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Morphology and properties of polyurethane-based blends

GIOVANBATTISTA DEMMA, EZIO MARTUSCELLI
Istituto di Ricerche su Tecnologia dei Polimeri e Reologia, C.N.R., Via Toiano, 2 – Arco Felice, Napoli, Italy

ALVISE ZANETTI, MAURO ZORZETTO
Nordica S.p.A., Via Montebelluna – Trevignano, Treviso, Italy

The morphology and the properties of injection-moulded samples of thermoplastic polyurethane-based blends have been investigated as function of composition and chemical nature of the second component (ABS, PS, ASA and SAN). The examination of the overall morphology shows that in the blend specimens the minor component forms separated domains. This evidence is gained using etching techniques in the case of ABS-, ASA- and SAN-containing blends while for TPU/PS the domains of PS are directly visible by SEM on the surface of fractured samples. Dynamic mechanical investigation supports the morphological findings. In the case of TPU/ABS blends it has been found that on increasing the ABS content, materials are obtained with a higher flexural modulus and Shore hardness but with a reduced density. On the other hand, TPU/ABS blends show a reduction of the elongation at break and the tensile strength. The impact behaviour of TPU/ABS alloys depends on temperature and composition. At low temperatures ABS acts as a toughening agent. At higher temperatures ABS acts as reinforcing agent. The influence of processing conditions on the morphology and properties of TPU/ABS blends is also discussed.

1. Introduction

The formulation of polyurethane-based alloys has developed over the past years as a means of improving some properties and/or reducing manufacturing costs. Polyurethane-based alloys generally contain a rigid thermoplastic polymeric material (ABS, PVC and SAN) as a second component. For example, Hooker's Ruco (Rucothane Division for the elastomer line) has put on the market a family of materials consisting of polyurethane (TPU), PVC and ABS combinations. From a cost/performance point of view, these alloys are competitive with pure TPU and with other elastomeric materials which are processed by injection moulding [1].

According to Howe [2], ABS may be used as a second component to reduce the costs of polyether-based TPU without deteriorating their properties in any significant way.

The Termofil Inc. Industry produces a TPU/ABS alloy, by injection moulding, which is used for manufacturing goods such as take-ups for cinematographic projectors. These alloys have a very good resistance to abrasion [3]. Generally the addition of a thermoplastic polymeric component with a high modulus (PVC, SAN, ABS) to an elastomeric TPU matrix results in an improvement of the tensile/tear strength and of the rigidity. Furthermore, it also results in an increased resistance to abrasion and a reduction of the "cut-growing" and "flex-cracking" phenomenon.

Very little information is found in the literature on these systems and no systematic studies have been published with the objective of correlating the processing variables and the composition with the morphology on one hand and properties on the other. In the present paper we report a study concerning the influence of the composition and

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TABLE I Polymers used in this research as blend components and their characteristics

Polymers	Code	Density ($g\,cm^{-3}$)	Tensile strength ($kg\,cm^{-2}$)	Elongation at break (%)	Tensile modulus ($kg\,cm^{-2}$)	Melt index 200° C, 5 kg (g/10 min)
Polyurethane (Desmopan from Bayer)	TPU	1.24	570	535	1 000	–
ABS resin (Novodur P.K. from Bayer)	ABS	1.05	350	30	20 000	5
Polystyrene (Edistir- from Montedison)	PS	1.05	500	3	32 500	9
Styrene–acrylonitrile copolymer (Luran from BASF)	SAN	1.08	750	5	36 000	0.7
Acrylonitrile–styrene–acrylic ester terpolymer (Luran-S- from BASF)	ASA	1.07	440	20	23 000	8

the processing conditions on the mechanical properties (tensile, flexural, impact) and on the overall morphology (modes and states of dispersion of the dispersed phase) of injection-moulded samples obtained by mixing a polyester-based thermoplastic polyurethane (low modulus type) with an ABS resin. For comparison, blends of TPU with polystyrene, styrene–acrylonitrile copolymer (SAN) and with an acrylonitrile–styrene–acrylic ester terpolymer (ASA) were also investigated.

2. Experimental details

2.1. Materials, mixing and processing conditions

A commercial low-modulus polyester-based polyurethane (Bayer, Desmopan) based on diphenylmethane diisocianate (MDI), poly(ethylene glycol) adipate and butandiol as chain extender, was used throughout. The polymers used as the second component together with their characteristics are listed in Table I. The composition of ABS was: acrylonitrile 20% (wt/wt); styrene 54% and butadiene 26%. The following standard procedure for the preparation of the blend samples was used:

(1) chips of the two components were weighed in their exact proportions and mechanically mixed;

(2) this mechanical mixture was used to feed a Sandretti 270 injection-moulding press.

For TPU/ABS mixtures a set of sheets were obtained by using a processing temperature of 195° C (i.e., the temperature of the fluid mass) and another set by using a processing temperature of 225° C. Blends of TPU with ASA, PS and SAN were all processed with the fluid mass temperature of 195° C.

All experimental tests were performed on samples directly obtained by blanking the injection-moulded sheets previously submitted to post-curing treatment. This treatment consists in keeping the moulded sheets in a thermostatic oven for 12 h at 80° C.

2.2. Mechanical characterization

The experimental conditions used for tensile and flexural mechanical tests and for abrasion-loss measurements are reported in Table II. For the mechanical tensile and flexural tests an Instron Mod. 1026 Dynamometer was used with cells from a 50 to 500 kg load and from 5 to 50 kg, respectively. Flexural tests were performed to measure the tangent modulus of elasticity often called the "modulus of elasticity" defined as the ratio, within the elastic limit of stress, to corresponding strain. It is calculated by drawing the tangent to the steepest initial straight-line portion of the load–deflection curve.

Complex shear moduli (G' and G'') and damp-

TABLE II Experimental conditions for tensile and flexural mechanical tests and for abrasion loss measurements

Tensile tests	
ASTM method	638
Specimen (shape and dimensions according to ASTM)	412/C
Cross-head speed	$100\,mm\,min^{-1}$
Temperature	23° C
Flexural tests	
ASTM method	D 790
Specimen dimensions	25 mm × 80 mm × 3 mm
Speed	$50\,mm\,min^{-1}$
Temperature	23° C
Abrasion loss	
According to DIN	53516 Method
Temperature	23° C

ing were measured using a torsion pendulum (DIN 53445) equipped with a temperature chamber. The temperature dependence of the moduli was measured, according to ASTM DIN 53445 NORM (the frequency varies from 0.1 to 10 Hz) within the range − 140 to + 200° C (the dimensions of the sample were 2 mm x 8 mm x 50 mm).

Samples were initially quenched from room temperature to approximately − 150° C and then heated at a rate of 1° C min^{-1}.

