STUDY AND CONTROL OF POLYMER BLENDS MORPHOLOGY AND RELATED PROPERTIES

Ph. Teyssié, R. Fayt and R. Jérôme Laboratory of Macromolecular Chemistry, University of Liège, B6, SART-TILMAN 4000 Liège, Belgium

<u>Abstract</u> - It will be shown how a combination of techniques allows to gain a rather precise idea of the (un)miscibility situation in polymer blends at different size scales (i.e. from ca.20 Å up to 1μ); typical examples include simultaneous use of TEM, SEM, NRET and ss. NMR.

On these bases, interesting blends have been studied and tailored, in which both morphology and interfacial adhesion have been controlled (in particular by the use of diblock copolymers) to provide for a better spectrum of properties. A number of situations will be described, implying commodity and engineering polymers, but also different types of fillers; their optimization has led to interesting applications in the field of better emulsion dispersions, very high impact resins, economical engineering plastics, controlled migration, filled materials, ...

INTRODUCTION

In recent years, when the production of new polymers from original monomers or processes faced difficult economical requirements, polymer melt-blending has provided one of the most efficient ways to generate new higher performance organic materials, from largely available products (1). The success of that approach is well illustrated by the large number of versatile blends introduced, fulfilling increasingly specific needs, and also by an impressive growth rate of that type of market, i.e. a 10 to 20% a year over the last decade (2).

Although a continuum of phase situations is documented as expected, two main classes of blends have been distinguished. The so-called "homogeneous" monophase mixtures, a famous example of which is General Electric Noryl, are very interesting in that they allow to broaden the "processing temperature window" of materials, and possibly to modify their melt flow; however, in that kind of blend, practically all properties become intermediate between those of the corresponding homopolymers, a situation which is often undesirable (3). It must also be stressed that the term "homogeneous" has a sometimes subjective meaning, since it depends on the widely different scales comprehended by the different investigation methods, and should be used rigorously only for those techniques unravellingshort-distance (10-20 A) interactions such as NRET, SAXS,... On the other hand, heterophase blends have the great advantage to provide for additivity of the phase properties, and we should thank mother Nature for having made among polymers such incompatible species. In such cases, however, a satisfactory overall spectrum of properties will critically depend on two demanding structural characteristics : a proper interfacial tension leading to a phase size small enough to allow the material be considered as "macroscopically homogeneous", and an interphase "adhesion" strong enough to accomodate stresses and strains without disruption of the desirable morphology. Unfortunately that is not the case for most immiscible polymer blends.

The answer of the chemist to that challenge has been the development of a macromolecular engineering approach, where precise (if sometimes minute) modifications of the interfacial situation combined with astute processing conditions control would lead to so-called tailored "alloys". It might be rightly argued though that the naïve joy of the chemist seeing a given modification at the molecular level strikingly reflected in a charge of the material bulk properties is not quite fair: this jump superimposes indeed many different problems involving microstructure, morphology, kinetics and thermodynamics, processing conditions and history. However, that phenomenological approach, if properly used in comparative terms,

still allows a very useful optimization of materials (each blend being a particular case by itself), and a continuous improvement of the available structure-properties relationships.

In this overview, it will be attempted to illustrate meaningful examples for the power of this molecular engineering approach, and further its <u>very general</u> character as applied not only to polymer-polymer alloys, but also to polymer-fillers, polymer-liquids (3), etc...

I.POLYSTYRENE-POLYETHYLENE ALLOYS AS MODELS FOR SCREENING THE MAIN PARAMETERS CONTROLLING MORPHOLOGY AND MECHANICAL PROPERTIES

The traditional answer to the structural requirements stated above for heterophase alloys has been the creation of a practically irreversible morphology in the polymerization process itself (establishment of covalent bonds between phases), as illustrated by the classical examples of HIPS and ABS resins.

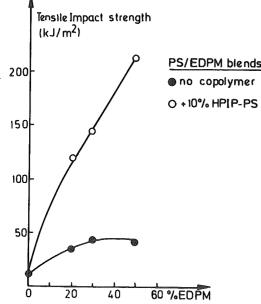
Simple blending of the same rigid and elastomeric polymers involved would, however, represent a more versatile approach, allowing the easy production of a continuum of different compositions by processing various proportions of these base polymers. Although other solutions are possible to promote the desired interfacial tension and adhesion (see section IV.2), the more versatile answer has been the use of block copolymers of suitably tailored composition and molecular weight (4).

