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Study of Surface Mechanical Characteristics of ABS/PC Blends Using Nanoindentation

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Abstract: Acrylonitrile butadiene styrene (ABS) and polycarbonate (PC) are considered a well-known class of engineering thermoplastics due to their efficient use in automotive, 3D printing, and electronics. However, improvement in toughness, processability, and thermal stability is achieved by mixing together ABS and PC. The present study focuses on the understanding of surface mechanical characterization of acrylonitrile butadiene styrene (ABS) and polycarbonate (PC) blends using nano-indentation. Polymer blends sheets with three different proportions of ABS/PC (75:25, 50:50, and 25:75) were fabricated via melt-processing and thermal press. Fourier transform infrared (FTIR) spectroscopy was performed to analyze the intermolecular interactions between the blends' components. To understand the surface mechanical properties of ABS and PC blends, a sufficient number of nano-indentation tests were performed at a constant loading rate to a maximum load of 100 mN. Creeping effects were observed at the end of loading and start of unloading section. Elastic modulus, indentation hardness, and creep values were measured as a function of penetration displacement in the quasi-continuous stiffness mode (QCSM) indentation. Load-displacement curves indicated an increase in the displacement with the increase in ABS contents while a decreasing trend was observed in the hardness and elastic modulus values as the ABS content was increased. We believe this study would provide an effective pathway for developing new polymer blends with enhanced mechanical performance.

Keywords: ABS/PC blend; nano-indentation; modulus; hardness; creep

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1. Introduction

Acrylonitrile butadiene styrene (ABS) is a thermoplastic characterized by its notch insensitivity and low cost with poor flame and chemical resistance, and low thermal stability. Polycarbonate (PC), on the other hand, has high modulus, high toughness, and high impact strength but difficult processability. By mixing together ABS and PC, drawbacks are minimized and useful characteristics such as low temperature toughness, heat resistance and thermal stability, and ease of processing at lower cost are generated. The small addition of ABS to PC helps in improving processability, impact resistance, and cost, while adding a small amount of PC to ABS results in better thermal and mechanical properties [1–3]. Polymer blending has proved itself as an efficient way of developing materials with desired enhanced properties. It is an economical way of producing new materials with a required set of properties. The behavior change of blends can be evaluated with the change in composition of blends. While blending ABS with PC, ABS grafted butadiene chains usually stay insoluble, though they are bonded by styrene-acrylonitrile

side-chains. This imparts remarkable physical properties in the blends [3]. ABS and PC have been blended together commercially for the past few decades and have been used widely in many applications such as electronics, medical equipment, 3D printing, and automotive parts [4–6]. Material design and selection for these applications require the knowledge of surface mechanical properties.

Near-surface properties of polymeric materials are considered to be of vital importance as they govern many aspects of contact performance of surface engineering and tribological applications. It is also very challenging to evaluate these near-surface properties of materials. Nano-indentation is considered as a convenient and powerful method to investigate the surface mechanical properties of materials. It has been extensively used for micro- and nano-sized structures analysis in biomedical materials, nanocomposites, and functionally graded materials, where conventional testing methods are impractical [7–9].

Nano-indentation makes use of contact compliance method. It is based on the measurement of reaction force on an indenter as a function of contact depth. During an experiment, the indenter is penetrated into the test material's surface. Load, indentation depth, and time are monitored continuously during testing. A set of loading and unloading curves are produced for each indentation operation. These load-displacement curves also provide hardness, elastic modulus, and creep curves. Thus, this method does not require the measurement of residual contact area. The force-displacement curves are often based on the work of Oliver and Pharr, which assumes that loading is an elastic-plastic region, while only elastic recovery occurs in the initial portion of unloading curve [8,9]. However, this method does not produce accurate young modulus values, whenever there are viscoelastic effects of materials involved. To overcome this issue, Briscoe et al. suggested to give a long creep period at the peak load before unloading [10].