2.3. Morphological characterization

The morphology of the TPU/ABS alloys was investigated by optical (OM) and scanning electron microscopy (SEM) techniques. The OM investigation was effected on sections treated with an osmium tetroxide (2%) water solution for about 24 h. For SEM observations the following standard procedure was followed: the sheet samples, with a thickness of roughly 3 mm, were immersed in liquid nitrogen for 10 min and then broken. The fractured surfaces were successively etched with methylethylketone (4 h at room temperature) good solvents of styrene-acrylonitrile copolymers contained in ABS [4] resin. Alternatively the fractured surfaces were etched with tetrahydrofuran vapour (for 1 h), a good solvent of the TPU phase [5].

After this treatment the samples were dried under vacuum for about 24 h. Before SEM observation, the fractured surfaces were metallized with Au–Pd alloy. A scanning electron microscope (Philips model 501) was used.

The same procedure was followed in the case of TPU/SAN and TPU/ASA blends. The fractured surfaces of TPU/PS blends were examined by SEM without prior etching.

2.4. Impact tests

The impact resistance of TPU/ABS samples was investigated, as a function of the composition and temperature, by using a CEAST fracture pendulum, equipped with a low-temperature device, connected to an advanced fractoscope registration system. The impact tests were performed according to the Izod method (ASTM D 256 Rule) on specimens with the following dimensions: 3 mm x 13.2 mm x 50 mm.

The Izod impact tests were performed at room temperature by using specimens with a 1.00 ± 0.05 mm deep notch whereas for tests at lower temperatures specimens with a 2.50 ± 0.05 mm deep notch were used. For each composition and temperature at least eight specimens were fractured. The impact behaviour of TPU/ABS blends was investigated at the following temperatures: room temperature (about 20° C), − 10° C, − 30° C and − 50° C.

3. Results and discussion

3.1. Mechanical properties

3.1.1. Abrasion loss–density

From the analysis of stress–strain curves of TPU/ABS blends the following conclusions may be reached:

(1) only alloys containing a high percentage of ABS present a clear phenomenon of yielding. At the same time an increase in the initial elastic tensile modulus is observed when the ABS percentage increases (see Fig. 1);

(2) both the tensile strength and the elongation at break monotonically decreases when the ABS percentage increases (see Fig. 2a and b, respectively).

Fig. 2a shows that for the same blend composition samples moulded with a fluid mass temperature of 195° C, yield higher tensile strength values than those moulded with the fluid mass at 225° C. The same behaviour is observed for the elongation at break of alloys with a percentage of ABS higher than 30%.

TPU/ABS (80/20) alloys show elongation at break values which do not depend on the temperature of the fluid mass whereas the 90/10

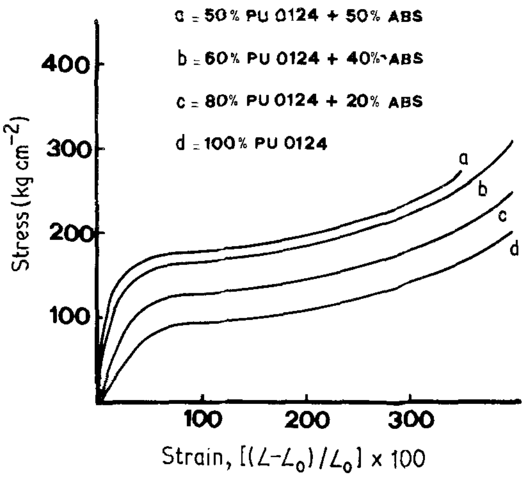


Figure 1 Stress–strain plots of TPU and TPU/ABS blends (room temperature). The experiments were performed up to a strain of 400%. Therefore, the end points of the curves do not represent the rupture of samples.

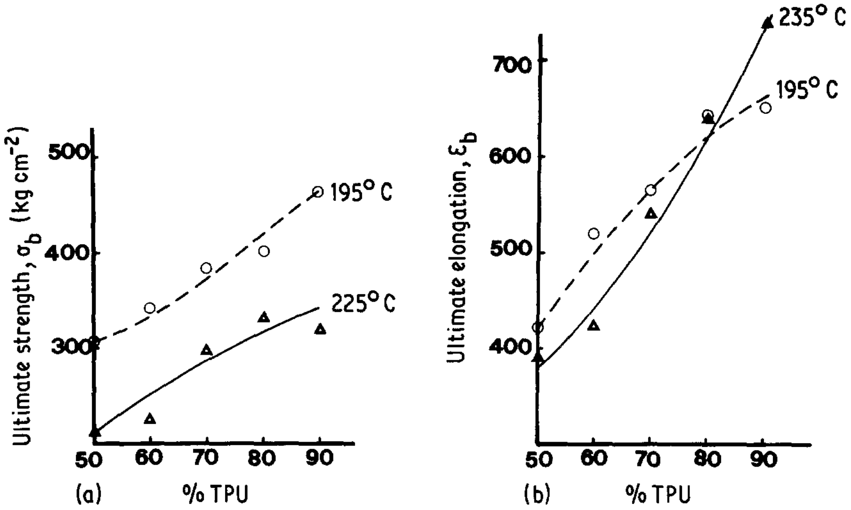


Figure 2 Dependence of ultimate strength (a) and ultimate elongation (b) on composition in TPU/ABS blends. The curves refer to two different moulding temperatures: 195 and 225° C.

alloys moulded at 225° C show a higher elongation at break than that moulded at 195° C. This tensile behaviour is probably related to thermal degradation phenomena that may occur when the TPU/ABS blends are processed at 225° C.

At the moment, we are unable to understand why alloys with low ABS content (20%), moulded with a fluid mass temperature of 225° C, show elongation at break values higher than those specimens moulded with the mass at 195° C. The elastic flexural modulus of TPU/ABS alloys monotonically increases with the percentages of ABS (see Fig. 3).

The mixtures 90/10 have moduli of 1100 kg cm^{-2} whereas 50/50 blends have moduli of 4100 and 4700 kg cm^{-2} for processing temperatures of 195 and 225° C, respectively. Thus passing from a 90/10 mixture to a 50/50 mixture an increase of over 300% is observed.

As shown in Fig. 3 there are no substantial differences between the values of the elastic flexural modulus measured on specimens moulded at temperatures of 225 and 195° C. The slight differences are in the range of experimental error.

Fig. 4a and b shows the composition dependence of density and Shore hardness of TPU/ABS alloys. The density diminishes linearly as the ABS percentage increases, whereas the Shore hardness increases monotonically. The moulding temperature does not seem to influence the density values, whereas it influences the Shore hardness only at low ABS concentrations. In the case of TPU/ABS mixtures the abrasion loss, as illustrated in Fig. 5, seems to increase with the ABS percentage.