In that prospect, poly(styrene-b-hydrogenated polybutadiene) diblocks (PSHBD), pure or "tapered", have been synthesized by anionic polymerization in non-polar medium, followed by hydrogenation using Ziegler-type catalytic systems (5). The resulting rubbery block thus contains about 90% of ethylene and 10% butane units, a structure very close to that of LLDPE often used with LDPE as the "soft" phase of these blends. The investigation of these model alloys did put in evidence the behaviour and key-structural features of the "emulsifying" diblock bridging of the compatibility gap between phases. The characteristics of the homopolymers used are of

course also important ⁽⁶⁾ but have been kept constant in most of these studies for comparative purposes.

- 1) Gross effects of the block copolymer in a melt-blended material can be summarized as follows:
- the phase mean size decreases enormously and comes down to a sub-micron situation (typically 500 nm, and even smaller depending on concentration and structural characteristics)(7)
- the morphology may change from dispersion to co-continuous (see below), as shown by selective extraction experiments of a 75% PS component, leaving intact the overall shape of the PE foam-like remaining phase (7);
- the bulk physico-mechanical properties of these alloys are strikingly improved, compared to the simple blend, by the presence of the copolymer (5,8) much higher elongation, stress and total energy at break, all over the composition range; much higher impact strength (see for example Fig. 1); and much smoother cold-fracture surfaces (a critical test for polystyrene) indicating an excellent phase adhesion.

Fig. 1
Tensile Impact
Strength of a
Polystyrene-EPDM
Alloy containing a
Poly(styrene-bhydrogenerated
isoprene) Diblock
Copolymer



- 2) Stability of these dispersions under processing has been ascertained in a number of cases. Either after roll-milling, compression molding and film stretching, or after injection molding, the fine sub-micron dispersion as well as the high level mechanical properties are maintained; they remain permanent characteristics of the samples (irrespective of composition) together with a high-gloss surface for the polystyrene-rich samples (/). These results indicate that, under proper conditions, good mixing results in a kind of a "machine equilibrium", i.e. reproducible samples from a well-controlled fabrication recipe. However, rheological characteristics are important here, and it was shown indeed that "tapered" diblock copolymers which have a lower melt viscosity than the corresponding "pure" diblocks, allow one to reach the final morphology and property plateau in a much shorter time than the latter ones(typically 30 seconds instead of 2-3 minutes) In specific cases, selective migration of the block copolymer to the surface must also be considered (9), a potentially interesting means to modify surface properties. It must also be stressed that solution casting techniques, which may lead closer to a true phase equilibrium and are more attractive in specific cases, are not as representative of "real life" processing conditions and have also their own drawbacks : selective dephasing in concentrated solu-
- 3) The location of the diblock copolymer in the processed samples is another important question, determining the final efficiency of the additive. In PE-PS alloys, a PSHBD containing a short central polyisoprene block (unreactive to the selective hydrogenation process) has been shown (osmium staining) to go as expected mostly to the phase interface $^{(10)}$; in polystyrene-rich products, it also promotes at the same time the formation of a certain amount of inversedphase, efficiently developing a "salami-type" of structure very similar to that encountered in HIPS, and ensuring highly desirable properties (high $\sigma_{\rm B}$ and $\varepsilon_{\rm B}$, high impact, excellent ageing).

tions, and very difficult drying under Tg.

- However, less clear-cut situations are often encountered (4), which then require further optimization (see Section II).
- 4) The amount of additive required is obviously a key parameter in terms of economics. For a given and already optimized diblock, the ultimate tensile properties are improved up to a plateau which is reached between 1 and 2% and then remains practically unaffected up to over 10% of that additive. Corresponding SEM micrographs clearly confirm that the more drastic change in morphology occurs with as low as 0.5% of the diblock, and that no major modification is seen above 2% (7,8), thus making this approach a perfectly acceptable one in economic terms.
- 5) The length of the blocks in the added copolymer has also been traced as an important molecular factor in those alloys where only non-polar interactions are at play. The resulting behaviour is however not straightforward, since tapered blocks of relatively lower molecular weight (20-40.000) promote higher $\sigma_{\rm R}$, sometimes with synergistic effects ($\sigma_{\rm R}$ higher than the homo-PS component), while higher M.W. (around 100.000) favours quite high $\epsilon_{\rm p}$. This behaviour might be explained by the fact that very high $\sigma_{\rm p}$'s seem to correspond to the presence of a co-continuous morphology, characterized however by a fragile-type failure in stress-strain curves, while high $\boldsymbol{\epsilon}_{R}$ are characterized by a yield curve characteristic of a ductile high-impact type of material (8). No doubt that this latter behaviour must be related at least in part to the efficiency of entanglements in providing for mechanical interphase adhesion under stress; in cases where strong interactions (strong dipoles, ions, or even covalent bonding established after morphology control) may develop between phases, this M.W. criterion becomes much less determinant, if not unsignificant, at least above a critical value probably not higher than a few thousands.
- 6) Finally, the microstructure of the block copolymer is also an important, if more subtle, molecular parameter. First of all, a comparison of literature data has clearly put in

evidence $^{(8,9)}$ that if one wants to improve simultaneously both the strength (σ_B) and the toughness (ϵ_B) of the alloy, the efficiency of block copolymer additives increases in the order: graft < triblock z stars < diblocks, as might be expected from simple statistical free distribution considerations.

