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# Morphology of poly(vinyl chloride)/poly(butadiene-co-acrylonitrile) latexes and cast and molded materials.

Ioannis Sionakidis

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MORPHOLOGY OF POLY(VINYL  
CHLORIDE)/POLY(BUTADIENE-co-ACRYLONITRILE)  
LATEXES AND CAST AND MOLDED MATERIALS

by

Ioannis Sionakidis

A Thesis  
Presented to the Graduate Committee  
of Lehigh University  
in Candidacy for the Degree of  
Master of Science  
in  
Polymer Science and Engineering

Lehigh University

1977

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This thesis is accepted and approved in partial fulfillment of the requirements for the degree of Master of Science.

Dec. 16, 1977  
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## ABSTRACT

Poly(vinyl chloride)/poly(butadiene-co-acrylonitrile) interpenetrating polymer networks (IPN's), 50/(25-25) by wt in composition were synthesized in latex form. Transmission electron microscopy along with dynamic mechanical spectroscopy agree with a graded composition in the latexes, in which the poly(vinyl chloride) seed latex network I is partially penetrated by the poly(butadiene-co-acrylonitrile) network II, thus giving increased amounts of polybutadiene in the exterior of the latex particles.

## INTRODUCTION

A latex interpenetrating polymer network (IPN) is a unique type of polymer blend synthesized by emulsion polymerization. A seed latex of crosslinked polymer I is first made; monomer II is then added together with crosslinker and activator (but no new soap), and polymerized on the original seed latex particles<sup>1-4</sup>. In a latex IPN, each interpenetrating network microcosm is contained within an individual latex particle<sup>1</sup>, as opposed to bulk IPN's where each network exists throughout the macroscopic samples<sup>5-7</sup>.

Polymers I and II are incompatible to a greater or lesser extent and usually show phase separation, similar to most polyblends, grafts and blocks. The presence of the double networks in a bulk IPN, however, is already known to control morphology, giving rise to phase domains ranging from 100Å to 1500Å<sup>5</sup>.

The morphology of latex IPN's is also expected to be complex, including possible core-shell and cellular structures. Core-shell morphologies of latex particles synthesized in two-stage polymerization are suggested by the work of Grancio and Williams<sup>8</sup>, while cellular structures are shown in bulk IPN's<sup>5,10</sup> and in graft copolymers<sup>11</sup>. As an example, ABS latex graft copolymers show both the core-shell structure and a cellular struc-

ture within the core<sup>12</sup>. Important variables in terms of latex IPN morphology include relative amounts of the two polymers, order of preparation, crosslink density, solubility of the polymer in the monomer, and the size of the latex particle relative to polymer chain dimensions. Thus, below a certain latex particle size the cellular structure may not form<sup>1</sup>, but a fine structure may still be possible.

Poly(vinyl chloride) (PVC) homopolymer is a stiff, plastic material that usually exhibits a slight degree of crystallinity<sup>13</sup>. When PVC is blended with an elastomeric second polymer, the modulus, glass-rubber transition behavior and impact resistance depend upon the amount of elastomer added and upon its compatibility with PVC<sup>14</sup>. Matsuo<sup>14</sup> showed that the compatibility of mechanical blends of PVC with poly(butadiene-co-acrylonitrile) [poly(B-co-AN)] increased with acrylonitrile content. Sperling, et al.<sup>15</sup> previously studied several prototype PVC/poly(B-co-AN) latex IPN compositions by means of dynamic mechanical spectroscopy (DMS) of cast films and molded sheets made from the latexes. They suggested that poly(B-co-AN) network II shows a gradient in composition within the seed latex polymer I network.

The present paper studies a particular composition of the PVC/poly(B-co-AN) latex system in detail. The

emphasis is on transmission electron microscopy (TEM) of the materials, to establish the morphologies in the latexes and their relation to the cast and molded forms. In combination with dynamic mechanical spectroscopy results, this gives a more complete picture of the effects of synthetic detail on phase size and continuity and the extent of molecular mixing between polymer networks I and II.

## EXPERIMENTAL

### Synthesis

The latex IPN's were synthesized by a two-stage emulsion polymerization technique as follows. The first stage consisted of making a seed latex of crosslinked PVC polymer I and then introducing the monomer II mixture of butadiene and acrylonitrile and crosslinker followed by a second polymerization. The recipe for the seed latex is the following:

Deionized H <sub>2</sub> O	115 ml
Sodium lauryl sulfate (SLS) emulsifier	0.3 g
Vinyl chloride monomer	≈ 20 g
Potassium persulfate (K <sub>2</sub> S <sub>2</sub> O <sub>8</sub> ) initiator	0.25 g
Tetraethylene glycol dimethacry- late (TEGDM) crosslinker	0.4% by wt of monomer

The above polymerization ingredients are charged as follows: 102 ml of deionized H<sub>2</sub>O is introduced into a 190 ml glass pressure bottle and is warmed to 50°C. Then 3 g of 10% (by wt.) water solution of the emulsifier is added and stirred. The bottle is cooled down to room temperature and then the potassium persulfate (K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>) initiator is added in solution form as 0.25 g/10ml H<sub>2</sub>O. The crosslinking agent was next added dropwise (about 4-5

drops). The bottle contents were then sparged for about 10 minutes with nitrogen gas to drive off the oxygen and the bottle was sealed immediately. The contents were next frozen and weighed. Vinyl chloride gas from a pressure cylinder was liquified (inside a 190 ml glass pressure bottle put in a liquid nitrogen bath) and introduced into the polymerization bottle on top of the already frozen contents. The monomer was allowed to boil off to the desired amount and the bottle was then capped with a gas-tight rubber seal, weighed and the exact mass of vinyl chloride in the bottle was determined and recorded. The capped bottle was then put in a 50°C water bath where it was tumbled at about one revolution per minute for no less than 7 hours, yielding a completely reacted product. A thin syringe needle was inserted in the bottle through the rubber seal to test for the presence of any unreacted monomer at the end of polymerization.