3.2. Impact resistance

Under our experimental conditions TPU/ABS specimens submitted to fracture at room temperatures do not break completely. In these specimens a stress-whitening region is observed, due to the formation of multiple crazes that propagate along the fracture surface, together with a transverse

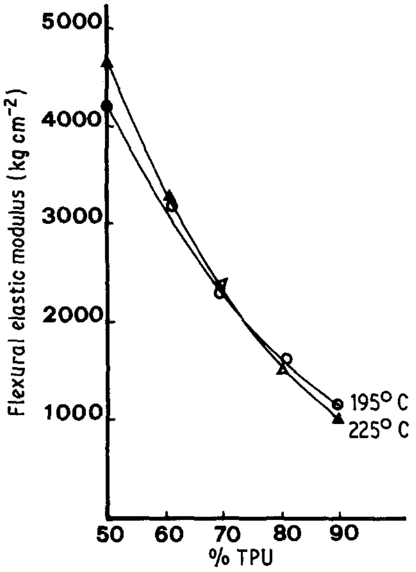


Figure 3 Variation with the composition of the elastic flexural modulus of TPU/ABS blends injection-moulded at 195 and 225° C.

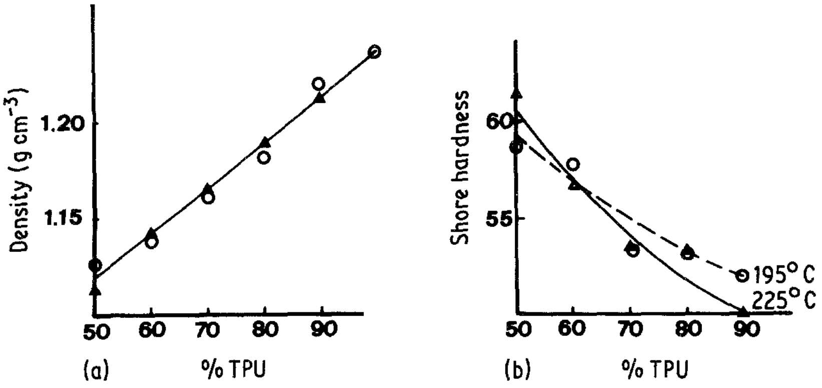


Figure 4 Density and Shore hardness of TPU/ABS blends as function of TPU content. Moulding temperature: ▲ 225° C ⊙ 195° C.

contraction. In the tests effected at – 10° C some samples break and others do not, whereas in the tests at – 30 and – 50° C all the samples undergo a complete fracture. It was possible to obtain accurate Izod impact strength values only on completely fractured samples.

Fig. 6 illustrates the impact strength dependence of TPU/ABS alloys as a function of composition for the two temperatures at which it was possible to effect the measurements. In the samples which were not completely broken, examined at room temperature and at – 10° C, a qualitative indication of their impact resistance may be obtained by measuring the depth of the fractures (L_1) and the depth of the area of formation of crazes (whitening areas) (L_2). L_2 is always greater than L_1. For the same test conditions a smaller impact resistance is expected for those samples which present higher L_1 and L_2 values.

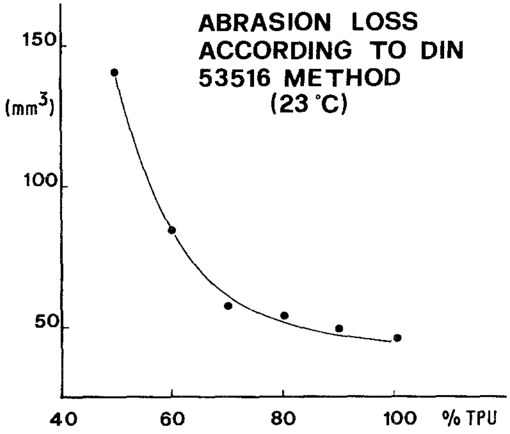


Figure 5 Abrasion loss in TPU/ABS blends moulded at 195° C, as a function of TPU content.

Therefore, as a quantity indicative of impact resistance we have used the ratios $1/L_1$ and $1/L_2$. The values of such quantities are shown as function of composition in Fig. 7. From the data in Figs. 6 and 7 it emerges that:

(1) at room temperature and at – 10° C the impact resistance of the alloys decreases when the ABS percentage increases,

(2) at temperatures – 30 and – 50° C the addition of ABS causes, on the other hand, a notable increase of impact strength values.

This behaviour may be accounted for if the glass transition temperatures of blend components are taken into consideration. In fact at – 30 and – 50° C the polyurethane matrix is at a temperature which is already below that of the T_g of the soft polyester segments [6] whereas the polybutadiene part of ABS is still rubbery (T_g about – 100° C) [7] and therefore the polybutadiene present in ABS as graft or free homopolymer acts as a toughening agent. At room temperature the ABS resin acts as a reinforcing agent due to the presence of the grafted or free SAN copolymer

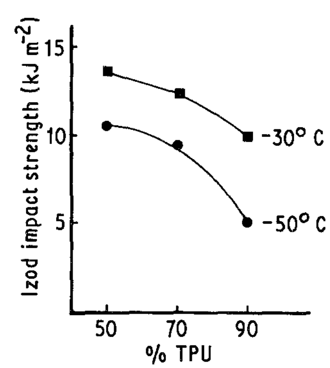


Figure 6 Izod impact strength of TPU/ABS blend as a function of TPU content at two different temperatures.

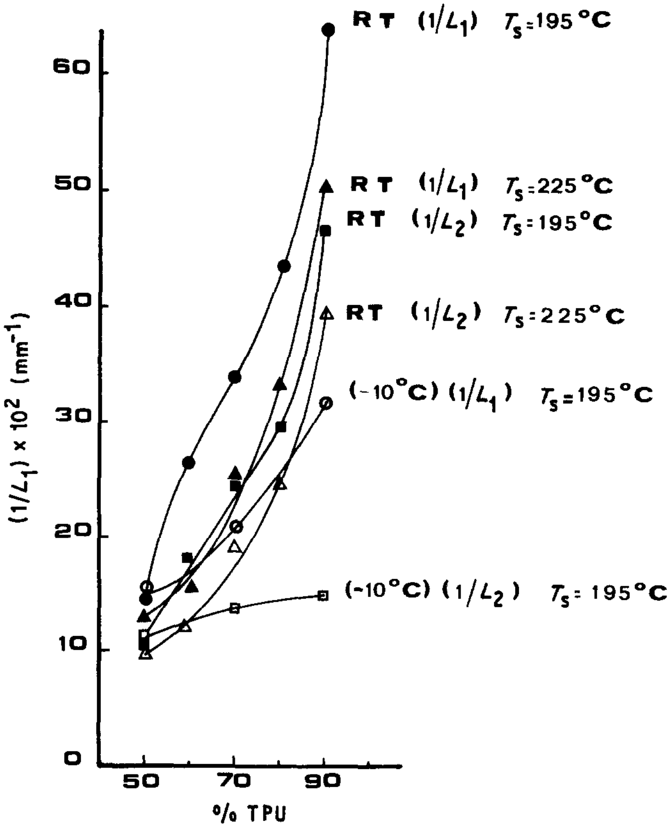


Figure 7 Variation of $1/L_1$ and $1/L_2$ with composition in TPU/ABS blends. The moulding temperature T_S and the temperature at which the tests were effected is given on the curves.

which is characterized by a high T_g value (about 100° C).