The "pure" or "tapered" character of the block is also of importance, both from rheology and ultimate properties viewpoint $^{(5,8)}$, each alloy being a case in itself.

The monomeric unit structure is finally another "tailoring" tool: e.g. PBD with very high 1.4 content will, after hydrogenation, better fit HDPE (11), while HPIP will match a propylene-rich EPDM. That hydrogenation process of dienes is a very versatile tool, since different monomers and microstructures are quite easily available: i.e. 1,2-PBD and 1,4-poly-2-methylpentadiene (12) will provide "amorphous polybutene" and "amorphous polypropylene" blocks respectively.

It may thus safely be said that these styrenics-polyolefine alloys, in addition to their interest as model blends, also represent a very attractive class of materials with desirable strength, high impact and good ageing properties.

II.EXTENSION TO OTHER MORE POLAR COMMODITY AND ENGI-NEERING POLYMERS

1. Principles

It is often very difficult, particularly on an industrial scale, to synthesize a block copolymer having practically the same structure as the corresponding homopolymers. It was thus proposed sometime ago (13) to synthesize A-C diblocks to tailor A and B homopolymer blends, granted that C would have strong enough (i.e. dipole) interactions with B. The problem was to find a kind of a "universal" additive (or family of diblocks) to achieve that goal as easily as possible. The best candidate for that C-block proved to be PMMA (highly syndiotactic, as obtained from anionic polymerization): it is indeed quite miscible with a number of important commodity and engineering polymers, the key ones being undoubtedly PVC (and other halogenated polymers) and SAN

resins. More practical synthetic ways were developed ⁽¹⁴⁾ for the synthesis of its block copolymers with styrenes, dienes, and hydrogenated dienes (see also above), thus opening the possibility to tailor blends of the above mentioned polar polymers with styrenics, diene rubbers (including Kraton's) and polyolefines. That family of diblocks was further expanded by using other methacrylates ⁽¹⁵⁾, new initiators for acrylates "living" polymerization ⁽¹⁶⁾, and hydrolysis of ter-(meth) acrylate monomeric units to yield blocks containing carboxylic units.

As an alternative to this really general purpose family of additives, polycaprolactone (PCL) block copolymers are also useful, particularly where polycarbonate is considered as a blend partner. In this case also, suitable synthetic methods have been devised $\binom{(17)}{2}$.

The basic validity of this scheme has been tested (18) and demonstrated by using a combination of two complementary sets of physical data: bulk-state NMR relaxation times and non-radiative energy transfer measurements. The data not only indicate (19) a nearly perfect mixing of PMMA with PVC at molecular scale (somewhat less efficient, although good, with SAN), but also show that the presence of a non-miscible block, i.e. PS, attached to PMMA does not significantly decrease its miscibility with PVC.

2) Application to PVC

On these bases was attempted the optimization of blends of PVC with other polymers, principally polystyrene or polydienes.

The PS-PVC alloy is an interesting model since involving two rigid partners. It is found nevertheless that PS block copolymers with either PPMA or PCL provide for a spectacular decrease in phase size, allowing casting of nice films from THF solutions, as well as very efficient mechanical blending. This is, however, a situation governed by a delicate balance of interactions, as indicated by the fact that PMMA-PS and PCL-PS copolymers largely diverge in some of their specific effects, although both provide interfacial adhesion in addition to phase size reduction (20): e.g. PMMA

copolymers have a favorable influence on Young's modulus of alloys of any composition, while PCL-PS's have a very detrimental "allergic" effect on the PS-rich composition. In this frame, it must be reminded that these two polymers have rather different solubility parameters, one higher and the other one lower than that of PVC.

