Determining the Elastic Properties of Modified Polystyrenes by Sound Velocity Measurements

Imran Oral,¹ Hatice Guzel,¹ Gulnare Ahmetli,² C. Hakan Gur³

¹Department of Physics Education, Selcuk University, Konya 42090, Turkey

²Department of Chemical Engineering, Selcuk University, Konya 42031, Turkey

³Department of Metallurgical and Materials Engineering, Middle East Technical University, Ankara 06531, Turkey

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ABSTRACT: The effect of the binding of various polyfunctional groups to polystyrene's (PS's) aromatic ring on the elastic properties of the PS were investigated by an ultrasonic method. Various sets of samples were prepared by chemical modification of pure PSs having different molecular weights with SA, maleic anhydride, and phthalic anhydride. The ultrasonic wave velocities of modified PSs were measured with the pulse-echo method at room temperature by a computercontrolled analyzer and a digital oscilloscope. The values of the acoustic impedance, Poisson's ratio, and elasticity constants of the samples were calculated by the measured values of the densities and sound velocities. The longitudinal and shear wave velocities and the values of all elastic constants increased with chemical modification of the pure PS. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 121: 3425–3432, 2011

Key words: mechanical properties; modification; polystyrene

INTRODUCTION

The use of ultrasound in physics and chemistry has been investigated for more than 70 years.¹ Recently, its potential in organic chemistry has become a fruitful area of research, offering the potential for new synthetic pathways.² Ultrasound is finding an increasing number of applications in the modern world. Included among these are medical imaging, dentistry, particle sizing, food processing, welding, wastewater treatment, and surgical processes.³ Furthermore, ultrasonic methods have been successfully used to monitor polymer processing,⁴ chemical reactions,^{5,6} film formation from aqueous polymer dispersions,⁷ glue processes, crystallization in polymers,^{8,9} and also, the characterization of polymers.

Polystyrene (PS) is one of the most versatile thermoplastics used in industrial engineering. PS is a thermoplastic substance; it is in a solid state at room temperature but flows if heated above its glass-transition temperature and becomes solid again when cooled.¹⁰ Typical applications of PS are nearly ubiquitous. For example, solid PS is used in disposable cutlery, optical tools, cases, drink cups, food trays, dishes, egg boxes, plastic models, video/audio cassettes, CD and DVD cases, toys, light diffusers, beakers, general household appliances, electronic housings, refrigerator liners, and smoke-detector housings. Expanded PS beads are used for packaging and cushioning. Because of its chemical inertness, PS is used to fabricate containers for chemicals, solvents, and foods, too.¹¹

The principal limitations of the polymer are its brittleness; inability to withstand the temperature of boiling water; mediocre oil resistance; poor chemical resistance, especially to organics; and susceptibility to UV degradation.¹¹ These defective properties of PS can be improved, and it is possible for PS to gain new properties and to ameliorate these defective properties by the binding of various functional groups to the aromatic ring of PS. Modified polystyrene (MPS) has been found to have higher mechanical, thermal, and elasticity properties than pure PS and to be more durable against impact. Therefore, various functional groups have been bound to the aromatic ring of PS through chemical modification. In the last decade, there has been great interest in the chemical modification of traditional polymers with the aim of enhancing their chemical properties and making them useful for special applications.¹²⁻¹⁸

Recently, attempts have been made to study the sound velocity and attenuation of polymers. When propagated in polymeric materials, acoustic waves are influenced by the polymer's structure and by molecular relaxation processes. It is possible to estimate the viscoelastic properties of polymeric materials from the

Correspondence to: G. Ahmetli (ahmetli@selcuk.edu.tr).

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velocity and attenuation of longitudinal or shear waves.¹⁹ Ultrasonic techniques are a versatile tool for investigating the changes in the microstructure, deformation process, and mechanical properties.²⁰ The various parameters upon which the elastic modulus of polymers depend can be studied by the measurement of the ultrasonic wave velocities.