As shown in Fig. 7 the processing condition seems to influence the impact resistance of TPU/ABS blends. In particular it may be observed that blends processed at a lower temperature of the fluid mass seem to show a better impact resistance. From the data of Fig. 7 it is interesting to point out that the processing conditions have little effect on the impact properties of the 50/50 blends. The influence of processing conditions on the impact resistance of blends may be probably accounted for if it is considered that following the injection moulding process, flow lines are generated in the moulded sheets which induce particular anisotropies and orientations in the material. In various literature papers [8] it has been found that the impact energy absorbed by an injection-moulded specimen is greater or smaller according to whether the impact direction of the hammer is, respectively, perpendicular or parallel to the flow lines.

The TPU/ABS sheets have been hit so as to have the components of the flow lines perpendicular to the impact direction rather than parallel to it.

On the basis of these considerations, the lower impact resistance of samples moulded at higher temperatures could simply be a consequence of the fact that in these conditions, owing to the greater fluidity of the material, the sheets are more isotropic.

Degradation phenomena, at higher temperatures, probably should also be considered in the interpretation of these effects.

3.3. Morphological studies

On examining non-stained thin sections of TPU/ABS alloys by optical microscopy the materials appear homogeneous. From the optical micrographs, the sections treated with an osmium tetroxide–water solution for 24 h, show distinct dark domains. The latter should represent areas richer in polybutadiene (present either as an homopolymer or as a grafted copolymer). These domains are uniformly distributed all over the surface of the section suggesting that the materials have a good degree of dispersion.

No distinct domains of the second component are visible on the cryogenically fractured surfaces of TPU/ABS, TPU/SAN and TPU/ASA mixtures when they are observed by SEM. However, after

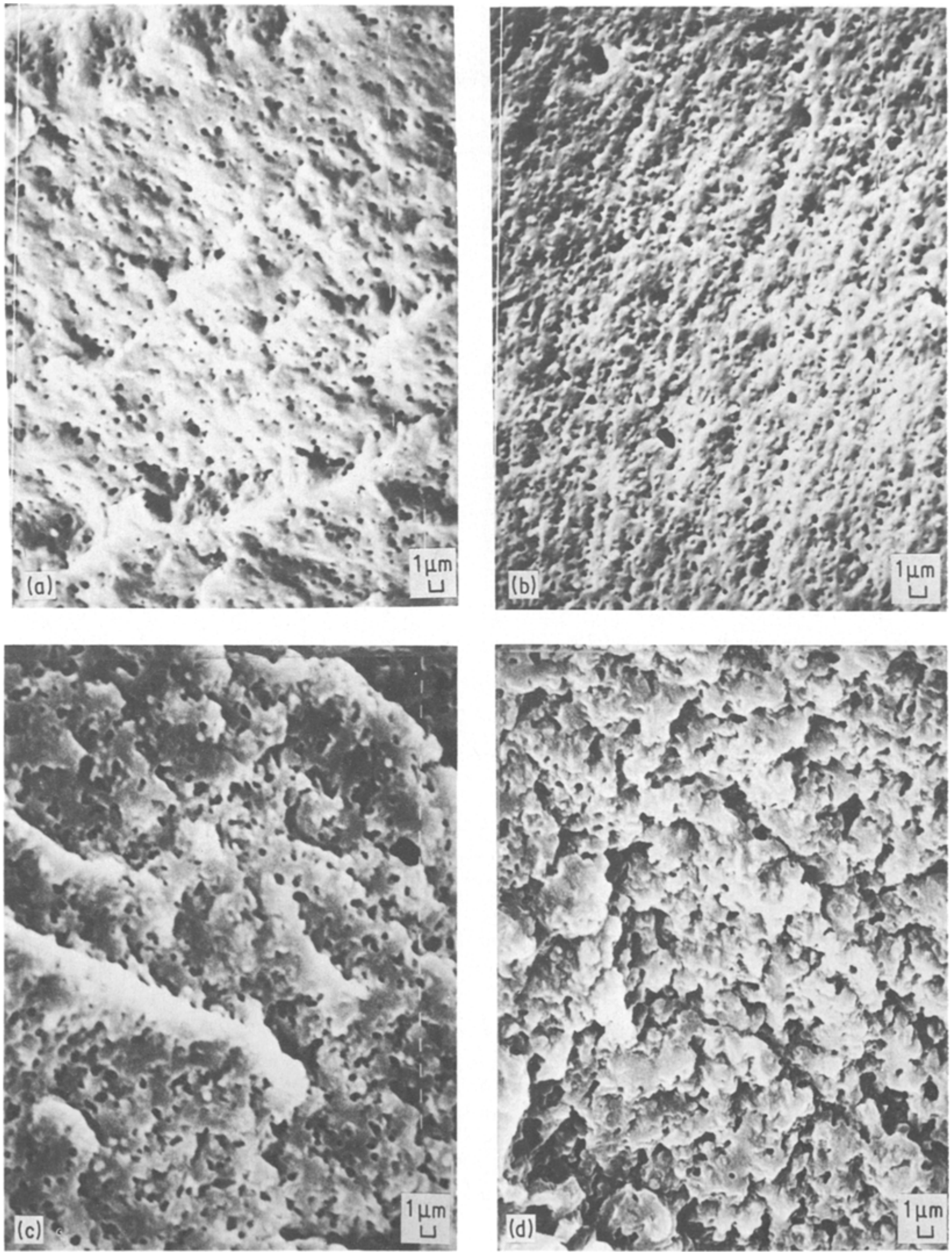


Figure 8 Scanning electron micrographs of cryogenically fractured surfaces of TPU/ABS blend specimens after etching with MEK. (a) TPU/ABS (90/10); (b) TPU/ABS (80/20); (c) TPU/ABS (70/30); (d) TPU/ABS (60/40).

etching with methylethylketone, the fractured surface of such blends show spherical cavities homogeneously distributed all over the surface. These cavities are caused by the solubilizing action of MEK which removes the free styrene–acrylonitrile copolymer from the surface. Owing to a mechanical effect it is also possible that a part of the grafted copolymer is removed.