The PVC latex was filtered to remove any traces of coagulated polymer and used as a seed latex for the second polymerization. New initiator (added in solution form as 0.25 g/10 ml H<sub>2</sub>O), plus TEGDM crosslinker (0.4% by wt. based on acrylonitrile plus butadiene) were stirred into the seed latex. The bottle was then sparged with nitrogen gas for about 5 minutes and the acrylonitrile monomer was weighed directly on top of the seed latex, 50% by wt. based

on PVC. Butadiene gas from a pressure cylinder was liquified (inside a liquid nitrogen bath) and introduced into the bottle directly on top of the floating acrylonitrile. The butadiene was then allowed to boil off to the desired amount. The final composition was a 50/(25-25) poly-(vinyl chloride)/poly(butadiene-co-acrylonitrile). The bottle was then capped and put into the water bath where it tumbled at 40°C for a minimum of 12 hours for a completely reacted product.

A portion of the finished latex (10-15 ml) was dried in open petri dishes at room temperature to form cast films. The rest of the latex was coagulated (a small portion of a saturated NaCl solution may be used), washed with water and 2-propanol, and finally molded at a pressure of about 5500 psi and at approximately 190°C temperature to form suitable sheets.

Because addition of the cold liquid butadiene may cause freezing and some coagulation of the seed latex, balloons were suggested for introducing butadiene<sup>15</sup>. After the new initiator, crosslinker, and acrylonitrile had been added, a balloon was hung inside the bottle above the surface of the seed latex and the floating acrylonitrile. The balloon was secured by stretching it over the outside lip of the bottle and the butadiene was poured into it. The balloon was slashed at 2 or 3 points on its

neck so that the butadiene would finally escape when tumbling began. The same weighing process was again employed before capping the bottle.

### Electron Microscopy

The latex particles of both the seed and the finished emulsions were viewed in the transmission electron microscope (TEM). The original latex was diluted with deionized water until the proper concentration was obtained (about 1 and 2 drops per 16 ml water for the seed and the finished latex, respectively). A drop of the diluted latex was placed on a microscope grid and excess fluid was removed. The grid was then inserted in the TEM for direct viewing of the latex particles. Shadowing the latex particles with a heavy metal (Pt) or staining the finished latex with osmium tetroxide ( $\text{OsO}_4$ ) before viewing was also done. When staining was done, a drop of the diluted finished latex was placed on the microscope grid and the grid was exposed in  $\text{OsO}_4$  vapor for about 3 hours. The  $\text{OsO}_4$  selectively attacks the double bonds of the polybutadiene component and hardens the latex particles. (The rubbery polybutadiene component will make the latex particles coalesce and flatten.) Micrographs were taken and the sizes of both the seed and the finished latex were established and compared.

The morphology of the cast films and molded sheets

was also examined by TEM. Specimens were "rough-trimmed" with a razor blade so that the end regions were tapered into the shape of a truncated pyramid having sides of  $\approx 0.2\text{mm}$ . The specimens then were exposed to  $\text{OsO}_4$  vapor for several days and subsequently vacuum treated to remove volatile material. Then a Porter-Blum MT-2 ultramicrotome equipped with a diamond knife was used to obtain sections about  $300\text{-}600\text{\AA}$  in thickness at room temperature. The thin sections were put on microscope grids and directly viewed in the TEM where photomicrographs of the morphology were obtained. Ultramicrotoming the specimens and then exposing the thin section to  $\text{OsO}_4$  for about 45 minutes before viewing in the TEM was also done. Soft cast or molded specimens were embedded in epoxy before sectioning.

#### Dynamic Mechanical Spectroscopy (DMS)

All DMS studies employed a Rheovibron direct reading viscoelastometer, model DDV-II (Vibron manufactured by the Toyo Measuring Instrument Co., Ltd., Tokyo, Japan) set at a frequency of 110 Hz. The temperature range employed was  $-120^\circ\text{C}$  to  $+150^\circ\text{C}$ , with a heating rate of about  $1^\circ\text{C}$  per minute. As per requirement of the instrument, the sample dimensions were of the order of  $0.02 \times 0.2 \times 1.4$  cm on the average.

## RESULTS

The seed and finished latexes showed considerable distribution of particle sizes. The seed latex particles, Figure 1, ranged from about 150 to 550 $\text{\AA}$  while the finished latex, Figure 2, showed bigger particles ranging from about 450 to 800 $\text{\AA}$ . The tiny particles seen in the stained finished latexes, Figure 2, are believed to be butadiene-acrylonitrile copolymer formed during the second polymerization. The total volume of these particles, however, is negligible and should not affect the morphology and the physical or mechanical properties of the bulk material. Unstained finished latexes showed much more coalescence in the electron microscope but the sizes were similar to those shown in Figure 2.

The films that were cast from finished latexes usually cracked during drying and showed low toughness. After heating in an oven at about 90 $^{\circ}\text{C}$  for about half an hour, they became softer and showed higher toughness; apparently the individual latex particles coalesced better at the elevated temperature forming a more continuous film. When balloons were used to introduce the butadiene monomer during the second polymerization, the resulting films showed varied behavior, ranging from discontinuous slightly tough to continuous tough films. The molded sheets showed in general leathery behavior with the one synthesized using

balloons showing again varied behavior. Morphology of the latter specimens in TEM, however, is no different.