Nano-indentation has been used widely to determine near-surface properties of materials. V. T. Nayar et al. reported macrocompression and nano-indentation of soft viscoelastic biological materials using a surrogate material agar for soft tissues. They discussed the challenges faced during mechanical behavior assessment, due to time-dependent mechanical response, inherent size, and shape limitations [11]. T. Iqbal et al. used continuous stiffness mode (CSM) nano-indentation to check the response of poly (methyl methacrylate) surfaces. The experimental work determined the load-displacement curves, elastic modulus, hardness, and plasticity index on the polymethyl methacrylate (PMMA) surface [12]. As experimental conditions are critical for the design of engineering components, S. Yasin et al. used nano-indentation to find the response of low-density polyethylene (LDPE) nano-indentation. Effect of frequency, peak load, strain rate, amplitude, and hold time on the mechanical properties (modulus, hardness, and creep) of LDPE was measured [13]. T. Nardi et al. performed nano-indentation of on the surface of polymer nanocomposites. Experimental results were simulated and mechanical properties of functionally graded nanocomposites were successfully found out [14]. Surface mechanical properties of some polymers such as polymethyl methacrylate (PMMA), polyetheretherketone (PEEK), polystyrene (PS), polycarbonate (PC), polypropylene (PP), and ultra-high molecular weight (UHMWPE) were discussed by T. Iqbal et al. using continuous stiffness nano-indentation. Considerable strain-rate hardening effect was observed for these polymers, with PMMA giving maximum hardness values [15]. Experimentation has been conducted to determine surface mechanical properties of viscous materials [16], polycarbonate, and syndiotactic polystyrene [17], polycarbonate/polymethyl methacrylate [18], poly(lactic acid)-based composites [19], α-Al_2O_3 (0001) [20], and so forth.

The present work reports on determination of surface mechanical characteristics (hardness, elastic modulus, creep rate, and creep) of neat ABS, PC and blends of ABS/PC blends through nano-indentation. In the past, work has been done to understand the behavior and mechanical properties of ABS, PC, and their blends [1–4,21–30], but there is limited data available on the surface mechanical characterization of ABS and PC. Therefore, this work mainly focuses on the nano-indentation study of neat ABS, PC, and three different blends of ABS/PC. ABS and PC blends were prepared in the lab and their FTIR analysis

was done. Later, all samples were experimented through nano-indentation to check the load-displacement, indentation hardness, elastic modulus, and creep behavior.

2. Materials and Methods

Materials used in this study were acrylonitrile butadiene styrene (ABS) and polycarbonate (PC), obtained from SABIC Pakistan Pvt. Ltd. Compared to other preparation techniques of polymer-blending, melt-blending is more attractive. It is a simple, cost-effective, environment-friendly technique where no organic solvent is used during preparation, unlike solution blending where extensive use of solvents is required to dissolve the polymers [31,32]. ABS and PC of different mass ratios (g:g) (ABS:PC = 100:0, 75:25, 50:50, 25:75, 0:100) were dried and mixed in internal mixer at about 220 °C. After mixing for about 6 min, material was taken out and dried in oven at about 120 °C to remove any moisture present. After drying, all samples were formed into 1.5 mm thickness sheets using laboratory thermal press of Gibitre Instruments srl, Italy (model: laboratory press, serial number: PRE 2004055), at about 240 °C. Samples were pressed at approximately 210 bar pressure. Schematic representation of the adopted methodology in the present study has been described in Figure 1.

2.1. FTIR Characterization

Chemical structure changes were characterized by FTIR data obtained from Fourier transform infrared spectrometer Bruker (model: Alpha, Germany) in the ATR (attenuated total reflection) reflectance mode. Resolution of 4 cm^{-1} and wave number range of 4000 cm^{-1} to 500 cm^{-1} was set for the spectrum collection. Scanning time of 20 s and 2 mm/s scanning rate was used for the characterization.