The polydiene-PVC alloys have been tested as possible candidates for high impact materials, and indeed the use of P(BD-b-MMA) diblocks conferred impressive impact resistance (20) to rigid PVC loaded with PBD or Kraton (values above 100 kJ/cm², notched Charpy, were currently achieved). However, slightly higher amounts of block polymer were needed to obtain the best performances: a possible reason for that observation might be found in the TEM micrographs of these alloys showing that, although enough of the added copolymer can still be found at the interface, a sizeable proportion of it is now dispersed in the PVC matrix as its own phase (4). Obviously a better balance of relative solubility is to be engineered for these alloys.

3) Application to SAN

A tempting idea was of course to mimick ABS resins by applying the concept developed hereabove to blends of SAN with rubbers, with the hope to produce along these lines, by simple mechanical blending, high impact SAN materials of varying composition, depending upon the feed ratio of the homopolymers in the machine.

After having found that Solprene star-type — thermoplastic elastomers—were — more efficient in that respect, it was also demonstrated that, for comparable compositions—(in AN and rubber), these alloys not only displayed better gross impact strength than classical (Cycolac) ABS resins, but a definitely more favorable failure profile (21): a profile which could further be somewhat tailored by engineering the additive and the mixing process.

From these two examples (and other additional recent data), it can be rationalized that the use of properly tailored block copolymer additives, together with specific rubbers, can help generating super-high-impact (SHI)

materials: this could be one of their most important applications, particularly when dealing with systems where other components or conditions significantly deteriorate the impact resistance.

This is an important point since it is clear that multicomponent blends can be, and will be, more and more the subject of these molecular engineering optimization procedures. As examples, one can cite the tailoring of ternary blends involving PS, polyolefines and PVC(i.e. the 3 main components of the plastic wastes), and the integration of fillers in polymer alloys (see Sections 4.1 and III.).

4) Application to Engineering Polymers

Poly(vinylidene fluoride) (PVDF)

Another attractive idea is the possibility to fill a very expensive engineering polymer matrix with a good amount of a definitely cheaper polymer or/and of a common inorganic filler. That might lead to some improvement in the set of properties together with more favorable economics of those high performance materials, provided they remain as the continuous phase and develop (thanks to the additive) a good adhesion with the filling phase. This approach has been put at work (22) in different allows of PVDF with polyolefines, poly- α -methylstyrene (PMS), or Noryl, using PMMA block copolymers with hydrogenated polydienes, PMS, or PS, respectively, since PMMA is well miscible with PVDF amorphous phase. The results are particularly comforting with Noryl (in terms of both $\boldsymbol{\sigma}_B$ and $\boldsymbol{\epsilon}_B$ of the alloys) despite the need for somewhat higher amounts of block copolymer when a very fine dispersion is required. Furthermore, some inorganic fillers, i.e. CaCO2, could also be integrated in the resulting alloys.

- Nylons

Of course, blends of some nylons with polystyrene or polyolefines can also be tailored this way, since the corresponding nylon 6 block copolymers are available (23). However, a general approach was desirable, that should be both easier and more versatile. An interesting answer to that challenge is the use of a C block carrying carboxylic groups (24), introduced either by carboxylation of a diene block

(maleic anhydride grafting) or by hydrolysis of t-butyl-(meth) acrylate units. These carbonyl groups interact very strongly with the amide, with which they even can eventually react by transamidification under proper conditions, so forming covalent bonds between the block copolymer and the polyamide phase. Anyhow, these strong interactions are sufficient to induce a fine sub-micron phase dispersion and an excellent interfacial adhesion (4), paving the way for very interesting semicrystalline - amorphous alloys.

III. EXTENSION TO COMPONENTS OTHER THAN POLYMERS

In the general scheme where an A-C block copolymer binds together two unmiscible phases A and B, it was of course another challenge to replace B by an inorganic filler. The molecular adhesion of a filler phase to the polymer matrix is not indeed a completely well-mastered problem: usually, a satisfactory adhesion is provided only by delicate and costly pre-treatments of the charge (25), and it would be most desirable that an additive were able to more or less fulfil that role during the processing of the alloy.

Again, it was expected that under usual processing conditions (high shear and temperature), a C block carrying electric charges (or functional groups highly reactive towards the filler) might ensure a sufficient interaction with that filler so as to disperse it and make it adhere to the polymer matrix A, through interaction of the A block with that matrix.

A clear example of these potentialities is given in fig.2, comparing the fracture SEM micrographs of a CaCO₃-LDPE blend with and without a copolymer containing a HPBD block bonded to another one carrying carboxylic groups, and showing greatly improved dispersion and adhesion.