The morphology of cast films and molded sheets stained with  $\text{OsO}_4$  was similar, Figures 3 and 4. They both showed a two-phase structure with the dark phase being the more continuous. The light phase is the PVC seed latex and the surrounding dark network is believed to contain more of the stained polybutadiene component. For comparison the morphology of an unstained material is shown in Figure 5.

The dynamic mechanical data of cast films and molded sheets showed broad glass transition regions with single well-defined  $\tan \delta$  and  $E''$  peaks. The appearance of these spectra are consistent with the earlier findings of Matsuo<sup>14</sup> that PVC forms semicompatible or compatible (depending on the amount of acrylonitrile present) blends with poly(B-co-AN).

## DISCUSSION

The TEM morphologies suggest a tendency toward core-shell latex structures, as found by Grancio and Williams<sup>8</sup>, upon the step addition of a comonomer during emulsion polymerization. On the other hand, the DMS results suggest extensive molecular mixing of the PVC and P(B-co-AN) in the bulk materials. The most reasonable overall interpretation is that the finished latex particles have a gradation of P(B-co-AN) content, increasing from the interior to a maximum at the exterior. Sufficient butadiene is present at the exterior to give strong OsO<sub>4</sub> staining and to permit film formation in cast latexes, although mechanical properties are improved considerably by further coalescence during heating cast films or molding under heat and pressure.

Although this picture remains qualitative, it is more complete than before<sup>15</sup>, and it relates latex and bulk characteristics rather than considering them separately. Support for this picture follows.

### Transmission Electron Microscopy

Two limiting cases can be considered for TEM image contrast of the finished latexes. In the first case, Figure 10a, the latex spheres are taken to be homogeneous in composition and to stain uniformly with OsO<sub>4</sub>. In TEM, the particles would be darkest in the interior, as expected

for the image of any sphere viewed in transmission.

In the second case, Figure 10b, the latex spheres are taken to have a stained shell that is sharply demarcated from an unstained core. The transmission image is darkest immediately outside the core, and the core is much brighter than in the homogeneous case.

The actual stained latex, Figure 2, appears to lie between the two limiting cases. Most particles are slightly but definitely darker near the exterior than in the center, consistent with a higher concentration of polymer II, P(B-co-AN), toward the exterior. An early stage of latex coalescence is apparent from the necks between particles, but these are light in the micrograph because they are only 70 to 150<sup>o</sup>Å thick.

For the core-shell limiting case, Figure 10c shows that ultramicrotomes thin sections can exaggerate the image contrast between core and shell. This effect is apparent in the micrographs of Figures 3 and 4, which show touching dark rings of the same diameter range as the finished latex particles.

Without staining, the contrast reversal found in the thin section of Figure 5 is expected, with the PVC-rich latex cores absorbing the electron beam more strongly than the P(B-co-AN)-rich shells. The latex particles still retain their identity in Figure 5, although not as strikingly as in Figures 3 and 4.

## Dynamic Mechanical Spectroscopy

The glass transitions of the cast or molded latexes, Figures 6-9, are much broader than usual for PVC, being over the range of about 0 to 100°C for all of the materials. This compares with  $T_g$ 's of about 7.5 and 100°C for the nominal composition of P(B-co-AN) and PVC homopolymer, respectively. For various mechanical blends of PVC with P(60B-co-40AN), however, Matsuo et al.<sup>16</sup> found fairly sharp transitions at temperatures between the  $T_g$ 's of the two polymers. The present materials, therefore, appear to have a range of compositions from PVC to P9B-co-AN), since the glass transitions are gradual and span the entire range between the individual polymers.

As can be noted in Figures 8 and 9, the transitions are somewhat sharper when butadiene is added by means of balloons, as described earlier. There is no ready interpretation of this finding. The butadiene might reach the emulsion particles later than the acrylonitrile and give a butadiene-rich shell, but there is no indication of the presence of polybutadiene with its low  $T_g$  of about -80°C.

All of the  $E''$  and  $\tan \delta$  curves show a broad, low  $\beta$ -transition peak at about -60 to -80°C. This is similar to the  $\beta$  peaks for PVC<sup>13,16</sup>, but displaced downward somewhat in temperature. Matsuo et al.<sup>16</sup> found a similar displacement in their mechanical blends, consistent with the present results.

## General

The combined experimental results agree with a graded composition in the latexes in which the crosslinked PVC seed latex is partially penetrated by butadiene and acrylonitrile monomers, which then polymerize in situ. Phase separation is not evident from either TEM or DMS results, but heavier OsO<sub>4</sub> staining indicates that the outer portions of the latex particles are richer in butadiene.

Intimate mixing of the monomers and polymers used here is reasonable, based both on solubility parameters, Table I, and the previously observed compatibility of the polymers<sup>16</sup>. Presumably, the polar acrylonitrile can swell readily into the PVC seed latex particles, along with appreciable amounts of butadiene. Polymer II, when formed, is highly compatible with the PVC, but its amount (and possibly butadiene content as well) increases toward the exterior of the finished latex particles. The presence of the rubbery copolymer makes film formation possible, particularly in view of its concentration at the outside where the particles are in contact during coalescence.

We are not aware of other comparable studies of multicomponent latexes and bulk materials made from them. In ABS materials, however, Kato<sup>12</sup> has shown that individual polybutadiene latex particles can develop P(styrene-co-acrylonitrile) glassy domains in their interiors as well as in surrounding shells that are either continuous or

intermittent. Molded materials have a continuous majority phase of the glassy polymer.

In the present system, the high compatibility of the polymers and the presence of crosslinking in both components probably precludes the formation of separate cellular phase domains. However, the morphology and properties of the bulk materials are clearly inherited directly from the size and composition gradations in the finished latex particles.