2.2. Nano-Indentation Studies

Load-hold-unload experiment was conducted on the surfaces of ABS, PC, and ABS/PC blends using a nano-indenter (Zwick GmbH and Co. KG, Ulm, Germany) and a Berkovich tip. Quasi-continuous stiffness mode (QCSM) module was used to record and measure the force and penetration depth of the indenter in the form of load-displacement curves (also known as compliance curves). Hardness and modulus were calculated using these compliance curves. Oliver and Pharr developed the method to determine these properties [33]. Hardness H, may be defined as:

$$H = \frac{P_{max}}{A_c} \quad (1)$$

where P_{max} is maximum applied load while A_c is projected contact area of indenter tip on testing sample surface. Further, elastic modulus E of the samples was calculated as

$$E = \left(1 - v_s^2\right)\left[\frac{1}{E_r} - \frac{1 - v_i^2}{E_i}\right]^{-1} \quad (2)$$

where E_r is reduced modulus, v_s is Poisson's ratio of the specimen, and v_i is the Poisson's ratio of the indenter [7,9,33,34]. Typical indentation parameters used for all measurements were as follows: number of indents: 36, maximum load for all indents: 100 mN, holding time at maximum load: 20 s [35].

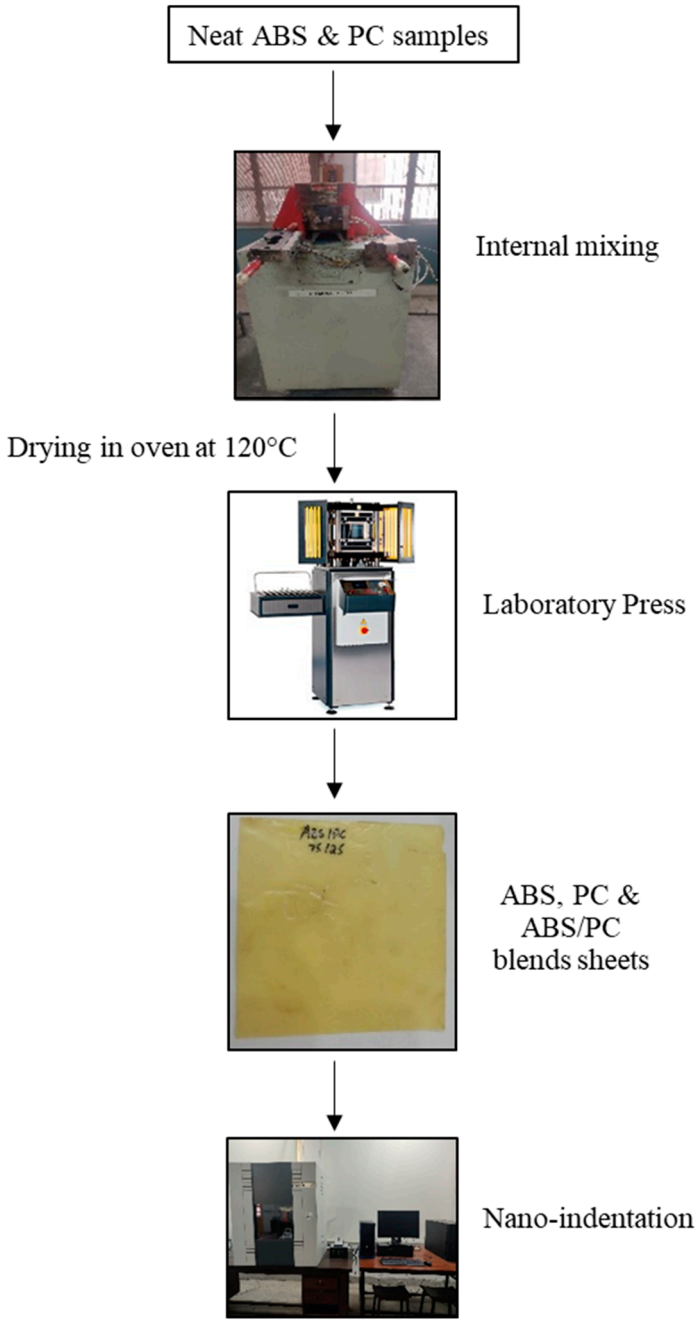


Figure 1. Schematic demonstration of the adopted procedure.