Elastic constants are physical properties that relate stress and strain. Because solids resist both volume change and shape change, they have at least two independent elastic constants. A low-symmetry crystal may have as many as 21 independent elastic constants, but polycrystalline, noncrystalline, and amorphous isotropic materials can be characterized by only two independent elastic constants: the longitudinal modulus (L) and the shear modulus (G). Many functional and differential relationships between elastic constants have been reported;²¹⁻²⁴ these have emerged from the theory of elasticity and from the physical meanings of the constants. However useful these relationships may be, they only represent the functions relating a dependent variable to two independent variables. Two independent elastic constants are required to characterize isotropic materials with the presently available relations. Hence, the identification of any new relationship that reduced the number of required independent elastic constants to one would be beneficial. This hypothesis is based on the belief that there must be a hidden relationship between the two independent elastic constants that is based on the microstructure, atomic bonding, electronic configurations, and so on. A number of researchers have given empirical relationships to relate the L, G, Young's modulus (E), and bulk modulus (K) for various elements.²¹⁻²³ Poisson's ratio (μ) , defined as the lateral contraction per unit breadth divided by the longitudinal extension per unit length in simple tension,²⁵ is reported to provide more information about the character of the bonding forces than any of the other elastic constants.²⁴

Although a number of researchers have evaluated the elastic properties of pure PS and MPSs by destructive techniques, the evaluation of elastic properties of these materials by nondestructive techniques has not been reported in the open literature. The ultrasonic velocity method is a nondestructive, economical, and very precise method.26,27 Moreover, the sound velocity through solids mainly depends on the intermolecular and intermolecular interaction potentials. Thus, ultrasonic evaluation of the elastic properties of these materials is important from both a scientific and technological point of view. Therefore, in this study, we aimed to obtain MPSs with various functional groups and to investigate their elastic properties by sound velocity measurements.

PS samples with different molecular weights [pure

Materials

polystyrene with a molecular weight of 5.0×10^5 (PS₅₀₀) and pure polystyrene with a molecular weight of 3.5×10^5 (PS₃₅₀)], succinic anhydride (SA), maleic anhydride (MA), phthalic anhydride (PhA), chloroform as a solvent, methanol as a precipitator, and BF₃·O(C₂H₅)₂ as a cationic catalyst were purchased from Merck (Darmstadt, Germany).

EXPERIMENTAL

Chemical modification

A reactor consisting of a mixer, cooler, and thermometer was used in this experiment. To modify PS₅₀₀, first, 7.8 g of SA (20% of the polymer amount) was added to a solution of 39 g of PS in 300 mL of chloroform (CHCl₃) by mixing. After the anhydride was dissolved completely, 10 mL of $BF_3 \cdot O(C_2H_5)_2$ was added drop by drop and stirred for 3 h at 25°C to end the reaction. The mixture was poured into a beaker. MPS was precipitated by methanol (500 mL) from the reaction mixture, filtered, and dried in *vacuo* at 60°C for 5 h. So, the modified polystyrene with a molecular weight of 5.0×10^5 (MPS₅₀₀) modified with SA (SAI) was obtained. The chemical modifications of the other PS_{500} samples with MA and PhA in the presence of the catalyst $BF_3 \cdot O(C_2H_5)_2$ were examined with the same procedure. MPS₅₀₀'s modified with MA (MAI) and PhA (PhAI) were also obtained.

The PS₃₅₀ samples chemically modified with SA, MA, and PhA in the presence of the catalyst BF₃·O(C₂H₅)₂ were examined with the same procedure, too. Modified polystyrenes with a molecular weight of 3.5×10^5 (MPS₃₅₀) modified with SA, MA, and PhA (SAII, MAII, and PhAII, respectively) were obtained.

Measurements

The Fourier transform infrared (FTIR) spectra of polymers were recorded on a PerkinElmer 1605 FTIR instrument (Perkin Elmer 1605, USA). The density (ρ) of the samples were measured according to the Archimedes principle with water as the immersion liquid by an analytical balance (Radwag AS220/C/2, capacity = 220 g, readability = 0.1 mg) and a kit of ρ . First, the temperature of the room was inserted into the balance; next, the masses of the samples were measured in air and in water, and finally, the densities of the samples were measured automatically by the balance. The accuracy of the measurements was about 0.001%.