TABLE I  
Solubility Parameters  
of Selected Monomers and Polymers<sup>17</sup>

<u>Structure</u>	<u>Solubility Parameter</u>	
	<u>Monomer</u>	<u>Polymer</u>
Poly(vinyl chloride)	7.8	10
Polybutadiene	7.1	8
Polyacrylonitrile	10.5	13

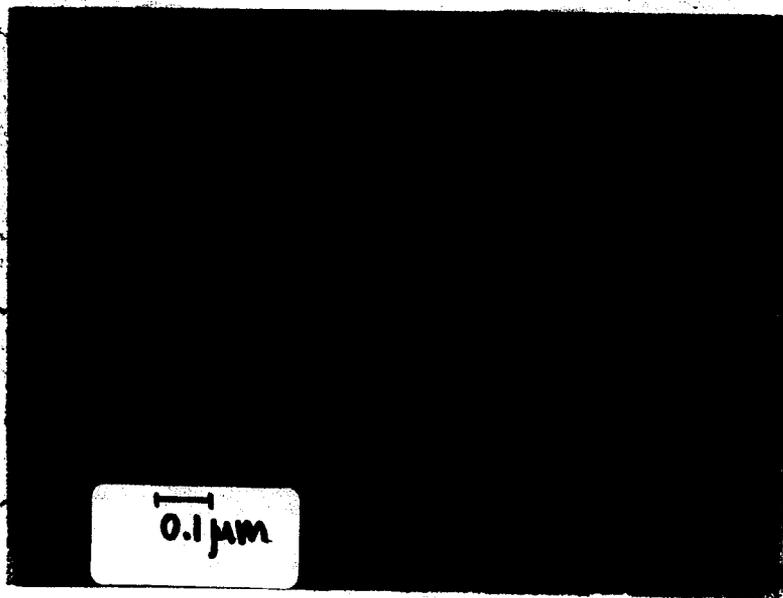


FIGURE 1. Electron micrograph of PVC seed latex, shadowed with Pt metal.

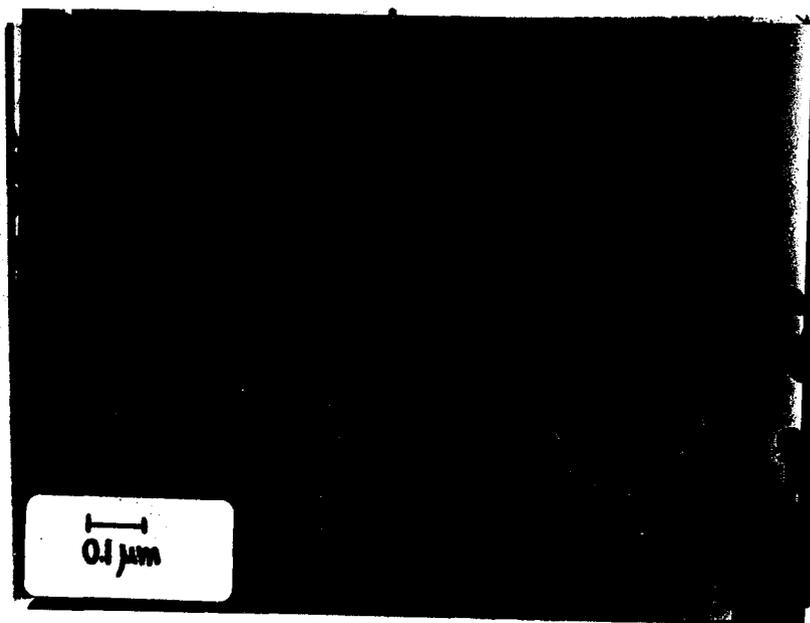


FIGURE 2. PVC/P(B-co-AN) finished latex. Electron micrograph of  $\text{OsO}_4$ -stained particles.

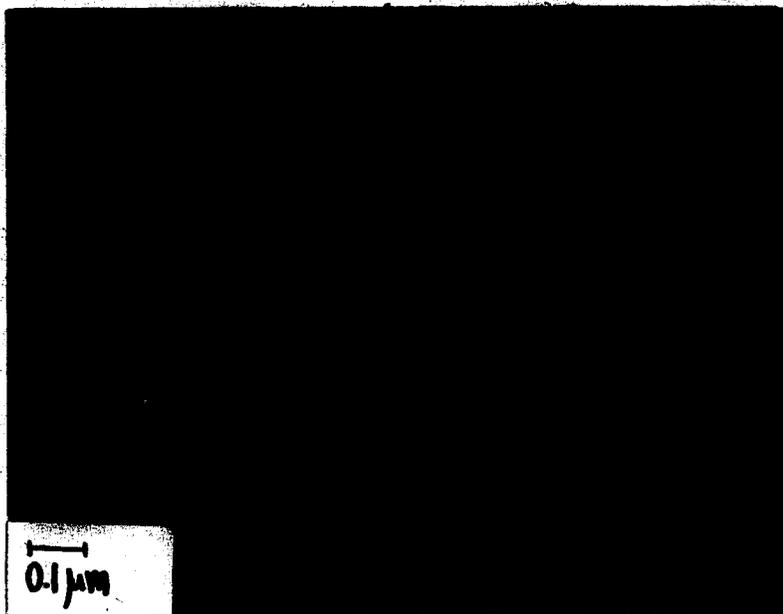


FIGURE 3. PVC/P(B-co-AN) cast film. Electron micrograph of film exposed to  $\text{OsO}_4$  for about 4-5 mins. after it was sectioned. Film was preheated at  $90^\circ\text{C}$  for about 30 mins. before sectioning.