3. Results and Discussion

3.1. FTIR Analysis

FTIR analysis is a useful tool to identify the promising interaction of polymers and presence of specific chemical groups [35]. Figure 2 shows the FTIR absorbance spectrum of neat ABS, 50% ABS/50% PC blend, and neat PC. For neat ABS, vibrations of both aromatic and aliphatic C-H bonds in ABS could be noted in the range of 3200–2800 cm^{-1}. The small peak around 2228 cm^{-1} could be an indication of C≡N stretching. Vibration at 1632 cm^{-1} represented C=C from butadiene unit while stretching vibration of styrene aromatic ring appeared at about 1491 cm^{-1}. For PC, methyl group was detected in the range of 2800 to 3000 cm^{-1}, carbonyl group near 1768 cm^{-1}, and C-O bond was detected in the range of 1300–1100 cm^{-1}. It is evident from the spectrum of ABS/PC 50–50 blend

that peaks at 2963 cm^{-1}, 2230 cm^{-1}, and 1771 cm^{-1} indicate the presence of both ABS and PC in the sample [4,5].

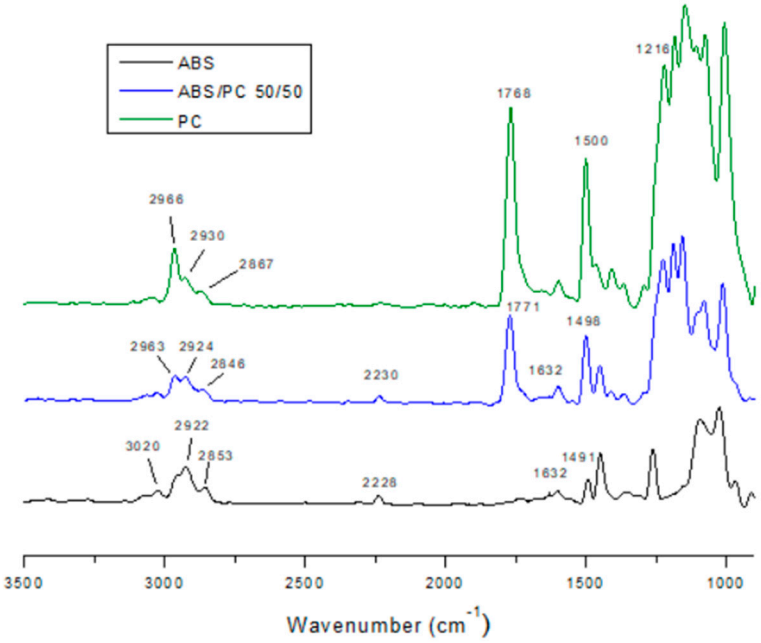


Figure 2. FTIR spectra of acrylonitrile butadiene styrene (ABS), ABS/polycarbonate (PC) 50/50 blend, and PC.

3.2. Nano-Indentation

Typical loading-unloading curves for pure ABS, PC, and the blend samples of various concentrations are presented in Figure 3. Peak load of 100 mN was used to check the maximum contact depth of all samples. The loading section of all five samples started at zero, reached a maximum depth at a peak load of 100 mN, while the unloading section was concluded between 3 and 5 µm. As the figure shows, neat ABS and PC indicates a maximum contact depth of 8.1 µm and 7.2 µm. T. Fang et al. reported depth of approx. 0.12 µm, at a load of 100 µN for PC [36], while T. Iqbal et al. achieved penetration depth of 5 µm at 50 to 60 mN load [15]. This difference may be due to different material sources and sheet preparation conditions. The blends of ABS/PC showed 7.87 µm, 8.96 µm, and 7.3 µm contact depth for 75% ABS/25% PC, 50% ABS/50% PC, and 25% ABS/75% PC mixtures, respectively. These values showed that the penetration depth was increased with the addition of ABS in PC. ABS/PC 50:50 exhibited a maximum depth of 8.96 µm, even higher than pure ABS. It may be due to the presence of cracks or void spaces when PC was mixed with ABS.