The density of the cavities and the average dimensions increase when the ABS percentage

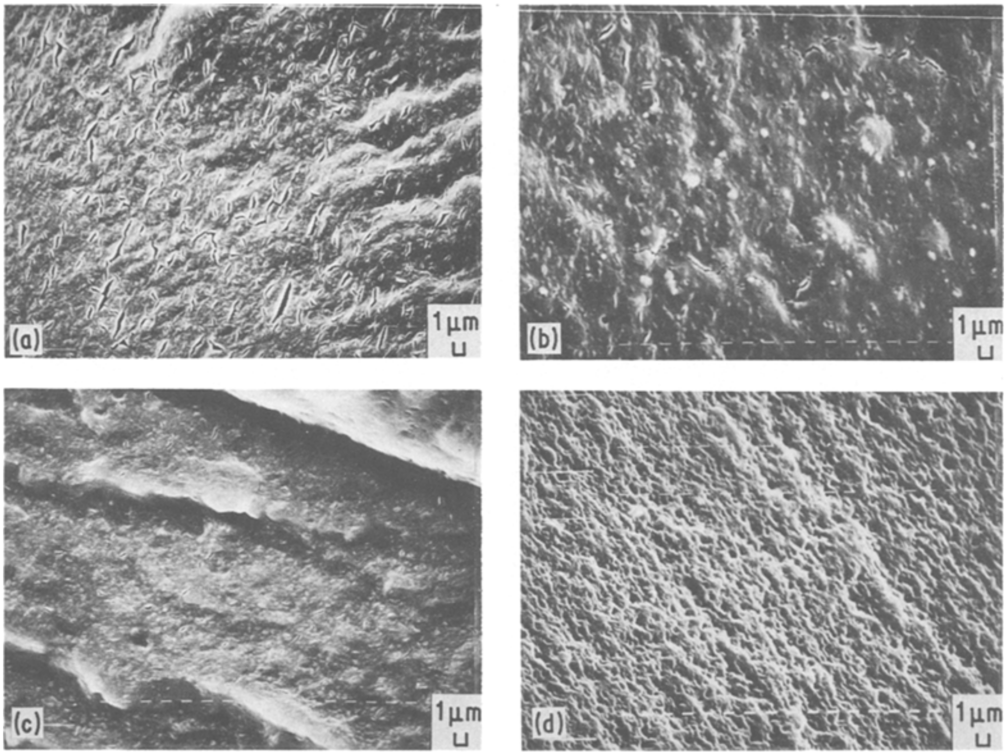


Figure 9 Scanning electron micrographs of cryogenically fractured surfaces of TPU and TPU/ABS specimens after etching with THF vapour. (a) TPU; (b) TPU/ABS (90/10). (c) TPU/ABS (80/20); (d) TPU/ABS (70/30).

in the alloy increases. At a higher ABS content the surfaces show an interconnected morphology (see scanning electron micrographs of Fig. 8).

The fractured surfaces which are not treated with MEK show no evidence of ABS domains which are separated from the matrix.

As shown in the electron micrographs of Fig. 9 the surface fracture of TPU samples subjected to warm tetrahydrofuran vapour show signs of cracks or crazes. Similar crazes are also visible on the surface of THF-treated samples of low ABS content TPU/ABS mixtures but with notably lower density and dimensions (see Fig. 9b and c). The surfaces of TPU/ABS specimens with a higher percentage of ABS, treated with hot THF vapour show a cellular morphology that according to the composition is more or less regular (see Fig. 9d). Furthermore, the presence of crazes is not observed therein.

These observations lead us to the interesting conclusion that it is possible to increase the TPU resistance to formation of crazes in a corrosive environment, that is, an environment created by hot THF vapour, by adding a certain amount of ABS.

The morphology developed in cryogenically fractured surfaces of TPU/SAN and TPU/ASA 70/30 blends after etching with methylketone is shown by the scanning electron micrographs of Fig. 10.

The examination by SEM of the cryogenically fractured surface of TPU/PS 70/30 blends reveals the presence of separated spherical shaped PS domains together with cavities. Both the surface of the cavities and the PS domains are smooth denoting low adhesion between the matrix and the dispersed phase (see Fig. 11).

Finally, it may be concluded that the preparation conditions of TPU/ABS mixtures are sufficient to obtain a fine dispersion of components even though they are incompatible from a thermodynamic point of view. The fact that the domains of the disperse phases are not directly visible on the surfaces of the cryogenically fractured samples of TPU/ABS, TPU/ASA and TPU/SAN blends is probably related to the high adhesivity at the interphase between these domains and the TPU matrix.

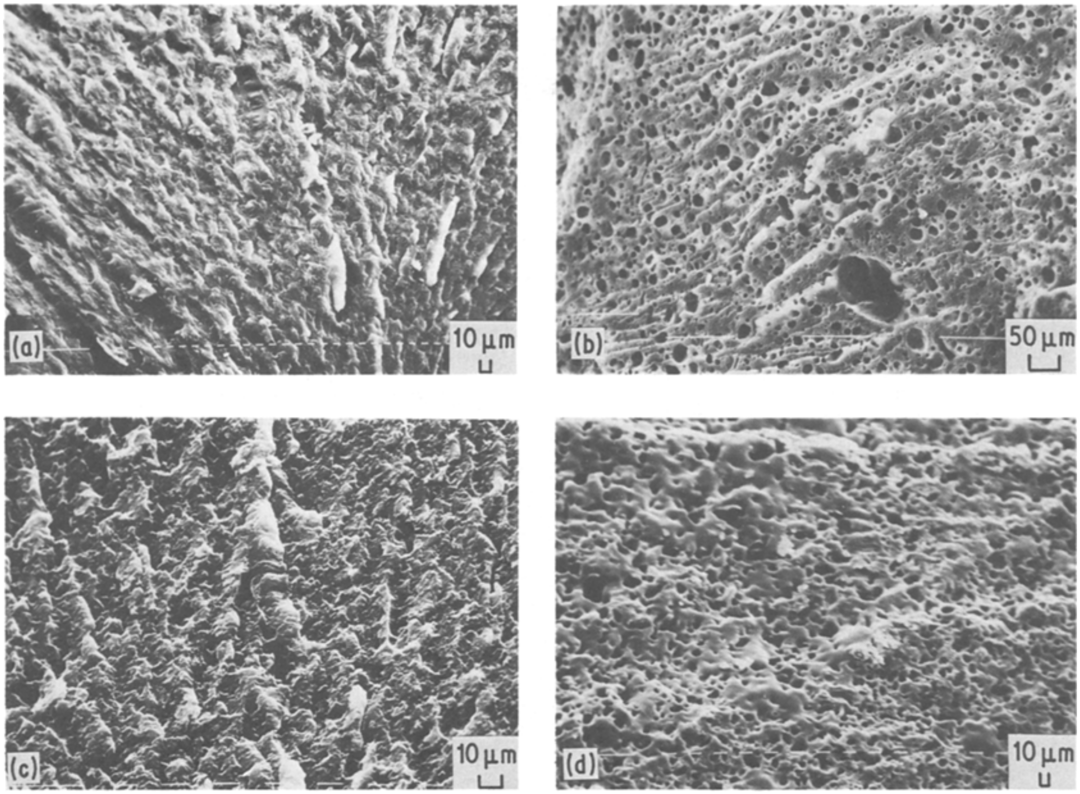


Figure 10 Scanning electron micrographs of cryogenically fractured surfaces of: (a) TPU/SAN (70/30); (b) TPU/SAN (70/30) after etching with MEK; (c) TPU/ASA (70/30) blend; (d) TPU/ASA (70/30) after etching with MEK.