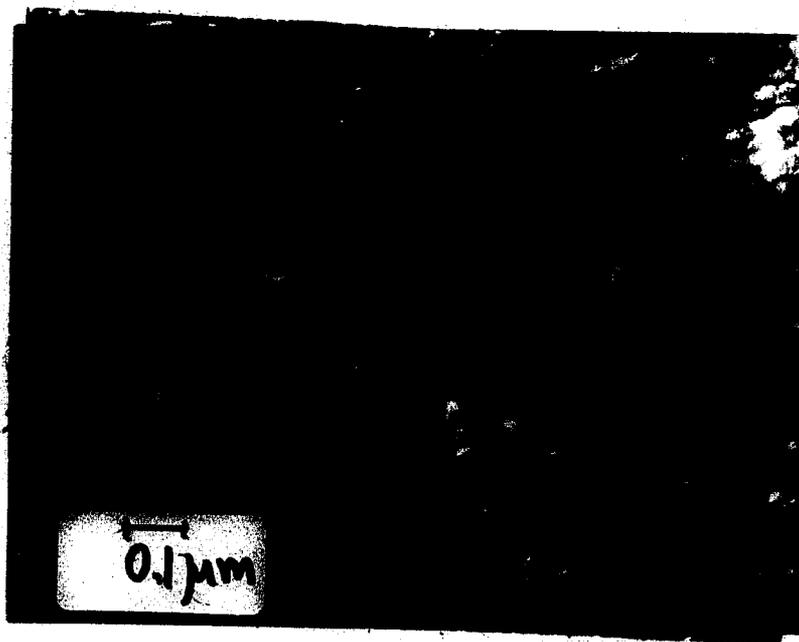


FIGURE 4. PVC/P(B-co-AN) molded sheet. Electron micrograph of sample stained with  $\text{OsO}_4$ . Sample was stained and then sectioned.



FIGURE 5. PVC/P(B-co-AN) cast film. Electron micrograph of unstained sample.

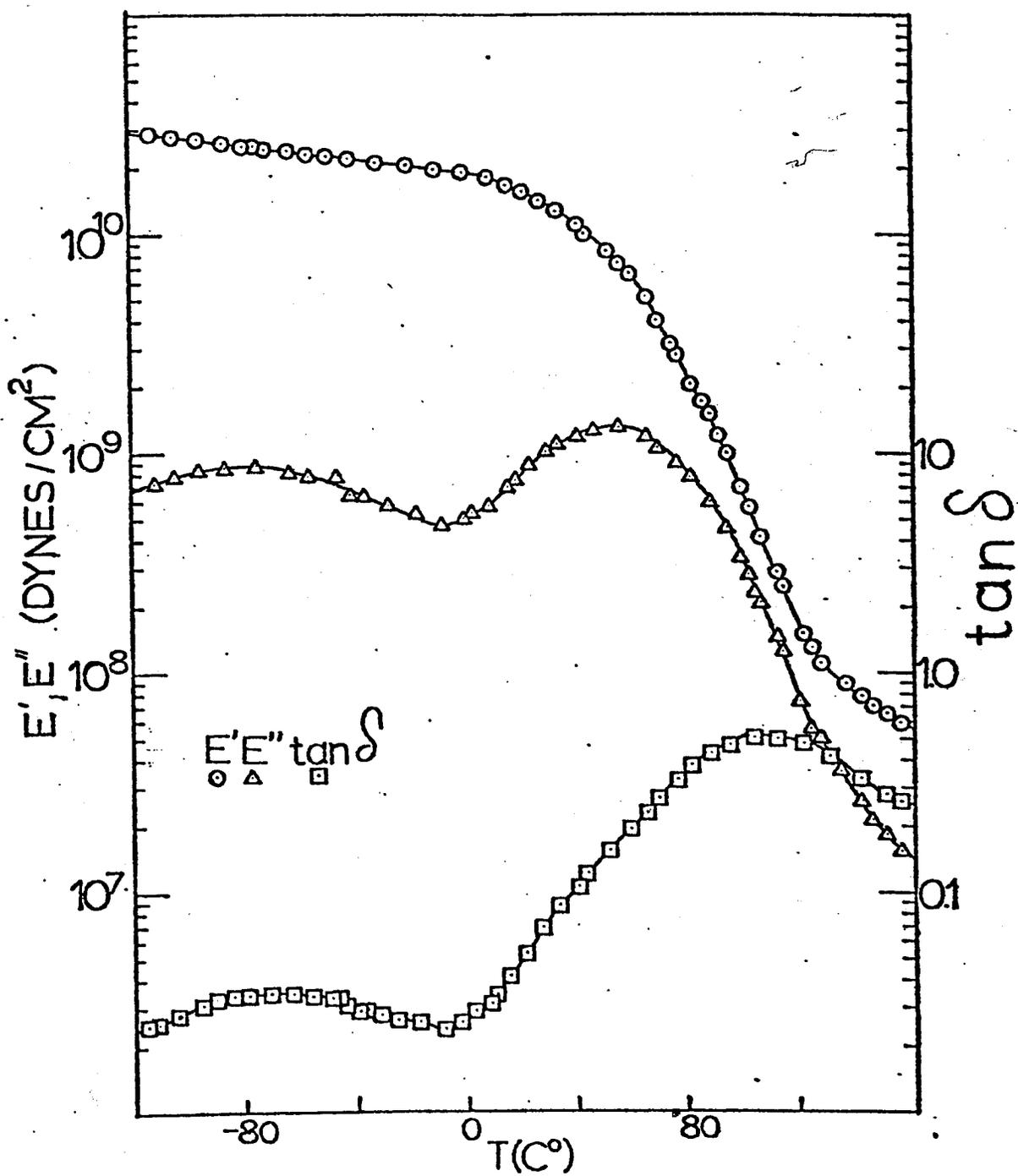


FIGURE 6. Dynamic mechanical spectroscopy of molded sheet of PVC/P(B-co-AN).