Figure 4 presents the indentation hardness of neat ABS, PC, and blends of ABS/PC as a function of indentation depth. At low penetration depths, there were remarkable fluctuations in the hardness values. These fluctuations were thought to be because of tip geometry defects and errors in the determination of surface. All samples showed high hardness values at low penetration depths (0–100 nm). It is evident from the Figure 4 that there is a decreasing trend of hardness values with the increase in contact depth. With the addition of PC, a harder behavior in polymer blends was provoked. ABS and PC showed 0.22 and 0.27 GPa average hardness values. Values of 75% ABS/25% Pc and 25% ABS/75% PC lay between those of neat ABS and PC. Generally, blends represented a harder behavior when the PC content was increased. An abrupt increase in hardness was recorded by R. Krache et al. when PC phase inversion occurred from dispersed to continuous phase [3]. T. Iqbal et al. used nano-indentation to determine surface mechanical properties of PC and reported hardness of 0.2 ± 0.02 GPa [15]. Interestingly, the blend sample 50% ABS/50% PC exhibited a significantly lower hardness value of 0.16 GPa compared to the values of

0.22 and 0.27 GPa for neat ABS and PC, respectively. This difference may have risen due to some surface effects during sample preparation or environmental effect [37].

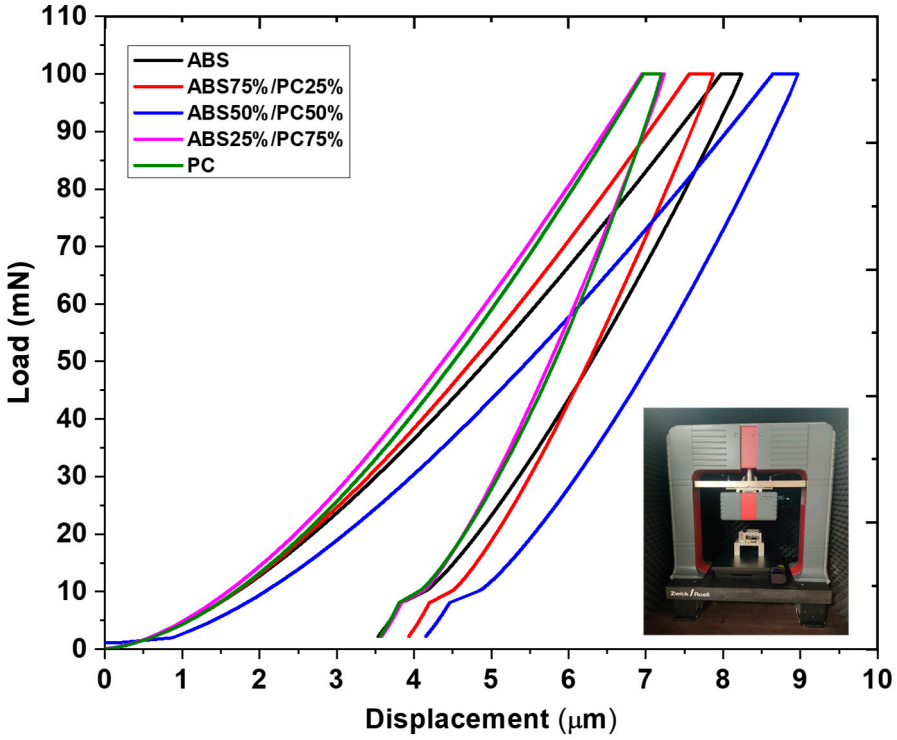


Figure 3. Load-displacement curves for five different samples of ABS, PC, and ABS/PC blends.