3.2. Fractographic analysis

In order to clarify the influence of temperature and composition on the fracture mode of TPU/ABS blends, a fractographic investigation was performed on the Izod impact fractured specimens.

At a high temperature $\geqslant -10^\circ$ C the specimens did not break. A stress-whitening phenomenon around the notch tip, together with a transversal contraction was observed. The specimens at these temperatures suffered macroscopic permanent deformation i.e. they yielded.

Composition seems to influence this type of fracture, as the depth of crack propagation within the whitened region increases with the percentage of ABS in the blends. Scanning electron micrographs of failure surfaces of Izod impact specimens (at -10° C) are shown in Fig. 12. As can be seen, the blends show failure surfaces with distinct tear

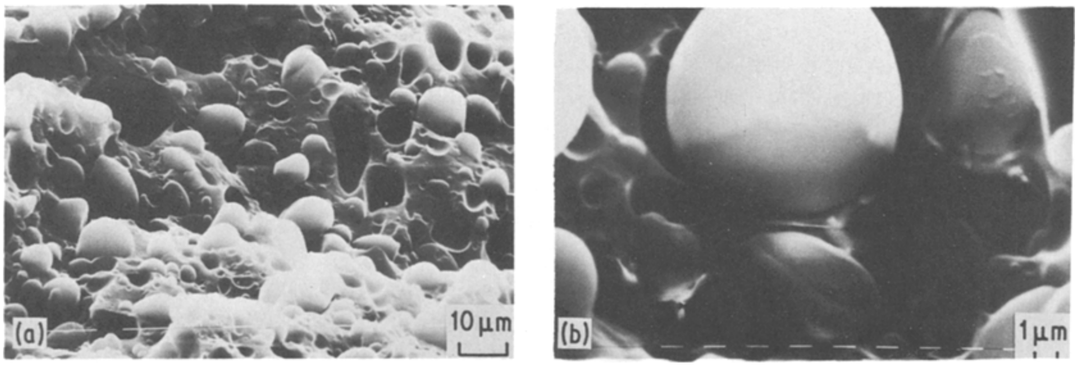


Figure 11 Scanning electron micrographs of cryogenically fractured surfaces of TPU/PS blend specimens.

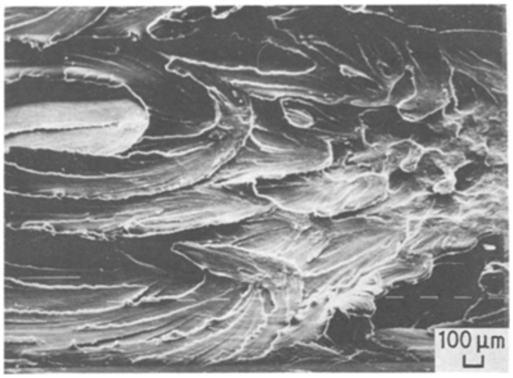


Figure 12 Scanning electron micrograph of fractured surface of TPU/ABS (70/30) blend specimen after Izod impact tests effected at – 10° C.

lines extended all over the broken area and a considerable amount of transverse contraction. The amount of material plastically deformed on the failure surface seems to decrease with the percentage of ABS. No evidence of a distinct induction region is observed.

From the above it may be concluded that TPU/ABS blends at a high temperature ($\geqslant -10^\circ$ C) show a ductile fracture mode characterized by the co-existence of a shear yielding process and multiple craze formation. At lower temperatures (– 30 and – 50° C) the fractured surfaces of specimens appear completely brittle. The specimens broke into two pieces with no evidence of stress-whitening, permanent macroscopic deformation or yielding.

Examination of the failure surfaces of these specimens by SEM reveals the presence of a small region localized near the centre of the notch where the failure initiates (see Fig. 13). In the immediate vicinity of this induction zone the broken surfaces show a macromorphology that is much more disturbed than that of regions far from the notch line.

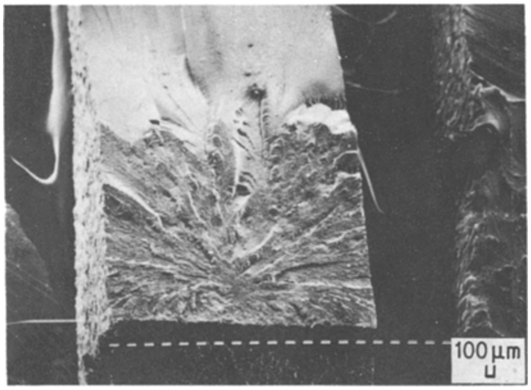


Figure 13 Scanning electron micrograph of fractured surface of TPU/ABS (90/10) blend specimens after Izod impact tests effected at – 30° C.

3.5. Dynamic mechanical properties

Fig. 14 shows the shear modulus, G', and the mechanical damping, $\tan\delta$, as a function of the temperature for pure TPU and TPU/ABS blends. The loss modulus, G'', is shown in Fig. 15.

In the temperature range explored (– 60 to + 150° C) TPU has one peak at about – 10° C in the plot of loss modulus G'' against temperature. This peak corresponds to the glass transition temperature of flexible polyester segments [7]. In the vicinity of this glass transition an abrupt drop in the shear modulus, G', and a maximum at about + 10° C in the mechanical damping, $\tan\delta$, are observed. The plots of G', G'' and $\tan\delta$ against temperature for TPU/ABS blends are much more complicated.

Three peaks are observed in the G''-temperature plots: at about – 85, – 30 and + 100° C. Corresponding to these temperatures the shear modulus, G', drops and the dissipation factor, $\tan\delta$, goes through maxima. As can be seen in Figs 14 and 15, the intensity of these transitions is composition dependent. In fact, the height of the G''-temperature plot peaks and the area and sharpness of the $\tan\delta$–temperature plot maxima at – 85 and + 100° C increase with the percentage of ABS, whereas the opposite trend is found for the transition at – 30° C. The transition at – 85° C corresponds to the glass transition of the polybutadiene phase, whereas that at + 100° C corresponds to the glass transition of the SAN phase.