The sound velocity measurements were done by the pulse-echo method at room temperature. The

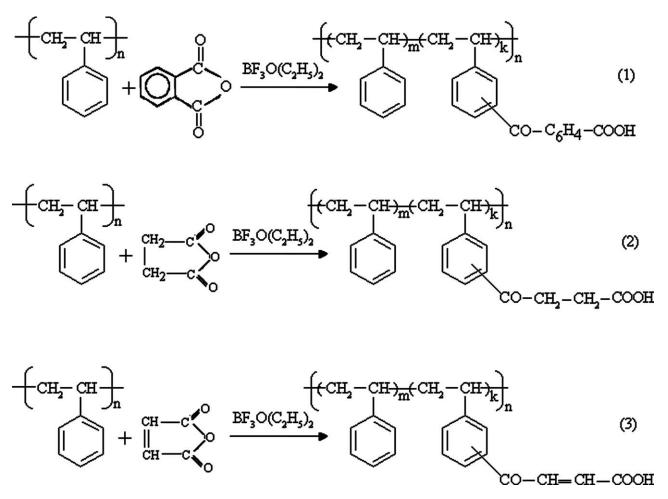


Figure 1 Modification reaction of PS with various organic anhydrides: (1) PhA, (2) SA, and (3) MA.

ultrasonic pulses were provided by a Ultrasonic Pulser Receiver 5800PR generator (35 MHz Panametrics Olympus). An electrical impulse with a high amplitude and a short duration excited the piezoelectric transducer vibrating on the fundamental mode through the sample and, after reflections on the opposite face, returned to the transducer. After propagation in the material, the output signal was displayed on the screen of a numerical oscilloscope (60 MHz GW Instek GDS-2062, Taiwan). We used 5-MHz shear (V155, Panametrics Olympus, USA) and 5-MHz (V109, Panametrics Olympus, USA) longitudinal contact transducers. As the coupling medium, glycerin (Couplant B, Olympus, USA) was used for the longitudinal wave measurements, and an Shear Wave Couplant (SWC, Olympus, USA) was used for the shear wave measurements. The knowledge of the transit time through the thickness of the sample allowed us to determine the wave velocities by eq. (1):

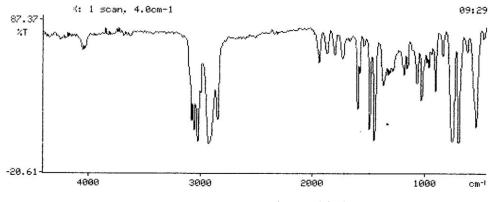


Figure 2 FTIR spectrum of unmodified PS.

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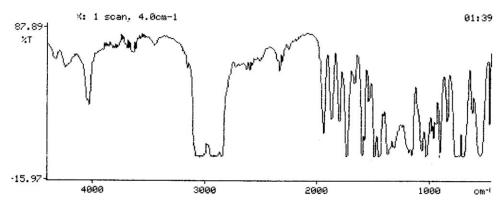


Figure 3 FTIR spectrum of PS modified with SA.

$$V = 2d/t \tag{1}$$

where *V* is the velocity of sound, *d* is the thickness of the sample, and *t* is the time of flight between subsequent backwall signals on the oscilloscope, respectively. The measurements were repeated 10 times to check the reproducibility of the data. The accuracy of the velocity measurements was about 0.04%. The elastic properties were calculated according to the following formulas.^{28–30}

$$L = \rho V_L^2 \tag{2}$$

$$G = \rho V_{\rm s}^2 \tag{3}$$

$$K = L - (4/3)G \tag{4}$$

$$u = (L - 2G)/2(L - G)$$
(5)

$$E = 2G(1 + \mu) \tag{6}$$

$$Z = \rho V_L \tag{7}$$

where V_L is the longitudinal wave velocity, V_S is the shear wave velocity, and *Z* is the acoustic impedance, respectively. The estimated accuracy of the elastic constants and *Z* was about 0.04%, and the estimated accuracy of the μ measurements was about 0.02%.

