assumed that the phase structure formed in molten state persist if a sample is quickly quenched. This assumption should be verified especially for blends where one or both components are semicrystalline polymers. On the other hand, optical microscopy and light and X-ray scattering, which were recently applied for the phase structure determination in molten polymer blends flowing in rheometers, have also strong limits. But comparison of their and electron microscopy results can help to decision if the phase structure formed in molten state persists in the quenched sample. The second general problems of microscopic methods is that 2D pictures of 3D structures are only determined. It is serious problem especially for discrimination between co-continuous and dispersed structure. In this case, study by electron microscopy should be completed by dissolution of individual components with specific solvents. For reliable determination of 3D structure of blends containing dispersed particles, orientation of anisometric and or ordered particles to the sample plain should be considered. Determination of 3D morphology of samples containing particles which are polydispersed in size and or shape is difficult task.

Usually it is assumed that evaluation of the sample area containing several hundreds of particles provides correct average particle size and reliable particle size distribution. It was found, however, that nonuniform phase structure containing areas with strongly different average particles size is formed in a number of blends [1]. An example is shown in Fig. 1. Results of evaluation of the average diameter of polyamide particles in four areas of polystyrene/polyamide/polyethylene (PS/PA/PE) blends are summarized in Table 1. They clearly show that differences among average particle diameters are larger than those which are considered as significant at studies of the dependence of the particle size on system

parameters. It clearly demonstrates that neglecting nonuniformity of the phase structure at these studies can lead to the totally erroneous results. Therefore, uniformity of the phase structure must be checked prior evaluation of the dependence of particle size on system parameters. It should be stressed especially in cases when the blend phase structure is studied by transmission electron microscopy, where small test pieces are observed.

Growth of the size of dispersed particles during blend annealing is so far not fully understood and satisfactorily described [2]. Uniformity of the phase structure of polypropylene/ethylene-propylene rubber (PP/EPR) blends were carefully checked prior start of experimental study growth of EPR particles during annealing. Qualitative information from the SEM micrographs of annealed blends was converted to quantitative structure descriptors, using image analysis technique. Plausibility of this method was verified. Results from the phase structure evolution, during annealing of quiescent immiscible PP/EPR blends, revealed growth in the particle size and shape relaxation phenomena. Comparison of experimental results with the theoretical relations for the growth rate of particle size indicated that coalescence is the main mechanism that contributes to the phase structure coarsening. Increase in the EPR concentration led to a higher growth rate of the size of dispersed particles.

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3. Financial support of the Grant Agency of the Academy of Sciences of the Czech Republic (grant No. IAA 200500903) is gratefully acknowledged.

Table 1. Comparison of the average equivalent diameter $\langle d \rangle$ of PA particles in areas of PS/PA/PE blend containing small (area 1, 2) and large (area 3, 4) particles.

	Area 1	Area 2	Area 3	Area 4
No. of particles	456	263	161	210
Mean size $\langle d \rangle$ (μm)	3.24	3.95	5.50	5.13

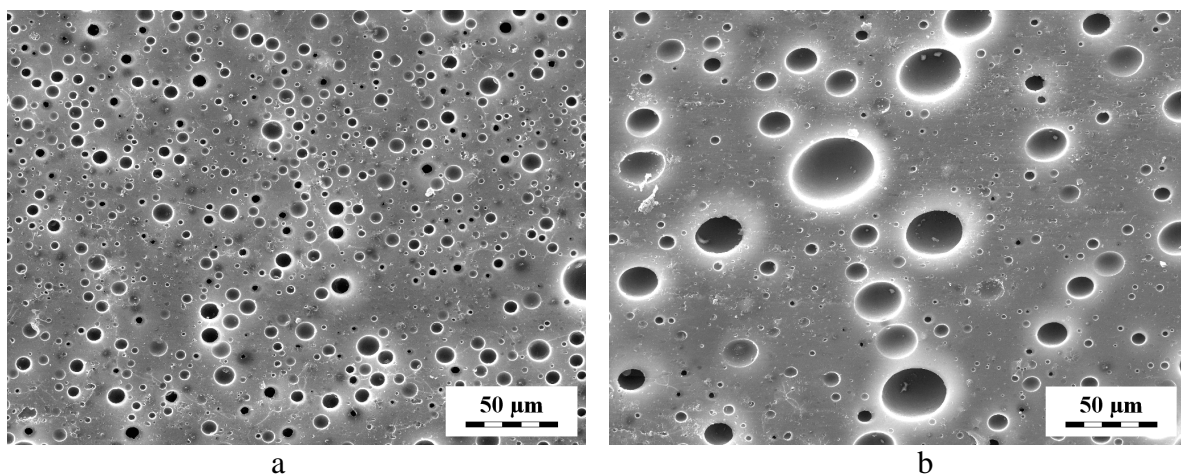


Figure 1. Morphology of two areas in one compression moulded sample of PS/PA/PE1 (75/20/5) blend. a – area containing only small PA particles, b – area containing large PA particles.