The dynamic mechanical behaviour of TPU/ABS blends indicates that TPU and ABS components are almost incompatible. The shift of the soft segment glass transition temperature (from – 10° C for pure TPU to about – 30° C for TPU/ABS blends) observed on blend specimens suggests that probably a certain amount of low molecular weight polybutadiene molecules may act as a plasticizer for TPU.

Fig. 16 shows the shear modulus, G', and the dissipation factor, $\tan\delta$, plotted against temperature for TPU/SAN, TPU/ASA and TPU/PS (70/30) blends. The temperature dependence of loss modulus G'', corresponding to the same blends, is shown in Fig. 17. Examination of the plots indicates the presence of distinct transitions at temperatures corresponding to the glass transitions of the components. Thus, it may be concluded that TPU is incompatible with materials such as SAN, PS and ASA.

These observations agree with the results of the morphological investigations.

4. Conclusions

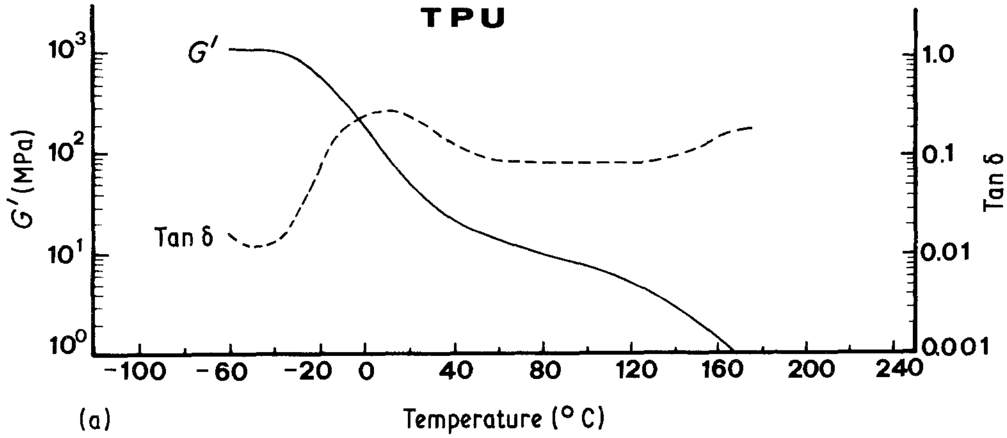
The addition of ABS to TPU causes substantial modification of the properties of the polyester-based thermoplastic polyurethane (low-modulus type).

With the addition of ABS, materials are obtained which show an increased flexural modulus and Shore hardness but a reduced density. Thus it is possible to process items with an increased rigidity having the same volume and lower weight, with respect to those produced with TPU only.

TPU/ABS mixtures have some disadvantages, i.e. the reduction of the elongation at break and the tensile strength. These disadvantages, however, may be maintained within certain acceptable limits if the ABS content is not too high.

The impact properties of the TPU/ABS alloys depends on the temperature at which the tests are performed. At low temperatures (<– 30° C) ABS acts as a "toughening" agent and the impact resistance of the mixture increases with the ABS percentage. At higher temperatures ABS simply acts as a reinforcing agent and consequently the impact reistance decreases when the ABS content increases. The sense of the change in properties when ABS is added to TPU depends on the composition and temperature.

Finally, it is important to point out that with one processing operation (that is to say, injection moulding) it is possible to obtain a TPU/ABS mixture with a high degree of dispersion of the minor component.



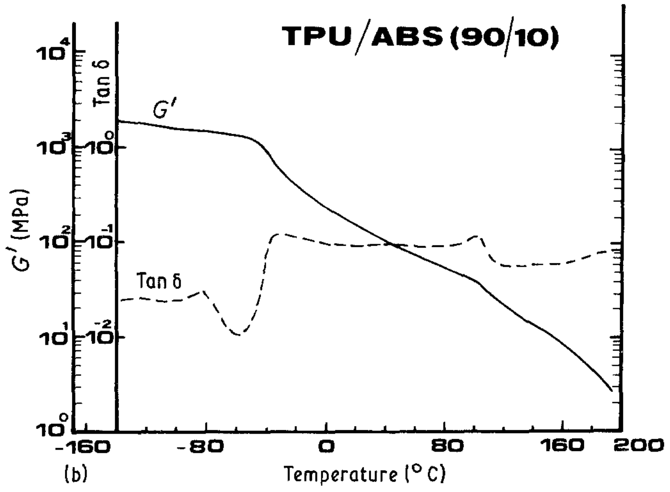
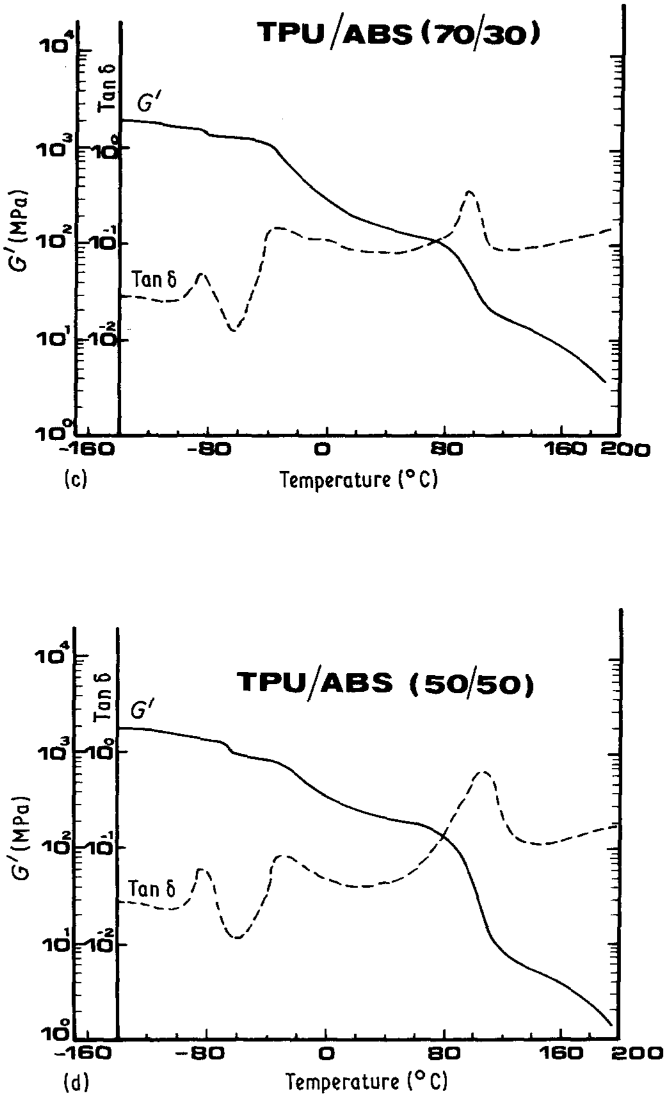


Figure 14 Shear modulus, G', and mechanical damping, tan δ, as function of temperature for: (a) pure TPU; (b) TPU/ABS (90/10) blend; (c) TPU/ABS (70/30) blend; (d) TPU/ABS (50/50) blend.