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RESULTS AND DISCUSSION

FTIR spectra

The bonding of the functional groups to the aromatic ring of pure PS with the $BF_3 \cdot O(C_2H_5)_2$ catalyst is shown in Figure 1. The data collected from the FTIR F2-F5 analysis are given in Figures 2-5. The aromatic-carbonyl group (Ar-CO) in the composition of three MPSs was seen between 1680 and 1700 cm^{-1} , the carbonyl (CO) of the carboxyl group was seen in the vicinity of 1724 cm⁻¹, and the carboxyl of the OH was seen between 3438 and 3450 cm⁻¹. The aromatic-carbonyl-aromatic structure (Ar-CO-Ar) of the PS modified with PhA was observed at 1690 cm⁻¹. In the FTIR spectrum of PS modified with SA, the carbonyl group (CO) near CH₂-CH₂ was observed at 1732 and 1100 cm⁻¹. In the FTIR spectrum of the PS modified with MA, the carbonyl group (CO) near C=C was observed at 1670 cm⁻¹.

ρ and sound velocity

The variations in ρ of PS and MPSs are shown in Table I and Figure 6. The densities ranged between 1035 and 1063 kg/m³ for MPS₅₀₀ and 1037 and 1044 kg/m³ for MPS₃₅₀. These values of the ultrasonic velocity of the pure PSs were close to those in related literature results.^{31,32} The ρ values of PS₅₀₀ and the

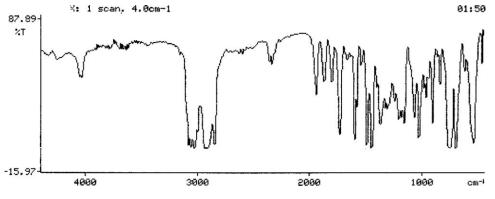


Figure 4 FTIR spectrum of PS modified with MA.

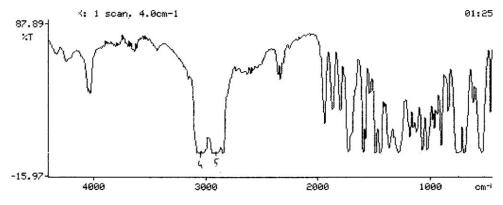


Figure 5 FTIR spectrum of PS modified with PhA.

MPSs obtained from PS₅₀₀ were higher than the ρ 's of PS₃₅₀ and the MPSs synthesized from PS₃₅₀. The chain length and the internal forces between atoms and molecules could have been the reason for these differences. The chain length of a polymer is related to its molecular weight. Thus, because the molecular weight of PS₅₀₀ was bigger than that of PS₃₅₀, the chain lengths of PS₅₀₀ and MPS₅₀₀ were longer. The other important result was the higher ρ obtained for PhAI, which showed that the internal forces were stronger for PhAI.

The variations in V_L and V_S of the SA, MA, and PhA MPSs of different molecular weights are illustrated in Table I, Figures 7 and 8. The V_L values of all of the MPSs were higher than that of the pure PS. The biggest increase for V_L was seen in SAI and PhAII. When pure PS_{500} was modified with SA, the average V_L increased from 2355 to 2414 m/s, and when pure PS₃₅₀ was modified with PhA, the average V_L increased from 2352 to 2419 m/s. As shown in Figure 8, the V_S values of all of the MPSs were higher than that of the pure PS. The biggest increase for V_S was seen in PhAI and PhAII. When pure PS_{500} was modified with PhA, the average V_S increased from 1155 to 1179 m/s, and when pure PS_{350} was modified with PhA, the average V_S increased from 1153 to 1178 m/s.

The velocity of ultrasonic waves in a material is dependent on the composition of the material, the elasticity properties, the compressibility, and ρ . It is