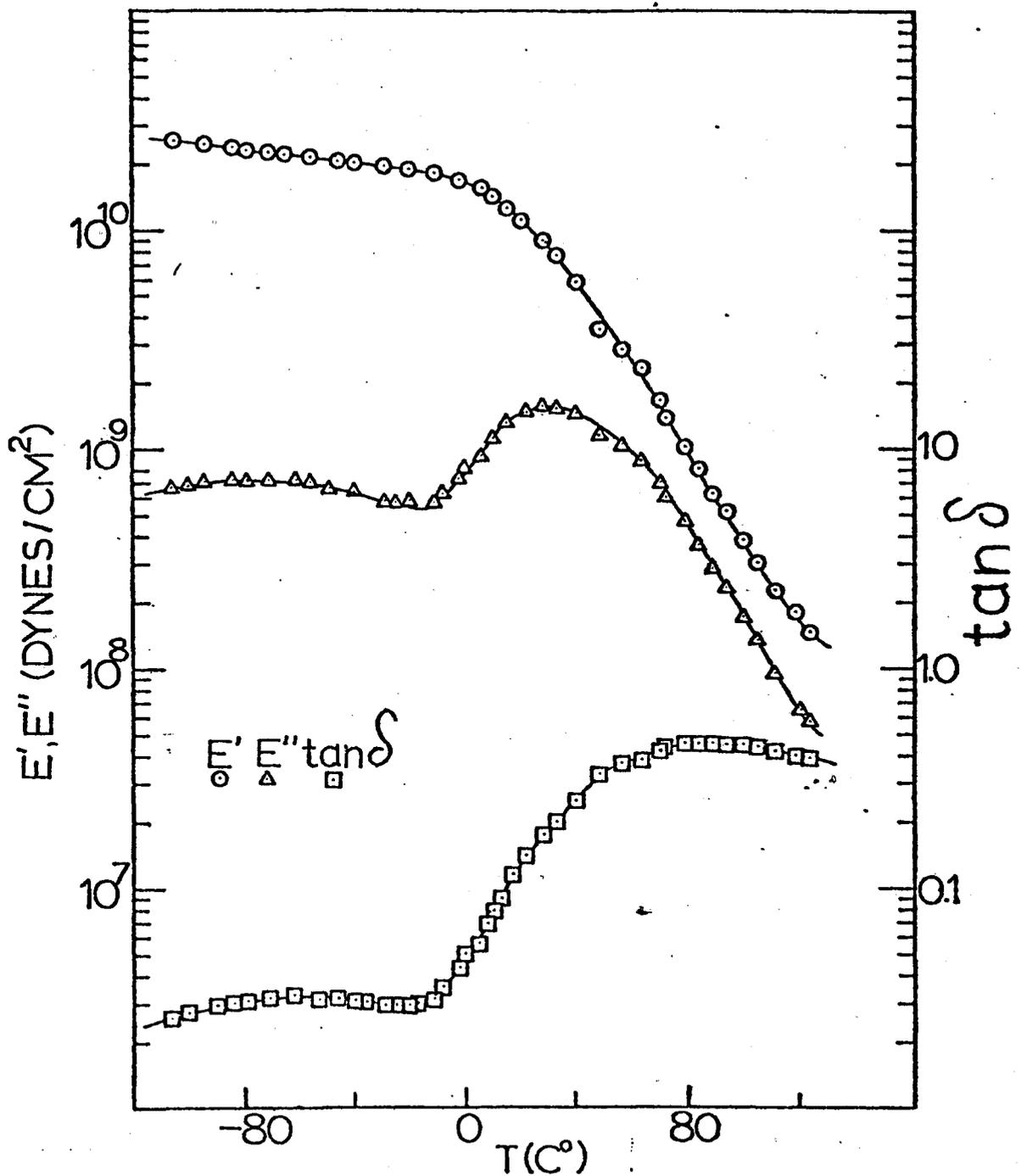


FIGURE 7. Dynamic mechanical spectroscopy of cast film formed from the same emulsion as in Figure 6. The peaks here are shifted to lower temperatures. Film was preheated at  $90^{\circ}\text{C}$  for about 30 mins. before testing.