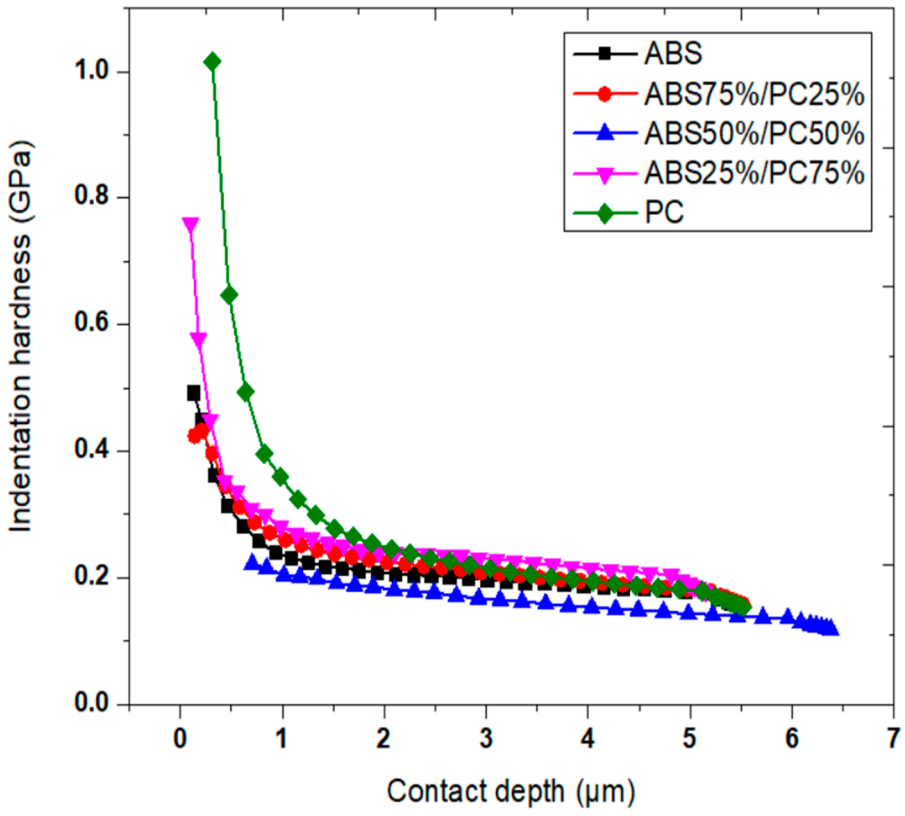


Figure 4. Hardness of ABS/PC samples as a function of contact depth.

Elastic modulus of all ABS/PC samples at different penetration depths has been presented in Figure 5. A decreasing trend in the elastic modulus values was observed with the increase in contact depth. This decrease may be explained as geometrical dislocations, which give rise to the strengthening mechanism [36]. It was also detected from Figures 4 and 5 that large variation in hardness and modulus values, up to 3 µm of contact depth, occurred during experimentation. This variation may have been caused due to uneven surface of the sheets or there may be certain surface changes involved due to environmental effects. Surface determination in nano-indentation is also quite important and poor determination causes fluctuations in the hardness and modulus values. At a penetration depth of 3 µm, ABS showed 1.28 GPa, compared to the value of 1.7 GPa for neat PC. A. Jee et al. measured modulus of PC as 2.27 Gpa using Oliver and Pharr method and 1.98 GPa using atomic force microscope (AFM) technique [38]. Other values noted from Figure 5 were 1.47 GPa, 1.23 GPa, and 1.66 GPa for 75% ABS/25% PC, 50% ABS/50% PC, and 25% ABS/75% PC blends, respectively. With the increase in PC content, the modulus of ABS/PC blends gradually increased as reported by R. Krache et al. [3]. Conversely, 50% ABS/50% PC showed little deviation from the trend values, as its value appeared to be less than the value of neat ABS. This deviation may be attributed due to the same reasons as explained earlier in the case of load-displacement curves and indentation hardness values.