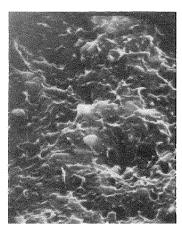




Fig. 2: Phase Adhesion as seen by SEM in LDPE/CaCO₃ Alloys Containing (left) or not (right) a Diblock³ Copolymer (HPBD - Polycarboxylic acid)

That approach is of course very versatile and extrapolable to other charges such as ${\rm Al}_2{\rm O}_3$ and ${\rm Al}\left({\rm OH}\right)_3$; inversely, interaction of a block containing basic groups with an acidic-type charge is also possible (e.g. vinylpyridine units with acidic silica).

Another very promising field is the <u>stabilization of</u> fine insoluble polymer dispersions in liquid matrix, wherein the A block interacts with the dispersed polymer (physically or even by chemical bonding), while the C block creates a coalescence barrier around the polymer particles. Since the pioneering work of Riess $^{(26)}$ in this field, interesting applications have been found both in organic and in aqueous media $^{(15)}$.

IV. OTHER APPROACHES

From what has been said above, it is obvious that any type of strong interaction developing at the phases interface will be able to achieve the two fundamental goals of anyone interested in tailoring the properties of polymer alloys. Block copolymers, interacting or reacting spontaneously with the homophases to be alloyed, represent a very versatile and interesting tool to achieve these goals, playing the molecular engineering game with entanglements, as well as hydrophobic, dipolar and electrostatic forces, in going from non-polar to polar and even charged components for the alloy. However, other approaches can also be combined with, or even substituted for, that use of block copolymers.

In the first place indeed, one may wish to freeze-in definitivelly the interesting morphologies which can be developed and stabilized with the help of these block additives. The interfacial reaction of suitable groups located on different phases is of course one way to achieve that purpose, since there is a great development of the interface in these fine dispersions, but it usually raises the chemical and economical problem of previously grafting these groups, often on low reactivity polymers. This is, however, a sometimes efficient approach $^{(15)}$, particularly when using reactive group located on the block copolymer itself. When applicable, γ -irradiation is probably the best and easier tool to definitively stabilize at least one, if not all, phases involved.

On the other hand, <u>block copolymers (linear or graft)</u> can be <u>generated "in situ"</u> in a number of cases. Although generally less efficient than diblock ones, graft copolymers are usually easy to generate in a standard polymer processing unit (30), hence the great industrial interest for this particular reactive processing approach.

Finally, interactions developed by other means than block copolymers can also be used. As a potential example of that, immiscible telechelic polymer blends have been investigated, in which component A is an α , ω -diacidic polymer (carboxylic or sulfonic), while component B is an α , ω -dibasic one, terminated for instance by hindered secondary amine groups, a typical pair involving PS and PIP. It appears that, contrarily to situations where non-functional or monofunctional polymers are used, the salt formation between different

end-groups promotes again a stable sub-micron phase dispersion with good interfacial adhesion $^{(27)}$, in agreement with results previously obtained by Eisenberg $^{(28)}$ on polyfunctional polymers acting as ionomers.

AS A SHORT CONCLUSION, it may be claimed that this molecular engineering of the bulk properties of polymer alloys, particularly when using block copolymers, represents a very efficient tool for a tremendous broadening and diversification in the use of available classical polymers.

The creation along these lines of complete families of new, higher performance materials is certainly a very worthwhile scientific, technological and economical goal in these times of penetration of polymer-based products and systems in practically all areas of human activity.

Acknowledgment

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POLYMER BLENDS - CURRENT STATE OF PROGRESS AND FUTURE DEVELOPMENTS FROM AN INDUSTRIAL VIEWPOINT

Hans Rudolph

Bayer AG, Research and Development, 5090 Leverkusen, Germany

Abstract - Technical and economic considerations have given polymer blends a major share in the increasing sales of plastics. This applies both to general-purpose and to the higher value-added materials. Thus it is scarcely surprising that almost all polymer manufacturers have now developed comprehensive ranges of blends having particular property profiles. The principal types of blends currently available are described.

The variety of products already developed should not be allowed to obscure the fact that certain conceivable and highly attractive property combinations have not so far been realized. If progress is to be made in this area, interdisciplinary cooperation between chemists and physicists in the fields of polymer chemistry, polymer physics and materials science is essential.

TNTRODUCTION

The significance of materials for human society is underlined by the fact that the various ages of our history have been named after the material used at the time:

the Stone Age, the Bronze Age and the Iron Age.

We would be oversimplifying things for ourselves if we were to refer to the present time as the "Polymer Age". One thing, however, is certain: there is no way of stopping the upward trend in the consumption figures for plastics (Fig. 1); metals are losing ground to plastics right across the board. The process is accompanied by occasional setbacks but they do not have a fundamental influence on it.