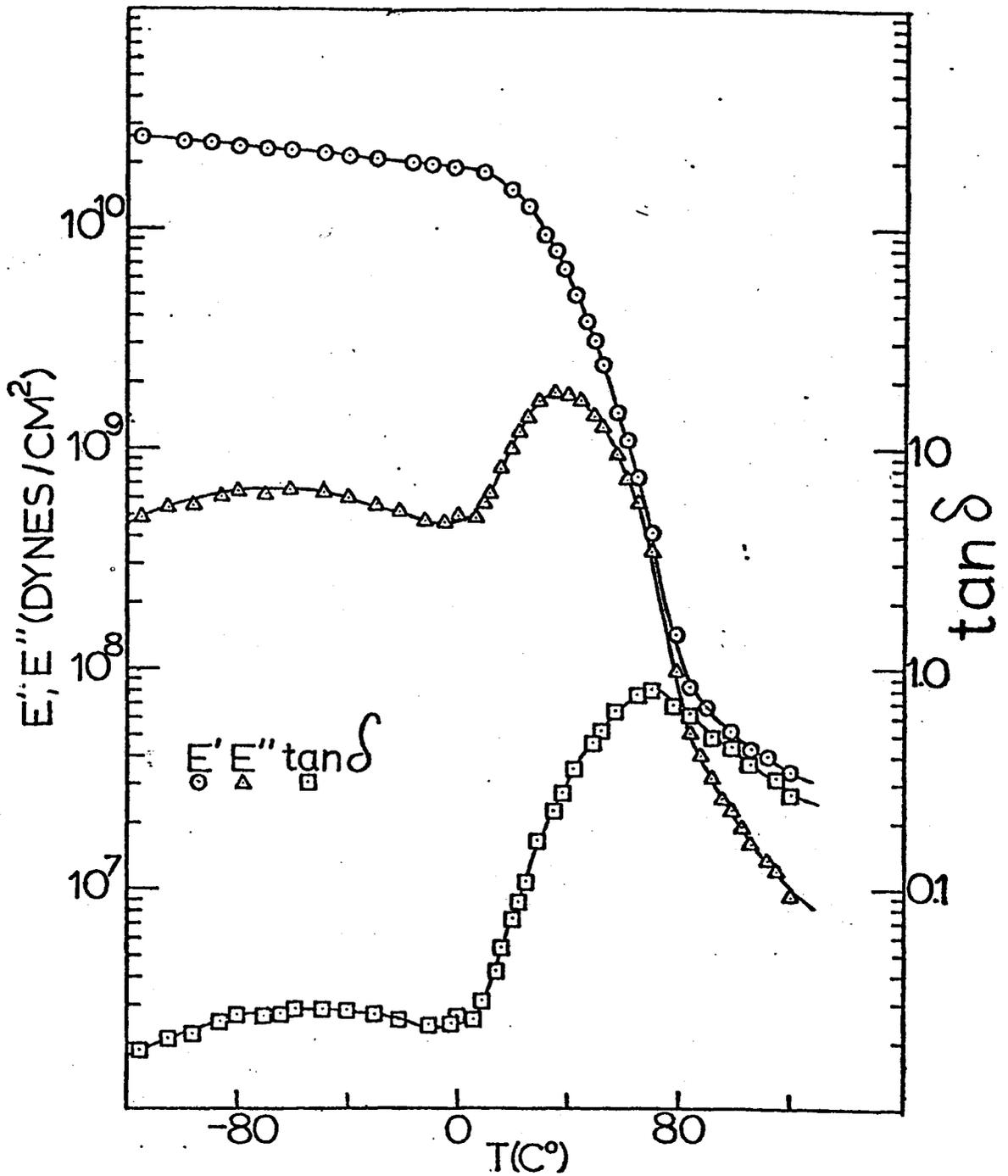


FIGURE 8. Dynamic mechanical spectroscopy of molded sheet. Emulsion was synthesized using balloons to add butadiene. Less gradation in PVC compared to Figure 6. Material is also softer, because shell is richer in polybutadiene.

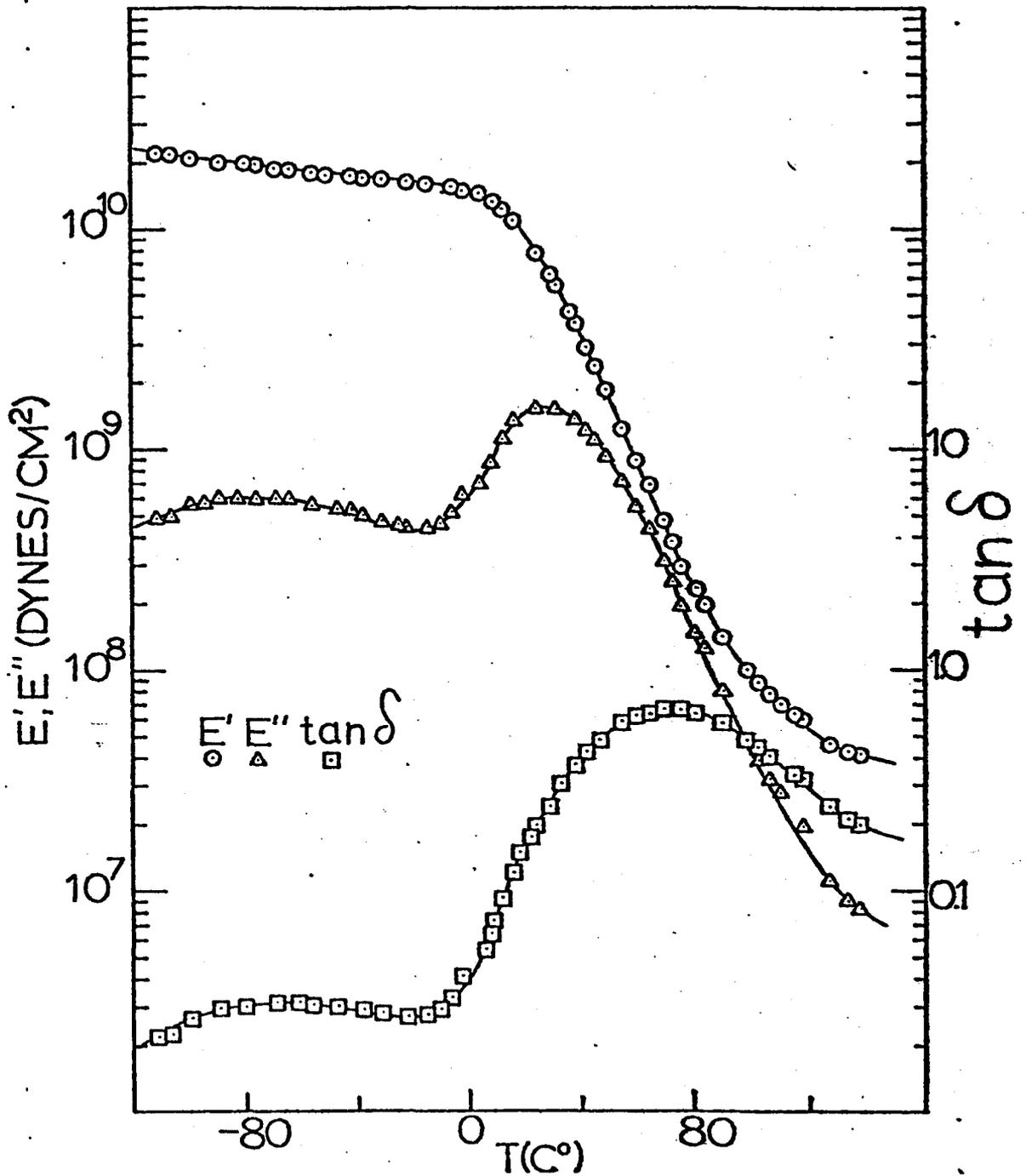


FIGURE 9. Dynamic mechanical spectroscopy of cast film formed from the same emulsion as in Figure 8. A more continuous and softer film than in Figure 7 is formed, because shell is richer in polybutadiene.