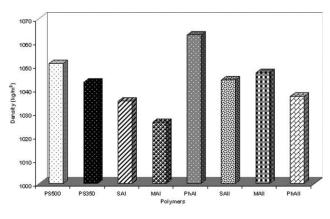
TABLE IVariation of ρ , V_L , and V_S

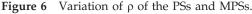
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Polymer	$\rho (kg/m^3)$	$V_L ({ m m/s})$	V_S (m/s)			
PS ₅₀₀ SAI MAI PhAI PS ₃₅₀ SAII	1051 1035 1026 1063 1043 1044	$\begin{array}{c} 2355 \ \pm \ 0.04 \\ 2414 \ \pm \ 0.03 \\ 2385 \ \pm \ 0.03 \\ 2400 \ \pm \ 0.04 \\ 2352 \ \pm \ 0.04 \\ 2403 \ \pm \ 0.03 \end{array}$	$\begin{array}{c} 1155 \pm 0.03 \\ 1175 \pm 0.03 \\ 1175 \pm 0.04 \\ 1179 \pm 0.03 \\ 1153 \pm 0.03 \\ 1158 \pm 0.04 \end{array}$			
MAII PhAII	1047 1037	$\begin{array}{r} 2391 \pm 0.04 \\ 2419 \pm 0.04 \end{array}$	$\begin{array}{c} 1156 \pm 0.03 \\ 1178 \pm 0.04 \end{array}$			

also effected by the microstructure (e.g., porosity, phases, imperfections). The initial increases in V_L and V_S of the pure PS by modification with some organic anhydrides was caused by the structural rearrangement of the PS. In Figure 1 and in the FTIR spectra of the unmodified and modified polystyrenes (MPSs), we observed that the different polyfunctional groups were bound to PS's aromatic ring. So, we can state that that these different groups affected the velocities of the unmodified PS and MPSs.

Elastic parameters

 μ , *L*, *G*, *K*, *E*, and *Z* of the unmodified PSs and the MPSs were calculated with eqs. (2)–(7). The results are given in Table II and Figures 9–11. For MAI, *L* and *K* tended to increase, but *G* and *E* tended to decrease. The results given in Figure 10 indicate that all in the other MPSs, *L*, *K*, *G*, and *E* increased with the chemical modification of the pure PSs. *L* ranged from 5.838 to 6.132 GPa, *G* ranged from 1.375 to 1.479 GPa, *E* ranged from 3.702 to 3.967 GPa, and *K* ranged from 4.004 to 4.162 GPa. The large difference between *L* and *G* arose from the volume effects. The change in the volume due to compressions and expansions involved in longitudinal strains was





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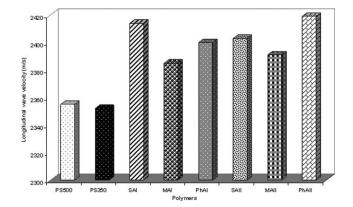


Figure 7 Variation of V_L of the PSs and MPSs.

pronounced, whereas no change in volume was involved in the shear strains. This may have accounted for the increase in ρ and the elastic moduli as well.

 μ was calculated with eq. (5). μ ranged from 0.3411 to 0.3486. μ decreased from 0.3415 to 0.3411 when pure PS₅₀₀ was modified with PhA. However, μ increased from 0.3417 to 0.3443 when pure PS_{350} was modified with PhA. Z was calculated with eq. (7). Z ranged from 2447 to 2554.10^6 kg m⁻² s⁻¹ (Table II). As shown in Figure 11, Z increased from 2475 to 2554.10⁶ kg m⁻² s⁻¹ when pure PS₅₀₀ was modified with PhA. Z increased from 2454 to 2509.10^6 kg m⁻² s⁻¹ when PS₃₅₀ was modified with SA and PhA, too. These results show that the modification of pure PSs with PhA supplied the best effect on the elasticity properties of the pure PSs. All elastic parameters are related to internal forces between the atoms and molecules of materials. Between the monomers of the linear polymers, which create the chains, there are very strong covalent bonds, and these covalent bonds provide the mechanical resistance. There are weak electrostatic gravitational forces (e.g., van der Waals forces, hydrogen bonds) between the molecular chains. These van der Waals forces and hydrogen bonds keep the chains of molecules close to each other. These forces are inversely proportional to the sixth power of the distance between the molecular chains. A smaller distance between the molecular chains

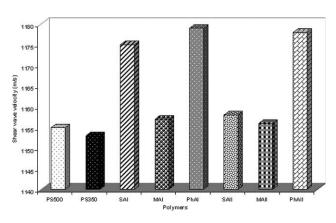


Figure 8 Variation of V_S of the PSs and MPSs.