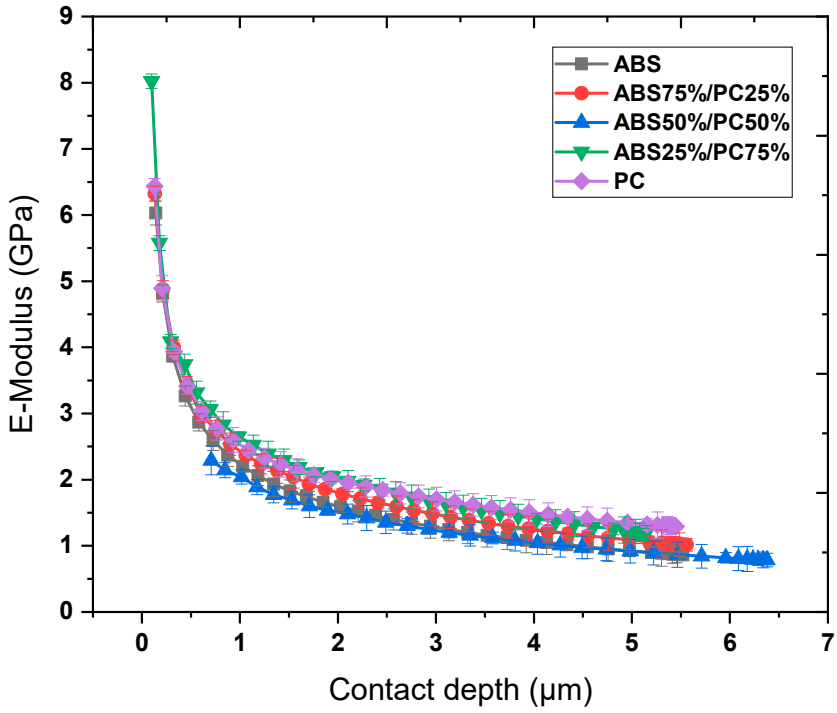


Figure 5. Modulus of neat ABS, PC, and blends of ABS/PC.

Creep is the tendency of a material to distort under constant load. During recording of loading and unloading data, there may be a region where creep appeared at the end of the loading and start of unloading sections. As the unloading section starts, penetration depth increases slightly, even though the imposed load was decreased continuously [8]. Here, creeping effect was readily detected at the correspondence of loading-unloading curves, at peak point of the load-displacement curves. Figure 6 represents the creep rate of ABS, PC, and ABS/PC blends at a constant load of 100 mN. Creep rate showed a decreasing trend with the rise in time. ABS creep rate changed from 16.7 to 10.9 nm/s while ABS 75%/PC 25% value decreased from 15.3 to 10 nm/s. Creep rates of the blends were lower than the value of ABS due to an increase in the amount of PC.

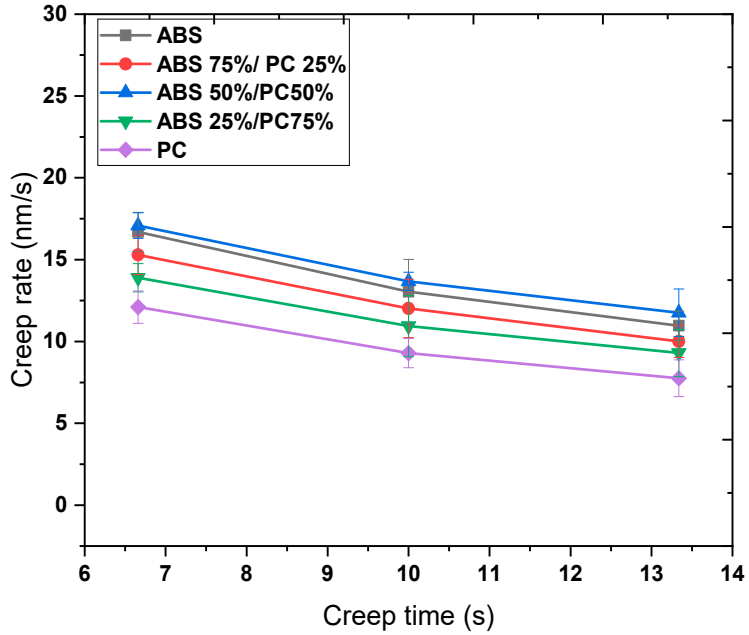


Figure 6. Creep rate vs. time for ABS, PC, and ABS/PC blends.