Figure 14 Continued.



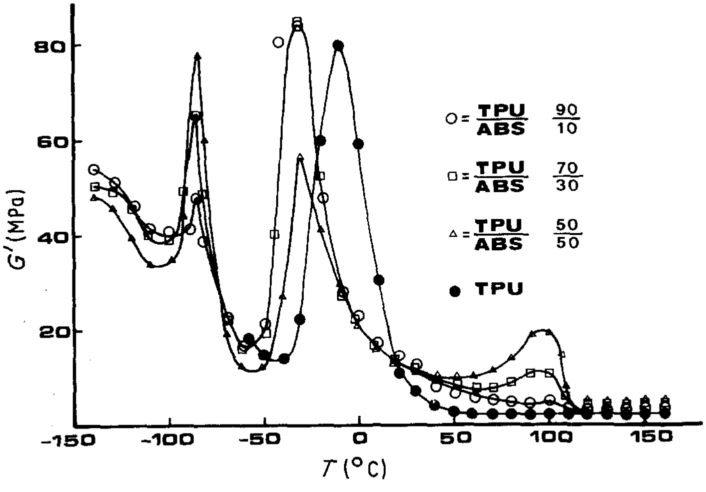
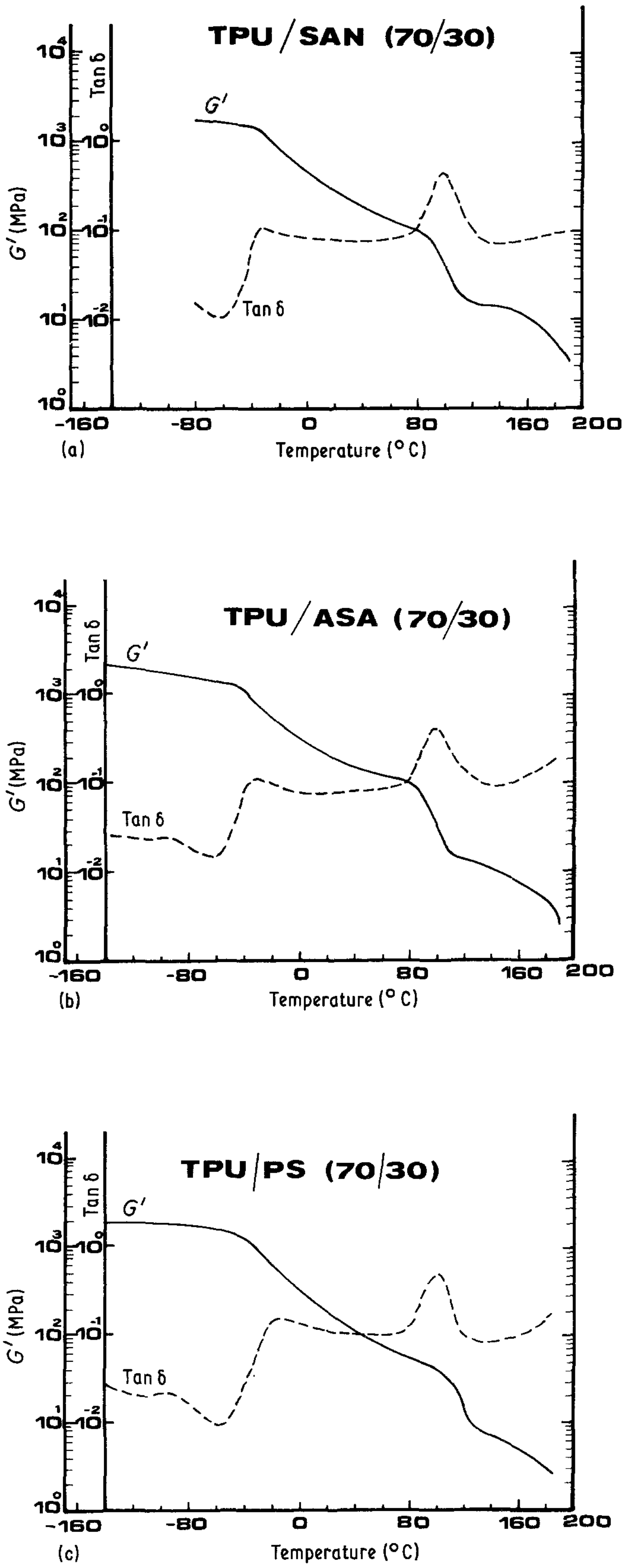


Figure 15 Loss modulus, G'', for TPU and its blends with ABS.

Figure 16 Shear modulus, G', and mechanical damping, $\tan\delta$, as function of temperature for: (a) TPU/SAN (70/30) blend; (b) TPU/ASA (70/30) blend; (c) TPU/PS (70/30) blend.



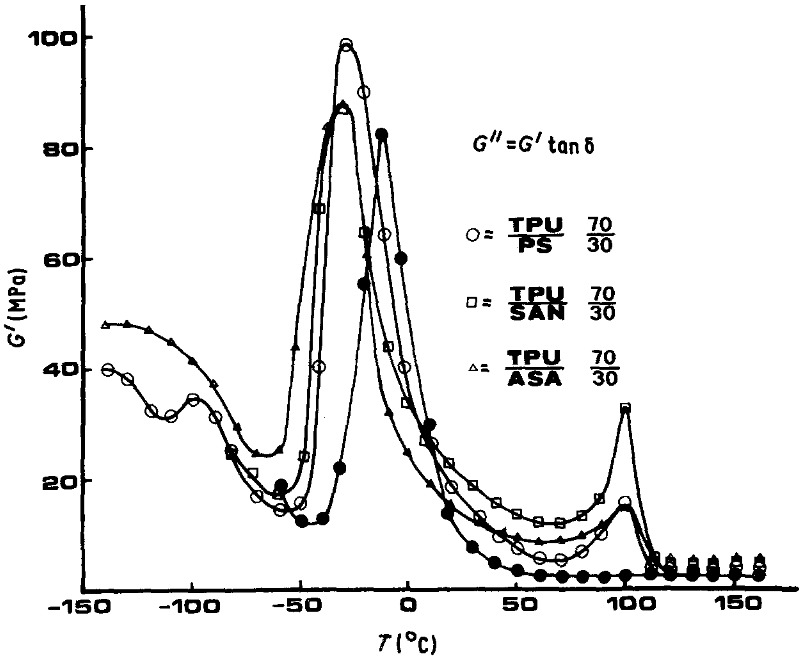


Figure 17 Loss modulus, G'', for TPU and its blends with PS, SAN and ASA.

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