increases the gravitational force between the molecular chains. $^{\rm 33}$

As shown in Figure 1, when pure PS was modified with SA, the -CO-CH2-CH2-COOH polyfunctional group was bound to the aromatic ring of pure PS. When pure PS was modified with MA, the -CO-CH=CH-COOH polyfunctional group was bound to the aromatic ring of pure PS, and when pure PS was modified with PhA, the -CO-C₆H₄-COOH polyfunctional group was bound to the aromatic ring of pure PS. The main reason for the difference in the values of the elastic parameters of MPSs (SAI, SAII, MAI, MAII, PhAI, and PhAII) was that different polyfunctional groups were bound to the aromatic ring of the pure PSs. The concentration of the functional groups connected to the structure of the PS changed with the molecular weight of PS, and more functional groups connected to low-molecular-weight PS.17 As shown in Table II, the modification of PS₅₀₀ and PS₃₅₀ with MA showed different results for all of the elastic parameters (μ , L, G, K, E, Z). The values of all of the elastic parameters of MAII were higher than those of MAI. The first reason for this was that the molecular weight of PS₃₅₀ was much smaller than the molecular weight of PS_{500} , and the second reason was that the polyfunctional group -CO-CH=CH-COOH had one double covalent bond. A double covalent bond can provide a strong electrostatic gravitational force between polymer chains. So, because more

TABLE II Variation of μ, *L*, *G*, *K*, *E*, and *Z* of the Samples

μ	L (GPa)	G (GPa)	K (GPa)	E (GPa)	$Z (10^6 \text{ kg m}^{-2} \text{ s}^{-1})$	
0.3415 ± 0.02	5.831 ± 0.04	1.403 ± 0.03	3.959 ± 0.05	3.766 ± 0.06	2.475 ± 0.04	
0.3447 ± 0.01	6.037 ± 0.03	1.431 ± 0.04	4.129 ± 0.04	3.848 ± 0.07	2.500 ± 0.05	
0.3459 ± 0.01	5.838 ± 0.04	1.375 ± 0.03	4.004 ± 0.06	3.702 ± 0.06	2.447 ± 0.05	
0.3411 ± 0.02	6.132 ± 0.04	1.479 ± 0.03	4.160 ± 0.05	3.967 ± 0.06	2.554 ± 0.04	
0.3417 ± 0.02	5.772 ± 0.04	1.388 ± 0.03	3.921 ± 0.05	3.725 ± 0.06	2.454 ± 0.04	
0.3486 ± 0.01	6.032 ± 0.03	1.402 ± 0.03	4.162 ± 0.04	3.781 ± 0.06	2.509 ± 0.05	
0.3473 ± 0.02	5.991 ± 0.04	1.401 ± 0.03	4.121 ± 0.05	3.777 ± 0.07	2.505 ± 0.05	
0.3443 ± 0.02	6.070 ± 0.03	1.441 ± 0.04	4.148 ± 0.05	3.876 ± 0.06	2.509 ± 0.04	
	$\begin{array}{c} 0.3447 \pm 0.01 \\ 0.3459 \pm 0.01 \\ 0.3411 \pm 0.02 \\ 0.3417 \pm 0.02 \\ 0.3486 \pm 0.01 \\ 0.3473 \pm 0.02 \end{array}$	$\begin{array}{c c} \mu & L (\text{GPa}) \\ \hline 0.3415 \pm 0.02 & 5.831 \pm 0.04 \\ 0.3447 \pm 0.01 & 6.037 \pm 0.03 \\ 0.3459 \pm 0.01 & 5.838 \pm 0.04 \\ 0.3411 \pm 0.02 & 6.132 \pm 0.04 \\ 0.3417 \pm 0.02 & 5.772 \pm 0.04 \\ 0.3486 \pm 0.01 & 6.032 \pm 0.03 \\ 0.3473 \pm 0.02 & 5.991 \pm 0.04 \\ \hline \end{array}$	$\begin{array}{c c c c c c c c c c c c c c c c c c c $	$\begin{array}{c c c c c c c c c c c c c c c c c c c $	$\begin{array}{c c c c c c c c c c c c c c c c c c c $	