Neat PC, on the other hand, showed a lower value and its rate changed from 12.1 to 7.8 nm/s. ABS 50%/PC 50% blend showed a maximum value of creep rate at the same conditions, which is not following the trend of all other samples. This difference may have risen due to improper mixing of the blend contents. Overall, a decrease of 8.4% and 16.7% was observed in the case of ABS 75%/PC 25% and ABS 25%/PC 75%, respectively, with the addition of PC in ABS. Figure 7 shows the creep time plotted as a function of depth change. It represented the creep time effect on the depth change of ABS, ABS/PC blends, and PC at a peak load of 100 mN. The depth of ABS varied from 106.6 to 339.5 nm while PC depth changed from 79.4 to 232.8 nm. Blends of ABS/PC showed mixed trends; depth was decreased as the PC content was increased. Although 50% ABS/50% PC trend was different from other blends, it showed highest depth achieved at 348.9 nm. This difference may be due non-uniform mixing and presence of voids in the sheet.

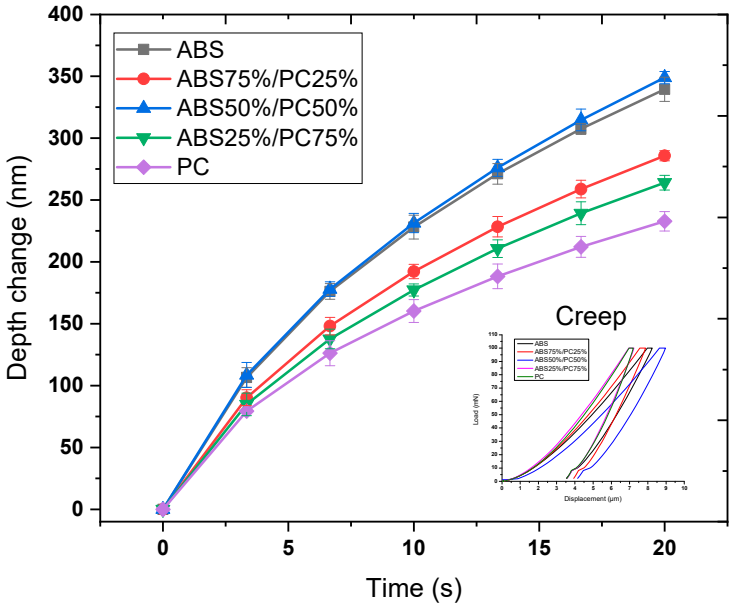


Figure 7. Creep time vs. depth change of neat ABS, PC, and blends of ABS/PC.

4. Conclusions

In this work, an experimental study was conducted to study the nano surface mechanical properties of neat ABS, PC, and ABS/PC blends. Five different sample sheets (ABS, 75% ABS/25% PC, 50% ABS/50% PC, 25% ABS/75% PC, and PC) were prepared by melt-processing followed by hot-pressing. FTIR technique was performed to analyze the chemical structure of all the samples. Blending of the samples was physical and there was no decomposition of polymers during the blending stage. At maximum load of 100 mN and 20 s hold time, ABS showed penetration depth of 8.1 µm and PC indicated 7.2 µm. Penetration depth was increased with the increase in ABS content. Elastic modulus and indentation hardness values were also noted. With the increase in penetration depth, hardness and elastic modulus of all samples was decreased with the increasing content of ABS. As the PC content was increased, general increase in indentation hardness and elastic modulus was observed. On the other hand, a decrease in creep rate and creep was observed with the increase in PC content, although 50% ABS/50% PC trends were different from other samples' results. These uncertainties were thought to be due to preparation issues, environments effects, presence of voids and cracks, and surface determination errors.

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