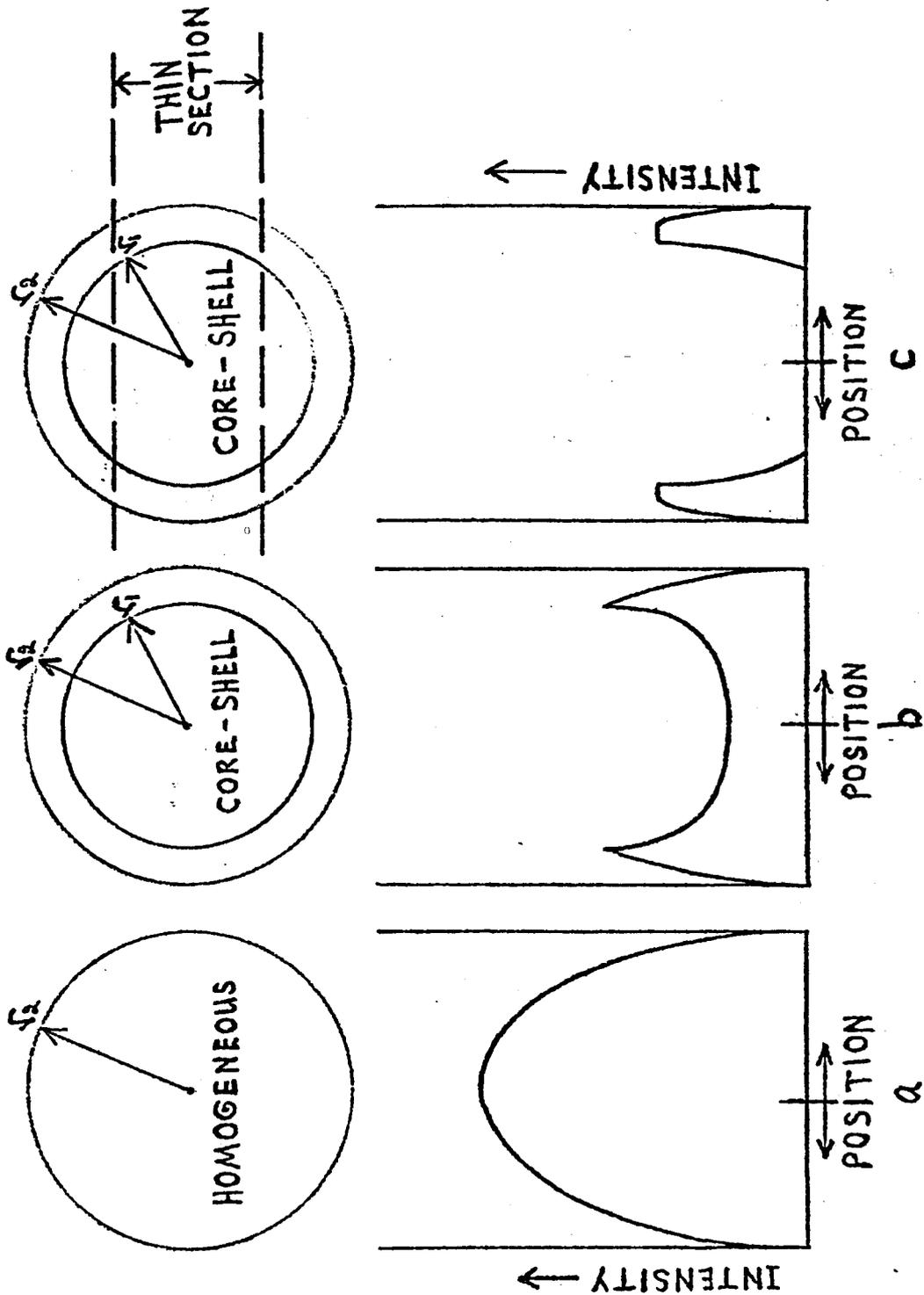


FIGURE 10. Idealized image intensities for transmission electron microscopy of latex particles: (a) homogeneous latex, (b) core-shell latex, (c) ultramicrotomed thin section through core-shell latex. Core/shell ratio, calculated assuming 50 wt. % PVC/50 wt. % P(B-co-AN) and polymer densities of 1.39 and 1.06 g/cm<sup>3</sup>, respectively, gives  $r_2 = 1.31 r_1$ .

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## VITA

Ioannis Sionakidis was born April 4, 1951, in Katerini, Greece, the youngest child of Mr. and Mrs. Demetrios Sionakidis. He attended elementary school in Katerini and graduated from the Gymnasium of Katerini High School in June 1969.

Entering Malone College of Canton, Ohio, in December 1970, he graduated with honors in June 1975, receiving the degree of Bachelor of Arts. He was admitted to the Graduate School of Lehigh University of Bethlehem, Pennsylvania, the same year, being granted a research assistantship, and became a candidate for the degree of Master of Science in Polymer Science and Engineering.