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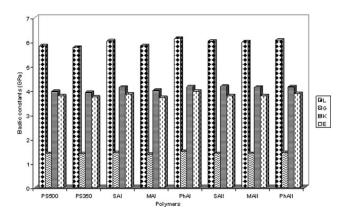


Figure 9 Variation of the elastic constants of the PSs and MPSs.

functional groups that had a double covalent bond were connected to the aromatic ring of pure PS₃₅₀, the values of all of the elastic parameters of MAII were higher than MAI. Although more polyfunctional groups were bounded to the aromatic ring of pure PS₃₅₀ by modification with SA and PhA. The values of the elastic parameters of SAI and PhAI were found to be higher than those of SAII and PhAII (Table II). The reason for this condition was that more polyfunctional groups filled the area between the molecular chains of SAII and PhAII. On the other hand, when pure PSs was modified with SA and PhA, the polyfunctional groups bound to the aromatic ring of pure PSs did not have a double covalent bond (Fig. 1). Therefore, the electrostatic gravitational forces between the molecular chains of SAII and PhAII were not as big as those between the molecular chains of SAI and PhAI.

In summary, the differences in ρ , ultrasonic wave velocity, and the elastic properties were related to the compositions of the samples. The force of attraction in PS is mainly due to short-range van der Waals attractions between neighboring chains and to the covalent bonds between the carbon atoms in the chains. Due to the molecules and long hydrocarbon chains, which consisted of thousands of atoms, the total attractive

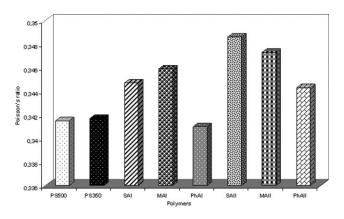


Figure 10 Variation of the μ values of the PSs and MPSs.

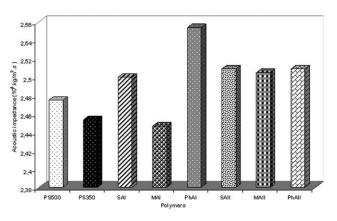


Figure 11 Variation of Z of the PSs and MPSs.

force between the molecules was large. The differences in *L* were mainly due to covalent bonds between the carbon atoms in the chains. However, the differences in *G* were mainly due to the weak Van der Waals forces and hydrogen bonds between neighboring chains.³³ When pure PS was modified with SA, MA, and PhA, different polyfunctional groups were bound to the aromatic ring of pure PS. These polyfunctional groups affected the length of the polymer chains and the relationship between chains, atoms, and molecules. Thus, this condition decided the intramolecular force's effects. With increasing attractive forces between the polymer chains, atoms, and molecules, the mechanical properties of the PSs were improved.

CONCLUSIONS

The effects of binding various polyfunctional groups to PS's aromatic ring by chemical modification on the elastic properties were investigated by an ultrasonic method. Various series of samples were prepared by the chemical modification by SA, MA, and PhA of pure PSs with different molecular weights. The elastic parameters of the samples were nondestructively determined by measurement of the densities and velocities of the ultrasonic longitudinal and shear waves. The sound velocities and elastic modulus were very sensitive to intermolecular interaction. The MPSs had better elastic properties and impact durability in comparison with the pure PSs. The improved elastic properties of the MPSs were explained by the polyfunctional groups that were bound to the aromatic ring of PS.

The ρ values, ultrasonic wave velocities, and elastic properties of the pure PSs and MPSs revealed the following conclusions:

- 1. The ρ of PS modified with PhA (PhAI) was higher than that of the pure PS.
- 2. By chemical modification of the pure PSs with SA or PhA, the sound velocities increased, and all of elastic parameters were improved.

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- 3. By chemical modification of the pure PSs with MA, the sound velocities, *L*, *K*, μ, and *Z* values increased; however, the *G* and *E* values decreased.
- 4. The binding of polyfunctional groups to the aromatic ring of pure PS had positive effects on the elastic properties of the MPSs.
- 5. Pure PSs modified with PhA gave the best mechanical properties.

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